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10-13 September 2023

22nd EUROPEAN CONFERENCE ON THERMOPHYSICAL PROPERTIES

Book of Abstracts

CONTENTS

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ABOUT THE CONFERENCE

The European Conference on Thermophysical Properties is held in a three-year cycle that includes the Asian Thermophysical Properties Conference and the American Symposium on Thermophysical Properties. All the European conferences are listed in the table below:

1	ECTP	1986	Nov. 11-13	Baden-Baden, Germany	E. Fitzer
2	ECTP	1970	Apr. 7-10	Salford, UK	A. Stuckes
3	ECTP	1972	Jun. 20-23	Turin, Italy	G. Ruffino
4	ECTP	1974	Sep. 4-6	Orleans, France	F. Cabannes
5	ECTP	1976	May 18-21	Moscow, USSR	A.E. Sheindlin
6	ECTP	1978	May 18-21	Dubrovnik, Yugoslavia	K. Maglic
7	ECTP	1980	Jul. 26-30	Antwerpen, Belgium	R. de Coninck
8	ECTP	1982	Jun. 30-Jul. 4	Baden-Baden, Germany	H.-E. Schmidt
9	ECTP	1984	Sep. 27-Oct. 1	Manchester, UK	R. Taylor
10	ECTP	1986	Sep. 17-21	Rome, Italy	G. Ruffino
11	ECTP	1988	Sep. 22-26	Umea, Sweden	G. Backstrom
12	ECTP	1990	Jun. 13-16	Vienna, Austria	W. Neumann
13	ECTP	1993	Sep. 24-28	Lisbon, Portugal	C.A. Nieto de Castro
14	ECTP	1996	Aug. 30-Sep. 3	Lyon, France	J.F. Sacadura
15	ECTP	1999	Sep. 16-19	Wurzburg, Germany	J. Fricke
16	ECTP	2002	Sep. 5-9	London, UK	W.A.Wakeham
17	ECTP	2005	Sep. 1-4	Bratislava, Slovakia	L. Kubicar
18	ECTP	2008	Sep. 5-8	Pau, France	J.-L. Daridon
19	ECTP	2011	Aug. 31-Sep. 4	Thessaloniki, Greece	M.J. Assael
20	ECTP	2014	Aug. 28-Sep. 1	Porto, Portugal	L.Santos, J. Coutinho
21	ECTP	2017	Sep. 3-8	Graz, Austria	G. Pottlacher
22	ECTP	2023	Sep. 10-13	Venice, Italy	A. Muscio

For more information, please visit the following web page:

https://www.thermophysicalpropertiesconferences.com/

This 22nd edition of the ECTP is organized by the Associazione Italiana Proprietà Termofisiche (AIPT—Italian Society for Thermophysical Properties) in cooperation with the University of Modena and Reggio Emilia (DIEF/EELab), the National Research Council of Italy (CNR-ITC) and the IUAV University of Venice.

The objective of the conference is to provide a forum for academic and industrial researchers to meet and share experiences in the field of thermophysical properties of a wide variety of systems, covering both fluids and solids. The conference will concentrate on theory, modeling, measurement and applications on the following topics:

Properties

Thermal conductivity, thermal diffusivity, viscosity and non-Newtonian properties, mass-diffusion, optical and radiative properties including emissivity, reflectivity and absorptivity, solubility, phase equilibrium including liquid-solid, calorimetric and volumetric properties, speed of sound, interfacial properties including solid-solid and wettability.

Materials

Metals and alloys, ceramics, glasses, composites, multi-functional materials, superconductors, insulation materials, solar-reflective materials and thermal shields, porous materials, granular and thin-film materials, foams, gels, emulsions, soft materials, nano-materials, near critical and super critical fluids, polymers, food and biomaterials, environmentally friendly fluids, aqueous systems, petroleum fluids, ionic liquids, molten salts.

Methods

Measurement techniques (including methodologies for data evaluation and prediction), engineering applications (polymerization, casting, sintering, plasma spraying, distillation, refrigerant techniques, thermoelectric cooling, insulation structures in civil engineering).

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CONFERENCE INFORMATION

Conference venue

IUAV University of Venice – Cotonificio Veneziano, *Dorsoduro, 2196, 30123 Venezia VE.*

The building was inaugurated in 1883.

The previous year a company for cotton spinning in Venice had been established.

Partially destroyed by fire in 1916, it was rebuilt.

The factory employed about a thousand workers. It remained in operation until 1960 and was then abandoned for thirty years before being restored in the 1990s to a project by Gino Valle Studio.

Today, the Cotonificio is one of the busiest university buildings and home to Archivio Progetti, ArTec archive on techniques and materials for architecture and industrial design, and a number of classrooms.

A selection of models from Archivio Progetti is displayed in the "Gino Valle" exhibit space, which is also venue for temporary exhibits.

Cotonificio

reception office 041 2571801/2

Accessible route available at the following link: https://www.iuav.it/Ateneo1/Sedi/Sedi-venez/cotonifici/mappe/V1-Itinerario-verso-sede-Cotonificio-Santa-Marta.pdf

GUIDELINES FOR ORAL LECTURES AND POSTER CONTRIBUTIONS

Oral lectures

- Plenary: 40 minutes + 10 minutes discussion.
- Keynote: 25 minutes + 5 minutes discussion.
- Regular: 15 minutes + 5 minutes discussion.

The time for oral contributions will be strictly limited as specified above! Chairpersons are encouraged to be seated no later than 5 minutes before the start of their session and to ensure that all presentations start and finish on time as scheduled, in order to allow participants to follow presentations in different parallel sessions.

Please note that all speakers are responsible for using only slides for which they own the copyright. Note also that other participants might take pictures of the presentations.

Poster sessions

Posters must be mounted at the beginning of the day in which their specific poster session is scheduled and taken off at the end of that day. All the posters will be displayed in the building that houses the Conference venue. Participants will be encouraged to look at the posters during the poster sessions, which will be held immediately after lunch each day. Materials to fix posters like adhesive tape or pins will be provided by the organizers.

The poster format has to be A1 portrait (594 mm wide x 841 mm height).

PLENARY LECTURERS

Plenary Lecture 1

The role of light from Galileo's Starry Messenger to modern measurement in infrared astronomy

Prof. Alessandro De Angelis

Graduated from the University of Padua, Alessandro De Angelis is professor in Experimental Physics in Padua and at IST Lisboa. His research interests range from general physics to high energy astrophysics. After seven years spent at CERN, Geneva, in the 1990s, he has conceived and implemented with NASA and the Max-Planck Institute in Munich essential experiments for the detection of cosmic photons of high energy. Since 2022 he his scientific counsellor for the permanent delegation of Italy to the International Organizations, and represents Italy in the Bureau International de Poids et Mesures in Paris. De Angelis has published numerous scientific articles and textbooks on Astroparticle Physics, and three popular books on Galilei and the history of science in the Renaissance. He is a member of the Accademia Galileiana.

The role of light from Galileo's Starry Messenger to modern measurement in infrared astronomy

Alessandro De Angelis

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"In 1610 Galileo published in Venezia his book Sidereus Nuncius, the first astronomy book based on observations with an astronomical instrument: the telescope. Visible light was the messenger from the stars. Since the 1930s, the spectrum of frequencies (and therefore energies) of the light waves we are capable of observing has extended: this is multiwavelength astronomy/astrophysics. New colors invisible to the human eye allow us to see previously unknown phenomena. Think about how different your hand is when you see it through an infrared camera, with your eyes, or in a X-ray scan: something similar happens with astrophysical objects.".

Plenary Lecture 2

Thermophysical Properties for Sustainable Goals Development – Contributing to a New Research Approach

Prof. Carlos A. Nieto De Castro

Carlos A. Nieto de Castro was born in Portugal in 1949. Full Professor at the Faculty of Sciences of the University of Lisbon since 1980, jubilated in 2019. Studied Chemical Engineering, and obtained his PhD in Engineering Sciences (Chemical Thermodynamics) from Instituto Superior Técnico, Portugal (1977). Received the Portuguese Stimulus to Excellence in Research Award, FCT-MCTES, Portugal (2005), the University of Lisbon Medal for distinguished service and has about 400 scientific publications including 38 books and chapters, one European patent and 550 conference presentations.

He directed over 40 national and international funded projects, part of them involving industrial companies. Member of several international scientific boards and learning societies, he is an international evaluator under European, National, and International programs, associate editor of the Journal of Chemical and Engineering Data (ACS), editorial board member of several journals, IUPAC and International Association for Advanced Materials fellow.

Prof. Carlos Nieto de Castro is one of world's most cited top scientists in Chemical Engineering/Physical Chemistry (top 2%), Stanford University Ranking, US, 2020, 2021, 2022. With more than 45 years dedicated to the research in thermodynamics and transport processes of fluids and materials, his actual scientific activity covers the field of molecular thermophysics and fluid technology, ionic liquids, molten salts, nanofluids, ionanofluids and nanosystems, including new heat and storage fluids with industrial impact in the area of energy and environment, and the use of ionic liquids as solvating and reaction media to synthesize and functionalize nanomaterials, for industrial and domestic applications.

Thermophysical Properties for Sustainable Goals Development Contributing to a New Research Approach

Carlos A. Nieto de Castro
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Thermophysics is the science and technology of the most important properties of materials. The thermophysical properties of fluids and materials play an important role in several processes in the chemical, extraction and manufacturing industries, especially in those involving simultaneous heat and mass transfer. Most of the problems that affect our society need values of these properties to design and characterize new products and processes, to replace unacceptable processes and compounds and to optimize energy balances and efficiency.

Under the United Nations Sustainable Development Goals tasks, research in this area requires innovative approaches that can respond for affordable and clean energy, industry (including fishing industries), innovation and infrastructure, sustainable cities and communities, and climate action, among others possible. In the recent years most of our group research was directed to cutting-edge problems, which include ionic melts (ionic liquids, molten salts), molten metals, nanomaterials, IoNanofluids and new engineering fluids. The environmental and safety requirements on the use of nanomaterials and nanosystems will also be analyzed on the basis of a recent proposed strategy, Lourenço et al., (2021) [1].

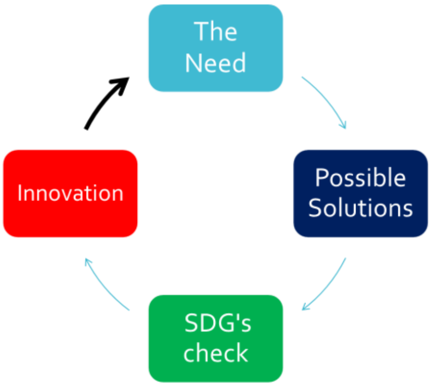

Fig. 1. Thermophysical Properties Sustainable Development Goals Approach (TPSGDA), a scheme

This talk will show how the studies performed in the last years, linking scientific knowledge and technological developments, are contributing to the resolution of some problems that affect our society. Recommendations for a new research strategy on thermophysical properties (TPSGDA) in the area of thermophysics will be discussed.

Acknowledgment

This study was supported by Fundação para a Ciência e a Tecnologia, IP, through projects UIDB/00100/2020 and UIDP/00100/2020 of Centro de Química Estrutural (CQE). Institute of Molecular Sciences is an Associate Laboratory funded by FCT through project LA/P/0056/2020.

Significant references

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Plenary Lecture 3

Investigating the liquid phase of high melting metals and alloys - A review of selected Pulse Heating Experiments

Prof. Gernot Pottlacher

Gernot Pottlacher, born 1956, studied physics at the University of Technology in Graz (TUG), Austria and is Dipl.-Ing. Dr. Techn. He was a professor at the Institute for Experimental Physics at TUG. His main field of activity are investigations of thermophysical properties of liquid metals and alloys with various techniques such as subsecond pulse heating, DSC (Differential Scanning Calorimetry), and four-point probes.

Further he gave courses in basic physics, for lighting engineering, for temperature measurements, as well as physics didactic courses for teachers in training. One of his hobbies is performing public experimental demonstrations-shows. In his retirement he leads the museum echophysics in PöLlau.

More details for Gernot Pottlacher regarding lectures, seminars and tutorials held at TU-Graz, area of expertise, performed projects, publications and supervised diploma and master theses please find in TUG – Online: https://online.tu-graz.ac.at/tug_online/webnav.ini, person: Pottlacher

Investigating the liquid phase of high melting metals and alloys - a review of selected Pulse Heating Experiments

Gernot Pottlacher

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During the last 40 years an increasing demand for thermophysical data of liquid metals and alloys has been noticed. Properties of matter at very high temperatures are useful for high-temperature technologies such as used in aerospace (e. g. simulation of the melting of re-entering space debris), for nuclear energy, for the establishment of temperature reference points above the freezing point of gold, for modelling of various casting processes, electro arc refinemet processes and metal powder production in steel industry, as well as for laser powder 3d printing and finally to obtain phase diagrams up to the critical point.

Starting 40 years ago with a very fast ohmic pulse heating system the Thermophysics and Metalphysics Group at TU-Graz continuously improved their experimental equipment to extend the number of thermophysical properties to study and to improve the accuracy of the data to be measured.

At that time in the eigthies many different groups all over the wold were using ohmic pulse heating techniques to obtain thermophysical data of metals and alloys close to melting in the solid state and in the liquid state. The first part of this talk will be to give a reviev of pulse heating experiments existing in the eighties and the according persons operating them.

The second part of this talk will deal how the following thermopysical properties as function of temperature for solid and liquid metals and alloys have been measured at TU Graz: Specific enthalpy, H, heat of fusion, ΔH, isobaric/isochoric heat capacity, cp/cv, density, d, electrical resistivity, ρ, thermal conductivity, λE, thermal diffusivity, a, phase transition temperatures, melting temperature (for pure metals), Tm, solidus/liquidus temperature (alloys), TS/TL, normal spectral emissivity, ελ, critical pressure, pc, critical volume, Vc, critical temperature, Tc, equation-of-state (EOS) parameters, viscosity, η, surface tension, γ. The data obtained have been reported in about 150 scientific papers.

Due to my retirement in the year 2021 the ohmic pulse heating system situated at TU-Graz has been transferred to VINCA INSTITUTE OF NUCLEAR SCIENCES, NATIONAL INSTITUTE OF THE REPUBLIC OF SERBIA, UNIVERSITY OF BELGRADE, 11001 Belgrade, P. O. Box 522 and will be operated in the future by Dr. Nenad M Milosevic.

ORAL CONTRIBUTIONS

OS1-1 L1A – Monday, 11 Sep. 11.40-12.00 – ROOM O2

Solubility of CO in water and NaCl (aq) at high pressures

Qiaoyun Chen[1], J. P. Martin Trusler[1]*

[1] Department of Chemical Engineering, Imperial College London, London (UK)
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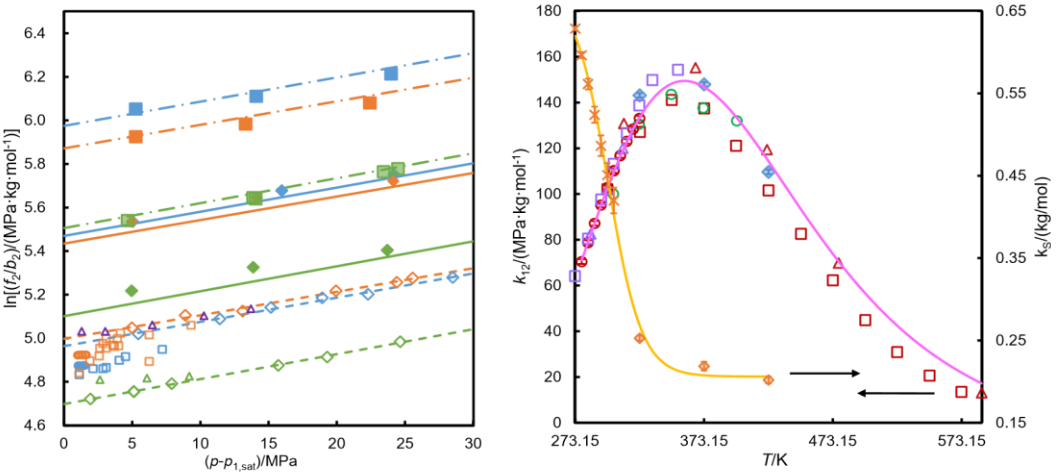
CO is one of the impurities maybe found in a CO_2 stream destined for geological carbon storage (GCS). The solubility of CO in formation brines is an important parameter in the design of a GCS project. In this study, we report the solubility of CO in water and NaCl(aq) solutions at pressures from 2 to 28 MPa and at temperatures from 323.15 to 423.15 K. This study provides the first data for CO solubility in brine with high molality of salt under GCS conditions.
The measurements were conducted by a static synthetic method [1] using a high-pressure view cell. Initially, the view cell was filled with a known amount of gas, after which water or brine was injected under stirring until all the gas dissolved. The bubble pressures were determined by a combination of graphical analysis of pressurevolume data and visual observation. The relative uncertainty of the CO solubility at a given temperature and pressure is estimated to be 4%. The new results from this study were used to develop a simple model to predict CO solubility in water and NaCl brines as a function of temperature, pressure and salt molality up to 4 mol/kg. This model is an extended Krichevsky-Kasarnovsky (KK) equation [2] given by:

$$\ln(f_2/b_2) = \ln(k_{12}) + k\,b_{ss} + (v_2/RT)(p - p_{1,\mathrm{sat}}). \qquad (1)$$

Here, f_2 is the fugacity of CO in the gas phase, b_2 is the molality of CO in the aqueous phase, k_{12} is Henry's constant, k_s is the Sechenov coefficient, b_s is the molality of salt, v_2 is the partial molar volume of CO in aqueous solution, p is pressure and $p_{1,\mathrm{sat}}$ is the solvent vapour pressure. Three parameters were regressed for each isotherm: k_{12}, v_2 and k_s. Fig. 1 (a) shows the experimental results from this study in comparison with both the KK model and literature data, while Fig. 1 (b) shows the k_{12}, and k_s as a function of temperature.



(a) (b)

Fig. 1. (a) Plot of ln (f_2/b_2) against (p-$p_{1,\mathrm{sat}}$). CO in water: △, ○ and □, literature data; ◇, this work; - - -, model. CO in 2 mol/kg NaCl (aq) solution: ◆, this work; ——, model. CO in 4 mol/kg NaCl (aq) solution: ■,this work; — · —, model. Colour codes for temperature: blue, T = 323.15 K; purple, T = 366.48 K; orange, T = 373.15 K; green, T = 423.15 K. (b) Plot of Henry's constant k_{12} and Sechenov coefficient k_S as a function of temperature T. △, ○, □ and ×, literature data; ◆, this work; ——, model.

Significant references

1. Torín-Ollarves, G.A. and J. P. M. Trusler, *Fluid Phase Equilib.*, vol. 539 (2021), pp. 113025.
2. Krichevsky, I.R. and J.S. Kasarnovsky, *J. Am. Chem. Soc.*, vol. 57 (1935), pp. 2168-2171.

OS1-1 L1B – Monday, 11 Sep. 11.40-12.00 – ROOM O1

Measurements of density, surface tension and viscosity of liquid steels and superalloys at high temperatures: influence on welding pool topology

J. Delacroix[1*], S. Gounand[2], S. Pascal[2], D. Borel[3], J. Delmas[3], P.-O. Barrioz[4], A. Brosse[4]

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[3] EDF R&D, Département PRISME, 6 quai Watier, 78400 Chatou, France
[4] FRAMATOME-DTIM, 2 Rue Professeur Jean Bernard, 69007 Lyon, France
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Within the framework of the welding of steel components in pressurized water reactors, the optimization of the welding pool penetration is a major issue, both from safety and economical viewpoints. Among other phenomena, the thermalhydraulics of the molten steel pool may be driven by Marangoni surface flows at the top of the welding bath, which can in turn influence bulk phenomena by viscous shearing. To compute these flows, such fundamental properties as density ρ, surface tension γ and viscosity η as functions of the temperature, are required. These properties at liquid state are scarce for austenitic steels and missing for both ferritic steels and superalloys. To our knowledge, there are no available experimental data above 1850°C, due to technological issues.

The VITI test facility has been designed to implement various techniques for the measurements of steel properties, such as Sessile Droplet (SD), Maximum Bubble Pressure (MBP) and Torsional Pendulum Viscometer (TPV) techniques. These techniques allowed us to perform original experimental measurements and temperature correlations from the melting point up to 2500°C, completing the data previously obtained at lower temperatures [1]. In particular, concerning surface tension, an experimental evidence of Sahoo [2] model is provided, emphasizing the decisive role played by sulfur and oxygen concentration on surface tension. The experimental uncertainties are presented together with a post-test chemical analyses for discussing the influence of sample composition.

In parallel, the measurements obtained on the VITI facility have been used as input data in dedicated modelling tools. Indeed, these data made it possible to carry out a benchmark and a sensitivity study involving the CEA, EDF and FRAMATOME calculation codes which are Cast3M, code_Saturne and COMSOL Multiphysics respectively. In the work presented in this paper (welding line without filler metal based on Gas Tungsten Arc Welding technique), the results showed on the one hand a good agreement between the three predictions, and on the other hand that the numerical simulations are extremely sensitive to surface tension, which implies knowing this parameter precisely to enhance the predictability of the models.

Acknowledgment

This work has been carried out thanks to the subsidized project for improving numerical simulation of welding operations in nuclear industry, jointly funded by CEA, EDF and FRAMATOME within a tripartite agreement.

Significant references

1. Delacroix, J., Piluso, P., Chikhi, N., Asserin, O., Borel, D., Brosse, A. and Cadiou, S. (2022), Measurements of Liquid AISI 304L Steel Density and Surface Tension, and Influence of Surface-Active Elements at High Temperatures. steel research int., 93: 2100624.
2. P. Sahoo, T. Debroy and M. J. McNallan, "Surface tension of binary metal-surface active solute systems under conditions relevant to welding metallurgy," Metall. Mater. Trans. B, vol. 19, pp. 483-491, 1988.

OS1-1 L1C – Monday, 11 Sep. 11.40-12.00 – ROOM N2

SO_2 Solubility in Polyethylene Glycol Dimethyl Ether (PEGDME) and its Correlation by using Macro ASOG Model

Tomoya Tsuji[*,1,2], **Ryosuke Wakasa**[3], **Andrea J. X. Lai**[1], **Hiroaki Matsukawa**[3], **Taka-aki Hoshina**[4,1], **Hiroyuki Matsuda**[2], **Katsumi Tochigi**[2], **Katsuto Otake**[3]

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[2]College of Science and Technology, Nihon University, Tokyo (Japan)
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[4]College of Industrial Technology, Nihon University, Narashino (Japan)
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Removal of sulfur compounds is compulsory for natural gas processing, exhaust gas treatment and so on [1]. Noles and Zollweg reported vapor-liquid equilibrium (VLE) for SO_2 - dimethyl ether (DME) at 283.15 – 425.9 K, and found the concave VLE with the azeotropic at 283.15, 323.59 and 363.15 K [2]. Fig.1 (a) shows the VLE at 283.15 and 323.59 K. The VLE suggests a bridge-like interaction between SO_2 and DME. In this research, SO_2 solubilities were measured in low molecular weight polyethylene glycol dimethyl ether (PEGDME), with the average molecular weight of 240, by using a static apparatus at 298.15 K. The PEGDME is composed of 4 or 5 repeating units of DME. Therefore, PEGDME was expected to have strong interactions with SO_2. Fig.1 (b) shows SO_2 (1) solubility in PEGDME (2) at 298.15 K. The experimental solubilities were far lower than those from Raoult law. The result suggested that a strong interaction is maintained among SO_2 molecule and repeating units of PEGDME. The data, shown in Fig. 1 (a), were correlated with an activity coefficient model similar to ASOG [3]. In the original ASOG [2], three groups, $-CH_2-$, -O- and SO_2, should be considered for these systems. In this model, two groups, $-CH_2OCH_2-$ and SO_2, were just considered, and their group interaction parameters were determined by the data shown in Fig. 1 (a). The modification was newly named as 'macro ASOG' in this research. Fig 1 (b) shows the estimation of the SO_2 (1) solubilities in PEGDME (2), where the effectiveness was set to 92.2% for number of atoms (other than hydrogen atoms) in the group $-CH_2OCH_2-$ of PEGDME. The detail measurements and calculation will be described in the conference.

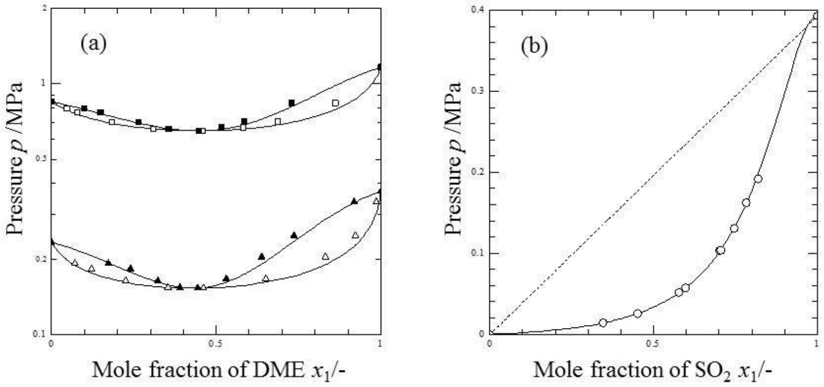

Fig. 1. (a) VLE for DME(1)-SO_2(2) **and (b)** SO_2 (1) solubility in PEGDME (2); (▲, △): Noles and Zollweg [2], 283.15 K; (■, □): Noles and Zollweg [2], 323.59 K; (○): This work, 298.15 K; (—): macro ASOG.

Acknowledgment

This research is one of the activities in the Grant for Joint Research Project 2021, which promoted by Japan International Cooperation Agency (JICA).

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2. J. R. Noles and J. A. Zollweg, *Fluid Phase Equilib.*, vol. 66(1991), pp. 275-289.
3. A. J. X. Lai, T. Tsuji, K. Tochigi, L. S. Tan, H. Matsuda and K. Kurihara, *J. Adv. Res. Fluid Mech. Therm. Sci.*, vol. 102 (2023), pp. 66-80.

OS1-1 L1D – Monday, 11 Sep. 11.40-12.00 – ROOM N1

Effect of interfacial reactions on contact angles of CaO-MgO-Al_2O_3-SiO_2 melts and thermal barrier coatings

M. Hayashi1*, K. Tsukamoto1, R. Endo2, T. Watanabe1 and M. Susa1
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Dept. of Materials Science and Engineering, Shibaura Institute of Technology, Tokyo (Japan)
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The surface of Ni-based superalloy turbine blades for jet engines is coated with a thermal barrier coating (TBC) to improve heat resistance. Yttria-stabilized zirconia (YSZ) coated by EB-PVD is often used as TBC. The YSZ is subjected to the CaO-MgO-Al_2O_3-SiO_2 oxides (CMAS), which are the main constituents of volcanic ash, and the molten CMAS spreads wetting and penetrating into the YSZ thin film, causing the YSZ to detach from the base material [1]. Thus, it is important to investigate the effect of interfacial reactions on the wettability of CMAS and YSZ. Although there have been reports on the interfacial reaction [2.3] and on the wettability [4.5], respectively, between YSZ and CMAS, to the best of our knowledge, there have been no reports with respect to the relation between wettability and interfacial reaction. The aim of this study is to investigate the effect of interfacial reactions on the contact angle by fabricating a suspension-type sessile drop method measurement system, determining the time change of the contact angle, and analyzing the microstructure of the reacting interface.

The substrates used were 18.8 mol% $YO_{1.5}$-ZrO_2 sintered (polycrystalline) plate (17YSZ), 23 mol% $YO_{1.5}$-ZrO_2 single crystal plate (surface orientation {100}) (13YSZ), and Al_2O_3 sintered plate. 20 mg of CMAS melt was attached to a 0.15 mm diameter R-type thermocouple wire and cooled in air to vitrification. Fig. 1 shows the contact angle measurement system by the sessile drop method. After confirming that the experimental system including the sample reached thermal equilibrium at 1573±5K, the CMAS melt was attached to the substrate. Then, the images were taken for 30 min using a backlight optical system with a He-Ne laser. The contact angle between the YSZ or Al_2O_3 substrate and the CMAS melt was calculated from the obtained images by image analysis using the $\theta/2$ method.

Fig. 2 shows the time change of the contact angle between the substrate and the CMAS melt. It can be seen that the contact angle decreases even after 600 s for the YSZ substrate, while, the contact angle is almost constant after 600 s for the Al_2O_3 substrate. For the Al_2O_3 substrate, anorthite and spinel were formed at the interface with the CMAS melt, suggesting that anorthite layer with a high melting point has prevented contact between the melt and the substrate preventing interdiffusion. [4,5] On the other hand, at the interface between YSZ and CMAS, Y^{3+} and Zr^{4+} were replaced by Ca^{2+} and Mg^{2+}, and ZrO_2 once dissolved to supersaturation in the CMAS melt was reprecipitated. This interfacial reaction is considered to have caused the decrease in contact angle.

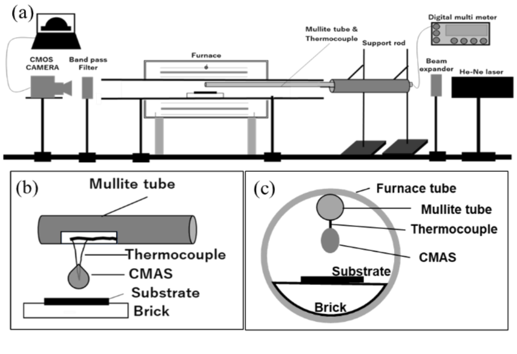

Fig. 1. Contact angle measurement system by sessie drop method (a) Schematic diagram, (b) Sample section, and (c) Sampl e section viewed from the side.

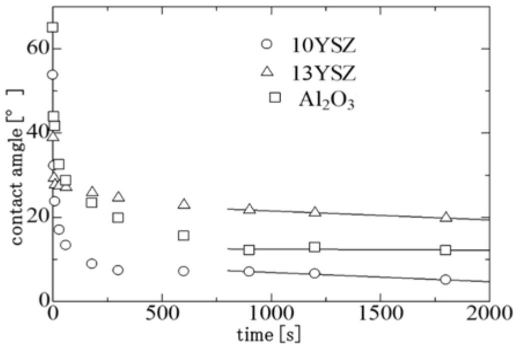

Fig. 2 Time change of the contact angle between the substrate and the CMAS melt.

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OS1-1 L1E – Monday, 11 Sep. 11.40-12.00 – ROOM M2

Heat Capacity and Thermodynamic Functions of $NaREF_4$

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Sodium rare earth ternary fluorides, $NaREF_4$ (RE = rare earth), are intermediate phases for rare earth element extraction and have important technological applications. To better understand their physical properties, energetics, and stability, we measured the heat capacity of three β-structured $NaREF_4$ compounds, $NaNdF_4$, $NaYbF_4$, and $NaYF_4$, from 1.8 to 300 K. Our measurements show an upturn in the low-temperature heat capacity of each sample which we attribute to the splitting of degenerate nuclear magnetic states. We provide calculations of the effective field causing the splitting for each sample. We also report standard entropy, enthalpy, and Gibbs energy increments at selected temperatures from 0 to 300 K. The enthalpy of formation of the three $NaREF_4$ from the elements ($\Delta H^\circ_{f,el}$) and the fluorides ($\Delta H^\circ_{f,fl}$) were measured previously, enabling us to calculate the Gibbs energies of formation from the elements and fluorides. The Gibbs energy of $NaREF_4$ relative to the elements at 298.15 K was calculated to be −1483.5 kJ·mol^{-1}, −1502.2 kJ·mol^{-1}, and −1497.8 kJ·mol^{-1} for $NaNdF_4$, $NaYF_4$, and $NaYbF_4$ respectively. The Gibbs energy of $NaREF_4$ relative to the fluorides at 298.15 K was calculated to be –5.39 kJ·mol^{-1}, −18.12 kJ·mol^{-1}, and −19.1 kJ·mol^{-1} for $NaNdF_4$, $NaYF_4$, and $NaYbF_4$ respectively. Each sample's negative Gibbs energy relative to both the elements and the fluorides indicates stability relative to these compounds. The results confirm that these three $NaREF_4$ compounds are thermodynamically stable relative to the elements and binary fluorides.

OS1-1 L2A – Monday, 11 Sep. 12.00-12.20 – ROOM O2

Development of a Custom High-Temperature, High-Pressure Phase Behavior Apparatus

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In this presentation, a design of a custom apparatus to measure the phase behavior and density of highly asymmetric mixtures to pressures of 300 MPa and temperatures of 673 K is presented. The major components of the instrument consist of a variable-volume view-cell and camera to monitor the phase behavior and an LVDT coupled with a metal bellows to track the internal volume of the view-cell. Density is determined with knowledge of the mass of sample loaded to the view cell. Both the view cell and thermostat were designed using finite element analysis to ensure the operability of the instrument to high pressures and stable temperature control could be achieved at high temperatures. The finite element simulations used to design the apparatus are discussed in detail in this talk. Additionally, image analysis tools that can be used to accomplish automated data acquisition of bubble points, liquid-liquid phase separation, and solidification phase behavior are discussed.

OS1-1 L2B – Monday, 11 Sep. 12.00-12.20 – ROOM O1

Surface tension measurements of steels by aerodynamic levitation. Influence of the chemical composition.

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Thermophysical properties of liquid steels are data of interest for a better understanding of industrial processes such as welding or additive manufacturing and for their numerical models. We are developing at IRDL an experimental device to measure density, surface tension, and viscosity of liquid steels. The method used for surface tension is the oscillating drop aerodynamically levitated. The levitation permits the drop to reach high temperatures (above 2 000 °C) and avoid pollution. The oscillations are obtained with acoustic excitation that sweeps a range of frequencies, then a fast Fourier transform is used to determine de resonance frequency of the drop. Finally, the surface tension is calculated thanks to Rayleigh's equation. Some results on pure metals and steels have been published in the last years [1, 2].

For steels, the presence of some alloy elements can be challenging because of evaporation or chemical reactions. For example, the evaporation of manganese is observed once the melting point of the steel is reached because of its low boiling point. Few seconds at high temperature will lead to the evaporation of nearly all the manganese. The resulting modification in chemical composition is thus likely to have an impact on the surface tension. To address this issue, the surface tension of steels with different chemical compositions is measured. In addition, a chemical analysis of the samples before and after experiment is performed. The experimental data are analyzed in order to establish a link between the surface tension and the chemical composition. The change of the composition due to evaporation of some components is also discussed.

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OS1-1 L2C – Monday, 11 Sep. 12.00-12.20 – ROOM N2

Peng-Robinson and Helmholtz-type Equations of State for CO_2-rich Multicomponent Mixtures Relevant to CO_2 Transport: Density and VLE Predictions

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The accurate prediction of thermodynamic properties of CO_2-rich streams is a crucial research issue for the deployment of the CCUS (CO_2 Capture, Utilization and Storage) value chain, especially concerning the design and operation of CO_2 transportation options, such as pipelines or ships. In pipelines CO_2 is transported either in the supercritical dense phase at atmospheric temperatures (e.g., from 100 to 150 bar and 20 °C) or as a pressurized vapor stream (e.g., 40 bar and 20 °C). Density and Vapor-Liquid Equilibria (VLE) are key thermodynamic properties for a proper prediction of the design and operation of CO_2 pipelines.

In this study, the predictive capability of three thermodynamic models is compared for the calculation of densities and phase envelopes of two multi-component CO_2-mixtures, one ternary blend ($CO_2/N_2/Ar$) presented by Ke et al. [1] and one seven-components based mixture ($CO_2/N_2/Ar/H_2/CH_4/O_2/CO$) tested by Nazeri et al. [2], whose results will be reported and discussed during the ECTP2023 conference presentation; the following models have been assessed: the standard Peng-Robinson cubic Equation of State (EoS) with Peneloux volume translation (PRPeneloux), the Peng-Robinson with Wong-Sandler mixing rule EoS (PR-WS) with specifically tuned binary interaction coefficients and the GERG-2008 multi-parameter Helmholtz-based model (in the version originally developed for natural gas streams). The major goals are: (i) for the cubic PR model, to assess the impact of Peneloux volume translation on the density prediction; (ii) for PR, to evaluate the role of the Wong-Sandler mixing rule compared to the classic one on the phase equilibrium predictive capability; (iii) to compare the accuracy of the cubic PR-WS model against the empirical and more computationally intensive GERG-2008 one.

Results, reported for the ternary $CO_2/N_2/Ar$ case study in Figure 1, show that GERG-2008 is more accurate for density prediction with deviations between -1 and +2%, while the Peneloux vol. translation significantly improves the PR behavior especially at supercritical pressures (where errors are in the ±2% range for PR-Peneloux and up to 6% for the PR model without Peneloux). Concerning the phase envelope prediction, GERG-2008 and the calibrated PR-WS model feature a very similar and satisfactory accuracy, with deviations of the order of around 1% point on dew/bubble point pressures at a given T in nearly the whole range, except than very close to the cricondentherm, while the standard PR with Peneloux fails to reasonably predict the bubble point curve.

This work represents the starting point for a further development and tuning of the selected models, which are going to be used not only in industrial applications for CO_2-flow measurement, but also for planning future labscale experiments related to new VLE, density and heat capacity data for multicomponent CO_2-based mixture.

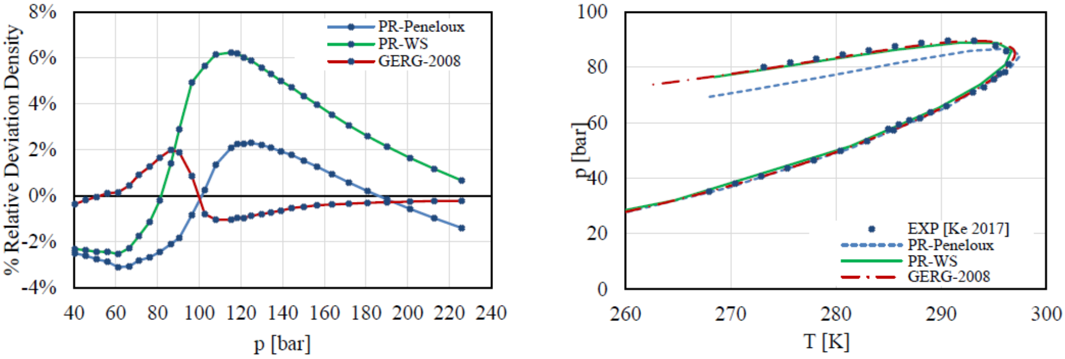

Figure 1: Results from models assessment against experimental data for the $90\%_{mol}\ CO_2 + 5\%_{mol}\ Ar + 5\%_{mol}\ N_2$ ternary mixture (Fig.1 - left) Relative deviation for Density evaluation at 303.15 K and pressures greater than 40 bar, calculated as $\frac{\varrho_{model} - \varrho_{exp\,data}}{\varrho_{exp\,data}}$ % (Fig.1 - right) Phase envelope of the mixture above 260 K.

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OS1-1 L2D – Monday, 11 Sep. 12.00-12.20 – ROOM N1

Metrological Facilities for Thermophysical Properties Measurements at High Temperatures and Determination of the Adhesion of Coating Systems

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Industries such as glass, space and aeronautics as well as power plants continuously need to design new products and improve the existing ones in order to remain competitive in their market. This often involves the use and the development of new materials, which are to be exposed to high temperatures up to 3000 °C and which are mechanically stronger, thermally more resistant, etc. Environmental and thermal barrier coatings (EBCs and TBCs) are for example used in stationary gas turbines and aircraft turbines in order to protect metallic components from the harsh operating conditions and high temperatures. For coating systems, the adhesive quality is of great importance, which are closely correlated with the thermal contact resistance. Usually, local delamination occurs first as a precursor to large-scale delamination and finally to complete coating failure. Therefore, the development of contactless methods for detecting local delamination during operation is of major interest. Existing investigation methods either are based on destructive testing (e.g. cutting the specimen) or require contact sensors (e.g. ultrasonic testing). Non-contact methods are not sufficiently reliable so far.

Hence, in the EMPIR Hi-TRACE project reference facilities and new methodologies have been developed for quantifying debonding and thermal contact resistance of layered systems by non-contact methods as well as for measuring the relevant thermophysical properties (thermal diffusivity, specific heat and emissivity) at high temperatures up to 3000 °C. In the developed setup, the surface of the investigated object is thermally excited with a non-contact heating method and the resulting temperature distribution on the surface is recorded using a thermographic device. Since an existing delamination increases the thermal contact resistance between the coating and the substrate, the heat transfer from the coating to the subjacent substrate is locally reduced, which results in a locally higher temperature as visualized in Fig. 1.

For performing quantitative analysis of the layer adhesion from the derived results, the underlying thermophysical properties of the coatings and substrates have been measured in this work at high temperatures using a laser flash facility [1] and an emissivity measurement apparatus [2]. Thus, the measured spectral emissivity (and transmissivity where applicable), thermal diffusivity and specific heat of the materials are presented together with the evaluation of the thermal contact resistance and the debonding of the investigated coating systems. Furthermore, the developed facilities and setups are introduced as well as the performed uncertainty assessments.

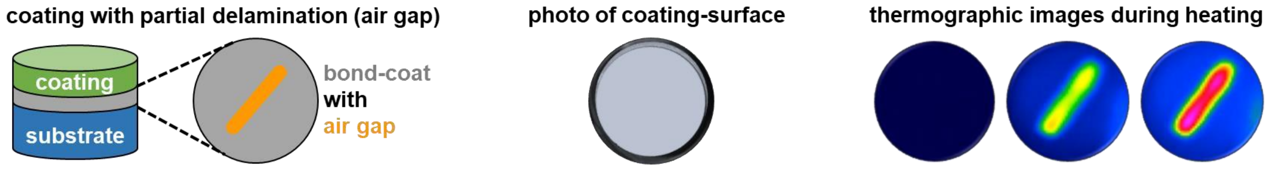

Fig. 1. Coating on a metal substrate with defined partial delamination (on the left) and temperature distributions on the coating surface determined by active thermography during the heating phase (on the right), which detects the partial delamination, although no delamination is visible on a photo of the coating surface (in the middle).

Acknowledgment

This research was funded by EMPIR program co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation program (grant number: 17IND11-Hi-TRACE).

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Mass transport control of remodeled microvascular system using bio-3D-printing

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In fundamental biomedical research field, remodeling of vascular system and controlling the mass transport of chemicals via the capillary vessel is extremely valuable for the tissue engineering. However, in the conventional technique, the complex 3D vascular system has not been established yet because of the difficulty to combine the thermal and fluid system design in the remodeled vascular system. Here, an organ-on-a-chip is one of promising approach to remodel the organs with vascular system in the microfluidic devices as shown in Fig. 1. By using the organ-on-a-chip, rapid and highly reproducible analysis can be achieved compared with other bulky test using animals. In this study, a bio-3D printer based on the femtosecond laser processing of collagen hydrogel has been newly developed. The proposed bio-3D printer can control the spatial resolution of processing and the mass transport properties of processed material by changing the fluence of the femtosecond laser. When the pulse energy is low, a photoablation occurs in the hydrogel. Photoablation is a non-thermal process, minimize the thermal effects on non-irradiated areas, hence, precise processing of several microns is accomplished. On the other hand, when the pulse energy is high, a cavitation occurs in the hydrogel. The mass transport of chemicals via the processed hydrogel was monitored for the thermal and fluid system design. In this presentation, the validity of proposed method to remodel the tissue by the femtosecond laser processing is discussed by comparing the mass transport characteristics of processed hydrogel using two different processing modes.

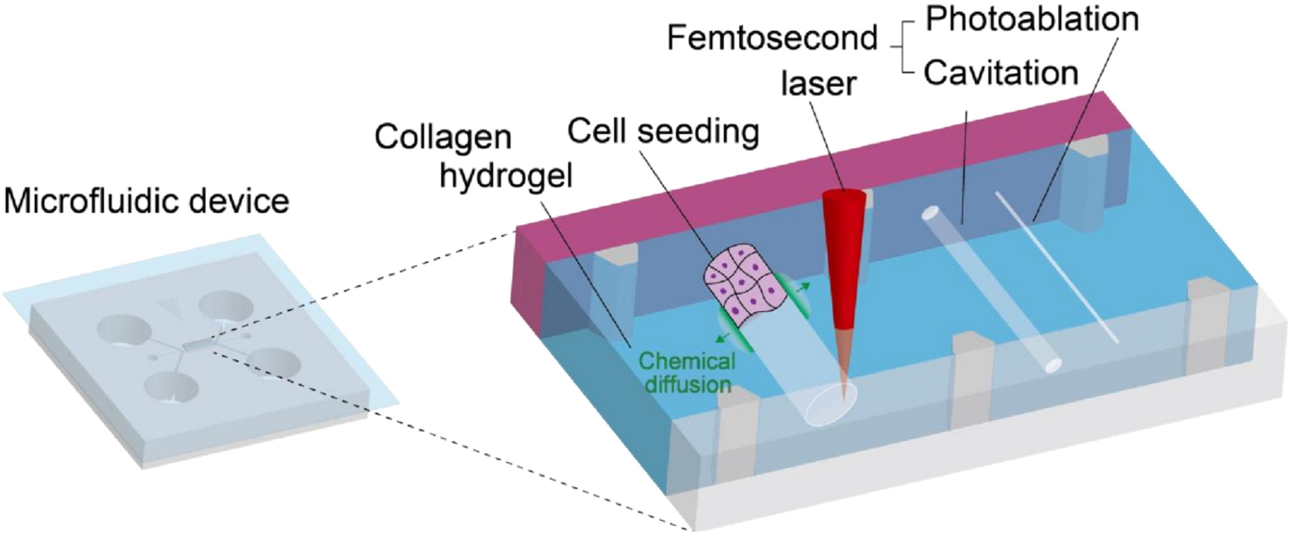

Fig. 1. Organ-on-a-chip

Acknowledgment

This study was partially supprorted by JSPS KAKENHI Grant Number 22H01418.

OS1-1 L3A – Monday, 11 Sep. 12.20-12.40 – ROOM O2

Simultaneous CCS and NG Storage in Salt Caverns

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Since the start of the industrial revolution, the world has seen unprecedented technological progress and accelerated energy demand, which is currently more than 90% carbon-intensive combustion. Necessarily, an equally proportional increase in CO_2 emissions has followed, together with an increment in the global average temperatures of more than 1°C. Clearly, a significant reduction in CO_2 emissions is not realistic in the forthcoming future. Even if a large proportion of the energy production is converted to renewable sources, negative emissions will still be necessary to stabilize or even reduce the CO2 content of the atmosphere. Yet, the primary energy source will continue to be carbon-based, leaving efficient Carbon Capture and Storage (CCS) innovation as the only sustainable option for our modern society and the only way to meet the Paris climate accord targets. Before the Russia-Ukraine conflict, Natural Gas (NG) represented 24% of Germany's primary energy use. Significant domestic and industrial renovations and investments were carried out in favor of NG, which has quickly become a more expensive and scarcer commodity. The demand for NG is heavily seasonal-dependent; the winter peak demand almost doubles that of the summer, requiring buffering the supply with reserves, 24% of which are stored in deep salt caverns operating up to 200 bar. However, a minimum operating pressure of about 70 bar is required to balance the lithostatic pressure, resulting in around 30% mass of the NG in salt caverns being permanently stored. Therefore, due to the current geopolitical situation, using part of these potential reserves would greatly alleviate Germany's current energy pressure, possibly also in other countries. The current German salt cavern storage capacity is above 15 billion m^3, while the estimated total annual German CO_2 emissions are equivalent to just 1 billion m^3 of CO_2 at 30 ºC and 100 bar. However, when mixed with some fluids, CO_2 develops very peculiar behavior, such as phase barotropicity. This behavior may allow the simultaneous storage of NG and CO_2 in salt caverns, resulting in innovative scenarios for CCS, providing an excellent contribution to Germany's carbon neutrality commitments and those of many other countries. However, the benefit of this approach would not only be ecological. Still, it would also allow for the liberation of large amounts of NG reserves currently trapped in salt caverns just to maintain the minimum lithostatic pressure. Given Europe's current geopolitical situation, we believe that the ideas discussed in this presentation are of high ecological and economic relevance.

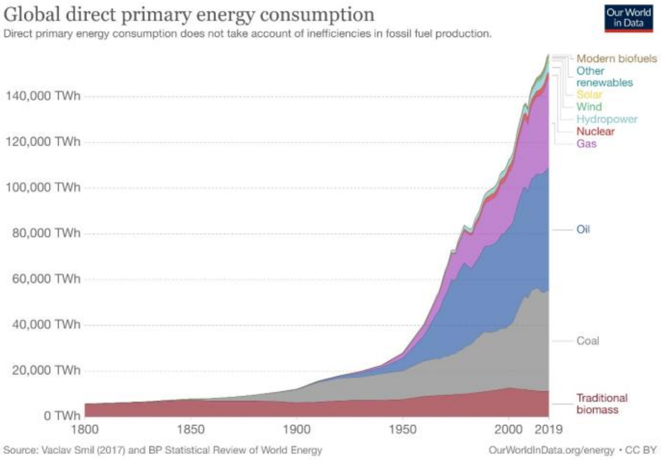

Fig. 1. Energy consumption trend.

OS1-1 L3B – Monday, 11 Sep. 12.20-12.40 – ROOM O1

Surface tension measurement of Cu_2S-FeS melts by aerodynamic and electromagnetic levitation techniques

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In smelting processes for pyrometallurgy of copper, a matte phase, which consists of molten Cu_2S and FeS, is separated from a slag phase by settling. In the settling process, the small size of matte droplets suspends in the slag phase, which causes a copper loss. The sedimentation velocity of the suspended matte droplet is described by Stokes law [1]. In addition, the adhesion of the matte droplet to gas bubbles affects the sedimentation phenomenon of the suspended matte droplets in the slag phase. Adhesion of the matte droplet to the bubble depends on the surface and interfacial tension of matte and slag [2]. The thermophysical properties of matte and slag affect the yield of matte production. Accurate thermophysical properties of molten matte and slag are required to design the separation process of suspended matte droplet in the slag phase. This study measured the surface tension of the Cu_2S-FeS melts using aerodynamic levitation method.

A sample with a diameter of 1.5 to 2.0 mm was placed on the conical nozzle, and the chamber was then evacuated using a rotary pump. The chamber was filled with normal pressure of Ar gas. By supplying Ar gas through the conical nozzle, the sample was levitated. The sample was heated and melted by irradiation of CO_2 laser, then, sample temperature was measured by a single-color pyrometer calibrated using the liquidus temperature of each sample. The surface tension can be evaluated from the fundamental oscillation frequency of the surface oscillation of the levitated droplet. In this study, after the sample temperature stabilized, surface oscillation of the levitated droplet was excited acoustically using two opposite speakers installed at the gas inlet for the levitation of the sample [3]. A resonance frequency of the fundamental surface oscillation was explored by observing the surface oscillation of the droplet. The surface oscillation of the levitated droplet was excited, after that, the excitation was turned off. The surface oscillation of the droplet decay, then, the droplet images at the oscillation decay were observed by high-speed camera. The surface oscillation frequency was evaluated using the droplet images at the oscillation decay. Surface tension of Cu_2S, FeS, and 31, 49, 69, 81mol%FeS-Cu_2S melts was measured in this study.

For all the compositions, Surface tension of Cu_2S-FeS melts decreased with increasing temperature. The surface tension of Cu_2S-FeS alloy was smaller than the value calculated from the additivity of surface tension of pure Cu_2S and FeS. It is considered that the surface concentration of FeS for the Cu_2S-FeS melt is higher than the bulk concentration of FeS.

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Fuel-Nitrogen Vapor-Liquid Equilibria Modeling Using the Helmholtz Energy Equation of State Approach

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The aviation sector is estimated to be responsible for 2% of global carbon emissions, and sustainable aviation fuels (SAF) have been considered as low-emission alternatives for petroleum derivatives commonly used in the sector. In the design and optimization of aircraft engines, the knowledge of thermophysical properties of the fuel-air blend is fundamental and is driven by their chemical structures. The vapor-liquid equilibria (VLE) of fuel-N_2 mixtures are of interest in the evaluation of the thermophysical state of the fuel-air mixture in a combustion chamber considering nitrogen as surrogate of the air. The present work aims to develop new mixture models of fuel-N_2 mixtures based on the Helmholtz energy equation of state (EoS) by fitting the binary interaction parameters. The fitting is based on the dataset available in the NIST ThermoDataEngine (TDE) and considers a wide range of chemical families that are gaining interest in the emerging sustainable fuels field. The equations of state available in Refprop10.0 for the pure fuels have been considered in the work, as well as some preliminary EOS for other fuel candidates. Finally, a correlation is proposed to calculate the binary interaction parameters for fuels-N_2 mixtures based on their chemical structure.

OS1-1 L3D – Monday, 11 Sep. 12.20-12.40 – ROOM N1

Anisotropy Study on Thermal Conductivity of MoS_2 and WS_2 Thin Films

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MoS_2 and WS_2 thin films have high carrier mobility and excellent mechanical properties, and have great application potential as thermoelectric materials in micro-nano flexible thermoelectric devices [1]. In this paper, MoS_2 and WS_2 films were prepared by magnetron sputtering, and 3ω method thermal conductivity measurement system was built to measure the thermal conductivity of the films. The temperature range of the experiment was 25 ° C ~ 200 ° C. According to experimental results, it is found that MoS_2 and WS_2 thin films have low thermal conductivity ($\leqslant 1.0\ W\cdot m^{-1}\cdot K^{-1}$), and their thermal conductivity increases with the increase of temperature. Due to the existing methods of measured the anisotropic thermal conductivity of the thin film usually need the complex thermal model and the high-precision manufacturing process such as suspended wire [2], the heating wires with width of 200 microns and 5 microns were used to study the anisotropy of thin films, respectively. From the measurement results of 5 μm wrie are significantly higher than the results of 200 μm wire, it can be known that in-plane heat conduction is also a main component. Fig. 1 shows the experimental results of MoS_2 and WS_2 thin films measured with 200 μm wide and 5 μm wide wires, respectively. Then, the thermal conduction of the anisotropic multilayer film-substrate system is derived using 2D thermal conduction model. Finally, in-plane and cross-plane thermal conductivities of the films were calculated separately.

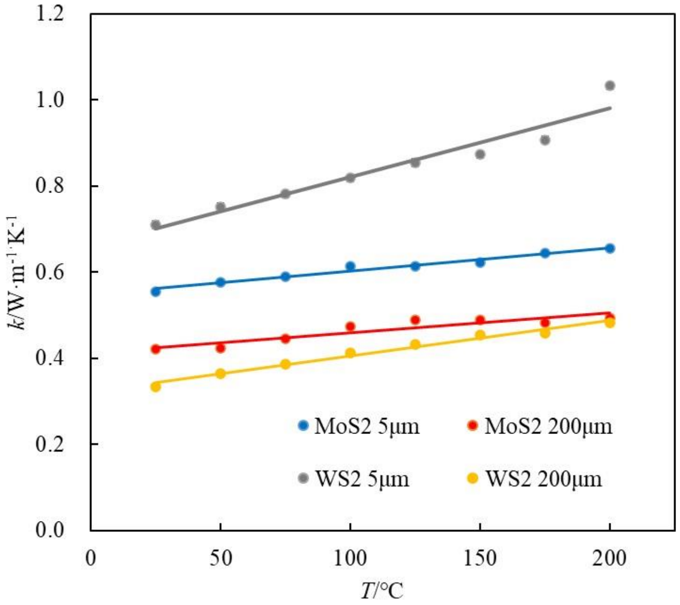

Fig. 1. Thermal conductivity measurement results of MoS_2 and WS_2 Thin Films

Acknowledgment

The supports provided by the National Natural Science Foundation of China (No. 41941018) for the completion of the present work are gratefully acknowledged.

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OS1-1 L3E – Monday, 11 Sep. 12.20-12.40 – ROOM M2

Wideband Mid-Infrared Thermal emitter based on multilayered Nanocavity Metasurfaces

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The efficiency and bandwidth of mid-infrared (M-IR) light sources will radically determine the performance of many applications including thermal imaging, thermal sensing and infrared spectroscopy. Compared to the conventional components, the excellent ability to control light makes metasurfaces an ultra-thin thickness and high-performance platform for infrared applications. In the recent past, various metasurfaces have been proposed to achieve high thermal radiation. Up to now, increasing the radiation bandwidth of metasurface emitter has become a key goal to enable extensive applications. Herein, we experimentally demonstrate a broadband M-IR thermal emitter using multilayered nanocavity metasurface consisting of two pairs of circular-shaped dielectric (Si_3N_4)–metal (Au) layers. We attribute the broadband high emissivity to the combination of multiple gap plasmon resonance (GPR) and propagation surface plasmon resonance (PSPR). The GPR is strongly supported by the metal-dielectric-metal (MDM) cavity with different dielectric thicknesses. The PSPR is connected with the period of metasurface. The meta-emitter offers a nearly perfect emissivity of more than 80% in the M-IR atmospheric window of 8~14 μm, providing broadband absorptance for both p- and s-polarisation states over a wide observation angle of 0° to 60° . The experimental description and the theoretical framework establish a basis for designing wideband thermal emitters, which, as expected, will initiate a promising approach for M-IR sources.

OS1-1 L4A – Monday, 11 Sep. 12.40-13.00 – ROOM O2

Thermophysical characterization of Deep Eutectic Solvents using the soft-SAFT EoS for applications in Greenhouse Gas Capture and Separation

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In the recent years, the themophysical study of Deep Eutectic Solvents (DES) has become a hot topic [1, 2]. These eutectic mixtures are based on the combination of a Hydrogen Bonding Acceptor (HBA) compound (normally a halide salt), and a Hydrogen Bonding Donor (HBD), (typically a neutral complexing agent). As a consequence of the hydrogen-bonding effect between both compounds, a substantial decrease of the melting point compared to that of the individual components occurs, far below that of an ideal system. This fact widens the range of the liquid state, while keeping a negligible volatility. In addition, DES are as tunable as Ionic Liquids, as the HBA:HBD combination can be easily modified, changing the hydrogen-bonding dominance and the thermophysical properties. However, those mechanisms are still being investigated so as to better describe their thermophysical behaviour. In this regard, the use of multiscale simulation provides a useful path to obtain additional information to guide the experimental work into the right direction.

In this contribution, a practical methodology to thermophysically characterize DESs for greenhouse gas capture applications is described. For this purpose, the soft-SAFT equation of state has been used in combination with other modelling tools, such as Turbomole-COSMO. Based either on molecular simulation data or charge analysis distribution, a rational choice of a molecular model and the number of dominating hydrogen bonds is proposed. An individual component approach, where each entity forming the DES is treated as an independent compound, is followed to describe density and viscosity of DESs, as well as the impact of water addition. Next, the solubility of CO_2 and fluorinated refrigerants in different DESs is provided, highlighting the impact of different variables (pressure, temperature) and structural characteristics of the DES (type of HBA, HBD, number of fluorine atoms, etc.) in the results [3-5]. The solid basis of soft-SAFT allows to present an integrated framework where it is possible to predict the behavior of these solvents at different conditions, facilitating a quick screening so as to select the best conditions for a particular gas capture or separation.

Acknowledgment

This research is supported by the Spanish Ministry of Science and Innovation (project STOP-F-Gas, PID2019108014RB-C21) and NEW-F-TECH (TED2021-130959B-I00).

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OS1-1 L4B – Monday, 11 Sep. 12.40-13.00 – ROOM O1

Evaluation of thermophysical properties of liquid Ti-Al-Cr-Nb alloys: surface tension and viscosity

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The surface tension and viscosity of liquid Al-Ti-Cr-Nb alloys were analysed with respect to the Ti-Al system consisting of the major alloying elements and estimating the effects of the other alloy components on the two properties. In the case of the surface tension, the analysis was extended to the Al-Ti-Nb (Fig.1) and Al-Ti-Cr ternary subsystems. The surface tension of these systems was calculated by the Compound Formation Model (CFM) and/or Quasi Chemical Approximation (QCA) for regular solution [1]. Preliminary test of the models used to calculate the viscosity isotherm of liquid Al-Ti alloys indicated the Terzieff model as the most appropriate. Subsequently, the surface tension and viscosity experimental data of the Ti48Al48Nb2Cr2 [2] was compared to the model predicted values as well as with the literature data.

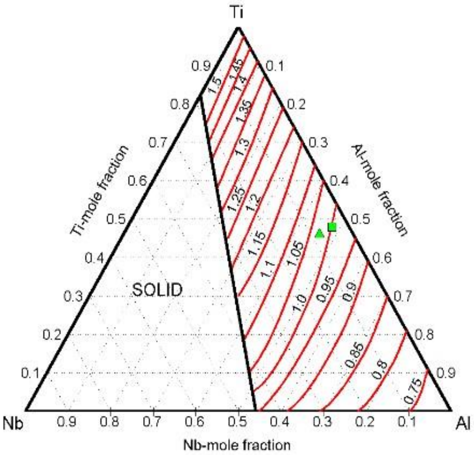

Fig. 1. Iso-surface tension lines of liquid Ti-Al-Nb alloys calculated for T=1973 K. In the Gibbs triangle the square symbol indicates the compositional locations of the Ti48Al48Nb4 (in at%) that approximates the Ti48Al48Nb2Cr2 alloy.

Acknowledgment

This work has been performed in the framework of the ESA MAP Programme under contract no. AO-99-022 (14306/01/ NL/SH).

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OS1-1 L4D – Monday, 11 Sep. 12.40-13.00 – ROOM N1

Large thermal conductivity switching ratio of Gd hydrides thin film with hydrogenation and dehydrogenation reactions

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A thermal switch is one of the emerging techniques to control heat flow in electronics, automobiles, satellites and so on. Control of switching is basically based on a sharp contrast of the thermal conductivities between two states induced by external fields [1] and chemical reactions [2]. For the VO_2 thin film, the thermal conductivity changes by a factor of 1.5 [3] by the metal-insulator transition around 341 K, where heat carriers also change from free electrons and phonons in the metal state to phonons in the insulator state. It is necessary to farther improve thermal conductivity contrast. Here, we focus on a switchable mirror [4] as a strong candidate of thermal switch materials, that enables reversibly switching between metal and semiconductor using hydrogenation and dehydrogenation reactions. The electrical conductivity of GdH_2 (metal, 2.3×10^6 S m^{-1} [5]) is 12 times larger than that of the VO_2 (metal, 1.9×10^5 S m^{-1} [3]). In this study, thermal conductivities of Gd hydride films were investigated in terms of hydro-/dehydrogenation reactions.

A Pd/Gd/Mo three-layered film was fabricated on a synthetic quartz glass substrate by dc magnetron sputtering using each metal target under 1.0 Pa of Ar (99.999 % in purity). Nominal thicknesses of the Pd, Gd and Mo were 5 nm, 300 nm and 102 nm, respectively. The thermal conductivity of the film was measured using a time-domain thermoreflectance apparatus. A Pd/Gd film was also prepared under the same sputtering conditions for electrical conductivity and X-ray diffraction (XRD) measurements. The data for GdH_3 were obtained after being maintained for 60 min under a mixture of Ar and H_2 (3 %) gases atmosphere. On the other hands, the data at the dehydrogenated state was measured after exposure to air for 60 min.

Figure 1 shows XRD patterns of the Pd/Gd two-layered film in the as-deposited, hydrogenated and dehydrogenated states. The hydro-/dehydrogenations reactions resulted in structural changes from Gd (asdeposited) to GdH_3 (hydrogenated state) and GdH_3 to GdH_2 (dehydrogenated state), respectively. Figure 2 shows the thermal and electrical conductivities of the Gd, GdH_3 and GdH_2 film. The thermal conductivity increases by a factor of 7.3 when the GdH_3 was dehydrogenated to the GdH_2, which was corresponding to change in the electrical conductivity.

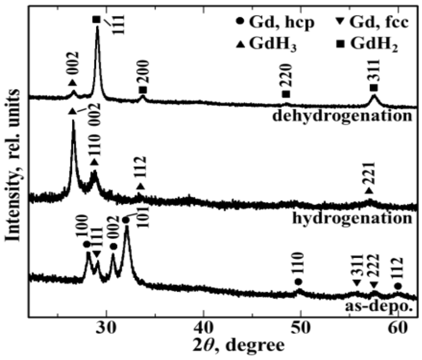

Figure 1. XRD patterns of the Pd/Gd film for the as-deposited, hydrogenated and dehydrogenated states.

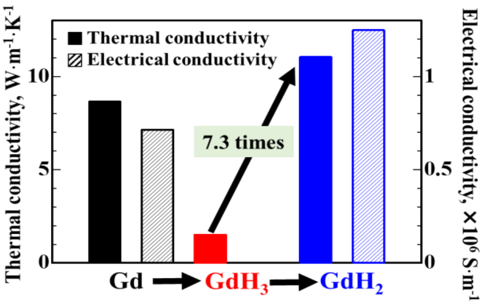

Figure 2. Thermal and electrical conductivities of the Gd, GdH_3 and GdH_2 film.

Acknowledgement

A part of this work was supported by JSPS Grants-in-Aid for Scientific Research, Grant Number 23H01362.

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OS1-1 L4E – Monday, 11 Sep. 12.40-13.00 – ROOM M2

Broadband and ultra-thin cryogenic radiator based on MIM structure for infrared astronomical observations

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A cryogenic radiator is required for high-sensitivity infrared astronomical observations to passively cool instruments to cryogenic temperatures and reduce observation noises. In order to increase the efficiency of radiative cooling, a radiator with high emittance at cryogenic temperatures is required. However, conventional radiator materials show low emittance at cryogenic temperatures as the wavelength of infrared radiation at cryogenic temperatures is long and the influence of the substrate increases. Although it is possible to increase the thickness of the material to increase emittance, however, this approach could lead to an increase in mass, contamination, and thermal resistance. For above reasons, no suitable material has been found that shows high emittance at cryogenic temperatures.

Therefore, we propose a novel radiator based on periodic Metal-Insulator-Metal (MIM) sandwich structure. This structure shows absorption peak at specific resonant wavelength by exciting surface plasmon resonance. In other words, the radiator shows high emittance at the resonant wavelength, which is mainly determined by the size and shape of periodic metal array. This feature benefits us in terms of ultra-thin thickness and selectivity of absorption wavelengths. In order to achieve the broadband radiation, multi-sizing periodic structures has been analytically investigated, but it is still challenging to fabricate three-dimensional structure and few fabrication results have been reported.

In this presentation, a radiator with high absorption near 150 μm, the peak wavelength of 20 K blackbody radiation, was designed by using the finite-difference time-domain (FDTD) method. The periodic structure was multi-sized in two and three-dimensional direction to achieve the broadband radiation required of spacecraft radiators. In addition, the sample aiming at broadening the absorption peaks was fabricated using nanofabrication techniques and the thermo-optical properties of the fabricated samples were also measured by Fourier Transform Infrared Spectroscopy (FTIR).

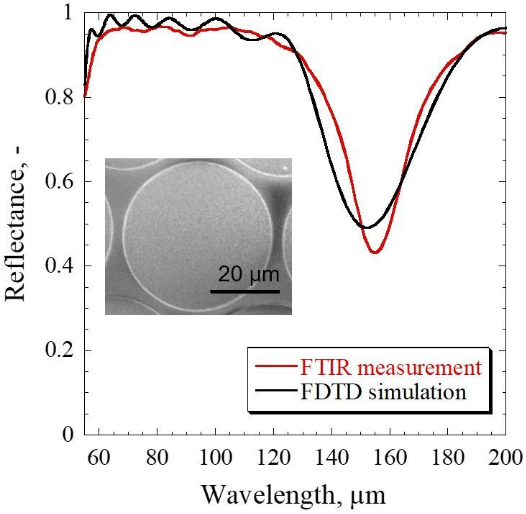

Fig. 1. Reflectance and SEM image of a basic MIM structure.

Acknowledgment

A part of this work was supported by “Advanced Research Infrastructure for Materials and Nanotechnology in Japan (ARIM)” of the Ministry of Education, Culture, Sports, Science and Technology (MEXT). Proposal Number JPMXP 1222UT1151.

OS1-2 K1A (Keynote) – Monday, 11 Sep. 14.30-15.00 – ROOM O2

The Thermal Conductivity of Galinstan ($Ga_{68.4}In_{21.5}Sn_{10}$) Alloy

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Matthias H. Buschmann[3]

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The need for new heat transfer agent for many applications, namely in the consumer electronics industry, requires materials, liquid at room temperature, with high thermal conductivity [1,2]. From the different possibilities, Galinstan, a eutectic alloy of Gallium, Indium and Tin with a melting point (T_m = 283.4 K)[1] has been proposed for many applications, namely for replacing mercury, a toxic element used for many years [2,3].
It is the purpose of this paper to report thermal conductivity measurements of Galinstan, produced by Haines & Maassen Metallhandelsgesellschaft mbH, Bonn, Germany, with product name Gallium/Indium/Tin Eutectic (NL-011). The composition of the eutectic alloy was given by the manufacturer, as $Ga_{68.4}In_{21.5}Sn_{10}$, although there is the presence of some components like Cu and Pb (around 0.0035 wt%) and Al and Zn (< 0.00005 wt%). The method used was the transient hot-strip, THS, and the details of the data acquisition system and platinum metal-film sensors, produced by PVD in ceramic substrates, have been presented recently [4] for measuring the thermal conductivity of air, at temperatures up to 800 ºC. In the current project the sensors had to be electrically insulated from the liquid conducting alloy, by developing a halogen-free, flame retardant coating, graded up to 600V insulation. Details of the measuring procedure will be reported. Measurements were performed between 28 and 103 ºC, at atmospheric pressure, with an estimated uncertainty of 5%, were fitted to a linear correlation. Results obtained are shown in Fig. 1, along with data of Plevachuk et al (2014)[1], with an estimated uncertainty of 7%.

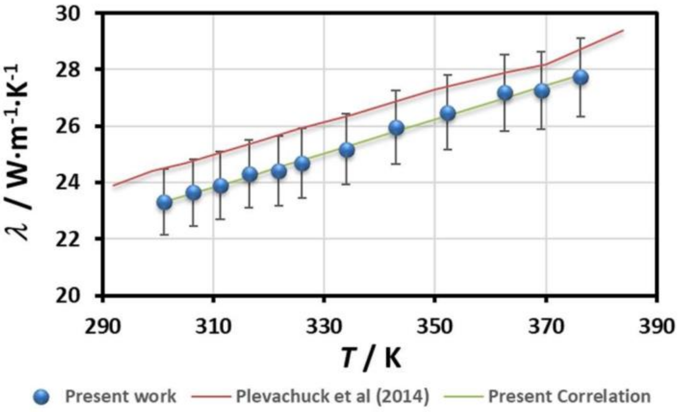

Fig. 1. Thermal conductivity of Galinstan as a function of temperature. Comparison with data of Plevachuk et al. (2014) data and the present linear correlation are also presented.

Acknowledgment

This study is supported by Fundação para a Ciência e Tecnologia, I.P. Portugal through projects of Centro de Química Estrutural (CQE) under research grant UIDB/00100/2020 and Bundesministerium für Wirtschaft und Klimaschutz der Bundesrepublik Deutschland under research grant 49MF200081.

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OS1-2 K1B (Keynote) – Monday, 11 Sep. 14.30-15.00 – ROOM O1

Solidification process of SS316L containing B_4C by a combination of ultra-high temperature thermal analysis and microstructural observation

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During a core disruptive accident in a sodium-cooled fast reactor, complex behavior of core damage is observed due to eutectic reaction between control rod material (B_4C) and reactor structural material (type 316 austenitic stainless steel (SS)). To simulate this eutectic reaction behavior, the authors have obtained thermophysical property data of B_4C-SS melts by combining the electromagnetic levitation method and static magnetic fields [1, 2]. In order to analyze the eutectic reaction mechanism in detail, it is necessary to clarify the melting behavior and solidification process of the B_4C-SS system. However, SS containing high concentration of B_4C has high liquidus temperature, which makes thermal analysis difficult using existing measurement equipment. In this study, we report the results of thermal analysis of melting and solidification behavior of high concentration B_4CSS materials using a blackbody radiation type ultra-high temperature thermal analysis method [3] developed in our laboratory. In order to elucidate the solidification process, we also observed the microstructure of samples obtained by rapid cooling of samples melted by the electromagnetic levitation (EML) method. Each experimental method is described below.

Ultra-high temperature thermal analysis: A BN crucible with blackbody cavity containing the sample (14, 17mass%B_4C-SS) was set in the furnace and then heated in an Ar gas atmosphere. The temperature was controlled by a radiation pyrometer installed in the lower part of the furnace, and the sample temperature was measured by the radiance from the blackbody pores by a radiation pyrometer installed in the upper part of the furnace. The radiation pyrometer at the top was calibrated with the eutectic temperatures of Ni-C and Ru-C in advance.

Solidification microstructural analysis: The samples were electromagnetically levitated and completely melted, then the temperature was gradually lowered to below the liquidus temperature or below the eutectic temperature, and then quenched. The surfaces and cross sections of the solidified samples were observed by SEM-EDX, and the phases were identified by an X-ray diffractometer.

At the conference, we will report the results of thermal analysis and solidification microstructure observation, as well as a discussion of the solidification process of B_4C-SS based on these results.

Acknowledgment

This study was supported by the "Technical development program on a common base for fast reactors" entrusted to the Japan Atomic Energy Agency (JAEA) by the Ministry of Economy, Trade and Industry (METI).

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OS1-2 K1C (Keynote) – Monday, 11 Sep. 14.30-15.00 – ROOM N2

Assessment of Refrigerant Property Modeling with SAFT-based Thermodynamic Models

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The thermodynamic properties of refrigerants (densities, phase equilibria, speed of sound, etc.) have been extensively studied over the last decades. The most accurate thermodynamic models are of the multi-fluid paradigm, based upon highly accurate pure fluid equations of state combined with mixing rules of varying complexity, from estimation schemes to departure functions, depending on the availability of high-quality data. The crux of the problem in modeling new refrigerants is the frequent complete absence of experimental data for many mixtures, and even for many pure refrigerants. Thus, the multi-fluid approach is not feasible because extensive high-quality data are not available, and simpler models must be used that can be parameterized based upon more limited data.

The "SAFT approach" has spawned a large family tree of thermodynamic models based on a perturbation approach. Perhaps the most popular variant in this genealogy is the PC-SAFT approach introduced by Gross and Sadowski. In recent years the SAFT-VR-Mie approach of Lafitte et al., with its more physical treatment of the molecular interactions, has been shown to yield more accurate thermophysical properties, especially caloric ones. Adding polar contributions (as was done by Gross and Vrabec) offers yet another route to improve the model fidelity.

This work will consider the models available in the open-source teqp library to ascertain which modeling approach is most suitable for the accurate representation of the properties of refrigerants. This will include PC-SAFT and SAFT-VR-Mie and their polar variants.

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OS1-2 K1D (Keynote) – Monday, 11 Sep. 14.30-15.00 – ROOM N1

Devices and methods for nano- and micro-scale measurement of heat transport properties of softmaterials; prospects from materials informatics to process informatics

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The hierarchical structures found in materials are the basis for multifunctionality that is sensitive to temperature and thermal conduction at the micro- and nanoscale. The interfaces between the constituent structures, such as the boundaries between crystalline lamellae and amorphous layered structures, are factors that contribute to the control of heat. Although phonons are inherently present in condensed systems, their behaviour is strongly dependent on the scale of the system and material, and they play a major role in electrical, optical[1], thermal[2] and mechanical[3,4] properties. Therefore, nanostructuring has proven to be very effective in controlling phonons and altering material properties. In this context, methodologies[5] to measure thermal conduction are important.

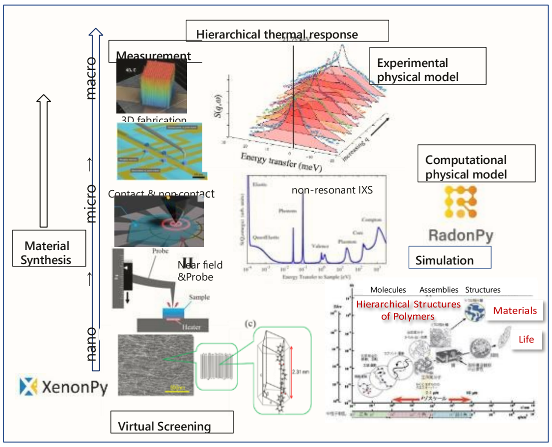

Fig. 1: Space-time hierarchy of materials; crosshierarchical thermal response from micro to macro-scale level, inter-hierarchical heat transfer mechanisms.

The research aims to form the basis for the development of accurate nano- and micro-scale thermal design of polymers and molecular crystals. Specifically, a nanoscale heating point is generated using a heatable probe-type AFM tip used in nanoscale lithography, and the temperature response to the heating is measured using a nano-and micro-scale temperature sensor designed by electron- or photo lithography to construct a measurement system that dramatically improves the spatial resolution of conventional thermophysical property measurements.

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In parallel, the spread of data-driven materials research has increased the need for systematically designed materials property databases, [6] where the bias and variance in the MD-calculated properties should be calibrated by a machine learning technique. Coupled usage will promote the basis of the process informatics through the virtual screening of the materials design validated through the processing conditions and measurements.

Acknowledgment

This work was partially supported by a Japan Society for the Promotion of Science (JSPS) grant, a Japan KAKENHI grant (Grant No. 20H04663, 22H02137), and a JST CREST grant (Grant No. JPMJCR19I3).

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Hydrothermal synthesis of vanadium dioxide nanoparticles for architectural glazing applications

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Windows, owing to their low thermal resistance and static transmittance to solar radiation, are often considered the least-efficient elements in the building envelope. Thermochromic coatings, which passively adjust their thermo-optical properties in response to temperature, have emerged as a promising solution to enhance energy efficiency. Among the various thermochromic coating technologies, Vanadium Dioxide (VO2) thermochromic coatings have garnered significant attention due to VO2's phase transition between solar transmitting and blocking states at 68°C. However, such a temperature is deemed to be too high for architectural glazing applications.

Tungsten (W) has been identified as the most efficient means to lower VO2's transition temperature to near room temperature. In this article, pure VO2 and W-doped VO2 nanoparticles were synthesized under high-pressure, high-temperature hydrothermal conditions for 72 hours, aiding in the formation of thermodynamically stable phases. The hydrothermal method is one of the most attractive and environmentally benign techniques as it is relatively simple, cost-effective, generates minimal waste, and operates at lower temperatures compared to other nanomaterial processing techniques. The synthesized nanoparticles were further characterized using X-Ray Powder Diffraction, Differential Scanning Calorimetry, Scanning Electron Microscopy, and Energy Dispersive XRay Spectroscopy. The results revealed that the nanoparticles are identified as pure monoclinic VO2, transforming into a rutile phase upon heating. The phase transition temperature of the W-doped VO2 nanoparticles decreased gradually with increasing W content, ultimately reaching near room temperature. Additionally, the nano-scale particle sizes, coupled with the uniform morphology and size distribution, is promising for fabricating thermochromic nanocomposites for architectural glazing applications.

Methyl myristate-in-water nanoemulsions as phase-change secondary working fluids for cooling applications

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Phase change nanoemulsions are colloidal systems in which a phase change material (PCM) is suspended in the form of nanometer-sized droplets within a based fluid, usually water [1]. When going from liquid to solid (or vice versa), dispersed droplets release or absorb significant amounts of thermal energy under nearly constant temperature conditions [2]. Thus, PCM nanoemulsions are interesting secondary working fluids to improve the storage capacity and thermal management in a wide range of heating and cooling applications [1-3]. A revision of the literature evidences that most investigations used paraffins as dispersed phase [3]. However, other organic materials such as sugar alcohols or fatty acid (esters), which are non-toxic and can be obtained from bio-sourced feedstocks, are still relatively unexplored. This work presents novel methyl myristate-in-water nanoemulsions as potential heat transfer and storage media to work at temperatures around 287 K. A mixture of non-ionic Brij^{TM}S2 and anionic sodium dodecyl sulfate (SDS) was used as emulsifier, while *n*-hexadecane, methyl stearate and combinations of them were tested as nucleating agents. The stability and thermophysical profile of suspensions with 2-12 wt.% contents of dispersed phase were investigated in the temperature range of 278-333 K. A mixture of *n*-hexadecane and methyl stearate with a mass proportion of 1:3 proved effective to reduce the size of dispersed droplets below 200 nm, improve temporal stability and reduce the super-cooling of the methyl myristate nano-confined in the nanoemulsions. Owing to the lower thermal conductivity and density of organic materials in comparison to water, these two properties reduced with the loading of dispersed phase present in the sample. Rheological analyses showed that 2-4 wt.% suspensions were mainly Newtonian while a slight shear-thinning behaviour was observed for 6-12 wt.% samples, particularly at 278 K when dispersed droplets were solid.

Acknowledgments

Project "PCM Cool" financed by CNR Italy. Grant PID2020-112846RB-C21 funded by MCIN/AEI/10.13039/501100011033. D. Cabaleiro is a recipient of a "Juan de la Cierva" contract (agreement IJC2020-043779-I) supported by the Spanish "Ministerio de Ciencia e Innovación" and the European Union NextGenerationEU/PRTR. D. Hermida-Merino acknowledges a "Maria Zambrano" contract for the University of Vigo, financed by the Spanish "Ministerio de Ciencia e Innovación" and the European Union NextGenerationEU/PRTR.

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Applying thermoelectric device using decay heat in wireless communication module

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Small Modular Reactors (SMRs) are advanced nuclear reactors that have a power capacity of up to 300 MW(e) per unit, which is about one-third of the generating capacity of traditional nuclear power reactors [1]. SMRs integrate major devices (*e.g.*, reactor, steam generator, etc.), so they are small - physically a fraction of the size of a conventional nuclear power reactor. Also, SMRs are modular - making it possible for systems and components to be factory-assembled and transported as a unit to a location for installation, and reactors - harnessing nuclear fission to generate heat to produce energy. Currently, more than 80 types of SMRs are under development. One of the configuration diagram of the instrumentation line expected for SMRs is shown in Figure 1. The sensing data near the reactor vessel (RV) is transferred from the outer wall of the RV through the mineral insulator (MI) cable. It also needs to transfer data through the penetration room penetrating the wall of the containment vessel (CNV). In addition, a power supply line for the data transmission must also be provided through the through-hole. Thus, wireless communication technology can help and provide huge benefits in developing SMRs. The absence and/or decrease of any through-holes and MI cables will improve the safety and cost related to SMRs. The wireless communication module needs power and the reactor will generate the decay heat, so thermoelectric elements will have key roles in supplying power. Here, we suggest a methodology to apply thermoelectric elements to supply power to wireless communication module under the SMR environment.

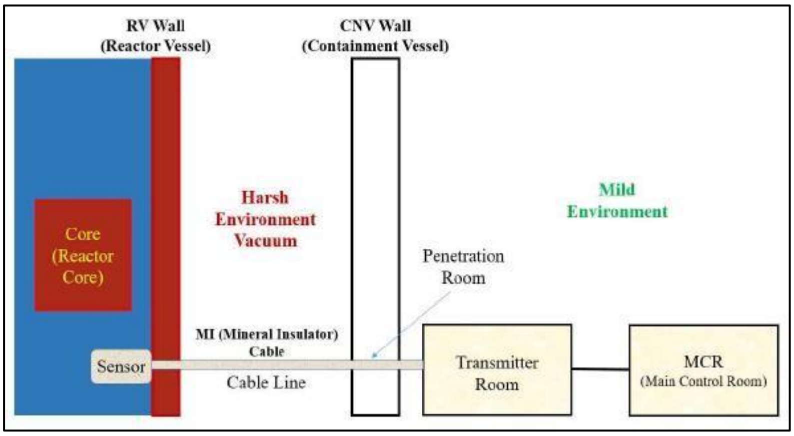

Fig. 1. Instrumentation and control line structure expected for small module reactors (SMRs).

Acknowledgment

We acknowledge the Korean government, Ministry of Science and ICT, for support (No. RS-202200144000).

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Calculating phase equilibrium properties of the reactive N_2O_4-NO_2 system

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Nowadays all power plants producing electricity use inert substances (for example, CO_2 and water) as working fluids in their thermodynamic cycle. The selection of the working fluid, which can be either a pure fluid or a mixture, is one of the main drivers towards the increase of their efficiency. However, such improvement starts revealing some limitations. The idea of the ERC REACHER project [1] is to use reacting fluids as working body instead of inert ones. That will allow the coupling of chemical reactions with classical thermal energy conversion. Preliminary calculations proved the promise of this approach [2]. One of such fluids may be dinitrogen tetroxide N_2O_4, which is able to rapidly decompose into 2 molecules of NO_2, and vice versa, according to the reversible reaction $N_2O_4 \leftrightarrow 2NO_2$. In order to assess the efficiency of a thermodynamic cycle operating with such a fluid, it is necessary to have a thermodynamic calculation tool enabling the determination of the thermodynamic properties of these fluids, and especially when more than one phase and chemical reactions simultaneously occur. The goal of this work is to provide a thermodynamic model describing the properties of the system in a wide range of temperatures and pressures, and a Fortran code available upon request, to perform these calculations.

Peng-Robinson equation of state combined with advanced mixing rules with a zero-residual excess Helmholtz energy model [3] is chosen to describe the N_2O_4-NO_2 system. The application of this equation of state requires as an input the knowledge of properties of pure N_2O_4 and NO_2 (their critical temperature, critical pressure, acentric factor). However, this system always exists as a mixture whose composition is dictated by chemical equilibrium. As a consequence, the thermodynamic properties of pure NO_2 and pure N_2O_4 are not measurable. In this work critical properties of N_2O_4 and NO_2 were estimated by molecular Monte Carlo simulations. Moreover, chemical equilibrium calculations in single phases are performed by modified RAND method [4]. This method is based on the Gibbs energy minimization under the material balance constraints. Calculations of VLE are based on finding a solution of the system of equations: for a given value of temperature, the system of equations describing properties of the phases, phase and chemical equilibria are solved for pressure, molar volumes and molar compositions of the phases. Calculated pressures were compared to the experimental values provided by DIPPR database. The coordinates of the unique critical point of the reacting system were calculated by a modification of an algorithm proposed for non-reactive systems [5].

Acknowledgment

The project has received funding from the European Research Council (ERC) under the European Union's Horizon Europe research and innovation programme (grant agreement No. 101040994).

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OS1-2 L1D – Monday, 11 Sep. 15.00-15.20 – ROOM N1

Measurement of the thermal conductivity at cryogenic temperature

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For storing and transporting liquid gas, it is useful to know the thermos-physical properties of materials. Liquid hydrogen is now an important subject of development. This study concerns the development of a device for characterizing thermal conductivity at low temperature. The device is based on a steady-state measurement of the thermal gradient on a bar of known section. The most commonly used method for measuring the thermal conductivity of metals is the guarded bar method. This is based on the heating of a metal bar in steady state and by measuring the temperature gradient. This method is unavoidable at high temperature where the radiation represents significant losses. At cryogenic temperature, the most significant losses are conductive and convective losses. In this study, the choice is to remove the thermal guard. To avoid exchanges with the air, the solution is to put the device in a high vacuum (1E-5mBar). We heat the sample with an electrical resistor on one side of the bar and we cool on the other side by plunging the enclosure in liquid nitrogen. The measurements made it possible to highlight the differences in behaviour of certain metals after having undergone annealing cycles. By using a sinusoidal heating regime, it was possible to identify the volumetric heat capacity as a function of the temperature. For composite materials, we measure the conductivity in two direction of the sample according to the direction of the fibre and according to the perpendicular direction of the fibre. Next step: 20K to reach the temperature for storing liquid hydrogen.

OS1-2 L1E – Monday, 11 Sep. 15.00-15.20 – ROOM M2

Thermophysical properties of wooden material exposed to high temperatures

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Timber is the only structural material of biological origin and is attracting growing attention in the construction industry within the last decades. It makes a significant contribution in terms of improving the overall energy efficiency of buildings and is nowadays not only used in traditional areas such as detached houses, but also in high-rise buildings in urban regions regarding a sustainable densification of urban areas. With the growing use of wooden material in high-rise buildings also fire protection requirements are increasing; the fire resistance of timber must be known and verified by cost and time consuming fire tests. The development of numerical models to predict the fire resistance of timber structures is a promising approach in terms of shortening the development cycles and a reduction of development costs. However, the development of numerical models requires an extensive knowledge of the temperature dependent thermophysical properties of wooden materials in different states (moist, dry, charred). Nevertheless, timber is the only structural material for which data of thermophysical properties at elevated temperatures are sparse or only treated in a limited manner, e.g., not considering the moisture content or the anisotropic behavior.

In this work thermophysical properties of wooden material in a wide temperature range are presented. The material was investigated in different states: moist, dry and charred, as well as during pyrolysis. Thermogravimetric measurements were performed to investigate the mass loss of wooden material at different heating rates up to 900°C. The thermal expansion was measured using a pushrod dilatometer. From thermal expansion and density at room temperature the temperature dependent density of the material was calculated over a wide temperature range. The (apparent) specific heat capacity of the wooden material was measured using differential scanning calorimetry and simultaneous thermal analysis; the material was investigated in the moist, dry and charred state, as well as during pyrolysis. The laser flash method was used to measure thermal diffusivity of the material. As wooden material shows an anisotropic behavior, thermal diffusivity, thermal expansion and thermal conductivity depend on the fiber direction. To consider the anisotropic behavior of wooden material, the corresponding measurements were performed on specimens cut along the fiber (longitudinal direction) and across the fiber (transversal direction).

Thermal conductivity was calculated from thermal diffusivity, specific heat capacity and density. These results are reported at room temperature of the moist material; from room temperature to the determined stability limit of wooden material at approx. 200°C of the dry material. Results of wooden material in the charred state are reported up to 900°C. From the measurements derived it can be concluded that the laser flash method is an efficient method to determine thermal diffusivity of wood and charred material in a wide temperature range and in selected orientations (in longitudinal and transversal direction). The thermal expansion of wooden material is significantly higher in transversal direction than in longitudinal direction, whereas thermal diffusivity and thermal conductivity are higher in fiber direction than across the fiber.

Acknowledgment

This work was supported by the "ACR Strategische Projekte" funding program coordinated by the Austrian Cooperative Research (ACR) and funded by the Austrian Federal Ministry of Labor and Economy (BMAW).

Effect of the size and coating agents of the NPs and the dispersion method on the stability time of non-aqueous nanofluids

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Nanofluids present several advantages as great thermal conductivities and high antifriction and antiwear capabilities. Both advantages are critical for the development of transmission fluids for electric vehicles [1], among other applications. However, the use of nanoadditives has an important disadvantage: the difficulty of achieving homogeneous and stable dispersions over time. In the case of non-aqueous fluids of low or no polarity, one of the most efficient strategies to avoid this problem is to coat the surface of the nanoadditive with long nonpolar chains through covalent bonds, which provides a high affinity of the nanoparticles with the base oils. For this purpose, modifying agents are used, being the most common organic acids, amines, polymers, organic phosphates, and silanes.

Several parameters affect the stability of the nanodispersions, such as the morphology, size, and concentration of the nanoparticles (NPs), or the viscosity of the base oil, the coating agent, and the preparation method. In this work, we have analyzed all these effects, based on our recent literature review [2]. Among the conclusions the following ones can be highlighted a) the dispersion methods involving evaporation of a volatile solvent led to greater stability results compared to the two-step method; b) the most common NP morphology in the more stable nanofluids was spherical with mean diameter lower than 20 nm (Carbon Quantum Dots, Cu, Ag, Pd, WS_2, Fe_3O_4, SiO_2); c) these nanofluids have concentrations lower than 1 wt% in most cases, being the highest stability (longer than 11 months) for the 0.015 wt% NP concentration [3]; d) the best stabilizing performance occurs when the modifying agent contains a nonpolar CH_3 termination and alkyl chains with at least 12 carbon atoms, such as oleic acid, oleylamine or polymers; e) the similarity in the polarities of the agent and of the base oil, thus following the rule 'like seeks like', which means that solvents will better disperse nanomaterials with similar surface chemistry; f) highly viscous base oils lead to better stabilities, but for low viscosity nanofluids high stability times can be achieved with small spherical NPs coated with an appropriate modifier agent.

Acknowledgment

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OS1-2 L2B – Monday, 11 Sep. 15.20-15.40 – ROOM O1

Coupled modelling of structural, thermodynamic and physicochemical properties of NaF-KF-UF_4 fuel salt

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Molten Salt Reactors (MSRs) are one of the Generation IV nuclear reactor designs that hold the promise of generating cheaper, stable and carbon-neutral energy in a modular and fast scalable fashion [1]. They contain the fuel in the liquid phase - a molten salt mixture of the fissile material, e.g., uranium fluoride. It is of the utmost importance to know the accurate thermo-physical and transport properties of the liquid fuel such as solidification temperature, density, viscosity, heat capacity, thermal conductivity and vapour pressure, utilized at different stages of the design, operation of the reactor and risk analysis.

The hazardous nature of the actinide-containing salts and the advances in the accuracy of *in-silico* research make *ab-initio* and thermodynamic modelling attractive in the study of ions within molten salt systems, reducing the experimental input.

The reference fuel salt for the MSR design is a NaF-KF-UF_4 eutectic mixture, known under an acronym as *FUNaK*, for which the comprehensive thermodynamic assessment and key physicochemical properties are still missing [2].

In this work we have focused on the development of coupled model of the structural and thermodynamic properties of the NaF-KF-UF_4 system from a microscopic (local structure) to macroscopic scale, combining experimental measurements, molecular dynamics (MD) simulations and a CALPHAD modelling approach. We have investigated the local structure of the selected relevant compositions in the NaF-UF_4 and KF-UF_4 binary sub-systems and ternary system NaF-KF-UF_4 using high temperature X-ray absorption spectroscopy (XAS) measurements using beamline at the synchrotron facility [3]. Moreover, we have coupled XAS with MD simulations using the Polarizable Ion Model (PIM) [4]. We have determined the coordination number of uranium, nature of neighbouring atoms and identified the formation of molecular complexes in the melt as a function of temperature, composition and change of the solvent. In overall, we validated molecular dynamics simulations of the binary and ternary systems and improved prediction capabilities of MD. An attempt to regress parameters of the U^{3+}-F^- PIM potential was taken.

MD simulations using the Polarizable Ion Model (PIM) allowed us to calculate relevant thermo-physical properties (density, thermal expansion, mixing enthalpies, heat capacity, viscosity, thermal conductivity), which are validated against the existing experimental data.

Finally, the experimental and simulation results served subsequently as input to build a comprehensive model of the NaF-KF-UF_4 system, linking structure and thermodynamic properties based on the CALPHAD methodology and modified quasi-chemical model in the quadruplet approximation (MQMQA) [5].

Acknowledgment

The authors would like to acknowledge Seaborg Technologies ApS for fully funding this project.

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Phase Behaviour of Isobutane + CO_2 and Isobutane + H_2 at Temperatures between 190 and 400 K and at Pressures up to 20 MPa

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Mixtures containing isobutane, carbon dioxide (CO_2), and/or hydrogen (H_2) are found in various industrial processes, from refrigeration to alkylation, and in pipelines. Understanding the thermophysical properties of these mixtures is essential for these processes. Isobutane and CO_2 are both considered green refrigerants and their use as pure fluids in low to moderate temperature refrigeration cycles has become well established [1], though use of the binary mixture has been hindered by limited data availability. New vapour liquid equilibrium (VLE) measurements of this system, and similarly of the isobutane + H_2 system, are needed to address the gaps in the literature. Therefore, in this study, measurements have been made of the phase behaviour of binary mixtures of isobutane with CO_2 or H_2, extending the range of available data for both mixtures. The results have been used to optimise model parameters for cubic and multiparameter equations of state (EoS). Compressed liquid density measurements have also been carried out for some mixtures.

The VLE measurements were performed using a static-analytical apparatus described previously [2], which has a maximum working pressure of 20 MPa and an operating temperature range of (183 to 473) K. Samples of the vapour and liquid were withdrawn through ROLSI electromagnetic sampling valves and carried to a gas chromatograph for analysis using a thermal conductivity detector. Density measurements were carried out using a vibrating tube densimeter, with an operating temperature range of (283 to 473) K and maximum working pressure of 70 MPa. The densimeter was calibrated with water and under vacuum. Mixtures were prepared gravimetrically in a variable volume cell using a high precision balance [3].

Modelling has been carried out primarily using variants of the Peng-Robinson EoS, with either van der Waals mixing rules or the modified Wong-Sandler mixing rule of Orbey and Sandler coupled with the NRTL free energy model [4]. The binary interaction parameters, expressed as functions of temperature, were regressed against the new experimental data and selected high-quality literature data. Some mixture reducing parameters for the multiparameter Helmholtz EoS in the REFPROP software were also regressed, as these were previously absent.

VLE measurements of the binary system isobutane + CO_2 were carried out along three isotherms (240, 280, and 310) K at pressures from the lower limit of the sampling system (~0.5 MPa) to the mixture critical pressure, finding good agreement with the literature data. VLE measurements of isobutane + H_2 were carried out on nine isotherms (190, 240, 280, 311, 339, 363, 375, 390, and 400) K at pressures up to 20 MPa, covering a much broader pT range than the one prior investigation [5]. Binary mixtures of isobutane + CO_2/H_2 were found to display type II and III behaviour, respectively, according to the classification of Scott and van Konynenburg [6]. In both cases, it is expected that the upper critical end point will not manifest before the formation of solid phases occurs.

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OS1-2 L2D – Monday, 11 Sep. 15.20-15.40 – ROOM N1

Probe based set up for the determination of local thermal diffusivity in soft materials

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Soft materials are one of the most promising materials for the control of the heat diffusion in a microscale as they have wide variety of its high order structures. As the integration of the electrical devices improved, the use of soft materials is sophisticated to the thermal management in a microscale, and the appropriate methodology to determine the thermal diffusivity in a microscale is demanded. Among the methodology for measuring thermal diffusivity, the method / analysis in which periodic heating is applied and the temperature response is treated as a temperature wave is widely used in soft materials as the temperature wave analysis method (TWA) [1-2]. In this study, a novel method to determine thermal diffusivity using cantilever nano thermocouples was developed based on the principle of the periodic heating method. The method was applied to the microstructure of soft materials fabricated with chemically amplified negative-tone photoresists to prove the concept.

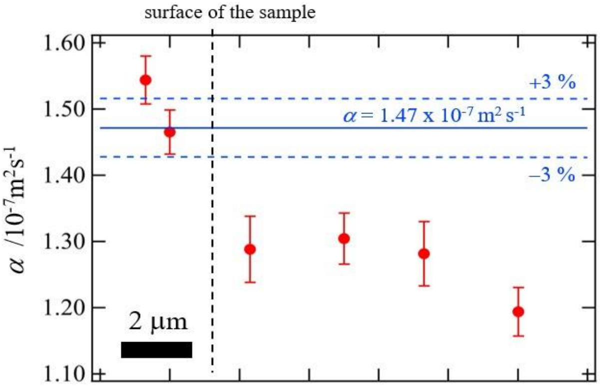

Fig. 1. The comparison of the estimated thermal diffusivity of the SU-8 micro cylinder-shaped structure measured during the completely discrete probe approach process. The error bar of each data point indicates the 66% confidential region of the analysis of the frequency dependency. Blue solid line is the total average valu e of the measurement, and the dashed lines indicate ±3% deviation from the average.

The proposed system consists of a thermocouple nanoprobe, micro heater arrays, and micro structured samples. The samples were fabricated using lithographic techniques on a micro heater, and also using a thin metal layer lift-off process. As thermal contact between the top surface of the sample and the probe tip is important for the contacttype measurements of thermal diffusivity, a protocol to ensure constant thermal contact was also developed in this study. Prior to measurements with AC Joule heating, the sample was DC heated and the DC temperature profile along the z-direction of the system was measured using the probe. The existence of an inflection point in the temperature profile perpendicular to the sample surface was found to be experimentally reproducible and this was measured and used as the reference position for the thermal diffusivity measurement [3].

The thermal diffusivity measurements were also performed at positions far from the surface and showed that the apparent thermal diffusivity changed significantly (Fig. 1).

In conclusion, a novel method for measuring thermal diffusivity using a thermocouple nanoprobe has been developed, with a protocol to ensure constant thermal contact between the probe tip and the sample surface.

Acknowledgment

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OS1-2 L2E – Monday, 11 Sep. 15.20-15.40 – ROOM M2

Thermo-physical characterization of plasters containing phase change materials

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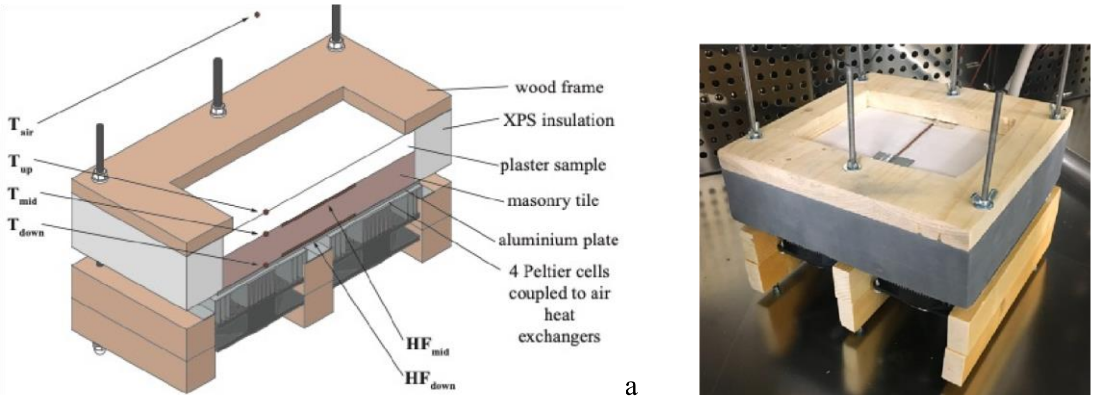
Phase change materials (PCMs) added into building materials have largely demonstrated their effect in improving the thermal properties by increasing the overall thermal capacity, thus contributing to the reduction of the energy demand for heating and/or cooling. Knowing the thermo-physical properties of the enhanced materials is essential to maximize the achievable effect, most importantly the latent heat capacity. The most used technique to do so is the Differential Scanning Calorimetry (DSC), but in case of inhomogeneous materials this method turns out to be poorly reliable, mainly due to the small amount of material usually needed to carry out the test [1]. To overcome the main limitations, an alternative set up was designed and realized as depicted in Fig.1, which allowed an indicative characterization of different plaster samples enhanced with granular PCM and also of a masonry tile underneath [2]. More specifically, plasters enhanced with 10% by mass of two different PCMs with melting temperatures of 28°C and 27°C, respectively, were considered (here named *10AS28* and *10TK27*).



a b

Fig. 1. (a) Materials and sensors position of the set up realized, (b) set up inside the climatic chamber.

At first, a steady-state test was conducted and constant thermal gradients, hence constant heat fluxes, were maintained through which the thermal conductivity of the materials used was estimated. Then, an unsteady-state test consisting of a two-step ramp was conducted: two ramps identical in terms of duration, heating and cooling rates, difference between the maximum and minimum temperatures were set, with the only difference that one was shifted from the other so that in one case the phase change of the material was fully included while in the other case it was completely avoided. The specific heat was estimated considering the total amount of heat absorbed by the material during the heating not in case of the phase change, while the latent heat was estimated by comparing the results obtained from the two ramps whose differences, since the ramps were identical, could only be attributed to the latent heat capacity of the PCM. The properties thus estimated are reported in Table 1, and with particular reference to latent heat, results showed a good agreement with values provided by the PCM suppliers.

Table 1. Plaster samples estimated thermal properties.

	ρ [kg/m^3]	λ [W/(m·K)]	c_p [kJ/(kg·K)]	Lh_{PCM} [kJ/kg]	$Lh_{plaster}$ [kJ/kg]
Reference (no PMC)	1646	0.31	0.89	-	-
10AS28	1522	0.28	1.07	92	6.8
10TK27	1365	0.24	1.29	160	11.9
Masonry tile	1608	0.52	0.83	-	-

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OS1-2 L3A – Monday, 11 Sep. 15.40-16.00 – ROOM O2

An approach to characterize the nanolayer for a nanofluid: thickness, density and molar mass

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The aim of this work is to develop a theoretical model to calculate the thickness, density and molar mass of the interfacial nanolayer around nanofluid particles. It is shown that its molar mass is equal to that of the base fluid. Expressions for nanolayer thickness and its density are derived. These properties of the nanolayer depend, in general, on temperature, on the nature of the base fluid, the nature of nanoparticles, their geometry and their concentration. It is shown that the model, under its hypothesis, does not support the equation of Pak and Cho [1]. The model is compared to other models from literature [2-4] and it is applied to nanofluids with nanoparticles of spherical geometry. An example is given in Table 1. With the prediction of the properties mentioned above it will be more achievable to predict other properties of the nanolayer.

Table 1. Values of the nanolayer properties of thickness, *t*, density, , molar volume, V^* , calculated for the nanofluid CuO (11 nm) + water, at different temperatures, *T*, at the pressure of 0.1 MPa at different nanoparticle concentrations. Nanolayer molar mass is that of the base fluid M^* 1.8015 10 kg · mol at all molar fractions, x_p.

weight fraction %	x_p	t /nm	ρ / kg□m^{-3}	V^* □10^6 m^3□mol^{-1}
T=283.15 K				
1	0.00228	0.89	2000	9.01
1.75	0.00402	0.28	2000	9.01
2.5	0.00577	0.05	2000	9.01
T=293.15 K				
1	0.00228	0.90	1997	9.02
1.75	0.00402	0.29	1997	9.02
2.5	0.00577	0.19	1997	9.02
T=303.15 K				
1	0.00228	1.08	1992	9.04
1.75	0.00402	0.42	1992	9.04
2.5	0.00577	0.29	1992	9.04

Acknowledgment

This work was founded by grant ED431C 2020-06 provided by the Xunta de Galicia (Spain) and the European Union H2020-MSCA-RISE-2019 PEPSA-MATE project.

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OS1-2 L3C – Monday, 11 Sep. 15.40-16.00 – ROOM N2

A Helmholtz Energy Equation of State (EOS) for N,N-Difluoromethane (DMF) And Development of Equations of State for Metals

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A first fundamental equation of state is presented for N, N-Dimethylformamide (DMF). DMF is an organic solvent used in many research settings and industrial processes, such as manufacturing polyurethane and pharmaceutical and agrochemical products. DMF is also used in the energy industry for manufacturing batteries and photovoltaic cells and in the semiconductor industry for manufacturing uniform semiconductor films. Despite the wide use of DMF, no fundamental equation of state had been developed until now. The equation is explicit in the Helmholtz energy with independent variables of temperature and density and comprises two parts. The first is the energy contribution of the ideal gas, representing properties at ideal states analytically formulated from a correlation for the ideal-gas isobaric heat capacity. The second part represents the residual contribution from the influence of intermolecular forces, which is determined empirically from experimental data. We will present newly developed fitting methods and plots of derived thermodynamic properties such as speed of sound, heat capacity, and the phase identification parameter illustrating that the equation shows physically correct behavior even when extrapolated beyond experimental data found in the literature. In addition to our recent equation of state development for the organic solvent DMF, we will present our current efforts to fit a fundamental equation of state for metallic elements.

Discussion on the current state and lessons learned will be presented as well as gaps in experimental data that could inhibit accuracy in regions of interest for industry applications.

Acknowledgment

Elizabeth G. Rasmussen acknowledges the NIST-NRC postdoctoral fellowship support administered by NIST in cooperation with the National Academies of Science, Engineering and Medicine (NASEM)/ National Research Council (NRC).

OS1-2 L3D – Monday, 11 Sep. 15.40-16.00 – ROOM N1

Development of a Micro Guarded Hot Plate apparatus for lab scale samples

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In this work, a micro guarded hot plate setup (micro-GHP) was developed in order to measure small samples with low and direction dependent thermal conductivity.

In the early stages of material development, samples are mostly small, thus requiring accordingly special measuring setups for all the relevant parameters. In the case of thermal conductivity, small specimen are mostly measured by means of transient methods such as flash method, hot-wire and hot-disc method. All these methods have specific limitations. Using the flash method a comparatively small specimen (roughly 5 mm to 20 mm in diameter) is used and requires additional measurement of the density and the specific heat. Furthermore, they are not suitable for materials with low thermal conductivity. The hot-wire technique measures radially around the wire and is not suitable for specimen with direction dependent thermal conductivity values. Finally, hot-disc sensors are also smaller (roughly 1 mm to 30 mm) leading to a spherical measuring geometry when measuring in the far field, again making measurement of materials with direction dependent thermal conductivity difficult.

The developed guarded hot-plate system has a total quadratic area of (5x5) cm² and a round metering area of $7.1 \cdot 10^{-4}$ m² and a 30 mm diameter. Due to its small size the apparatus can be easily transferred into various environments, such as a vacuum chamber for gas pressure dependent measurements or a climate chamber for temperature dependent measurements. It can measure solid specimens by clamping the stack of apparatus plates and specimen together or it can measure powders using a special encasement.

In this work, measurements on low conducting specimens, such as nano-porous polyurethane foam and silica aerogels are presented. The results are compared with the results from measurements performed on larger specimen of the same material using standard GHP and heat-flow-meter technique. A good agreement of the measured values within 10 % can be determined.

OS1-2 L3E – Monday, 11 Sep. 15.40-16.00 – ROOM M2

Thermal Assessment of Hollow Micro-Sphere Based Ceramic Composite for High Temperature Application

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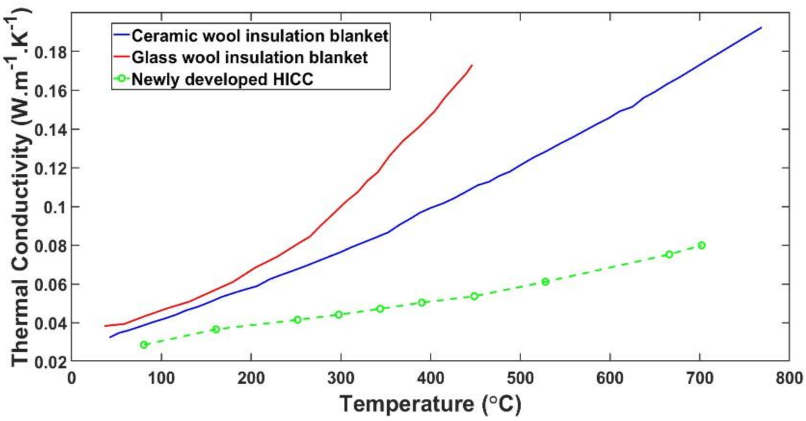

Figure 1: Comparison of high-temperature thermal conductivity of ceramic fibers insulation (ρ = 96 kg/m^3), glass wool blanket (98 kg/m^3) and HICC.

Thermal insulation materials are essential for restricting heat transfer and lowering overall energy usage. Hollow ceramic-microspheres (HCM) have recently gained popularity as insulation due to their high thermal resistance and thermal integrity at elevated temperature at low cost [1]. In this investigation, a heat-insulating ceramic composite (HICC), comprised of HCM beads, ceramic (alumina) fibre, and silica along with binding agents, was developed and characterised. With the help of a high-speed stirrer, the raw ingredients were mixed with water and a dispersing agent (Nonylphenol Ethoxylate) to make a homogenous mixture. The microstructural morphology of the composite was examined using scanning electron microscopy (SEM), and its thermal conductivity was measured to investigate the effects of alumina fibre and microspheres on its performance. Combining ceramic fibres and hollow particulates of different sizes to form multi-scale pore sizes significantly minimises high-temperature conductive and convective heat transport [2]. At temperatures between 100℃ and 700℃, the heat-insulating properties of the composite were compared to those of conventional insulation blanket. The result (Fig.1) shows that the thermal conductivity of the HICC is lower than that of the traditional inorganic insulating materials at the corresponding temperatures. Moreover, the developed composite was applied over a cylindrical high-temperature thermal storage (DN, 150 mm) to demonstrate its applicability as a high temperature thermal insulator. The surface temperatures were recorded in real-time. The result (Table-1) shows that the ceramic composite provided excellent insulating qualities; compared to the conventional ceramic-wool blanket, 65% less thick layer of HICC is required to provide the same thermal insulating performance. This research provides valuable insights into developing and characterising a new ceramic composite insulation material for high-temperature applications, offering substantial energy savings and reducing greenhouse gas emissions. Excellent flexibility and thermal insulating capabilities make HICC an appealing substitute for conventional inorganic insulation materials.

Table 1. Comparison of ceramic blanket and HICC applied over high temperature thermal storage.

Insulation material	Pipe internal surface avg. temperature (°C)	Average outside temperature (°C)	Ambient temperature (°C)	Thickness (mm)	Weight (kg/m)
Ceramic wool blanket	630	59.6	33	154.2	19.6
HICC		54.4		51.3	14.1

Acknowledgment

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OS1-3 L1A – Monday, 11 Sep. 16.30-16.50 – ROOM O2

LH2 Facility: Advanced Measurement of Ortho-Para Conversion and Boil-Off Gas in Liquid H2

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Current hydrogen liquefaction technologies are limited by interconnected and related challenges, including ortho-para (O-P) hydrogen conversion and boil-off gas management. The substantial deficiency of LH2-relevant experimental data regarding the catalyzed O-P conversion kinetics and boil-off gas (BOG) during liquefaction must be addressed and validated. This work presents a unique laboratory capability at the University of Western Australia (UWA) that has been recently developed and consists of two major sections: (A) Ortho-para (O-P) hydrogen conversion and (B) hydrogen liquefier and LH2 boil-off monitoring, as shown in Figure 1. A singlestage cryocooler is deployed to enable ortho-para measurements at various amounts of catalyst, temperatures ranging between (350 and 30) K, and pressure up to 5 MPa, where ortho-para ratio is monitored by in-situ Raman spectroscopy using in-line fibre optic probe. A dual-stage cryocooler is used to liquefy hydrogen (at around 20 K and 1 bar), which is then transferred to a BOG test cell, where temperature stratification and boiloff rates is studied at different amounts of heat ingress, liquid level, and system pressure. The BOG testing rig has been fabricated, which includes vacuum shields (aluminum and copper cans) and LH2 stainless steel cell. Detailed thermal analyses were performed to minimize heat ingress into the hydrogen liquefier and BOG testing cell. The results were directly applied to the material selection, vacuum considerations, cooling capacity, instrumentation, and the dimension of every component. Overall, the LH2Facility design temperature is 20-350 K with the maximum pressure and volume of 5 MPa and 7 ml for (i) o-p conversion cell; and (ii) 1 MPa and 7 L for the boil-off testing cell. The design, engineering drawings, HAZOP, safety review, SOP, and construction of the LH2Facility have been finalized, and the apparatus is currently being commissioned and tested.

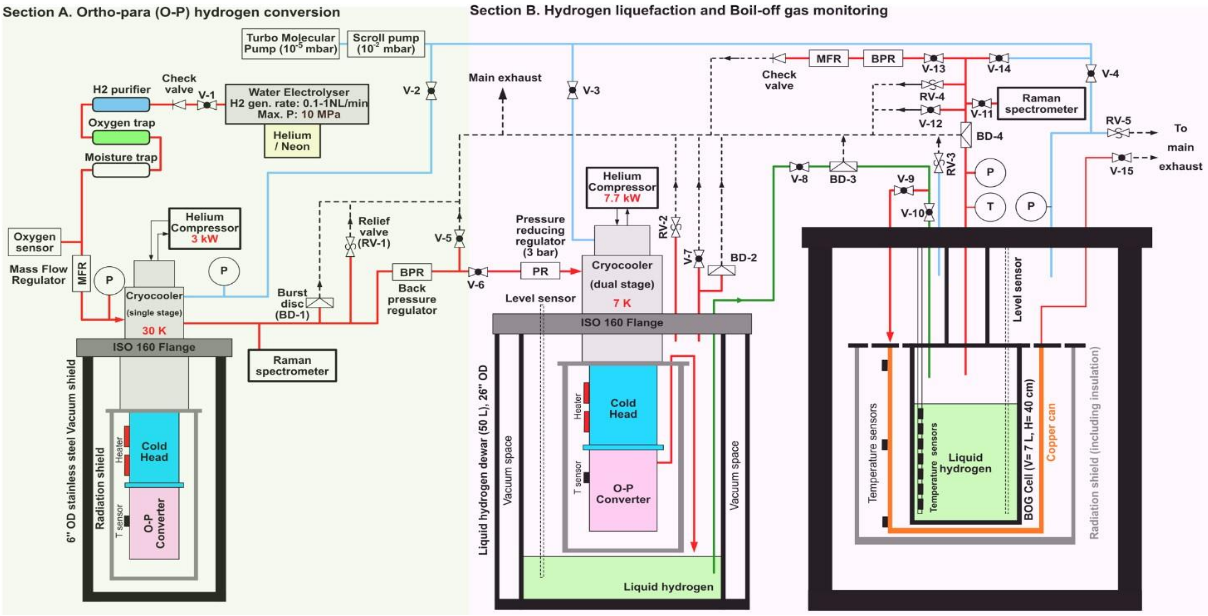

Fig. 1. A schematic diagram of the proposed LH2 and ortho-para conversion apparatus.

Acknowledgment

This work was supported by the Future Energy Exports Cooperative Research Centre, whose activities are funded by the Australian Government's Cooperative Research Centre Program.

Challenging ab initio calculations - Measurements of thermophysical properties of gases with dielectric-constant gas thermometry

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Dielectric-constant gas thermometry (DCGT), developed in the seventies in the U.K., and later improved by PTB, is now a well-established primary-thermometry method and, as part of the *Mise en Practique for the definition of the kelvin in the SI*, an allowed way of realizing the base unit kelvin. The basic idea of DCGT is to replace the density in the state equation of a gas by the dielectric constant and to measure this constant by the capacitance changes of a capacitor filled with gas at different pressures and constant temperature (measurement of isotherms). For a real gas, both input equations (gas and Clausius-Mossotti equation) are power series containing different virial coefficients. Historically, the method was applied in the low-temperature range at low pressures, but recently, due to the work on the determination of the Boltzmann constant at the triple point of water (TPW, 273.16 K) [1], the temperature and pressure range was extended. The extreme demands concerning pressure, capacitance and temperature measurement led to unique experimental capabilities at PTB. Recent measurements with argon, neon and helium have been performed at the TPW up to pressures of 7 MPa [2]. Furthermore, for neon and helium, isotherms with low density (pressures up to 0.3 MPa) were measured recently in the complete temperature range from their normal boiling-point temperatures to 273 K [3,4]. The results for the virial coefficients deduced from these data sets are compared against latest ab initio calculations. Until now, the most challenging test for ab initio calculations is an alternative pressure standard using helium [5, 6]. In summary, all these test show clearly, where ab initio theories have taken the lead and where experiment is still one step ahead.

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OS1-3 L1D – Monday, 11 Sep. 16.30-16.50 – ROOM N1

Thermophysical properties and microstructure of lead-free solder joints reinforced by metal deposited nanoparticles

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A rapid development of the electronics industry and the corresponding demands for the miniaturization of soldered joints require the development of new lead-free solders (LFS) with improved thermophysical and mechanical properties [1]. Ternary Sn-Ag-Cu (SAC) alloys of eutectic or near-eutectic compositions are considered the most promising and are widely used as solder materials. In this regard, it is necessary to improve various thermophysical and mechanical properties, such as electrical and thermal conductivity, thermal resistance, fatigue and creep resistance, yield strength, etc.

The reliability of solder joints is especially determined by the intermetallic compound (IMC) formed at the joint interface. Unlike traditional lead-tin solders, SAC solders generally have a higher melting point and tin content. Therefore, formation and growth of the IMC layer occurs faster in the SAC solder joints, which leads to brittle fractures and a decrease in the service life of the joints due to thermal fatigue.

To improve the properties and strengthen the base solder matrix, various nano-sized admixtures, in particular ceramic and carbon, are added [2]. In contrast to metal nanoparticles, they are non-wettable by metal melts. To solve this problem, metallic coatings is applied to their surface to form core-shell structures and to improve adaptation to the solder matrices. As a result, the metal-coated layer forms a strong "bridge" that reacted with the LFS matrix to form an intermetallic layer during soldering. Another important requirement for the LFS application is their reliability in a wide range of operating temperatures, including sub-zero temperatures. In this work, the effect of carbon nanotubes, carbon nanospheres, ceramic TiO_2, SiO_2, Al_2O_3 and ZrO_2 admixtures coated with Ni, Au, Au-Pd and Pt on the properties and microstructure of the SACbased solder joints was studied both at elevated and sub-zero temperatures.

Acknowledgment

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OS1-3 L1E – Monday, 11 Sep. 16.30-16.50 – ROOM M2

Geometric optimization of thermochromic meta-surfaces using machine learning

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Radiative heat transfer among the Sun, Earth, and Space has a vital role in determining the temperature on the surface of the Earth. A device temperature-dependently modulating its spectral emissivity has the potential to reduce loads for air-conditioners in buildings. For example, promoting sunlight absorption and infrared reflection is helpful to keep warm in winter, while both need suppression for cooling in summer. A meta-surface consisting of vanadium dioxide (VO_2), which exhibits metallic or glass-like characteristics at a high or low temperature, has been proposed to control emissivity in a range from visible to infrared wavelength [1]. In this study, an improved geometry consisting of titania (TiO_2) and VO_2 is proposed to reduce the phase transition temperature of VO_2 to room temperature [2]. As shown in Fig. 1, the meta-surface has multiple VO_2–TiO_2 slabs with different widths periodically placed on the gold substrate layer. The thicknesses of VO_2 and TiO_2 layers affect sunlight absorption. At the same time, the width of each slab determines the resonant wavelength of the short-range surface plasmon polariton relating to the infrared emission [3]. Since geometric parameters complicatedly correlate the broadband emissivity, repetitive numerical simulations utilizing Bayesian optimization were conducted to obtain the best combination of parameters. Although the total number of candidates for the current condition reaches up to dozens of thousand, the machine learning following the Bayesian process contributed efficiently to suppressing the consumption of computational resources. Eventually, the obtained meta-surface exhibited much better emissivity than the previous work for any temperature condition.

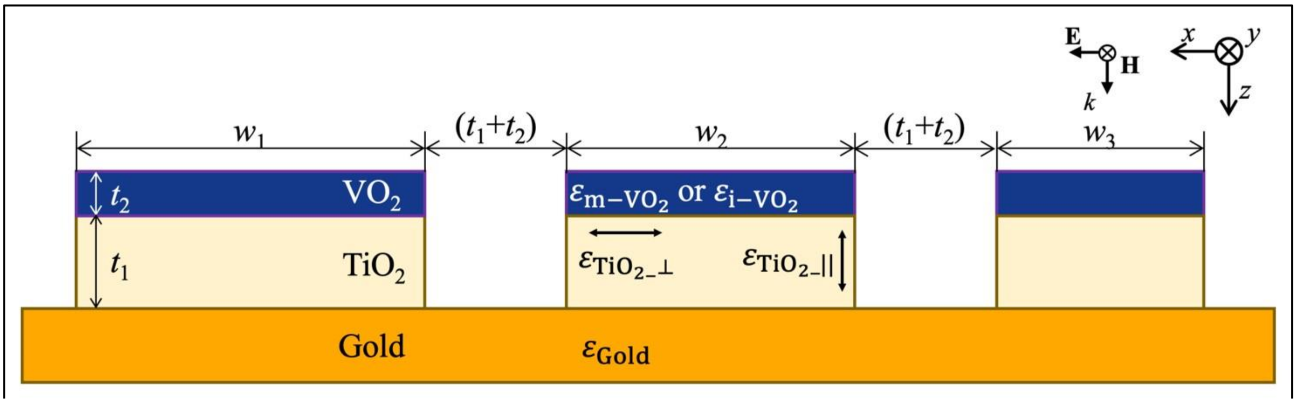

Fig. 1. Schematic image of the meta-surface with multiple slabs.

Acknowledgment

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OS1-3 L2A – Monday, 11 Sep. 16.50-17.10 – ROOM O2

Crystal Structure Behavior Determination of Oxygen and Argon for Safer Liquefied Hydrogen Delivery

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Solid oxygen and argon are of increasing interest to the growing hydrogen economy – part of the efforts towards net-zero carbon emissions. This is because these two molecules are among the main impurities in hydrogen production via water vapour plasmolysis [1]. They pose risks to freeze out in hydrogen liquefaction, a common process to transport large amount of hydrogen in the absence of pipelines. Accurate descriptions of oxygen and argon in the solid phase at cryogenic temperatures relevant to hydrogen liquefaction are required to prevent unanticipated shutdown of liquefaction plants.

Unfortunately, there are limited data for solid oxygen and argon, let alone at conditions related to the liquefied hydrogen. Under most circumstances, engineers have to rely on empirical methods for rough estimations. These approaches, as expected, are untrustworthy: the calculated freeze-out temperatures of solid oxygen deviate by up to 23 K from the available experimental data [2]. Moreover, there are completely no data for solid argon solubility in hydrogen, and the available datasets for oxygen are self-contradictory. Thus, reliable measurements of crystal oxygen and argon thermodynamic properties are in demand for accurate solid freeze-out prediction.

This work generates accurate crystal structure behaviour of oxygen and argon via the high-intensity diffraction instrument Wombat from liquefied hydrogen operation temperatures to the melting point. The achieved results, with the measurement uncertainty estimated to be around 0.4% (k = 1), resolve the data scarcity issue and identify the outliers from the old literature. The measured cell volumes of oxygen and argon will then be utilised to construct reference equation of state for the two crystals so that the solid freeze-out temperature will be described with accuracy to provide reliable guidance to the industry.

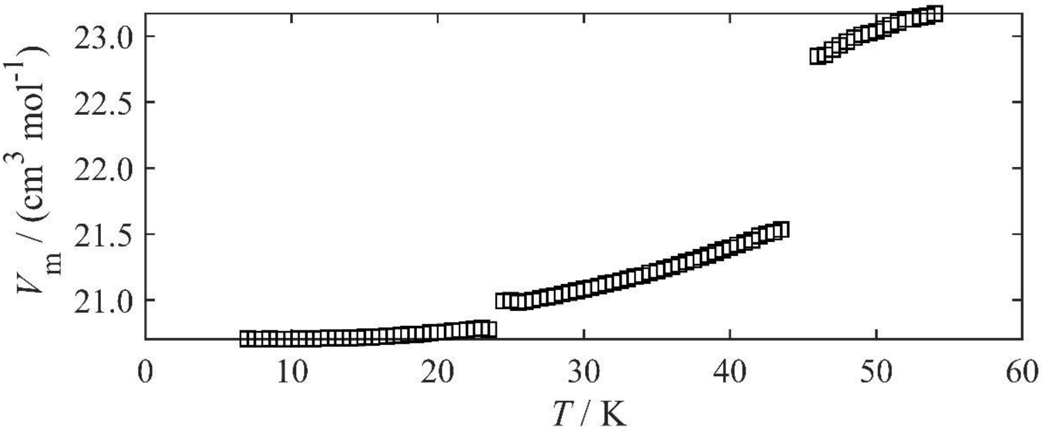

Fig. 1. Extracted volume per molecule for oxygen diffraction.

Acknowledgment

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OS1-3 L2C – Monday, 11 Sep. 16.50-17.10 – ROOM N2

How well does the Mie potential describe the thermodynamic properties of spherical fluids?

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The determination of thermodynamic properties using experiments in the laboratory is a time-consuming and expensive process. Especially the handling of toxic, corrosive or explosive substances can significantly impede the measurements. Molecular simulation provides a viable alternative to access thermodynamic properties without the use of laboratory experiments. These simulations are based on potential functions that characterize the behavior of molecules based on their position relative to each other. For the development of more complex molecular models, it is essential to understand the basic properties and behavior of spherical structures in detail. Intermolecular forces between approximately spherical molecules, such as those of noble gases[1] and methane, can be approximated by radially symmetric pair potentials. A well-known interaction function is the (λ-6) Mie potential that combines London-dispersion and a variable repulsion term between two molecules. With this potential model, a fluid is characterized by its parameters for size σ, energy ε, and repulsion λ.

The generalized fundamental equation of state of Pohl *et al.*[2], developed on the basis of simulation data, is used to discuss the influence of repulsive forces between spherical molecules on thermodynamic properties. The noble gases neon, argon, krypton, and xenon as well as the spherical alkane methane are analyzed. Investigations are made with respect to saturation properties, such as the vapor pressure, and with respect to properties in the homogeneous state region.

Pareto optimization is applied to determine suitable parameters for σ, ε, and λ, considering experimentally determined fluid properties from the literature. The calculated potential parameters are compared with values from the literature. It is shown that the application of pair potentials does not describe the thermodynamic properties of spherical fluids with sufficient accuracy.

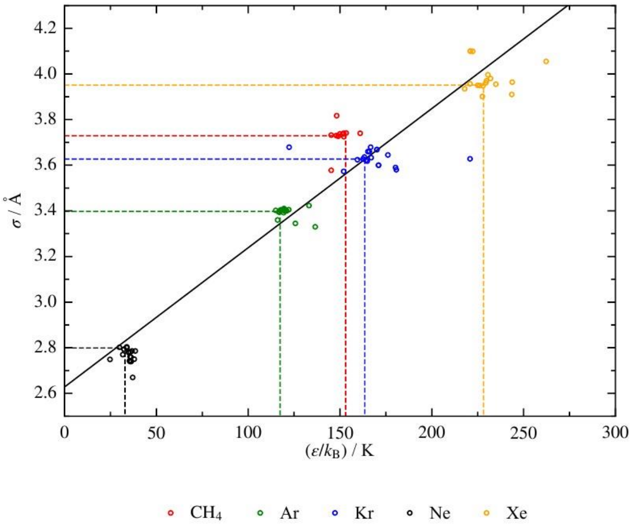

Fig. 1 Potential parameters σ and ε for selected noble gases and methane. The dashed lines indicate the values estimated in this work. The bullets show values from different literature sources.

Acknowledgment

This work was supported by Deutsche Forschungsgemeinschaft (DFG) [Grant Nos. SP 507/15 and VR 6/16].

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OS1-3 L2D – Monday, 11 Sep. 16.50-17.10 – ROOM N1

Thermophysical Characterization of 1D Micro/Nanoscale Materials

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The last decade has witnessed significant advances in miniaturization in material synthesis and manufacturing from micron to nano, even atomic scales. Great challenges have arisen in thermal science in terms of characterizing material properties and probing thermal and mechanical behavior during material synthesis/manufacturing at the micro/nano/atomic scale. This talk will focus on our experimental work to measure the thermophysical properties (e.g. thermal diffusivity, thermal conductivity, and specific heat) of free-standing fiber/wires/films with a thickness from micron down to single atomic layer thickness. Four new techniques will be introduced: transient electro-thermal (TET), transient photo-electro-thermal (TPET), pulsed laser thermal relaxation (PLTR), and energy transport state-resolved Raman (ET-Raman) techniques. Discussions will be presented on the advantages and disadvantages of each technique. Results will be provided for samples down to single atomic layer thickness. Our down to 10 K measurement has enabled determination of residual thermal reffusivity at the 0 K limit and determine the structure domain size for energy carrier scattering.

OS1-3 L2E – Monday, 11 Sep. 16.50-17.10 – ROOM M2

Metrological Framework for Passive Radiative Cooling Technologies and Development of Setups for Reproducible In-Field Performance Testing

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Almost 20 % of the global electricity consumption is caused by cooling systems. As the demand for cooling is expected to grow tenfold by 2050, improving the efficiency of cooling systems plays a critical role in addressing the global climate challenge. Passive Radiative Cooling (PRC) materials, which can dissipate heat into the surrounding as thermal radiation (especially through the atmospheric infrared window between 8 µm and 13 µm) have recently emerged. Hence, the project PaRaMetriC (Metrological Framework for Passive Radiative Cooling Technologies) aims to develop a comprehensive metrological framework with standardized performance indicators and testing protocols to enable comparable evaluation of their cooling performance on-site and the determination of potential energy savings that could derive from the deployment of such technologies.

One work package within this project deals with the design of a testing setup and the development of a protocol for determining the figures of merit of candidate PRC materials by in-field measurements with a relative uncertainty below 10 %. For this purpose, on-site prototype setups are planned to be realized in different climatic regions, e.g. Barcelona (maritime Mediterranean climate), Madrid (continental Mediterranean climate), Torino (warm temperate climate) and Würzburg (moderate temperate climate), in order to cover a wide range of environmental conditions. With these prototype setups, measurements will be performed on candidate benchmark materials. Additionally, the environmental and atmospheric conditions (temperature, solar irradiance, humidity, wind speed, etc.) will be monitored during the measurements using appropriate sensors. Up to now, the design work has been started.

This presentation gives an overview of the PaRaMetriC project and provides an outlook on the aimed future activities regarding the setup for in-field measurements, including the configuration of the setup (thermal insulation, thermal load, etc.), the selection of appropriate sensors and the implementation of PRC materials. Furthermore, previous work will be presented. So far, first PRC materials with high solar reflectance and high thermal emittance have been prepared. Beside the infrared-optical properties, the thermal conductivity of the prepared layers has also been measured. At these samples, the derived surface temperature has been measured for sky-facing surfaces in dependence on the surrounding conditions. Together with measurements on reference materials, the correlation of the surface temperature with the solar reflectance index (SRI) has been investigated. The SRI value has been determined by measurements of the solar reflectance and thermal emittance of the prepared PRC materials according to ASTM E 1980 - 11. As a preliminary result, a temperature drop below ambient air temperature has been recorded for some prepared PRC materials even during the day in sunshine.

Acknowledgment

The project 21GRD03 PaRaMetriC receives funding from the European Partnership on Metrology, cofinanced by the European Union's Horizon Europe Research and Innovation Programme and from the Participating States.

Solid-State hydrogen storage: Magnesium hydride with MXene catalysts in metal hydride tanks

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Increasing energy consumption has led to a rise in carbon emissions and depletion of natural resources, highlighting the need for renewable energy sources and energy storage systems. Hydrogen storage has emerged as a promising and environmentally friendly alternative for energy storage in various applications, owing to its impressive energy density and ability to produce zero emissions during usage. Solid-state hydrogen storage systems, specifically metal hydrides, have advantages over traditional methods in terms of safety, efficiency, and cost-effectiveness. However, the slow hydrogen release kinetics of magnesium hydride (MgH_2) has hindered its practical use. Recent studies have shown that MXenes, a group of two-dimensional transition metal carbides, nitrides, and carbonitrides, exhibit high surface area and superior electronic conductivity and can function as catalysts for hydrogen storage, specifically Ti_3C_2 MXene. This project aims to study an offgrid photovoltaic-hydrogen system with a metal hydride storage tank integrated with MXene catalysts for hydrogen storage. The system generates electricity through solar panels, produces hydrogen using an electrolyzer, stores it in the metal hydride tank until required, and converts it back to electrical energy using a fuel cell. The experimental results demonstrate that the addition of Ti_3C_2 MXene to MgH_2 resulted in enhanced dehydrogenation kinetics and a lower starting dehydrogenation temperature compared to pure MgH_2. The onset dehydrogenation temperature was reduced by 5°C, indicating that Ti_3C_2 MXene acted as a catalyst, improving the dehydrogenation performance of MgH_2. Additionally, the apparent activation energy of MgH_2-Ti_3C_2 was reduced by 18%, signifying that Ti_3C_2 MXene effectively lowered the energy barrier required for the dehydrogenation process. These findings suggest that Ti_3C_2 MXene holds promise as a catalyst for enhancing the hydrogen storage properties of MgH_2. In our future research, we will focus on testing the application of MgH_2-Ti_3C_2 for hydrogen storage in buildings, with a focus on its potential for integration with off-grid photovoltaic-hydrogen systems, as part of sustainable building energy storage solutions.

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Calculation of thermodynamic properties of neon using path integral Monte Carlo simulations and *ab initio* potentials

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Based on Feynman's path integral formulation of quantum mechanics, nuclei can be treated quantum mechanically in a classical Monte Carlo simulation by representing particles as finite ring polymers within the classical isomorphism. In this work, the methodology of Lustig [1-2], with which rigorous expressions for all thermodynamic properties can be derived in any ensemble, is extended to the path integral framework for the canonical (*NVT*) and isobaric-isothermal (*NpT*) ensembles. With the derived expressions, thermodynamic properties such as the speed of sound or Joule-Thomson coefficient can be calculated fully incorporating quantum effects without uncontrolled approximations. The derived expressions are verified by simulations of neon at selected state points at temperatures below 100 K using highly accurate *ab initio* potentials for pairwise and nonadditive three-body interactions [3-4]. We observe good agreement of our results with experimental data, which demonstrates the necessity to consider quantum effects as well as non-additive three-body interactions when predicting thermodynamic properties by solely theoretical means.

Acknowledgments

This work has been funded by dtec.bw – Digitalization and Technology Research Center of the Bundeswehr (project H2MIXPROP). Computational resources (HPC cluster HSUper) have been provided by the project hpc.bw, also funded by dtec.bw. dtec.bw is funded by the European Union – NextGenerationEU.

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OS1-3 L3D – Monday, 11 Sep. 17.10-17.30 – ROOM N1

Uncertainty evaluation associated with the measurement of thermal conductivity at the nanoscale

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Thermal management is a key issue for miniaturizing electronics devices (overheating, local hot spots). To anticipate these failures, manufacturers require knowledge of the thermal properties of the used materials at the nanoscale. Indeed, thermal properties of materials at nanoscale can be completely different from those of the bulk materials. The Scanning Thermal Microscopy (SThM) is a relevant technique to perform local measurements of thermal properties with a high spatial resolution of tens of nm [1]. It has been widely used to characterize various types of nanostructured materials such as nanowires, 2D materials, nanoparticles [2].
In this contribution, we use the SThM in its active mode to determine the local thermal conductivity of materials by studying the variation of the electrical resistance of a hot thermoresistive tip in contact with a sample. The proposed calibration approach relies on three main steps: 1) the evaluation of the uncertainty associated with SThM measurements; 2) the establishment of a theoretical model of the calibration curve that describes the link between the thermal conductivity and the variation of electrical resistance based on heat transfers between the probe and the sample.3) the development of a Bayesian approach to estimate the calibration curve with uncertainty on both axes and to predict the thermal conductivity of unknown materials and their associated uncertainty.
In our application, the experimental calibration curve (Fig. 1) is determined for our SThM Pd probes on a set of samples with well-known thermal conductivity (5% relative expanded uncertainty). We show that this methodology allows to evaluate the range of sensitivity of the SThM technique for quantitative thermal conductivity measurements and the uncertainty associated to the measurements as well as to identify main influencing factors in order to decrease the uncertainty.

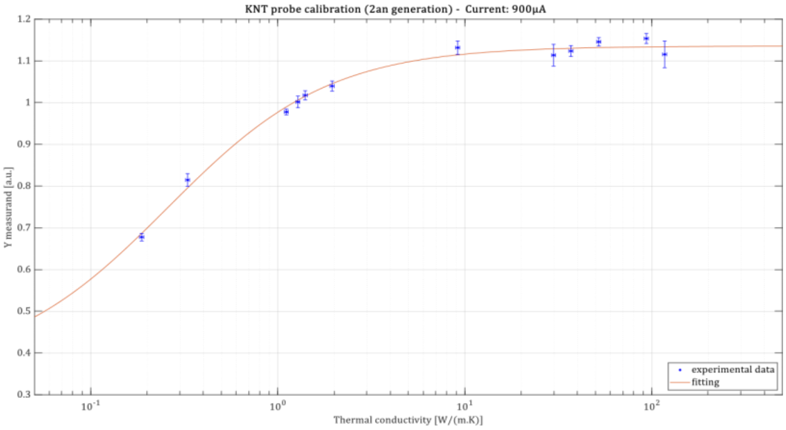

Fig. 1. Experimental calibration curve determined for KNT probe.
Uncertainty bars associated to experimental data are standard uncertainty.

Acknowledgment

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Emissivity and Reflectivity Measurements for Passive Radiative Cooling Technologies

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The project 21GRD03 PaRaMetriC is a three-year EU research project aiming to develop a metrological framework for comparable performance evaluations of passive radiative cooling (PRC) technologies. Effective optimization and development of these technologies is one of the keys to improving the global climate challenge since cooling systems are responsible for nearly 20 % of electricity consumption and 10 % of greenhouse gas emissions worldwide. Considering the inevitable growth of the cooling needs, and consequently the electricity consumption, the PRC technologies will necessarily have long-term economic, social and environmental impacts. Thanks to their optical properties, PRC materials can effectively reflect solar radiation but at the same time dissipate heat through the 8 μm to 13 μm infrared transparency window by using space as a cold and renewable heat sink. This makes it possible to achieve sub-ambient temperatures even in direct sunlight without using any electricity.

As the leader of a work package, the Physikalisch-Technische Bundesanstalt, together with partners of the PaRaMetriC project, will develop accurate and traceable methods to determine the thermophysical properties of PRC materials such as reflectance and emittance in the broad spectral range from 250 nm to 50 μm, encompassing both the solar spectrum (250 nm – 2500 nm) and the infrared transparency window of the atmosphere (8 μm – 13 μm) with an absolute uncertainty of less than 0.03. For this purpose, several candidate benchmark passive cooling materials will be selected and measured with various approaches by several partners. Characterizations of, and comparisons between, reference and end-user measurement techniques applied for the measurements of selected PRC materials will not only allow accurate determination of the thermophysical properties, but also identification of measurement problems and suitable approaches. Scientific community and industrial sector will benefit from new measurement data as well as guidelines developed within the project.

Acknowledgment

The project 21GRD03 PaRaMetriC receives funding from the European Partnership on Metrology, co-financed by the European Union's Horizon Europe Research and Innovation Programme and from the Participating States.

OS1-3 L4A – Monday, 11 Sep. 17.30-17.50 – ROOM O2

Cryogenic Microwave Resonant Sensors for Inline Measurements of *ortho-para* Hydrogen Composition and Catalyst Kinetics

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With the growing demand for a cleaner energy future, hydrogen has emerged as a viable option to transport, store, and utilise decarbonised energy. Due to the low energy density of hydrogen in its ambient gaseous phase, denser forms, including liquid hydrogen, are actively being developed to reduce the delivered cost of hydrogen. In the case of liquid hydrogen, the extreme cryogenic conditions required for liquefaction (20 Kelvin) presents a significant challenge in designing efficient and low-cost liquefaction processes. This challenge is compounded by the unique quantum behaviour of hydrogen associated with each molecule's isomer state; to achieve stable equilibrium in the cryogenic liquid phase, the majority fraction of ambient hydrogen (of the *ortho*-hydrogen isomer state) is required to undergo isomer conversion (to *para*-hydrogen). This isomer conversion is an exothermic process with enthalpy greater than what is required for hydrogen vaporisation. The natural conversion to the equilibrium state is slow (on the magnitude of days); thus if liquid hydrogen is produced in the unconverted state, natural conversion has the potential to cause uncontrolled boil-off and product losses. Catalysts are used within industrial liquefiers to reduce the isomer conversion time (by around five orders of magnitude), enabling production of equilibrium liquid hydrogen product. Whilst these catalysts are effective, the kinetics of how they operate are not well understood. This is due both to the limited data available [1], and the unestablished effect of impurities and poisoning on catalyst performance [2]. Overall, understanding the impact of hydrogen's isomer state on liquid hydrogen production and utilisation requires accurate measurement instruments. Currently, the most accurate and widely used sensors require sampling and have limited applicably in an industrial context due to their high cost, regular requirement for calibration, and specific operating ranges.

In this work, we introduce a novel microwave resonant cavity sensor capable of real-time inline *ortho-para* hydrogen compositional measurements at high pressures and down to cryogenic temperatures. Microwave resonant cavities have been applied to a range of fluid property measurements, each one optimised to sense the property or behaviour of interest [3-4]. Microwave cavities are well suited to industrial sensing as they are robust with no moving parts, and their readout can be easily automated. They are advantageous compared to other *orthopara* isomer sensors due to their extensive operating range of temperature and pressure, robust construction with no moving parts, and relatively low cost. As will be presented in this work, microwave resonant cavities are capable of extremely precise measurements, allowing the small differences between *ortho* and *para*-hydrogen's properties to be resolved, particularly for optimised cavity designs. Along with the novel sensor, we will introduce an integrated experimental cryogenic apparatus that has been established at the University of Western Australia (UWA) for characterising the kinetics of catalysts used in hydrogen isomer conversion. The test system includes Raman spectroscopy of cryogenically converted hydrogen, to independently validate the performance of the novel microwave sensing technology and catalysts being developed and studied. We will present data showing in-situ monitoring of hydrogen isomer conversion at temperatures below 20 Kelvin, and characterisation of the kinetics of a hydrous ferric oxide catalyst at temperatures down to 40 Kelvin.

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OS1-3 L4D – Monday, 11 Sep. 17.30-17.50 – ROOM N1

Measurement of 2D-distribution of water rotational relaxation time by short-wave infrared (SWIR) micro spectroscopy

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Water is essential for biomolecules to perform their functions by mediating physiochemical reactions. In particular, the water rotational relaxation time is an important index for estimating such functions, e.g. the degradation rate of proteins has the scaling law relationship with the solvent water rotational relaxation time [1], and the red oak seeds lose their desiccation tolerance when the rotational relaxation time of their retained water is longer than critical value [2]. The authors' group have recently shown that the SWIR spectroscopy could exclusively measure the water rotational relaxation time equivalent to the dielectric relaxation time of water as well as the amount of water in solutions or biological matters [3-4]. Applying this technique to an infrared micro-spectroscope, the 2D-distributions of the molarity and rotational relaxation times of water in a sample can be obtained with a spatial resolution of several tens of micrometres.

A 0.25mm thick glass or an alginate bead (c.a. ϕ2mm, Na-Alg 1wt% and $CaCl_2$ 10wt%) was enclosed in pure water between two parallel CaF_2 window plates in a 0.4mm distance for SWIR micro-spectroscopy. The 2Ddistributions of molarity and rotational relaxation time of water averaged along the thickness direction of the sample are shown in Figures 1 and 2 respectively. Water molarity of the sample in Figure 1 is lower than that in Figure 2 because glass does not absorb water. On the other hand, the water rotational relaxation time of the sample in Figure 1 is identical to surrounding pure water, whereas that in Figure 2 is c.a. 0.4psec slower than that of surrounding pure water because the retained water in the alginate bead includes bound water.

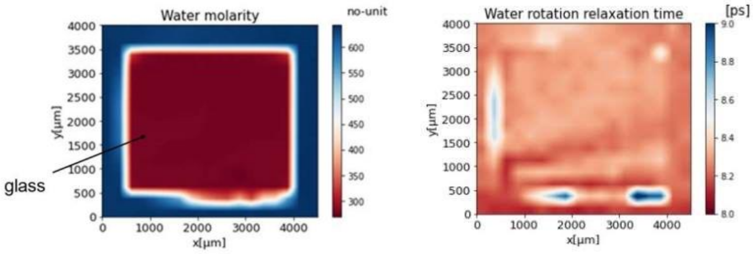

Fig.1 The 2D-distribution of water molarity and water rotational relaxation time for glass

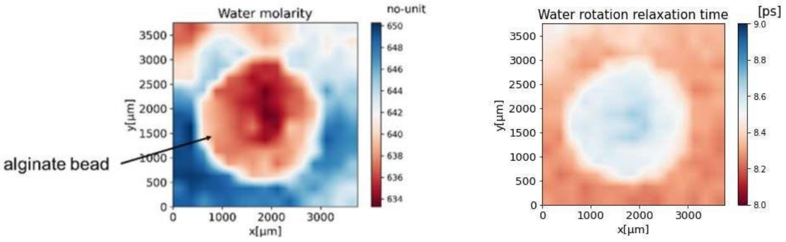

Fig.2 The 2D-distribution of water molarity and water rotational relaxation time of alginate bead

Acknowledgment

This work was supported by JST, the establishment of University fellowships towards the creation of science technology innovation, Grant Number JPMJFS2108 and a part of JSPS Grant (22H01409).

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Solar Spectrum Reflectometer measurements on profiled and small samples: design and testing of adapters

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Summer overheating, one of the main causes of the Urban Heat Island effect, can be limited through solar reflective materials which remains cold under solar radiation. These materials are characterized by a high solar reflectance, the ratio between reflected and incident solar radiation, and a high thermal emissivity, the ratio of the radiant emittance of heat of a surface to that of a standard blackbody. In particular, solar reflectance can be measured in three different ways: the use of pyranometers or albedometers are regulated by ASTM E1918 [1], the use of a spectrophotometer with an integrating sphere is described by ASTM E903 [2] while according to ASTM C1549 [3] the measurements can be performed by means of a solar spectrum reflectometer (SSR).

So far testing of new solar reflective building materials was mainly performed in the laboratory by an SSR or a spectrophotometer. On site measurements is performed by pyranometers or albedometers, which present issues caused both by high-pitched roof and by solar and sky requirements. In several places in southern and middle Europe, for instance, conditions of unobstructed sunlight and zenith angle ≤ 45°, mandatory to perform measurements, are not so common: during summer the sky is often hazy and during autumn, winter and spring zenith is lower than 45°. In addition, the requirement of horizontal surface excludes from measurements most of the residential buildings, since traditionally they are made with at least 15° slope. According to Sleiman et al. [4] the solar reflectance of building materials should be tested also after few years of exposure due to the evolution of thermophysical properties. In particular, the decay of solar reflectance after three years of natural exposure or after accelerated aging performed on laboratory should be considered [5]. Reflectometer measurements performed on field, powered by a portable battery, could be a good solution to obtain reliable measurements in every climate, lightning, and slope condition, but the shape of the roof surfaces remains a critical issue since most of the clay tiles are characterized by elaborated morphologies.

In this study two sets of cylindrical adapters for SSR were produced and tested, inspired by adapters designed for portable emissometer, and made via fused deposition modeling. The adapters were coated with different finishing (solar reflective white matt paint and specular reflective coating) and were mounted to the top of the SSR measurements head. Measurements were performed as described by ASTM C1549. The adapters were tested by using a set of 14 ceramic tiles characterized by different values of solar reflectance and the results were compared to those obtained by measurements according to ASTM E903 and to ASTM C1549 without SSR adapters. The data show two clusters, one for adapters coated with aluminum foil and one for adapters coated with white matt paint. However, if we consider each type of data we have a strong linear correlation between the results with and without the adapters for the series coated with aluminum foil while, for samples with white matt paint, the correlation is better fitted by a quadratic function. In conclusion, the designed and prototyped adapters allow, with reliable results, on-field measurements of solar reflectance on roof coated with profiled tiles whereever ASTM E1918 and other standard test methods cannot be used.

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OS2-1 L1A – Tuesday, 12 Sep. 09.50-10.10 – ROOM O2

The Thermal Conductivity of Ionic Liquids: A Key for New Heat Transfer Fluids

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Ionic liquids (ILs) are salts in the liquid state at room temperature with some relevant properties, namely their low vapor pressure over a large temperature range, and higher thermal conductivity than molecular fluids and molten salts are considered sustainable novel solvents in chemical technology, making them possible candidates for heat transfer applications. Among all known ILs, the ones with imidazolium-based cations are the most studied, given their safety, low cost, low viscosity, non-toxicity, and biodegradability. [1,2]. Recent efforts to recommend reference values for the thermophysical properties of an IUPAC standard ionic liquid, $[C_6mim][(CF_3SO_2)_2N]$, showed that there are still problems in experimental data [3]. As so, there is a need to fully understand their properties at a molecular/ionic level in order to understand the thermal conduction in these ILs. This work analyses recent work on the thermal conductivity of pure ionic liquids, reporting new data for $[C_2mim][(N(CN)_2]$ [4], $[C_4mim][(N(CN)_2]$ and $[C_2mim][SCN]$, while analyzing the effect of the cation and anion on the macroscopic thermal conductivity. The thermal conductivity was measured with a transient hot-wire probe, in the temperature range 303.15 K < T < 348.15 K, with an estimated expanded uncertainty Ur (λ) = 0.02. [5]

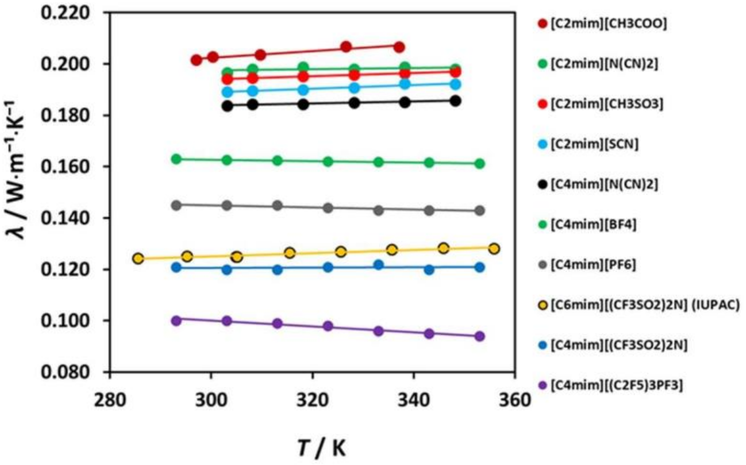

Fig. 1. Thermal conductivity of several ionic liquids as a function of temperature, measured in our group.

Acknowledgment

This study was supported by Fundação para a Ciência e a Tecnologia, IP, through projects UIDB/00100/2020 and UIDP/00100/2020 of Centro de Química Estrutural (CQE). Institute of Molecular Sciences is an Associate Laboratory funded by FCT through project LA/P/0056/2020.

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Thermal and mutual diffusivity of cyclohexane and ethanol mixture by the dynamic light scattering and Raman spectra

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In the process of the heat and evaporation of purification, heat and mass transfer has to be considered. At present, research activities in connection with the modelling of the evaporation of ethanol and cyclohexane are based on theoretical diffusivity data. For their check and validation, there is, however, a lack of reliable experimental data. In this work, a dynamic light scattering apparatus combined Raman spectrum which was especially developed for the simultaneous determination of both thermal and mutual diffusivity for liquid mixtures is presented. The binary mixtures composed of ethanol and cyclohexane were investigated at temperatures 298K, 323K and 348 K. The mole fractions of cyclohexane are 9.1%, 23.1%, 50%, 70%, and 90%, respectively. A total of 30 experimental data points and corresponding Raman spectra were obtained. At the same concentration, the thermal diffusivity decreases with increasing temperature. At the same temperature, there exists a minimum value for the mutual diffusivity as the concentration changes. From the peak position and intensity of the Raman spectrum, it can be seen that the ring vibration at the 802cm^{-1} Raman shift increases with the concentration of cyclohexane. Based on the information of the characteristic peaks, a linear relationship related to concentration can be quantitatively analyzed. The average expanded uncertainty of thermal diffusivity and mutual diffusivity was 5% and 5%, respectively. These findings provide new insights into shed light on industrial purification and separation of volatile liquids with opposite water solubilities, such as cyclohexane and ethanol [1-2].

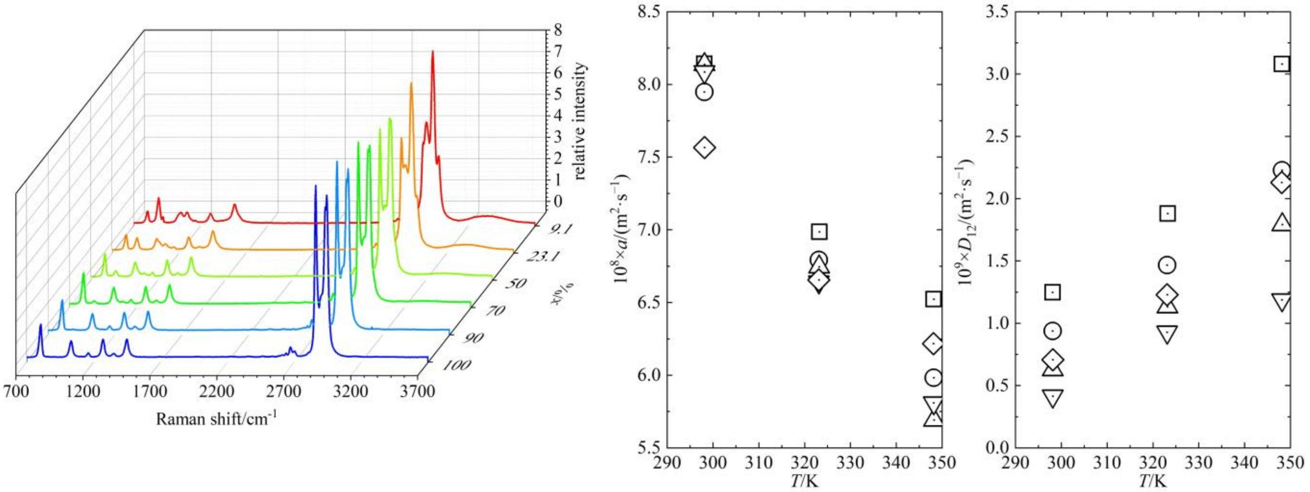

Fig. 1. Left; Raman spectra of binary mixture of cyclohexane and ethanol at 298K; Right; Thermal and mutual diffusivities of binary mixture of cyclohexane and ethanol. (□) x=9.1%; (○) x=23.1%; (△) x=50%; (▽) x=70%; (◇) x=90%.

Acknowledgment

The authors appreciate the financial support from the open funded projects of State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation (No. PLN2021-06) and the Natural Science Basic Research Project of Shaanxi Province (No. 2023-JC-YB-389).

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OS2-1 L1C – Tuesday, 12 Sep. 09.50-10.10 – ROOM N2

Accurate Measurement of Speed of Sound in Fluid for Improving Prediction Precision of PC-SAFT Equation of State

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Speed of sound, as a second-order derivative thermodynamic property, is one of the most demanding challenge for any thermodynamic model performance. The accurate speed of sound is promising to be introduced into the parameter estimation of equation of state (EoS) for improving prediction precision [1]. The light scattering method has unique advantage in speed of sound measurement, however, whose accuracy remains inferior to that of acoustic methods. To improve measurement accuracy, this paper analyzed the uncertainty sources of the speed of sound based on the basic principle of Brillouin scattering method. It was found that the approximation of scattering angle in the measurement formula and the measurement of Brillouin frequency shift are the major error sources, accounting for 8.2% and 90.9% of the total uncertainty severally. Therefore, modifications in both aspects were conducted in our experimental system. Inspired by geometric optics, a symmetric optical path of the scattering angle twice than incident angle ($\Theta_S = 2\Theta_{In}$) was proposed, as shown in Figure 1, eliminating the approximation error. Meanwhile, the doubled Brillouin frequency shift in new optic path and a novel algorithm for Brillouin peak determination allowed for a more accurate Brillouin frequency shift under constant sampling rate. By reassessment, the uncertainty of speed of sound was reduced from 1.05% to 0.23% (cover factor $k = 2$), achieving the same magnitude order as acoustic methods. Then the speeds of sound of n-butanol, a potential biodiesel, were measured at temperatures up to 600 K within 0~8 MPa for supplementing missing data. Compared with existing literature value, the average relative deviation is 0.24% and the maximum deviation is 0.52%, that confirmed the elevated accuracy. Furthermore, besides of saturated vapor pressure and density, the speeds of sound as additional data were put into parameter fitting for the crossover PC-SAFT (Perturbed-Chain Statistical Association Fluid Theory) EoS, which has excellent performance in near critical region [2]. The results show that its prediction precision is significantly improved on the second-order derivative thermodynamic property while keeping acceptable precision for the primary property, with the average absolute relative deviations (AARD) of 1.6% and 10.8% for density and speed of sound respectively.

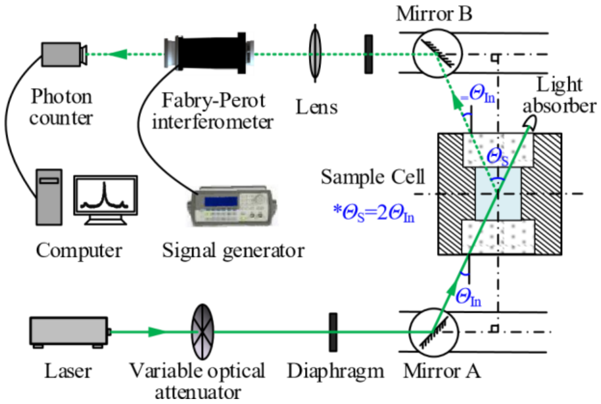

Fig. 1. The Brillouin scattering experimental system with symmetrical optical path for speed of sound measurement.

Acknowledgment

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OS2-1 L1D – Tuesday, 12 Sep. 09.50-10.10 – ROOM N1

Correlations for the density, viscosity and thermal conductivity of liquid titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum, and tungsten

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In a series of recent papers [1-6], new reference correlations were proposed for the density, viscosity and thermal conductivity of liquid aluminium, antimony, bismuth, cadmium, cobalt, copper, gallium, germanium, lead, mercury, indium, iron, lead, nickel, silicon, silver, thallium, tin, and zinc. In this presentation, the work is finalized for the liquid metals of Group IV.

The available experimental data for the density, viscosity and thermal conductivity of liquid titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum, and tungsten have been critically examined with the intention of establishing reference correlations whenever possible for these properties. All experimental data have been categorized into primary and secondary data according to the quality of measurement, the technique employed and the presentation of the data, as specified by a series of criteria. The primary data are employed for the development of the reference correlation, while the secondary data are only used for comparison reasons.

In the case of the density, new reference correlations are proposed for liquid titanium, zirconium, hafnium, vanadium, niobium, tantalum, chromium, molybdenum. However, in the case of the viscosity and thermal conductivity, the lack of data for some of these liquid metals, do not allow the proposition of a reference correlation. In such cases, this lack of experimental data is emphasized.

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OS2-1 L1E – Tuesday, 12 Sep. 09.50-10.10 – ROOM M2

Effect of stretching on the in-plane and out-of-plane thermal conductivities of natural rubber strips- Elastocaloric effect for the generation of solid-state cooling

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The elastocaloric effect in elastomers is mostly due to the crystallization induced within natural rubber during its elongation, typically by a factor of 3 to 6 [1-3]. This may lead to refrigeration without greenhouse gases, and is referred to as "solid-state cooling generation". The devices performances are deeply influenced by the thermal properties of the materials, which are unknown for elastomers under large deformations. This communication aims to measure the thermal conductivity tensor of natural rubber as function as the stretching factor. An original measurement process allowed to measure the 3 main components of the thermal conductivity tensor (transverse k_z, in the direction of stretching k_x and in the in-plane perpendicular direction k_y).

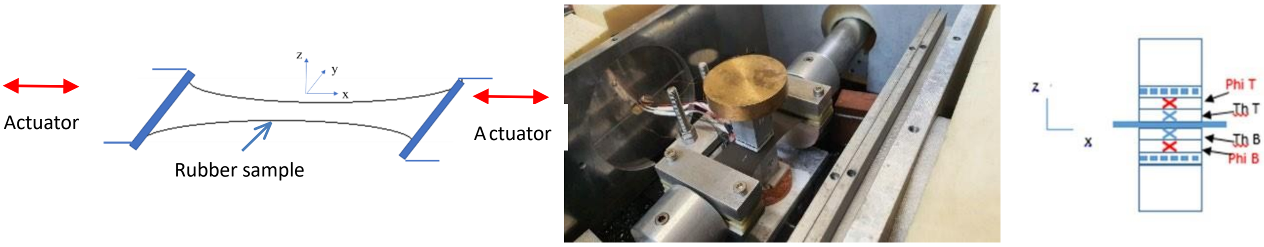

Figure 1. Experimental setup for k_x, k_y and k_z measurement vs. stretching factor

As shown on Figure 1 (right) on both sides of the stretched rubber sample, two sensors were implemented:

- a temperature sensor (80µm diam., type K) inserted in a 1mm thick aluminum plate (18 x 18mm^2) for k_z measurement or a thicker block (6 mm) with a particular geometry to get sensitivity to the k_x or k_y values so to the in plane thermal conductivities,
- a thin (0.5mm) heat flux sensor highly accurate (~3%) from GreenTEG (18 x 18mm^2).

The results are presented on Figure 2 showing decreasing values for k_z and increasing one for k_x and k_y as the elongation is increased.

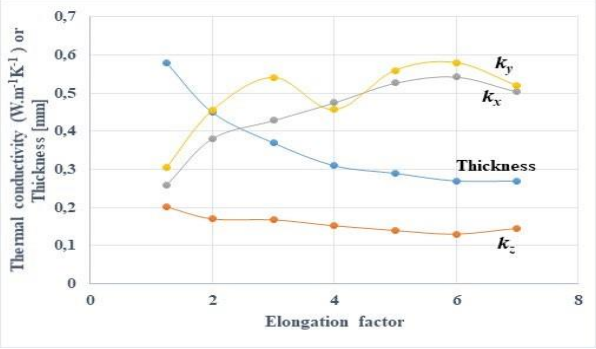

Figure 2. Obtained thermal conductivities for Xinyinte Rubber sample

Acknowledgment

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OS2-1 L2A – Tuesday, 12 Sep. 10.10-10.30 – ROOM O2

The thermodynamic effects of using reactive working fluids in the Stirling cycle

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Due to its high theoretical efficiency and flexibility to exploit green energy sources, the Stirling engine is being investigated for the role it is capable of partaking in the current energy transition. Operating on a closed thermodynamic cycle, the choice of the working fluid is of utter importance when designing the real Stirling engine. To date, helium, hydrogen, and air – all inert gases – are among the most commonly used working fluids in a Stirling cycle. However, to further improve the system's thermodynamic performance, alternate chemically reactive working fluids have been investigated. This was inspired by the observation that the fluids' molecules dissociate during expansion and recombine during compression, positively impacting the expansion and compression work quantities. Thus, reactive fluids accommodate chemical reactions and undergo molecular transformations throughout the cycle in response to changes in the fluid's temperature and pressure, steering the reaction to a new equilibrium and leading to a new molar composition.

Chemically reactive dinitrogen tetroxide (N_2O_4) has been extensively studied in the past as a working fluid for the Stirling engine. This fluid is based on an equilibrated chemical reaction that reversibly shifts in either direction (association at low temperatures or dissociation at high temperatures), satisfying the chemical equilibrium. Furthermore, due to the chemical reactivity of the fluid, an additional degree of freedom is unveiled and discussed pertaining to the engine's operation: the nonadiabaticity of the internal regenerator. Therefore, various discrepancies arise between the conclusions of previous studies on the effect of utilizing this reactive working fluid. This is due to the difference in the assumptions applied to model the Stirling engine regenerator. Therefore, the thermodynamic effects of utilizing chemically reactive fluids in a Stirling cycle are not well understood, and the comparison between inert and reactive working fluids is not well established.

Hence, this work is aimed at understanding the behavior of reversibly chemical reactive fluids, as well as their effect on the system's performance, specifically the thermal efficiency and net specific work output. After investigating the behavior and impact of utilizing reactive working fluids in heat pumps [1] and other power cycles [2], this work aims at assessing a wide range of fictive chemically reactive gases as working fluids in an ideal Stirling engine, by means of the ideal gas mixture thermodynamic model. These fictive gaseous mixtures are identified by the reaction coordinates: standard enthalpy and entropy change of reaction.

Results show a maximum increase of 110% in the net specific work output compared to inert ideal gases. The fluids that yield this increase are characterized by a slightly lower thermal efficiency compared to that of the inert fluids. However, there exists a range of reactive fluids that offer a substantial increase in thermal efficiency, up to 77% compared to 55% for inert fluids. The corresponding cycles are characterized by a nearly adiabatic internal regenerator, similar to that of comparable inert cycles.

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OS2-1 L2B – Tuesday, 12 Sep. 10.10-10.30 – ROOM O1

Prediction of partition coefficients of formic acid in water-soybean oil system

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The knowledge of thermodynamic properties is essential for the development of chemical processes. Depending on the complexity of the system, the prediction of these properties becomes interesting. There is a considerable number of thermodynamic models based on group contribution, i.e., works with a sum of contributions of parts of the molecule. Examples are the Universal Quasichemical Functional-group Activity Coefficients (UNIFAC) and the Conductor-like Screening Model for Segment Activity Coefficient (COSMO-SAC). The specified models were applied to estimate partition coefficients associated with the epoxidation of soybean oil, a reaction to produce green components of plasticizers. The reaction system is typically biphasic, with a liquid aqueous phase and a liquid organic phase, with formic acid participating in reactions in both phases [1-3]. Therefore, in order to detail a model that represents this reaction system, the knowledge of phase equilibrium is essential, since it affects mass transfer effects. In the present work, the models UNIFAC and COSMO-SAC were applied to predict the partition coefficients of formic acid in a system containing soybean oil and water, considering the effects of temperature, soybean oil:water ratio, and formic acid:(soybean oil + water) ratio. The predicted results indicate the tendency of formic acid to solve preferably in the aqueous phase. The results suggest that temperature is the major effect on the partition coefficients for this system, although this effect was more accentuated in the UNIFAC prediction. Figure 1 illustrates a comparison between the two models, considering a soybean oil:water ratio of 1 (in weight) and a formic acid:(soybean oil + water) ratio of 0.03 (in weight).

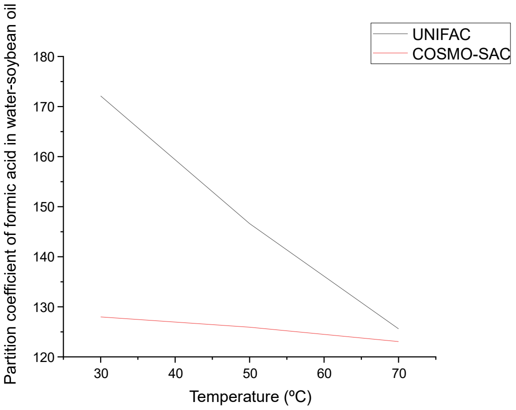

Fig. 1. Effect of the temperature on the partition coefficients of formic acid in water-soybean oil system.

Acknowledgment

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OS2-1 L2C – Tuesday, 12 Sep. 10.10-10.30 – ROOM N2

Development of an Instrument to Measure the Sound Speed of Molten Metals at Extreme Conditions

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Thermodynamic property measurements of liquid-phase metallic elements can offer fundamental insight into material behavior and guide the successful execution of industrial designs. Reference quality measurements are especially important given the growing number of next-generation energy, defense, and manufacturing systems operating in extreme environments. Specific applications for materials in extreme environments include fusion energy, armor, metal additive manufacturing, and semiconductor manufacturing. Nevertheless, a gap in the literature exists for reference quality data of molten metals above atmospheric pressure to 100 MPa. To fill this gap, an instrument derived from the pulse-echo technique is being developed and tested to measure the speed of sound of molten metals at conditions of up to 750 K and 50 MPa. [1] The thermodynamic property speed of sound is of particular value as it offers the prospect of high accuracy over wide temperature and pressure ranges where other direct measurements, such as density, have limited accuracy. Additionally, speed of sound measurements can be used to obtain three additional thermodynamic properties of density, isobaric heat capacity, and isobaric expansivity. We describe the setup, processing, measurement methods, and measurement results to date.

Acknowledgment

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OS2-1 L2D – Tuesday, 12 Sep. 10.10-10.30 – ROOM N1

Thermophysical properties of Al-Ti binary liquid alloys

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Binary and ternary Al-Ti alloys play important roles in medical and aeronautical applications. They are used under harsh conditions as turbine blades. Yet, systematic thermophysical property data of the Al-Ti binary liquid alloy system does not exists. This is partly due to the high chemical reactivity of Ti- alloys. Containerless investigations using levitation techniques are therefore mandatory.

In the present work, liquid binary Al-Ti alloys, covering the entire compositional range, including the pure elements, are processed in electromagnetic levitation (EML). Density, surface tension, molar specific heat (c_p), and normal spectral emissivity ε are studied as functions of temperature and composition.

In EML, forces generated by an inhomogeneous magnetic AC - field stably position the specimen against gravity. Melting is achieved by inductive heating. Denisty (volume) is determined from the droplet's edge curve in the shadow graph profile. Surface tension is determined from the frequency spectrum of the time dependent radius. In order to determine c_p and ε, a magnetic DC field is used in addition to suppress the fluid flow. c_p is then determined by means of laser modulation calorimetry and ε is measured from the normal spectral radiance measured by a multichannel spectrometer calibrated by quasi-black body radiation.

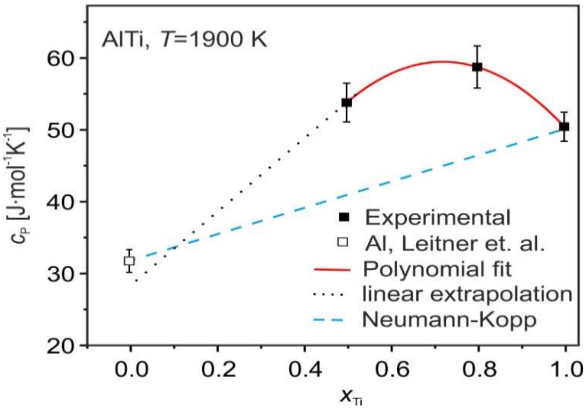

Fig. 1. Molar specific heat of Al-Ti binary alloys at 1900 K versus Ti-mole fraction.

The results are compared to analogous results for similar systems (Al-V, Al-Ni, …). They are analyzed in terms of solution models. It is found that, with respect to all investigated properties, Al-Ti forms a highly non-ideal solution. Figure 1 shows for example the molar specific heat of Al-Ti at 1900 K.

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OS2-1 L2E – Tuesday, 12 Sep. 10.10-10.30 – ROOM M2

Thermophysical properties of wooden material exposed to high temperatures

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Timber is the only structural material of biological origin and is attracting growing attention in the construction industry within the last decades. It makes a significant contribution in terms of improving the overall energy efficiency of buildings and is nowadays not only used in traditional areas such as detached houses, but also in high-rise buildings in urban regions regarding a sustainable densification of urban areas. With the growing use of wooden material in high-rise buildings also fire protection requirements are increasing; the fire resistance of timber must be known and verified by cost and time consuming fire tests. The development of numerical models to predict the fire resistance of timber structures is a promising approach in terms of shortening the development cycles and a reduction of development costs. However, the development of numerical models requires an extensive knowledge of the temperature dependent thermophysical properties of wooden materials in different states (moist, dry, charred). Nevertheless, timber is the only structural material for which data of thermophysical properties at elevated temperatures are sparse or only treated in a limited manner, e.g., not considering the moisture content or the anisotropic behavior.

In this work thermophysical properties of wooden material in a wide temperature range are presented. The material was investigated in different states: moist, dry and charred, as well as during pyrolysis. Thermogravimetric measurements were performed to investigate the mass loss of wooden material at different heating rates up to 900°C. The thermal expansion was measured using a pushrod dilatometer. From thermal expansion and density at room temperature the temperature dependent density of the material was calculated over a wide temperature range. The (apparent) specific heat capacity of the wooden material was measured using differential scanning calorimetry and simultaneous thermal analysis; the material was investigated in the moist, dry and charred state, as well as during pyrolysis. The laser flash method was used to measure thermal diffusivity of the material. As wooden material shows an anisotropic behavior, thermal diffusivity, thermal expansion and thermal conductivity depend on the fiber direction. To consider the anisotropic behavior of wooden material, the corresponding measurements were performed on specimens cut along the fiber (longitudinal direction) and across the fiber (transversal direction).

Thermal conductivity was calculated from thermal diffusivity, specific heat capacity and density. These results are reported at room temperature of the moist material; from room temperature to the determined stability limit of wooden material at approx. 200°C of the dry material. Results of wooden material in the charred state are reported up to 900°C. From the measurements derived it can be concluded that the laser flash method is an efficient method to determine thermal diffusivity of wood and charred material in a wide temperature range and in selected orientations (in longitudinal and transversal direction). The thermal expansion of wooden material is significantly higher in transversal direction than in longitudinal direction, whereas thermal diffusivity and thermal conductivity are higher in fiber direction than across the fiber.

Acknowledgment

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OS2-1 L3A – Tuesday, 12 Sep. 10.30-10.50 – ROOM O2

Experimental thermophysical and rheological profile of cellulose nanocrystal nanofluids based on propylene glycol: water

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Global primary energy demand increased by 5.8% in 2021, exceeding 2019 levels by 1.3%. Moreover, the use of fossil fuels in the last 5 years accounted for 85% of primary energy [1]. The sustainability of the energy system is closely related to the performance of the heat transfer processes. Propylene glycol and its aqueous mixtures are common working fluids in these processes. In this work, nanofluids were designed by dispersing cellulose nanocrystals (CNC) of 10 nm average width at concentrations from 0.15 to 0.30 wt% in a binary mixture of propylene glycol:water 20:80 wt% (PG:W). With the aim to observe the temporal stability, a Zetasizer Nano ZS (Malvern Instruments, UK) was used. The studied nanodispersions showed a constant size throughout a period of at least two weeks. Isobaric heat capacities were determined by a Q2000 calorimeter (TA Instruments, USA) under the quasi-isothermal temperature-modulated differential scanning calorimetry method from 283 to 323 K. Decreases around 3% in this property with nanoparticle loading were obtained by comparing the nanofluids and PG:W. Experimental thermal conductivity measurements were carried out by a THW-L2 device (Thermtest, Canada). Figure 1 shows the temperature dependence of this property within the 383 to 313 K range and as it can be observed, increases with respect to PG:W for all designed samples are found.

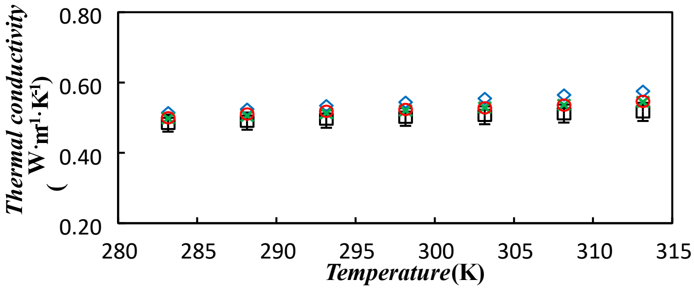

Fig. 1. Temperature dependence of thermal conductivity for base fluid (□), 0.15 wt% CNC/PG:W (◇), 0.20 wt% CNC/PG:W (○) and 0.30 wt% CNC/PG:W (□).

Density and rheological tests were also performed by a DMA501 densimeter (Anton Paar, Austria) and a Physica MCR 101 rheometer (Anton Paar, Austria), respectively, from 283.15 K to 313.15 K. The density of nanofluids increases up to 0.12% respect to PG:W. Newtonian behavior was observed for the 0.15 wt% and 0.20 wt% nanofluids (viscosity increases up to 34% respect to PG:W), while shear thinning behaviour was detected for the 0.30 wt% nanofluid. In conclusion, CNC/PG:W nanofluids show good stability and decreases in isobaric heat capacity, improvements in thermal conductivity and slight increases in density with respect to the base fluid were examined. Furthermore, the increasing amount of CNC nanoadditive leads to shear thinning behaviour.

Acknowledgments

Grant PID2020-112846RB-C21 funded by MCIN/AEI/10.13039/501100011033, Grant PDC2021-121225-C21 funded by MCIN/AEI/10.13039/501100011033 and by "European Union NextGenerationEU/PRTR". Authors also acknowledge the financial support by Xunta de Galicia, GRC ED431C 2016-034. M.A.M. acknowledges the foundership by the European Union-NextGenerationEU, the Spanish Ministry of Universities and the Recovery, Transformation and Resilience Plan, through a call from the University of Vigo (Spain). J.P.V. thanks the Defense University Center at the Spanish Naval Academy (CUD-ENM) for all the support provided for this research.

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OS2-1 L3B – Tuesday, 12 Sep. 10.30-10.50 – ROOM O1

Determination of Diffusion Coefficients from Constant Volume Diffusion Tests through Numerical Simulation

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Molecular diffusion is a fundamental mass transport phenomenon crucial to many scientific and industrial fields. Its accurate description relies on diffusion coefficients that can be either experimentally measured or theoretically estimated. The constant volume diffusion (CVD) method [1] is widely used to measure liquid-phase diffusion coefficients at high pressures. However, it requires an analytical or numerical solution for data interpretation, with the latter generally involving fewer assumptions. In this study, we described an algorithm that can determine constant or composition-dependent diffusion coefficients from the CVD data using the orthogonal collocation method. We further coupled the simulation code using this algorithm with five diffusion coefficient correlations, including the Wilke-Chang (WC) [2], Hayduk-Minhas (HM) [3], extended Sigmund (ES) [4], Riazi-Whitson (RW) [5], and Leahy-DiOSFiroozabadi (LDF) [6], to investigate their performance in predicting or regressing the CVD data for methane-n-alkane and nitrogen-n-alkane systems at elevated pressures. We found that the CVD results are insensitive to the gas-phase diffusion coefficients. The simple WC and HM give the closest predictions despite their empirical nature. In contrast, the predictions by LDF under the rigorous Maxwell-Stefan framework are not satisfactory, which may be attributed to an inherent problem of LDF in modeling gas-liquid diffusion. Regression results using different correlations and a constant liquid-phase diffusion coefficient are almost the same. Different correlations result in different ranges of the regressed coefficients, and those determined using the assumption of constant diffusion coefficients are always within these ranges. We also compared and illustrated the differences in the profiles of diffusion coefficients between these correlations. In general, ES predicts a decreasing diffusion coefficient towards the interface whereas WC predicts an increasing trend. RW and LDF show an increasing trend in most cases. The study demonstrates that the algorithm provides a reliable and effective approach for processing CVD data. It also highlights how the data interpretation depends on the assumed composition-dependence.

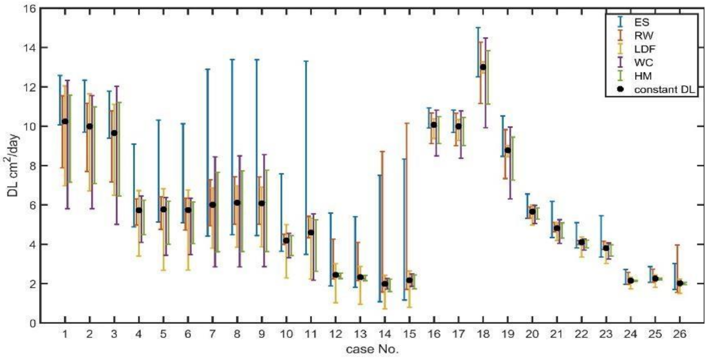

Fig. 1. Regressed liquid-phase diffusion coefficients D_L using different correlations for methane-n-alkanes and nitrogenn-alkanes at elevated pressures.

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OS2-1 L3C – Tuesday, 12 Sep. 10.30-10.50 – ROOM N2

Liquid-Phase Speed of Sound measurements of R-1130(E) at Temperatures Ranging from 230 K to 420 K and Pressures up to 25 MPa

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Liquid-phase speed of sound data are reported for the low global warming potential refrigerant *trans*-1,2Dichloroethylene (R-1130(E)). The data were measured using a dual-path pulse-echo instrument at temperatures ranging from 230 K to 420 K and pressures up to 25 MPa. The uncertainties in the speed of sound measurements were estimated to be lower than 0.04% over the entire range of state points investigated. Presently, no other speed of sound data for the R-1130(E) refrigerant are available for comparison to the data reported in this study. Therefore, the data were compared to the extended corresponding states model of Tanaka et al.[1] which was previously compared to their own density, vapor pressure, and surface tension measurements of R-1130(E). The R-1130(E) data reported in this study compared to the ECS model of Tanaka et al. exhibited consistent deviations from -4.1% to -3.8% over the range of conditions investigated.

Acknowledgment

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OS2-1 L3D – Tuesday, 12 Sep. 10.30-10.50 – ROOM N1

A new approach for estimating the thermal diffusivity of molten metals at very high temperature

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The development of numerical simulations of industrial processes such as welding and additive manufacturing requires the knowledge of physical properties of metals from solid state to vaporization. In the case of thermal diffusivity, the higher the temperature, the more difficult it is to estimate experimentally the value of this property. Indeed, multiple issues are encountered such as radiative heat loss, pollution of the sample or motion inside the molten sample, which makes the estimation of this property a challenging task.

In this communication, we develop a methodology to estimate the thermal diffusivity of liquid metals with a fusion point above 1500 °C. The set-up is based on the traditional front face laser flash method [1]. To avoid undesirable reaction of the molten metal with oxygen, the set-up is placed inside an inert chamber filled with protective argon gas, so the oxygen content in the surrounding atmosphere is less than 5 ppm. The sample, in form of a thin metallic sheet (220 µm) is heated by a 1070 nm fiber laser, and a system of reflective mirrors allows a synchronized visualization of both the sample faces. Given the thin thickness of the sample, the resulting heat diffusion time is very short, so a high speed camera (10 000 FPS) is used to capture the emitted radiation during the experiment and the temperature field is then estimated by applying a gray-scale/temperature conversion [2]. Moreover, when the temperature exceeds the fusion point, the resulting melt pool remains attached to the solid surroundings due to the surface tension, but random motions are observed inside the melt pool, and they should be integrated in the estimation process. To address this issue, an image processing technique based on a non-rigid registration [3] is implemented. The local transformations during the experiment are thus estimated allowing the characterization of the velocity field of the melt pool. Different strategies to validate the obtained data are presented. The extracted temperature and velocity fields are used as input data to estimate the thermal diffusivity by inverse techniques. Experimental and numerical challenges are also addressed. The general framework of the proposed approach is illustrated in figure 1. The methodology is applied on pure iron samples and first outcomes in both solid and liquid states are presented.

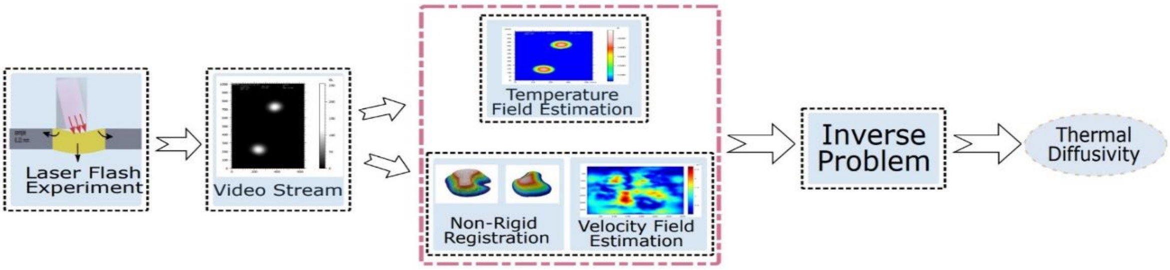

Fig. 1.A graphical representation of the proposed methodology

Acknowledgment

This work is funded by Université Bretagne Sud and Région Bretagne.

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OS2-1 L3E – Tuesday, 12 Sep. 10.30-10.50 – ROOM M2

Effect of the delay time correction of laser pulse in thermal diffusivity measurement in a laser flash method

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Laser flash method is the most widely used for evaluating the thermal diffusivity of solid materials such as metals, alloys, and ceramic materials. As semiconductor integration increases and substrates become thinner, measuring high thermal conductivity is more important. In this study, it was found that delay time varied with the power of the single laser pulse incident on the specimen. When the input laser voltage changes from 1538 Volt to 2306 Volt, the delay time varies from 0.186 to 0.120 milliseconds due to the change of the single pulse width. The delay time directly affects the half-time of the laser flash measurement, and it can cause to distort the results of thermal diffusivity. We describe a procedure for correcting the thermal diffusivity by reflecting the delay time to fit the half-time at each voltage condition. All measurement values of the thermal diffusivity were arranged with the intrinsic method that were extrapolated to the zero-input power of the incident laser, and Cape and Lehman's curved fit was used to calculate thermal diffusivity[1-3]. In order to validate this procedure, the experimental works were carried out with five graphite samples with a thickness of (1~4) mm. At a thickness of 1 mm, the effect of delay time was more evident due to fast thermal diffusion with the short half-time. In addition, the validity of thermal diffusivity correction was once again verified with Al6061 samples with a different thickness between 1 mm and 4 mm.

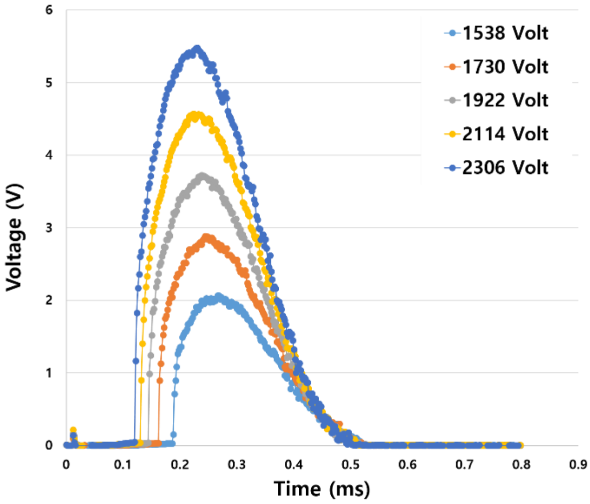

Fig. 1. Delay time of single pulse changed laser power voltage

Acknowledgment

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OS2-1 L4A – Tuesday, 12 Sep. 10.50-11.10 – ROOM O2

Parameter Estimation of Iron Oxide Nanofluids by Montercarlo Markov Chains in the Hyperthermia Treatment by Laser Induction

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The objective of this work was to estimate the parameters of iron oxide nanofluids of a model for laser heating for cancer treatment by hyperthermia. A linear regression model was proposed as a solution to the forward problem trained with a database obtained from multiple simulations of the complete model. A Bayesian approximation was performed in the estimation of the parameters with the Markov Chain Monte Carlo (MCMC) method, which was implemented with the Metropolis-Hastings algorithm. Experimental temperature measurements were obtained through an infrared camera from the fluid surface [3]. Iron oxide nanoparticles dissolved in distilled water in two proportions (0.025 and 0.050 wt) were considered and heated by a laser in the near-infrared range. The results indicate that it is possible to obtain the estimation of the parameters of different nanofluids in a short period. Fig. 1 shows the histogram for absorption coefficient of the iron oxide nanofluids of 0.050 wt.

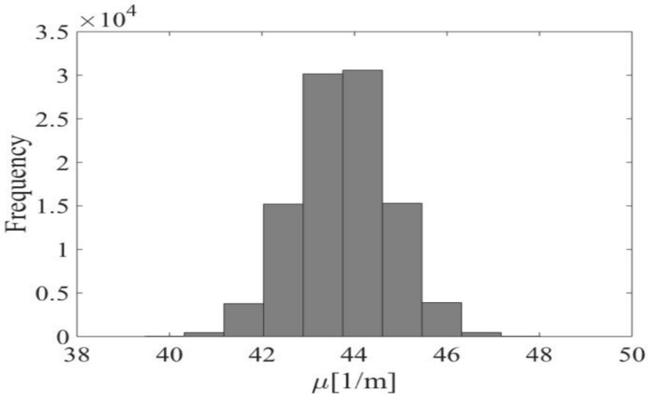

Fig. 1. Histogram for absorption coefficient of de nanofluids of 0.050 wt.

Acknowledgment

The authors are thankful for the support provided by Conselho Nacional de Desenvolvimento Científico e Tecnológico; Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES); Fundação Carlos Chagas Filho de Amparo à Pesquisa do Estado do Rio de Janeiro, (FAPERJ) Brasil; Fundação de Amparo à Pesquisa do Estado do Amazonas – FAPEAM and Dirección General de Investigaciones (DGI) of Universidad Santiago de Cali, Colombia.

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OS2-1 L4B – Tuesday, 12 Sep. 10.50-11.10 – ROOM O1

Measurement and Evaluation of Diffusion coefficients of water and propylene glycol in CO_2 under different pressure using the DPDVA method

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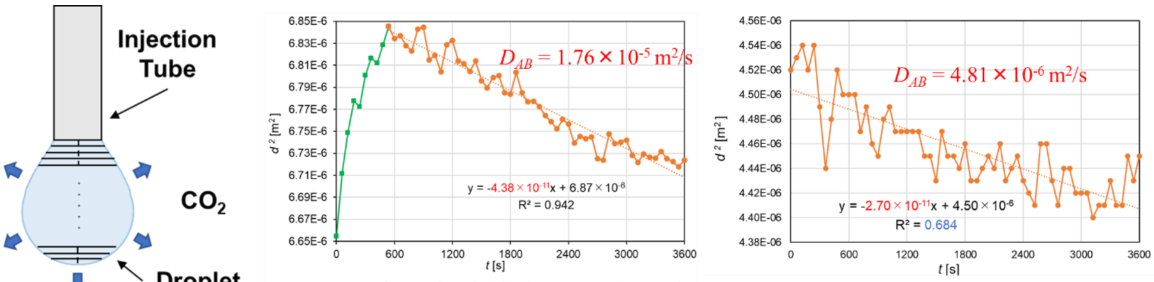
Soil pollution by heavy metals and volatile organic compounds has become a significant problem all over the world. From the viewpoint of conservation of nature and our healthy life, technologies to reuse soil are important and remediation methods using supercritical CO_2 (sCO_2) are attracting solution. Supercritical fluids have gas-like diffusivity and liquid-like solubility, and are expected to be a highly efficient and safe method for soil remediation. It is important to understand and measure the mass diffusion of pollutant under supercritical conditions. In this study, in order to evaluate the phenomenon at supercritical conditions, the diffusion coefficients of water and propylene glycol to CO_2 were measured by the Dynamic Pendant Drop Volume Analysis (DPDVA) method under ambient and high- pressure conditions[1].

A schematic of the DPDVA method and the time variation of propylene glycol’s d^2 (d : Droplet diameter) are shown in Fig. 1. A droplet is kept at the tip of the injection tube and the diffusion coefficient is derived from the time variation of d^2.



Propylene glycol-Air (298 K, 0.10 MPa) Propylene glycol-CO_2 (298 K, 5.05 MPa)

Fig.1 Schematic of DPDVA method and an example of time variation of d^2

At normal pressure, d^2 was well approximated linearly against time. However, at high pressure, 5.05 MPa, the droplet changed to a vertically stretched shape and measurement data had disturbances. It can be considered that the increase in CO_2 absorption due to the increased pressure may have caused the viscosity drop of droplet and enhanced the influence of local convection. The value of the diffusion coefficient obtained by the experiment were 1.15×10^{-5} m^2/s (Water-Air, 298 K, 0.10 MPa), 1.76×10^{-5} m^2/s (Propylene glycol-Air, 298 K, 0.10 MPa), 4.81×10^{-6} m^2/s (Propylene glycol-CO_2, 298 K, 5.05 MPa). The diffusion coefficient at ambient temperature and pressure was compared with the experimental data using the Fuller’s equation. The value were 1.15×10^{-5} m^2/s (Water-Air), 0.97×10^{-5} m^2/s (Propylene glycol -Air), which is in relatively good agreement with the experimental data[2].

Acknowledgment

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OS2-1 L4C – Tuesday, 12 Sep. 10.50-11.10 – ROOM N2

Self-diffusion coefficients of pure fluid using residual entropy scaling with PC-SAFT and group contribution method

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The residual entropy scaling can simplify the complex variation of transport properties with temperature and pressure into a single valued function of residual entropy [1-2]. The self-diffusion coefficient can characterize the mass transfer behavior driven by molecular microscopic motion. The entropy scaling of self-diffusion coefficients for real fluids has been built [3-4], however, previous work required separate fitting parameters for each substance. In this work, a residual entropy scaling model of self-diffusion coefficient is developed for pure fluids based on group contribution method. The relation between reduced diffusion coefficient D^* and reduced residual entropy s^* can be represent by:

$$\ln\left(D^*\right)=\ln\left(\frac{D}{D^{\mathrm{ref}}}\right)=\sum_k A_k s^{*k}+B\left(1-\exp(s^*)\right) \tag{1}$$

where D_{ref} is calculated by Chapman-Enskog equation, $s^* = s_{\mathrm{res}}/mR$ is obtained by PC-SAFT EoS, A_k and B are parameters determining by group contribution approach. According to Eq. (1), some functional group parameters are fitting by self-diffusion coefficients data of about 40 pure substances, including *n*-alkanes, branched alkanes, alcohols, ethers, esters and ketones, as shown in Table 1. Figure 1 shows the calculation results for residual entropy scaling of self-diffusion coefficients in *n*-alkanes based on group contribution method, with a relative deviation of 6.73%.

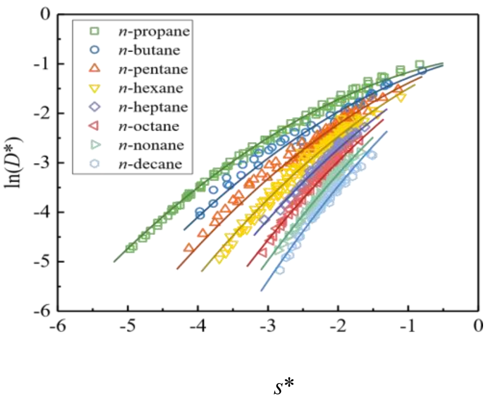

Fig. 1. Residual entropy scaling of self-diffusion coefficients in *n*-alkanes.

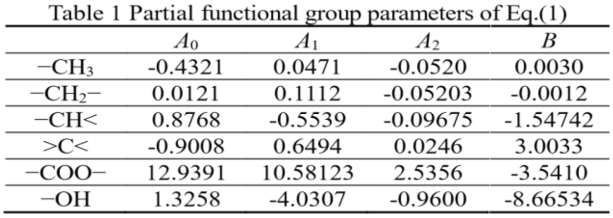

Table 1 Partial functional group parameters of Eq.(1)

	A_0	A_1	A_2	B
−CH$_3$	-0.4321	0.0471	-0.0520	0.0030
−CH$_2$−	0.0121	0.1112	-0.05203	-0.0012
−CH<	0.8768	-0.5539	-0.09675	-1.54742
>C<	-0.9008	0.6494	0.0246	3.0033
−COO−	12.9391	10.58123	2.5356	-3.5410
−OH	1.3258	-4.0307	-0.9600	-8.66534

Acknowledgment

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OS2-1 L4D – Tuesday, 12 Sep. 10.50-11.10 – ROOM N1

Thermophysical property measurements of vanadium melts and discussion on thermal diffusivity of transition metals based on Mott's theory

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Over the past 80 years, the correlation between the density of states (DOS) and physical properties such as electrical resistivity [1], Seebeck coefficient [2], thermal conductivity (κ) [3], and thermal diffusivity (α) have been investigated. In 1936, Mott [4, 5] reported that the electrical resistivity of the transition metals is attributed to the scattering process of the transition of electrons in the *s*-state to the unoccupied *d*-state at Fermi level. It is called "Mott's *s*-*d* scattering model".

For vanadium melts, Zinovyev et al. [6] measured α using plane-temperature wave method, and Pottlacher et al. [7] evaluated α via the Wiedemann-Franz law. Thermal diffusivity reported by Zinovyev is 18% higher than the value reported by Pottlacher. Thus, α of pure vanadium melts has yet to be established.

Our research group developed electromagnetic levitation (EML) technique with a static magnetic field for the accurate measurement of thermophysical properties such as density (ρ), normal spectral emissivity, heat capacity (C_P), and κ, in a liquid state. The static magnetic field can suppress the convection in the levitation melts and translation motion, and surface oscillation of the levitation melts [8]. In our previous study, the relation between α and reciprocal DOS for six transition metals in a liquid state has been investigated, in consequence, a good linear correlation between α and reciprocal DOS at Fermi level was demonstrated. This linear correlation is supported by Mott's *s*-*d* scattering model.

In this study, ρ, C_P, and κ of vanadium melts were measured to obtain α using EML with a static magnetic field, and the correlation of the α and reciprocal DOS at Fermi level of vanadium melts was discussed based on Mott's *s*-*d* scattering model.

Acknowledgment

This study was supported by Japan Society for the Promotion of Science (JSPS) KAKENHI Grant Number 20K22464 and 21K14447.

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OS2-1 L4E – Tuesday, 12 Sep. 10.50-11.10 – ROOM M2

Measuring the in-plane thermal diffusivity by photothermal spatially random pattern

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A laser pulse, with duration 2 ms, and characterized by a random spatial distribution in a square area of 1 by 1 cm, is projected on the surface of a specimen. An IR camera collects a sequence of images of the specimen's surface temperature. Those images are quite sharp immediately after the pulse, resembling the light pattern randomly distributed that was projected (see Fig. 1). As the time goes by, the pattern decreases in intensity and blurs as well, due to the diffusion of heat. The components of the spatial Fourier Transform of the temperature field, collected by the IR camera, are analysed in time. Their amplitudes are decreasing with time according to an exponential decreasing function. The time constants depend on the spatial frequencies and on the thermal diffusivity of the specimen as well. Fitting the data allows finally to estimate the time constant and therefore, once the spatial frequency is known, the thermal diffusivity. Other spatial distributions, like the point, the line, and the periodic grid, were proposed in the past [1], while the random spatial distribution was considered but with a different data analysis [2]. In this study, because of the randomness of the pattern, several spatial frequencies are stimulated and can be analysed. That make the estimation of thermal diffusivity more robust. Eight random patterns have been prepared by gold spattering on glass substrate, in the facilities of Tokyo Tech Laboratory. Preliminary results are given in Tab.1, for a specimen of clay brick.

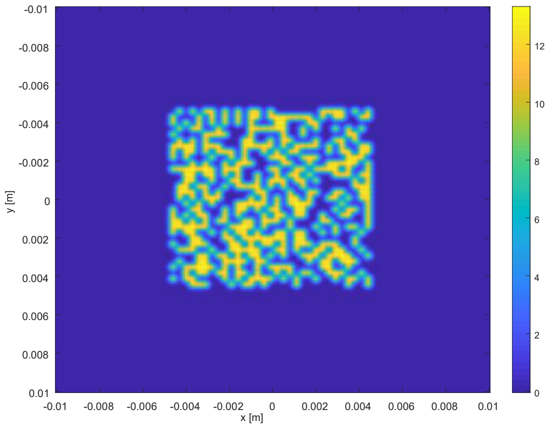

Fig. 1. Surface temperature immediately after the laser pulse with random spatial distribution.

Table 1. Thermal diffusivity results obtained by using 8 different random patterns projected on a specimen of clay brick.

	Pattern #1	Pattern #2	Pattern #3	Pattern #4	Pattern #5	Pattern #6	Pattern #7	Pattern #8
Thermal Diffusivity [m^2s^{-1}]	6.19E-07	6.20E-07	6.35E-07	6.27E-07	6.28E-07	6.57E-07	6.23E-07	6.27E-07
Standard deviation [m^2s^{-1}]	4.89E-09	1.06E-08	2.30E-09	7.29E-09	4.30E-09	5.73E-09	5.35E-09	1.76E-08

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OS2-2 L1A – Tuesday, 12 Sep. 11.40-12.00 – ROOM O2

A thermodynamic modeling of CO_2 absorption with amino acid ionic liquids

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Amino acid ionic liquids (AAILs) are new solvents synthesized from amino acids and their derivatives, which are attractive as promising candidates for CO_2 absorption due to high solubility and selectivity of CO_2 and low solvent loss [1-2]. An impediment to developing IL-based CO_2 capture technology is the lack of thermodynamic models that can accurately describe the CO_2-IL absorption process to guide the selection of solvents. In this contribution, cubic plus association equation of state (CPA EoS) with three cross-association schemes ($N_{1:2}$, $N_{1:1}$ and $N_{2:1}$) proposed based on reaction mechanisms was used for modeling CO_2 chemisorption in AAILs with various amino species and numbers as listed in Table 1. The results show that $N_{1:2}$ scheme is applicable to [Glu], [Tyr] and [Gln]-IL, $N_{1:1}$ scheme is applicable to [Pro], [Gly], [Ala], [Val], [His] and [Arg]-IL, and $N_{2:1}$ scheme is applicable to [Lys] and [Arg]-IL. The average absolute relative deviation of CO_2 solubility in AAILs in the range of temperature (283-318 K) and pressure (10-400 kPa) is 4.6%, as shown in Fig. 1. The prediction capability of model was verified by the good quantitative agreement with solubility data in literature. The developed model was then used to estimate the CO_2 absorption enthalpy, solvent cyclic capacity and regeneration energy to evaluate the CO_2 capture performance of selected AAILs. The highest cyclic capacity and greater energy saving were obtained for [Cho][Gly]. These results indicate that this model provides a feasible approach to preliminary screen suitable IL solvents and design CO_2 capture process.

Table 1. Amino acid ionic liquids studied in this work and number of NH_2/NH groups.

AAIL	Number of amino groups	AAIL	Number of amino groups	AAIL	Number of amino groups
[Cho][Pro] [VBIm][Pro]	NH(1)	[BMIm][Val] [Cho][Gly] [Cho][Ala] [Cho][Glu] [Cho][Tyr]	$NH_2(1)$	[VBIm][His] [Cho][His]	$NH_2(1)$ & NH(1)
[VBIm][Gly] [VBIm][Val] [BMIm][Ala]	$NH_2(1)$			[VBIm][Lys] [Cho][Gln]	$NH_2(2)$
				[VBIm][Arg] [Cho][Arg]	$NH_2(2)$ & NH(2)

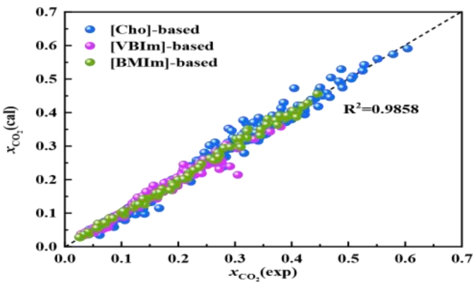

Fig. 1. Experimental CO_2 solubility (x_{co_2}(exp)) in AAILs versus calculated CO_2 solubility (x_{co_2}(cal)) by CPA EoS.

Acknowledgment

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OS2-2 L1C – Tuesday, 12 Sep. 11.40-12.00 – ROOM N2

On the hunt for sustainable refrigerants fulfilling technical, environmental, safety, and economic requirements by using robust models

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Given the F-gases emission prospects in the near future, Kigali's amendment has committed to restrict the manufacture and further supply of high global warming potential (GWP) 3rd generation refrigerants in nowadays refrigeration and air conditioning applications (RAC). Particularly, up to one-fifth of today's RAC systems that operate with hydrofluorocarbons (HFCs) will have to be completely replaced in the years to come in harmony with eco-friendly European policies [1,2]. To tackle this issue, the polar soft-SAFT molecular-based equation of state (EoS) has been used in a predictive manner to screen and design alternative low-GWP fluorinated-based refrigerants valid as drop-in replacements for the most commonly used HFC refrigerants in RAC today, R134a (GWP=1300) and R410A (1924). For this purpose, a robust and accurate thermodynamic model has been built to reproduce refrigerants' properties, including vapor-liquid equilibria, derivative properties and binary systems at a coarse-grain level [3]. The modeling framework is applied in a systematic manner for assessing compliance of drop-in replacements towards ensuring retrofitting scenarios based on a multi-criteria assessment (4E analysis) on energy, exergy, environmental, and economic variables, in addition to a thorough examination of safety and technical features. In addition, Artificial Neural Network (ANN) models [4] are also implemented, in combination with the SAFT models, to predict thermophysical properties of designed blends for which experimental data is non-available, exploring the novelty of artificial intelligence on the hunting for low-GWP refrigerants without the need of further experiments. This holistic approach is seen to be valid at a broad spectrum of working conditions and system configurations, emphasizing the applicability, transferability and accuracy of the accomplished methodology. Additional environmental and cost rate evaluation (geographically dependent based on electricity cost) is provided to quantify the impact associated with their use and emissions, allowing to identify suitable replacements from a techno-environmental-economic point of view. The compatibility KPIs are also reproduced for R513A (608), R450A (682), and R32 (675), the best-known domestic and commercial drop-ins for R134a and R410A, fulfilling an in-depth comparison among mid- and low-GWP cooling agents. Finally, a sensitivity analysis is explored based on legislative agendas that can be realistically implemented in upcoming years to boost its economic viability and effectively promote a transition to their manufacturing and deployment. Overall, this work proves the value of using an accurate molecular-based EoS to generate the required data for the design of low-GWP refrigerants with enhanced safety and technical features.

Acknowledgment

This research is supported by the Spanish Ministry of Science and Innovation MCIN/AEI/10.13039/501100011033, under R + D + I projects STOP-F-Gas, (PID2019-108014RB-C21) and NEW-F-TECH (TED2021-130959B-I00), and by Khalifa University of Science and Technology (project RC22019-007). Carlos G. Albà acknowledges a FI-SDUR fellowship granted by the Catalan Government.

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OS2-2 L1D – Tuesday, 12 Sep. 11.40-12.00 – ROOM N1

Melting of oxide film containing Si formed on iron plate utilizing the heat of oxidation reaction

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In the hot rolling process of steelmaking, an oxide film (oxide scale) is formed on the steel plate due to hightemperature oxidation. Particularly in the case of Si-containing steels, a Si-rich layer (consisting of Fe_2SiO_4 and FeO, etc.) is formed between the steel and the FeO scale. The Si-rich layer is difficult to remove from the steel by descaling, and rolling with scale remaining on the surface would result in surface defects. According to thermodynamic data, Fe_2SiO_4 and FeO formation reactions (oxidation reactions) are exothermic [1]. Thus, the oxidation reaction could melt the oxide scale even though the temperature of the steel plate is lower than the melting point of the oxide scale. From the above, to examine the effectiveness of utilizing the oxidation reaction of iron against the melting of the Si-rich layer, this study aimed to investigate the effect of SiO_2 addition on the oxidation of iron at high temperatures. The sample were iron plates (99.99 mass%) with 3 mm thick and 15 mm square. The surfaces and sides of the samples were polished, and R-type thermocouples were spot-welded to the top, bottom, and sides of the sample. SiO_2 powder (99.99 mass%) was applied to the top surface of the iron plate with 0.15 g. The samples were placed in a horizontal electric furnace, and the temperature was raised to 1373 K, 1423 K, and 1473 K in a nitrogen atmosphere, switched to an air atmosphere, and held for 30 min each. The temperature change of the sample was measured using the attached thermocouples. Figure 1 shows a postexperimental photograph of the iron plates held at 1373 K and 1423 K. The applied SiO_2 powder remained on the surface of the iron plate in the case of the sample held at 1373 K, whereas the surface of the sample held at 1423 K was oxidized and further roughening due to melting was observed. Similar results were obtained at 1473 K. The temperature measurements showed that at a holding temperature of 1423 K, the top surface temperature of the sample temporarily reached 1516 K. X-ray diffraction analysis showed that Fe_2SiO_4 and iron-based oxides were present in the sample after the experiment. From the above, it was confirmed that melting of the oxide scale occurred using the exothermic reaction of the oxidation of the iron plate, even though the temperature was kept at 1423 K, which is lower than the eutectic temperature of Fe_2SiO_4 and FeO[2].

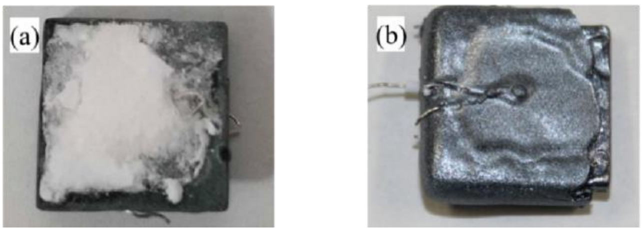

Fig. 1 Appearance of samples after the experiment at (a) 1373 K and (b) 1423 K

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OS2-2 L1E – Tuesday, 12 Sep. 11.40-12.00 – ROOM M2

Lock-in thermography of green powder metallurgy component flaws using preventive non-destructive imaging

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Pre-sintered ("green") compressed metal powder components were studied using Lock-In Thermography (LIT) to assess the capability of this methodology through non-destructive imaging to produce quantitative information on crucial material integrity properties used in automotive manufacturing that impact mechanical strength and integrity, such as density nonuniformity. Using pre-optimized experimental parameters, including infrared-camera phase stabilization by means of ultra-high sampling frequency rates, LIT was shown to reveal distinct phase shift bi-modalities and widespread non-uniformity in an industrial sample which exhibited surface cracks. In juxtaposition, LIT images from another sample without cracks were much more uniform across the entire sample. A regression fitting program was used to determine the local thermal diffusivity at critical phase-varying locations of the cracked sample and a positive correlation was found between the thermal diffusivity and the phase shift with a lone exception occurring at the exact location of the crack. Optical imaging of the sample also revealed a positive correlation between the phase shift and the local sample porosity which determines local density. The combination of these procedures led to the concept of preventive non-destructive evaluation (P-NDE) as an important new inspection approach which hinges on the premise of developing adequate analytical and measurement tools to investigate material integrity deficit factors during manufacturing that may lead to catastrophic flaws / cracks before the emergence of such flaws.

OS2-2 L2A – Tuesday, 12 Sep. 12.00-12.20 – ROOM O2

Isobaric heat capacities at high pressures of DEAE-water and EAE-water mixtures for CO_2 capture.

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Carbon capture and storage has become a promising technology for dealing with climate change. Amine-based carbon dioxide (CO_2) capture is a well-known and widely used technology in this field [1]. Thermophysical properties are crucial for the characterization of any fluid, including those involved in carbon capture processes. In this sense, isobaric heat capacity is fundamental in change in Gibbs energy, enthalpy, and entropy [2]. However, the necessary thermophysical properties over wide ranges of concentrations, temperatures, and pressures of mixtures involved in the process, are practically non-existent in the literature.

To cover this gap, we measured the isobaric heat capacity of two aqueous solutions of amines with a similar structure, being 2-diethylaminoethanol (DEAE) a tertiary amine and 2-ethylaminoethanol (EAE) a secondary amine. Experimental measurements were performed at amine mass fractions of 0.1; 0.2; 0.3 and 0.4 over a range of temperatures from (293.15 to 353.15) K and pressures up to 20 MPa. For this purpose, an automated quasi-isothermal flow calorimeter was used, with an expanded relative uncertainty better than 1 %. This calorimeter was developed in our laboratory and details are given in [3].

The influence of these parameters (amine mass fraction, temperature, and pressure) was studied. Both aqueous solutions of amine showed a similar behavior. An example can be seen in figure 1. For isobaric heat capacity, temperature has a slight effect and pressure has almost no influence on isobaric heat capacity. Increase amine mass fraction involves a rise in this property. There is no experimental data available in literature for isobaric heat capacity of these systems, therefore comparison was not possible.

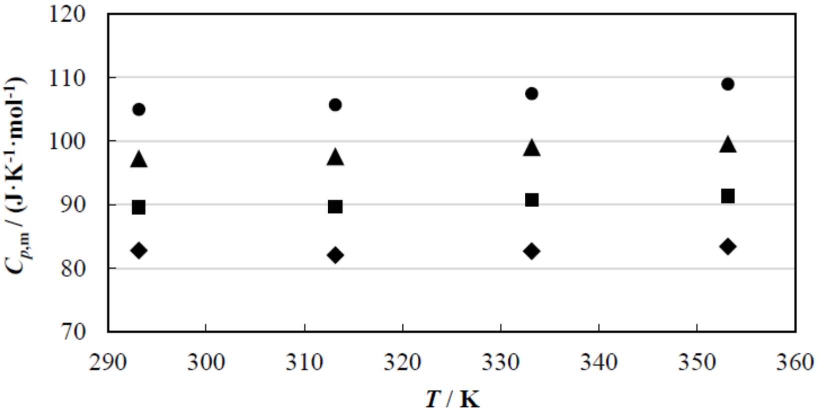

Fig. 1. Experimental molar isobaric heat capacity of EAE (1) + H_2O (2); at the atmospheric pressure (0.1 MPa) as function of temperature and amine mass fraction: (♦) $w_1 = 0.1$; (■) $w_1 = 0.2$; (▲) $w_1 = 0.3$; (●) $w_1 = 0.4$.

Acknowledgment

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Combining Machine Learning with Thermodynamic Group-Contribution Methods

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Information on thermophysical properties of mixtures is paramount for process design and optimization in chemical engineering. However, as experimental data are in general scarce, prediction methods are essential in practice. During the last decades, an enormous effort has been carried out for developing and refining physical prediction methods for mixtures. Among them, group-contribution (GC) models of the Gibbs excess energy (GE), like UNIFAC, are in particular successful, since they can be used for describing a very large number of components based on a relatively small number of group-specific parameters. However, there are still significant gaps in the tables of the pair-interaction parameters, which describe the binary interactions between structural groups of the components in the mixture, which is due to the fact that fitting new parameters is extremely elaborate or partly impossible due to insufficient amounts of experimental data. This still greatly limits the applicability of GC models to date.

We have recently introduced Matrix Completion Methods (MCMs) from Machine Learning (ML), where they are widely used in recommender systems, as an alternative to established physical methods for predicting different properties of mixtures [1-6]. The basic idea of using MCMs is that the data for the properties of binary mixtures can conveniently be organized in matrices with rows and columns representing the components that make up the mixtures. These matrices are usually only sparsely occupied with experimental data and MCMs were used for predicting the missing entries, achieving higher accuracies than the physical baselines.

In the present work, we have developed a new hybrid approach by combining the MCM concept with GC GE models [7]. The idea thereby is to use an MCM to complete the matrix of pair-interaction parameters among structural groups of the GC model, which substantially extends the scope of the GC model. We exemplify our hybrid approach by embedding an MCM into the UNIFAC model, and using it for predicting the pair-interaction parameters between the UNIFAC main groups. The hybrid model was trained on synthetic data for activity coefficients obtained with published UNIFAC parameters as well as on experimental data from the Dortmund Data Bank. Excellent prediction accuracy for activity coefficients and phase equilibria for binary and multicomponent mixtures is found, while the scope of the model is substantially larger than that of the currently available versions of UNIFAC.

Our proposed hybrid approach thereby enables to combine the strengths of both, the physical and the ML world in an elegant way: it combines the ability of the physical GE model to consistently generalize over temperature and concentration and to extrapolate from binary to multi-component mixtures, with the ability of the MCM to give predictions for unreported parameters. Moreover, our approach is not restricted to the prediction of interaction parameters of GC GE models, but can in principle be applied to any model relying on binary parameters and therefore opens perspectives to a new generation of prediction methods and models in chemical engineering.

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OS2-2 L2D – Tuesday, 12 Sep. 12.00-12.20 – ROOM N1

Electrical Conductivity of CaO-Al_2O_3-CaF_2 melts

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The electro slag remelting (ESR) is one of the methods used to produce high cleanliness steel. In the ESR method, the product steel electrode is immersed in flux, and the electrode is melted by the Joule heat of the flux generated by the high current. The molten steel then passes through the flux, where it absorbs impurity sulfur and non-metallic inclusions, resulting in a clean steel. CaF_2-based melts are mainly used as fluxes, but the electrical conductivity of CaF_2-based melts varies widely among researchers and measurement methods. Therefore, the purpose of this experiment is to measure the electrical conductivity of CaF_2-based melts based on the van der Pauw method[1] , which does not require the cell constant, which is a fundamental error factor in electrical conductivity measurement at high temperatures.

The composition of the CaF_2-CaO-$AlO_{1.5}$ melt used in this study is shown in Table 1. Samples were melted in an Ar atmosphere after weighing and mixing reagents. The measurement temperature was set at 16001450 °C, the open circuit voltage was 2.0 V, and dehydrated Ar flowed through the furnace at 200 ml/min. Electrodes were fabricated by passing Pt-30mass%Rh through four alumina insulating tubes, and the electrodes were connected to an impedance analyzer using a four-terminal probe. The electrodes were immersed in the melt, and the impedance and phase angle at immersion depths of W= 5 mm, 10 mm, 15 mm, and 20 mm were measured by AC frequency sweep (50 Hz to 200 kHz). The electrical conductivity was then calculated by utilizing the real part values of the impedance when the imaginary component of the impedance was zero at two immersion depths.

The relationship between temperature and electrical conductivity of the CaF_2-CaO-$AlO_{1.5}$ melt measured is shown in Fig. 1. The electrical conductivity showed a positive temperature dependence. From No.1, 3 and 5, it was found that the electrical conductivity increased with increasing CaF_2 content, and decreased with increasing $AlO_{1.5}$/CaO ratio from No.2~4 and 5~7. The reason for the temperature dependence of electrical conductivity is thought to be that ions contribute greatly to electrical conductivity in CaF_2 melts, and as the temperature increases, the diffusion path of ions increases. The increase in the $AlO_{1.5}$/CaO ratio decreases the electrical conductivity because the Ca^{2+} that compensates for the charge on Al^{3+} increases with the increase in the $AlO_{1.5}$/CaO ratio. The increase in the $AlO_{1.5}$/CaO ratio is thought to be due to a decrease in the amount of Ca^{2+} that serves as a carrier.

Table 1. Chemical compositions of samples(mol%).

No.	CaF_2	CaO	$AlO_{1.5}$
1	40.0	30.0	30.0
2	30.0	40.0	30.0
3	30.0	35.0	35.0
4	30.0	30.0	40.0
5	20.0	40.0	40.0
6	20.0	26.7	53.3
7	20.0	22.0	58.0

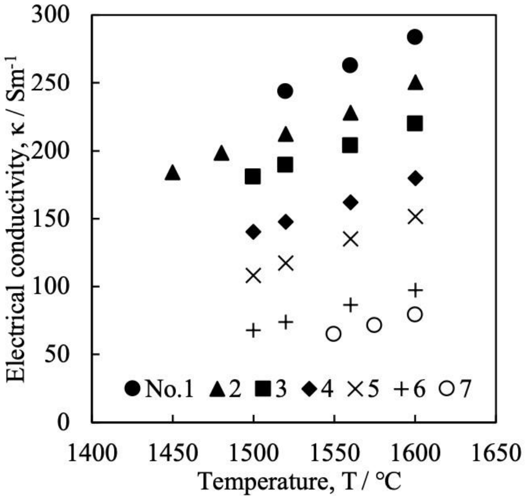

Fig. 1. Temperature dependence of electrical conductivity of CaF_2-CaO-$AlO_{1.5}$ melts.

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OS2-2 L2E – Tuesday, 12 Sep. 12.00-12.20 – ROOM M2

Thermal Diffusivity Measurement for µm-scale Single Wires Using Lock-in Thermography

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As heat dissipating materials for power electronics components, composite materials have been developed with high thermal conductivity fillers such as AlN and BN. There are researches to increase thermal conductivity with low filler content by controlling the direction of heat flow with fibrous filler with high aspect ratio. Although it is essential to understand thermophysical properties of the fibrous filler itself, which is the key to high thermal conductivity, it is quite difficult to measure the properties because fillers are very fine with µm scale. In this study, we developed a single-wire measurement method using a lock-in thermography (LIT) periodic heating method. This method uses a laser for heating and a LIT for measuring Temperature, and it is non-contact and does not require scanning, therefore, it is possible to measure quickly and accurately. In this paper, we report on the evaluation of the validity of the method for measuring fibrous fillers added to composite materials. Cu and Ni metallic wires were selected as samples and their surfaces were coated with Bi to increase laser absorption and infrared emissivity. The measurements were conducted under vacuum to reduce heat loss and were carried out at several frequencies. As a result, the measurements were within ±1.7% and ±11.3% of the bulk reference value for the Cu and Ni, respectively, which confirms the validity of the measurements. It was also confirmed that sample diameter and Bi coating had little effect on the results.

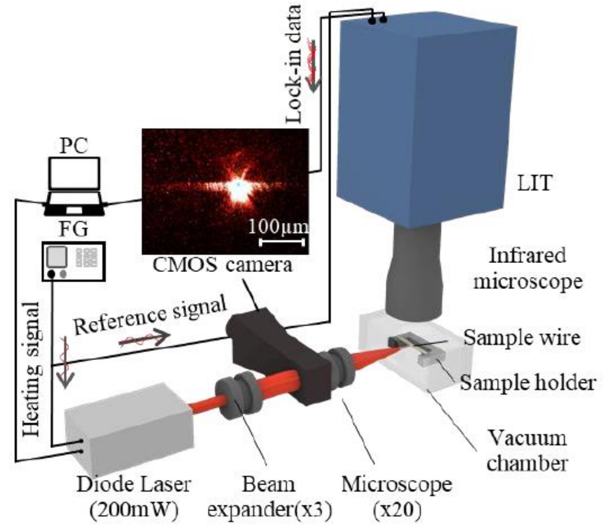

Fig.1 Schematic of measurement apparatus

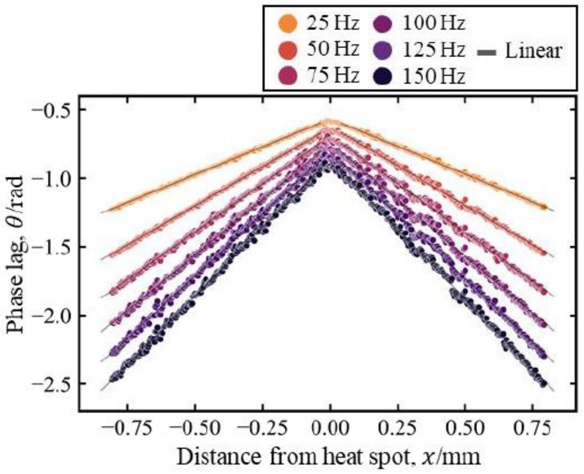

Fig.2 Phase lag profile of Bi-coated Cu 50µm plotted against distance from heating spot.

Table 1. Thermal diffusivity results of Cu and Ni wires.

Material	Diameter (µm)	Coating	D (10^{-6} m^2·s^{-1})		
			This work	Ref.	Difference
Cu	50	Bi	117	117	0%
	25	Bi	119		+1.7%
Ni	50	Bi	20.3	22.9	-11.4%
	50	-	20.5		-10.5%

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OS2-2 L3A – Tuesday, 12 Sep. 12.20-12.40 – ROOM O2

Measurement and modelling of thermophysical properties of CO_2-brine mixtures

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Carbon capture and storage (CCS) and geothermal energy extraction are alternative approaches to fossil fuels considered to effectively reduce CO_2 emissions in the atmosphere. One possibility is to store CO_2 in depleted petroleum reservoirs or saline aquifers. Another approach is to use CO_2 as a working fluid to enhance heat transfer from depth to surface in geothermal reservoirs. The correct prediction of the phase equilibrium conditions of CO_2 + brine mixtures and the long-term trapping of CO_2 in the saline aquifer require the accurate knowledge of the PT-x properties of these fluids at reservoir conditions. While the majority of experimental and modelling works have used NaCl as the only salt component in the brine [1], the presence of other salts can affect the brine properties differently. Therefore, it is important to obtain new dataset for the thermophysical properties of CO_2 + brines containing multiple salts. In this work, the solubility of CO_2 in brine solutions containing NaCl, $CaCl_2$, $MgCl_2$, $SrCl_2$, KCl, $NaHCO_3$, $BaCl_2$ and Na_2SO_4 was measured by using an experimental gravimetric method, at 303 and 333 K, from 5 to 40 MPa, and for salinities up to 2 m. A vibrating U-tube densimeter and a capillary viscometer were used to measure the density and viscosity of brines loaded with controlled amounts of CO_2 at the same conditions. For solubility modeling, the Søreide-Whitson model [2] was generalized to brine containing NaCl, KCl, $MgCl_2$, $CaCl_2$ and Na_2SO_4 for temperatures between 273 and 473 K, pressures up to 200 MPa and salinities up to 6 M. The generalized model could also be used to calculate the solubility in multiple-salt brine. The results of our study showed that the modified model considerably improves the description of the individual salt brines and calculates the CO_2 solubility in a large variety of brine solutions, including our measurement, with good accuracy when compared with previous parameterisations. The excess volume method [3] was used for the density modeling, and a friction-theory approach was used for the viscosity modeling.

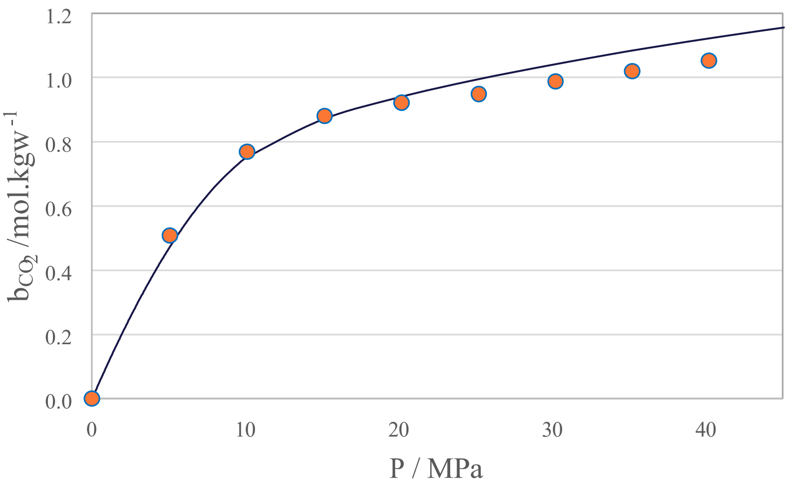

Fig. 1. Experimental data for CO_2 solubility in a synthetic brine at 60 °C and modelling with a single-salt approach.

Acknowledgment

Funding from the Greensand Project Consortium is gratefully acknowledged.

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OS2-2 L3C – Tuesday, 12 Sep. 12.20-12.40 – ROOM N2

Group contribution-based graph convolution network: A versatile model for estimating thermodynamic properties

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Properties data for chemical compounds are crucial for designing and operating chemical processes. The group contribution method is widely used and straightforward for predicting properties. However, this method has inherent inaccuracy due to the simplified representation of the molecular structure. We propose a compromise between a complex machine-learning algorithm and a linear group contribution method to address this [1]. A functional group graph represents molecules, reducing the number of adjustable parameters for machine learning and resulting in higher accuracy than conventional methods. The process was examined in various aspects, including incorporating uncertainties in the data, the robustness of the fitting process, and detecting outlier data. In addition, we suggest merging a machine learning model with the COSMO-SAC activity model to predict vapor-liquid equilibrium (VLE) [2]. The quantum chemical calculations in the COSMO-SAC model are computationally demanding. In this work, COSMO calculations are replaced by employing a machine learning model. We assessed the accuracy of machine learning models by comparing their predictions of both COSMO calculations and VLE results using molecules that were not part of the machine learning training process. A new COSMO calculation database was generated to improve model performance.

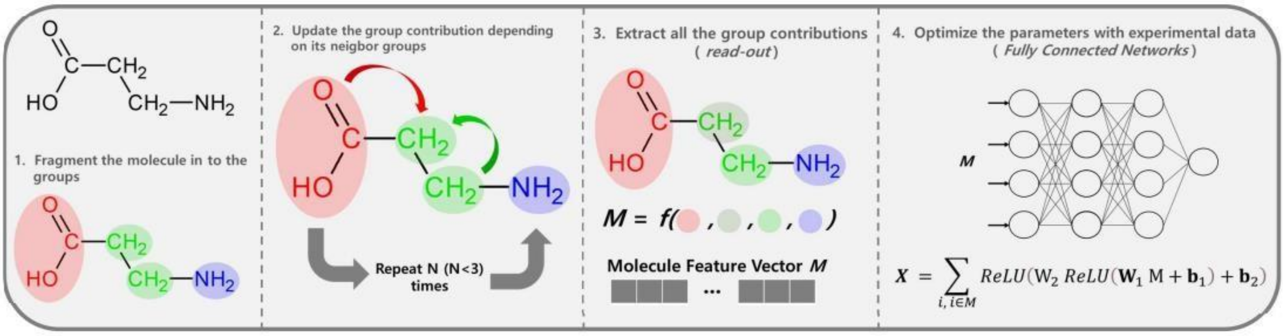

Fig. 1. Schematic diagram calculation process for the Group Contribution Graph Convolution Network.

Acknowledgment

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OS2-2 L3D – Tuesday, 12 Sep. 12.20-12.40 – ROOM N1

Electrical conductivity modelling of multicomponent molten slags

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Electrical conductivity is one of the fundamental properties determining the performance of pyrometallurgical processes, e.g., the power supply requirements of operating electric smelting furnaces. Together with viscosity and other thermophysical properties, they help understand molten slags' structure to solve specific industry challenges and improve product quality [1]. However, measuring electrical conductivity still involves difficulties and high uncertainties (sometimes up to 30%). Therefore, predicting electrical conductivity with a reliable model is highly important. Almost all the available models are polynomial fit to a range of slag compositions without considering the slag structure and conduction mechanisms. The present electrical conductivity model incorporates the contributions from all cations including the network-forming cations modeled as neutrals in the two-sublattice ionic liquid thermodynamic model. The electrical conductivity is predicted taking into account of both the ionic conductions and the electric conductions with Fe^{+2}/Fe^{+3} bearing slags. Combing with TCOX12 [2], the thermodynamic database of oxides which predicts the liquid structure e.g., the distribution and connectivity of cations in the oxide melts, the electrical conductivity is readily calculated in the whole composition and a broad temperature range. In the present work, comparisons between experimental and calculated data are provided. The performance of the model is systematically analyzed and discussed.

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OS2-2 L3E – Tuesday, 12 Sep. 12.20-12.40 – ROOM M2

Optimal Experiment Design for Thermal Property Measurement of Orthotropic Materials using Infrared Thermography

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The optimal set-up for a two-dimensional (2D) transient heat conduction experiment aimed at estimating simultaneously the thermal properties of orthotropic materials when using infrared (IR) thermography is designed. To this aim, a D-optimum criterion [1-2] ensuring the minimization of the confidence region of the estimated parameters is applied to define the optimum heating and experiment times. The optimal experiment is also sought in terms of the aspect ratio of the sample and the width of the heated region. In fact, the experimental apparatus here considered consists of a thin electrical heater between two larger samples of the same material and thickness. Moreover, temperature measurements useful for the estimation procedure are obtained non-intrusively from the unheated surface of the sample (exposed to the environment) using an IR camera [3]. For this reason, the heat transfer coefficient (h) is regarded as an unknown of the inverse problem, as well as the directional thermal conductivities (k_x and k_y) and volumetric heat capacity of sample (C).
The 2D heat conduction problem is modeled through a rectangular plate (i.e., the sample) partially heated at the front boundary through a surface heat flux, while only the opposite boundary is subject to convection with the ambient air (all the other boundaries are kept insulated). Once the temperature solution to this problem is obtained starting from a generalized analytical solution available in the literature [4], the superposition principle is also applied to account for a finite heating period. Then, the so-called scaled sensitivity coefficients of temperature with respect to the parameters of interest are computed, and a sensitivity analysis is performed (see Fig. 1). Finally, the optimal experiment is designed through the so-called Δ^+ criterion [1-2].

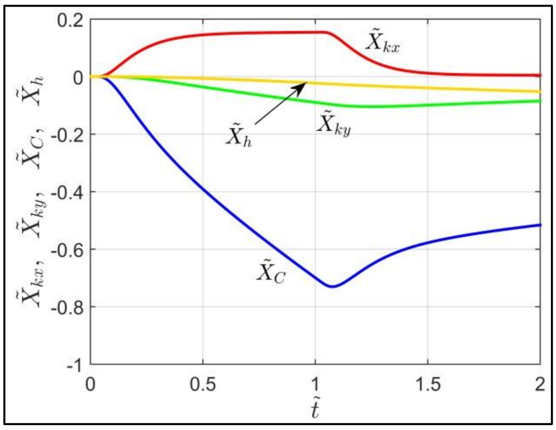

Fig. 1. Dimensionless scaled sensitivity coefficients at the unheated surface of the sample with respect to k_x, k_y, C, and h, as a function of time and for a heating per $\tilde{t}_h = 1$

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OS2-2 L4A – Tuesday, 12 Sep. 12.40-13.00 – ROOM O2

Molecular modeling of the CO_2 interactions in Phosphonium-based Ionic Liquids through SAFT and COSMO approaches

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While amines remain as the most common type of solvent for CO_2 capture, it is well known that they evaporate and degrade during process operation, with environmental and economic consequences. In addition to these problems, there is nowadays a need to find alternatives that can be used at different gas compositions, even though at very low ones, as it occurs in applications related to Direct Air Capture.

Ionic Liquids (ILs) have seen during several years as an interesting type of fluids due to their low vapor pressure and the possibility to "design" them by combining different cations and anions for each particular case, considering the flue stream composition and specific conditions. Particularly, some phosphonium cation/anion combinations have been studied in literature with promising results. Nonetheless, restricted and limited laboratory data is available and a complete characterization of these compounds is still required to select the most appropriate CO_2 absorber.

In this work, the potential of phosphonium based ILs as CO_2 absorbers at different operating conditions for industrial application is assessed through the use of an accurate statistical mechanics-based equation of state (EoS). Particularly, the soft-Statistical Association Fluid Theory (soft-SAFT [1]) is found to provide a suitable description of ILs and their behavior in mixtures, and will be applied here, in combination with quantum-chemical approaches, such as Turbomole-COSMO, to obtain the charge distribution profiles and describe the key interactions in these compounds. The work departs from the trihexyltetradecylphosphonium cation $[P_{66614}]^+$, which is combined with different anions. The resulting ILs are characterized by a complete description of their pressure-temperaturedensity diagrams, and derivative properties [2]. Transport properties, such as the viscosity, are also modeled using the Free-Volume theory coupled to soft-SAFT EoS. Then, CO_2 absorption isotherms are described and compared to experimental data when available. The CO_2 absorption capacity of the different ILs, considering both diluted and concentrated mixtures in CO_2, is assessed through the calculation of Henry's law constants and the solvation enthalpies and entropies at different conditions, proposing a preliminary list of potential compounds for different types of flue gases.

Acknowledgment

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OS2-2 L4C – Tuesday, 12 Sep. 12.40-13.00 – ROOM N2

Similarity-based Prediction of Activity Coefficients

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For process design and optimization in chemical engineering, the knowledge of the thermodynamic properties of mixture is essential. Among the most important mixture properties are activity coefficients, which describe the deviation from an ideal mixture and are the crucial quantities for the description of, e.g., phase equilibria. However, since experimental data for activity coefficients, as for mixture properties in general, are missing for many mixtures of interest in practice, prediction methods for activity coefficients are highly needed.

In the present work, we have developed a novel, completely data-driven approach for the prediction of the activity coefficients $\gamma_{i,j}^{\infty}$, of solutes *i* at infinite dilution in solvents j using the concept of similarity-based imputation. The underlying idea is that "similar" solutes (solvents) should lead to similar $\gamma_{i,j}^{\infty}$, if mixed with the same solvents (solutes). Following this concept, $\gamma_{i,j}^{\infty}$, for unstudied mixtures can, thus, be predicted based on available data for similar mixtures. For quantifying the similarity among solutes (solvents), we introduce a new metric based on quantum-chemical component descriptors used in the COSMO-SAC model [1], which is then applied in the prediction of $\gamma_{i,j}^{\infty}$, for unstudied mixtures. We have trained the model on data for 416 solutes and 255 solvents from the Dortmund Data Bank and have evaluated the predictive accuracy using a leave-one-out strategy. The results demonstrate that the new method outperforms physical state-of-the-art models like COSMO-SAC [1] and UNIFAC [2] in terms of predictive accuracy. Furthermore, we have combined the approach with matrix completion methods from machine learning [3-8], which led to a further substantial increase in both scope and prediction accuracy.

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OS2-2 L4D – Tuesday, 12 Sep. 12.40-13.00 – ROOM N1

Link between Structures and Thermophysical Properties of Melts for the Primary Elements of Metallic Glasses

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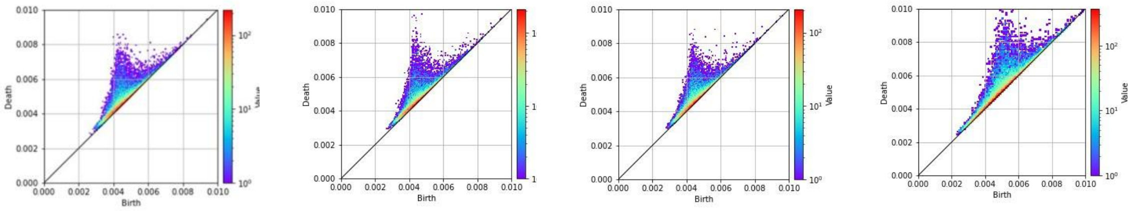
The link between glass-forming ability of metallic glasses and the structure of their melts has been a longstanding issue. In recent years, significant progress has been made in the study of structure-property relations in liquid and amorphous metal oxides using machine learning [1]. These approaches are also applicable to metallic melts at high temperatures. Therefore, we measured structure data of high-temperature melts, which are main constituent elements of metallic glasses, using synchrotron radiation X-rays combined with aerodynamic levitation techniques. Then, reverse Monte Carlo (RMC) modelling to the X-ray diffraction data and persistent homology (PH) analysis [2] were performed to investigate the relations with thermophysical properties such as molar volume and entropy [3]. The melt structure features extracted from the atomic arrangements from the RMC results are in good agreement with the thermophysical properties investigated in this study.



(a) Fe at 1820 K (b) Zr at 2120 K (c) Hf at 2510 K (d) Si at 1700 K

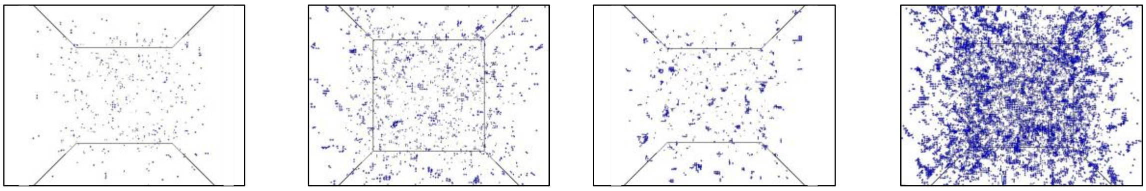


Fig. 1. Persistence diagrams (upper side) and void distributions (lower side) obtained from the RMC results of liquid metals and Si.

Acknowledgment

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OS2-2 L4E – Tuesday, 12 Sep. 12.40-13.00 – ROOM M2

IR thermography applied to the characterization of thermoelectric materials

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IR thermography, thanks to its imaging capability is an interesting instrument to carry out photothermal experiments for the characterization of the thermal conductivity components of an orthotropic material (see Fig. 1). Two active thermographic techniques are considered. In the first a laser pulse, spatially localized, heats the material under test. IR thermography collects a sequence of images of the transient phenomena that are spatially decomposed by means of the Fourier Transform and successively analysed in time to determine the thermal diffusivity [1]. In the second, a laser source is modulated to produce thermal waves. The temperature field is successively analysed in phase and amplitude to retrieve the components of thermal diffusivity, in-plane and through the thickness [2]. The measurement of the specific heat and the density allows for the assessment of thermal conductivity in the aforementioned directions. The technique is applied to polyaniline doped materials for thermoelectric applications (see Fig.2).

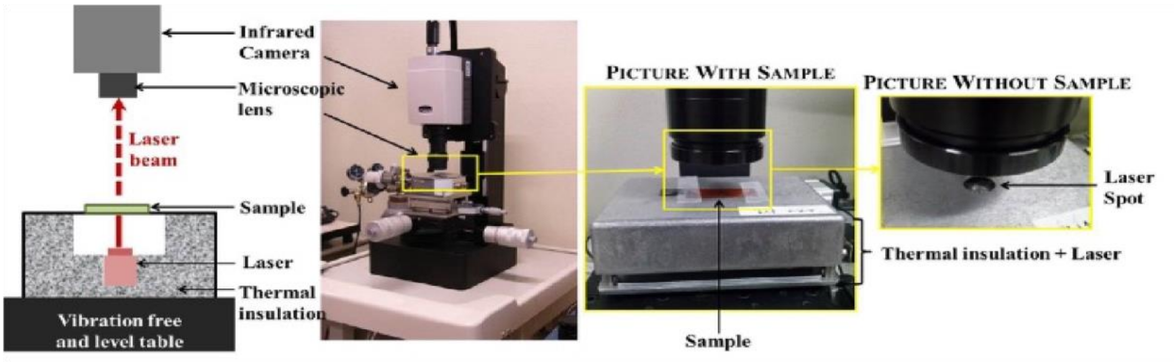

Fig. 1. Experimental lay-out for IR thermographic microscopy.

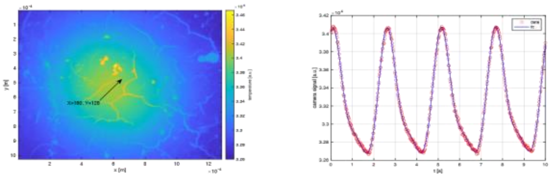

Fig. 2. On the left IR image of polyaniline doped materials; on the right the temperature oscillation on the pixel indicated in the IR image.

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Surface Tension of HFCs, HFOs, and Their Mixtures Probed by the Linear Gradient Theory and Molecular Dynamic Simulations

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To reduce the environmental issues associated with traditional refrigerants, extensive studies have recently been increasingly focused on the low global warming potential (GWP) refrigerants, also known as fourth-generation refrigerants. Surface tension plays an essential role in phase transitions as well as technical processes like boiling and condensation, which is a crucial fluid property for associating and predicting heat transfer and pressure drop during a phase change. Thus, accurate surface tensions of the refrigerants are necessary for predicting their flow behavior and convection characteristics in numerous refrigeration apparatuses.

In this work, we collect the available experimental data after a careful literature survey. The surface tensions for HFCs, HFOs, and their mixtures are well modeled. The predictive method combines the Peng−Robinson (PR), Volume−Translated SRK (VTSRK), and Cubic−Plus−Association (CPA) Equation of States (EoS) with the linear gradient theory (LGT). To minimize the deviations between the predicted data and the experimental data, a new correlation for the influence parameter is proposed that is a function of the densities of the bulk phases. Moreover, molecular simulation offers a complementary approach to predicting reliable thermophysical properties and providing molecular-level understanding. The surface tensions of HFCs, HFOs, and their mixtures have been predicted using molecular dynamics (MD) simulation over a broad temperature. A good consistency is achieved between the experimental measurements, LGT calculations, and MD simulations. The density profiles in the vapor-liquid interfacial region are also fully investigated for certain components. These findings are expected to provide a useful tool for investigating the properties of heat and mass transfer in refrigeration systems.

Acknowledgment

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OS2-3 L1B – Tuesday, 12 Sep. 14.30-14.50 – ROOM O1

Improving the transport properties of ionic liquids by blending with fluorinated carbonates

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Ionic liquids (ILs) have got significant attention in the fields of lithium metal batteries (LMBs) owing to a range of desirable properties such as large liquid range, nonflammability and wide electrochemical voltage windows etc. However, it suffers from high viscosity and poor ionic conductivity [1-2]. Mixing ILs with conventional carbonate solvents as additives/cosolvents is considered as immediate practical solution. While the conventional carbonate solvents are less compatible with lithium metal anode, hindering the application of ILs in LMBs. As an additive derived from carbonate solvents, fluorinated carbonates not only retain the low viscosity of carbonate solvents, but also provide better cycling performance for LMBs [3]. In this work, fluorinated carbonates are used as additive to reduce viscosity and improve ionic conductivity of ILs. The densities, kinematic viscosities and ionic conductivities of 1-Methyl-1-propylpyrrolidinium bis(fluorosulfonyl)imide（$[Pyr_{13}][FSI]$） + fluorinated carbonates mixtures were measured in the temperature range from (298.15 to 343.15) K and at pressure of 0.1 MPa with the pycnometer, Ubbelohde capillary viscometer and conductivity meter, respectively. The results show that fluorinated cyclic carbonates can greatly reduce viscosity, increase the ionic conductivity and density in the most case. The dependence of temperature on viscosities and ionic conductivities have been fitted to the Arrhenius equation with high precision, respectively. Additionally, the effect of concentration of fluorinated cyclic carbonates on the electrical conductivity has been examined using the Walden rule.

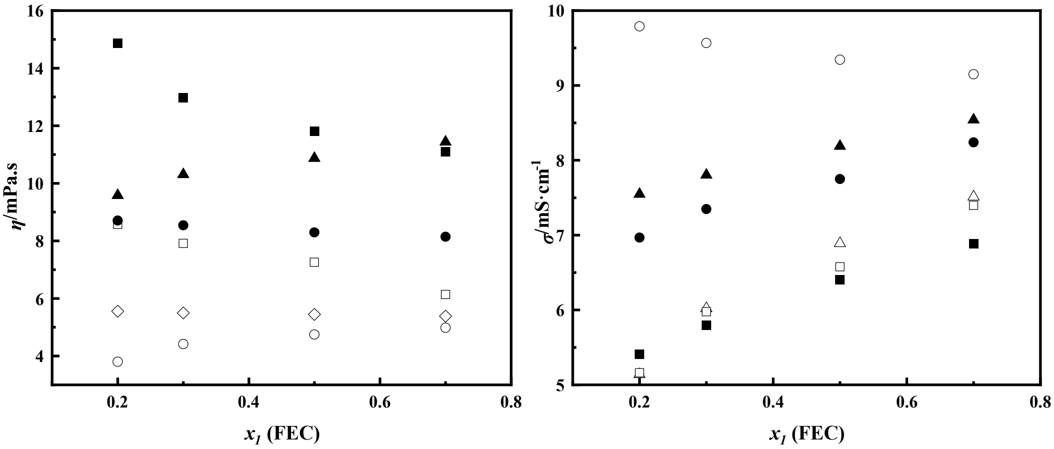

Fig. 1. Properties of the mixtures of $[Pyr_{13}][FSI]$) + fluorinated carbonates at 298.15 K and 0.1 MPa: (a) viscosity; (b) electrical conductivity. ●, $[Pyr_{13}FSI]_7:[FEC_{x1}:EMC_{x2}]_3$; ○, $[Pyr_{13}FSI]_5:[FEC_{x1}:EMC_{x2}]_5$; ▲, $[Pyr_{13}FSI]_7:[FEC_{x1}:FEMC_{x2}]_3$; △, $[Pyr_{13}FSI]_5:[FEC_{x1}:FEMC_{x2}]_5$. ■, $[Pyr_{13}FSI]_7:[FEC_{x1}:HFDEC_{x2}]_3$; □, $[Pyr_{13}FSI]_5:[FEC_{x1}:HFDEC_{x2}]_5$.

Acknowledgment

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OS2-3 L1C – Tuesday, 12 Sep. 14.30-14.50 – ROOM N2

Experimental and numerical study of the thermal radiative properties of Al/air flames

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Aluminum microparticles have been proposed as green energy vectors, given their high combustion enthalpy and the availability of clean recycling routes for the oxide products. Unfortunately, most studies in the field have dealt with either nanometric or very large Al particles, each with their own disadvantages. Combustion of Al spheres in the 1-10 µm range has been suggested as a compromise solution, but the properties of these systems are poorly known.m range has been suggested as a compromise solution, but the properties of these systems are poorly known. This contribution presents an experimental setup and an optical model to study the radiative properties of these flames [1].
The studied flame is formed by independent Al particles burning in vapor phase, from which liquid Al_2O_3 nanoparticles are formed by condensation. In contrast to the gas phase, the emission by the condensed products does not have an exact solution. A direct electromagnetic simulation of this system is computationally expensive, so a homogenization procedure has been used, where the particulate nature of the alumina cloud is modeled using the Maxwell-Garnett approximation. The emissivity of each burning particle can be solved exactly using the core-shell Lorenz-Mie solution, allowing for different temperatures in each layer. We found that the complex refractive index of liquid alumina had to be modified with a term corresponding to pre-gap optical absorption (the Urbach tail) to correctly reproduce the results in the visible range. This simplified model constitutes a starting point for more in-depth studies of heat transfer and pyrometric temperature measurements in Al/air flames.

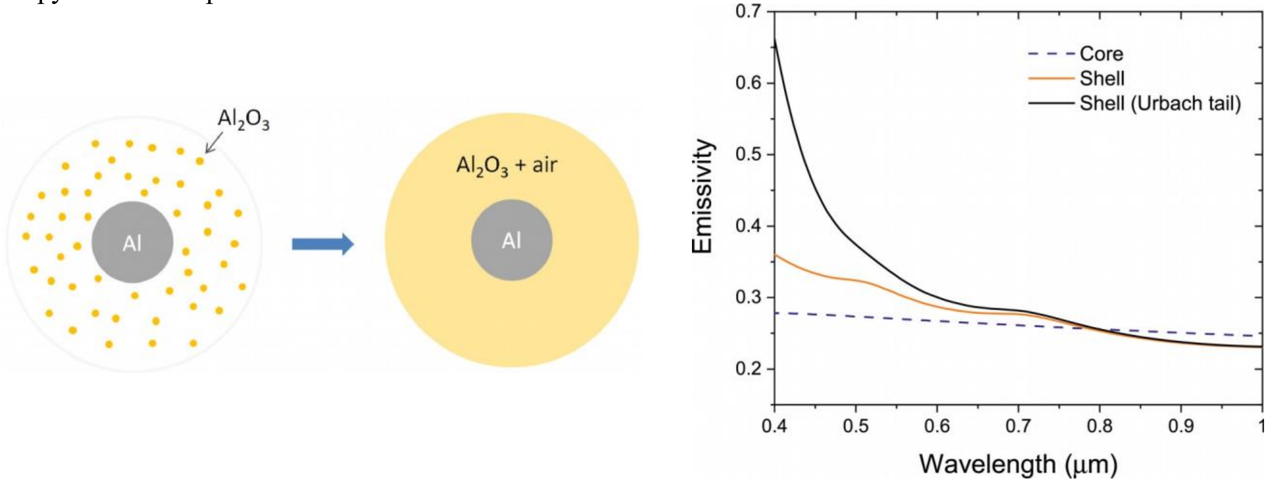

Fig. 1. Left: Geometry of the burning process and homogenization approach. Right: Calculated emissivities for the Al core at 2790 K and Al_2O_3/air shell at 3200 K. The effect of accounting for the Urbach tail in the calculation is shown.

Acknowledgment

This work is part of two projects funded by the Agence Nationale de la Recherche (ANR-19-CE05-0021, ANR18-CE05-0040). I. González de Arrieta acknowledges support from the Basque Government by means of a postdoctoral grant (POS-2021-2-0022).

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OS2-3- L1D – Tuesday, 12 Sep. 14.30-14.50 – ROOM N1

Metrological improvement of a high temperature commercial calorimeter by embedding an in-situ electrical calibration system

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The main drivers behind the development of high-temperature advanced materials motivate the increasing demand of high temperature measurement techniques for thermal conductivity, thermal diffusivity, specific heat and enthalpy of fusion. Differential scanning calorimeters (DSCs) which are applicable to a wide range of scientific and technological research fields involving physical, chemical and biological processes can be used to study the thermophysical properties at high temperatures. The calibration of these instruments is usually done using certified reference materials in temperature, enthalpy of fusion or specific heat depending of the required measurement. The use of in-situ electrical calibration system, enabling to have experimental conditions of calibration and measurement as similar as possible, yields to highly accurate determination of the sensitivity of the DSC.

A new in-situ high temperature electrical calibration system was developed at LNE-LCM and integrated into a commercial heat flux calorimeter MultiHTC from the company Sétaram (France) in order to perform accurate and reliable measurements of enthalpy of fusion that are directly traceable to the International System of Units (SI). First determinations of the enthalpy of fusion of pure copper performed at LNE at 1084 °C (as illustrated in figure 1) are promising.

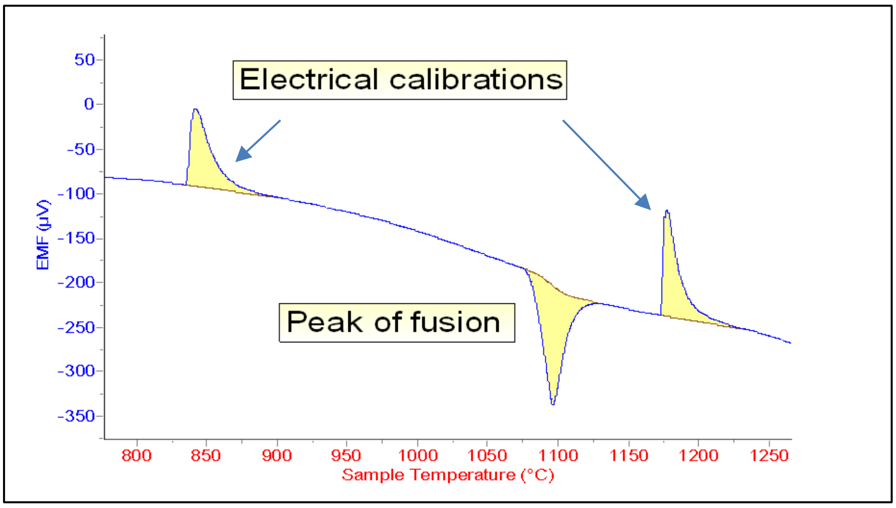

Fig. 1. Electrical calibration and measurement of enthalpy of fusion performed during the same heating run at 5 K/min.

This paper gives a description of the new metrological improvement of the commercial DSC for the measurement of the enthalpy of fusion at high temperature, where the offer of certified reference materials above 660 °C (aluminum) is totally inexistent.

Thermal conductivity measurements for the hydrochloroolefin R1130(E)

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The identification of new refrigerants characterized by low GWP (<150), as required at international level by several agreements and regulations, is still far from the conclusion. In particular, for a proper selection, the thermophysical properties of hydro(chloro)fluoroolefins (H(C)FOs) are required, but their knowledge is still scarce for several of these fluids. Amongst these, R1130(E) has recently get some attention as a component, with R1336mzz(Z), of the azeotropic binary mixture (R514A), that could be applied as a substitute for R123 in centrifugal chillers, high-temperature heat pumps, and organic Rankine cycles. R1130(E) is a hydrochloroolefin characterized by a relatively high normal boiling temperature (320.88 K) and belongs to the ASHRAE safety group B1. Its properties are still not widely studied and, in particular, no data are available to our knowledge for the thermal conductivity. Thus, in this paper, a set of experimental thermal conductivity data, performed with a double THW apparatus, will be presented. The data are measured in the range of temperatures between 250 K and 320 K, with pressures up to 10 MPa.

Monte-Carlo-based data evaluation for surface light scattering (SLS) experiments in the presence of line-broadening effects

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Surface light scattering (SLS) is a well-established technique for an absolute determination of viscosity and interfacial tension of transparent fluids in a non-invasive way at macroscopic thermodynamic equilibrium with typical expanded uncertainties ($k = 2$) of 2% and below [1-2]. Continuous developments of the SLS technique open up further application possibilities in thermophysical property research with strong reference to process engineering, including its on-line or in-line operation. Here, process-relevant fluids are often opaque and nontransparent, which is why SLS experiments need to be performed in reflection geometry employing small wave vectors q of the probed surface fluctuations. The use of small wave vectors is not only benefical with respect to higher scattering intensities allowing for faster data acquisition, but also because of the shift towards an oscillatory behavior of surface fluctuations reflected by the measured correlation functions (CFs). This favors a simultaneous determination of viscosity and interfacial tension.

In the range of small wave vectors, i.e. q values below about 1×10^5 m^{-1}, the evaluation of the CFs is, however, hindered by the presence of line-broadening effects [3]. They originate from the finite sizes of the illumination and detection optics and result in a geometrical and diffraction-based spread of wave vectors in combination with possible shifts in the main wave vector. Until today, empirical evaluation methods commonly employed in literature are not able to account for line-broadening effects in the range of small q values reliably, which is reflected by erroneous values for viscosity and interfacial tension. To address this issue, physically solid evaluation approaches are required, yet lacking so far.

The aim of the project behind the present contribution is to develop the SLS method for an accurate determination of viscosity and interfacial tension of non-transparent fluids in the range of small wave vectors. The CFs obtained in this range represent a distribution of damped oscillations of varying frequency and damping, which cannot be decomposed in an analytical way. Therefore, a non-empirical data evaluation strategy was established which decomposes the measured CF into a sum of damped oscillations via a Monte-Carlo-based algorithm and subsequently performs an in-depth analysis of the corresponding amplitude distributions [4]. Based on measurements in reflection geometry on the transparent reference fluid toluene, it is demonstrated that the application of the new evaluation strategy allows for a reliable determination of viscosity and interfacial tension also in the presence of line-broadening effects. Even for q values down to 0.8×10^5 m^{-1}, the obtained values for viscosity and interfacial tension agree within 3% with reference values.

Acknowledgment

The authors gratefully acknowledge funding of the Erlangen Graduate School in Advanced Optical Technologies (SAOT) by the Bavarian State Ministry for Science and Art.

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OS2-3 L2B – Tuesday, 12 Sep. 14.50-15.10 – ROOM O1

Effective thermal conductivity and specific heat capacity measurements of battery cells

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The effective thermal conductivity $\lambda_{eff}(T)$ in different directions of battery cells as well as their specific heat capacity $c_p(T)$ is of essential importance for the understanding of the thermal behaviour and thermal management of battery packs [1]. Especially for cylindric cells lithium-ion batteries, destructive radial thermal conductivity measurements can be conducted via the pipe method [2].

This study focuses on the use of already available and standardized measurement devices like Laser Flash (LFA), Differential Scanning Calorimetry (DSC) and Heat Flow Meter (HFM) to evaluate these properties. LFA and DSC were used to determine the thermal diffusivity $a(T)$ in plane direction and $c_p(T)$ of anode, cathode, separator, and pouch materials of a lithium-ion pouch cell. The HFM method was applied to evaluate $c_p(T)$ and $\lambda_{eff}(T)$ of a lithium-ion pouch cell perpendicular to the pouch surface at different State of Charge (SoC). Additionally, the HFM method was validated for the evaluation of $\lambda_{eff}(T)$ of cylindric battery cells as passive component by development of a measurement procedure based on the embedding of cylindrical cells in a cuboid matrix.

Fig. 1. left: Pouch cells stacked in HFM; right: Cylindric lithium-ion battery cells in HFM

The HFM results of the investigated pouch cells through plane measurement (Fig. 1 left) showed a good reproducibility for $\lambda_{eff}(T)$ with $\lambda_{eff} = 0{,}715$ W m^{-1} K^{-1} at $T = 25$ °C with an expanded combined uncertainty of $U_{(k=2)} = 0{,}02$ W m^{-1} K^{-1}. Differences of $\lambda_{eff}(T)$ due to the SoC could not be resolved in the results. The $c_p(T)$ of the pouch cell based on HFM resulted in $c_p(T) = 0{,}944$ J g^{-1}K^{-1} with $U_{(k=2)} = 0{,}035$ J g^{-1}K^{-1}. The determined data for $c_p(T)$ and $a(T)$ in-plane direction of the pouch component were processed in a Finite Element (FE) Modell to calculate the in-plane thermal conductivity of the whole pouch cell with $\lambda_{eff} = 52{,}54$ W m^{-1} K^{-1}.

The validation procedure for $\lambda_{eff}(T)$ of the HFM method for cylindric samples with standard materials (Borosilicate glass) showed deviations below 5% between the measurement and FE simulation results. After the validation, cylindric cells were measured with the HFM (Fig. 1 right) resulting in $\lambda_{eff} = 1{,}045$ W m^{-1} K^{-1} at $T = 20$ °C with $U_{(k=2)} = 0{,}09$ W m^{-1} K^{-1}.

The evaluated results show, that HFM is a suitable non-destructive method to analyze the eff. thermal conductivity through-plane for pouch cells and tangential/radial for cylindric cells as well as for specific heat capacity measurements. The eff. thermal conductivity in-plane direction can be determined by dissecting the cell to its components to determine the in-plane thermal diffusivity as well as the specific heat capacity and density.

This data can be processed in a FE Modell to evaluate the in-plane effective thermal conductivity.

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OS2-3 L2C – Tuesday, 12 Sep. 14.50-15.10 – ROOM N2

Measuring the spectral emissivity up to 3000 K

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In high temperature applications (e. g. space aeronautics and fuel-based energy production) material safety, the cost and energy use during production and chemical resistance are critically affected by the thermophysical properties of the materials in use. Especially optical properties such as the spectral emissivity play a key role in radiative energy transfer and temperature measurement via radiation thermometry. Because surface conditions are easily affected by chemical reactions at elevated temperatures and the dominant mode of heat transfer is conducted via radiation, the emissivity itself is notoriously difficult to measure.

The Joint Research Project EMPIR Hi-TRACE ("Industrial Process Optimization Through Improved Metrology of Thermophysical Properties") aims to provide reliable and SI-traceable measurement techniques for thermophysical properties in the high temperature range up to 3000 K. For the measurement of the emissivity of solid materials measurement set-ups at PTB, CAE and TU Graz, which are based on radiometric or calorimetric principles and use pulse or induction heating, were advanced, validated with respect to the achievable measurement uncertainty and experimentally compared.

The PTB uses a dynamic method to measure the spectral emissivity (ADeM) in the temperature range from 750 °C up to 2500 °C based on a modified Laser-Flash- apparatus [1]. The sample is inductively heated to the desired temperature and is than hit by a short, high energy laser pulse. The emissivity at the laser wavelength of 1064 nm can be determined by measuring the absolute laser energy incident at the sample front side and the absolute, adiabatic temperature rise of the backside of the sample. An additional array-spectrometer is used to extend the measurement capabilities to the spectral range from 500 nm to 1100 nm.

The CAE uses the emissivity measurement apparatus (EMMA) which compares the spectral radiance of the sample to that of a blackbody reference at the same temperature to determine the spectral emissivity in the wavelength range from 1 μm to 20 μm [2]. Sample and blackbody are inductively heated in a temperaturecontrolled vacuum vessel to account for background radiation.

At TU Graz the normal spectral emissivity of solid and liquid samples is measured by a combination of an ohmic pulse heating (OPA) apparatus and a μs division of amplitude polarimeter (DOAP) [3]. The OPA rapidly heats up the wire shaped sample while the DOAP analyses the polarisation states of a laser beam reflected at the sample to determine the emissivity at the laser wavelength of 684.5 nm.

Reference measurements of the spectral emissivity have been conducted on samples made of isotropic graphite, tungsten and molybdenum in order to compare the three methods. All measurement methods are in good agreement in the overlapping wavelength range from 600 nm to 1100 nm with regards to their respective uncertainties.

Acknowledgment

This project (17IND11) Hi-Trace has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

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OS2-3- L2D – Tuesday, 12 Sep. 14.50-15.10 – ROOM N1

Relationship between configurational heat capacity and liquidus viscosity of glassforming melts

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For design of glass materials, information on glass-forming ability (i.e., resistance to crystallization) of inorganic melts with a desired composition is indispensable, as is design of optimal physical properties for the application. However, it is difficult to predict glass-forming ability of multi-component systems such as practical compositions, or even simple composition systems.

In this study, we focused on liquidus viscosity (η_L) as one of the indicators of glass-forming ability. η_L is the viscosity at the liquidus temperature (T_L), which is known to be correlated with glass-forming ability and negatively correlated with critical cooling rate of melt [1]. In addition, it is also an indicator of formability in glass production and is an industrially valuable feature. For example, Zheng et al. proposed a method to calculate η_L by simultaneously measuring the T_L and predicting the viscosity curve by DSC measurements [2]. However, that method requires multiple calorimetric measurements that require precision.

As mentioned in Adam-Gibbs theory [3], configurational entropy (S_{conf}) is dominant in temperature dependance of viscosity of glass. Considering that the change in S_{conf} of melt is related to the stability of glass, η_L of inorganic liquids (glasses) of various composition systems, as well as configurational heat capacity ($C_{p\ conf}$) and S_{conf} of the melt. η_L was evaluated from viscosity curves measured by the inner-tube rotation method (DSR502, Anton Paar) and T_L obtained by direct observation of heat-treated glass. $C_{p\ conf}$ and S_{conf} were calculated from the calorimetric changes of glass and supercooled liquid measured by DSC method (DSC404F3, NETZSCH). The obtained $C_{p\ conf}$ and S_{conf} showed a good correlation with the $\log\eta_L$ in various compositions with various η_L (Fig. 1), indicating that these properties related to structural relaxation are one of the factors determining liquidus viscosity.

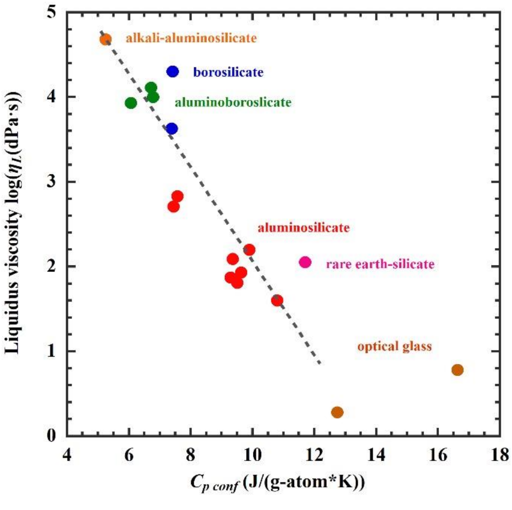

Fig. 1. Relationship between configurational heat capacity obtained from DSC curve on cooling and Liquidus viscosity $\log\eta_L$.

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Development of a clinical diagnostic device for non-contact in-situ measurement of tear viscosity

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Recently, dry eye syndrome has been causing eye diseases such as keratoconjunctival epithelial disorders, and the number of patients is increasing as the use of PCs and smartphones increases. Nowadays, the economic loss caused by dry eye syndrome has become a social issue due to the opportunity loss and economic loss. The Schirmer test is a conventional technique to diagnose dry eye syndrome; however, invasive techniques like Schirmer that cause pain and take a long time to measure reduce the patient's quality of life. Therefore, we propose a non-invasive in-situ diagnostic method measuring the viscosity of tear fluid as a biomarker for dry eye syndrome.

The principle of this method is to observe the thermal expansion and its decay behaviour due to latticeshaped laser heating of the object to be measured, as illustrated in Fig.1. The tear fluid membrane on the eye surface is sinusoidally excited by an interference of two pulsed-laser beams, and the lattice-shaped capillary wave is generated by the thermal expansion. Since the attenuation behaviour of the amplitude of the capillary wave varies with viscosity and surface tension, the thermophysical properties of the sample can be measured by the time variation of the amplitude. This non-contact optical viscometer is a measurement technique that has potential to be applied to point-of-care-testing (POCT).

In the case that the sample surface has a spherical shape, it is quite difficult to guide a signal light from a sample surface to a photodetector because of the diffuse reflection from the sample surface. To overcome these challenges, a precise control of optical path was introduced to enhance the sensitivity and reproducibility of the measurement of tear viscosity. In the presentation, the validity of proposed method for the clinical application is discussed.

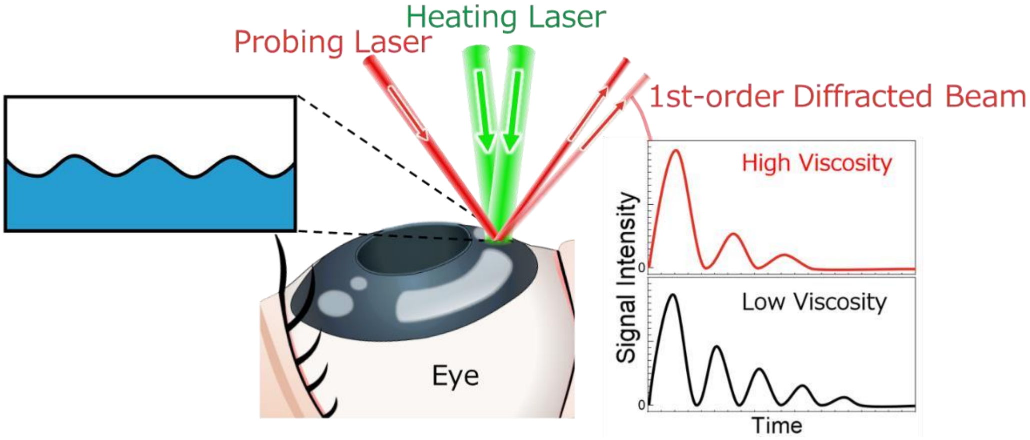

Fig.1 Principle of viscosity measurement

Acknowledgment

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OS2-3 L3A – Tuesday, 12 Sep. 15.10-15.30 – ROOM O2

Dynamic Viscosity and Interfacial Tension of N_2 and *n*-tetradecane System by Dynamic Light Scattering Method and Molecular Simulation

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CO_2 and N_2 enhanced oil recovery (EOR) can reduce costs and improve oil recovery. The viscosity and interfacial tension of CO_2/N_2 and crude oil system are the key parameters affecting the EOR process. The study of viscosity and interfacial tension of N_2 and alkanes (typical crude oil components) can provide key data for the EOR process.

In this work, the viscosity and interfacial tension of the $N_2 + n\text{-tetradecane}$（$n$-$C_{14}H_{30}$） system were obtained experimentally by dynamic light scattering method at the temperature range of 323.15~473.15 K and the pressure range of 1~5 MPa. A coarse-grained force field for n-$C_{14}H_{30}$ was established by the Martini model. The viscosity and interfacial tension of $N_2 + n$-$C_{14}H_{30}$ were calculated by molecular simulation, and the average relative deviations between the calculated and experimental data are 12.6% and 5.7%, respectively The $N_2 + n$-$C_{14}H_{30}$ system and $CO_2 + n$-$C_{14}H_{30}$ system were compared by molecular simulation, and the viscosity and interfacial tension of the two systems were analyzed from the perspective of molecular level[1,2].

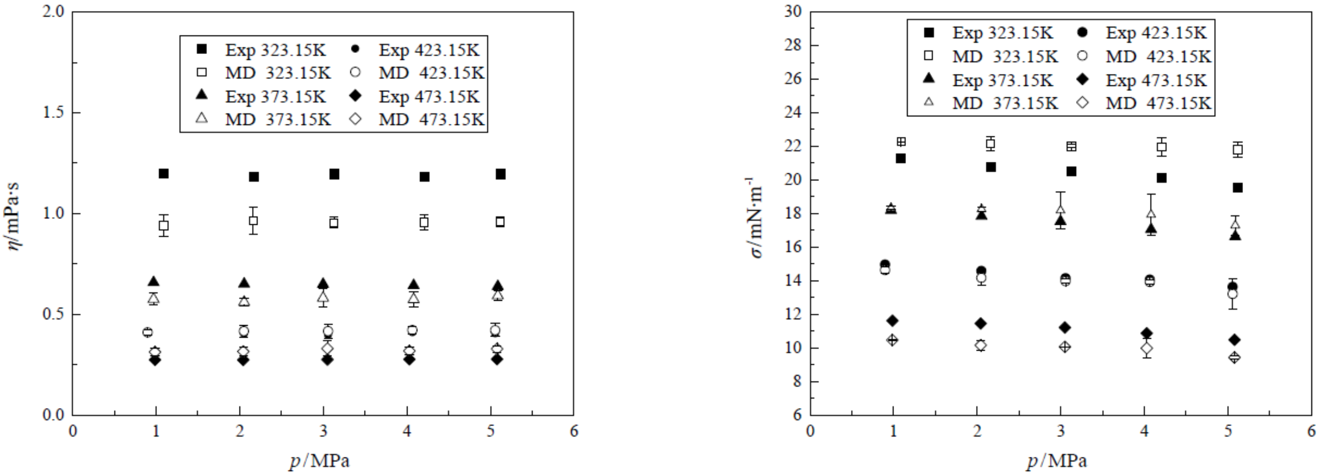

Fig. 1. Experimental and simulation results of viscosity and interfacial tension in N_2+n-$C_{14}H_{30}$ system

Acknowledgment

The authors appreciate the financial support from the open funded projects of State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation (No. PLN2021-06) and the Natural Science Basic Research Project of Shaanxi Province (No. 2023-JC-YB-389).

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OS2-3 L3B – Tuesday, 12 Sep. 15.10-15.30 – ROOM O1

A modified method for measuring the mutual diffusion coefficients in liquid binary sodium-ion battery electrolytes

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The diffusion coefficient is a basic thermophysical parameter, which characterizes the electrolyte transport process inside the battery and determines the power density and safety performance [1-2]. This paper presents a modified image processing method for measuring the mutual diffusion coefficient in binary transparent liquid by using digital holographic interferometry, as shown in Figure 1. In the image preprocess stage, a fast adaptive bidimensional multivariate empirical mode decomposition (FABMEMD) is used to eliminate the zero frequency component and noise in interferograms effectively. The spectrogram is extract by Fourier transform, and the phase is unwrapping by iterative least square method. By analyzing the phase curve at different diffusion time, the mutual diffusion coefficient of binary solution can be obtained by the discretizing equation of Fick’s second law based on the finite volume method (FVM). The measurement principle can be determined by:

$$D_j = \frac{h^2}{\tau} \frac{\left(\varphi_j^{i+1} - \varphi_j^i\right)}{\left(\varphi_{j+1}^i - 2\varphi_j^i + \varphi_{j-1}^i\right)}$$

where φ^i_j is the phase at node j and time layer i, h is the spatial step, τ is the time step, D_j refer the mutual diffusion coefficient at node j The expanded relative uncertainty of the mutual diffusion coefficient measurement is estimated to be 2.2% with the level of confidence of 0.95 (coverage factor $k = 2$). The modified method can eliminate the influence of uneven initial concentration distribution, and effectively improve the accuracy of experimental measurement.

As a substitute for lithium ion battery, sodium-ion battery has received more and more attention in the field of energy storage [3]. In this paper, the mutual diffusion coefficients of $NaPF_6$/NaTFSI in PC/DEC were measured from 0.1 ~ 2 mol/L by the modified digital holographic interferometry. We investigated the effects of temperature, concentration and different anions on the diffusion in electrolyte.

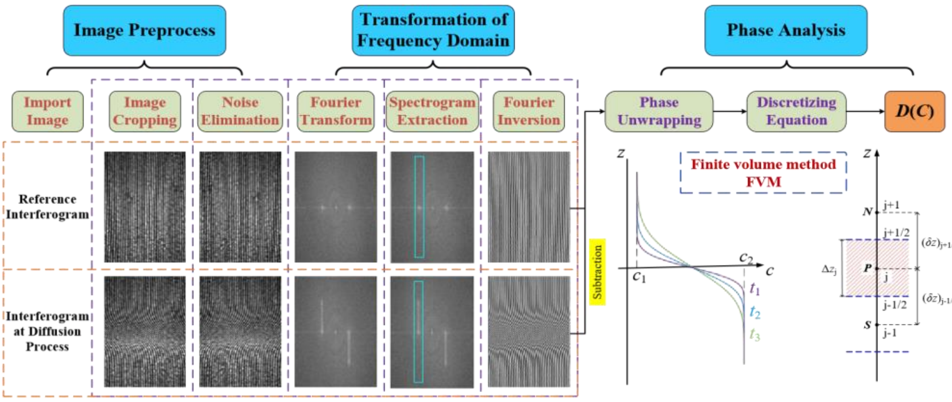

Fig. 1. Image processing of the digital holographic interferometry.

Acknowledgment

This work is supported by the National Natural Science Foundation of China (No. 51576165) and the Fundamental Research Funds for the Central Universities (No. xzy022022033).

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OS2-2 L3C – Tuesday, 12 Sep. 15.10-15.30 – ROOM N2

Emissivity measurements of NiTi shape memory alloys with different surface properties

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The superelasticity of NiTi alloys makes them unique for their application in cardiovascular stents [1]. Whereas, in the present work, the shape memory effect of thin NiTi wires is exploited to employ them in a hybrid actuator. For contactless analysis of the temperature distribution along such wires (diameter $\geqslant 75\ \mu$m), accurate emissivity data are needed, however, literature reveals an insufficient data situation for NiTi alloys. Here, we present an apparatus for measuring the emissivity of metals on a microscopic level, covering the temperature range from 308 K to 423 K. This apparatus consists of three principal components, i.e., a state-of-the-art infrared camera (type: ImageIR 8300, InfraTec GmbH, Germany), a reference quality black body cavity with an effective emissivity of 0.999948 [2], and contact thermometry calibrated on ITS-90. Within the scope of the present study, the emissivity of NiTi samples having rough, sightly oxidized, and heavily oxidized surfaces was measured by comparing them with the black body cavity. The expanded combined uncertainty of the experimental emissivities was estimated to be 0.015 at a confidence level of 95% ($k = 2$). Fig. 1 exhibits the experimental emissivities of two rough NiTi samples as a function of temperature. With increase in temperature, the emissivity increases rapidly from 0.1 to 0.28 in the martensite phase, while it decreases slowly in the austenite phase. This behavior is due to the temperature-dependent change in microstructure on the sample surface. Moreover, it will be presented that the emissivity of NiTi is low on a shiny rough surface, and it increases with the thickness of the oxidation layer.

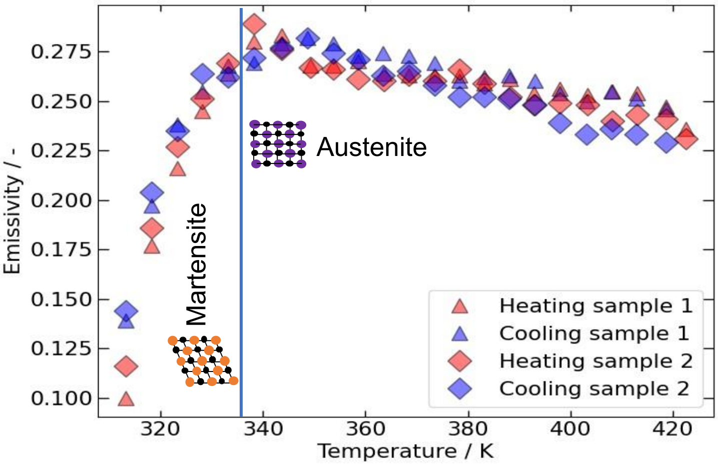

Fig. 1. Behavior of the emissivity of two rough NiTi samples with temperature.

Acknowledgment

The authors would like to thank the German Federal Ministry of Economic Affairs and Climate Action for supporting this project.

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OS2-3- L3D – Tuesday, 12 Sep. 15.10-15.30 – ROOM N1

NIST database for thermophysical property data of metal systems

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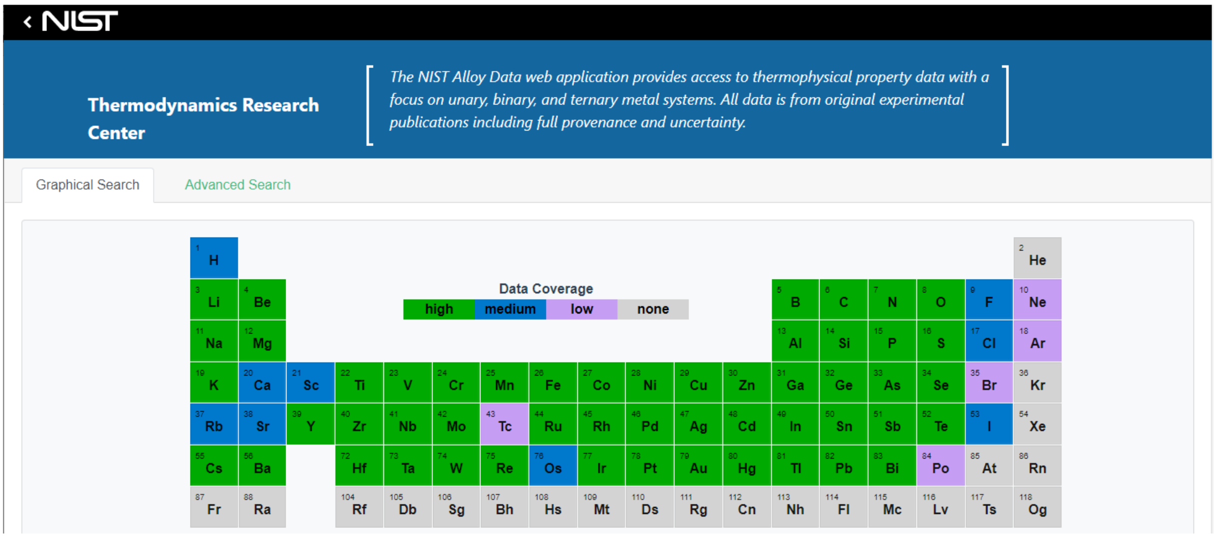
Research into new metallic materials is heavily based on a framework of integrated advanced modelling, computational and experimental tools, and quantitative data. This approach allows researchers to harness the power of experimental materials data, rapidly model properties needed for additive manufacturing processes, use them in support of the CALPHAD approach to computational material development or to address the challenges and the complex multiphysics in the development of theoretical and computational solutions they need to predict material properties in a wide spectrum of time and length scales. Very often parameters must be scaled to, or models relay on verification with original experimental thermophysical property data. This requires a comprehensive data infrastructure with improved data reliability and interoperability compared to what was available for historic data from various print formats.

The presented NIST database from the Thermodynamics Research Center (TRC) provides such an online repository for thermophysical property data (e.g. Enthalpy, viscosity, electrical resistivity, …) of mostly unary, binary, and ternary systems. It is publicly accessible at http://trc.nist.gov/metals_data (DOI: 10.18434/M32153) and free of charge for non-commercial users.

This talk provides an update on the coverage of captured data that is now available in a well-structured machine-readable format with metadata and full provenance. It highlights how to access the data via our web UI and, for larger scale applications, programmatically via our RESTful API and discusses the available example code in our Github repository.



Thermodynamics Research Center

The NIST Alloy Data web application provides access to thermophysical property data with a focus on unary, binary, and ternary metal systems. All data is from original experimental publications including full provenance and uncertainty.

Figure 1: Screenshot of the NIST Alloy Data web interface [1].

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OS2-3 L3E – Tuesday, 12 Sep. 15.10-15.30 – ROOM M2

Transport Properties Measurement of CF_3I

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This study emphasizes the transport properties of CF_3I, particularly its viscosity and thermal conductivity. CF_3I, also known as trifluoro iodomethane, has favorable properties, such as non-flammable, low toxicity, low ozone depletion potential, and low global warming potential. In addition, CF_3I's good thermodynamic properties, such as its low boiling point and high critical temperature, make it appropriate for use in refrigeration systems. Therefore, CF_3I has regained the interest of different scholars and is regarded as a promising component in mixtures with other refrigerants. As CF_3I is an integral part of mixture refrigerants, it is necessary to comprehend its thermophysical properties. Here, the tandem capillary tube method is used to evaluate the viscosity of CF_3I; this involves placing two capillary tubes side by side in a series connection to cancel out the end effects of pipes. Nevertheless, CF_3I's thermal conductivity is measured experimentally using the transient hot-wire method, in which two platinum wires are linked in parallel to nullify any impacts from axial heat conduction. The experimentally measured viscosity and thermal conductivity are reported in liquid and vapor phases. The results show that viscosity and thermal conductivity have an average absolute deviation (AAD) of about 5% with the preliminary calculated Extended Corresponding States model (ECS).

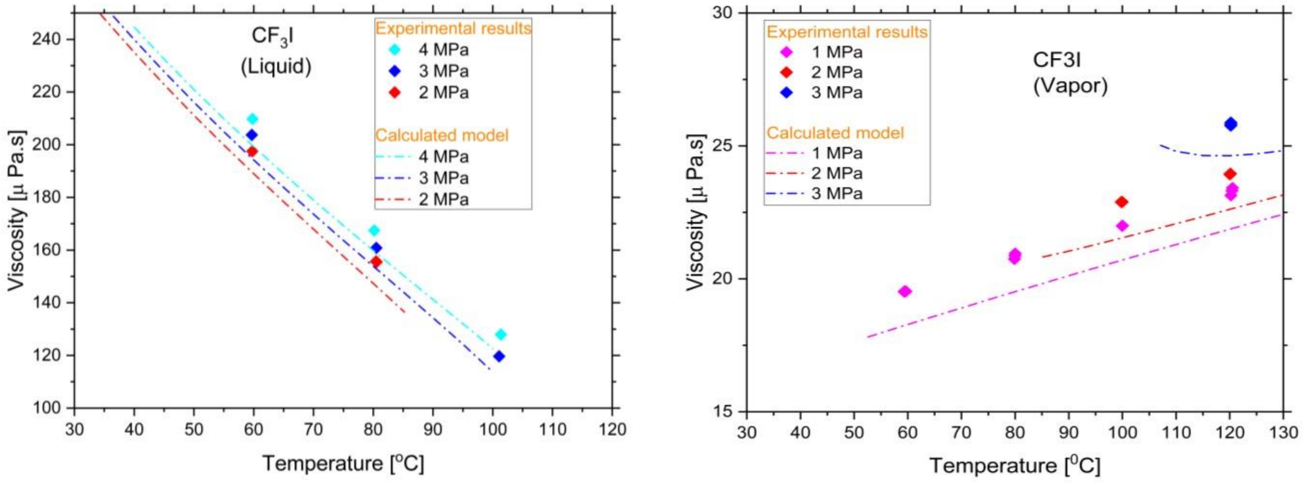

Fig. 1. Experimental viscosity with respect to temperature

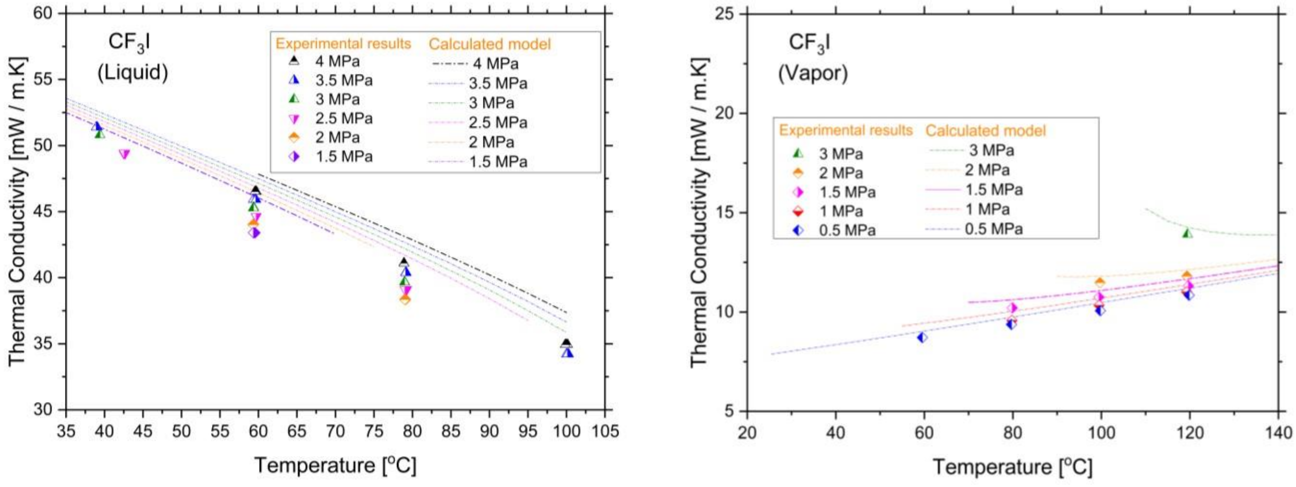

Fig. 2. Experimental thermal conductivity with respect to temperature

OS2-3 L4A – Tuesday, 12 Sep. 15.30-15.50 – ROOM O2

An equation and an artificial neural network for the surface tension of organic sulfides

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Surface tension is an important property of fluids for different industrial applications. It is fundamental in the design of equipment for many chemical processes, such as distillation and extraction, among others. As concern organic sulfides, the knowledge of their surface tension can help to remove these compounds from fossil fuels, which is one of the main objectives of the oil refining process [1].

Although various models were proposed to describe the surface tension of organic compounds, such as empirical and semi-empirical correlations [2], none was developed explicitly for organic sulfides. For this reason, a scaled semi-empirical correlation recently developed by our group of work for other families of fluids [3,4] was refitted and tested for their surface tension. To develop the model on a reliable dataset, the experimental surface tension data of the studied fluids were collated from the DIPPR database and the Dortmund Data Bank and selected based on statistical analysis.

Moreover, a multi-layer perceptron neural network is also proposed to calculate the surface tension of the studied organic sulfides. It was trained, validated, and tested on the selected experimental data.

The accuracy of the presented models was proved by comparing their results with that of various well-known correlations available in the literature.

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OS2-3 L4B – Tuesday, 12 Sep. 15.30-15.50 – ROOM O1

Determination of effective thermal conductivity of Li-Ion cells – an uncertainty analysis

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The effective thermal conductivity of Li-ion cells is a critical parameter that affects the thermal behavior and performance of a Li-ion battery. Accurate measurement and analysis of the effective thermal conductivity can help to improve the design and optimization of Li-ion cells, and ensure their safe and reliable operation. However, there are various challenges associated with the determination of the thermophysical properties of Liion cells, including thermal diffusivity. One of the most widely used techniques for measuring thermal diffusivity is laser-flash analysis. Several challenges and sources of uncertainty must be taken into account to obtain accurate results.

In this study commercial Li-Ion cells were investigated. The cells were opened and their components were characterized with the laser-flash method and differential-scanning calorimetry (DSC), in order to obtain the thermal conductivity of anode, cathode, separator and the cell housing. The thermal diffusivity of the layers was measured through-plane and in-plane, in order to characterize the anisotropy of the layer setup inside the battery cell.

The effective thermal conductivity of the entire cell was calculated from the measured values of the individual layers via the series and parallel connection of thermal resistors. A detailed uncertainty analysis was carried out for the calculation of the effective thermal conductivity of the entire cell. The results are compared with measurements using guarded-hot-plate apparatus on closed cells. For these measurements a new measurement strategy was applied to quantify the thermal contact resistance between the cell and the apparatus.

In conclusion, while laser-flash analysis is a useful technique for the determination of thermal diffusivity, several challenges and sources of uncertainty must be carefully considered and addressed to obtain accurate results. The thermophysical characterization of Li-ion cells is a critical aspect of battery technology, necessary for improving the performance and safety of Li-ion batteries. Accurate measurement and analysis of the thermophysical properties of the cell can provide valuable insights into its thermal behavior, allowing for the design and optimization of more efficient and reliable batteries.

Acknowledgment

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OS2-3 L4C – Tuesday, 12 Sep. 15.30-15.50 – ROOM N2

Upgraded emissometer at the University of the Basque Country

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The HAIRL emissometer (High Accuracy Infrared Radiometer, Leioa) is a mid-infrared emissometer that was designed and built at the University of the Basque Country (UPV/EHU) [1]. The instrument can measure spectral directional emissivity in the range between 0.83 and 25 µm, and it is equipped with an electric heater that can increase the surface temperature of the samples up to 1273 K. During 20 years of service, the instrument has undergone instrumental and methodological updates, such as the development of specific uncertainty budgets and the implementation of a Monte Carlo method to propagate uncertainties when integrating spectral and directional data [2].

However, the emissometer required an update to replace some of its core components, such as the FTIR spectrophotometer, which led to an integral upgrade that went beyond the mere substitution of the instruments. We present the total renovation performed to the HAIRL, which consists of three blocks: hardware, software and methodology. The hardware renewal is based on the use of control-design paradigms that allow the construction of scalable and maintainable instruments. Based on this philosophy, we have upgraded or replaced most of the devices that constitute the instrument. Among other modifications, we replaced the previous FTIR by a Bruker Vertex 80v, improved the sample heating system, redesigned the temperature measurement system and installed a new low-temperature blackbody. Regarding the software, we have not only rebuilt all the communication protocols between the different devices and the control-computer using an expandable software-design, but we have also implemented a monitoring system based on new technologies such as InfluxDB and Grafana. Finally, all these technical improvements permitted us to adopt a new uncertainty budget, which treats statistically magnitudes such as the spectra measured by the FTIR or surface temperatures, and allows discarding anomalous data.

The new instrument presents several improvements compared to its previous version. On the one hand, the system can be completely controlled and monitored remotely, and it will be able to measure autonomously. On the other hand, the abstraction achieved after the renovation will allow exploring further improvements in order to measure new types of materials, such as semitransparent ceramics and coatings.

Acknowledgment

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OS2-3- L4D – Tuesday, 12 Sep. 15.30-15.50 – ROOM N1

Optical Differential Scanning Calorimetry for Modern Material Science at High Temperatures

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Modern material science is of high importance for further development in many fields. The increase of the process temperature of thermal energy conversion processes and therefore the development of highly temperature stable materials is one of the most important issues on the way to higher efficiencies. In some cases under operating conditions, the temperature of the thermal barrier coating of modern gas turbine blades is far above the melting point of the protected body material. In additive manufacturing techniques as laser sintering material is locally molten by a laser beam. Hereby, an increase of precision and the enhancement of process control are aims of further development. In these and also in many other fields, the reliable prediction of the behavior of different materials at harsh operation conditions and therefore the exact knowledge of material parameters at high temperatures above 1000 °C and up to 2000 °C is needed. In this regard, the thermal diffusivity and the specific heat capacity are of great relevance. For the determination of the thermal diffusivity even at high temperatures above 2000 °C, the laser flash method can be used as a verified measurement method. The specific heat capacity at temperatures below 1000 °C can already be measured by differential scanning calorimetry (DSC). Today, in most DSC Setups thermocouples are utilized for the necessary temperature measurement. However, up to now, thermocouples are not suitable for higher temperatures. For this reason, a reliable measurement method to provide verified information about the specific heat-capacity of material samples at temperatures above 1000 °C is still needed.

To resolve this lack of information in future, within the project Optical Differential Scanning Calorimetry For Modern Material Science At High Temperatures (OptiMa) it is intended to develop a DSC method for measurements at temperatures between 1000 °C and 2000 °C. In this new built DSC setup (Fig. 1), several optical detectors like radiation thermometers and thermographic cameras will be tested and analysed, to gather the required temperature information without fault-prone thermocouples. Simultaneously, different methods of sample heating will be tested e.g. inductive heating and laser-heating. Furthermore, it is planed utilize the triple-cell sample holder design, which reduces the number of necessary measurement cycles [1][2].

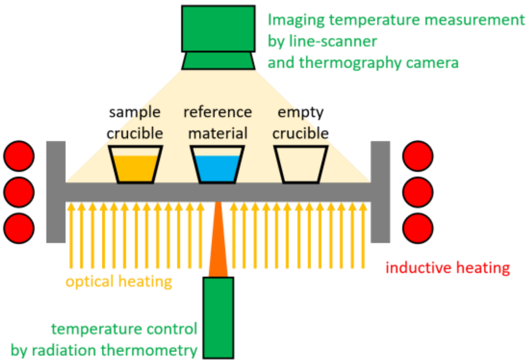

Fig. 1. Principle drawing of the new DSC setup with optical temperature measurement for determination of the specific heat capacity at temperatures between 1000 °C and 2000 °C.

Acknowledgment

This Project (contract number: 13FH070KX0) has been sponsored as a part of the funding program Forschung an Fachhochschulen by the German Federal Ministry of Education and Research (BMBF).

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OS2-3 L4E – Tuesday, 12 Sep. 15.30-15.50 – ROOM M2

Measurements and modelling of density and viscosity of methyl dodecanoate and ethyl tetradecanoate

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To reduce the net carbon emissions in maritime and long-distance road transport, biological fuels are feasible options since battery technology is not yet mature enough to ensure, e.g., intercontinental shipping. If these fuels are used, e.g., on ships equipped with the CCS on ships technology, even negative carbon emissions are possible. Biodiesel, a mixture of numerous fatty acid esters (FAE) derived from the transesterification of fatty acids, is already worldwide used with a fraction of up to 10% in blends with fossil diesel in the transportation sector. Pure biodiesel can be used in most common diesel engines, bearing the potential of drastically minimizing carbon emissions. To allow for efficient process design, thermophysical properties of biodiesel such as density, viscosity or heat capacity have to be known at various temperature and pressure conditions since they influence, e.g., flow and pumping behaviour and spray quality. As the composition of biodiesel depends on the composition of the fatty acid feedstock and the alcohol used for transesterification, the thermophysical properties of biodiesel vary. To predict the properties of possible biodiesel compositions, several mixture models exist in the literature, mostly relying on the properties of pure FAE. However, the availability of experimental data for most of the pure FAEs is limited in temperature and often lacking at elevated pressures [1].

Therefore, the density and viscosity behaviour of methyl dodecanoate and ethyl tetradecanoate, typical compounds of biodiesel, were determined in this work, enhancing data availability for both substances. Using a coupled vibrating-wire vibrating-tube viscometer-densimeter, measurements were conducted at pressures from (0.1 to 100) MPa and temperatures from (298 to 423) K. Relative expanded uncertainties of the results were estimated to be within 0.1% for density and 1.2% for viscosity. The new data set was further used to elaborate correlations to predict density and viscosity at arbitrary conditions. For methyl dodecanoate, deviations of the experimental data from the developed Tait equation for density and Tait-Andrade equation for viscosity are exemplarily shown in Figure *1*. With deviations lower than 0.1% for density and lower than 0.8% for viscosity, the correlations represent the new data within their experimental uncertainty. In addition, the correlations agree as well with experimental data available in the literature within their stated measurement uncertainty.

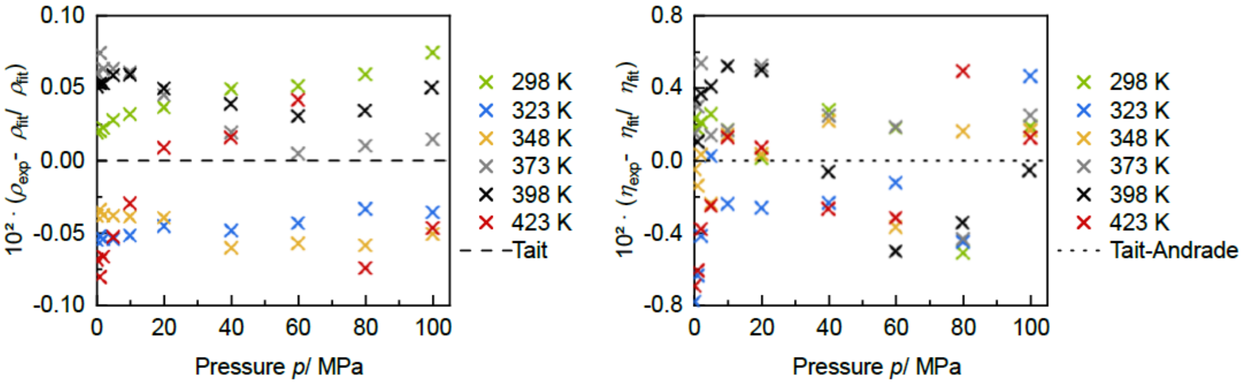

Figure 1. Deviations of the experimental data for methyl dodecanoate for density (left) and viscosity (right) from the developed Tait equation and Tait-Andrade equation, respectively.

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OS2-3 L5A – Tuesday, 12 Sep. 15.50-16.10 – ROOM O2

Pulsed laser viscometer: A contactless and high-speed technique to measure viscosity and surface tension with small sample volume (Re-examination of working equation)

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We have developed a pulsed laser viscometer (PLV) which is a high-speed viscosity sensing technique using pulsed volume heating laser of near-infrared wavelength. In this technique, two pulsed laser beams intersect on a sample liquid surface which generate an interference grating pattern with a period of about 80 -100 μm. The temperature distribution of the thermal grating creates corresponding nanometer order displacement on the surface mainly driven by the thermal expansion of the sample. After the nano-second order pulse heating, the generated displacement starts damping oscillation (capillary wave). The temporal behavior of the capillary wave can be detected by observing the intensity of the first-order diffracted beam irradiated on this heating area because the capillary waves act like a reflection grating. Since the temporal behavior of the capillary wave principally depends on the viscosity and the surface tension, it is possible to determine viscosity and surface tension within μs to ms which in an extremely short period of time for a viscosity measurement. The pulsed laser viscometer measurement has the following characteristics: (1) non-contact and *in situ* measurement, (2) spatial resolution of 10 ~ 100 μm, (3) high speed measurement of within μs ~ ms, (4) small amount of sample of μL ~ mL, and (5) wide viscosity range of 10^{-1} ~ 10^4 mPa·s [1-2].
Even though we have successfully measured a wide variety of Newtonian liquids (Acetone, Toluene, Water, Ethanol, 2-Propanol, 1-Hexanol, Ethylene Glycol and JS100) and whole human blood, the working equation employed was based on the surface heating laser-induced capillary wave (LiCW) technique, which is not perfectly capable of expressing the entire wave form of volume heating PLV. In the present paper, we are attempting to provide appropriate working equations to theoretically calculate the behavior of PLV capillary waves for the determination of the viscosity of liquids with an aimed accuracy of at least a few percent.

Acknowledgment

The work described in this paper has been supported by JSPS KAKENHI Grant Number 23K03698.

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OS2-3 L5B – Tuesday, 12 Sep. 15.50-16.10 – ROOM O1

Development process of interfacial chemical reaction in Ni-rich layered cathodes for all-solid-state batteries

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All-solid-state batteries (ASSBs) have attracted much attention in the fields of energy storage, electric vehicles and portable electronic devices due to their higher safety and energy density. Ni-rich layered ternary materials ($LiNi_{1-y-z}Co_yMn_zO_2$, $1\text{-}y\text{-}z \geq 0.7$) are considered to be among the most promising candidates for cathode materials in ASSBs due to their unique advantages [1-2]. Nevertheless, the interfacial reaction between ternary cathode (NCM) and solid-state electrolytes (SSEs) has become the main issue to limit the long-cycle stability of the cathode. Related studies have shown that when NCM materials are in direct contact with sulfide-based SSEs, by-products generated by the interfacial chemical reaction accumulate at the interface, resulting in increasing interfacial impedance [1-3].

However, up till now, the formation mechanism of the NCM/SSEs interfacial chemical reaction, as well as its property characteristics and evolution process still lack detailed characterization. In this paper, SEM, EDS, STEM and other characterization means are used to reveal the dynamic development process of the chemical reaction from the cathode-electrolyte interface to the interior of the particle, and determine the effect of the chemical reaction on the irreversible decay of the battery capacity.

On this basis, a surface coating of lithium niobate is adopted to establish a passivation protection layer at the cathode electrolyte interface. The coated battery has been subjected to 2000 charge/discharge cycles at a rate of 1 C and achieved a capacity retention rate up to 82%.

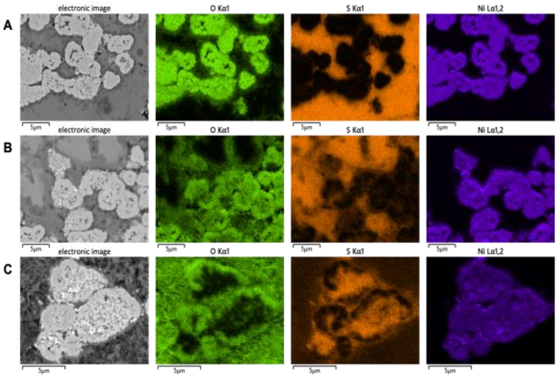

Fig. 1. Features of cathode cross sections. Chemical components of the cathode cross sections measured after cycling, showing the changes in the characteristic elements at the cathode-electrolyte interface during cycling. The SEM-EDX mappings of: (A) 200-cycled; (B) 500-cycled; and (C) 1000-cycled. The left to right panels (except the first panel) in each part are the SEM images corresponding to O, S, Ni maps, respectively.

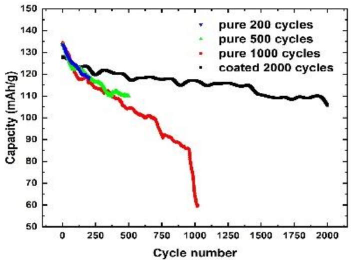

Fig. 2. Plot of discharge capacity versus cycle number for pure NCM and coated LNO@NCM in cells revealing that the $LiNbO_3$ coating improves capacity retention. Charging and discharging were performed for 200, 500, 1000, and 2000 cycles at a rate of 1 C.

Acknowledgment

This work was supported by the National Natural Science Foundation of China (Grant Nos. 52130602 and 51827807), the Science and Technology Major Project of China National Machinery Industry Corporation (SINOMAST-ZDZX-2019-04), and the Special Funds for Innovation Driven Development of Guangxi Zhuang Autonomous Region (AA17204061).

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Development of a test bench for measuring emissivity at low temperature for the space sector

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The scientific and technical fields requiring knowledge of the emissivity of surfaces are extremely numerous. Emissivity is essential to calculate and optimize heat exchange by radiation. The emissivity of a material strongly depends on the nature of the material; it can also depend on the wavelength, the morphology of the surface (e.g. roughness) and the temperature.

In the space industry, it is necessary to control heat exchange to maintain the thermal housekeeping within acceptable ranges. Heat exchanges between the spacecraft and the surrounding environment are only radiative exchanges. For this, knowledge of the emissivity with good precision is essential. Typical applications are radiators, Multi-Layer Insulation materials, and other thermal-control surfaces, which frequently operate at cryogenic temperatures.

There are few devices capable of measuring emissivity at low temperature ($T<77K$) with sufficient precision. THEMACS Engineering on behalf of the European Space Agency (ESA) has developed a measuring bench to measure the emissivity on any substrate. At these temperatures, reflectometric methods for high emissivity are difficult to use because the useful spectral range is between 10 and 200µm and the reflected flux is low. Consequently, a calorimetric approach is usually more feasible and provide lower uncertainties. The measurement bench is inspired by existing devices [1-3] with the novelty of not needing to produce a specific sample geometry. For this we made a fluxmeter made with an emissive surface at a temperature slightly higher than the sample to be tested. To maintain it at a constant temperature, it is supplied with an electrical power that is continuously measured. The advantage of this approach is that it does not have to regulate the temperature of the sample. Another advantage is to be able to cool down quickly. The fluxmeter is painted with an emissive paint which must be characterized at all temperatures. The first results allowed us to adapt the design in order to significantly lower the level of uncertainty for measurements on different types of materials.

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OS2-3- L5D – Tuesday, 12 Sep. 15.50-16.10 – ROOM N1

Specific heat capacities of Al-Si based alloys in a liquid state

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Further effective utilization of energy is becoming a necessity to achieve carbon neutrality. Thermal energy storage materials are attracting attention from this perspective, and phase change materials (PCMs), which store heat as latent heat during melting and solidification of materials, focus much attention. Among these, metallic PCMs are promising candidates for thermal energy storage materials because of their large storage capacity per unit volume and high thermal conductivity, which enables rapid heat transfer. When thermal energy storage materials are combined with thermal power plants, it is necessary to develop metallic PCMs in various temperature ranges. In this study, two Al-Si based alloys are selected as metallic PCM materials in the 550650°C (823-923 K) range. One is an ADC12 alloy and the other is an Al-5.9mass%Si-1.6mass%Fe alloy with a liquidus temperature of about 630°C (900 K). ADC12 is a Japanese Industrial Standards (JIS) alloy for die casting and is an Al-Si-Cu alloy with Si content of 9.6-12 mass% and Cu content of 1.5-3.5 mass%. This study presents the results of a comparative study of the specific heat capacity of these alloys in the molten state measured by differential scanning calorimetry (DSC), adiabatic calorimetry, and laser modulation calorimetry methods [1-2] as shown in Fig. 1. The specific heat capacities of these alloys in the molten state had a negative temperature dependence, which will be discussed at the conference.

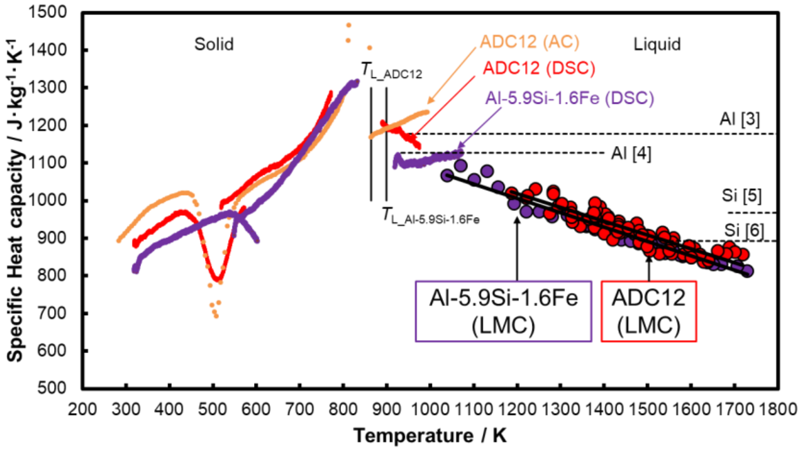

Fig. 1. Specific heat capacities of ADC12 and Al-5.9mass%Si-1.6mass%Fe alloy. LMC: laser modulation calorimetry, DSC: differential scanning calorimetry, and AC: adiabatic calorimetry.

Acknowledgment

This study was supported by a project, JPNP16002, commissioned by the New Energy and Industrial Technology Development Organization (NEDO).

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OS2-3 L5E – Tuesday, 12 Sep. 15.50-16.10 – ROOM M2

Viscosity measurements of (hydrogen + methane), (hydrogen + ethane), and (hydrogen + carbon dioxide) mixtures

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Hydrogen is widely discussed as an alternative, more sustainable resource, since the world seeks for measures to reduce the impact of climate change. Hydrogen, produced from renewable energy sources by electrolysis of water, or natural gas reforming in combination with carbon capture and storage (CCS), provides a climateneutral energy source and chemical feedstock. The existing natural gas grid can be used for the transport and distribution of hydrogen, to gradually transition towards a carbon-neutral economy. Furthermore, underground storage of hydrogen in depleted oil and gas reservoirs offers large scale storage capacities, which can be used to balance the energy supply and demand mismatch of volatile renewable energy sources. In this context, the transport of hydrogen mixtures, with methane, ethane and carbon dioxide, plays an important role. One of the key thermophysical properties in transport processes, is the dynamic viscosity, which can be calculated from viscosity correlations or viscosity prediction models for fluid mixtures. However, currently existing models are not validated for hydrogen-rich mixtures, due to a limited experimental data base. Therefore, the viscosity of (hydrogen + methane), (hydrogen + ethane), and (hydrogen + carbon dioxide) binary mixtures was measured, to enhance the existing data base. The measurements were carried out, utilizing a rotating body viscometer in the temperature range of (253.15 to 473.15) K and pressures up to 20 MPa. The relative expanded combined uncertainty was estimated to be (0.35 to 0.70) % (k = 2). The measurement principle is based on the deceleration of a cylindrical rotating body, due to the viscous drag of the surrounding fluid. The rotating body is vertically suspended inside the pressure tight measuring cell by a magnetic suspension coupling. The experimental data were compared to the Extended Corresponding States model (ECS) [1], as implemented in the thermophysical reference data base REFPROP (v10.0) [2]. Relative deviations between the experimental and calculated data exceed 8 %, 4%, and 4 % for the systems (hydrogen + methane), (hydrogen + ethane), and (hydrogen + carbon dioxide), respectively. The measurements carried out in this work extent the existing data base over a broad range of temperature, pressure and composition and, thus, can be used for the improvement of viscosity models for hydrogen mixtures.

Acknowledgment

The authors acknowledge funding from the Fraunhofer High-Performance Center DYNAFLEX and the German State of North Rhine-Westphalia.

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The Temperature of Maximum Density for Aqueous Solutions

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The temperature of maximum density (TMD) at 4 C is the most widely known thermodynamic anomaly of liquid water. The origin of this intriguing behavior has motivated many experimental and theoretical studies since long ago. Nowadays, it is an accepted fact that the TMD comes from a competition between two forms of water: one characterized by a highly ordered hydrogen-bonded structure, less dense and prevalent at low temperature and the other form, less structured, denser, and prevalent at high temperature [1]. The addition of solutes to the water, since distorts this hydrogen bond network, changes the TMD location, a fact noted as far back as XIX century [2]. The amount of the change in the TMD depends on the solute concentration and also on its chemical nature. As a rule, TMD goes to lower temperatures for most compounds and it decreases as solute concentrations increases. However, some chemicals, mainly short chain alcohols, slightly increase the TMD. While these general features regarding the TMD are a well-known fact since many years ago, a significant amount of new experimental works have been carried out in last years [3-7], a fact that encourage a global picture to be drawn. Therefore, this work reviews the results of the TMD for aqueous solutions comprising a wide variety of solvents, inorganic and organic, and also the effect of pressure over this magnitude. The main tendencies are sketched out, and the results are rationalizing taking into account the chemical nature of the solutes.

Acknowledgment

The author acknowledges financial support from the Spanish Ministry of Science and Innovation under Grant No. PID2020-115722GB-C22.

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Speed of sound measurements in hydrogen up to 100 MPa and an equation of state for normal hydrogen

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The availability of thermophysical properties of fluids is crucial for any process simulation and design. Especially concerning a shift towards a hydrogen-based economy, the thermophysical properties of hydrogen are decisive for a successful energy transition. As the trade of hydrogen will take on a similar significance to that of natural gas, establishing flow metering standards for hydrogen with, e.g., critical flow venturi nozzles (CFVN) is essential, which is the task of the MetHyInfra project funded by the European metrology programme for innovation and research (EMPIR). To determine the flow in CFVN, the speed of sound in the fluid is a required quantity. However, the equation of state (EOS) for normal hydrogen developed by Leachman et al. [1], predicts the speed of sound with a relative uncertainty of about 0.5%. This large uncertainty results from the fact that for the development of this EOS, no speeds of sound in gaseous hydrogen were available at temperatures above 34 K and 0.1 MPa. Therefore, new speed of sound data were measured within this work at temperatures from (273 to 323) K and pressures from (1 to 100) MPa. A cylindrical resonator was used for pressures up to 10 MPa and a dual-path pulse-echo system from 20 MPa upwards. The relative expanded uncertainty ($k = 2$) of the data was estimated from (0.03 to 0.05)%. As shown in Figure 1, the EOS of Leachman et al. [1] shows larger deviations from the experimental data than attributed to the measurement uncertainty, increasing significantly with increasing pressure. Therefore, a new EOS was developed based on the new speed of sound data and other thermodynamic property data from the literature. This equation was developed for the implementation in measurement sensors as well as CFD simulations of CFVN and is kept as short and simple as possible with polynomial terms and integer exponents only. Nonetheless, the new EOS calculates the experimental speeds of sound within deviations of 0.09% (see Figure 1), thus, the deviation is considerably lower. By using data for other thermodynamic properties from the literature, the EOS is valid at temperatures from (140 to 370) K and pressures up to 100 MPa, whereby 140 K is the lower limit of temperatures occurring in CFNV.

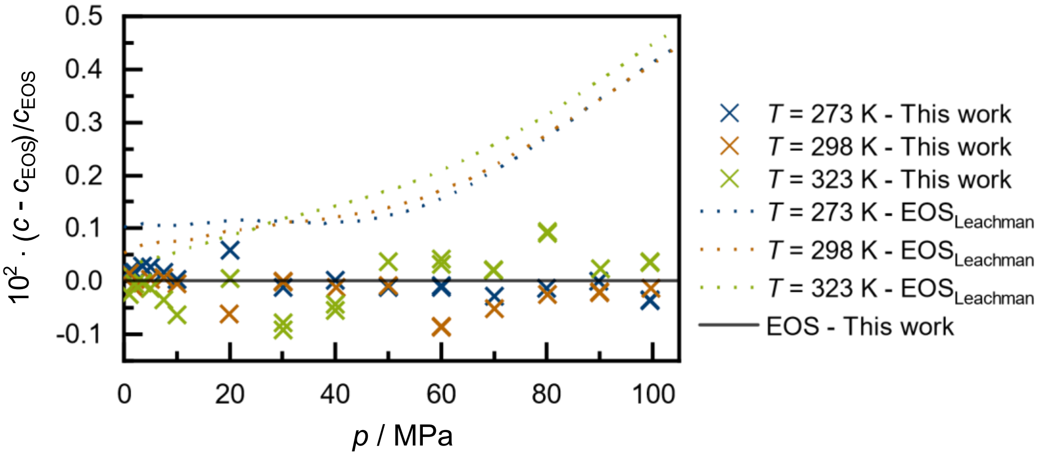

Figure 1. Relative deviations of the experimentally determined speed of sound in hydrogen at three different temperatures and the EOS by Leachman from the newly developed EOS.

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OS3-1 L1D – Wednesday, 13 Sep. 09.50-10.10 – ROOM N1

Effect of Fe_nO and CaO/SiO_2 on the viscosity of EAF-type slags

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The viscosity of slag is the one of the important physical properties to optimize the metallurgical processes at high temperature. Especially, it is well known that the viscosity among the physical properties most affects the slag foaming phenomena in Electric Arc Furnace(EAF) processes. With the advent of the carbon neutrality, the EAF processes are expanding and developing for low emission of carbon dioxide. The EAF slag compositions have also been changing and it is going to affect the slag foaming phenomena. Therefore, it is important to investigate physical properties of various slag systems. In present work, the viscosities of $CaO\text{-}SiO_2\text{-}Fe_nO$ slags system, simplified actual EAF slag, were investigated.

The compositions of present work were selected with a relatively high basicity compared to previous studies[1,2)]. Viscosity measurement was determined with a rotating crucible viscometer, and the atmosphere was maintained at an oxygen partial pressure of $1\text{-}3\times10^{-13}$atm using Ultra High Purity(UHP) argon. The experimental temperature was from 1873K to 1623K. This study was conducted every 50K intervals or continuous with 5K/min on cooling. The continuous method was introduced to measure as the slags were crystallized during experiment. In the experiment, Fe_2O_3 was maintained in UHP Ar atmosphere for 1.5 hours at 1873K to perform when ratio of Fe^{2+}/Fe^{3+} was equilibrium.

Fig. 1. showed temperature dependence of the viscosities at constant basicity. These results satisfied the Arrhenius equation over entire temperature region. Moreover, the viscosity values appeared in two types according to crystallization behavior. The viscosities of non-crystallization slag were lower than previous work1,2) with similar conditions because the oxygen partial pressure was relatively low, resulting in the increase of ferrous ions. To understand the effect of iron oxide, it was important that the ratio of Fe^{2+}/Fe^{3+} was determined through chemical analysis. In order to confirm the effect of each component, the experiment was conducted after keeping one component constant, respectively. It was found that the viscosities are decreased with increasing total Fe and slag basicity. By contrast, the viscosities in crystallization slag were influenced by solid fraction rather than the effect of each component.

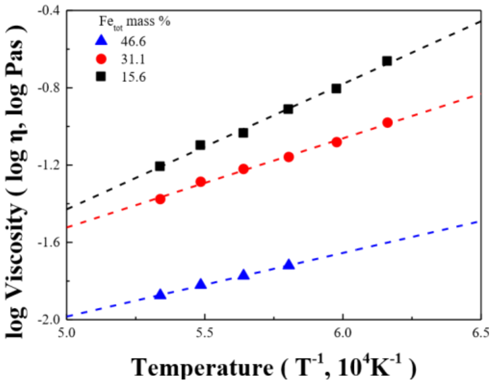

Fig. 1. Temperature dependences of the viscosities at constant basicity (CaO/SiO_2=1)

Acknowledgment

This study has been financially supported by the New Energy and Industrial Technology Development Organization (NEDO) under Project No. JPNP21014.

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Evaluation of multilayer systems using advanced laser flash analysis

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The Laser-Flash method (LFA) is a well-established method for measuring the thermal diffusivity of solid specimens with low to medium thermal resistance [1]. By heating a flat specimen of known thickness with a short laser pulse, a heat wave propagates through the material to the backside. The recorded temperature rise over time at the backside of the sample is fitted using analytical models with the thermal diffusivity as a fit parameter. The laser flash method has also been successfully applied to layered systems, by application of an appropriate analytical model. However, in case of thin layers with low thermal resistance, the conventional laser-flash method comes to its limits. In order to expand the scope of the method, front side detection of the surface temperature as function of time using a radiation thermometer is added to the experimental setup [2-3]. In this work, the recorded data are evaluated using a new adapted analytical model for the front side temperature in addition to a conventional evaluation of the backside temperature increase [4]. A pulse correction method is added to both, the front and back side fitting process [5-6]. In order to test the developed two-sided evaluation model with integrated pulse length correction, measurements were carried out on a homogeneous reference sample with known thermal diffusivity. To investigate the applicability to two-layer systems, synthetic measurement data were generated with a 2dimensional numerical model [7]. The fit results are compared with the input data of the simulation.

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OS3-1 L2A – Wednesday, 13 Sep. 10.10-10.30 – ROOM O2

Study of adhesion of cationic surfactant-added ice on various test plates under voltage application in a nanoscale field

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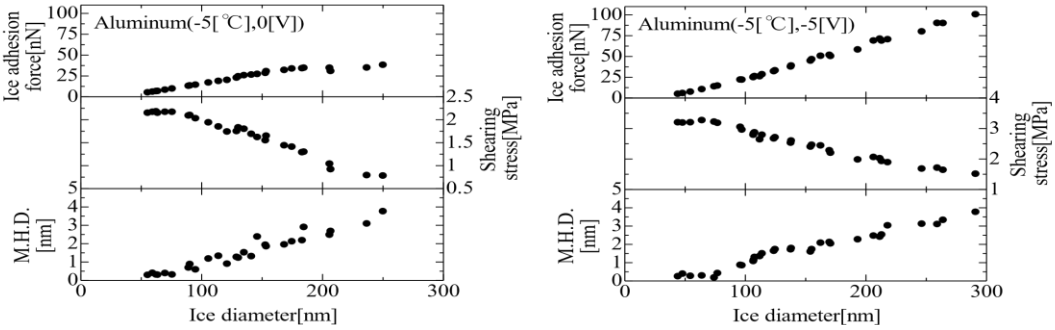
Ice and snow on solid surfaces can cause various problems such as energy loss and performance degradation. We focused on the possibility of efficient deicing by reducing the adhesion of ice. Conventional research has mainly focused on the microscale. However, due to the roughness of the solid surface, the adhesion force measured includes the force that breaks the ice, making it impossible to accurately measure the ice adhesion force. The scanning probe microscope[1] (SPM) developed in previous research has been used to accurately measure adhesion forces on the nanoscale. Since previous studies have shown that nonionic surfactants can inhibit ice adhesion, this study will examine how the application of voltage changes the adhesion inhibition by using cationic surfactants. A test plate to be used is placed inside the SPM, and ice to which a cationic surfactant is added is generated and peeled off. The adhesion force will be determined based on the data obtained during this process. The experimental conditions for SPM are shown in Table 1 and the experimental results of adhesion are also shown in Fig.1.



(a) No voltage applied (b) 5[V] applied

Fig. 1. Adhesion results on aluminum test plate.

Table 1. Experiment conditions of SPM.

Test plate	Aluminum, Stainless
Scanning range	10[μm]*10[μm]
Scanning speed	20[μm/s]
Number of measurement points	256*256
Temperature of test surface	-5[℃]
Pressing force(LFM mode)	1000[nN]
Surfactant	Hexadecyltrimethylammonium Bromide(cation)
Applied voltage	0[V], ±5[V], ±9[V]

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OS3-1 L2C – Wednesday, 13 Sep. 10.10-10.30 – ROOM N2

Experimental and Predicted Speed of Sound and Virial coefficients for Mixtures in Gaseous Phase

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Due to the imperfection of pure refrigerants [1], the refrigerant mixtures are developed to take into account the thermodynamic performance and safety of the third-generation refrigerant hydrofluorocarbons (HFCs) and the environmental friendliness of the fourth-generation refrigerant hydrofluoroolefins (HFOs). Speed of sound is a fundamental thermodynamic property which characterizes the physical system and can derive other important properties including virial coefficient, density, and ideal heat capacity.

In this work, we modified the experimental procedure of the cylindrical fixed-path acoustic resonance method [2] for the speed-of-sound measurement of mixtures. Then we obtained speed of sound data for the binary mixture 2,3,3,3-tetrafuoropropene (HFO1234yf) + 1,1,1,2-tetrafluoroethane (HFC134a) at two molar fractions in the temperature and pressure range of (295 to 363) K and (29 to 1010) kPa, respectively. The combined relative uncertainty (k=2) is within a few parts of 10^4. The accurate molar fractions along each isotherm line were determined the standard with the absolute uncertainty of 0.0015 by extrapolating the experimental speed of sound to zero pressure. The interaction virial coefficients were derived from the experimental data. Additionally, we modified a model to predict the speed of sound with no requirement of experimental data.

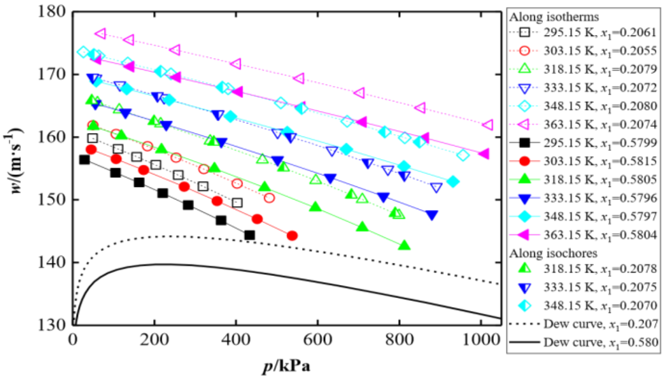

Fig. 1. Experimental gaseous speed of sound for HFO1234yf(1) + HFC134a(2) binary mixture.

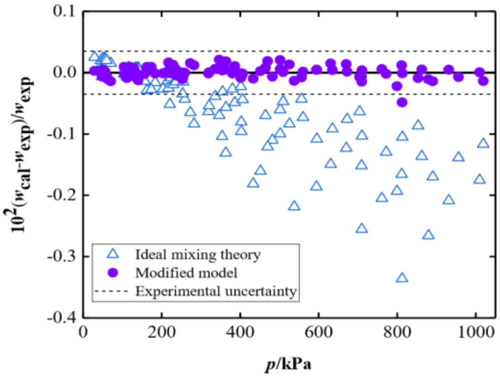

Fig. 2. Predicted gaseous speed of sound for HFO1234yf(1) + HFC134a(2) binary mixture.

Acknowledgment

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OS3-1 L2D – Wednesday, 13 Sep. 10.10-10.30 – ROOM N1

Liquid iron viscosity measurements by aerodynamic levitation, both numerical and experimental approaches

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Thermophysical properties measurements on liquid metals is a challenging process due to both high temperatures and complex behavior of the liquid phase. Several ground-based methods are studied by some laboratories and IRDL has developed since 2017 an aerodynamic levitation apparatus allowing high temperatures (1000 °C - 3000 °C) and non-contact measurements (Figure 1). This device is currently operational for density [1] and surface tension [2, 3] measurements and now in development for viscosity estimation. The classic methodology with levitation consists first in finding of the resonating frequency of the aerodynamically levitated sample. The second step is the excitation that is brutally stopped to observe the relaxation time (Figure 2).

This communication will present an actual limitation observed by the team where the gravity seems to be impacting the measurement for low viscosity metals like iron ($\mu<10$ mPa·s). First results with liquid iron and comparison with literature show some differences.

Then, a multiphysics numerical model will be presented. This model solves heat transfer and fluid flow, and moving mesh in both the levitating gas and the liquid metal is developed in the finite elements code Comsol Multiphysics. It will be used to study and discuss the impact of earth gravity and/or the surrounding gas during measurement. Indeed, the gravity can be removed easily in the model to compare the apparent viscosity determined with the estimation algorithm. Finally, outlooks and conclusions will be given to improve ground-based measurements methods.

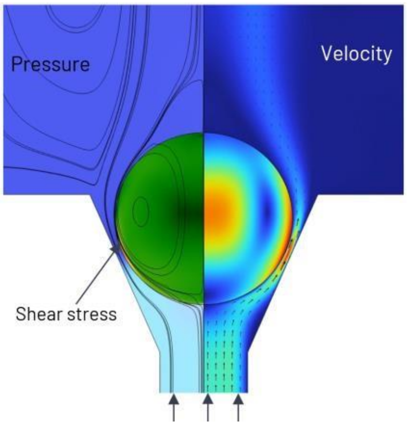

Fig. 1. Multiphysics modeling of the experiment.

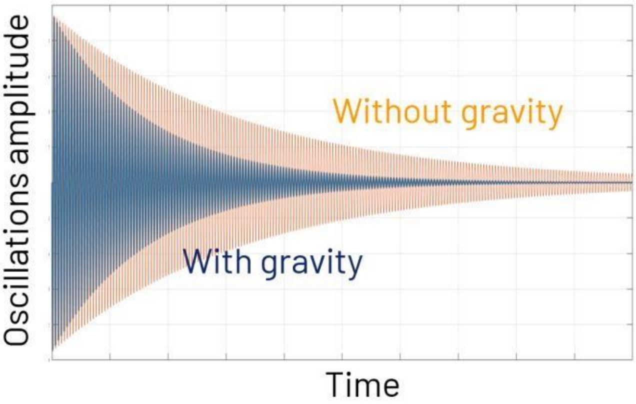

Fig. 2. Effects of gravity impact.

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OS3-1 L2E – Wednesday, 13 Sep. 10.10-10.30 – ROOM M2

Methodology for estimating uncertainty on high-temperature thermal diffusivity measurements

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The measurement capabilities of the homemade laser flash apparatus (LFA) from LNE have been enhanced in order to perform accurate and reliable thermal diffusivity measurements of homogeneous solid materials up to 3000 °C. Uncertainties associated with these measurements have been estimated for the first time for ultra-high temperature, by applying a proven metrological approach composed of four successive steps (as illustrated in figure 1).

The measurement process is analysed in a first step by determining the measurand which is the quantity intended to be measured, by identifying the input quantities and by establishing the measurement model linking the input quantities to the measurand. In a second step, the uncertainty sources associated to the input quantities and to the measurement model are identified and quantified. The final expression of the uncertainty associated with the measurand is calculated in the two last steps by propagating the uncertainties of the influence quantities.

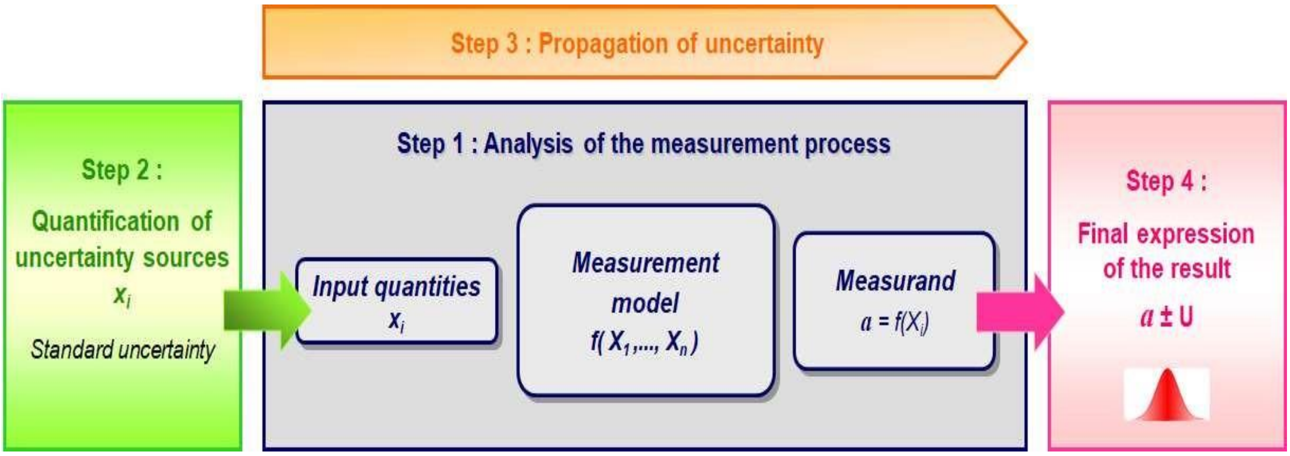

Fig. 1. Assessment of uncertainties in four successive steps.

This new configuration of the LNE's LFA has been applied for measuring the thermal diffusivity of pure refractory materials as well as of some typical advanced materials used by nuclear energy, space and aeronautics industries. The relative expanded uncertainty (k=2) of the thermal diffusivity measurement is estimated to be between 3 % and 5 % in the temperature range from 23 °C to 3000 °C for the investigated refractory materials. This paper presents in detail the assessment of uncertainty associated with these very high temperature thermal diffusivity measurements.

Acknowledgment

The project 17IND11 Hi-TRACE has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

Isobaric Heat Capacity via MD and Quantum Corrections: the case of water.

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The calculation of the heat capacity has been a fundamental part of the evaluation of the thermodynamic properties of molecular fluids from theoretical models. Since its development in the 1970's, Molecular Dynamics (MD) has been used widely to evaluate this property. To capture the complex behavior of molecular fluids, force fields of increasing complexity have been proposed. Such is the case of water where a plethora of force models have been proposed and parametrized, each one following a different philosophy. However, despite its widespread use, standard MD is not capable of capturing the quantum effects inherent in complex fluids due to its classical nature. To mitigate such limitations quantum correction schemes have been proposed, however their performance is rarely evaluated especially for complex force fields. The present work aims to evaluate the impact of quantum effects on the computation of isobaric heat capacities of molecular systems via classical all-atoms MD simulations.

The quantum correction approaches proposed by Berens et al. [1] and by Horn et al. [2] are evaluated for two of the most accurate polarizable water models available: the iAmoeba [3] and Amoeba14 models [4]. For these two models, a series of MD simulations of water molecules were performed at different temperatures (273K360K) and a fixed pressure of 1 atm. Enthalpies and velocity autocorrelation functions are extracted from the simulations along with isobaric heat capacities obtained from fluctuation formulae. Additionally, enthalpies and isobaric heat capacities are quantum corrected using both the Berens et al. [1] and Horn et al. [2] approaches.

Our results show a strong influence of the quantum corrections in the computation of the heat capacity for both models. Compared to the approach of Horn et al., the scheme of Berens et al. shows larger quantum corrections at lower temperatures, affecting the qualitative behavior of the isobaric heat capacity. Comparison with experimental data of isobaric heat capacity of water shows that the Berens et al. scheme produced better results for both water models when quantum correction is applied to the isobaric heat capacity obtained from enthalpy fluctuations. Quantum corrections based on the scheme by Berens et al. are demonstrated to be a very effective and powerful way to compensate for the intrinsic limitations of classical MD simulations allowing for a computationally inexpensive way to improve transferability of a molecular model.

Acknowledgment

We acknowledge partial funding from the Australian Research Council Grant ID: DP190103538. This work was performed on the OzSTAR national facility at Swinburne University of Technology. The OzSTAR program receives funding in part from the Astronomy National Collaborative Research Infrastructure Strategy (NCRIS) allocation provided by the Australian Government, and from the Victorian Higher Education State Investment Fund (VHESIF) provided by the Victorian Government.

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OS3-1 L3C – Wednesday, 13 Sep. 10.30-10.50 – ROOM N2

Speed of sound measurements in binary mixtures of hydrogen with pentane and hydrogen with iso-pentane using a clamp-on ultrasonic flow meter

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New thermodynamic models are requested for supporting the adoption of hydrogen as an energy vector since the available standard formulations do not allow to predict the properties of mixtures, with the necessary accuracy, when the content of hydrogen is higher than 10 %. For supporting the implementation of updated equations of state, measurements of speed of sound have been performed in a mixture of hydrogen and pentane, and a mixture of hydrogen and iso-pentane both at concentrations of the second components of 1 %, 2 % and 5 %. The clamp-on ultrasonic flow meter has been calibrated in the temperature range of (250 and 330) K and pressure up to 5 MPa, using helium as reference fluids.

The choice of using a clamp-on flow meter represents a preliminary step in the path for the adoption of transfer standards of speed of sound for the *in-situ* verification and calibration of ultrasonic flow meters. The outcome of the experiments will be used to characterize hydrogen-enriched gases by their thermodynamic properties as part of the EURAMET project “Metrology for decarbonizing the gas grid” (Decarb).

OS3-1 L3D – Wednesday, 13 Sep. 10.30-10.50 – ROOM N1

Densities, Surface-tensions, and Viscosities of Molten Ni-based Superalloys

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To perform casting simulations with high accuracy, determining the thermophysical properties of Ni-based superalloys with high precision is necessary. In thermofluidic analysis, viscosity is the most important thermophysical property. However, the viscosities of alloys are difficult to measure because they are less than that of molten glass, and data are limited. In this study, to derive the viscosities of Ni-based superalloys, their densities are first measured using the maximum bubble pressure method [1]. The viscosities and surface tensions are then evaluated using the vibrating crucible method [2] and maximum vapor pressure method, respectively. Measurement results of the average densities and surface tensions of the molten Ni-based alloys in the temperature range of 1653–1773 K are listed in Table 1, and their viscosities based on temperature are presented in Fig. 1. We plan to discuss the relationships among surface tension, density, and viscosity based on these data.

Table 1 Densities and surface tensions of molten Ni-based alloys
(Averages derived from the temperature range of 1653–1773 K)

Sample	Density (kgm^{-3})	Surface tension (mNm^{-1})
Alloy 65	7.52×10^3	1.55×10^3
Alloy WA	7.43×10^3	1.54×10^3
Alloy 718	7.82×10^3	1.47×10^3
Alloy 720	7.22×10^3	1.51×10^3

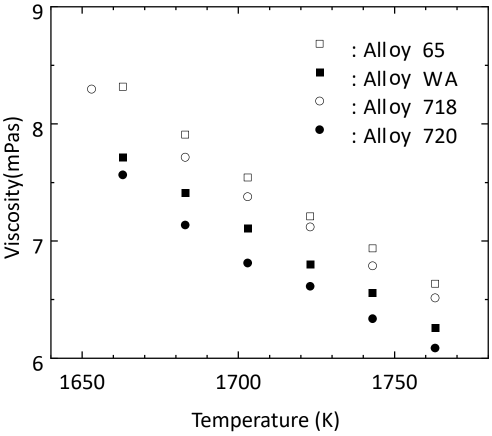

Fig. 1. Viscosities with temperature of Ni superalloys.

Acknowledgments

This work was supported by the sabbatical program at Ibaraki University. We appreciate the support of R. Sato, and H. Nara at Ibaraki University as well as H.-P. Heller, A. Yehorov, N. Levchenko, N. Tinkova, O. Kovtun, and W. Xingern of TU Bergakademie Freiberg and R. Endo of the Shibaura Institute of Technology. This work was also supported by funding from Daido Steel Co. Ltd.

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Characterization of thermal properties of mold in petri dishes by laser flash

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Microbiology standard procedures for bacteria or micro-organisms detection in liquid or gas media usually require incubation of the samples for a few days [1]. The complete work focuses on culturebased methods to perform a count of the number of bacterial cultures that reproduce after the standardized test for respective regulation. But, for preliminary approach, this presentation aims at the fast detection of the micro-organisms by laser flash excitation on a plate with culture media containing molds, which temperature is measured with an infrared camera. Temperature variations during the cooling are compared with temperatures solution of a direct lumped model. This model depends on several physical and geometrical parameters, that are estimated through Bayesian inference with Monte Carlo Markov Chain method and Metropolis-Hastings algorithms [2][3]. Results are discussed and compared with data encountered in the literature.

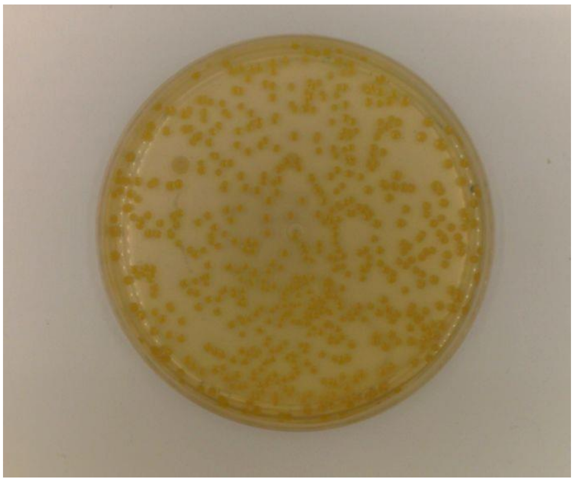

Fig. 1. Example of bacteria colonies.

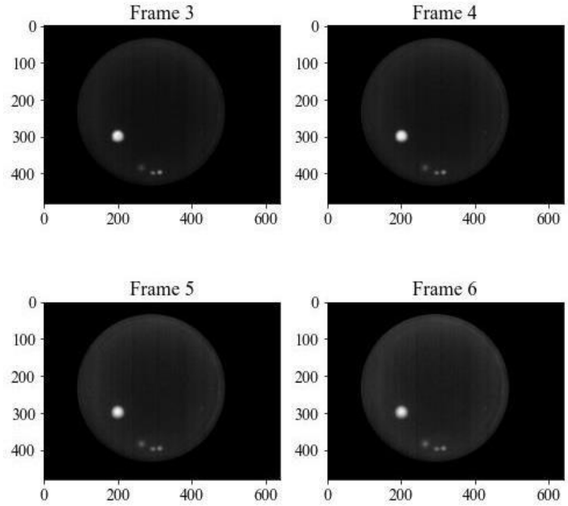

Fig. 2. Examples of IR frames after light flash (the values are the *x*- and *y*-direction pixels).

Acknowledgment

The support provided by CNPq and FAPERJ, agencies of the Brazilian and Rio de-Janeiro state governments, as well as of DGI of Universidad Santiago de Cali, Colombia is gratefully appreciated. Also, the Brazilian company Baktron laboratory, for provide us materials for the experiments.

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OS3-1 L4A – Wednesday, 13 Sep. 10.50-11.10 – ROOM O2

Reference Correlations of the Viscosity and Thermal Conductivity of 1-Hexene from the Triple Point to High Temperatures and Pressures

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1-Hexene (IUPAC name), also known as hex-1-ene, hexylene, or butyl ethylene, is an industrially significant linear alpha olefin, with primary use as a co-monomer in the production of high-density and linear low-density polyethylene. It is also employed in the production of linear aldehyde, isoprene, methylisobutylcetone, and higher fatty alcohols. Furthermore, 1-hexene is employed as a hydrophobe in oil-soluble surfactants, as a lubricating fluid, and as a catalyzer.

There is currently no reference correlation for the viscosity, nor the thermal conductivity of 1-hexene, probably attributed to the fact that an equation of states for 1-hexene has only just now been published [1].

Hence, new wide-ranging correlations for the viscosity and thermal conductivity of 1-hexene based on critically evaluated experimental data are presented. All experimental data have been categorized into primary and secondary data according to the quality of measurement, the technique employed and the presentation of the data, as specified by a series of criteria. The primary data are employed for the development of the reference correlations, while the secondary data are only used for comparison reasons. The viscosity correlation is valid from the triple point to 580 K and up to 245 MPa pressure, while the thermal conductivity is valid from the triple point to 620 K and 200 MPa pressure. Both correlations are designed to be used with a recently published equation of state that extends from 140 K to 535 K, at pressures up to 245 MPa. The estimated uncertainty at a 95% confidence level is 2% for the viscosity of low-density gas (pressures below 0.5 MPa), and 4.8% over the full range of application. The thermal conductivity of the low-density gas has an estimated uncertainty of 3%, and 4% over the full range of application.

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Speed of sound measurements and correlation of {(1-x)3,3,3-trifluoropropene (HFO-1243zf) + x2,3,3,3-tetrafluoropropene (H FO-1234yf)} with x= (0.1582, 0.4625, 0.7623) at temperatures from 243.15 to 343.15 K and pressures up to 90 MPa

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In the search for a fourth generation of environmentally friendly refrigerants capable of meeting the numerous requirements of the EU F-Gas Regulation (REGULATION (EU) No. 517/2014) and the Kigali Amendment to the Montreal Protocol, hydrofluoroolefins (HFOs) have attracted increasing interest in the HVAC&R industry as promising alternatives to the established hydrofluorocarbons (HFCs) and hydrochlorofluorocarbons (HCFCs), currently being phased out. Despite their potential in terms of thermodynamic and regulatory requirements, a paucity of data has been found in the literature on the thermodynamic properties of HFOs and their mixtures, which are required for the development of dedicated equations of state (EoS) [1]. In the present study, the speed of sound of the binary mixture {(1-x)R1243zf + xR1234yf} was measured with a double-path pulse-echo device, previously calibrated with toluene, using literature data from Dhakal et al. [2]. Measurements were made for three mixtures with x= (0.1582, 0.4625, 0.7623) along 8 isotherms ranging from 243.15 to 343.15 K and at pressures from near saturation to 90 MPa. The presented data, which to date represent the only available data set for the speed of sound of the selected mixture, were correlated by a dedicated empirical equation as a function of temperature and pressure. Furthermore, the experimental data were used to re-parameterise a Helmholtz free energy EoS which was previously optimised solely on the basis of experimental VLE data [3]. Using the EoS thus improved, good agreement was observed between experimental and correlated data for the binary system, both for speed of sound and VLE measurements.

Acknowledgment

This work was supported by the Royal Society through an International Exchanges 2021 Cost Share CNR award, reference number IEC/R2/212092.

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Thermophysical properties of liquid Al-Ni based alloys: experiments vs modelling.

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The present study is the state of the art in the thermophysical properties of liquid Al-Ni-based alloys, widely used as functional and structural materials. Manufacturing of simple or complicated parts by different casting processes involve complex interactions between various parameters related to material composition and operating conditions, and often, the manufacture of defect free casting products is almost impossible. Therefore, to prevent formation of casting defects, much attention has been paid to the modelling of solidification. Development of numerical optimization techniques and availability of commercial software packages together with new generation of powerful supercomputers and accurate property data are needed for engineering design of materials by controlling composition and microstructure. However, the use of such mathematical and numer ical tools for the modelling of solidification is often limited by the lack or paucity of reliable thermophysical properties data, such as surface tension and density, thermal conductivity, diffusivity and viscosity of relevant liquid metals and alloys, needed as input parameters for the computational models. Indeed, the high reactivity of Ni-based superalloys, together with impossibility to find chemically inert crucible or support materials to avoid the reactions at the interface, is the main problem when deal with conventional experiments. In order to overcome these limitations, in the framework of the ESA-MAP Thermolab and Thermoprop projects, the containerless processing using non-contact diagnostic tools has been applied reducing the interactions between the melt and its environment. Practical benefits of containerless processing include suppression of heterogeneous nucleation enhancing undercooling of the melt and making it possible to achieve isothermal solidification near its liquidus or much lower temperatures. In this work, the thermophysical properties data of Ni-based industrial alloys are collected and compared to the corresponding model predicted values.

OS3-1 L4E – Wednesday, 13 Sep. 10.50-11.10 – ROOM M2

Photothermal investigation of original and degraded asphalt

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Asphalt is one of the most common materials used in road construction. It is subject to both chemical and structural aging processes during use. At least to our knowledge, it is not currently known whether these aging processes also lead to a measurable change in the thermal properties of asphalt. If so, these changes could be exploited for non-destructive testing of the aging condition in situ. Photothermal analysis of a component surface involves looking at the time course of the surface temperature during and after pulse-like heating with an expanded laser beam. In the case of concrete surfaces, this method works well under laboratory conditions, as recently published [1]. It allowed the determination of the thermal effusivity. Now it should be investigated whether the photothermal signal allows conclusions to be made regarding the aging state of the asphalt.

Within the scope of this paper, 2 asphalt specimens were investigated: a closed asphalt with 3% void content (SMA 11S) and an open-pore asphalt with 25% void content (PA 8). Both samples were artificially degraded according to a standardized procedure, leaving a portion of the surface unaffected [2]. Subsections from both areas were then separated for photothermal testing. Figure 1 shows the results of these investigations.

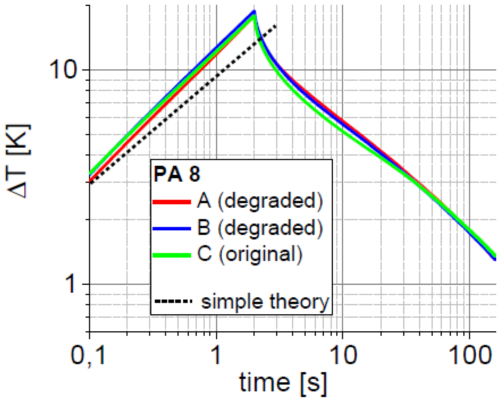

Fig. 1a. Transients of temperature change on the surface of the open-pore asphalt, measured on different parts (A, B degraded, C original state)

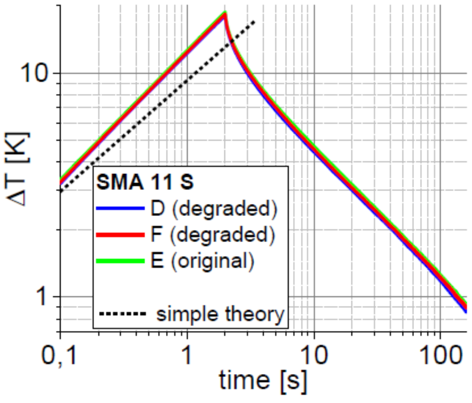

Fig. 1b. Transients of temperature change on the surface of the closed asphalt, measured on different parts (D, F degraded, G original state)

In the case of the PA8 porous asphalt, cooling takes place somewhat faster in the original state than in the degraded state. For the other sample SMA 11 S, no measurable difference could be found. This means, first of all, that the photothermal method is apparently not sensitive enough to detect aging on asphalt in general.

However, it is noteworthy that both asphalt types heat up significantly faster than would be expected from the theory of heat conduction. These results will be discussed in more detail in the paper, also taking into account the respective fine structure of the asphalt.

Acknowledgment

The authors like to thank Philipp Rückert of the ASPHALTA Prüf- und Forschungslaboratorium GmbH Berlin for providing the samples.

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Thermal expansion coefficients of laser surface-modified bimorph microactuator

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Thermal bimorph microactuators which convert external thermal energy into kinetic energy are expected to be applied to micro-robots. The thermal bimorph microactuators are driven by the difference in coefficient of thermal expansion (CTE) of two materials. This study proposes the femtosecond laser surface modification method to create the soft bimorph microstructure. First, a three-dimensional microstructure of PDMS was built by two photon polymerization 3D printing. Next, the surface of 3D printed PDMS microstructure was modified using a femtosecond laser. To determine CTE of the modified surface, we fabricated the microscale bimorph cantilever by using the method written in above. The curvature changes of the bimorph cantilever are dependent on CTE, Young's modulus, and temperature rise. The Young's modulus, which was the necessary parameter to estimate the CTE of the surface-modified area, was measured by a Nanoindenter. Figure 1 shows experimental results of the curvature changes due to thermal stimulation. The displacement of the bimorph cantilever was captured by CCD camera. The curvature changes due to heat stimuli were extracted from the captured images. The curvatures were 2.95×10^{-3} /µm at 300 K and 2.16×10^{-3} /µm at 373 K, respectively. From the curvature change, the CTE at the surface-modified is estimated to be about three times lower than that of PDMS. The precise design and control of the thermal bimotph microactuators can be achieved based on the estimated CTE and Young's modulus of modified surface.

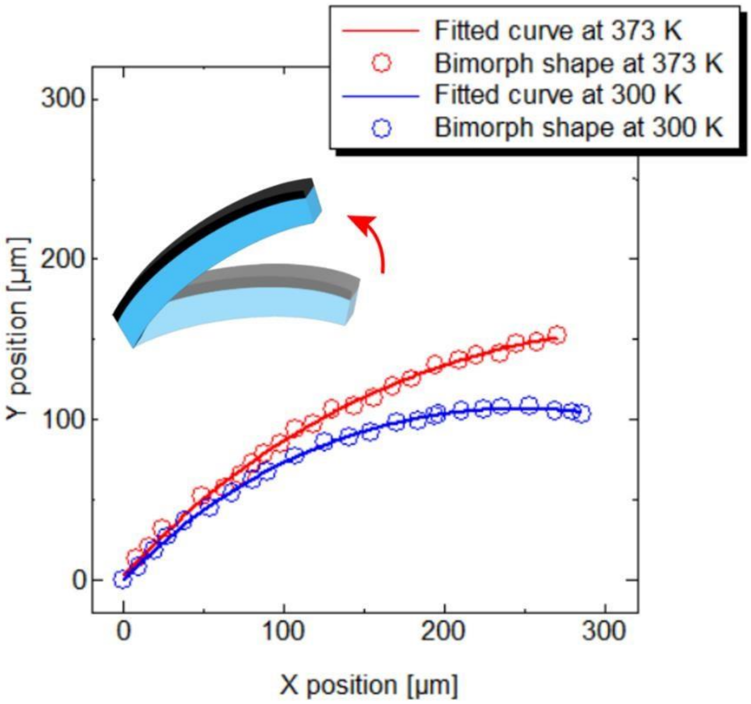

Fig. 1. The bimorph curvature changes due to thermal stimulation.

Acknowledgment

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Thermodynamic Properties of Binary Refrigerant Blends Containing R-32, R-152a, R-227ea, R-1234yf, and R-1234ze(E): Vapor and Liquid (*p*-*ρ*-*T*-*x*) Measurements and Mixture Models

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The pressure-density-temperature-composition (*p*-*ρ*-*T*-*x*) data of binary refrigerant mixtures containing R-32 (difluoromethane), R-152a (1,1-difluoroethane), R-227ea (1,1,1,2,3,3,3-heptafluoropropane), R-1234yf (2,3,3,3-tetrafluoropropene), and R-1234ze(E) (*trans*-1,3,3,3-tetrafluoropropene) were measured in both the vapor and liquid phases using a two-sinker, magnetic-suspension densimeter. The specific samples in this study comprised two compositions of approximately (0.3/0.7) molar and (0.7/0.3) molar for each of the following four binary refrigerant blends: R-32 + R-1234yf, R-32 + R-1234ze(E), R-1234yf + R-152a, and R-1234ze(E) + R-227ea. Single-phase vapor densities were measured over a temperature range of approximately (253 to 293) K and pressures from (0.05 to 0.98) MPa. Single-phase liquid and supercritical densities were measured over a temperature range of approximately (230 to 400) K and pressures up to 22 MPa; for refrigerant blends containing R-1234yf the maximum pressure was limited to 14 MPa. Overall relative combined, expanded uncertainties in density ranged from 0.025% to 0.191%, with an average uncertainty of approximately 0.05%. The density measurements were part of a larger measurement campaign that included speed of sound and phase equilibria; these data were combined with available literature data to develop improved thermodynamic models for these systems. For all four systems, the new models result in density deviations of 0.1% or less. Here we present the density measurement results, along with a brief overview of the new thermodynamic models.

Accurate Measurements of the Thermal Conductivity of *n*-Docosane, *n*-Tetracosane, 1-Hexadecanol, 1-Octadecanol, 1,6-Hexanediol, and 1,8-Octanediol in the Solid and Liquid Phases

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New measurements of the thermal conductivity of *n*-docosane, *n*-tetracosane, 1-hexadecanol, 1-octadecanol, 1,6hexanediol, and 1,8-octanediol, in the solid and liquid phase, are presented. The technique employed is the transient hot-wire technique, based on a full theoretical model. The technique is absolute and is characterized by an uncertainty of 1%.

One of the main issues when measuring the thermal conductivity of a solid material, especially when employing methods based on thermal contact, whether they be the transient hot-wire technique (THW), or any other, is the thermal resistance between the solid sample(s) and the source of heat or thermometer. The resistance is often attributed to a thin layer of air that is present in the space between the imperfectly smooth surfaces of the test material and the other surfaces. To avoid this thermal resistance, for the first time, the THW technique is employed by immersing the wires directly into a melted organic Phase Changing Material (PCM) and letting the system cool down. In this way, upon solidification, the contact between the heating wire and the solid sample is excellent, and the value of the thermal conductivity can be attained directly from the analytical solution as well as from the numerical solution.

The experimental measurements of the thermal conductivity for the six PCMs were fitted to a straight line as a function of the temperature. Table 1 shows the standard deviations (at the 95% confidence level) of the measurements from the straight line, in the solid and liquid phases, respectively.

Table 1. Standard deviations, at the 95% confidence level.

	Solid phase		Liquid phase	
	Std.Deviations / %	Temperature range / K	Std.Deviations / %	Temperature range / K
n-docosane	0.40	267 – 308	0.60	330 – 360
n-tetracosane	0.60	265 – 306	0.30	339 – 363
1-hexadecanol	0.65	266 – 315	0.16	334 – 361
1-octadecanol	0.46	259 – 324	0.33	340 – 363
1,6-hexanediol	0.70	263 – 296	0.40	327 – 351
1,8-octanediol	1.10	265 – 312	0.50	344 – 356

Acknowledgment

The help of A. Assimopoulou and M.C.M. Sequeira is gratefully acknowledged.

Experimental and numerical study of the effect of surface texture on the emissivity of metallic materials

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Pyrometric and thermographic techniques are frequently required in the metal industry in order to obtain the necessary metallurgical properties during synthesis processes. However, non-contact temperature measurements are particularly complicated in metals, because of their low and highly variable emissivity, which is strongly influenced by the surface finish of the material. For this reason, this work presents a combined experimental and numerical investigation of the emissivity of metallic materials of interest taking into consideration their surface texture and roughness.

First, results corresponding to a model system are presented. Directional spectral emissivity measurements have been performed for a sample of pure rolled aluminum in the relevant temperature range for non-contact temperature applications ($T = 423$ - 823 K) in vacuum and for both S and P polarizations [1,2]. Moreover, rigorous coupled-wave analysis (RCWA) has been used to model the optical response of the material while considering its surface roughness. We prove that the cold-rolling process induces a multi-scale roughness profile that significantly enhances emission at all wavelengths, and that a small band in the P-polarized component at oblique angles is formed by a native oxide layer. The strong influence of the surface state on the emissivity explains the significant discrepancies observed in the literature and demonstrates that an accurate characterization of the surface texture is mandatory for the comparison of data.

In addition, preliminary results corresponding to metallic materials of industrial interest will be shown. The directional spectral emissivity and bidirectional spectral reflectivity of Inconel 718 samples with different surface patterns have also been studied, in order to assess the impact of the surface structure on its radiative properties. These results are being analyzed by numerical simulations based on ray tracing simulations.

In conclusion, these findings demonstrate the importance of characterizing the radiative properties of metallic materials with surface textures, and the possibility of reproducing them using numerical methods. These aspects could play an important role in the design and optimization of metallic materials and pave the way to further research. In addition, the work presents emissivity data of great importance for the application of thermographic techniques in the metallurgical industry.

Acknowledgment

This work was funded by the University of the Basque Country, Spain (GIU19/019) and the Basque Government, Spain (IT-1714-22 and PIBA-2021-1-0022). J. Gabirondo-López, M. Sainz-Menchón and I. González de Arrieta also acknowledge financial support from pre- and post-doctoral fellowships by these institutions (University of the Basque Country, Spain: PIF 21/06; Basque Government, Spain : PRE-2022-10086, POS-2021-2-0022).

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OS3-2 L2B – Wednesday, 13 Sep. 12.00-12.20 – ROOM O1

Validation of a Simple but Versatile Thermal Contraction Measurement Setup for Temperatures between Room Temperature and 1.8 K

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High-energy accelerators require the use of cryogenics to cool down the superconducting magnets that guide and focus the particle beams. Understanding the thermo-mechanical behavior of the constituent materials of the magnets, particularly of Nb_3Sn-based superconducting coils, is paramount for magnet performance because of their highly strain-sensitive superconductivity. Due to the scarcity of commercial systems capable of measuring the thermal contraction of the magnet coils, a customized dilatometric test bench for cryogenic temperatures has been developed in collaboration between the Mechanical Measurement Laboratory at CERN and the external company attoCUBE. The dilatometric setup features an optical displacement sensor based on Fiber-Optic FabryPérot interferometry and is integrated into a closed-cycle cryostat providing a minimum temperature of 1.8 K.

During an initial extensive validation campaign, we focused on two main aspects: optimizing and determining the measurement accuracy and elaborating a suitable sample preparation to avoid limitations of the measurable materials caused by the requirements of the optical dilatometry. To quantify the accuracy in the form of a measurement error, we compared measurements on single-crystal silicon samples as a particularly suitable reference material for dilatometric instruments with highly accurate literature data [1]. In addition, the expanded uncertainty was determined according to the GUM [2] to analyze and reduce the sources of error.

Considering both approaches, we achieved an uncertainty in the relative change of length of better than $0.03 \cdot 10^{-3}$, corresponding to about 1 % of the total contraction of copper at 1.8 K. Therefore, our optical dilatometric setup is sufficiently accurate for most cryogenic engineering applications, while featuring a high versatility in the materials to be tested.

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OS3-2 L2C – Wednesday, 13 Sep. 12.00-12.20 – ROOM N2

Thermodynamic Property Measurements of Binary Refrigerant Blends HFO1123 + R290

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In order to prevent global warming, there is an urgent need to find alternative refrigerants to the hydrofluorocarbons (HFCs) currently in use, and hydrofluoroolefins (HFOs) with carbon-carbon double bonds are considered promising candidates because of their low global warming potentials (GWPs). HFO1123 (trifluoroethene) is one of the most notable HFOs, especially for low-temperature refrigeration applications, and a fundamental equation of state (EOS) for HFO1123 has recently been formulated on the basis of the latest experimental data [1]. However, its disproportionation reaction is concerned, and HFO1123 blends with R290 (propane) are considered to suppress this reaction. In the present study, *PVTx* properties, saturated densities, and critical parameters of binary refrigerant blends of 80 mass% HFO1123 + 20 mass% R290 and 65 mass% HFO1123 + 35 mass% R290 were measured. The *PVTx* properties were obtained in the temperature range from 310 to 400 K and at pressures up to 6.9 MPa by the isochoric method, and the experimental results of 80 mass% HFO1123 + 20 mass% R290 are shown in Fig. 1. The saturated densities and critical parameters were determined by the visual observation of the meniscus disappearance and the intensity of the critical opalescence. In addition, vapor-liquid equilibrium of the blends was measured from 263 to 323 K based on the recirculation method. These comprehensive measurements are useful for verifying the thermodynamic consistency and determining the mixing parameters of the EOSs for HFO1123 + R290 blends.

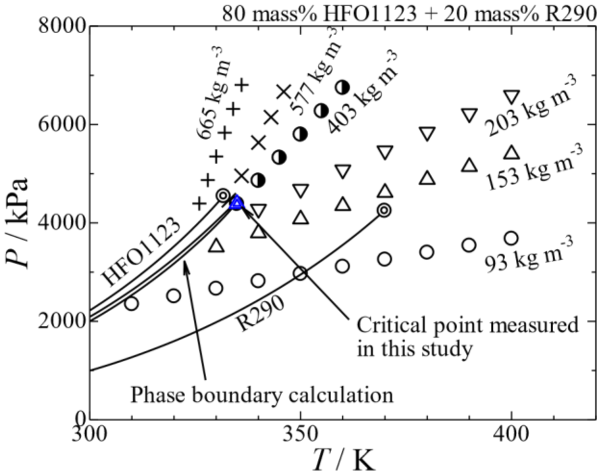

Fig. 1. Experimental results of *PVTx* properties of 80 mass% HFO1123 + 20 mass% R290. The solid lines of the vapor pressures of the pure substances and phase boundary of the blend were calculated by REFPROP 10.0 [2].

Acknowledgment

This research has been supported by a grant from New Energy and Industrial Technology Development Organization (NEDO), Japan. We are greatly thankful to AGC Inc. for providing the sample of HFO1123.

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OS3-2 L2D – Wednesday, 13 Sep. 12.00-12.20 – ROOM N1

Investigation on influence of applying voltage to a mixture of two surfactants with different molecular diameters on supercooling degree

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Recently, ice slurry, a solid-liquid two-phase mixture consisting of water and fine ice, has been attracting attention in the field of thermal storage. One method of producing ice slurry is the supercooling method, in which the liquid is cooled to a supercooled state and then the supercooling is dissipated. However, the supercooling state is unstable and must be actively controlled. The authors added two types of nonionic surfactants with different molecular diameters and confirmed further improvement of supercooling by allowing surfactant molecules with smaller molecular diameters to penetrate between the molecules of surfactants with larger molecular diameters added at the C.M.C. concentration.[1] In this study, with the aim of further improving the controllability of supercooling degree, we investigate the effect of a mixture of nonionic and anionic surfactant solutions with different molecular diameters on supercooling degree. Furthermore, we will apply a voltage to the solid-liquid interface and study the effect of the voltage application on supercooling degree. Fig.1 shows the average supercooling degree at each concentration resulting from the experiment. This figure shows the mean supercooling degree on the vertical axis and the concentration on the horizontal axis. Fig.1 shows that the addition of two surfactants with different molecular diameters further improved supercooling degree even when the concentration of the base metal reached C.M.C. concentration. Furthermore, due to the characteristics of the anionic surfactant, even after interfacial saturation, the supercooling resolution was not promoted by the micelles, and a concentration interval was observed in which the maximum supercooling degree was maintained.

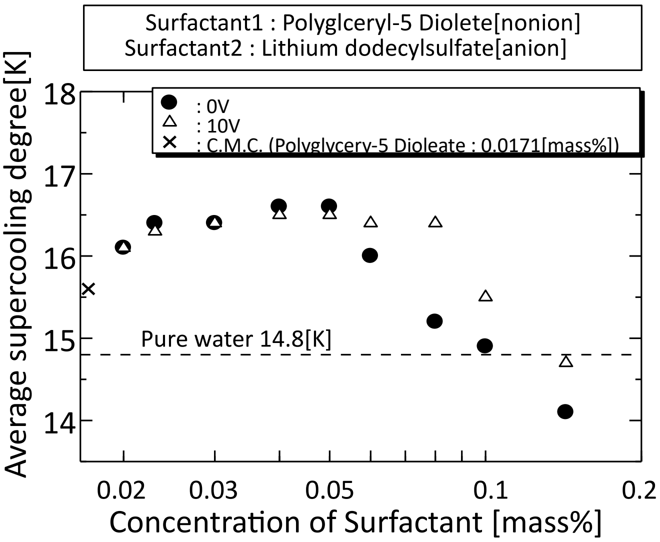

Fig.1 Average supercooling degree at each concentration

Acknowledgment

Use this section for the acknowledgment such as the supporting grants, etc.

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OS3-2 L2E – Wednesday, 13 Sep. 12.00-12.20 – ROOM M2

Study on MEMS heat flux sensor calibrated for low frequency component

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Heat flux sensors for the mechanical product (*e.g.*, internal combustion engines) enables the investigation of the heat transfer between the wall and fluid in the metal body. Therefore, we have developed MEMS heat flux sensor using metal substrate for the engine use [1]. The sensor can be calibrated by the self-heating of the surface RTD for high frequency component (10 Hz~10 kHz), however, there is a difficulty in calibrating low frequency and DC component of the heat flux. In this study, in order to calibrate the low frequency component, the estimation of thermal properties using cyclic heating was proposed, and the preliminary experiment and analysis was performed. The fabricated sensor, and the cross-section is shown in Fig. 1. The heat flux is calculated by the transient heat conduction analysis using the surface temperature measured by the thin film RTD, and the internal temperature (x = 2.2 mm) by a tiny RTD (Pt 100). The internal thermometer is glued in the hole drilled in the ϕ10 mm Al alloy (A5052) substrate, thus, the thermal resistance and the capacity between the internal thermometer and the substrate should be estimated and reflected into the analysis model.

For the investigation of thermal properties around the internal thermometer, the top surface of the sensor was uniformly and cyclically illuminated by the UV LED. The temperature amplitude of the surface RTD ($\Delta T_{x=0\ \mathrm{mm}}$) and the internal Pt 100 ($\Delta T_{x=2.2\ \mathrm{mm}}$) was measure by the lock-in amplifiers, and the ratio was plotted as shown in Fig. 2. The ratio decreases as frequency increases from 0.001 to 10 Hz, as with the result of semi-infinite heat conduction under cyclic surface heating. The increase around 100 Hz was the effect of the electrical noise. The 1D transient heat conduction simulation including the internal thermal resistance, was also plotted in Fig. 2. The internal thermal resistance (R_{in} = 7.5×10^{-6} $(\mathrm{W/m^2/K})^{-1}$) was estimated by the steady radiative heating [2], and the resistance was reflected into the control volume with 0.1 mm thick just above the internal thermometer. The heat capacity around the internal thermometer was also estimated to be consistent with the experimental value at 1 Hz. The plot was agreed by substituting 38 times the capacity of A5052 to the control volume.

In the present estimation procedure, the characterization of thermal properties consistent with both 1 Hz and 10 Hz was not realized. Further study will be performed to construct the calibration protocol consistent with wide range of frequency, by considering the effect of the thermal resistance between the surface thin film and substrate, and improving the experimental system for the estimation of the internal thermal resistance.

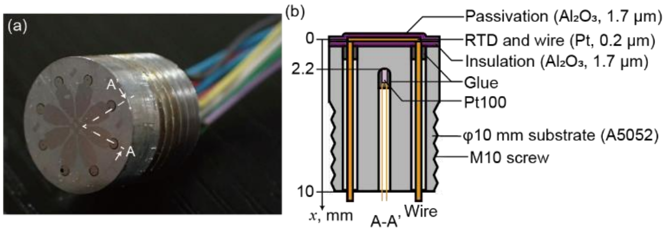

Fig. 1 (a) Fabricated sensor, and (b) cross sectional image.

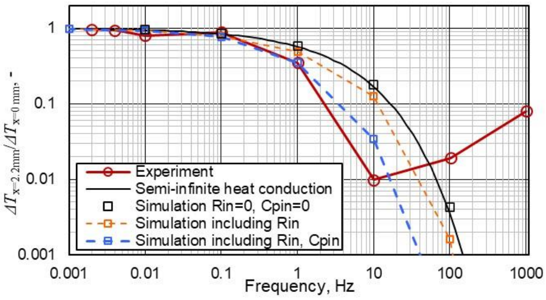

Fig. 2 Frequency characteristics.

Acknowledgment

This work was partially supported by JSPS KAKENHI JP23K13268, and the research association of automotive internal combustion engines (AICE). The fabrication of the sensors was performed in the clean room in "Mechano-Micro Process Room" of Tokyo Institute of Technology, Yokohama, Japan.

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OS3-2 L3B – Wednesday, 13 Sep. 12.20-12.40 – ROOM O1

Feasibility Study of Rayleigh Backscattering Optical Fibre Strain Measurement Technique for Determining the CTE of Lightweight Composite Structures

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The coefficient of thermal expansion (CTE) is an important mechanical property of lightweight composite structures, such as honeycomb panels. However, measuring the CTE of these structures is challenging, as they are typically not possible to measure with other well-established methods due to their complex geometries. In this study, we investigate the feasibility of using the Rayleigh backscattering optical fibre strain measurement technique to determine the CTE of composite structures.

To determine strain using Rayleigh backscattering technique, an optical fibre is attached to a surface. When the surface experiences deformation, the deformation causes strain in the fibre which leads to changes in the backscattered light intensity. However, in this study, we propose a novel approach where the fibre is only bonded in two points, allowing to decouple thermal effects over the fibre by comparison with an unstrained fibre. To validate the feasibility of this technique, the CTE measurements obtained with Rayleigh backscattering technique over well-known materials, as invar, were compared with measurements over the same materials performed with a horizontal push-rod dilatometer.

Our results show that this novel approach is a practical method for determining the CTE of assembled composite structures. The technique is non-destructive, requires minimal sample preparation, and can provide highaccuracy measurements. These findings have significant implications for the design and optimization of composite structures in various applications, such as particle detectors, aerospace, and automotive industries.

OS3-2 L3C – Wednesday, 13 Sep. 12.20-12.40 – ROOM N2

Gaseous *pvTx* experimental measurements and models for the binary mixtures of carbon dioxide(CO_2) with 3,3,3-trifluoropropene(R1243zf) or 1,3,3,3-tetrafluoroprop-1ene(R1234ze(E))

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As low GWP refrigerants, hydrofluoroolefins (HFOs) and its mixtures have wide application prospects. Carbon dioxide has also drawn widely attention in thermal systems. In this work, the gaseous *pvTx* properties for binary mixtures of CO_2 + R1243zf and CO_2 + R1234ze(E) were measured by the Burnett apparatus from 333.15 to 393.15 K and from 0.6 to 6 MPa. The maximum standard measurement uncertainty (k=1) of temperature, pressure, mole fraction and mole density were estimated to 10 mK, 0.7 kPa, 0.002 and 0.05%, respectively. The virial equation of state (virial EoS) was established based on the experimental data. The REFPROP 10.0 and the Peng-Robinson equations of state (PR EoS) with the van der Waals mixing rule were used to predict the experimental data and compared with the virial EoS. The binary interaction coefficient in the mixing rule was from the vapor-liquid equilibrium data in literature. The virial EoS shows excellent correlation performance for the experimental data, and the predictive deviation of PR EoS is limited over a wide temperature range.

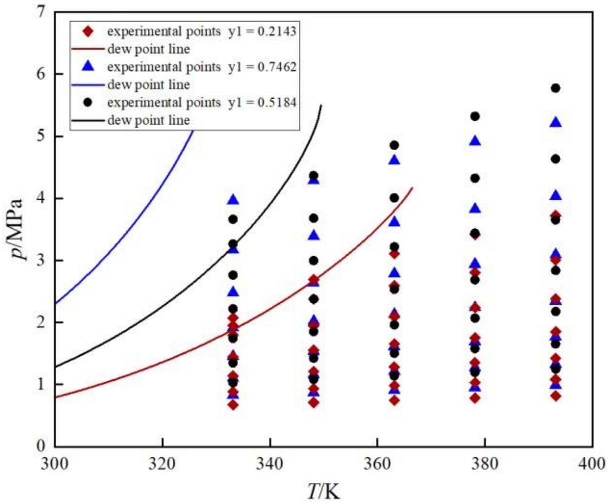

Fig. 1. Gaseous *pvTx* property diagram for the CO_2 (1) + R1243zf (2) binary mixture

OS3-2 L3D – Wednesday, 13 Sep. 12.20-12.40 – ROOM N1

Nano-enhanced isopropyl palmitate for cold storage applications

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Europe is a world leader on clean energy research and is at the forefront of green technology [1], but coal, gas and oil constitute about 80% of the world's energy consumption. Phase change materials (PCM) are attracting attention and, in particular, isopropyl palmitate (IPP) is an interesting eco-friendly solution. Its phase transition temperature is between 283 K and 286 K and its heat of fusion is ~113 J/g [2], so it is suitable for thermal energy storage applications. Nano-enhanced PCM were designed dispersing graphene nanoplatelets of 2 nm thickness (GnP2) in IPP at nanoadditive concentrations from 0.50 to 2.0 wt%. Samples were subjected to a treatment with CTAB as surfactant, with a mass ratio GnP2:CTAB 1:1. A Zetasizer Nano ZS (Malvern Instruments, UK) was used to study the temporal stability of the dispersion. The size of the studied sample remained constant over a period of three weeks. Solid-liquid phase changes have been observed using a Q2000 differential scanning heat flux calorimeter (DSC) (TA Instruments, New Castle, DE, USA). Figure 1 shows the modification of the onset temperature for cooling and freezing processes, where the reduction of the temperature difference between the freezing and the melting for the GnP2 (1.0 wt%)/IPP sample point means a 10% sub-cooling reduction with respect to IPP.

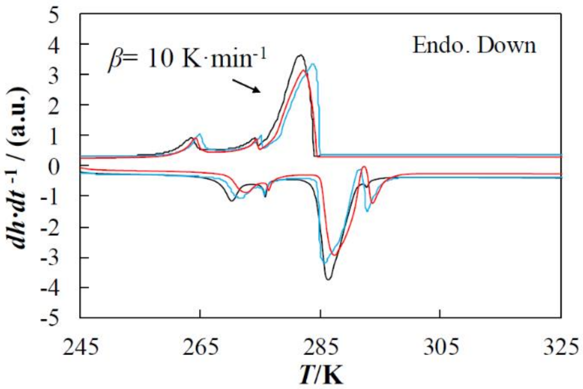

Fig. 1. DSC cooling and heating thermograms at scanning rates of 10 K·min^{-1} for base fluid (—), GnP2(0.50wt.%)/IPP (—), and GnP2(1.0wt.%)/IPP (—). (CTAB:GnP2 1:1).

The experimental determination of thermal conductivity was carried out by means of a THW-L2 device (Thermtest Inc., Hanwell, NB, Canada). The dispersion of graphene nanoplatelets improves the thermal conductivity of the isopropyl palmitate for all studied dispersions. The observed thermal conductivity enhancements increase with the loading of GnP2, reaching a 6.1% improvement for the maximum concentration (2.0 wt.%).

In summary, the temporal stability, phase change transitions and thermal conductivity for GnP2/IPP were experimentally studied. The dispersions of GnP2 in IPP show good stability with the addition of CTAB with a mass ratio GnP2:CTAB 1:1. An increase in thermal conductivity of 6.1% was observed for the highest GnP2 concentration (2.0 wt.%), as well as a reduction in subcooling of 10% for a concentration of 1.0 wt.% of GnP2.

Acknowledgments

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OS3-2 L3E – Wednesday, 13 Sep. 12.20-12.40 – ROOM M2

Emissivity-free two-dimensional radiation thermometry using a high-speed CCD camera

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The radiation thermometry is an essential technique on thermophysical properties measurement for the molten materials at high temperature, especially in the measurement using levitation techniques. However, the emissivity of the samples needed to be calibrated at know temperature, and to be assumed that calibrated emissivity is constant against temperature when we use the radiation thermometry. To overcome these difficulties, dual-wavelength reflectance-ratio method has been developed to measure the temperature without emissivity data [1, 2]. When we apply this technique for the sample, which is moving or having rapid temperature change, high sampling rate and trailing of the sample is required. In this research, the applicability of the two-dimensional emissivity-free radiation thermometry for spherical solid metals at elevated temperature was experimentally investigated using a high-speed CCD camera.

A high-speed CCD camera, which pixel brightness of the images was calibrated using a quasi-blackbody, was used for the experiment. The nickel and copper solid spheres (diameter: 8 mm), which imitate the levitated liquid metal, were electromagnetically heated at the center of rf coil in Ar-5vol%H_2 atmosphere. The sample temperature was monitored using a calibrated K-type thermocouple inserted into the spheres. After the experiment, the monitored temperature was converted to the surface temperature by measuring the temperature difference between the surface and inside of the sample. The radiance of the samples at elevated temperature with and without reflection of the auxiliary light were obtained using the high-speed CCD camera.

The sample temperature was calculated based on the dual-wavelength reflectance-ratio method [1,2]. The obtained temperature agreed with the surface temperature determined by a thermocouple within 50 K in the temperature range of 1100 - 1300 K.

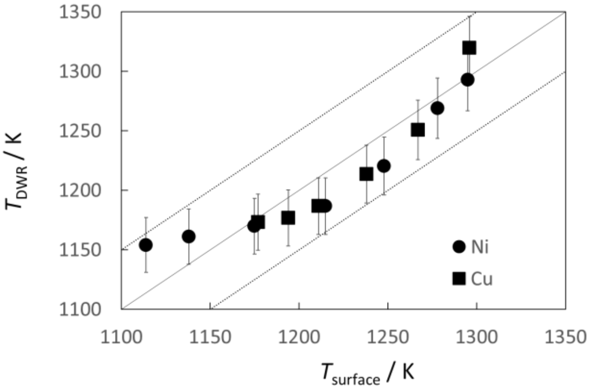

Fig. 1. Correlation between the temperatures measured by the DWR using the high-speed CCD camera and by the thermocouple.

Acknowledgment

This work was supported by JSPS KAKENHI Grant-in-Aid for Challenging Research (Pioneering) Grant Number JP16K14169 and Grant-in-Aid for Scientific Research (B) Grant Number 20H02490. Measurements were conducted using a high-speed camera loaned from Photron Co., Ltd.

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Expanding the Application Range of Vibrating-Tube Densimeters – Performance Test at Cryogenic Temperatures

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Vibrating-tube densimeters (VTDs) enable reasonably low complexity of an experimental apparatus while providing fast and robust measurements with comparatively low uncertainties, particularly in liquid states. VTDs are primarily used at elevated temperatures of up to 723 K [1]. However, in recent studies, a VTD was employed at temperatures as low as 203 K [2,3], yielding reliable results even though the utilized VTD is only rated for temperatures down to 263 K by the manufacturer. Here, we present a performance test of a widely used commercial VTD model (Anton Paar, Austria, type: DMA HPM) at even lower temperatures, i.e., down to 100 K. The low temperatures were achieved by setting up the VTD inside a liquid nitrogen-cooled thermal chamber. The VTD was calibrated with methane and propane as reference fluids over the temperature range from (120 to 200) K at pressures up to 10 MPa, covering a density range from (22 to 702) kg·m^{-3}. Different calibration models [4,5] were used to determine the densities from the measured oscillation period, temperature, and pressure. It was found that the VTD is subject to a shifting vacuum oscillation period when exposed to thermal cycling to temperatures below 180 K but it still operates at temperatures as low as 100 K. The observed shifts diminished during the course of the study but did not stop completely. Accordingly, the calibration models were modified with respect to the treatment of the vacuum oscillation period to account for the shifting characteristics. The best results were obtained with the calibration model of Outcalt and McLinden [4]; evaluations with this model reproduced reference densities within ±0.11 kg·m^{-3}. The calibration was validated with comprehensive density measurements of ethane and argon with the result that the measured densities agree with the reference data within ±0.47 kg·m^{-3}. Estimations of the expanded combined uncertainty (k = 2) in density range from (0.52 to 1.14) kg·m^{-3} for all ethane and argon validation measurements. This corresponds to relative uncertainties of (0.083 to 0.098)% for ethane and (0.144 to 0.346)% for densities in the gas phase of argon. Our study shows that the DMA HPM allows accurate density measurements under cryogenic conditions provided a proper calibration against reference fluids has been performed and that the vacuum oscillation period is repeatedly measured and correctly accounted for in the data analysis. This makes the VTD an accessible alternative to more complex cryogenic densimetry, especially since VTDs can be easily deployed in the field.

Acknowledgment

The authors thank Anton Paar for supporting the present study at Chemnitz University of Technology.
Roland Span acknowledges the support of the ERC-2021-ADG ThermoPropHy.

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Experimental densities of hydrogen-isobutane mixtures with three different compositions

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To target the gas grid decarbonisation and carbon capture and storage, accurate thermophysical properties models are the tools able to ensure the efficiency of the involved processes. This is the framework of goals of the project EMPIR Metrology for Decarbonising the Gas Grid (20IND10 Decarb). In the work here presented, the results of accurate experimental densities of hydrogen and isobutane ($H_2 + iC_4H_{10}$) are discussed. The measurements are carried out by means of a vibrating tube densimeter, in the temperature range of (270 and 330) K and at pressure up to 4 MPa. The mixtures are measured with three different compositions, with 5 %, 10 % and 15 % of iC_4H_{10} respectively. The vibrating tube densimeter has been preliminary characterized by using nitrogen and vacuum as reference fluids.

All terms contributing to the uncertainty in determining the density are considered in order to obtain a final relative uncertainty of the mixture density better than 0.1 %.

The measurements presented will be adopted for reviewing the standard equation of states for custody transfer today unable to model mixtures with a hydrogen content higher than 10 %.

OS3-2 L4D – Wednesday, 13 Sep. 12.40-13.00 – ROOM N1

Solid-liquid phase equilibrium: alkane systems for low temperature energy storage

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The incessant and increasing need for energy requires urgent and effective solutions. The supply of renewable energies is mainly intermittent, which often leads to a gap between the availability of energy and its consumption. Therefore, energy storage becomes imperative for increasing the efficiency in the use of the produced energy. Concerning this, the use of phase change materials (PCMs) has taken a very important role, especially for active cooling systems. In particular, eutectic systems can be designed with characteristics that make them suitable for each application, allowing significant energy savings, which is very relevant in what concerns economic and environmental sustainability [1].

This work aims to the characterization of selected systems that can be used for energy storage at low temperatures, starting by the construction of solid-liquid phase diagrams using differential scanning calorimetry (DSC) and Raman Spectroscopy results. Phase diagrams are crucial to characterize the behaviour of these systems and reveals the robustness of the PCM, and for new PCMs, equilibrium studies are essential to achieve the desired melting temperature [2].

This work is focused on binary systems composed by normal alkanes, in particular, the binary mixtures C_8-C_{10} and C_{10}-C_{12}. These systems revealed to behave as eutectic systems at low temperatures, showing a promising applicability for thermal energy storage for low temperature applications. The eutectic points are around -61ºC and -35ºC, respectively. Additionally, a fitting equation for the experimental liquidus lines of the phase equilibrium diagrams is proposed for each system.

Acknowledgment

Centro de Química Estrutural and Centro de Química de Coimbra are Research Units funded by Fundação para a Ciência e Tecnologia through projects UIDB/00100/2020 and UIDP/00100/2020, and UIDB/00313/2020 and UIDP/00313/2020, respectively. Institute of Molecular Sciences is an Associate Laboratory funded by FCT through project LA/P/0056/2020. M.C.M. Sequeira acknowledges the PhD grant funded by FCT ref. UI/BD/152239/2021.

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Multi-Wavelength Pyrometer for Ultra-Fast Temperature Measurements

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The investigation of thermopysical properties of materials is impossible without accurate temperature measurements. To measure the temperature in the range from one to a few thousand Kelvin, a new multiwavelength pyrometer was built. It is very precise, flexible, and portable and allows temperature measurements with a time resolution of nanoseconds and spatial resolution down to 100 µm.

The pyrometrical determination of surface temperatures by an analysis of thermally emitted light is often the only available method for high temperature measurements, starting from the solid state to the Warm Dense Matter temperature region. The pyrometrical technique is based on measurements of wavelength-dependent thermal radiation or spectral radiance, and its comparison to that of blackbody (Planck) radiation or its Wien's approximation. The main uncertainty in traditional pyrometry is the surface emissivity, which is generally unknown and hard to measure. A commonly employed approach to deal with this problem is to measure the thermal emission at multiple wavelengths – an approach called multi-wavelength pyrometry [1]. The emissivity in that case is approximated by some function of the wavelength. In practice, the grey body approximation $\varepsilon(\lambda) = const$ is often used, but it is accurate only for certain materials in a narrow temperature and wavelength range. In more advanced techniques a polynomial dependence of $\varepsilon(\lambda)$ on λ is employed.

Our device (Fig. 1) has 5 channels and is based on PIN-diodes detectors. Light collected from the sample by parabolic mirrors is transported to the pyrometer by fiber optics. A typical application of the pyrometer is, for example, temperature measurements during the irradiation of samples with intense swift heavy ion beams at the FAIR facility. Software has been written to analyze the spectral radiance with either a linear, a polynomial, or an exponential emissivity model [2].

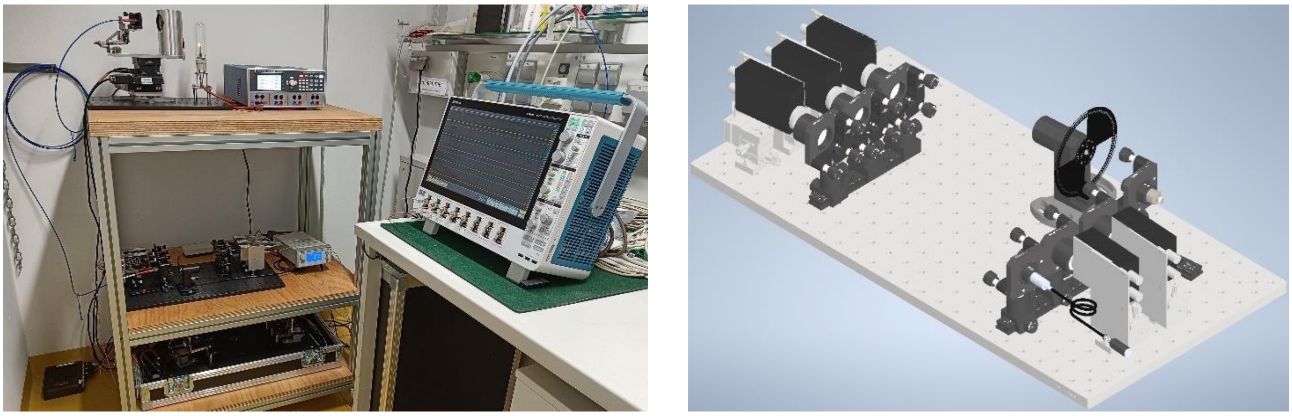

Fig. 1. Photo and scheme of the multi-wavelength pyrometer.

The device has shown its universality and applicability for temperature measurements in the temperature range from 1 kK up to a few thousand Kelvin in various experiments within a broad timescale from nanoseconds to milliseconds.

Acknowledgment

Authors acknowledge support by the BMBF under grant number 05P21RFFA2.

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Thermophysical properties of bicyclic hydrocarbon liquid organic hydrogen carriers

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For the optimization of processes and apparatuses related to the liquid organic hydrogen carrier (LOHC) technology, accurate thermophysical property data at process-relevant conditions are required. However, in the case of the technically promising bicyclic hydrocarbon LOHCs, such data are only sparsely available in the literature. This contribution summarizes current research activities on various thermophysical properties of the LOHC system based on diphenylmethane (DPM, H0-DPM) and benzyltoluene (BT, H0-BT) at process-relevant temperatures T up to 593 K using light scattering and conventional techniques. Experimental data for the viscosity η, surface tension σ, thermal conductivity λ, and density ρ of pure H0-DPM and its partially and fully hydrogenated derivatives H6- and H12-DPM as well as their mixtures at ambient pressure allow for interpretations on the influence of T, degree of hydrogenation (DoH), and mixture composition [1,2]. These measurement results also served for the validation of force fields used for molecular dynamics simulations on η, σ, and ρ [1]. η shows a strongly nonlinear behavior as function of the DoH and also depends on the exact mixture composition, while σ and ρ are nearly independent of the mixture composition at a given DoH. Parallel to surface light scattering experiments, depolarized Raman spectroscopy could be successfully applied for the determination of the DoH with an average absolute deviation from the analytical values below 0.02 [3]. In a next step, the solubility of hydrogen (H_2) in the DPM-based system and the influence of dissolved H_2 on η, σ, and ρ was determined experimentally [2,4]. Despite H_2 mole fractions in the liquid phase of up to 10% at 473 K and 10 MPa, a negligible effect on η as well as ρ and a weak decrease in σ by about 5% at 7 MPa relative to the values of the H_2-free systems was found independent of the DPM-based solvent and T. Measurements on η, σ, and ρ of mixtures of H0-DPM with its oxygenated derivative benzophenone as potential co-product within the LOHC cycle revealed an increase in all three properties, especially in η, with increasing benzophenone concentration [5]. Investigations on the recently proposed LOHC system based on a eutectic mixture of DPM with biphenyl show similar values for η, σ, and ρ as the system based on pure DPM [6]. In an analog manner as performed for the DPM-based system, current investigations focus on the thermophysical properties of the technically more relevant BT-based system. Since this system features position isomerism, the effect of the latter on the studied properties is particularly addressed here.

Acknowledgment

This work was funded by the Bavarian Ministry of Economic Affairs, Regional Development and Energy.

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OS3-3 K2B (Keynote) – Wednesday, 13 Sep. 14.30-15.00 – ROOM O1

Thermophysical properties of oxygen gas from first principles

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The second virial coefficient and the transport properties shear viscosity, thermal conductivity, and self-diffusion of low-density oxygen gas have been determined at temperatures up to 2000 K by first-principles calculations. A complete description of interactions between two ground-state triplet oxygen molecules requires three different potential energy surfaces (PESs), namely, a singlet PES, a triplet PES, and a quintet PES. We have developed new analytical formulations for these three PESs. The triplet and quintet surfaces are based on high-level quantum-chemical ab initio calculations at levels up to CCSDT(Q), whereas the singlet surface is based on interaction energies obtained from the respective triplet and quintet energies employing the Heisenberg Hamiltonian. The thermophysical properties have been derived from the PESs employing statistical thermodynamics and the kinetic theory of molecular gases. We compare the results with experimental and theoretical data from the literature.

Acknowledgment

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The fundamental role of multi-scale thermodynamics. An application to reactive media proposed as working fluids for thermodynamic cycles.

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Depending on the complexity of the thermodynamic behaviour of fluid mixtures, on the accuracy needed for their characterization, and on the -industrial or fundamental- knowledge need, the assessment of thermophysical and thermochemical fluid properties may require the complex use of multi-scale complementary approaches: experimental measurements, fluid models (i.e., equations of state), quantum chemistry, molecular Monte Carlo, molecular dynamics simulations and artificial intelligence.

A research energy engineering topic which has recently received the attention of the scientific community proposes the use of reactive fluids instead of inert ones as working fluids for thermodynamic cycles, backbone of thermal power plants, heat pumps and refrigeration systems. Such reactive fluids are characterised by reversible chemical reactions and are thus able to evolve in the two reactional directions along the thermodynamic cycle, when exposed to the modification of their temperature and pressure. Such a “reversible” evolution is driven by chemical equilibrium. A similar reactive system is the dissociation/association of dinitrogen tetroxide/nitrogen dioxide, $N_2O_4 = 2\ NO_2$, that we use in classes of Thermodynamics to introduce the concept of chemical equilibrium (see Figure 1).

Figure 1. Two ampoules containing a reactive mixture composed by N_2O_4 and NO_2, and following the reaction $N_2O_4 = 2\ NO_2$ according to chemical equilibrium. The ampoule on the left is at higher temperature and thus contains mostly NO_2, the ampoule on the right is at lower temperature and thus contains mostly N_2O_4.

It has been preliminary proved that the use of these reactive fluids enables the ground-breaking improvement of the efficiency of thermodynamic cycles. Understanding and modelling the thermodynamic behaviour of these reactive fluids is necessary, although being highly challenging. This presentation introduces such a difficulty, highlighting the importance of adopting a multi-scale and multi-approach methodology in order to assess mass and energy balances of these fluids along the thermodynamic cycle. We highlight that this research is not only focused on the use of $N_2O_4 = 2\ NO_2$, which is toxic, but on newly designed molecules, which similarly require a complete characterization. The methodology introduced hereinafter, focussed on $N_2O_4 = 2\ NO_2$, can be applied to other reactive systems that are being proposed as reactive working fluids for thermodynamic cycles.

More specifically, to determine the enthalpy of a reactive mixture, modelled as a real fluid, there is the need of knowing, for each molecule forming the mixture, the specific heat capacity in its ideal gas state, the standard enthalpy of formation and, to finally apply a cubic equation of state, the acentric factor, the critical temperature and pressure. The problem is that pure N_2O_4 and NO_2 are not experimentally accessible at any temperature and pressure since N_2O_4 and NO_2 always coexist, with a composition dictated by chemical equilibrium. Reliable modelling approaches are thus required to determine all these properties on the one side, while experimental data are needed on the other, to enable the optimisation of the mixing rule of the considered equation of state. For N_2O_4 and NO_2 the appeal to Quantum Chemistry is discussed to calculate enthalpies of formation and specific heat capacities, Monte Carlo simulations are presented as necessary tools to calculate critical properties and acentric factors. The application of equations of state for the characterization of these fluids is presented and their optimisation on vapour-liquid equilibrium thermodynamic data available for the reactive mixture is discussed.

Acknowledgment

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OS3-3 K2D (Keynote) – Wednesday, 13 Sep. 14.30-15.00 – ROOM N1

Simultaneous density and thermal conductivity depth profile reconstructions from noised thermal-wave amplitude and phase data using a combined integral-equation and imperialist competitive algorithm method

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An efficient new thermal-wave inverse-problem approach based on an integral-equation boundary-value method coupled with an imperialist competitive algorithm (ICA) was developed. The methodology was successfully applied to simultaneously reconstruct density and thermal conductivity depth profiles in a sintered powder metallurgy sample from an industrial automotive manufacturer with inhomogeneous density depth profile and a surface layer of higher density than the bulk. The density depth profile was validated independently using the manufacturer's data and in-house porosity measurements. The present non-destructive inverse problem approach represents a generalized formalism to thermal-wave reconstruction optimization of dual depth profiles using frequency scan data measured from the interrogated surface. From a fundamental viewpoint, the method adds significant insights into the relationship between thermal conductivity and density distributions in inhomogeneous solids.

OS3-3 K2E (Keynote) – Wednesday, 13 Sep. 14.30-15.00 – ROOM M2

Isothermal VLE measurements of tricosan in supercritical carbon dioxide

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Accurate phase equilibrium data (VLE, *PTxy*) of heavy hydrocarbons in the supercritical CO2 is required for simulation of the oil reservoir and enhancing oil recovery process with supercritical fluids. This study reports the isothermal VLE data of triclosan (heavy *n*-alkane) in supercritical CO_2 at two temperatures of 323 K and 343 K over the pressure range from (1.66 to 34.88) MPa. The measurements were made using a high-temperature and high-pressure VLE optical cell [1]. The combined expanded uncertainty of the temperature, pressure, and concentration measurements at 0.95 confidence level with a coverage factor of $k = 2$ is estimated to be 0.15 K, 0.0022, and 0.035, respectively. The measured isothermal VLE data for the mixture at selected temperature are presented in Fig. 1. The measured VLE data has been used to determine the critical parameters (T_C, P_C, xx_{CC}) of the binary tricosan+CO_2 mixture. The derived critical properties data of the binary tricosan+CO_2 mixture are: T_C=343 K, P_C=38.84 MPa, $xx_{CC} = 0.071$ mole fraction of triclosan. The measured critical curve data for the mixture has been used to study the mixture- and pure-like behavior of the thermodynamic properties of the mixture near the critical point of pure solvent (CO_2) based on isomorphism principle of criticality in binary mixture. The Krichevskii parameter of the mixture, where CO_2 is the critical solvent, has been calculated based on the critical curve data.

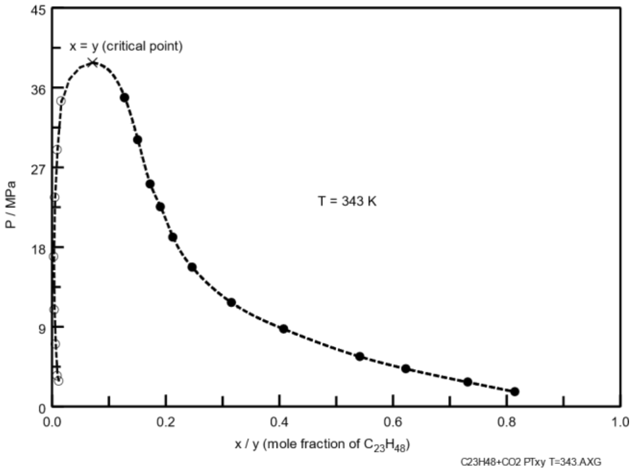

Fig. 1. Measured VLE data of tricosan+CO2 binary mixtures at selected isotherm of 343 K.

Acknowledgment

The study was supported by a grant from the Russian Science Foundation, Project number № 22-7910129, https://rscf.ru/en/project/22-79-10129/.

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OS3-3 L1A – Wednesday, 13 Sep. 15.00-15.20 – ROOM O2

Modelling of Boil-off From LNG and Liquid Hydrogen

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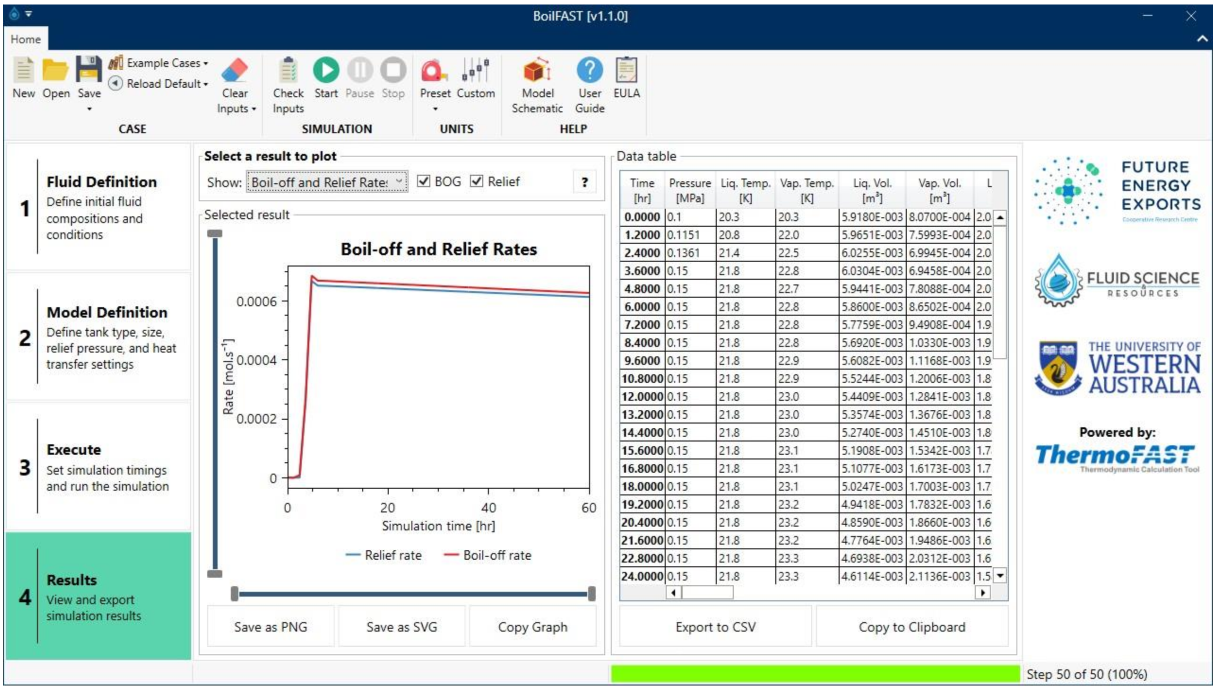
Increased trade in Liquefied Natural Gas (LNG) and growing interest in liquid hydrogen (LH2) as an environmentally friendly alternative underscore the need for reliable predictions of the boil-off gas (BOG) production which occurs when storing and transporting these two fuels. This has both operational and economic consequences, due to the associated loss of liquid product, and can have significant safety implications, should a rollover event occur and release significant quantities of BOG. To this end, a new software application, BoilFAST, has been developed. BoilFAST can simulate self-pressurisation in, and BOG production from, various LNG mixtures and LH2 for a range of tank geometries and thermal insulation systems. Currently, BoilFAST uses a non-equilibrium superheated vapour (SHV) model that treats the vapour and liquid phases separately, accounts for heat transfer from the ullage into the liquid, and uses highly-accurate Helmholtz equation of state models for thermodynamic property and flash calculations. The SHV model has been validated by testing against experimental and industrially relevant data for LNG and LH2, exhibiting excellent agreement with this data across a range of simulated conditions. BoilFAST is being extended to include simulation of thermal auto stratification and prediction of rollover events in LNG and LH2 mixtures.



Show: Boil-off and Relief Rates ☑ BOG ☑ Relief

Time [hr]	Pressure [MPa]	Liq. Temp. [K]	Vap. Temp. [K]	Liq. Vol. [m³]	Vap. Vol. [m³]	L
0.0000	0.1	20.3	20.3	5.9180E-003	8.0700E-004	2.0
1.2000	0.1151	20.8	22.0	5.9651E-003	7.5993E-004	2.0
2.4000	0.1361	21.4	22.5	6.0255E-003	6.9945E-004	2.0
3.6000	0.15	21.8	22.8	6.0304E-003	6.9458E-004	2.0
4.8000	0.15	21.8	22.7	5.9441E-003	7.8088E-004	2.0
6.0000	0.15	21.8	22.8	5.8600E-003	8.6502E-004	2.0
7.2000	0.15	21.8	22.8	5.7759E-003	9.4908E-004	1.9
8.4000	0.15	21.8	22.8	5.6920E-003	1.0330E-003	1.9
9.6000	0.15	21.8	22.9	5.6082E-003	1.1168E-003	1.9
10.8000	0.15	21.8	22.9	5.5244E-003	1.2006E-003	1.8
12.0000	0.15	21.8	23.0	5.4409E-003	1.2841E-003	1.8
13.2000	0.15	21.8	23.0	5.3574E-003	1.3676E-003	1.8
14.4000	0.15	21.8	23.0	5.2740E-003	1.4510E-003	1.8
15.6000	0.15	21.8	23.1	5.1908E-003	1.5342E-003	1.7
16.8000	0.15	21.8	23.1	5.1077E-003	1.6173E-003	1.7
18.0000	0.15	21.8	23.1	5.0247E-003	1.7003E-003	1.7
19.2000	0.15	21.8	23.2	4.9418E-003	1.7832E-003	1.6
20.4000	0.15	21.8	23.2	4.8590E-003	1.8660E-003	1.6
21.6000	0.15	21.8	23.2	4.7764E-003	1.9486E-003	1.6
22.8000	0.15	21.8	23.3	4.6938E-003	2.0312E-003	1.6
24.0000	0.15	21.8	23.3	4.6114E-003	2.1136E-003	1.5

Step 50 of 50 (100%)

Fig. 1. A screenshot of the BoilFAST software showing a BOG profile for LH2.

Acknowledgment

This work was supported by the Future Energy Exports Cooperative Research Centre, whose activities are funded by the Australian Government's Cooperative Research Centre Program.

OS3-3 L1B – Wednesday, 13 Sep. 15.00-15.20 – ROOM O1

Molecular Insight on the Properties of Ionic Liquid + Water Mixtures: From Experiment to Modelling

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Chemical process design need, for the sake of quality of investment, operation and safety, accurate values of the thermophysical (and toxicological) properties of liquids. Ionic liquids as possible replacements of currently chemicals for which European and International agencies have mandatory requirements, in actual and future chemical processes, generate a plethora of mixtures, that need an effort to devise reliable prediction and estimation methods, in the impossibility of measuring all properties of all systems known [1,2]. Water, as the main chemical engineering fluid, is a natural choice, but its use alone is not sufficient. In our laboratory a systematic study of the properties of [C_2mim][X] ionic liquids mixtures with water, by measuring properties that can give a molecular insight on the structure of the mixtures, combined with molecular simulation and spectroscopic information. It is the purpose of this contribution to report our findings, for ionic liquids where X = CH_3COO^-, $CH_3SO_3^-$ and $N(CN)_2^-$, for properties like density, speed of sound, viscosity, electric conductivity, thermal conductivity, infinite dilution diffusion coefficients and refraction index [3-5] and discuss the possible structure of their aqueous systems, in all range of compositions, schematically presented in Figure 1 for the [C_2mim][CH_3SO_3] + water mixture [4], extended to all systems.

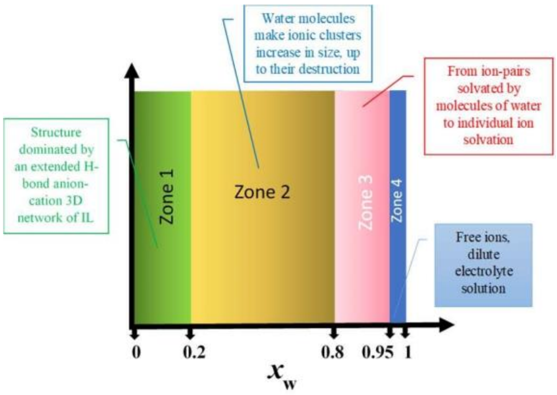

Fig. 1. Schematic representation for the different structural zones of the [C_2mim][CH_3SO_3] + water mixture. Scale in mole fraction is not proportional.

Acknowledgment

This work was partially supported by Centro de Química Estrutural - UID/QUI/00100/2013, UID/QUI/00100/2019, and UIDB/00100/2020, funded by FCT – Fundação para a Ciência e Tecnologia, Portugal. The author thanks all his collaborators contribution to the related publications.

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OS3-3 L1C – Wednesday, 13 Sep. 15.00-15.20 – ROOM N2

MD simulations and excess entropy scaling for the transport properties of dense HFEs

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Low GWP HydroFluoroEthers (HFEs) are appropriate for electronic cooling and have the potential to be used in the air-conditioning systems [1]. However, their transport properties need to be examined in the dense fluid region. Therefore, we report, validate, and test quantum mechanically-derived force fields and we used equilibrium and non-equilibrium MD simulations for the transport properties. The fractional Stokes-Einstein (SE) relation was tested to scale the predicted self-diffusion coefficient *vs* viscosity in the form of $(DT^{-1}) \propto (1/\eta)^s$, with s ranges between ≈ 0.89 and 0.92 for the HFE-7000 to -7500 in the reduced density range of $\rho\sigma^3$=0.56-0.75. The excess entropy scaling of the transport coefficients were performed via the formula $\Gamma/\Gamma^0 = \exp(aS^{ex}/kT)$, with Γ^0 being the dilute term, for the saturated and dense fluid properties. The formulation was tested for different HFOs, HFCs, and HFEs. The saturation entropies were used for the saturated properties predictions.

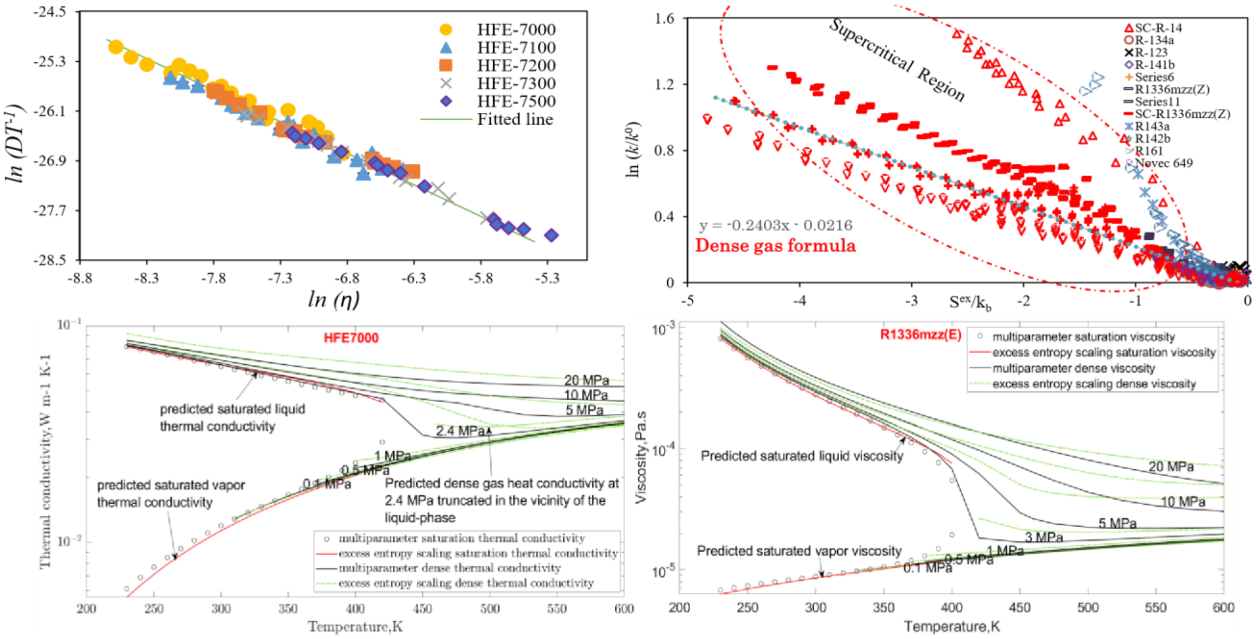

Fig. 1. Upper left corner: superposition of the translational self-diffusion vs viscosity in the fractional SE plot for these molecular family. Upper right corner: entropy scaling for the thermal conductivity of several refrigerants in the dense gas phase. Two lower plots: viscosity and thermal conductivity predictions compared to the multiparameters EoS [2-5].

Three general entropy scaling formula were developed for saturated and dense phases for the dynamic viscosity (η) of several refrigerants, but the three heat conductivity (k) formulas need to be substance-specific valid for the saturated and dense phases. Based on the comparisons with the experimental data (partly are mentioned in the REFPROP v.10), an estimated average error of 15% for the dense liquid and 37% for the dense gas included supercritical (SC) region of heat conductivity, while 19% for the dense liquid viscosity, 22% for the dense gas viscosity included SC fluid, and 45% up to supercritical region for the saturated viscosity were obtained.

Acknowledgment

This work was supported by the Czech Science Foundation Grant No. GA22-03380S and the institutional support RVO:61388998.

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OS3-3 L1D – Wednesday, 13 Sep. 15.00-15.20 – ROOM N1

Thermophysical properties of 3D-printed ceramics during binder burnout and sintering

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In the frame of this research, various thermophysical properties testing techniques have been employed to investigate the binder burnout and sintering of 3D-printed ceramic green bodies as used for medical implants or in special aerospace applications. Generative manufacture of such components is consecutively gaining preponderance as it allows for more efficient production of both, prototypes and small series, when compared to alternative or traditional ceramic manufacturing processes such as pressing or injection molding. Yet, dimensional accuracy, mechanical performance, and reproducibility of 3D-printed ceramic components meet or even exceed those being produced conventionally.

The samples examined in this work, were provided by Lithoz GmbH, Vienna, Austria, leading manufacturer of lithographic 3D printers for highly sophisticated ceramic components. Lithography-based ceramic 3D manufacturing (LCM) describes a process where an emulsion, consisting of a ceramic powder and UV-curing liquid binder, is exposed to light, layer by layer, resulting in a three-dimensional ceramic green body. Subsequently, this green body is subjected to a specific heat-treatment with the aim of burning out the organic binder (up to ~500°C) and sintering (up to ~1600°C), in order to obtain the final ceramic component.

Alumina green bodies, containing ~21% of organic binder (UV-cured organic), as assessed via Thermogravimetry (TGA), have been further investigated to gain insights into the development of thermophysical properties in the course of binder burnout and sintering. Therefore, Simultaneous Thermal Analysis (STA) was employed to assess the energetic effects throughout the decomposition of the organic binder. Moreover, Dilatometer (DIL) measurements were carried out both, in and normal to the samples' printing direction, to gain insights into the shrinking behavior throughout binder burnout and sintering. These results were further used to calculate the density change by considering the mass change from TG, and, subsequently, to correct thermal diffusivity results achieved via Laser Flash Analysis (LFA) for the sample thickness. Via Differential Scanning Calorimetry (DSC), the specific heat capacity was determined prior to and after the binder burnout. This collective data eventually allows for calculation of the thermal conductivity, being a product of thermal diffusivity, specific heat capacity and sample density. All these properties impact the binder removal and sintering of the 3D-printed Alumina green bodies. The results on the sintered material show typical values for fine grained dense Alumina. Furthermore, they are in line with the theoretical expectations of a ceramic material, in which the phonon contribution to the heat transfer plays the dominant role. The measured data helps to understand the changes in the material during heat treatment and allows for optimization of the temperature profile during debinding and sintering [1].

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OS3-3 L1E – Wednesday, 13 Sep. 15.00-15.20 – ROOM M2

An instrument for highly accurate density measurements of fluid mixtures including dew-point densities

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A further step towards advancing the vapor-liquid equilibrium modeling of fluid mixtures is the provision of accurate dew-point density measurements. Our previous studies [1-2] revealed the need for a more accurate measurement technique for vapor-phase density measurements that accounts for the distorting impact of sorption phenomena on solid surfaces near the dew point. Even the sophisticated two-sinker density measurement principle does not compensate for changes in the composition of mixtures due to sorption phenomena, both in the homogeneous vapor phase and in the vicinity of the dew point. Moreover, density measurements of pure fluids are also affected by adsorption in the region close to the dew line. Against this background, a novel foursinker densimeter (FSD) was designed [3], fabricated and set up. Here, the FSD will be described, and validation measurements on pure fluids and mixtures, as well as an uncertainty analysis will be presented.

This FSD combines the traditional two-sinker technique, by means of two 'density sinkers' for accurate density measurements, with two additional 'sorption sinkers', see Fig. 1. The two density sinkers have similar surface areas and are both gold-plated in order to have comparable adsorption loads. Both sinkers have the same mass but different volumes as one is made of silicon and the other of stainless steel. The large volume difference increases the resolution of density measurements even for low densities. The two sorption sinkers have much larger surface area (7.7:1) than the density sinkers, which will facilitate the detection of small amounts of adsorbed material onto the sinker's surface with relatively low uncertainty. Both sorption sinkers are made of stainless steel and are identical in construction except for different surface finishes: one is gold plated. The different surface finishes of the two sorption sinkers resemble the internal surfaces of the instrument (gold plated measuring cell, and stainless steel gas distribution line). Accurate dew-point densities can be determined based on the simultaneous measurement and correction for sorption phenomena. Furthermore, in conjunction with molecular dynamics simulations, which help interpreting the experimental results, it will be possible to estimate and correct for selective adsorption of gas mixtures. Densimeter that utilize a magneticsuspension coupling usually suffer from a systematic force transmission error (FTE), which increases the total measurement uncertainty. However, a major advancement of the new instrument is that all four sinkers can be weighed independently in the same coupling position, which largely cancels the FTE. The apparatus utilizes two Standard Platinum Resistance Thermometers (SPRTs) and three pressure transducers to measure the temperature and the pressure in different ranges with low uncertainty. Measurements will cover temperatures from (190 to 470) K at pressures to 15 MPa.

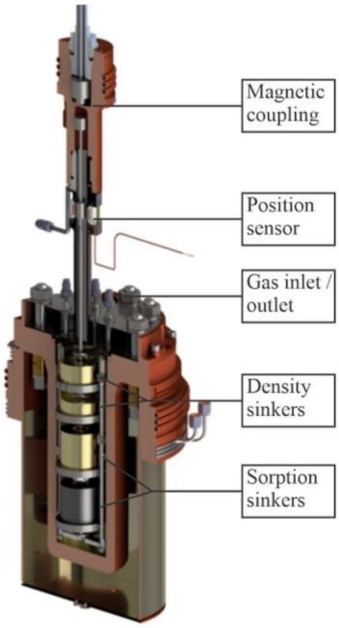

Fig.1. Cut-away of the FSD's measuring cell. The three gold-plated sinkers are near the top of the cell

Acknowledgment

This work is funded by the Deutsche Forschungsgemeinschaft (DFG, German Research Foundation) – Project Numbers 269357610 and 459051105.

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OS3-3 L2A – Wednesday, 13 Sep. 15.20-15.40 – ROOM O2

Effective extraction of benzene from benzene-*n*-hexane mixtures using polyols-based deep eutectic solvents at different temperatures

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In the petrochemical industry, the separation of benzene and *n*-hexane is one of the most difficult processes [1]. There is no effective separation method for mixtures containing less than 20 wt% benzene. In this study, the feasibility of two different deep eutectic solvents (DESs) as novel extractants for the separation of the mixture *n*-hexane + benzene was investigated. To determine the optimal solvent for this separation, a solubility test was performed at room temperature and atmospheric pressure using a series of DESs. Both DES 1 (choline chloride:triethylene glycol, molar ratio = 1:4 and 1:8) and DES 2 (choline chloride:diethylene glycol, molar ratio = 1:4 and 1:8) were chosen as DESs for this study (DES 2). The liquid-liquid equilibria (LLE) of the ternary systems {hexane + benzene + DES1} and {hexane + benzene + DES2} were determined at temperatures range from 298.2 K to 318.2 K and atmospheric pressure. The solute distribution coefficient and selectivity values for the studied ternary systems were calculated and compared to LLE data available in the literature. In addition, the experimental LLE data were successfully correlated using the nonrandom two-liquid (NRTL) model [2]. The obtained results indicate that DESs are promising extractants for the industrial separation of low concentration naphta streams. Moreover, the new binary interaction parameters (BIPs) could be widely applied for optimizing process design in the petrochemical industry process.

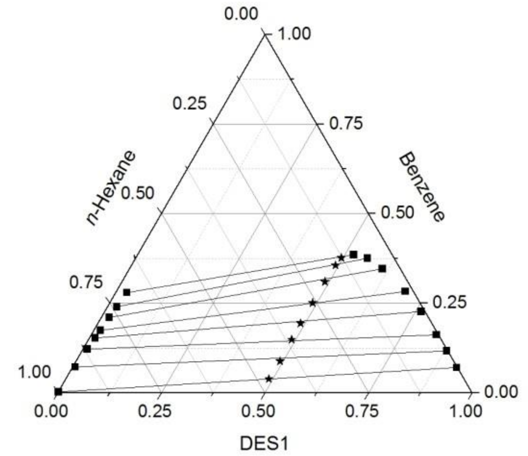

Figure 1. Experimental LLE tie-lines data for studied mixture of n-hexane + benzene + DES1 at 318.15 K

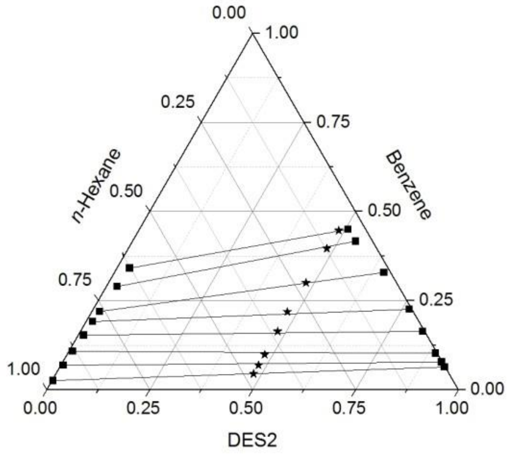

Figure 2. Experimental LLE tie-lines data for studied mixture of n-hexane + benzene + DES2 at 318.15 K

Acknowledgment

We gratefully acknowledged financial support from Ministry of Science and Technology (MOST), Taiwan, through grant MOST-111–2221-E-027–002.

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OS3-3 L2B – Wednesday, 13 Sep. 15.20-15.40 – ROOM O1

Viscosity of Methyl and Ethyl Esters: Experiments and modeling

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Saturated methyl and ethyl esters are naturally occurring chemicals that are widely used in industry as additives in the food industry, as environmentally friendly, non-toxic organic solvents, as well as ingredients in synthesizing surfactants in the chemical industry, and as raw materials in the pharmaceutical industry. They are also an important component of mixtures of fatty acid esters, present in biodiesel.

In this work we report on the measurements of viscosity and density for a number of ethyl esters (ethyl ethanoate, ethyl butanoate, ethyl dodecanoate and ethyl tetradecanoate), as well as their mixtures (four binary mixtures, one ternary and one equimolar, quaternary mixture) over the whole range of compositions in the temperature range 288.15 - 323.15 K. The viscosity was measured with a Stabinger viscometer, whilst the density was measured by means of a vibrating U-tube densimeter. The combined expanded uncertainty ($k \approx 2$) for the density is 1.7 $\mathrm{kg.m^{-3}}$, and relative one for the viscosity is 0.014.

The measured viscosity of pure ethyl esters, together with the published literature values, were correlated by means of the extended hard-sphere (EHS) model that has its basis in kinetic theory and the molecular description of the fluid. A separate correlation was developed for methyl esters, based only on the published viscosity values. The correlations, for both families of esters, are valid in the temperature range 283 to 373 K and pressures up to 200 MPa, for all the esters up to and including methyl tetracosanotae and ethyl eicosanoate. The EHS model was used to predict the viscosity of mixture of esters. The measured viscosity data were represented with the average absolute deviation of 2.5% and a maximum deviation of 6.4%.

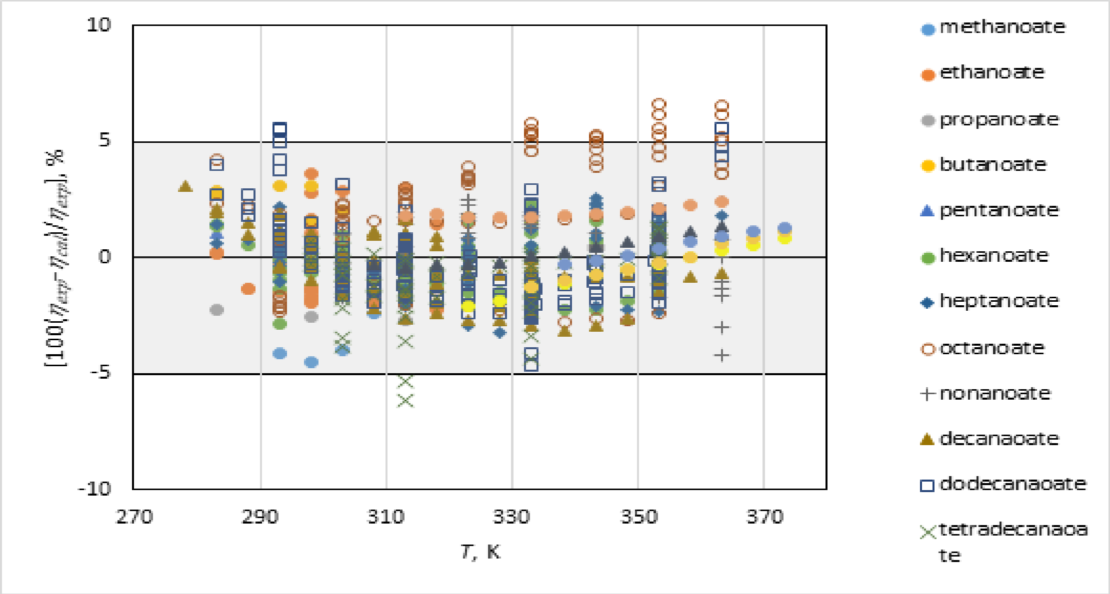

Fig. 1. Percentage deviation of the experimental data for a number of selected methyl esters from the calculated values, by means of the EHS model, as a function of temperature.

Acknowledgment

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OS3-3 L2C – Wednesday, 13 Sep. 15.20-15.40 – ROOM N2

Prediction on the solubility of Ionic Liquid with HFC by Graph Neuron Network

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Absorption refrigeration is a new research hotspot because it can be driven by low-grade heat, such as industrial waste heat and solar, geothermal energy.[1] The working pair, which including a refrigerant and an absorber, determines the cooling efficiency and the applicability of absorption refrigeration system. Hydrofluorocarbon (HFC) + ionic liquid (IL) is a new type of working pairs developed for absorption refrigeration system. Aiming to provide a guide for screening the optimal one from many candidates, a model based on graph neuron network (GNN) is presented to estimate the solubility of HFC in ILs from molecular structure. Considering the atoms as nodes and bonds as edges, the cations, anions of ionic liquids and HFCs can be transformed to their graph representation and input of the model. A dataset containing 1397 solubility data for 11 HFCs in ILs consisting of 10 cations and 13 anions at temperature from 273.13 K to 413.30 K and pressure from 0.99 kPa to 51640 kPa were established to train the model. The GNN model has great solubility regression ability indicated by the average absolute deviation of 0.003 from experimental data and has similar accuracy to group contribution method.[2] Nevertheless, because the smallest unit of the molecules are atoms but not groups, our GNN model can predict a wider range of working pairs structures. The prediction results show that [P6,6,6,14] [MeSO$_4$] has high solubility to HFC, especially to R152a, R161 and R1141.

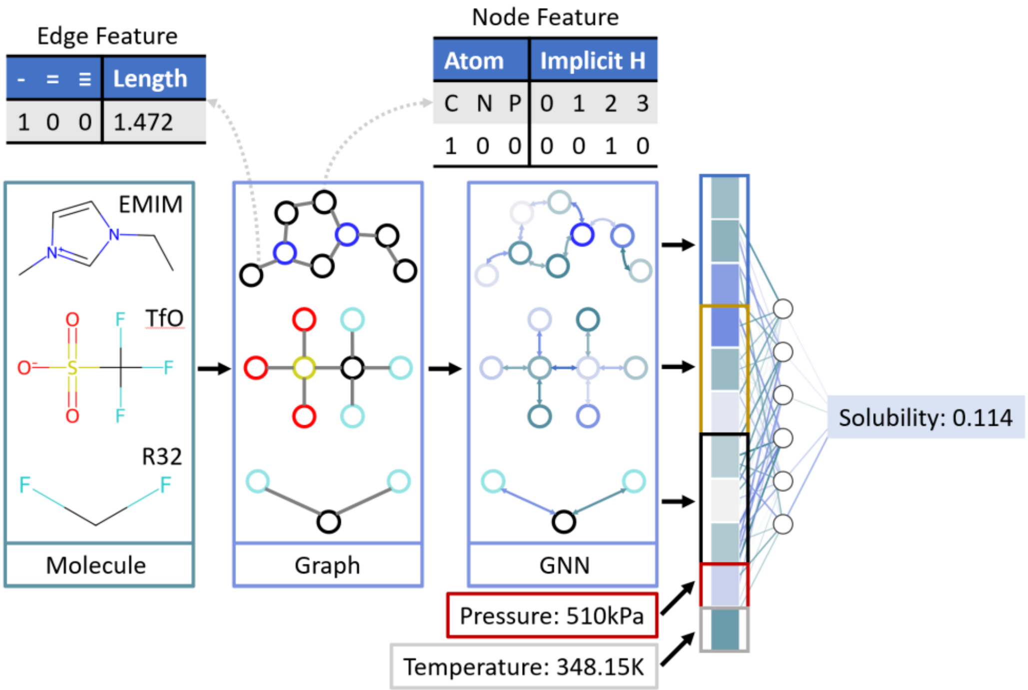

Fig. 1. 1. The workflow of the solubility prediction model.

Acknowledgment

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High temperature measurement for laser powder bed fusion

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Laser powder bed fusion (LPBF) is a popular additive manufacturing technique used to produce complex parts by selectively melting and fusing metal powder using a laser beam. However, the high-energy laser used in LPBF generates a large amount of heat that can cause thermal stress and distortion in the printed parts, affecting their mechanical properties and dimensional accuracy. Thermography can help identify these issues and improve the quality of LPBF parts. It can be used to monitor the temperature distribution of the powder bed, the melt pool, and the surrounding substrate as well as temperature gradients along the scan track. By analysing the thermal data, researchers can gain insights into the heat transfer and thermal behaviour of the system, which can help to optimize process parameters and to reduce defects in the printed parts.

However, the characteristics of the process introduce difficulties in terms of measuring absolute temperatures. Especially the high process temperatures, the rapid heating and cooling process as well as the emissivity of the material play a major role here. Therefore, proper calibration of the thermography system is important in order to ensure accurate and reliable temperature readings. The development of advanced sensing techniques that can measure temperature in real-time and at the required temporal and spatial resolution is still an active area of research in LPBF.

This contribution presents a method for measuring absolute temperatures and temperature gradients in LPBF based on the innovative Synchronised Path Infrared Thermography-approach, utilizing two synchronised scan heads. Further it gives insight into the calibration process of the thermography camera including a complex optical path for spatially independent high speed and high temperature measurements. Finally, it presents first results of in-situ measurements of absolute temperatures in the LPBF-process.

Vapor pressure measurements for very-low volatile compounds, new static apparatus and selection of standards

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Vapor pressure data for low-volatile compounds are important in many fields of human activities. From the chemical industry, through the formation of secondary organic aerosols in the troposphere, to models of the spread of so-called "non-volatile" pollutants (herbicides, polyaromatic hydrocarbons, or ionic liquids).

This work focuses on determining vapor pressures of compounds with $p^{sat} < 1$ Pa at 373 K, which is commonly performed using the Knudsen effusion method or its derivatives (quartz-crystal microbalance effusion and Knudsen effusion–mass spectroscopy) [1]. These methods are suitable for high temperatures and low pressures but are associated with larger uncertainty than the static method and may be subject to systematic errors due to uncertain molar mass of the effusing vapors or due to offset of the working steady-state from the true equilibrium state. The application of the static method under the same conditions is exacting because of the limited availability of high-temperature pressure sensors and intense desorption of light gases from vacuum manifold. Consequently, only a few static devices for low-volatile compounds were put into operation and proved to produce high-quality vapor pressure data.

Another obstacle with respect to these efforts is the unavailability of reliable reference data for the very-low pressure region. Proper calibration and testing require reference data of at least the same uncertainty as targeted, preferably over the entire pressure and temperature range of the device. So far, the least volatile vapor pressure standard is crystalline anthracene [2] for which a recommended vapor pressure equation was developed in 2019 for the region of 200 to 373 K. However, it is at least one to two orders of magnitude more volatile compared to the target compounds. The only possibility is to extend the range of available reference data step-by-step, supporting the results by the currently available standards and to establish new, even less volatile, benchmarks.

We are about to present a high-temperature static apparatus with two absolute capacitance diaphragm gauges operating in the temperature range 363 to 463 K and the pressure range 0.5 to 13000 Pa. Its performance was tested with an emphasis on the low-pressure region using five compounds with sufficiently reliable reference data: naphthalene, ferrocene, benzophenone, dibenzothiophene, and anthracene. An uncertainty of this new apparatus was proven to be $U_c\ (p^{sat}/\mathrm{Pa}) = 0.005\ (p^{sat}/\mathrm{Pa}) + 0.05$ and a reconsideration of the reference equations for anthracene and ferrocene at high temperatures was performed.

The new static apparatus was subsequently used to determine the vapor pressures of compounds targeted as new low-pressure standards: 1,3,5-triphenylbenzene, benzanthrone, and perylene. The vapor pressures of these three compounds were previously studied several times with reasonable agreement, but never using a static method. Based on the literature and new experimental data, the recommended vapor pressure equations can be derived using the simultaneous correlation method [3] that ensures thermodynamic consistency with calorimetrical sublimation enthalpies and difference in heat capacities and enables controlled extrapolation.

Acknowledgment

V. Š. and Š. K. acknowledge financial support from the Czech Science Foundation (GACR No. 23-05476M).

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An Equilibrium Approach to Modeling the Viscosity of Long Range Fluids

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Understanding the viscous behavior of complex fluids with long-range interactions is important. For short-range interactions, Chapman-Enskog theory provides an approach to calculating the viscosity based on hard-sphere systems using the Enskog equation [1].

$$\eta_{\mathrm{Enskog}} = \eta_0[g(\sigma)^{-1} + 0.8V_{\mathrm{excl}}\rho + 0.776V_{\mathrm{excl}}^2 g(\sigma)]$$

This equation can be fitted with some success to experimental data using effective parameters, such as an effective hard-sphere diameter, σ, and the Carnahan-Starling expression for estimating $g(\sigma)$ as well as the excluded volume V_{excl}. More complex versions of this have been proposed such as the Enskog-2σ model that performs quite well for simple gasses and liquids [2]. Although very useful in an experimental context for predicting the viscous behavior of such fluids, this approach offers limited theoretical insight into the underlying mechanisms. We generalize an analytical approach previously used for polar fluids [3] to estimate $g(\sigma)$ and link this property to the equilibrium properties of the fluid derived from the Helmholtz free energy. We show, by comparing our predictions to numerical simulations, that this approach performs well for a range of densities, for example for a Lennard-Jones fluid [4]. We investigate its limitations by testing other interaction potentials, including anisotropic potentials. We discuss our results also in the context of excess entropy scaling.

Acknowledgment

This work has been supported by the Research Council of Norway through its Centers of Excellence funding scheme, project number 262644, and FRIPRO project number 275507, as well as the National Infrastructure for Computational Science in Norway (UNINETT Sigma2), project number NN9573K.

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OS3-3 L3C – Wednesday, 13 Sep. 15.40-16.00 – ROOM N2

A Patel-Teja equation of state for R-1224yd(Z) refrigerant

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Despite the limited accuracy, cubic equations are widely adopted in chemical engineering thanks to their simplicityand acceptable accuracy when used for designing thermodynamic machines. In this work, a Patel-Teja equation ofstate is proposed for modeling thermodynamic properties of cis-1-chloro-2,3,3,3-tetrafluoro-1-propene, also known asR-1224yd(Z). Experimental measurements of vapor pressure and compressed liquid densities have been used to fit theequation parameters and specific heat capacities have been calculated using the equation and the ideal gas specificheat capacities. Differently, speed of sound is obtained using the expression derived from the equation of state, butfitting independent parameters obtained from compressed liquid speed of sound experimental measurements.Predictions of the model have been compared with available experimental measurements, showing deviations betterthan 0.5 % for vapor pressure, 2 % for compressed liquid densities up to 370 K, better than 1 % for compressed gasdensities up to 420 K, better than 2 % for speed of sound in liquid and in vapor phase and better than 5 % for isobaricspecific heat capacity.

OS3-3 L3D – Wednesday, 13 Sep. 15.40-16.00 – ROOM N1

Experimental set-up for dynamic material investigation at high temperatures

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Several applications in energy conversion, production systems and manufacturing techniques operate at very high temperatures above 1500 °C. E.g. the novel additive manufacturing (AM) systems allow unique fabrication processes by a layer to layer application of materials via selective melting. This melting process requires high temperatures, in particular for metallic and ceramic devices. A critical challenge in AM is the control of the induced power for melting the materials to ensure on the one side a definite melting, but preventing simultaneously an evaporation of these materials by excess temperature, which might lead to undesirable gas inclusion. Additionally, it must be ensured that the layer-to-layer process leads to a fabricated device, which ideally poses the same material properties as devices fabricated from the bulk material by other means. Also the increase of efficiency of conventional – i.e. gas turbine based – and future – i.e. fusion and solar reactors – power plants requires an increase of operation temperature, posing high requirements on the material used. Presently, the fabricated devices by AM techniques as well as the parts used in power plants are only investigated post fabrication or use by invasive methods, i.e. are destroyed for investigating their properties. Therefore, it is highly desirable to develop in-situ technologies to investigate material properties during the AM process and/or during the energy conversion process to increase efficiency and ensure on-line quality assurance.

THWS and CAE are developing innovative sensor systems to investigate high-temperature processes with high spatial and temporal resolution within their project "Experimental set-up for dynamic material investigation ExdyMa" [1]

This set-up is based on the laser flash principle and is improved by additional heating sources with different time varying possibilities to heat the investigated samples and the heat-spread is investigated with point like measuring devices or with imaging systems. By varying the wavelength of the optical heating and of the detection system as well as the time evolution of the heating, different information of opto-thermal parameters and other material properties, e.g. mechanical contact, electrical conductivity will be assessed.

Besides, the particular demand of AM and power conversion systems on temperature and material property measurements will be described and dedicated solutions to the mentioned challenges are presented.

Acknowledgement

This work is supported by the federal ministry of education and research, grant agreement number 13FH007IN6 and by the federal ministry of economic affairs and energy, grant agreement number 03ET7082 and by the Bavarian Ministry of Economic Affairs and Energy and Technology, grant number 6665e/366/2.

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A composite microwave resonator for vapour-liquid equilibrium measurement

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Cleaner forms of energy have received more publicity in recent times, as a key requirement to achieve emissions reduction targets and address climate change. These energy sources, such as natural gas and hydrogen, present new challenges in designing the most efficient and safe storage and transport processes. Engineering these systems relies on underlying thermophysical models, which are developed from fundamental experimental measurements of density, composition, and phase conditions. Fluid-mixture models are particularly sensitive to these properties at vapour-liquid equilibrium (VLE), making VLE data especially important for model development and tuning.

In this work, a new apparatus and analysis method is presented for measuring the VLE properties of binary fluid mixtures in-situ and non-invasively. The apparatus is based on a novel composite microwave resonator, designed for fast and reliable measurements of VLE composition, density, and vapour quality. The resonator supports multiple electromagnetic resonances, each with distinct spatial distributions within the cavity and thus sensitivity to the vapour and liquid phases. The resonator improves on previous microwave re-entrant resonators that have been demonstrated for VLE measurements [1-2], through optimising the distribution of sensing regions within the sample space, allowing it to achieve higher contrast between liquid and vapour properties. Furthermore, the composite design allows for a reduced sample space (less than 2 mL) and an innovative mixing capability has been implemented for faster equilibration of mixtures. The presentation will include an overview of the composite resonator apparatus conception and design, principle of operation, uncertainty analysis, and results from validation experiments. Of particular focus is the validation experiment where a binary mixture of methane and propane was compared against literature data acquired using conventional measurement techniques. The results show that this apparatus enables VLE characterization equivalent to the state-of-the-art but with the advantage of rapid data acquisition, where each VLE condition achieved equilibration and measurement of composition and density in less than two hours.

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OS3-3 L4C – Wednesday, 13 Sep. 16.00-16.20 – ROOM N2

Chemical engineers' needs and the value of research publications

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The chemical industry is a major sponsor and consumer of material and substance property research. It generates tax revenue, which contributes to public research funds. Research results are used for development and optimization of chemical production processes [1]. In the majority of cases, chemical engineers use process simulators for modeling. Simulators have specific minimum requirements to input data, sometimes depending on a process or application. In general, a full thermodynamic description equivalent to an equation of state (EOS) for the fluid plus one-atmosphere or saturation-state solid properties complemented by transport property models may be needed. The needs for mixtures usually include fluid phase equilibria expressed by EOS of activitycoefficient models, as well as solid solubilities and single-phase densities and transport properties. Interest in particular substances may be confidential and may not be disclosed, which creates challenges for research planning but also allows one to guess that any research may find a practical application.

Research projects usually address only one aspect of the full thermodynamic description. This means that in order to produce a complete thermodynamic description someone has to find, collect, and analyze the needed information from multiple sources and complement it by necessary predictions. Do this task routinely for every compound or mixture of interest makes the process extremely labor-intensive. No single provider can give all needed information, and even consolidated resources in the area hardly cover all published property data for materials and substances. A rational approach seems to be improving the existing publishing practices and establishing closer connections between the research and engineering communities. A traditional Thermodynamics Research Center (TRC) activity, cooperation with several important journals in the area, has brought a lot of experience and understanding of the situation. One conclusion that can be drawn from this activity is that the impact area needs to be wider than the journals involved in cooperation with focus on the completeness and quality of reporting. One of the efforts initiated by TRC/NIST and supported by a wide range of chemical engineers and subject experts was Good Reporting Practice, an IUPAC project, which summarized users' expectation of reporting research results [2]. Efforts to disseminate its results are being undertaken by the project participants, including its popularization and creation of resources for reviewers who can enhance the impact of the recommendations. Along with the problems needing attention, our activity has revealed good practices, which will be shown and appreciated in the presentation.

The presentation will illustrate data discovery and capture challenges and show cases of data reliability assessment and consistent property data evaluation for pure compounds and mixtures producing sets of equations suitable for engineering application. Possible ways to reconcile the interests of researchers and engineers and to further increase the impact of publicly funded research on practical applications will be discussed.

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OS3-3 L4D – Wednesday, 13 Sep. 16.00-16.20 – ROOM N1

Effect of build orientation on the thermal conductivity of Alsi10Mg samples fabricated by Selective Laser Melting

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In recent years, additive manufacturing techniques such as Selective Laser Melting (SLM) have gained attention as a promising way of producing complex metal components with excellent mechanical and thermal properties. SLM uses a high-energy laser beam to melt metal powders layer by layer, allowing the creation of structures that are impossible to obtain using traditional manufacturing techniques. Despite the benefits of this technology, the anisotropy of SLMproduced parts can have a significant effect on their thermal properties and thus it is important to investigate them, in particular the thermal conductivity. This study aims to experimentally measure the thermal conductivity of AlSi10Mg aluminium alloy cylindrical samples produced by SLM with several build orientations.

Using a Hot Disk equipment, the transient plane source method (TPS) was adopted to measure the samples' thermal conductivity. This method requires that a flat sensor is located between two specimen halves. During the tests, the sensor generates a heat flow rate while measuring the temperature rise on the specimen's active surfaces. To perform these measurements, an appropriate test time is needed to minimize the influence of the boundary conditions when using a finite-size specimen and it is necessary to know the volumetric specific heat of the material. The TPS method can also be used with anisotropic materials, if the thermal conductivity of the tested samples is the same in all the radial directions. Three sets of cylindrical samples were produced with different angles between the cylinder axis and the build direction (0°, 45° and 90°) to investigate the anisotropy of thermal conductivity. The samples had a diameter of 50 mm and a height of 16 mm, which were considerably larger than the diameter of the sensor. Therefore, the solution of the thermal conductivity equation was found with the assumption that the sensor was located in an infinitely large material, and the thermal penetration generated by the instrument did not reach the outer boundaries of the samples.

Experimental measurements were carried out at room temperature using the anisotropic module of the Hot Disk TPS 3500 instrument on all printed samples, and a negligible difference was observed between the thermal conductivity measured along the build and transverse directions. This result was further confirmed by measuring the thermal conductivity using the isotropic model of the instrument, leading to the same conclusions.

The measured values of the thermal conductivity for all samples are reported in Figure 1. The presented values are the mean of at least five repeated measurements. The highest difference between axial and radial components of the thermal conductivity was equal to 4.8% using the anisotropic model, while with the isotropic model the highest difference in the thermal conductivity of the tested samples was equal to 3.6%. In both cases, the measured difference is lower than the experimental uncertainty of the adopted instrument. Therefore, it is possible to conclude that no anisotropic behaviour was observed in the tested samples.

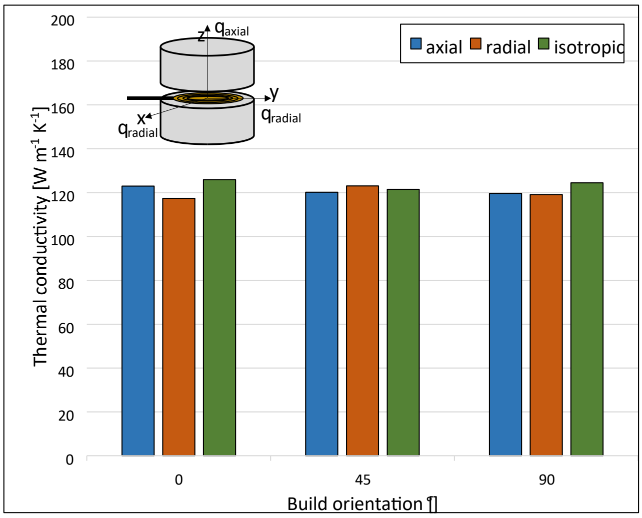

Figure. 1. Measured values of axial, radial and isotropic thermal conductivity for the tested samples.

Isothermal (vapour + liquid) equilibrium measurements and correlation of the binary mixture {3,3,3-trifluoropropene (R1243zf) + isobutane (R600a)} at temperatures from 283.15 to 323.15 K

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The F-gas Regulation and the Kigali Amendment to the Montreal Protocol introduced many constraints and led to intense research for alternatives to fluorinated greenhouse gases for air conditioning and refrigeration purposes [1]. However, a refrigerant is required to satisfy several criteria other than a low GWP and ODP values, such as good efficiency in thermodynamic cycles, non-flammability, non-toxicity, compatibility with the state of the art materials and cost [2]. In this perspective, hydrofluoroolefins (HFO) are considered interesting alternatives to hydrofluorocarbons (HFCs) and hydrochlorofluorocarbons (HCFCs) for HVAC and refrigeration applications [3]. The research for low GWP refrigerants is involving also a few hydrocarbons (HCs), such as propane (R290) and isobutane (R600a), which are not new in the market and commonly used in domestic refrigerators because of good efficiency, low charge and low price, even if they are highly flammable [4].

If HCs are well known fluids, with a large amount of experimental data and reliable Equations of State (EoS) available in literature, the same is not for HFOs and the knowledge of HFOs blends is even scarcer. [3]. In the present study, the vapor–liquid equilibrium (VLE) for the binary system {3,3,3-trifluoropropene (R1243zf) + isobutane (R600a)}, has been experimentally studied by means of a vapor recirculation apparatus. Only one set of VLE data is available in literature at the present for temperatures from 253.15 K to 293.15 K [4]. The measurements have been performed at isothermal conditions in the range of temperatures between 283.15 K and 323.15 K, while the composition of both phases in equilibrium has been measured by gas-chromatographic analysis. The data presented in this work, together with the ones available in the literature, have been used to fit a new mixture model based on Helmholtz free energy EoS type. The novel EoS shows good agreement with the experimental data.

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Transient absorption spectroscopy for evaluation of photothermal conversion in heterostructured nanoparticles

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Recently, the unique optical properties of metallic nanoparticles have attracted attention as a means to effectively utilize solar energy, a renewable energy source. Metal nanoparticles possess optical properties such as large optical absorption enhancement, spectral shift, and surface electromagnetic field enhancement at specific wavelengths of light through localized surface plasmon resonance (LSPR). In fact, a number of photothermal conversion devices, such as contrast agents for photothermal therapy and direct absorption solar collectors, have been proposed to effectively utilize LSPR. Iida et al. reported that the electronic dynamics in this photothermal conversion process can be modified by heterojunction of semiconductors with noble metals [1]. However, it is still unclear how this change in electronic dynamics contributes to the photothermal conversion process. In this study, we aim to clarify the mechanism of photothermal conversion in heterostructured nanoparticle dispersions. Changes in electronic dynamics of Au nanoparticles upon electronic excitation by semiconductor heterojunction evaluated by transient absorption spectroscopy (Fig. 1). Au/TiO_2 shows a significant decrease in absorbance around 545 nm (bleaching). Figure 2 shows the bleaching relaxation process. Link et al [2] showed that electronphonon scattering is the dominant contributor to fast relaxation after electronic excitation, while phonon-phonon scattering is the dominant contributor to slow relaxation.

In this study, we show that the change in electron dynamics during the photothermal conversion process due to the noble metal-semiconductor heterostructure may contribute to the change in thermalization time due to electron-phonon scattering.

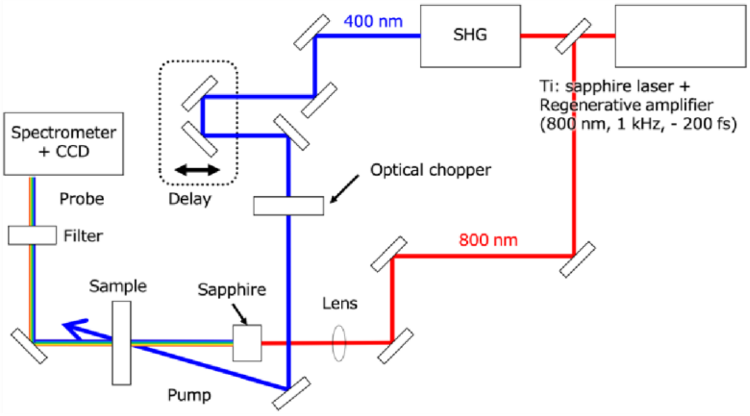

Fig. 1. TAS measurement system.

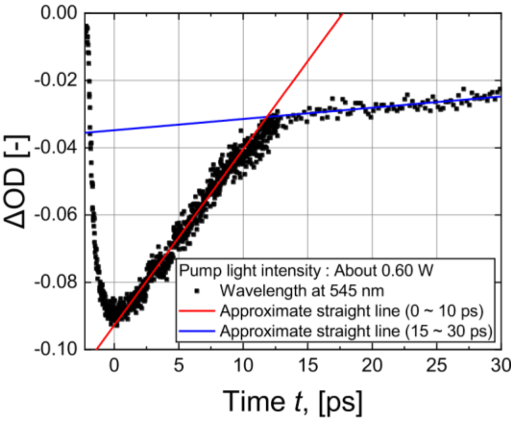

Fig. 2. Bleaching recovery of Au/TiO_2

Acknowledgment

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Measurement and prediction of speed of sound in diesel-biodiesel blends at high pressure

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In an effort to reduce harmful emissions from diesel engines, one promising approach is to improve combustion processes by increasing injection pressure. Additionally, the use of non-conventional fuels, such as diesel-biodiesel blends, may help reduce exhaust emissions without requiring significant changes to diesel engine design. Dieselbiodiesel blends are a particularly attractive alternative fuel in terms of reducing air pollution, as they offer several environmental benefits compared to petroleum diesel.

However, the actual benefits and drawbacks of using diesel-biodiesel blends vary significantly depending on the thermophysical properties of the blend. These properties are directly influenced by the temperature and pressure of engine operating conditions, as well as the proportion of diesel and biodiesel used. The fatty acid alkyl ester profile in biodiesel, which depends on the feedstock source, also plays a key role in determining the properties of diesel-biodiesel fuels. Therefore, in order to make the best possible use of biodiesel, accurate knowledge of the thermophysical properties of biodiesel is required, including its behavior with respect to temperature, pressure, diesel/biodiesel ratio, and fatty acid alkyl esters composition.

To provide data on the thermophysical properties of diesel-biodiesel blends under high pressure, we conducted measurements of the speed of sound in a petro diesel sample (B0) and three diesel-biodiesel blends (B10, B25, and B55) corresponding to the addition of 10%, 25%, and 55% (vol%) of rapeseed biodiesel, respectively, in diesel B0. Speed of sound measurements were carried out from atmospheric pressure up to 250 MPa, in the temperature range of 263 to 383 K, using a pulse technique operating at 3 MHz. The experimental results were compared to the predictions of a combining rule based on the product of the speed of sound (c) and molecular weight (M) raised to the power of gamma (γ=0.975), which has been found to be appropriate for group additivity rules in both the alkane family [1] and the fatty acid alkyl ester family [2]. As can be seen in Fig.1, a good agreement was observed between the calculated and experimental speed of sound values, with deviations never exceeding the experimental uncertainties.

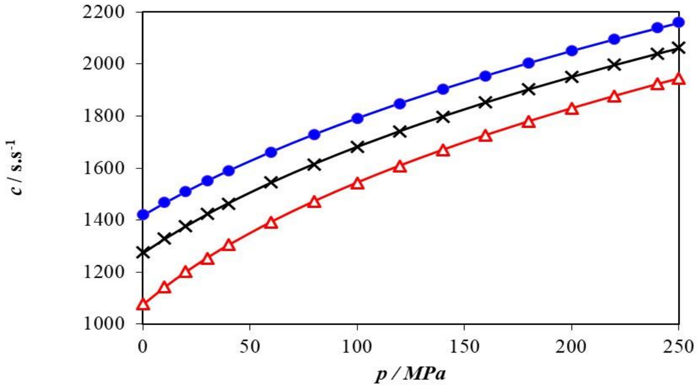

Fig. 1. Speed of sound in B55 as a function of pressure. Line: calculation; points: experimental data (□, 283.15 K; ×, 283.15 K; ●, 383.15 K).

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Phase behavior of the binary system CO_2 + indane from 293.15 K to 360.15 K: measurements and modelling

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Binary systems containing CO_2 and common hydrocarbons have been extensively measured and a large amount of data are available for such mixtures. However, a lack of data is still observed for systems containing less common compounds such as heterocyclic hydrocarbons like indane (C_9H_{10}, CAS 496-11-7). Naphthenoaromatic compounds (tetralin, indane and their alkylated components) can nonetheless be found in substantial proportion in some crude oils.

In this study, the phase behaviour of the CO_2 (1) + indane (2) binary system has been experimentally studied at temperatures ranging from 293.15 K to 360.15 K. To our knowledge, no literature data are available relating to this mixture. Measurements were performed using two experimental devices: a classical variable volume high– pressure cell allowing visual observation of phase transitions at constant overall composition and a small volume high pressure microscopy (HPM) cell.

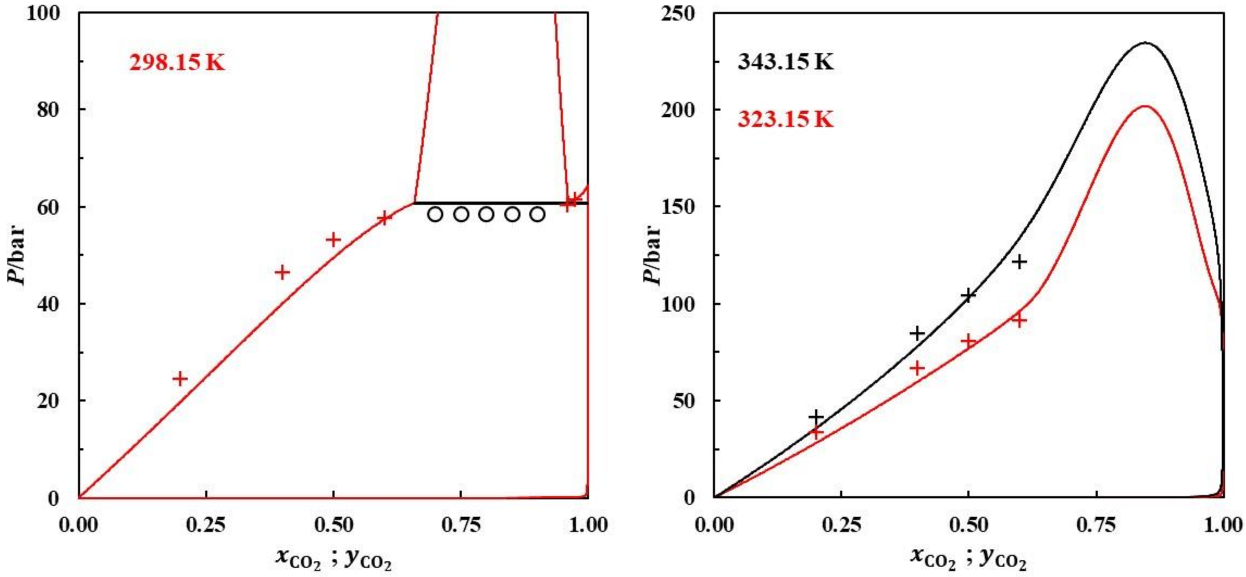

Fig. 1. Isothermal phase equilibria of the binary system CO_2 (1) + indane (2) at 298.15 K, 323.15 K and 343.15 K.
+: this work experimental bubble point; o: this work experimental point located on the three-phase line (VLLE)
Continuous curves: saturation curves calculated with the Peng-Robinson equation of state

The results obtained in this work reveal the occurrence of vapor–liquid, liquid–liquid and vapor–liquid–liquid phase transitions in the investigated temperature range. Bubble points and coordinates of the three-phase line were measured whereas the Peng-Robinson equation of state was used to calculate the phase diagram of the mixture. In some parts of the phase diagram, it was not possible to measure saturation pressures with reasonable accuracy. Indeed, in the liquid-liquid phase splitting region as well as for rich-CO_2 compositions, inspection of the fluid using the HPM cell revealed that the phase transition was not an abrupt phenomenon but rather a continuous process with, at the beginning, the appearance of hardly detectable thin droplets.

Preliminary results obtained in this study allow us to assume that the CO_2 + indane system belongs to type III binary system.

Viscosity measurement in diesel-biodiesel blends at high pressure

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Progress in fuel injection technology is crucial for the development of compression ignition engines with higher fuel efficiency and lower harmful emissions. The performance of diesel engines, in terms of fuel consumption and exhaust gas emissions, is closely related to the injection and atomization of fuel in the combustion chamber. Since the thermophysical properties of fuel play a critical role in the atomization process, it is essential to understand these properties, including volumetric acoustic and transport properties, for biofuels under high pressure in order to optimize engine performance with biodiesels in current and future injection pressure ranges.

Viscosity is a key property that can affect atomization, influencing spray pattern and droplet size distribution in the spray. Viscosity also impacts lubrication efficiency and leakage within the moving parts of the pump and injectors. Reliable data on viscosity is necessary for modeling the flow of biodiesel within the nozzle. Therefore, we have initiated a program to measure the viscosity of diesel and biodiesel samples at an extended range of pressures, up to 200 MPa.

At atmospheric pressure, viscosity was measured using a classical Ubbelohde capillary viscometer in the temperature range of 293 to 353 K. At higher pressures, a falling-body viscometer was used to measure viscosity. The samples tested included a petro-diesel sample (B0) and three diesel-biodiesel blends (B10, B25, and B55) corresponding to 10%, 25%, and 55% (vol%) of a rapeseed biodiesel in diesel (B0), respectively.

Analysis of oscillation behavior of droplet in aerodynamic levitation

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In this study, we analyzed the effect of gas jets on the oscillation behavior of levitated droplets in the aerodynamic levitation method and developed a correction formula for the measured surface tension. The oscillation behavior of droplets was analyzed by Smoothed Particle Hydrodynamics (SPH) simulation [1]. Thermophysical properties such as the viscosity and the surface tension of metals in the molten state are important for not only material science but also improving the efficiency of industrial processes. Since molten metals are generally high-temperature and highly reactiveness, it is difficult to measure thermophysical properties using conventional container-based methods, so that containerless methods such as the levitation method have been developed. In the aerodynamic levitation method, a sample is levitated by high-speed gas jet from the tip of a nozzle, thermophysical properties are measured from the shape and vibrational behavior of the levitated droplets [2,3]. Fluid pressure applied to the floating droplets was calculated by CFD calculations, and the obtained results were given as external forces in the SPH calculations to analyze droplet deformation behavior. As a result, simulation analysis shows that droplets levitated by the gas jet were affected by the fluid pressure, resulting in a smaller vibration frequency. This results suggests that the surface tension values measured by the aerodynamic levitation method may need to be corrected. From Young-Laplace's equation, surface tension is caused by the pressure difference between the two-phase interface, and the relationship between the pressure difference and the curvature of the interface is theoretically shown. Considering the fluid pressure obtained from the CFD calculations, the pressure difference between the two-phase interface results in a smaller surface tension than in the case without the gas jet. Comparison of the surface tension values calculated from the Young-Laplace equation with those obtained from the SPH oscillation analysis showed good agreement. This study proposes a correction method for surface tension measurements in the aerodynamic levitation method, suggesting the possibility of more accurate measurement of physical properties.

Acknowledgment

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Thermophysical properties in two supercritical fluids CO2+hydrocarbon system: study of clustering effect

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In CO_2+hydrocarbon systems, when carbon dioxide is present in excess and under supercritical conditions, solvent molecules (CO_2) tend to aggregate around solute hydrocarbon molecules, forming distinct structures called clusters. The nature and magnitude of the clustering effect depend on various factors, including the amount of solute, the asymmetry between solvent and solute components, as well as the temperature and pressure conditions of the system. The formation of clusters can significantly affect the thermophysical properties of the mixtures, such as density and viscosity, leading to unusual behaviors. For example, at infinite dilution, the partial molar volume of the solute component can reach negative values, resulting in a pressure drop when adding hydrocarbon molecules at constant volume to pure CO_2.

The clustering effect in supercritical CO_2-heavy hydrocarbon systems may have significant implications in various chemical engineering processes involving CO_2-heavy hydrocarbon mixtures, such as enhanced oil recovery by CO2 flooding, gas separation for carbon capture, and gas injection in reservoir fluid for carbon storage. It may also impact supercritical extraction processes where CO_2 in supercritical conditions is used to extract specific components from a solid or liquid carrier. Despite its importance, the clustering effect is poorly documented, with limited thermophysical measurements conducted under conditions of cluster formation, i.e., in the vicinity of the infinite dilution of the hydrocarbon component in carbon dioxide.

The objective of this work is to study the thermophysical properties of two supercritical CO_2-heavy hydrocarbon mixtures (CO_2+n-C_7 and (CO_2+n-C_7+n-C_{12}) through a combination of experiments, equation of states, and molecular simulations, with a particular focus on improving the modeling of these complex systems. A key emphasis is placed on investigating potential correlations between demarcation lines that define gas-like and liquid-like behaviors (e.g., Widom, Frenkel, and entropy) in pure supercritical fluids and the clustering effect that arises in a supercritical solvent upon addition of a small amount of solute.

Thermodynamic evaluation of solubility product of AlN in liquid Fe-Cr-Ni alloys for design of AlN solution growth method

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AlGaN-based ultraviolet light emitting diodes (UV-LED) are expected to use as an alternative for mercury lumps. Aluminum nitride (AlN) is an ideal substrate material for the UV-LEDs because of its high thermal conductivity, high transparency in the deep UV region, and good lattice matching with AlGaN. AlN also has a wide bandgap and high dielectric breakdown strength. Therefore, AlN is a promising material for highefficiency and high-breakdown voltage power devices. However, it is difficult to produce bulk AlN single crystals using melt solidification processes such as Czochralski method due to the high dissociation pressure of AlN at high temperatures. AlN single crystals are currently produced by the sublimation method. The sublimation method requires high temperatures above 2300 K, which increases the production cost.

Our group focuses on the solution growth method to grow AlN at lower temperatures than the sublimation method. To seek a suitable flux for the solution growth of AlN, it is important to know the solubility product of AlN in the flux. In this study, we focused on Fe-based alloy, in particular Fe-Cr-Ni alloy, as a flux for AlN growth. The thermodynamic data for Fe-based alloys have been reported abundantly. Based on the thermodynamic data [1], the solubility product in the flux can be evaluated. Cr and Ni in Fe increase the solubility product of AlN because the interaction coefficient of Cr on N and the interaction coefficient of Ni on Al were negative with large absolute values. In this study, the solubility product of AlN in the Fe-Cr-Ni flux was evaluated based on thermodynamic data. Fig. 1 shows the temperature dependence of the solubility product of AlN in six compositions of Fe-Cr-Ni. Based on the evaluated results, we designed the solution growth process of AlN using the Fe-Cr-Ni flux.

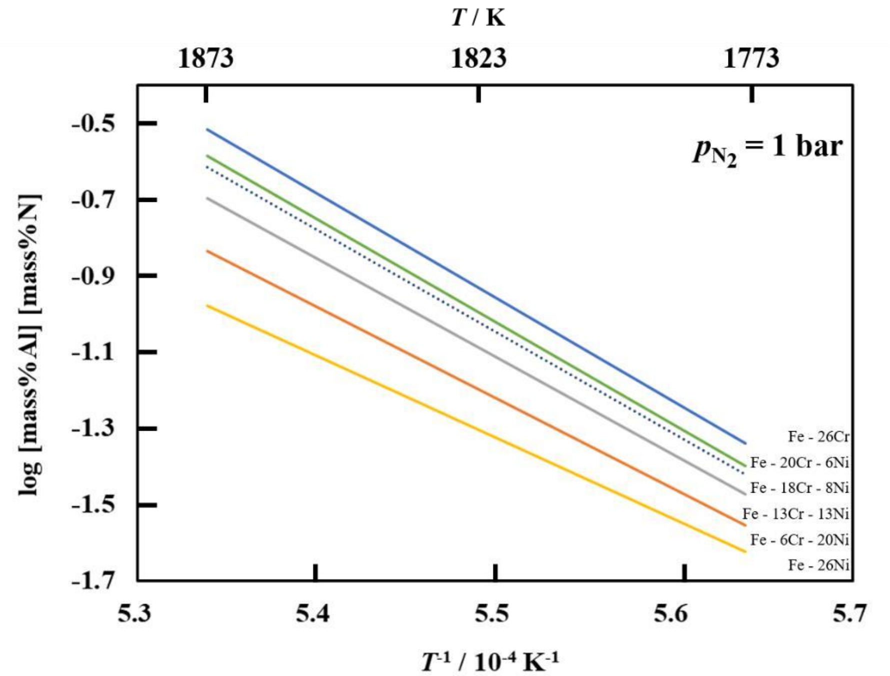

Fig. 1. Temperature dependence of AlN solubility product in Fe-Cr-Ni flux

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Thermal Transfer Properties of Porous Carbon Materials as a Function of Temperature up to 2800 °C: A Comparative Study of Selected Materials for Thermal Insulation

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Heat transfer properties, like thermal diffusivity and thermal conductivity, of porous carbon materials are important for designing and optimizing of high-temperature applications, such as thermal insulation, catalysis, and energy storage. In this study, we investigate and compare the thermal transfer properties of selected porous carbon materials from different precursers and manufacturing processes as a function of temperature up to 2800 °C.

In detail, the thermal diffusivity of carbon hard-felt, carbon xerogel, the ablative insulation material ZURAM® and non-porous graphite was determined by means of flash technique under Helium atmosphere.

Carbon xerogel[1] samples were synthesized using a sol-gel method and a drying process at ambient conditions followed by pyrolysis under Argon atmosphere. The carbon hard-felt is a commercially available product used for technical applications. ZURAM® is an ablative TPS material[2], which mainly consists of a rigid carbon preform and phenolic resin. Prior to the investigations, the material was pyrolised.

The effective thermal conductivity of all porous carbon materials increases with increasing temperature, regardless of their origin and manufacturing process. However, the thermal conductivity of the porous carbon samples also depends on their thermal history. We observed significant differences in the absolute values of the effective thermal conductivity among the different carbon materials, which can be attributed to variations in their porosity, degree of graphitization, and other structural features.

Our findings provide valuable insights into the thermal transfer behavior of porous carbon materials at high temperatures and have important implications for the selection of advanced high-temperature materials for various applications.

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Evaluation of An Acoustic Gas Thermometer for High Temperature Use

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National Metrology Institute of Japan (NMIJ, AIST) performs the measurements of thermodynamic temperature through the temperature dependence of the speed of sound in monoatomic gas using acoustic gas thermometer (AGT). It developed an AGT system that adopted a quasi-spheric resonator (QSR) fabricated using a diamondturn tool to have satisfactory finishing of its internal surface, using which the thermodynamic temperatures have been reported at temperatures below the gallium melting point [1]. To extend the thermodynamic temperature measurement to higher temperature range, a new QSR has been build.

The new QSR was a stainless steel 316L sphere with diamond-turned inner surface and three-liter volume. This QSR has ports that can be used to fix capsule type standard platinum resistance thermometers (cSPRTs) at the top, the bottom and the equator. The cSPRTs are for the use at temperatures up to indium freezing point (156.5985 °C). The QSR is set in a stainless steel 316L container, called pressure vessel, through which thermometer liners are connected to the QSR at the top, the bottom and the equator. These thermometer liners are used to place long-stem standard platinum resistance thermometers (SPRTs) for measurements at temperatures above the indium freezing point. The QSR is schematically shown in Fig. 1.

As the first step, the new QSR is evaluated for the use at the triple point of water and the gallium melting point (29.7646 °C). A liquid bath is introduced for these measurements. The evaluation results are reported in the presentation.

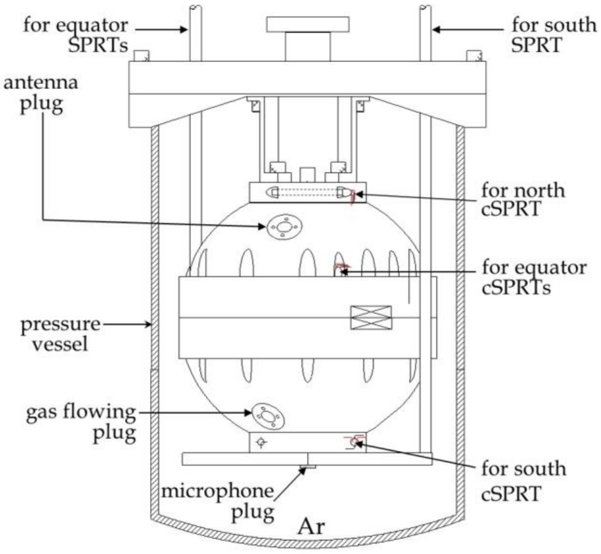

Fig. 1. Schematic diagram of the stainless-steel QSR

Acknowledgment

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Exclusion of Ohmic loss effect from dielectric loss spectra using Kramers-Kronig relations for evaluation of slow dynamics of water molecules

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Dielectric spectroscopy is known as a powerful tool to characterize the dynamics and structural properties of substances in a wide range of frequencies. However, in the measurement of conductive liquids such as biological samples, the contribution of the DC conductivity σ/ω, where ω is the angular frequency, overlaps with the dielectric loss ε'' up to GHz frequency range. This Ohmic loss effect σ/ω disturbs or sometimes prevents evaluation of dielectric relaxation of water molecules.

The Kramers-Kronig (KK) relations link the real and imaginary parts of the complex permittivity $\varepsilon^* = \varepsilon' - j\varepsilon''$ in the presence of DC conductivity σ, as expressed in Eqs. (1) and (2).

$$\varepsilon'(\omega) = \varepsilon_\infty + \frac{1}{\pi} P \int_{-\infty}^{\infty} \frac{\varepsilon''(\omega')}{\omega' - \omega} \mathrm{d}\omega' , \tag{1}$$

$$\varepsilon''(\omega) - \frac{\sigma}{\varepsilon_0 \omega} = -\frac{1}{\pi} P \int_{-\infty}^{\infty} \frac{\varepsilon'(\omega') - \varepsilon_\infty}{\omega' - \omega} \mathrm{d}\omega' , \tag{2}$$

where ε_∞ is the high-frequency limit of the complex dielectric permittivity, and ε_0 is the dielectric permittivity of a vacuum. The symbol P in Eqs. (1) and (2) denotes the Cauchy principal value of the integral. In this study, dielectric loss without the contribution of DC conductivity, $\varepsilon''(\omega) - \sigma/(\varepsilon_0\omega)$, has been calculated by the Hilbert transform of experimentally obtained real part $\varepsilon'(\omega)$ [1]. Practically, the frequency range is limited in experiments, although the KK relations involve an integration from $-\infty$ to ∞. We have circumvented this problem by the FFT of the periodized signal, which can be prepared by the extrapolation and symmetric expansion of the dielectric spectrum.

Our KK transform protocol [1] has been applied to the dielectric spectrum of aqueous lysozyme solution (3 mM, T = 300 K). Figure 1 compares the dielectric loss spectrum directly obtained by the measurement (raw data) and that calculated by the KK transform of experimentally obtained $\varepsilon'(\omega)$. Although the contribution of DC conductivity partly masked the dielectric relaxation of water in the lower frequency for the raw data of ε'', the dielectric loss spectrum without the Ohmic loss contribution has been obtained by the KK transform. Our KK transform procedure has a large practical interest for evaluation of dielectric relaxations related to the slow dynamics of water molecules distributing in sub-GHz frequency range.

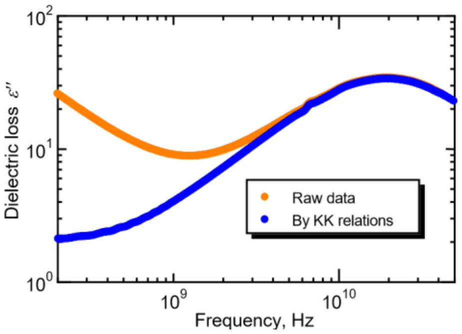

Fig. 1. Comparison of the dielectric loss spectrum directly obtained by the measurement (raw data) and that calculated by the KK transform of experimentally obtained real part of ε'' for lysozyme aqueous solution with the concentration of 3 mM at T = 300 K [1].

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Thermal conductivity change of yttrium oxyhydride thin films induced by UV light photo-reduction

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Reversible photochromic properties of yttrium oxyhydride (YHO) films have been recently reported [1]. Under UV illumination at ambient conditions, an insulating and transparent YHO film can be reduced and become a metallic YH_2 [2]. Such switchable reaction enables their utilization in not only optical and electrical applications, but also in the heat flow control by the contrast of thermal conductivities between YHO and YH_2.

YH_2 thin films with a thickness of 100 nm were deposited on synthetic quartz glass substrates and those with a 100 nm-thick Mo coat by reactive dc magnetron sputtering using a metallic Y target and mixture gases of Ar-30% H_2 in unheated conditions. The as-deposited YH_2 films were put in a desiccator shaded by a blackout curtain for 48 hours, resulting in conversion of YH_2 into YHO in the atmosphere. The photo-reduction behaviour of the YHO films under UV irradiation of 365 nm of a light emitting diode was investigated by X-ray diffraction, electrical conductivity, optical properties and thermal conductivity measurements. The YHO film was an insulator. After 2 h of the UV irradiation, the YHO film converted to YH_2 with an electrical conductivity of about 9.4×10^3 S/m. Turning off the UV irradiation, the electrical conductivity of the YH_2 film gradually decreased with time (Fig.1). The thermal conductivity between YHO and YH_2 with the UV irradiation up to 6 h was measured by the picosecond pulsed light heating thermoreflectance (Fig.2). The derived thermal conductivities of the YHO and YH_2 films were 1.2 and 1.3 W m^{-1} K^{-1}, respectively. The increase in the thermal conductivity was 0.1 W m^{-1} K^{-1}, which is in agreement with free electron's contribution in the YH_2 films estimated by the Wiedemann-Franz law.

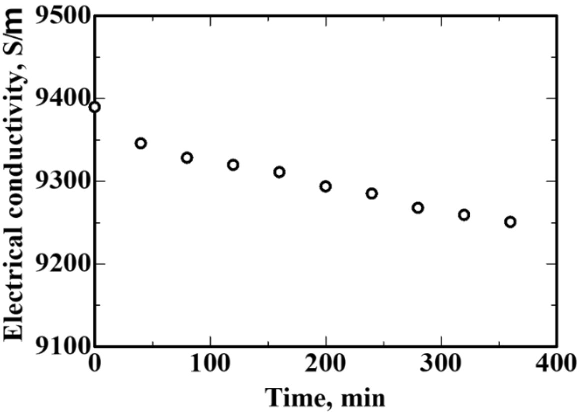

Figure 1 Electrical conductivity of the YH_2 film after 2 hours of the UV irradiation. The horizontal axis means elapsed time after the stop of the UV irradiation.

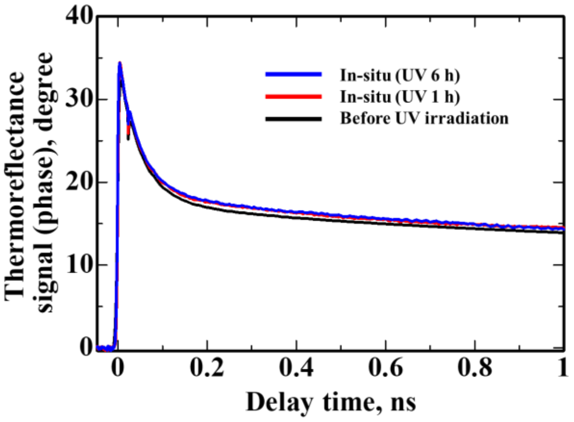

Figure 2 The thermoreflectance signals of the YHO/Mo two-layered films before and after the UV irradiation.

Acknowledgement

A part of this work was supported by JSPS Grants-in-Aid for Scientific Research (KAKENHI), Grant Number 23H01362.

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Development of time domain thermoreflectance measurement system for in-plane thermal conductivity of thin films: sensitivity analysis on two-layer film structure

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A figure of merit of thermoelectric thin films has generally been evaluated using in-plane electrical properties and out-of-plane thermal conductivity. There is an inherent inconsistency in this evaluation with respect to the measurement directions. The final goal of this study is to establish a measurement method of in-plane thermal conductivity of thin films using a nanosecond pulsed light heating time-domain thermoreflectance (TDTR) technique [1] for evaluating a true figure of merit. A concept of a proposed in-plane thermal conductivity measurement is shown in Fig. 1(a). A striped pattern area (shaded in red) on the sample surface is heated by a laser pulse, whereas a transient temperature signal (thermoreflectance signal) is detected at the space between the stripes as an intensity change of CW laser reflected on the transducer film. In this measurement method, twodimensional heat conduction across the striped patterns can be assumed as shown in Fig. 1(b). The specifications of the measurement system are a heating pulse duration of 1 ns, a heating repetition ratio of 250 kHz, stripe width, *L*, of 16 µm, and space width, *S*, of 16 µm. Mo/ZnO/R-plane sapphire substrate where Mo thickness of 100 nm was studied as a model specimen. ZnO thin films with thicknesses of 160 nm and 320 nm and inplane thermal conductivities of 10, 20, and 30 $Wm^{-1}K^{-1}$, and out-of-plane thermal conductivity of 25.8 $Wm^{1}K^{-1}$ were examined. Figure 2 shows the simulated normalized temperature for the ZnO film with thicknesses of 160 nm and 320 nm and in-plane thermal conductivities of 10 $Wm^{-1}K^{-1}$, 20 $Wm^{-1}K^{-1}$, and 30 $Wm^{-1}K^{-1}$ as a function of elapsed time from arrival of pump pulse. As a result, the temperature responses of ZnO thin films with thickness of 160 nm and 320 nm can be distinguished between 0 and 200 ns and between 0 and 800 ns, respectively. It is concluded that the proposed measurement method has a sufficient sensitivity for the inplane thermal conductivity.

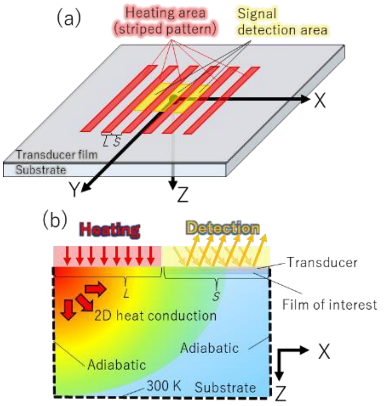

Fig. 1 schematic diagrams of (a) heating and detection area for the in-plane thermal conductivity measurement system and (b) heat conduction model and boundary conditions.

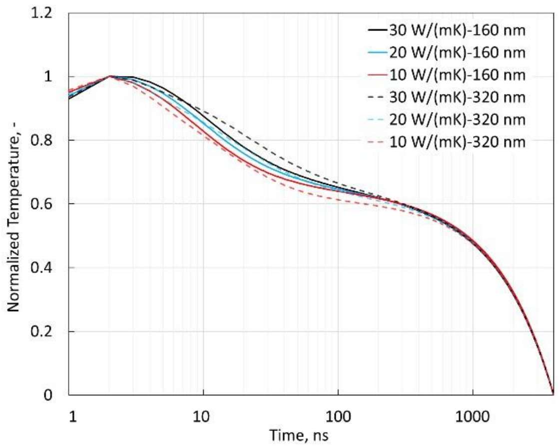

Fig. 2 simulated TDTR signals of Mo/ZnO/R-plane sapphire substrate with a striped-pattern heating on the surface.

Acknowledgments

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Density measurements of high-temperature oxide melts with electrostatic levitation furnaces onboard International Space Station and on the ground

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Investigating the properties of high-temperature liquids above around 2000 K poses significant challenges due to the inevitable chemical reactions between samples and containers, as well as the crystallizations from the containers. However, these challenges can be surmounted by using levitation techniques that prevent contamination and heterogeneous nucleation. The levitation techniques enable to precisely measure thermophysical properties of high-temperature liquids across a wide temperature range, including deep undercooling. Among several levitation techniques, our group has developed an electrostatic levitation furnace (ELF) using Columbo force and installed it in the International Space Station (ISS) to investigate metal oxides that are difficult to levitate under gravitational force due to insufficient charge accumulation on the samples [1]. To demonstrate the capability of the ISS-ELF, the thermophysical properties of molten lanthanoid sesquioxides were measured. Specifically, the densities of Ln_2O_3 (Ln =Y, Gd, Tb, Ho, Er, Tm, Yb, Lu) at their melting temperatures (T_m) were obtained over the wide temperature range (2700-3200 K) [2-4], and these densities exhibit a linear correlation with temperature. The molar volumes of these oxides at their T_m were calculated and compared with those of other sesquioxides. The volumes of the nonglass-forming sesquioxides (Ln_2O_3, Al_2O_3, and Ga_2O_3) exhibit linear correlations with the cubes of their cation radii, whereas those of the glass-forming oxides (B_2O_3, As_2O_3, and Sb_2O_3) show different correlations. Hence, molar volumes might serve as a useful indicator of a single component oxide's ability to form glass. In addition, the densities of molten Al_2O_3 - Y_2O_3 compounds were measured over wide temperature range including undercooled region. Since the compounds exhibited exceptionally large amount of charge and the high stability during heating, the measurements could be conducted with an electrostatic levitation furnace on the ground. The molar volumes of molten compounds showed a linear dependence on the concentration at superheated region. However, in the undercooled region, the molar volumes deviated from those predicted by ideal solution model due to attractive interactions between Al_2O_3 and Y_2O_3.

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Investigation by 3D FEM of the probe response for thermal conductivity measurements by SThM

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With the development of micro and nanotechnologies, traceable techniques for physical property characterization at micro and nanoscales are required. To investigate the thermal properties at these scales, the Scanning Thermal Microscopy (SThM) technique is promising [1]. In the frame of thermal conductivity (*k*) analysis, thermoresistive probes used in active mode are mainly used and a calibration of the probe is needed. Currently, the experimental calibration of SThM probes is based on the measurement of a set of bulk materials with well-known thermal conductivities. The resulting calibration curve represents a measurand linked to the probe electrical resistance variation, as a function of *k*. Another method to link the probe electrical resistance variation to k, is to develop an electrothermal model describing the heat dissipation within the probe-sampleenvironment system. Previous works have demonstrated that the simple analytical model proposed by Fischer [2] for microprobes still needs to be improved for nanoprobes [1]. In order to better reproduce the measurements with the KNT nanoprobe we have developed a 3D numerical electrothermal model of the sample-probe-environment system for the second generation of KNT probes using the finite element method (Fig. 1.a). The probe response (variation of the electrical probe resistance) in vacuum and in air out of and in contact with a sample were investigated. We find that the resistive element dimensions and room temperature (Fig. 1.b) are the most influencing model parameters on the probe response. Taking into account the contact thermal resistance *Rc* and the ballistic regime due to the nanosize of the probe–sample thermal contact in our model we also demonstrate through simulations that parameters such as *Rc* and the amorphous or crystalline nature of the sample have an impact on the *k* sensitivity range of the technique. The thermal radius of the nanocontact was also examined in order to improve the model, revealing that this thermal radius, which was assumed constant in 1D models, in fact depends on the sample thermal conductivity *k*. These results give new paths to improve the calibration methodology.

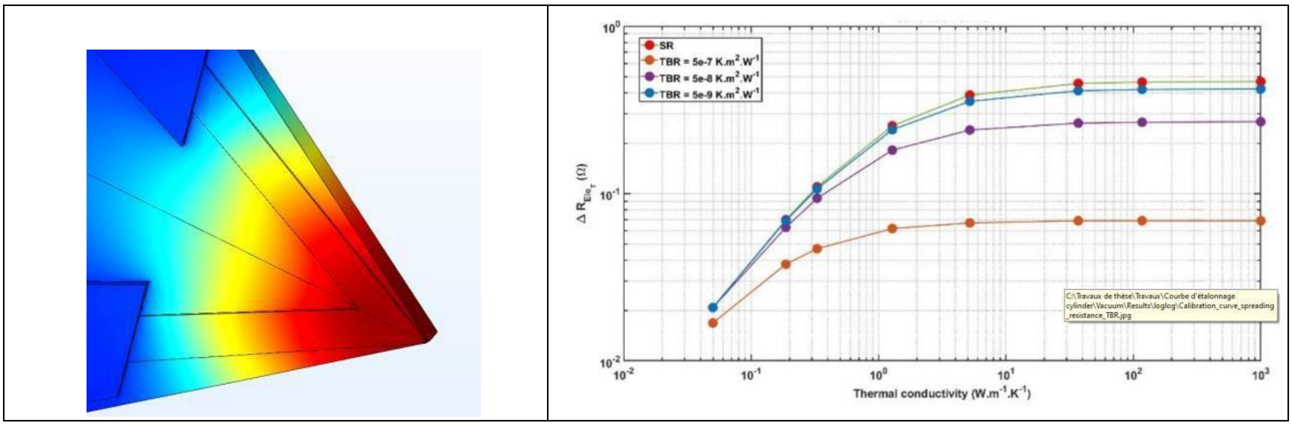

Fig. 1. Simulation with the 3D model: (a) temperature field within a KNT probe in active mode operated in vacuum and out of contact with a sample and (b) the contact thermal resistance R_c influence on the probe response.

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Thermal diffusivity determination using an atomic force microscope and modulated laser excitation

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This paper introduces a technique to measure opto-thermal parameters with sub-micrometer resolution using an atomic force microscope (AFM) and an additional laserheating to generate thermal waves. Thermal waves can be measured in various ways as shown by earlier photothermal techniques [1]. The use of an AFM as temperature probe allows the measurement of sub-micrometer properties through the sharp tip on the probe and thus small interaction areas within the sample [2]. The most common approach is Scanning Thermal Microscopy (SThM) to measure the temperature on the sample as well as the phase lag and amplitude of the thermal waves. Another way, which is planned in this study, is to use a basic AFM probe and measure the thermoelastic deformation of the sample surface caused by the temperature variation of the thermal waves. Similar to photothermal reflectance techniques the phase lag and, thus, the thermal diffusivity can be measured by either using spatial or frequency dependency [3-4]. The paper presents the theoretical aspects and used atomic force microscope. The intended experimental improvements necessary for the application of the AFM as thermal microscope as well as theoretical simulations and very first results are given.

Acknowledgment

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Evaluating silicon ring resonator as thermometer using dual-comb Spectroscopy

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Silicon has a high thermo-optic coefficient, and the resonant wavelength of its ring resonator shifts sensitively with temperature. The high thermo-optic coefficient implies that it can be used as a highly sensitive micro-scale photonic thermometer. In recent years, its potential for thermometer has been demonstrated [1], but the measurement uncertainty has been large and limited by the resonant wavelength measurements using spectrometers.
A little while ago, "Dual-Comb Spectroscopy (DCS)" using optical frequency combs which is used for laser frequency measurement has been reported [2]; and we have also developed an instrument with a relative uncertainty at the 10^{-9} level (1 fm level at 1550 nm) [3]. Using this system, more accurate evaluation of photonic thermometer is expected to be enabled. In this study, we evaluated the temperature dependent resonant wavelength of the silicon photonic thermometer by using dual-comb spectroscopy. The silicon waveguide has a 400 nm wide and 220 nm high silicon core surrounded by SiO_2 (Fig. 1). The ring diameters are 5, 10, and 20 µm, corresponding to FSRs of about 18, 9, and 4.5 nm, respectively. First, we evaluated the photonic thermometer using super luminescent diode light and optical spectrum analyzer which accuracy is approximately 10 pm of wavelength. The normalized resonance wavelengths $(T)/\lambda(T = 0\ °\mathrm{C})$ showed on a fitting function independent of the ring radius and the wavelength-temperature coefficient is 74 pm/K. The measurement uncertainty is about 150 mK which dominant factor is from the accuracy of optical spectrum analyzer. Second, the light source was switched from the super luminescent diode to a comb, where the comb was incident into and transmitted through the silicon ring resonator, and the resonator temperature was varied from 0 °C to 60 °C. The resulting wavelength-temperature coefficients are 74.4 pm/K and 74.0 pm/K for resonator #1 and #2, respectively. In addition, we observed abnormal resonance spectra, possibly due to nonlinear optical effects, with increasing power of optical frequency comb. We will report the detail of this result.

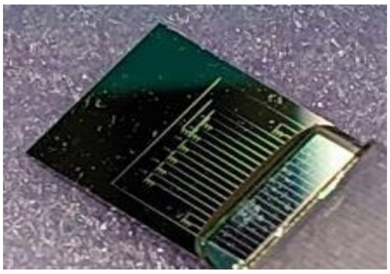

Fig. 1. Picture of the photonic thermometer chip

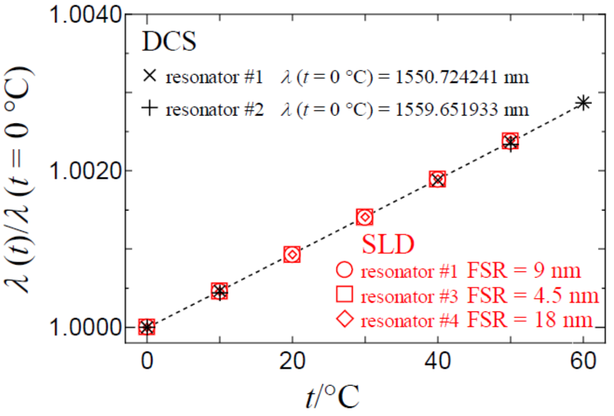

Fig. 2. Temperature dependent resonant wavelength of ring resonator evaluated by DCS (black data) and SLD (red data)

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This work was supported by Innovative Science and Technology Initiative for Security Grant Number JPJ004596, ATLA, Japan.

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Impurity effect on the triple point temperature of xenon

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The international temperature scale of 1990 (ITS-90) defines T_{90} which is close approximations to the corresponding thermodynamic temperature [1]. According to the ITS-90, T_{90} from 13.8033 K to 273.16 K is realized by using standard resistance thermometer (SPRT) calibrated by defining fixed points of the ITS-90. Recently, the investigation of the replacement of the triple point of mercury (234.3156 K), which is one of the important fixed points of the ITS-90, has been performed because of restriction of the mercury.

The triple point of xenon (161.406 K) is one of the candidates for replacing the triple point of mercury [2]. To use the triple point of xenon as fixed point for realizing temperature scale, factors that change the fixed-point temperature need to be investigated. One of the typical factors is impurity effect that cause freezing point depression. It is known that the krypton as impurity in the xenon has a strong influence on the transition temperature of xenon because krypton is similar noble gas as xenon and has a close transition temperature to xenon [2]. It is also important to check reproducibility with samples from different sources to use them as standards.

In this study, we evaluated the triple point temperature of xenon using samples of different purity supplied by different manufactures. Table. 1 shows the result of the impurity analysis of three xenon samples. The purity of samples is over 99.9997 % and the krypton concentration is 0.1 ppm, 0.4 ppm and up to 0.05 ppm in sample1, 2 and 3, respectively. Figure. 1 shows the melting curves of the triple point of xenon using different purity samples. The melting curve of sample1 with 0.4 ppm of krypton was slightly lower than that with 0.1 ppm of krypton, and temperature difference between two samples is about 0.1 mK in the range of $F < 0.4$. The difference decreased with increasing F and reached about 10 μK at $F = 1$. These results suggest that the triple point temperature of xenon has high reproducibility even if the purity of samples from different sources is over 99.9997 % and the concentration of krypton as impurity is up to 0.4 ppm. In addition, we also evaluated the influence of heat flux and non-uniqueness, and the uncertainty for the realization of the triple point of xenon. We will report the detail of this result in the presentation.

Table 1. The result of impurity analysis of three xenon samples

analyte	Sample1	Sample2	Sample3
Kr	0.1 ppm	0.4 ppm	<0.05 ppm
Ar	<0.1 ppm	<0.1 ppm	-
N_2	1.6 ppm	<0.1 ppm	<0.1 ppm
O_2	<0.1 ppm	<0.1 ppm	<0.1 ppm
H_2	<0.1 ppm	1.4 ppm	-
H_2O	<0.1 ppm	0.5 ppm	<0.1 ppm
CO	<0.1 ppm	<0.1 ppm	<0.1 ppm
CO_2	<0.1 ppm	<0.1 ppm	<0.1 ppm
purity	>99.9997 %	>99.9997 %	>99.9999 %

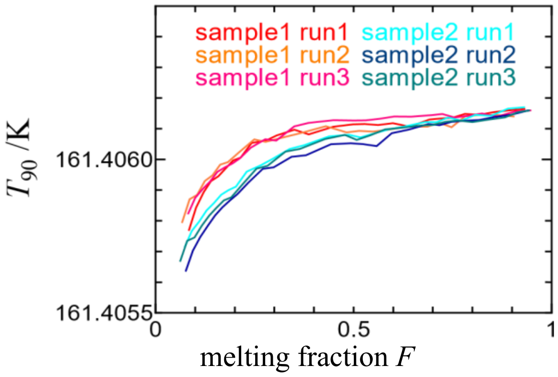

Fig. 1. Melting curves of the triple point of xenon with different samples

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An experimental method for the investigation of complex phase equilibria under high pressure. Application to CO_2 +Brazilian pre-salt crude oil systems

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The development of any petroleum field depends on the properties of the fluids produced. Once a new accumulation is discovered, the first property of the fluid to be measured is its phase behavior, with bubble point pressures being the main demand. However, there is an increasing demand for multiphasic equilibrium data, such as Liquid-Liquid Equilibria (LLE) and Liquid-Liquid-Vapor Equilibria (LLVE). This is particularly relevant for Enhanced Oil Recovery (EOR) processes, such as carbon dioxide flooding, which may result in one, two, or more phases in equilibrium, as carbon dioxide is not fully miscible with most crude oils in all circumstances. The occurrence of Liquid-Liquid Equilibria has a significant impact on the mechanisms of oil displacement and, consequently, on oil recovery efficiency. As liquid properties strongly depend on reservoir pressure, temperature, and composition, obtaining reliable LVE, LLE, and LLVE data under high pressure has a huge impact on the design of EOR projects.

However, techniques capable of assessing the complete phase behavior diagram for CO_2-crude oil mixtures are scarce. One of the main reasons is the unavailability of an experimental method capable of investigating such complex systems. To overcome this challenge, the development of an experimental setup capable of determining fluid phase transitions in opaque oils under reservoir conditions is presented here. This setup is based on the use of a new visualization system operating in the short-wave infrared range. The visualization system, which combines a high-pressure full visibility cell and a high-pressure microscopy cell, allows for direct and complete external scrutiny of fluid samples under high-pressure conditions, as well as focusing on dispersed elements of micrometer size. This reduces the study of LLE (Fig 1) and LLVE of opaque oil to the convenient investigation of transparent fluids. It enables the investigation of fluid phase equilibria, but also the detection of solid formation, such as wax precipitation or asphaltene flocculation (Fig. 2).

To demonstrate the capability of the technique, measurements were undertaken on a dead oil from a pre-salt reservoir recombined with CO_2 in various proportions. The results show a complex phase behavior with LLE observed at high temperature.

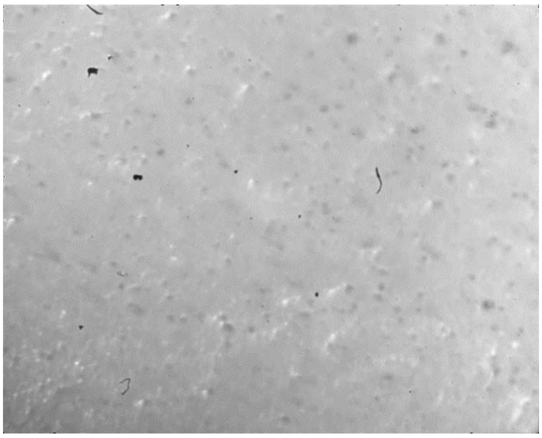

Fig. 1. Liquid-Liquid Equilibrium observed in a dark oil under high pressure with the full visibility setup equipped with a SWIR camera

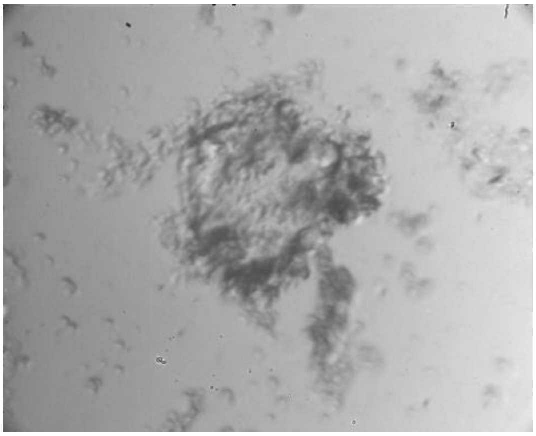

Fig. 2. Asphaltene flocs observed in a dark oil under high pressure with the HPM cell equipped with objective 50× and a SWIR camera

Speed of sound measurements for trifluoroiodomethane (R-13I1) and *cis*-1,1,1,4,4,4hexafluoro-2-butene (R-1336mzz(Z)) in the liquid phase

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Under the Kigali Amendment to the Montreal Protocol, refrigerants with low global warming potential (GWP) are widely required in the air-conditioning and refrigeration fields. Trifluoroiodomethane (R-13I1) and *cis*-1,1,1,4,4,4hexafluoro-2-butene (R-1336mzz(Z)) are both expected to be used as the low-GWP refrigerants without toxicity and flammability. It is essential to evaluate thermophysical properties of these refrigerants for accurate assessment of the thermodynamic cycle performance. The authors measured speed of sounds for R-13I1 and R-1336mzz(Z) in the liquid phase by using a commercially available speed of sound sensor, which utilizes an ultrasonic pulse propagation technique. The speed of sound data for R-13I1 were obtained in a temperature range from 280 K to 343 K and a pressure range from 310 kPa to 6850 kPa, and those for R-1336mzz(Z) were obtained in a temperature range from 283 K to 343 K and a pressure range from 40 kPa to 6550 kPa. Figure 1 shows relative deviations of the measured speed of sound data from the reference values, which are calculated from the equation of state by Lemmon and Span [1] for R-13I1 and that by McLinden and Akasaka for R-1336mzz(Z) [2]. It was found that the measured speed of sound data for R-13I1 were systematically smaller than the reference values of Lemmon and Span by typically 2.0×10^{-2}, whereas those for R-1336mzz(Z) were consistent with the reference values of McLinden and Akasaka within 0.2×10^{-2}.

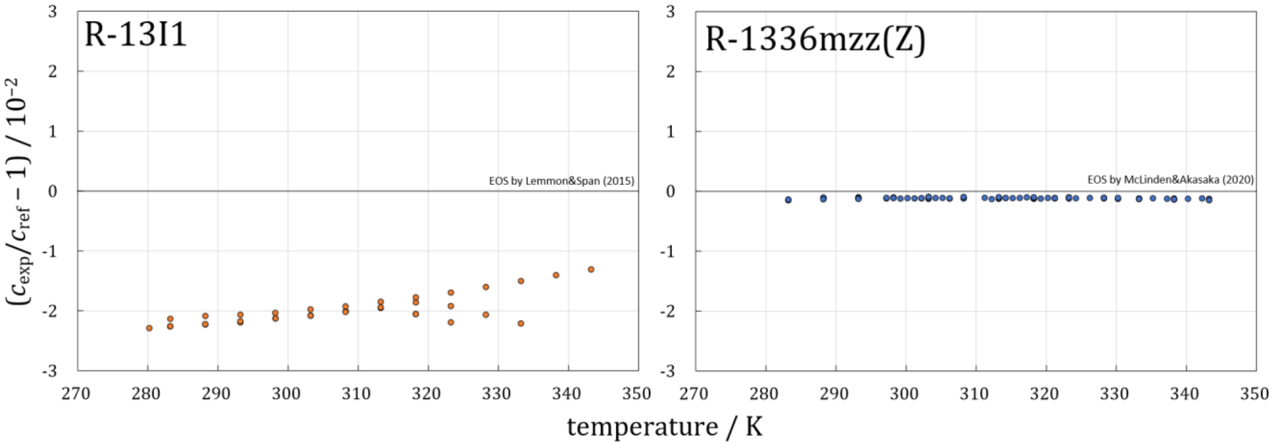

Fig. 1. Relative deviations of the measured speed of sound data for R-13I1 (left) and R-1336mzz(Z) (right) from their reference values calculated with the equations of state by Lemmon and Span [1] and McLinden and Akasaka [2].

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Density and surface tension measurement of Ti-Nb melts using electromagnetic levitation technique

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Ti-based alloys are widely used for medical implant production due to their high biocompatibility. Pure Ti has a higher Young's modulus compared with cortical bones, on the other hand, β-stabilized Ti-Nb-based alloys show low Young's modulus. Therefore, Ti-Nb-based alloys are good candidate for artificial joint material because it leads to reduction of mechanical mismatch between the elasticity of bone and implant. For manufacturing personalized artificial joints, three-dimensional printing (additive manufacturing) techniques have been expected. In the three-dimensional printing technique, large temperature gradient exists near the position of irradiated part of laser or electron beam. Thus, local high-temperature zones and deeply undercooled zones are formed in the melt pool. To improve the three-dimensional printing technique, it is necessary to understand the melting and solidification processes at the interface between the solid and the melt pool. To analyze the melting and solidification processes, accurate thermophysical properties of the three-dimensional printing materials over a wide temperature range are required. In this study, we focused on the basic binary Ti-Nb alloys, and, thermophysical properties of the Ti-Nb alloys were measured using an electromagnetic levitation technique.

Ti-15, 30, 45mass%Nb samples were prepared by arc melting with appropriate amounts of pure Ti (99.987 mass%) and Al (99.99 mass%). Ti-Nb samples were levitated under Ar-5vol.%H_2 with He atmosphere. The sample was levitated by applying a radio-frequency current to the levitation coil. A vertical static magnetic field of 3 T was applied to the levitated droplet to suppress surface oscillation and translational motion of the droplet using a superconducting magnet in density measurement [1]. In surface tension measurement, the vertical static magnetic field was not applied to observe surface oscillation and translational motion of the droplet [2]. Temperature of the sample was controlled by irradiation of heating laser and exposing the droplet to He gas flow. The sample temperature was measured using a single-color pyrometer. The temperatures were calibrated using the liquidus temperature of each alloy, and temperatures other than the liquidus temperature were determined by assuming that the normal spectral emissivity of the sample has no temperature dependence at the pyrometer wavelength (1.6 μm).

Fig. 1 shows the temperature dependence of the density of Ti-Nb melts. The density of the Ti-Nb melts decreased with increasing temperature as shown in Fig. 1. Details of the experimental procedures and surface tension of the Ti-Nb melts will be presented at the conference.

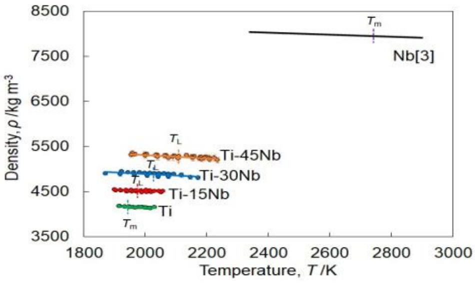

Fig. 1. Temperature dependence of the density of Ti-Nb melts.

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Wide-ranging speed-of-sound measurements in neon and derived third and fourth acoustic virial coefficients

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Neon is currently considered as an alternative working fluid to helium for different gas thermometry techniques. However, despite offering certain advantages, its application is hindered by the fact that the thermophysical properties of neon are far less accurately known than those of helium. Therefore, we carried out comprehensive and accurate measurements of the speed of sound in neon. The measurements were performed using a doublepath-length pulse-echo technique and cover the temperature range from 80 K to 420 K at pressures up to 100 MPa. To cover such a wide range of states, two thermostat arrangements were used, one for the cryogenic temperature range below 200 K and one for the higher temperatures. The relative expanded measurement uncertainty (at the 0.95 confidence level) is 0.015%. Furthermore, we derived third and fourth acoustic virial coefficients from the speed-of-sound data in the temperature range of the measurements by fitting a fourth-order acoustic virial expansion in pressure to the data using first-principles values for the second acoustic virial coefficient as constraints [1]. An auxiliary experiment was set up to measure speed of sound in neon between 200 kPa and 700 kPa at 273.16 K using a large volume copper spherical resonator. From the results of this experiment we determined the molar mass of the neon sample previously used for high-pressure speed of sound measurements and, additionally, the second acoustic virial coefficient β_a^{Ne} at 273.16 K. The results validate recent first-principles calculations of the higher virial coefficients of neon [2].

Acknowledgment

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Modified vibrating-wire viscometer for measurements of gas mixtures containing hydrogen

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A combined vibrating-wire viscometer and single-sinker densimeter [1] has been modified for measurements of gas mixtures of H_2 with CH_4, C_2H_6, C_3H_8, N_2 and CO_2. It enables simultaneous density and viscosity measurements at the same state point in the temperature range from 293 K to 493 K at pressures up to 30 MPa. The vibrating-wire viscometer is operated in the free-decay mode. The density measurements are characterized by a relative expanded uncertainty (at the 0.95 confidence level) of less than 0.1%, while the relative expanded uncertainty (at the 0.95 confidence level) in viscosity is estimated to be 0.25% to 0.3%. This poster discusses the modifications of the instrument in detail and presents results of first measurements. The magnetic field in the vibrating-wire sensors, which is used to excite the vibration of the wire and to measure the free decay of the vibration, is generated by two permanent magnets encapsulated by stainless steel plates. The magnetic field between the magnets was examined by simulations using the commercial software COMSOL. The results of these simulations revealed that the present design leads to a magnetic short-cut in the encapsulation. By exchanging the materials of the stainless steel plates, the short-cuts could be eliminated, which yields an improved signal-to-noise ratio of the measurement of the free decay of the vibration.

Because of hydrogen embrittlement, measurements of hydrogen containing mixtures impose strong requirements on the materials of the parts of the instruments which are in contact with the mixture. To meet these requirements, the materials of the vibrating-wire sensors had to be replaced by hydrogen compatible materials. Moreover, since the copper-beryllium alloy of the pressure vessel could not be qualified as a certified material for pressure vessels, the outer stage of the two-stage thermostat, the double-mantle thermostat, which surrounds the pressure vessel, is employed as shatter protection. This required a redesign of the double-mantle thermostat. Furthermore, the automation of the measurements was improved by employing a nitrogen-operated highly accurate pressure controller. The measured gas is separated from the absolute pressure transducers by a differential pressure transducer with a gold-plated membrane. The pressure controller automatically adjusts the pressure in the nitrogen-filled branch of the valve system between the differential pressure and absolute pressure transducers after reducing the pressure of the gas in the pressure vessel.

Acknowledgment

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Fatty acids loaded with graphene nanoplatelets as phase change materials for solar thermal energy storage

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Owing to the increasing awareness about energy-related issues such as global warming, the use of sustainable resources is gaining increased attention worldwide. Unfortunately, the intermittency of renewable alternatives such as solar is a major challenge in the development of this type of clean technologies. Thermal energy storage in the form of latent heat by using phase change materials (PCMs) is considered one of the most attractive strategies to address possible unbalances between energy production and consumption [1]. Over the last decades, fatty acids have been proposed as possible green PCMs to improve the energyefficient design and techno-economic performance of solar systems [2]. Compared to paraffins, usually extracted from crude oil, fatty acids are less toxic and can be produced from sustainable feedstock. However, like many other organic compounds, fatty acids exhibit lower thermal conductivity, which usually slows down the accumulation and release of stored energy. Among the strategies proposed in the literature to improve the thermal performance of PCM-based systems, the dispersion of carbon-materials has proved promising efficacy [1]. In this work, three different saturated fatty acids with carbon numbers of 12-16, namely lauric, myristic and palmitic acids, are investigated both pure and loaded with 0.25 wt.% of graphene platelets. Samples were characterized in terms of latent heat, melting temperature and thermal conductivity from 283 K to 343 K. A comparison between the obtained results will be shown, together with an analysis of the available literature data.

Acknowledgments

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A Burnett apparatus for experimental determination of the virial coefficients of hydrogen-containing gas mixtures

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With the Burnett technique [1], expansion series of a gas can be conducted to determine virial coefficients solely by measuring temperature and pressure at each expansion step of a series. In this work, a Burnett apparatus is designed to measure the virial coefficients of the hydrogen-containing binary gas mixtures H_2/CH_4, H_2/C_2H_6, H_2/C_3H_8, H_2/N_2, H_2/CO_2 with different molar compositions. The apparatus is designed for the temperature range from 233 K to 343 K with pressures up to 7 MPa. The materials of the parts of the apparatus in contact with the mixtures are chosen to be compatible with hydrogen. The expansion series are fully automated by employing motor-driven and pneumatic valves and a nitrogen-operated pressure controller. The measured gas mixture is separated from the absolute pressure transducers by a differential pressure transducer with a gold-plated membrane. The pressure controller automatically adjusts the pressure in the nitrogen-filled branch of the valve system between the differential pressure and absolute pressure transducers after each expansion step. To maintain a low uncertainty of the pressure measurements during a measurement campaign, the absolute pressure transducer and differential pressure transducer are calibrated automatically before each expansion series with a highly accurate nitrogenoperated piston gauge. A long-stem standard platinum resistance thermometer calibrated on ITS-90 is applied to measure the temperature in the wall of the pressure vessel. This poster discusses the Burnett apparatus in detail and presents results of first measurements.

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A new mixture model for carbon dioxide (CO_2) + difluoromethane (R32) systems

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In the framework of the international regulations on climate change, such as the EU F-Gas regulation and the Kigali amendment to the Kyoto protocol, mixtures of carbon dioxide (CO_2) and hydrofluorocarbons (HFCs) are gaining interest in the HVAC and refrigeration field. Carbon dioxide is a low-GWP, low-ODP non-flammable refrigerant characterized by a low critical temperature and a relatively high freezing point. These last two characteristics bound the efficiency of CO_2 appliances for heating and cooling. Although, HFCs are the most widely used refrigerants for a number of applications due to their good performance in the refrigerating cycles, but they are characterized by high GWP and thus restricted by international regulations. Among HFCs, difluoromethane (R32) gained interest in the last decade due to suitable thermodynamic properties and a relatively low GWP (675). At the same time, the application of R32 as refrigerant is subject to safety restrictions being mildly flammable (classified as A2L by the ASHRAE Standard 34-2019). Blending CO_2 with R32 might reduce the flammability and the environmental impact of the last one and extend the range of applications of the former. To better understand the thermodynamic behavior of CO_2/R32 systems, speed of sound measurements have been carried out with a acoustic-resonator-type instrument. Such measurements, together with vapor-liquid equilibra and density data available in the literature, have been used to build a new mixture model based on the Helmholtz energy equation of state by fitting either the binary mixture parameters and the departure function. The new model resulted to be in good agreement with the experimental data.

Surface Tension and Wettability of EV Transmission nanofluids containing $CaCO_3$ or CeF_3 Nanoparticles as Additives

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The rise of the electric vehicle (EV) as a more sustainable transport alternative makes it necessary to study new low-viscosity transmission fluids that adapt to the new requirements and improve its energy efficiency [1,2]. The use of nanoadditives in transmission fluids can lead to reductions in energy losses, wear, and emissions of pollutants as well as to improve the motor cooling. An important quality of the lubricants is their ability to spread on a metal surface, that is, the wettability of the lubricant. Wettability is usually characterized through properties like contact angle or surface tension, being relevant to study its dependence on temperature. Currently, there are hardly any studies related to the wettability properties by adding nanoparticles to lubricants [3]. In this work, surface tension and contact angle of nanolubricants composed of $CaCO_3$ or CeF_3 nanoparticles as additives and a transmission fluid, PAO8, were studied for different mass concentrations (0.05, 0.10, 0.15 and 0.20 wt%) at 298.15 K and 313.15 K. As Fig. 1 shows, decreases in surface tension with respect to the obtained for the PAO8 base oil were observed with the nanolubricants composed by the two nanoadditives, reaching the maximum reductions for the $CaCO_3$ nanolubricants, at 0.10 wt%, being 0.64% for T=298.15 K and 0.67% for T=313.15 K.

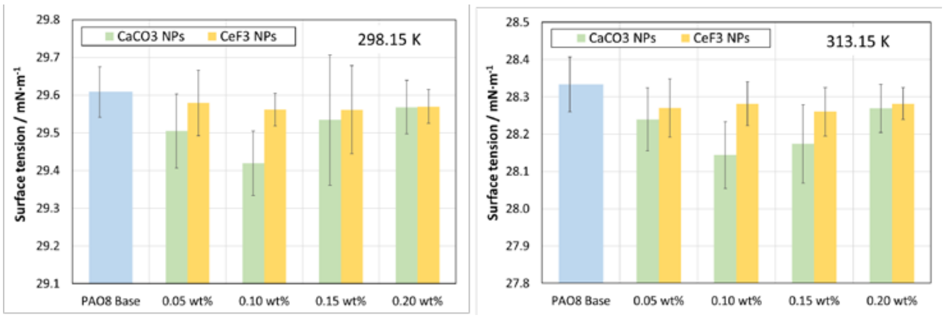

Fig. 1. Surface tension obtained for the base oil and $CaCO_3$ or CeF_3 nanolubricants at 298.15 K and 313.15 K.

Regarding the contact angle measurements of the $CaCO_3$ and CeF_3 nanolubricants and of the base PAO8 without additives, no clear trend can be observed in any of the cases that could indicate a significant change in the contact angle values by the addition of nanoparticles. Finally, it is clearly observed that when the temperature is increased, the values of the contact angles drop.

Acknowledgment

This work is supported by MCIN/AEI/10.13039/501100011033 and by the European Regional Development Fund (ERDF, FEDER in Spanish) through the PID2020-112846RB-C22 project as well as by Xunta de Galicia (ED431C 2020/10). JMLDR acknowledges the grant of the Margarita Salas program, funded by MCIN/AEI/10.13039/501100011033 and "NextGenerationEU/PRTR". M.J.G.G. acknowledges a postdoctoral fellowship (ED481B-2019-015) from the Xunta de Galicia (Spain).

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Phase Equilibria of Carbon Dioxide Capture from Binary Gas Mixtures using Tetraisopentylammonium Bromide Semiclathrate Hydrate

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Semiclathrate hydrates (SCH) are a type of gas hydrate that have shown promise in carbon capture and storage (CCS) applications. In these applications, the SCHs can be used to selectively capture and separate carbon dioxide (CO_2) from other gases in a mixture. By adjusting the pressure and temperature conditions, SCHs can be formed to capture CO_2, leaving other gases behind. Once the CO_2 is captured, the SCH can be thermally or mechanically destabilised, releasing the CO_2 for storage or use. This approach can contribute to decarbonization and net-zero energy production by capturing CO_2 from flue gas from combustion and precombustion capture of CO_2 from biogas. The aim of the research was to study the pressure-temperature phase stability of tetraisopentylammonium bromide (TiPAB) SCH in the presence of pure gases and binary mixtures relevant to flue gas and biogas and to measure the separation factors for CO_2.

Commercially available triisopentylamine and isopentyl bromide were used to synthesize TiPAB. Aqueous solutions of the TiPAB were prepared at concentrations ranging 0.027 and 0.271 mol·L^{-1}. The phase equilibrium of SCH phase with pure gases (CO_2, N_2 and CH_4) and binary mixtures representative of flue gas (80 mol% N_2 + 20 mol % CO_2) and biogas (50 mol% CH_4 + 50 mol % CO_2) were measured using the isochoric pressure search method in three identical stainless cell pressure cells at pressures up to 9 MPa. The temperature of these cells was controlled using a coolant jacket connected to a temperature-controlled circulation bath, and they were house inside an environmental chamber. For the gas mixtures, the gas trapped by the TiPAB SCH was analyzed using a gas chromatograph with a thermal conductivity detector, and the CO_2 split fractions and separation factors were calculated.

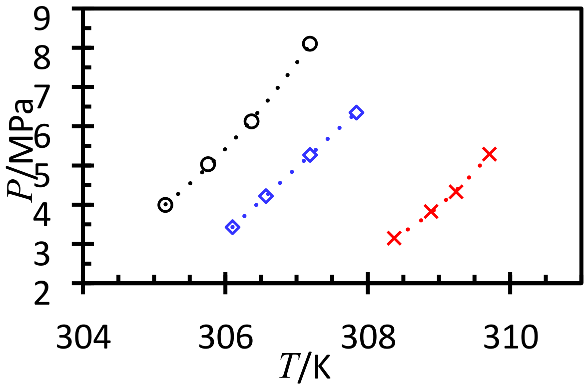

Fig. 1. Phase equilibrium for nitrogen (○), carbon dioxide (×) and flue gas (80 mol% N_2 + 20 mol % CO_2, ◊) at an aqueous concentration of TiPAB of 0.27 mol·L^{-1}

The measurements conducted revealed that SCHs formed with these gases are stable at temperatures above 296 K and up to 311 K. Increasing the TiPAB concentration increased the stability. The stability of the TiPAB SCHs formed with the flue gas mixture, as illustrated in Fig 1., was lower than that of pure CO_2 but more stable than the with pure N_2. Separation factors of up to 12.2 were observed for CO_2. Regarding the biogas mixture, biogas TiPAB was generally more stable than with pure CO_2 which was more stable than with pure CH_4. Separation factors of up to 4.9 were observed for the CO_2.

The high stability of TIPAB SCH is favourable for the development of new low-energy industrial carbon capture processes, as they may be simply dissociated using a low-grade heat source. However, to design an industrial separation process utilising TiPAB SCH further measurements, including kinetics, are required.

Acknowledgment

We thank Craig Grimm from UWA for his assistance in constructing the cells used in this work.

Density of aqueous alumina (15 nm) nanofluids at different temperatures

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Nanoscience deals with materials at the nanometre scale. At nanometric scale this scale matter presents properties distinctively different from those in the bulk and not surprisingly the concept of a nanofluid emerged in this field. Nanofluids are a new class of colloids consisting of solid particles with sizes typically in the range (1 to 100) nm dispersed in a base fluid.The literature has shown that the main factors involved in the behavior of their thermophysical properties are the size, morphology and content of nanoparticles, the purity of the base fluid and also the possible addition of surfactants. The results presented in this work were obtained without the addition of surfactants in order not to mask the effect of the nanoparticles and highlight the importance of the other factors mentioned above on the density behavior.

In this work the density of nanoparticles of the metal oxide of Al_2O_3 in water is studied. The measurements were carried out at fifteen concentrations (up to 2% in volume) and at six temperatures (from 293.15 K to 343.15 K). The nanoparticles are spherical with a size of 15nm.

The study shows the influence of the nanoparticle concentration and the temperature in the density behaviour. The enhancement of density, ρ/ρ_W, where ρ and ρ_W are the densities of the nanofluid and the water respectively, was calculated and its behavior was analyzed. From a fundamental standpoint, it is discussed whether the positive values of ρ/ρ_W can be considered greater than what would be expected. From an applied standpoint isobaric thermal expansivity is calculated at all temperatures and their values are compared with that of the base fluid.

From the theoretical point of view the contributions to density from water and nanoparticles are separated. Following the procedure proposed in [1] the thickness, density and molar mass of the interfacial nanolayer around nanofluid particles are predicted. The results obtained for the density deviation from the ideality do not support the Pak and Cho's equation [2] except as an approximation, [1].

Acknowledgment

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A reactive molecular dynamics study of HFO-1234ze(E) and CO_2/HFO-1234ze(E) pyrolysis mechanisms

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Owing to its low global warming potential, HFO-1234ze(E) and CO_2/HFO-1234ze(E) a suitable working fluid for the power cycle. Good thermal stability of the working fluid is crucial to create a safe power cycle. Understand the thermal decomposition mechanism of the working fluid to provide some theoretical guidance for the safe application of the working fluid. Herein, the thermal decomposition behaviors of HFO-1234ze(E) and CO_2/HFO-1234ze(E) are investigated via ReaxFF molecular dynamics simulations with a reliable force field validated by the density functional theory (DFT) method. For the first time, the HFO-1234ze(E) pyrolysis pathway and the effect of CO_2 on the HFO working fluid pyrolysis pathway were analyzed using the ReacNetGenerator package software. ReaxFF MD simulations lead to reasonable Arrhenius parameters compared with experimental results based on first-order kinetic analysis of HFO-1234ze(E) and CO_2/HFO1234ze(E) pyrolysis. The results show that the pyrolysis pathways of HFO-1234ze(E) are divided into 3 categories: (1) the cleavage of the C-C bond to form smaller hydrocarbon or fluorocarbon radicals; (2) the dehydrogenation or defluorination reaction to form an H or F radical; (3) H-abstraction reactions by small radicals including H, CH, CHF, or CF_3. CO_2 affects the pyrolysis pathway of HFO-1234ze(E). The present research demonstrates that ReaxFF reactive molecular dynamics is a valuable tool for the detailed study of pyrolysis mechanisms of HFO working fluid, which complements commonly used experimental and computational techniques. [1-3].

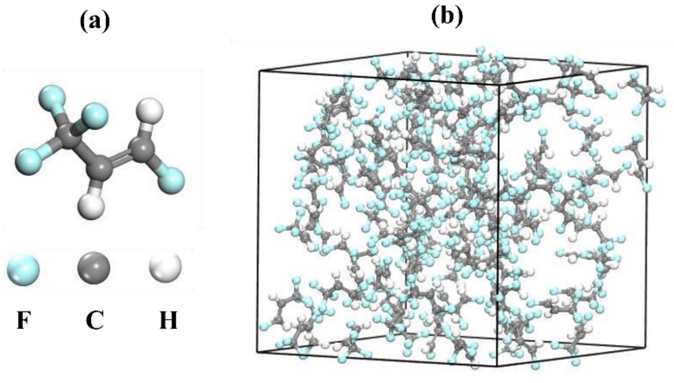

Fig.1. (a) Molecular structure of R1234ze(E) (b) R1234ze(E) pyrolysis system

Acknowledgment

This work was supported by the National Natural Science Foundation of China (No. 52022066).

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Numerical Study and Optimization of Doping Graded Oriented Extinction Phasechange Fiber for Enable Thermal Insulation Improving

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Phase change materials (PCMs) play essential roles in extending transient thermal insulation in the aerospace field; however, the extinction properties variations during the phase change process have seldom been investigated. The present study presents an extinction phase-change fiber (EPCF) design, i.e., composed of extinction material shell as well as the PCM core and dispersed in Al_2O_3 aerogel. The extinction performance (especially after the phase transition) and the insulation performances at steady and transient states according to specific infrared radiation wavelength modulation are numerically investigated. This study reveals that the extinction characteristic of VO_2@SiO_2 is superior to that of SiO_2 and TiO_2 fiber. The extinction performance is improved by ~8.37% after the phase transition of VO_2. Besides, the time constant of VO_2@SiO_2 increased by ~2.17 times the pure aerogel at the insulation aspect. Similar conclusions could be self-evidently found in some other EPCFs; furthermore, multi-layer EPCFs design is studied for spectral modulation principle combined with the transient heat transfer characteristic. Our developmental work could inspire some novel ideas on insulation material design in the future and improve the understanding of phase change insulation [1,2].

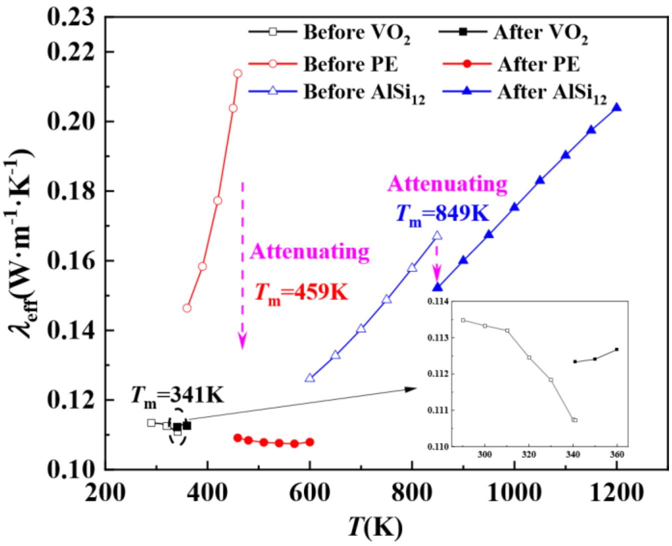

Fig. 1. Thermal insulation performance of EPCFs-aerogels under steady state. The λ_r decreases markedly due to the improvement of extinction property after PE and $AlSi_{12}$ phase transformation at T= 459 K, 849 K.

Acknowledgment

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Measurement Method for Thermal Conductivity of Porous Aerogel Composites based on High Temperature Gas Flow Heating

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The novel micro-nano porous aerogel composites have high temperature resistance and ultra-low thermal conductivity, and the accurate measurement of thermal conductivity is very important [1].

Due to micro-nano porous structure, the radiation heat transfer performance at high temperature is far more than that of solid heat transfer and gas heat transfer, which becomes the main factor affecting thermal conductivity [2].

However, thermal conductivity measurement methods such as conventional protective hot plate raise the surface temperature of the material by means of high temperature metal plate contact, whose radiation characteristics will increase the internal radiation intensity of aerogels greatly and make the measurement results significantly higher than the actual value [3-4].

Based on this, a low thermal conductivity measuring device of steady-state heat flux method using high temperature gas flow heating method is designed. During the test, the aerogel hot wall surface is heated by high temperature gas flow, and the cold wall surface is attached to the metal water cold plate. The heating test of aerogel is shown in Figure 1. The core area between the material and the metal water cold plate is embedded with a high precision thermal conductivity heat flux meter to measure the normal heat flux. In order to reduce the influence of heat leakage around the materials, a large temperature difference is formed in the normal direction of the measured sample to ensure that the heat flux in the normal direction is large enough. The test results show that the heat transfer in the aerogel reaches equilibrium at 1200 s continuously heated by the high temperature gas flow of 800℃. The temperature of cold and hot wall are 57℃ and 310℃ respectively. The heat flux measured by the heat flux meter is 700 W/m^2, and the calculated thermal conductivity is 0.036 W/(m.K). Compared with the thermal conductivity measured by the protective hot plate method, the thermal conductivity is reduced by 4.3%.

(a) (b)

Figure 1. Heating test of aerogel: (a) Outer wall surface; (b) Inner wall surface.

Acknowledgment

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Numerical study of stratified fiber volume-type extinction and reflective shields surface-type extinction for high temperature aerogel insulation

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Spherical opacifier doping aerogels for thermal insulation have been investigated in the past decades. However, the cylindrical fiber doping as well as reflective shields surface-type extinction are seldom studied. Particularly, there are no numerical optimization and comparative study on these two extinction dopant types. Our results reveal that the optimal diameter of non-silica fiber dopant is significantly lower than that of opacifier doping at identical temperature. Carbon fiber has incomparable infrared radiation suppression ability of about 4 times that of carbon opacifier in the whole temperature range. Compared with the opacifier doping, the nonsilica fiber doping method is demonstrated to provide stronger light shielding effect, lower thermal conductivity at a certain doping fraction, and much higher mechanical properties. Next, a stratified core-shell design for oxidizable carbon fiber is proposed to extend its excellent radiation suppression ability to high temperature. Moreover, reflective shields surface-type extinction is investigated and compared. Our findings provide useful guidance for aerogel high temperature thermal insulation. [1,2].

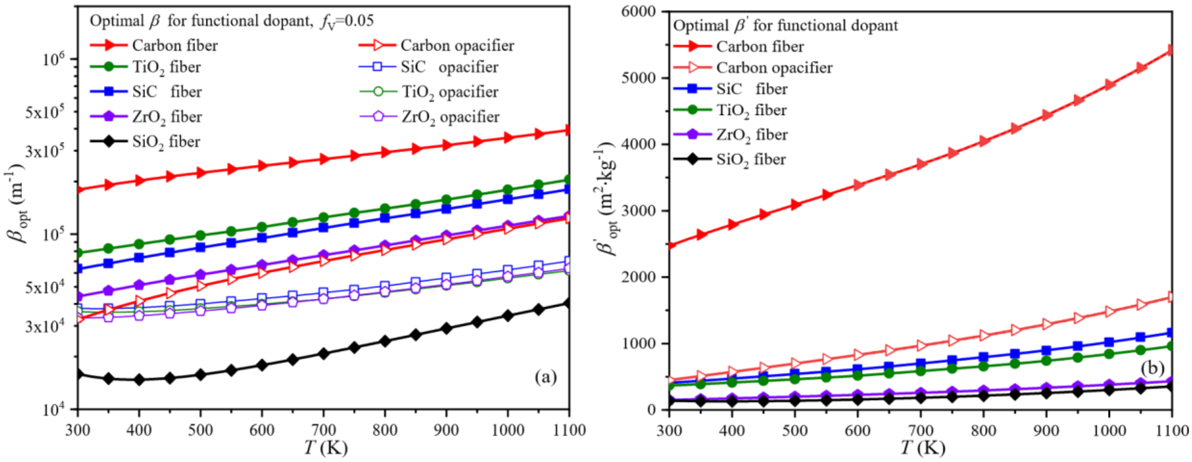

Fig. 1. Optimal temperature-dependent extinction coefficient b$_{opt}$ (a), and optimal temperature-dependent specific extinction coefficient b'$_{opt}$ (b) of dopants in function of temperature.

Acknowledgment

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POSTER SESSION PS2

Calibration of Thermocouple Wafer Sensor Using Point-to-Point Method

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For the evaluation of temperature accuracy and spatial uniformity inside the chamber being used in the semiconductor industry, wafer sensors with thermocouple junctions (so-called TC wafer) on the wafer surface have been used in many processes, such as doping and annealing. The accuracy of the TC wafer indeed depends on the calibration of each thermocouple junction on the surface. Usually, TC wafer calibrations have been done in the furnace with a large inside the working volume. Still, the exact temperature uniformity of the furnace could not be evaluated due to the lack of enough reference thermometers. To increase the accuracy of TC after, a new concept of calibration system with point-to-point contact between the reference thermocouple and TC wafer measuring junction is constructed. A total of 33 reference type B thermocouples are installed upwards from the bottom of the furnace chamber, and a TC wafer with a diameter of 300 mm can be placed on these thermocouples. Point-to-point contact can meet each TC wafer junction with a corresponding reference thermocouple junction just beneath the wafer. This direct contact can reduce the measurement difference between the reference and the TC wafer to improve the calibration accuracy. This work will prepare and test two TC wafers (one is designed with type R, and the other is a type B thermocouple) using this system from 600 ºC to 1000 ºC.

Acknowledgment

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Experimental Investigation of Convective Properties of Graphene Aerogels

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With the increasing ubiquity and performance of portable electronic devices, it is crucial to develop materials that are both lightweight and efficient in dissipating heat [1]. Open-cell porous materials have been identified as promising candidates to meet these technological demands, with graphene aerogels being particularly noteworthy due to graphene's exceptional thermal conductivity, high specific surface area, strong internal flow mixing properties, low density, good corrosion resistance, and potential flexibility and stretchability [2-3]. To lay the groundwork for the development of ultra-lightweight heat dissipation devices based on graphene aerogels, this study aimed to experimentally investigate the convective heat transfer properties of these materials, including flow permeability and convective heat transfer coefficient (Fig. 1).

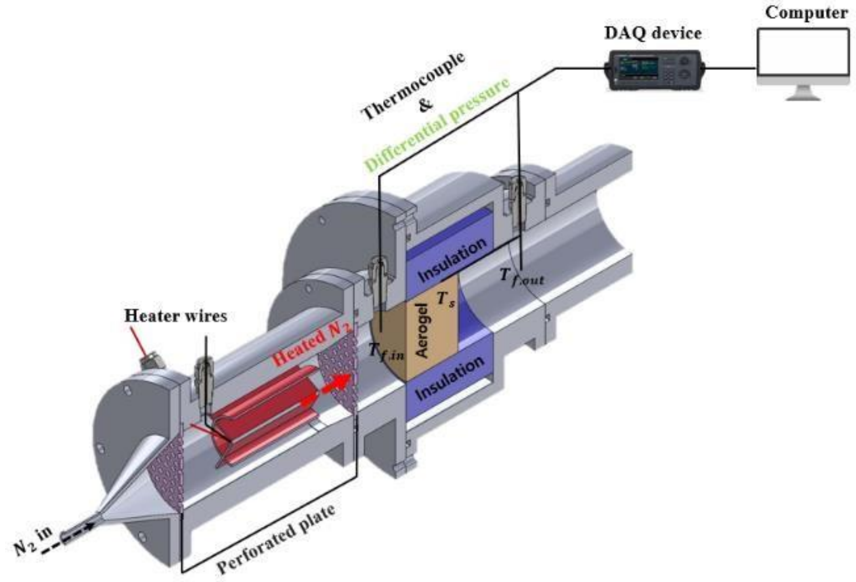

Fig. 1. Schematic diagram of the experimental setup.

Acknowledgment

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Development of a system for measuring specific heat of refrigerants

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There has been considerable research activity focused on identifying alternative refrigerants that exhibit a low global warming potential (GWP) [1]. Achieving an accurate equation of state (EOS) for newly developed refrigerants is crucial for designing efficient cooling systems for industrial use, and requires precise measurement and analysis of their thermophysical properties, including specific heat, thermal expansion coefficient, and compressibility [2]. The present study utilized a high-pressure sample injection system, comprised of two syringe pumps (Teledyne ISCO, 260D) and a mixer (Vinci BTSP-500-5), in conjunction with a Calvet calorimeter (Setaram BT2.15), to determine the specific heat of refrigerants at different pressures and temperature. The temperature of the system can be precisely regulated within the range of -40 to 6$_0$ °C by means of the calorimeter, while the pressure can be monitored and actively regulated in real-time during the mixing process, spanning from 1 bar to 80 bar. The system was checked by measuring and validating the specific heat of a widely accepted reference material. For the purpose of verifying the accuracy of the specific heat measurement system for refrigerants, the specific heat of R410a was measured in the liquid domain while varying the temperature and pressure. We present results showing excellent consistency with values found in the existing literature, with deviations of less than 5%.

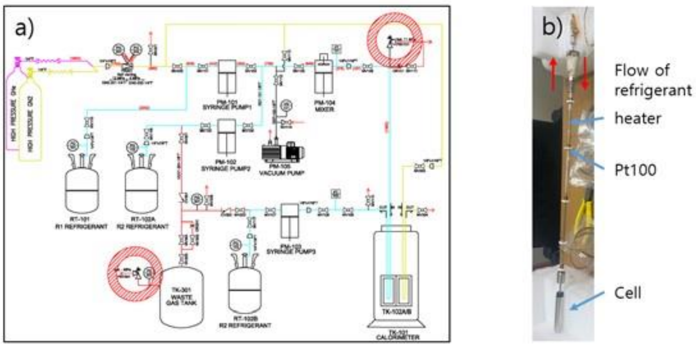

Fig. 1. a) Piping & instrument diagram, b) Modification of measurement cell in the calorimeter.

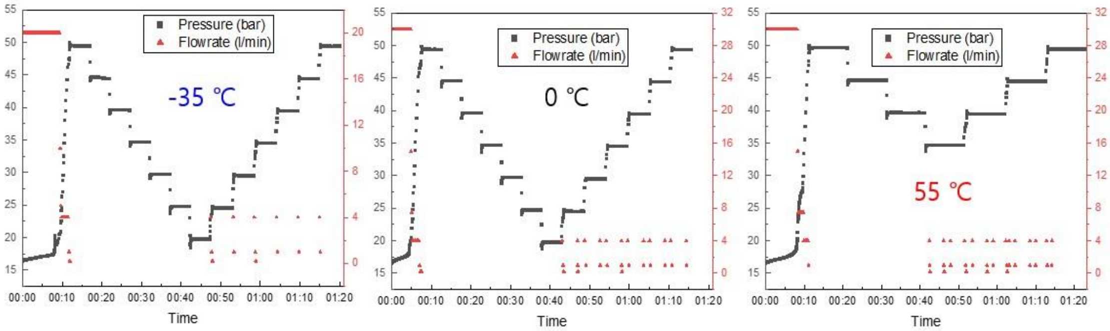

Fig. 2. The pressure test of the system using R410A.

Acknowledgment

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Measurement of phase fraction in simulated lunar regolith and evaluation of average thermal conductivity

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Regolith on the lunar surface has been noted to adhere to spacecrafts.Thermal interaction between regolith and the spacecraft should be considered and reflected in the design for a successful mission on the Moon. This analysis requires thermophysical properties of particles less than 1 mm in diameter. Endo et al.[1] reported the thermal conductivity of a regolith simulant(FJS-1) particle from the distribution of thermal property ($b=\sqrt{C\rho\lambda}$, C: specific heat, ρ: density, λ: thermal conductivity). Sakatani et al. [2] reported the thermal conductivity of JSC1A for a large number of particles using the line heat source method. The measured value was affected by both the space between the particles and voids in the sand. Based on the above background, the objective of this study was to measure the fraction of phases in a large number of particles for lunar regolith simulants and to calculate the average thermal conductivity.

Samples used were lunar regolith simulants, FJS-1 and JSC-1A. Samples were embedded in resin and polished with a mirror finish. The distribution of phases in the sand was observed and elementally analyzed using a scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (SEM-EDS). X-ray diffraction (XRD) was also performed to identify the mineral phase. The phase proportions and porosity were analysed on images of more than 10 taken by SEM. The average value of *b* and average thermal conductivity of the sand particles were evaluated from the value of *b*, density and specific heat of each phase. Fig. 1 (a) and (b) show backscattered electron images (BEI) of FJS-1 and JSC-1A. Both are composed of multiple phases, but JSC-1A has many voids. SEM-EDS and XRD results confirmed that anorthite, olivine, and ilmenite exist in FJS-1, while there are pyroxene, anorthite, and olivine in JSC-1A. From the phase proportions, the average thermal conductivity was evaluated to be 2.50±0.06 $Wm^{-1}K^{-1}$ for FJS-1 and 4.19±0.20$Wm^{-1}K^{-1}$ for JSC-1A. Furthermore, by considering the voids in the JSC-1A particle, the average thermal conductivity was 3.44 ± 0.39 $Wm^{-1}K^{-1}$.

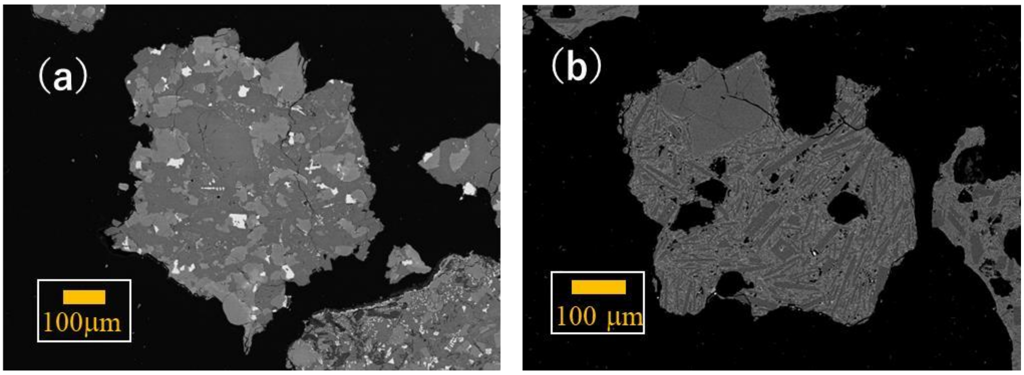

Fig. 1. BEI of (a) FJS-1 (b) JSC-1A

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Determination of average thermal conductivity for a lunar regolith simulant particle by using thermal microscope

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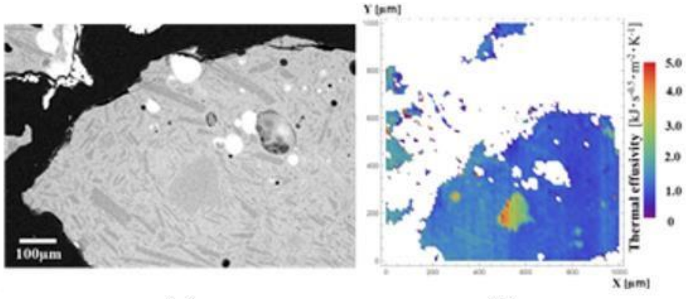
The thermal conductivity of a single grain of lunar regolith must consider the interaction between the spacecraft and the lunar regolith. This study aimed to determine the average thermal conductivity of a lunar regolith simulant through the thermal effusivity distribution measured by a thermal microscope. The thermal microscope is an apparatus used to measure thermal effusivity on the micrometer scale by combining cyclic heating and reflectance methods. Spatial resolution is about 10μm. The lunar regolith simulant (JSC-1A) particles were embedded in an epoxy resin and polished to a mirror finish. RF-spattering coated about 100-nm thick Mo film. The thermal effusivity was measured for a 1×1 mm^2 area with intervals of 10 μm by thermal microscopy. The measured area was analysed by scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM–EDS). X-ray diffraction (XRD) was performed to identify the mineral phases in JSC-1A. Fig. 1 (a) and (b) show the back-scattering electron image (BEI) of the sample and thermal effusivity distribution, including the area shown in Fig.1(a). Fig. 1(a) shows that the sample consists of several phases. Some phases have a size of 100 μm; however, most consists of phases with 10 μm or less, which is smaller than the resolution of a thermal microscope. The SEM–EDS and XRD results showed that a single sand particle comprised several minerals, such as anorthite, olivine, pyroxene, and ilmenite. All the phases are solid solutions. Fig. 1(b) seems to correlate with the BEI of the sample. For more detail, the measured thermal effusivity reflects (i) the chemical composition change in the solid solution for larger particles and (ii) the interfacial resistance between the small phases. Table 1 shows the average thermal effusivity and thermal conductivity calculated using the density and specific heat of the sand particles [12]. The difference in thermal effusivity and conductivity between two particles would be due to the difference in the ratio of existing mineral phases.



(a) (b)

Fig. 1. (a) BEI and (b) corresponding thermal effusivity distribution of a sand particle

Table 1. Average thermal effusivity and thermal conductivity of sand particles

	Thermal effusivity $[\mathrm{kJ\cdot s_{-0.5}\cdot m_{-2}\cdot K_{-1}}]$	Standard deviation $[\mathrm{kJ\cdot s_{-0.5}\cdot m_{-2}\cdot K_{-1}}]$	Thermal conductivity $[\mathrm{W\cdot m^{-1}\cdot k^{-1}}]$
Particle 1	1.2	0.4	1.0
Particle 2	1.3	0.7	1.2

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Detection of Hydrogen in Air by the Suspended 3 Omega Method

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The concentration of hydrogen can be calculated by measuring the mixture of hydrogen and air. Typical thermal conductivity-based hydrogen concentration sensors have the lowest detectable hydrogen concentration of 1% (volume fraction). In order to achieve further reduction in the lowest detection limit of the hydrogen concentration, it is essential to have the 3 omega sensor on a low thermal conductivity substrate such as polyimide to prevent heat from the microheater from being leaked to the substrate. The highest hydrogen concentration sensitivity can be achieved by isolating the microheater from the substrate, a suspended microheater. In this paper, conduction heat transfer modeling of a microheater, substrate, and sample gas is performed by utilizing a bi-directional 3 omega method. When measuring with 3 omega sensors fabricated on EXG glass or polyimide substrates, the minimum detectable hydrogen concentration was calculated as 10% and 4%, respectively. If a 3 omega sensor with a suspended microheater structure was used, the lowest measurable hydrogen concentration in air was reduced to ~0.25%. A 3 omega sensor with a suspended microheater structure is expected to enable real-time monitoring of localized hydrogen diffusion in air, with spatial and temporal resolutions of ~2 mm and ~1 s or less, respectively.

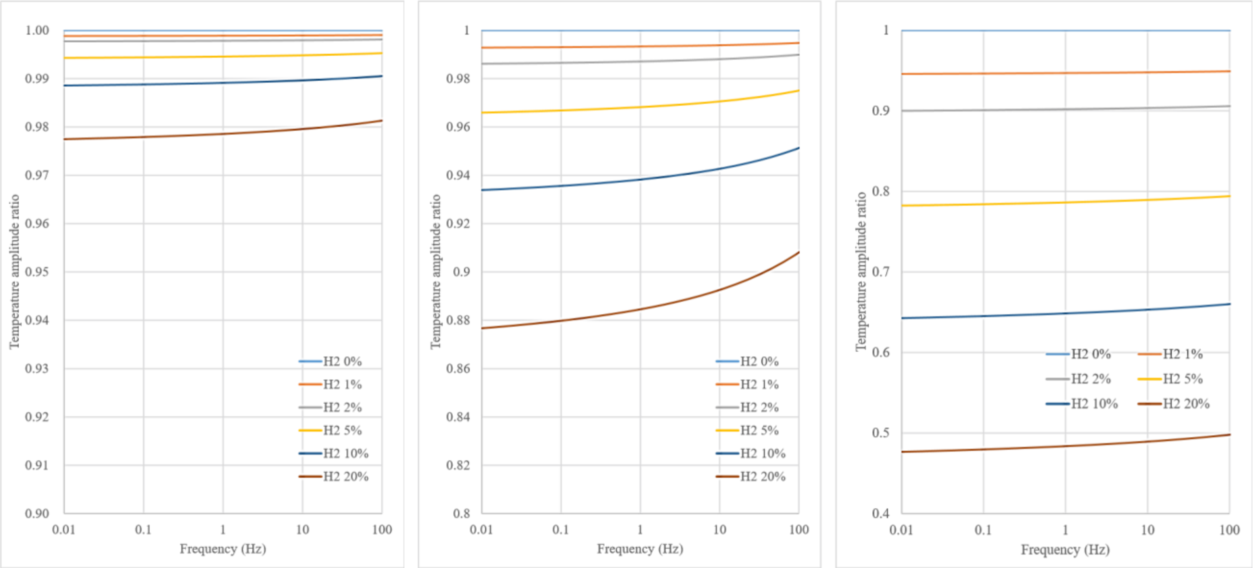

Fig. 1. Calculation of temperature amplitude ratio (with respect to temperature amplitude when 100% air and 0% hydrogen) for temperature oscillation frequency range of 0.01 to 100 Hz and (left) for EXG glass substrate, (middle) polyimide substrate, and (right) suspended 3 omega sensors.

Acknowledgment

This work was supported by the National Research Foundation of Korea(NRF) grant funded by the Korean government(Ministry of Science and ICT) (No. RS-2022-00144368).

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Development of viscosity and density measurement system for mixed refrigerants

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Due to climate change, there are many studies being conducted to develop refrigerants with low global warming potential (GWP)[1]. It is important to accurately measure the properties of refrigerants for the development of an accurate equation of state (EOS) to ensure their stable use once they are developed [2]. So, in this study, a system was developed and validated with a reference refrigerant for measuring the density and viscosity of mixed refrigerants, as shown in Fig. 1. The vibrating tube densimeter (Anton-Paar, HPM DMA) and the moving piston viscometer (Cambridge Viscosity, ViscoPro 2100) were applied to measure the density and viscosity of the refrigerant, respectively. To measure the properties of the liquefied refrigerant, the temperature and pressure of the refrigerant were controlled by an environmental chamber(Weiss) and a high-pressure syringe pump(ISCO). To verify the measurement equipment, density measurement was carried out from 15 bar to 50 bar at room temperature using R410a, which is well known refrigerant, and it was confirmed that the measurement results had a maximum difference of 0.1% compared to the results of REFPROP as shown in Fig. 2. In the future, this validated system will be used to measure the density and viscosity of newly developed mixed refrigerant.

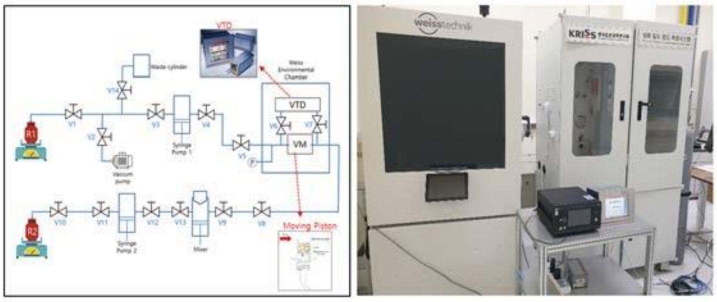

Fig. 1. Viscosity and density measurement system for mixed refrigerants in KRISS

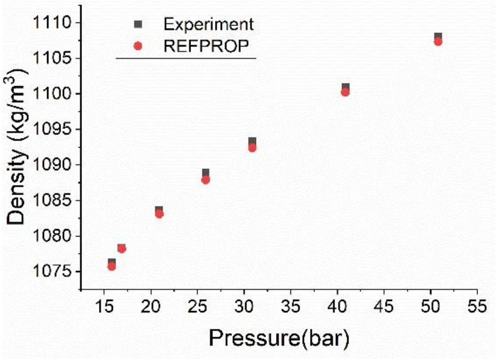

Fig. 2. Density measurement results compared to REFPROP with R410A for different pressure up to 50 bar

Acknowledgment

This work was partly supported by Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant funded by the Korea government (MOTIE) (20212020800070, Development of next-generation alternative refrigerant and efficient heat pump system).

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Development of a transient hot-wire apparatus for thermal conductivity measurement of mixed refrigerants

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Due to the global warming issue, next-generation refrigerants having very low global warming potential (GWP) have been interesting increasingly [l]. In order to develop high-profile refrigerants having GWP < 10, several thermophysical quantities, for examples, density, heat capacity, viscosity, and thermal conductivity, etc., should be measured precisely. Among them, the thermal conductivity of the refrigerant fluid has been studied due to the importance in caculating heat transfer coefficients for the application of the heat exchanger design [2]. A transient hot-wire (THW) apparatus with the temperature range from -40 oc to 80 oc and the pressure range between 1 MPa and 10 MPa for measuring the thermal conductivity of the refrigerant fluid and the binary mixture of refrigerants was developed. Temperature and pressure control performance checks of the apparatus were conducted with helium. The main THW cell was calibrated to obtain the resistance-temperature relation of the hot-wire sensor, which is conformed to Korean national standars established by the Temperature Standards Laboratory at the Korea Research Institute of Standards and Science (KRISS). The THW apparatus was validated with measurement of pure water and toluene in liquid phase and nitrogen, argon in gas phase over the entire temperature and pressure range. In order to validate our THW apparatus for thermal conductivity measurement of mixed refrigerants, the thermal conductivity of R41 Oa were measured as a function of temperature and pressure in liquid and vapor regions. The measured values shows good agreement with the literature values within less than 1%.

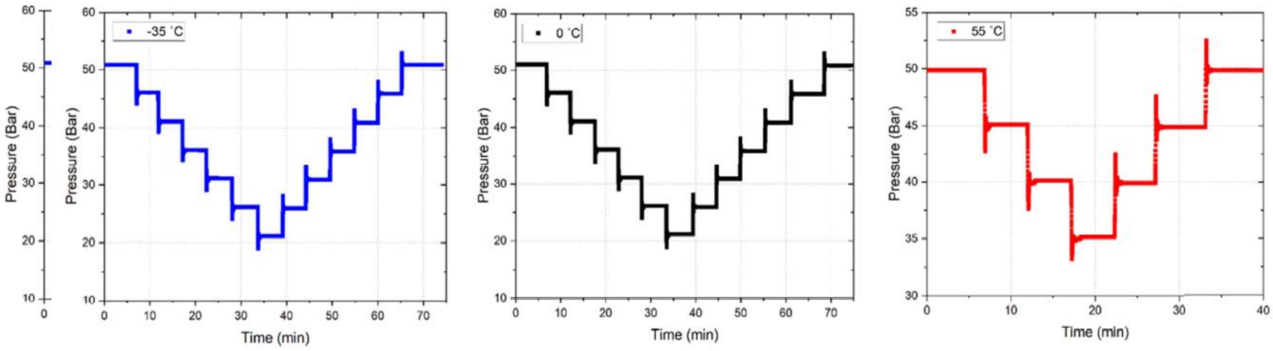

Fig. l. Temperature and Pressure Performance checks of the THW apparatus

Acknowledgment

This work was partly supported by Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant funded by the Korea government (MOTIE) (20212020800070, Development of next-generation alternative refrigerant and efficient heat pump system).

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Variation of effective thermal conductivity with oxide scale structure and its effect on cooling of steel

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During the hot rolling process of steelmaking, an oxide scale is formed on the steel plate, which affects the cooling rate of the steel. Wendelstorf et al.[1] showed spray cooling experiments on steel with oxide scale and reported the effective value of surface heat transfer coefficient. The reported value showed large errors. One of the factors contributing to the error is the thickness of the oxide scale varies with the location of the sample. Clarifying the effect of the oxide scale on the apparent surface heat transfer coefficient will provide guidelines for controlling the cooling rate of steel. Therefore, this study aims to observe the oxide scale formed on a practical steel plate to clarify the thickness and structure and to evaluate the effective thermal conductivity of the oxide scale. The cross-section of heavy steel was observed using a scanning electron microscope (SEM). A micro X-ray CT system also analyzed the distribution of each layer in the oxide scale. Fig. 1 shows the results of the cross-sectional observation of the sample by SEM. The thickness of the Si-rich layer was found to be uneven. Based on Fig. 1, the oxide scale could be expressed by the three models, as shown in Fig 2. The thicknesses of the $Fe_{1-x}O$ and Fe_3O_4 layers are the same in both of them, but the presence of a porous layer and the thickness of the Si-rich layer were different: (a) a model with a Si-rich and porous layers, (b) a model with a thin Si-rich layer, and (c) a model with a thick Si-rich layer. Proportions of models (a), (b), and (c) were 8.1%, 53.6%, and 38.2%, respectively**.** The effective thermal conductivity of each model was then evaluated [2-3]. In the evaluation, the heat was considered to transfer from the bottom of the model to the top during the cooling of the steel [4]. The effective thermal conductivities for models (a), (b), and (c) in Fig. 2 were 0.23, 1.2, and 1.8 $Wm^{-1}K^{-1}$, respectively. It was found that the porous layer decreases the effective thermal conductivity significantly, and the thickness of the Si-lich layer also has an effect.

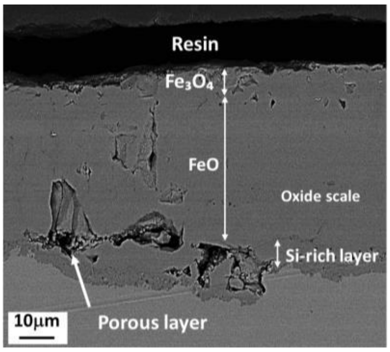

Fig. 1. Results of cross-sectional observation of Si-containing heavy steel by scanning electron

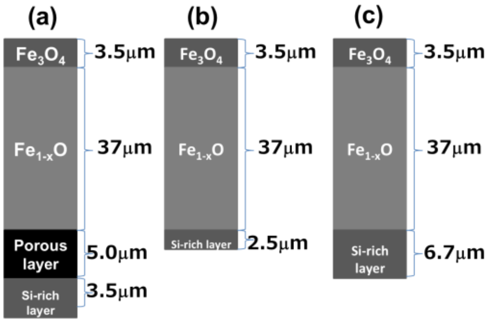

Fig. 2. Three oxidation-scale structural approximation models

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The authors would like to thank National Institute for Materials Science (NIMS), Japan, for their help with the micro X-ray CT measurements.

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Densities of aqueous solutions of amines up to 100 MPa

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The use of amines as chemical absorbents in postcombustion CO_2 capture is extending due to the knowledge of the technology involved in comparison to other methods, however, different amines or mixed amines are explored in the search of effectiveness and decreasing the energy demand.

A first step to propose alternative amines is the knowledge of their thermophysical properties such density, viscosity or heat capacity, to the simulation and the design of the absorption and desorption units. With this purpose, our group is involved in the accurate measurements of these properties for different aqueous solutions of amines and, densities of different amines, such as 2-aminomethylpropanol (AMP) and 3-methylaminopropylamine (MAPA), will be presented. For each amine, four mixtures (amine + water) at mass fractions $w_{\text{amine}} = 0.1$; 0.2; 0.3 and 0.4, were prepared by weighting and the measurements were carried out at six isotherms, from 293.15 K to 393.15 K, and pressures up to 100 MPa.

The measurements of density were carried out using a commercial vibrating-tube densimeter (Anton Paar DMA HPM). This technique was improved in our laboratory with different devices in such a way that ramps of pressure and temperature are programmed in a computer and the frequencies are recorded allowing an automated measurement. This equipment is able to measure density from (0 to 3000) kg/m^3 with an expanded uncertainty (k=2) less than 0.7 kg/m^3 [1].

As an example of the general behavior observed for the mixtures studied, density of the aqueous mixture AMP ($w_{\text{AMP}} = 0.4$) is represented as a function of pressure at different temperatures in Figure 1. It can be noticed the increase of density with increasing pressure or the decrease of density with increasing temperature.

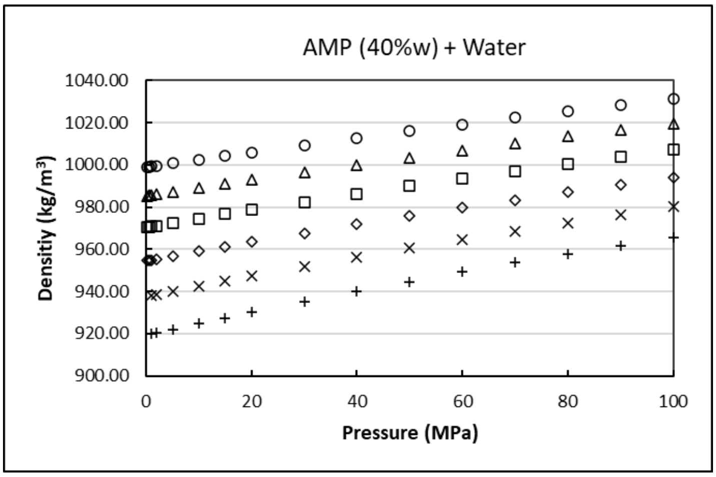

Fig. 1. Density of the mixture AMP + water (amine mass fraction 0.4) as a function of pressure at different temperatures: (○) 293.15K; (Δ) 313.15 K; (☐) 333.15 K; (◊) 353.15 K; (×) 373.15 K; (+) 393.15 K.

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Measuring humidity in gases using a microwave resonator

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Humidity is one of the main contaminants which can be found in different technological processes such as the manufacture of ultra-pure gases, production of semiconductors, energy processes or space applications. However, these industries require new measurement methods and standards to determine humidity in higher ranges of pressure and temperature for a variety of non-air gases [1].

Most primary humidity standards realize dew-point temperature and their measurement in air at atmospheric pressure are well established. In addition, dew-point is directly related to water vapor pressure and this is used to obtain other humidity parameters.

In the search of fulfilling the industrial needs and requirements in high accurate humidity measurement and traceability, microwave resonators offer a high potential as standard hygrometers. This paper presents the development of a new microwave resonant cavity designed in our group and the set-up of the technique to be used as standard hygrometer. The work is part of the project PROMETH2O funded by European Metrology Programme for Innovation and Research (EMPIR) from EURAMET. Our research group (TermoCal) in collaboration with the National Institute of Aerospace Technology (INTA), which is in charge of the national humidity standard, are involved in the determination of water vapor enhancement factors in the frost-point temperature range from −20 °C to −90 °C, at pressures from 0.1 MPa to above 4 MPa, focusing on selected ultra-pure gases such as nitrogen, argon and hydrogen.

The quasi-spherical cavity is made of stainless steel 316L, and its dimensions are an external diameter of 60 mm and inner diameters of (x=50.08 mm, y= 50.16 mm and z=50 mm). It is internally covered by a 15 µm gold layer to improve the electric conductivity of microwaves. The resonator is formed by two semi-spheres which are attached together in the equatorial part using screws. In addition, a tight pressure vessel has been designed to allocate the quasi-spherical resonator. The quasi-spherical resonator and the pressure vessel will be filled with the humid gas being the same conditions (pressure and temperature) inside and outside the resonator.

The pressure vessel is maintained at constant temperature by means of a thermostatic bath, and the temperature is measured using a platinum resistant thermometer calibrated with a standard uncertainty of 2 mK. On the other hand, a piezoelectric quartz transducer is used to measure the pressure being its relative standard uncertainty of 0.01%. Both magnitudes are traceable to national standards with coverage factors k equal 1.

Finally, a humid generator is added to the system to control the flow of humid gas entering the resonator. To complete the set-up of the technique, first, measurements of humid air are being undertaken to check the correct performance of the system. In this stage, a commercial humidity generator (Thunder 2500) is used. The humid air, provided by this equipment, is fed to the microwave resonator and to a precision dew point hygrometer (MBW DP30), calibrated to national standards, in order to compare the results and check the performance of the new microwave technique. Thenceforth, we will continue measuring other gases such as nitrogen and hydrogen.

Acknowledgment

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Measurements of speed of sound in hydrogen with propane

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The European Union is committed to tackle climate change and environmental degradation and the use of renewable energy or decarbonizing the gas grid are some of the actions promoted by EU to achieve those goals. Moreover, introduction of biomethane, hydrogen-enriched natural gas, pure hydrogen or carbon capture and storage are different strategies that may be applied.

On the other hand, gas industries have identified some specific priorities regarding the challenges of new energy gases: calculating flow of alternative energy gases is not possible and it requires new traceable facilities for flow meters for hydrogen or hydrogen-enriched natural gas, which is a key parameter since it is related to the determination of costs. Also, costs are linked to energy content which depends on composition, but there is a lack of traceable gas analysis methods. In both cases, physical property measurements are important for designing these decarbonized gas grids. Currently, there is a lack of experimental results necessary to validate numerical methods adopted by industries to predict the properties of the alternative gases.

Our research group is involved in the thermodynamic characterization of these new gas mixtures by measuring density, speed of sound or phase envelope, using high accurate experimental techniques.

In this paper, new speed of sound data of different mixtures of hydrogen and propane are presented. The measurements were carried out using a spherical acoustic resonator [1]. The cavity, which consists of two hemispheres fixed by electron beam welding, is made of 321 austenitic stainless steel and has an internal radius of 40 mm and thickness of 10 mm. The temperature is measured by means of two capsule-type platinum resistance thermometers whose standard uncertainty is 1 mK at 273.16 K. Two resonant quartz-crystal manometers are used for pressure measurements at ranges of (0 to 2) MPa and (1 to 20) MPa with a relative standard uncertainty of 0.01%. The total uncertainty of the speed of sound is not worse than 0.02%.

Acoustic resonance frequencies and the half-width of the resonance peaks are measured for four different acoustic radial modes (0,2), (0,3), (0,4) and (0,5) at each pressure, and the final speed of sound is the average obtained for these modes whereas the dispersion of the values is considered as a contribution to the uncertainty of the speed of sound.

Finally, the experimental results are compared with those predicted by GERG 2008 equation of state [2], which is the equation of state recommended for calculating properties for natural gas and currently serves as the ISO standard (ISO 20765-2). It estimates the thermophysical properties in the entire fluid region for natural gases and related mixtures of up to 21 components. However, it has only been validated using limited test conditions for hydrogen enriched natural gas. Our measurements will provide new data of binary gas mixtures needed to cover the lack of experimental data required to improve GERG-2008 formulation for this kind of mixtures.

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Thermodynamic (p, ρ, T) characterization of hydrogen-enriched natural gases

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Hydrogen-enriched natural gas, H2NG, is a mixture of natural gas and H_2 that can be used on existing natural gas infrastructure with little or even no modification to be applied. H2NG is thus an option in the transition from fossil fuels to a hydrogen economy, as it can reduce greenhouse gas emissions and can accelerate the adoption of H_2 from renewable sources. Theoretically, H_2 can be mixed with NG in any ratio, but H2NG mixtures with up to 20 vol-% of H_2 represent the most realistic near-term option due to technical and economic reasons.

The addition of H_2 to natural gas alters the thermodynamic properties of the mixture, which affects its transport, storage, and combustion characteristics. In this work, we present experimental density measurements for different H2NG mixtures, performed with a high-precision single-sinker densimeter [1], from (250 to 350) K and up to 20 MPa. The mixtures were prepared gravimetrically according to ISO 6142-1 [2] for maximum precision in their composition. Six mixtures were prepared: two standard 11-compound high-calorific (H_2free) basic natural gas mixtures, and four derived H2NG with 10 % and 20 % H_2 added. The experimental density results for the six mixtures are compared to the current reference equation of state for natural gas, the GERG-2008 [3]. While the H_2-free natural gas mixtures are well represented by the reference EoS, relative deviations of up to 0.44 % can be observed for the H2NG mixtures.

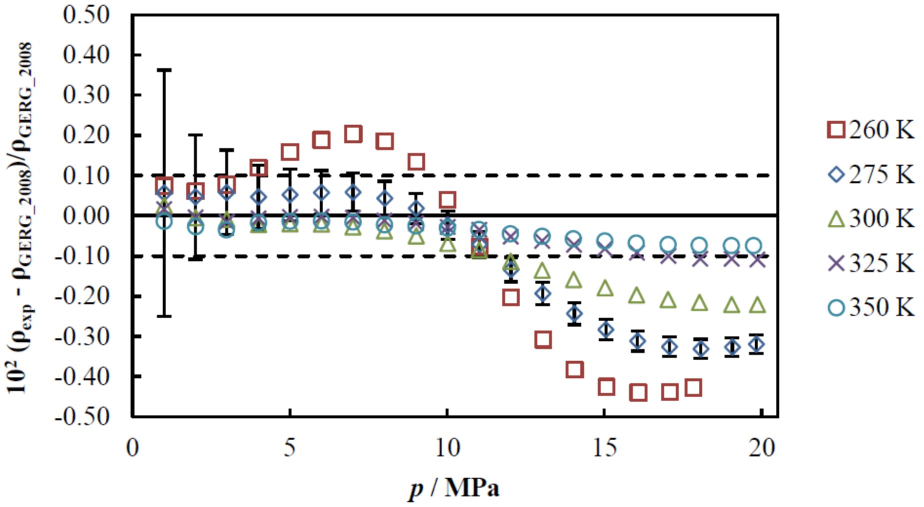

Fig. 1. Relative deviations of experimental density of a H2NG mixture with 10 % H_2 from density values calculated from GERG-2008 EoS for different temperatures and pressures. Dashed lines indicate the expanded ($k = 2$) uncertainty of the EoS. Error bars on the 275 K data set indicate the expanded uncertainty of the experimental density.

Acknowledgment

This work was funded by the European Metrology Programme on Innovation and Research (EMPIR), project 19ENG03/g07 MefHySto; and by the Regional Government of Castilla y León (Junta de Castilla y León), by the Ministry of Science and Innovation MICIN, and the European Union NextGenerationEU / PRTR.

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Towards accurate, high-throughput predictions of the thermodynamic properties of solids

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Absence of reliable experimental data for the thermodynamic properties of solids is a major challenge in electrolyte modelling and materials science. Obtaining accurate and reliable values for these properties is important in several chemical contexts including electronic waste recycling, thermal energy storage, carbon capture and storage and pharmaceutical production. In recent decades, electronic models rooted in quantum mechanics (QM) have paved the way for highly predictive simulations of solid-state thermodynamics [1]. Nonetheless, widespread use of these QM models is hindered by their complexity and computational expense, while novel atomistic models still lack the desired chemical accuracy [2]. Our work on an efficient methodology for high-throughput prediction of the temperature-dependence of thermodynamic properties based on the harmonic approximation (HA) for solids [3] along with the development of a novel model for the heat of formation will be presented and discussed. We find that the HA provides excellent quantitative description of the enthalpy and Gibbs free energy of numerous solids, suggesting that this may be a powerful and resourceful method for predicting, verifying and improving upon experimental values for the thermodynamic formation properties of solids. Upcoming work includes application of predicted thermodynamic properties to solid-liquid phase equilibria of thermal energy storage materials.

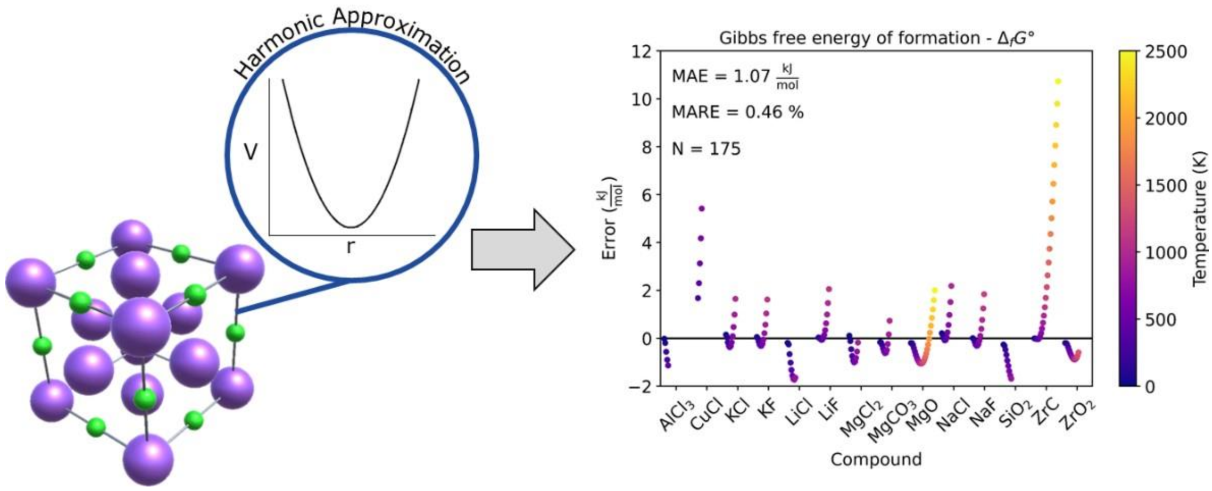

Fig. 1 (Left) The harmonic approximation in a solid, (Right) The error in the temperature-dependence of the Gibbs free energy of formation when using QM-based HA calculations [3].

Acknowledgment

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Passive Radiative Cooling Technologies for school and company buildings in Rwanda

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The effects of global warming increase the need for cooling systems worldwide. The topic of passive radiative cooling (PRC) technologies is gaining in relevance especially in developing and emerging countries where climate makes it difficult to work and learn indoors. The aim of the new cooperation project between Bundesverband Großhandel, Außenhandel, Dienstleistungen e. V. (BGA), the Business Scout for Development program of the Bundesministerium für wirtschaftliche Zusammenarbeit und Entwicklung (BMZ) and Physikalisch-Technische Bundesanstalt (PTB) is the development and implementation of PRC technologies, in particular the investigation of the cooling effect of special white paints on roofs of buildings. As part of the Cool White project, several school and company roofs in Rwanda and South Africa are being painted white together with local painters. Lower temperatures are expected to not only improve conditions for students and workers in these countries, but also enable energy savings.

PRC materials can effectively reflect solar radiation thanks to their optical properties, but at the same time dissipate heat through the 8 µm to 13 µm infrared transparency window by using space as a cold and renewable heat sink. This makes it possible to achieve sub-ambient temperatures even in direct sunlight without the need for electricity. An accurate determination of the thermophysical properties of PRC materials, such as reflectance and emittance, is the key to optimal and correct material selection, as well as effective optimization and development of PRC technologies. These optical properties should be measured in the broad spectral range from 250 nm to 50 µm encompassing both the solar spectrum (250 nm – 2500 nm) and the infrared transparency window of the atmosphere (8 µm – 13 µm).

PTB will bring its expertise in determining optical properties to investigate different white colors, select the best possible ones and provide training to the local Rwandan metrology institute on how to attach the temperature and humidity measurements to the roofs. As first results, we will show the emissivity data of 8 different PRC coatings available on the local market and the temperature data of the unpainted and painted buildings in Rwanda.

Thermal radiative properties of alumina from solid rocket plume according to optical properties and particle size

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This research investigated the influence of optical properties and particle size on the thermal radiative properties of alumina particles from solid rocket plume. Thermal heating generated from the plume of solid propellant rockets is important design factor in thermal protection system. And since this heating is mainly due to radiative heat transfer, it is significant to study the radiative properties of alumina, which plays a key role in plume radiation. For the effect of optical properties on the radiative properties, several data were carefully gathered and converted into the radiative properties with radiation calculation method such as Mie theory and etc. Subsequently, the radiative properties were calculated in accordance with particle size and temperature variation. As a result, the difference between optical properties for wavelength integrated efficiency factor is up to more than one order of magnitude for the 10 ㎛ particle at a temperature of 2320K, which shows that proper selection of optical properties matters. It is also observed that the particle emissivity increases as particle diameter increase at the same temperature and this tendency is applied to the temperature.

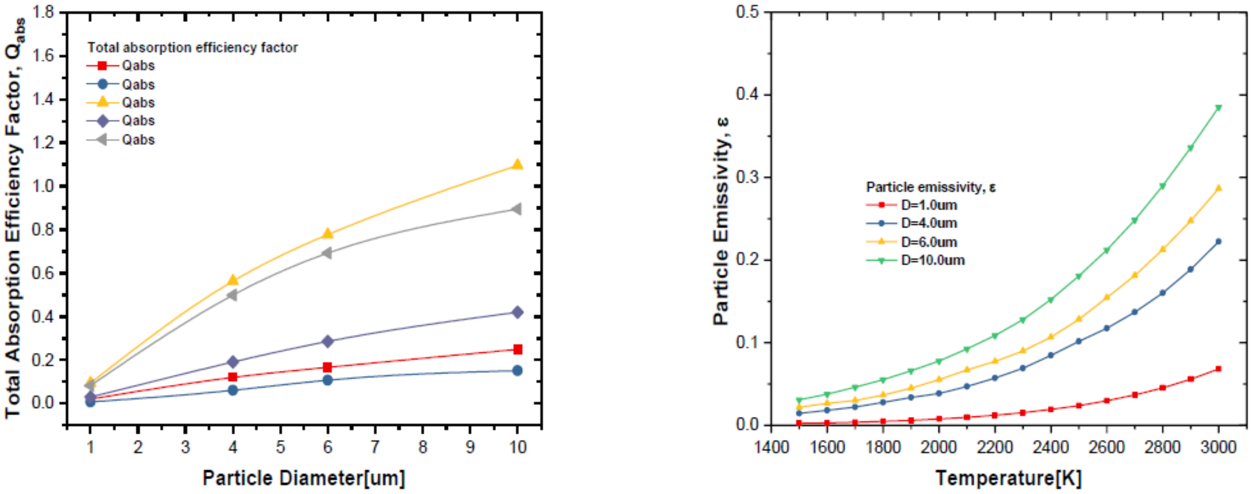

Fig. 1. Total (wavelength length integrated) absorption efficiency factor according to optical properties(left) and total particle emissivity for different particle diameter and temperature(right)

Table 1. Absorption and scattering coefficients for mixed particle distribution

Researcher	Optical properties (n − iκ), (n is refractive index, κ is absorption index)
Bakhir et al.	$\kappa = 0.002(0.06\lambda^2 + 0.7\lambda + 1)exp(1.847(T^* - 2.95))$, $T^* = T/1000$ (λ is wavelength, T is in K)
Konopka et al.	Experimental data (κ, n)
Kuzmin et al.	$\log \kappa = -2.19 + 0.089\lambda^{0.95} - 0.00056(3200 - T)\lambda^{-0.45}$ n $= 1.747 + 0.0066\lambda - 0.0068\lambda^2 + 0.00003 \times T$
Dombrovsky.	$n = [1 + \lambda^2 \left(\frac{1.024}{\lambda^2 - 0.00376} + \frac{1.058}{\lambda^2 - 0.01225} + \frac{5.281}{\lambda^2 - 321.4}\right)]^{0.5} \times [1 + 0.0202(T^* - 0.473)]$, $T^* = T/1000$

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Infrared emissivity of noble metals and the anomalous skin effect

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We report novel experimental results on the emissivity of pure metals using the new Reduced Background Calibration Facility 2 (RBCF2) at PTB [1] and corroborated at UPV/EHU using an upgraded version of its HAIRL emissometer [2]. The optical properties of noble metals (Cu, Ag, Au) can be described using the classical freeelectron optical theory with an additional term to account for the extra emission due to the anomalous skin effect [3]. Good-quality emissivity data was obtained at 473 K, which could be reproduced using the extended freeelectron theory, with only the surface specularity parameter p as a free variable. The importance of controlling roughness and surface contamination for obtaining reproducible data for these materials was also revealed, as well as differences between bulk and film samples. Tentative differences are observed between gold (which suggests better agreement to a $p = 1$ specular surface) and silver and copper (which are closer to the predicted values for $p = 0$ diffuse surface). A weighted linear regression of the spectral curves suggests that the results for gold and silver are statistically significant, whereas those of copper show a larger discrepancy with the theoretical prediction for $p = 0$, although still within the theoretically allowed range.

Acknowledgment

This work was supported financially by the Basque Government (grant numbers PRE-2019-2-0191, POS2021-2-0022, PIBA-2021-1-0022) and the University of the Basque Country (GIU19/019).

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Molecular Interactions of dihydrolevoglucosenone (DLGN) and C_2-C_5 *n*-alcohols

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New data of density (□), viscosity (η) and speed of sound (u) of liquid pure and their mixture have been determined for the binary mixture of {dihydrolevoglucosenone (DLGN) + ethanol, or + 1-propanol, or + 1-butanol, or + 1-pentanol} over the entire composition range at T = (288.15 - 303.15 K) and p = 92.71 kPa. The densities and speeds of sound of pure components and their solutions were measured using a commercial density and speed of sound analyzer, manufactured by Anton Paar (Model DSA 5000), which was calibrated with air and high-purity water. The equipment measures the speed of sound at a frequency of 3 MHz. The combined expanded uncertainty in the density and speed of sound measurements were 1.0 kg·m^{-3} and 1.1 m·s^{-1}, respectively. The temperature was automatically kept constant with an uncertainty of 0.02 K using the Peltier device built in the densimeter. The viscosities of pure components and their solutions were measured using a viscometer manufactured by Anton Paar (Model SVM 3000/G2). The reproducibility was ± 0.35% for the viscosity measurements and ± 0.02 K for the temperature measurements. The results were used to calculate the excess molar volume (V^{E}_{m}), the isentropic compressibility deviation (Δ□), viscosity deviation (□□), and Gibbs excess energy of activation
(ΔG^{E*}), which were correlated by a Redlich-Kister polynomial function. The excess molar volumes values were also used in the estimation of the partial molar volumes, excess partial molar volumes and apparent molar volumes. The empirical models developed by Grünberg-Nissan, Heric-Brewer, Katti-Chaudhary and van der Wyk were used to correlate the experimental viscosity data. Furthermore, FT-IR spectroscopy study was performed to explain possible interactions by hydrogen bonds between the compounds present in the mixtures. A Fourier Transform Infrared Spectroscopy Thermofisher Nicolet iS50 was used to obtain IR spectra. The spectroscopic analysis confirmed specific interactions through hydrogen bonds between the DLGN and the alcohols present in the mixtures. Finally, Density Functional Theory (DFT) calculations were performed to understand the intra- and inter- interactions between the compounds present in the mixtures.

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Towards an open database of optical and radiative data

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The digitalization of scientific journals and equipment has led to a huge increase in the digital data available for researchers. However, in order to ensure the availability of such a considerable amount of data, it is necessary to not only display them in figures or compile them in non-programmatically accessible tables, but to process and standardize the results of experimental works, and collect them in databases. For example, crystallographic data is usually stored and distributed using CIF files, which has permitted the development of databases such as the Protein Data Bank (PBD) and the Crystallography Open Database (COD) [1, 2]. Moreover, some of them also follow the open data philosophy: they allow users to access the original CIF files at no cost. Unfortunately, no standard file format has been established to record the results from thermo-optical experiments, and the availability of unified digital data is anecdotal [3].

We present the first approach to a standard file format definition and to a prototype of an open database of optical and thermal radiative properties: emissivity, reflectivity, absorptivity and transmittance. On the one hand, the file standard allows gathering all the information related to an experiment in a single file. On the other hand, the database can be accessed through a web application and it has a search engine to find data by multiple fields, such as material, type of experiment, author, article DOI, etc. Each entry contains information related to the publication metadata and an interactive data visualization tool. Additionally, other important details will be reported: uncertainty calculation methodology, sample purity, surface roughness, thickness, and thermal history. It is an open database: all data shown at the web application is available to download. Thus, the experimental data are linked to, but not constrained by, its original publications; and they can be reinterpreted, replotted and reused. That facilitates inter-comparisons between research teams, measuring methods and uncertainty budgets, and provides the industry with high-accuracy digital data.

In the future, the standard file format for optical and radiative data will need to be further developed to ensure that it is robust enough to avoid ambiguities, and that it provides an adequate flexibility to accept different materials and techniques. Regarding the database itself, researchers from around the world will easily access and share their data, allowing for greater collaboration and progress in the field of thermal radiation studies. Additionally, an open database will help to ensure the reproducibility of results and increase transparency in the scientific process, further strengthening the credibility and reliability of thermophysical research.

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Molecular dynamic simulation on the enhancement of the thermal physical properties of molten carbonates

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Carbonate molten is considered to be a potential heat storage material in concentrated solar power (CSP) station because of its wide working temperature range, low price, high heat of dissolution, low corrosion and other excellent characteristics. The specific heat capacity and thermal conductivity of molten salts have a significant impact on the efficiency of thermal transfer and storage. In this work, composite energy storage materials based on amorphous SiO_2 nanoparticles and binary carbonate were proposed and designed. Molecular dynamics method was used to calculate the specific heat capacity and thermal conductivity of carbonate molten-based nanofluid, and explore the microscopic enhanced mechanism of nanoparticle to heat storage and thermal conductivity. The results show that the thermal conductivity and specific heat capacity of carbonate molten-based nanofluid enhances significantly than that of the pure carbonate molten, with the maximum increase of 24.38% and 55.1% respectively. The density profile of atoms indicates that a compressed interfacial layer presents around the nano-SiO_2, which result in the increase of the specific heat capacity of carbonate molten-based nanofluid. And the collision of atoms in the compressed interfacial layer could enhance the thermal conductivity. According to analyze the distribution of RDF among atoms, it is found that the addition of SiO_2 particles changes the microstructure of the molten carbonate, altering the spacing between the cathode and cation particles, which is another reason for the enhancement on the specific heat and thermal conductivity of the carbonate. This work provides a theoretical basis for the application of molten carbonate based composite energy storage materials in CSP station.

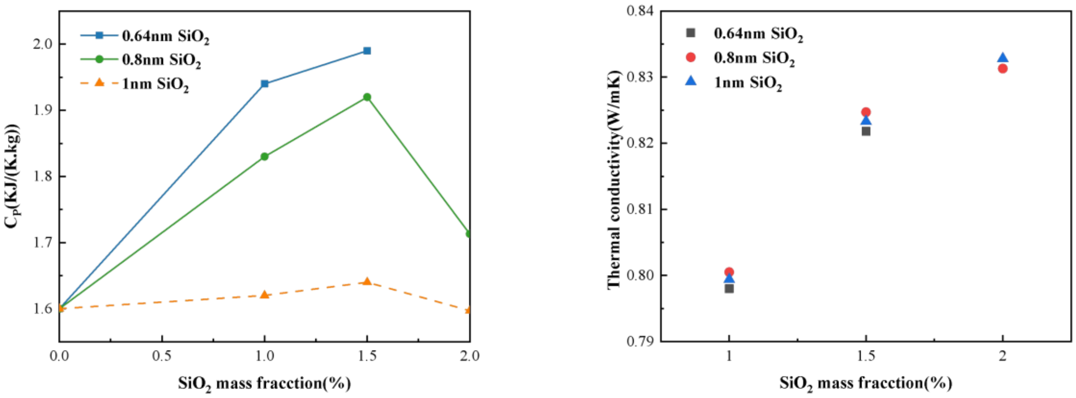

Fig. 1 The specific heat capacity and the thermal conductivity of carbonate molten-based nanofluid in different systems

Acknowledgment

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Development of Reusable Light-Weight Multi-layers TPS

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As missions in space diversify, the development of reusable vehicle such as space plane capable of flying in the atmosphere and space is becoming increasingly concrete. When flying in the hypersonic region or re-entering the earth from space, TPS (Thermal Protection System) is absolutely necessary to protect payloads and vehicle from extreme thermal environments. To develop such a TPS, it is fundamentally necessary to develop insulation and heat-resistant materials that have stable physical properties even at high temperatures. The development progress of a reusable lightweight multi-layered TPS with performance applicable to reusable vehicles or space plane is described. A reusable TPS must be durable, operable, cost-effective and lightweight with the primary function of keeping the vehicle structure within acceptable limits. Durability must be able to withstand environmental threats such as velocity impact and rain impact during handling. It must also have the ability to withstand some degree of damage without maintenance/repair [1]. The goal is to develop a multi-layered TPS tiles composed of coating, heat resistance, and insulation, which is benchmarked with latest reusable TPS TUFROC [2]. Oxide/carbon fiberbased porous materials are used as insulation layer, lightweight materials based on carbon fiber felt as heat-resistant layer, and high-emissivity waterproof glass heat resistant materials are used as coating layer. The composition was designed in such a way that an Al_2O_3-B_2O_3-SiO_2 based glass phase was formed by adding B_4C powder in certain ratio to cerak wool, an Al_2O_3-SiO_2 -based fiber, which is an oxide-based ceramic raw material for manufacturing insulation materials. The density after heat treatment was measured to be 0.35g/cm^3, and the microstructure of the insulation material produced by the shear mixer process is shown in Fig. 1. An important factor in improving the mechanical properties of ceramic tiles is the bonding of ceramic fibers, and it was confirmed that ceramic fibers, which are raw materials, were connected to each other in microstructure. Carbon fiber felt and polysiloxane, a precursor for SiOC manufacturing, were selected as heat-resistant materials. A polysiloxane slurry for impregnation was prepared by putting the polysiloxane primary ground in an alumina pestle mortar into an ethanol solvent and performing a ball milling process together with a ZrO_2 ball. The slurry was impregnated and treated in a low vacuum for a smooth penetration environment into the carbon fiber felt. The results of XRD crystal phase analysis for the heat-resistant material are shown in Fig. 2. Borosilicate and SiC are used as raw materials for the glass heat-resistant coating material. To optimize the strength, emissivity, and density of the glass coating layer, and $TaSi_2$-$MoSi_2$-SiB_6 was designed and coated. After the surface was immersed in the coating slurry to a certain depth, the coating was performed by drying, and the insulation layer and the heat-resistant layer were attached before heat treatment using RTV-650 silicone bond.

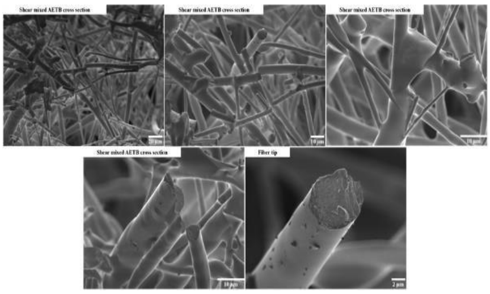

Fig. 1. Microstructure of Insulation Material.

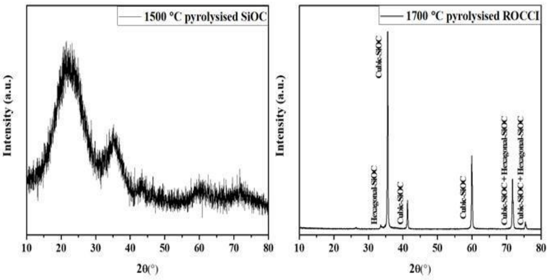

Fig. 2. XRD Formation Phase Analysis of Heat-resistant Material.

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Thermal conductivity of TPS material in vacuum condition

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A spacecraft contains various insulations or TPS (thermal protection system) materials to maintain its thermal condition as well as protect fuselage from external environment. Sometimes they occupy a high level of weight. Since weight is directly related to cost and performance of the spacecraft, it needs to be optimized. Thermal conductivity is a key property to decide thickness and weight of the insulation and is changes with vacuum levels. This paper introduces a measurement method and apparatus for thermal conductivity in vacuum condition and shows how it changes with measuring a TPS material.
The measurement apparatus uses a guarded hot plate [1] method and was firstly introduced by Kim et al. [2]. The measuring part is composed of a heater, guard, bottom plate and upper plate and it is installed in a vacuum chamber. The vacuum level can be controlled from atmospheric pressure to 0.1 Pa.
Thermal conductivity of a TPS material with 20mm thickness is measured using the apparatus in different vacuum condition. To verify its accuracy, thermal conductivity in atmospheric pressure is measured and compared using commercial measurement apparatus.

Table 1 Measurement result

Pressure [Pa]	Thermal conductivity [mW/m-K]		
	In-house apparatus	Commercial GHP	Commercial HFM
101325	79.2	79.3	79.6
50929	68.8	Not measured	
11865	64.09		
1029.25	52.69		
6.0795	43.16		
0.1149	43.15		

Table 1 shows measured thermal conductivity. In the atmospheric pressure, measured value shows very close to those of commercial equipment within 1% of relative error. It is decreased as vacuum level lowers and reached to 43 mW/m-K at 6 Pa and not decreased any further until 0.1 Pa. The minimum value is sum of solid conductivity and radiative conductivity. The difference between maximum at atmospheric pressure and minimum is around 36 mW/m-K and it is gas conductivity of contained gas in the specimen. From the gas conductivity, it is assumed that the gas is Methane which has very close thermal conductivity 34 mW/m-K at 25℃.
As shown in the result, thermal conductivity can be significantly decreased according to vacuum pressure. Therefore, if vacuum condition and corresponding thermal conductivity are considered when designing the insulation or TPS of spacecrafts, thickness and weight will be lessened. It means the performance enhancement and cost saving of whole system.

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Evaluation of moisture diffusivity from pore distribution curves: An application to a ceramic brick

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The moisture diffusivity of porous materials may strongly depend on the content of moisture in the materials, varying over an order of magnitude or even more. This dependence can be obtained by the Boltzmann-Matano method [1] that uses moisture profiles from experiments. However, such experiments are time consuming (and could have quite large measurement uncertainties). An alternative approach to evaluate the moisture diffusivity is to use measurements of the water absorption coefficient and pore size distribution for a given material [2], which take relatively short times and are rather accurate. In this paper we investigate this latter approach. We point out that a value of the diffusivity at a single value of moisture content (usually the point of saturation) is needed to properly determine the diffusivity. We also discuss the roles of small and large pores on the diffusivity and conclude that accurate experimental data on the distribution of small pores is crucial. We apply our results to an example of a ceramic brick.

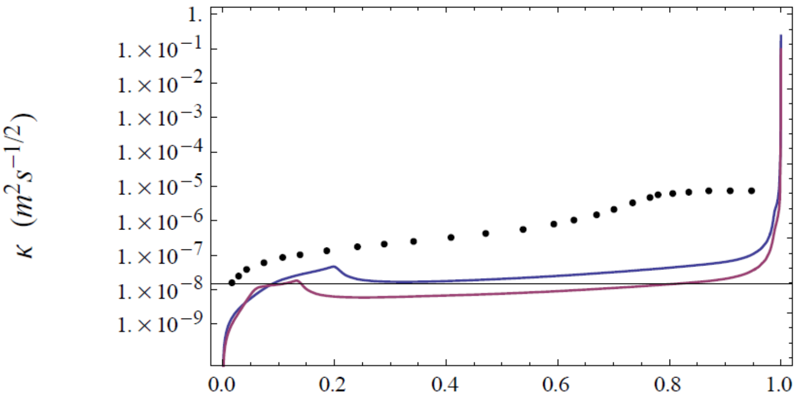

Fig. 1. The dependence of the moisture diffusivity κ on the moisture content w from the Boltzmann-Matano method (dotted) and from pore distribution curves (full).

Acknowledgment

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First results from the determination of density and dielectric virial coefficients of hydrogen and hydrogen methane mixtures

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The urgent need for sustainable energy sources particularly with regard to the climate crisis has led to increased interest in green hydrogen as a fuel. This applies to pure hydrogen, e.g. for fuel cell applications, as well as to mixtures with other substances, e.g., methane for power to gas applications with subsequent transport in the existing natural gas pipeline grid. To fully utilize hydrogen and its mixtures as a large-scale fuel source, accurate knowledge of the thermodynamic properties is essential. Since the underlying data of the currently valid fundamental equation of state for pure hydrogen by Leachman et al. from 2009 often dates back to the 1960s, we see the urgent need for verification and improvement [1]. We present a new apparatus particularly designed for the determination of the density and dielectric virial coefficients of hydrogen and later hydrogen-methane mixtures, using a combination of dielectric constant gas thermometry (DCGT) and the Burnett method. Initially, DCGT is used to obtain the molar polarizability and DCGT-virial coefficients, which are then complimented by expansion measurements. From the pressure and capacitance ratios, the dielectric and density virial coefficients are determined separately whereas knowledge of the absolute particle density is not required. The setup is a further development of the apparatus published in [2]. It also consists of four temperaturecontrolled measurement chambers, each equipped with a cylindrical capacitator and interlinked with a gashandling system to enable the expansions. In contrast to the previous apparatus, the expansion valves are inside the vacuum bell and are kept at measuring temperature while the volume of the gas-handling system (including the pressure sensors) which is stabilized to ambient temperature is further minimized. A similar approach utilizing the Burnett method exclusively was used by Sakoda et al. in 2012 to measure the density virial coefficients of hydrogen for temperatures between 353 K and 473 K [3]. We target a lower temperature range from 235 K to 350 K which is more relevant for the distribution around ambient temperature and the starting point for hydrogen liquefaction. The latest Burnett measurements in this temperature range have been conducted in the 1960s by Michels et al. [4]. First results of our measurements as well as experimental details and progress are shown and compared to the available literature.

Acknowledgment

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Prediction of the thermal conductivity of $H_2/CO_2/CO/CH_4/H_2O$ mixtures at high temperatures and high pressures based on the extended corresponding states principle

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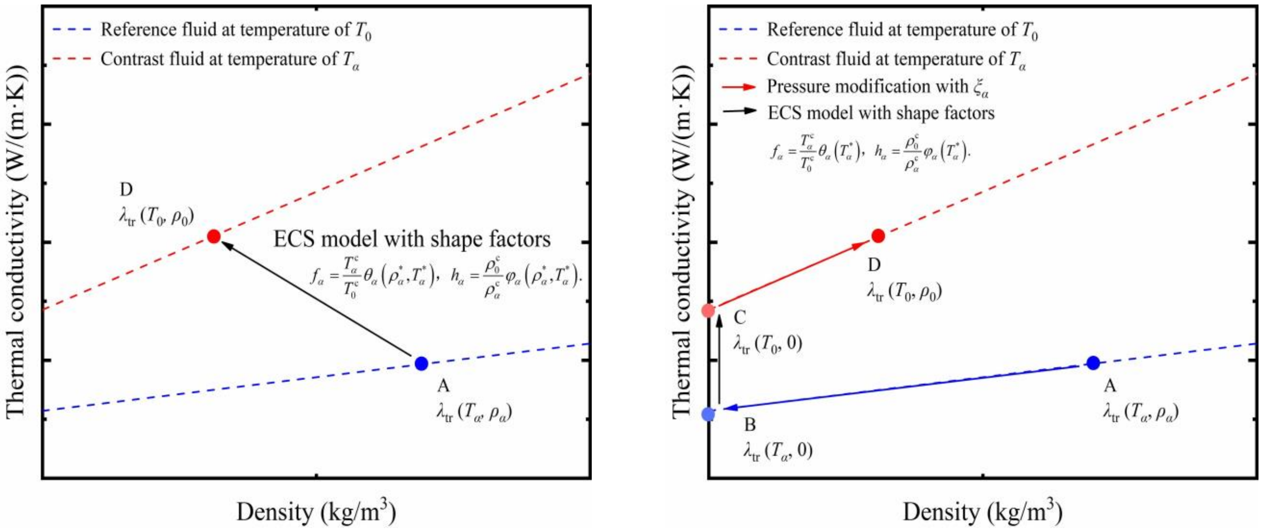
An improved extended corresponding states principle for predicting the thermal conductivity of $H_2/CO_2/CO/CH_4/H_2O$ mixture is presented. The model uses hydrogen as a reference fluid and employs shape factors and a density-modified parameter. Calculations for the thermal conductivity require only critical constants, molecular weight, the ideal gas heat capacity, the dilute gas viscosity and mole fraction for each mixture component as input. The model was tested for pure fluids, binary hydrogen-containing mixtures, a binary non-hydrogen-containing mixture, and quinary mixtures at temperature up to 915 K. The average absolute deviation between experiments and predictions is less than 4.52%. The present model is suitable for prediction at temperatures lower than 1000 K and pressures lower than 20 MPa with an uncertainty of 6.12% (k=2), which is necessary for implementation of hydrogen generation systems and has potential to be applied to more species and mixtures.



(a) (b)

Fig.1. Calculation difference between one-step method and three-step method:
(a) One-step method. (b) Three-step method.

Acknowledgment

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Thermodynamic-Based Crystallization Process Design for the Ultra-Purification of Organic Chemicals

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Melt crystallization is a promising separation technique for producing ultra-pure chemicals and reducing carbon emissions by increasing process energy efficiency. This Study presents a thermodynamic framework-based melt crystallization process for the purification of organic chemicals. Solid-liquid phase equilibrium was measured using synthetic and thermal methods and calculated using the excess Gibbs energy models to indicate the thermodynamic feasibility of the purification process. We carried out the crystallization process using a suspension-based melt crystallization technology in a continuous crystallization process by constructing a 1-liter lab-scale continuous crystallization system and analyzing the process conditions and particle size distribution for each crystallizer. By varying the residence time and slurry density, we determined the effect of these parameters on the median size of crystals and estimated the kinetic parameters based on the measured CSD. The scraper rotational speed was also varied to ensure adequate mixing and prevent agglomeration. The results showed that increasing the residence time and slurry density led to an increase in the median crystal size, while the scraper rotational speed had no significant effect. The purity of the crystals was found to be correlated with the initial composition of the product and impurities and the median crystal size. This study presents a thermodynamic framework-based melt crystallization process for purifying organic chemicals. We used suspension-based melt crystallization technology to construct a lab-scale continuous crystallization system and varied process parameters to determine their effect on median crystal size and estimate purity of the product. Results showed the purity of crystals was correlated with initial composition and median crystal size. Melt crystallization is a promising technique for producing ultra-pure chemicals and reducing carbon emissions.

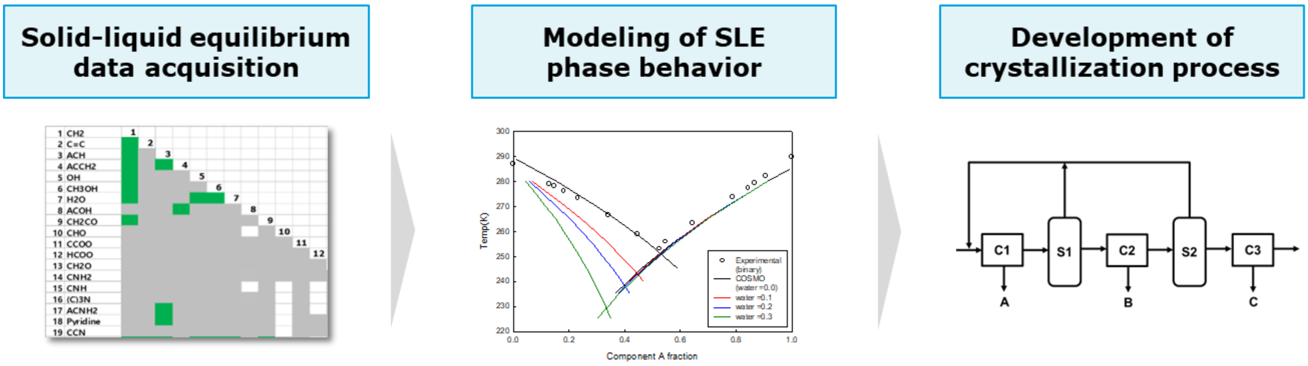

Fig. 1. Workflow of development of a crystallization process.

Table 1. Analysis of crystal purity as a function of crystallizer temperature, feed residence time, feed flow, and agitator speed, in a continuous crystallizer.

Composition	Temperature (Kelvin)	Residence time (min)	Agitator (rpm)	Slurry handling	Purity	CSD (*um*)
1	270.85	233	60	O	94.41	-
2	270.15	140	60	O	94.44	607.79
3	269.55	70	60	O	95.92	499.63
4	270.05	35	90	O	92.80	533.45

Thermodynamic Models and Properties for Optimal Entrainer Selection in Extractive Distillation Processes

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Extractive distillation is a critical process in the chemical industry for separating and purifying mixtures with closeboiling points. The aim of this study was to compare different properties for entrainer selection, including experimental data and thermodynamic models like UNIFAC and COSMO-SAC, to select an appropriate entrainer and thermodynamic model for extractive distillation. Accurate thermodynamic data, including vapor pressure, activity coefficients, and VLE data, were crucial in making this selection. Our results demonstrate that selecting a reliable entrainer and thermodynamic model significantly enhances the efficiency of extractive distillation. This information can assist in designing and optimizing distillation processes for various industries. In this study, we compared different properties for entrainer selection, including experimental data and thermodynamic models like UNIFAC and COSMO-SAC. Accurate thermodynamic data such as vapor pressure, activity coefficients, and VLE data were considered to select an appropriate entrainer and thermodynamic model for extractive distillation. The results indicate that using reliable data and selecting a suitable thermodynamic model can significantly enhance the efficiency of extractive distillation. Selecting a reliable entrainer and thermodynamic model is crucial for designing and optimizing extractive distillation processes. Our findings provide essential information to assist in making informed decisions for designing and optimizing distillation processes. [1-2].

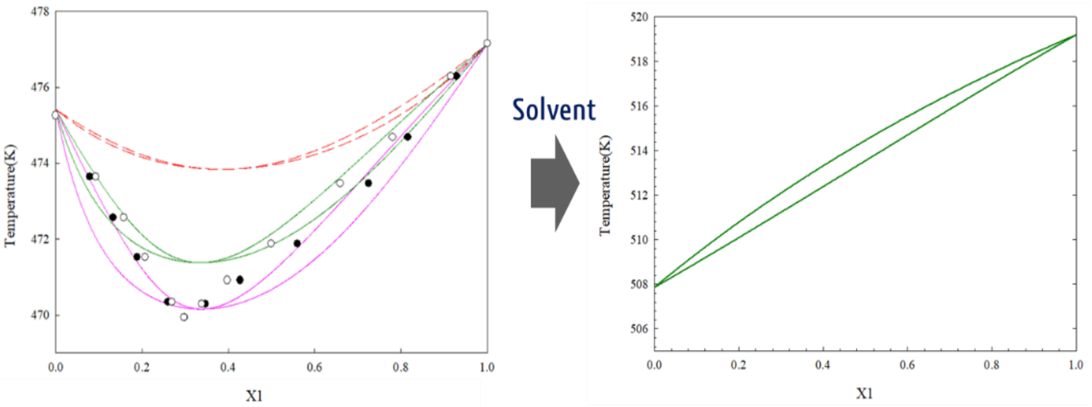

Fig. 1. The binary VLE behavior with appropriate entrainers.

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Volumetric Proprieties of Ternary Mixtures Containing 1-Chlorobutane , N-heptane and Ketone Measurements and Prediction

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The purpose of this work is the determination of the excess volume of ternary mixtures containing 1chlorobutane, n-heptane and Ketone. This work is part of a systematic program of research for the measurements and predictions of physical properties of ternary mixtures containing halogenated hydrocarbons, n-alkane and Ketone.

The present work reports experimental excess volume of 1-chlorobutane+n-heptane+hexan-2-one or cyclohexanone at the temperature of 288,15 K, 298,15 K, 308,15 K and atmospheric pressure.

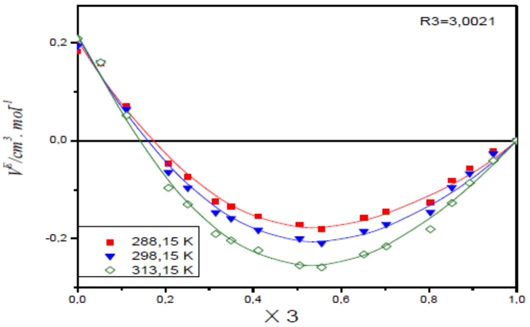
Excess molar volumes were determined from the densities of the pure liquids and mixtures, measured with an Anton Paar DMA 5000 vibrating tube densimeter. The apparatus was calibrated at atmospheric pressure before cach series of measurements using bi distilled and degassed water, density data were taken from litterature for water [1].

For cach ternary, the experimental data of V^E corresponding to the three binary mixtures were fitted to the variable degree polynomials suggested by Redlich-Kister [2]

Experimental excess molar volumes for the Ternary mixtures were fitted to the Nagata and Tamura equation [3]:

$$V_{123}^{E} = V_{bin}^{E} + x_1x_2(1-x_1-x_2)(B_0 + B_1x_1 + B_2x_2 + B_3x_1^2 + B_4x_2^2)$$

Experimental excess molar volumes data are compared with theoritical prediction using Prigogine-FloryPatterson model [4-8].



Excess molar volume for
1-chlorobutane (1) +n-heptane(2) + cyclohexanone(3)
at differente temperatures

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A Novel Sensitivity Model for High Temperature Thermopile Heat Flux Sensor with Air Gap

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Recent advancements in heat flux measurement have resulted in the development of a robust High Temperature Thermopile Heat Flux Sensor(HTTHFS) with air gap intended for use in extreme thermal environments in which the uncooled sensor temperature reaching up to 1000◦C[1].

The HTTHFS considered in this study are referred to as "total" heat flux sensor responding to all three modes of heat transfer[2], especially the intense radiation heat transfer between the surfaces of the air gap at elevated temperature, which significantly increases the nonlinearity and temperature dependence of the heat flux. Therefore, it is difficult to adapt the traditional calibration method with a given sensitivity coefficient to this form of heat flux sensor with a wide temperature range.

This article presents proposed a novel sensitivity model and parameter identification method for calibrating the HTHFS and other uncooled differential heat flux sensors at elevated sensor temperatures with minimal uncertainty. First, the theoretical sensitivity model of the HTHFS is established using simplified node thermal network based on numerical simulation results, indicating that the excellent insulation performance of the air gap contributes significantly to maintaining a suitable temperature difference range under high heat flux conditions, and the primary cause for the sensor's output temperature dependence is the change in the radiant heat transfer between the air gap and the thermal conductivity of the armored structure with temperature.

Then, the heat flux correction factor between the received and the absorbed radiant heat flux is derived accounting for the different temperature and viewing angle compared to the water-cooled Gordon meters on the other side of the slit heat source, which is very important to cut down calibration errors under high temperature conditions[34].

Last, the parameters of the simplified node thermal network of the heat flux sensor sensitivity model are identified using Least Squares identification methods based on the calibration test data set including seven operating conditions over the hot wall temperature range from 700K to 1400K. The deviations between model predictions and experimental results are within the range of 5%, which shows that the sensitivity model obtained for the HTHFS effectively characterized its temperature nonlinear dependence with acceptable uncertainty limits.

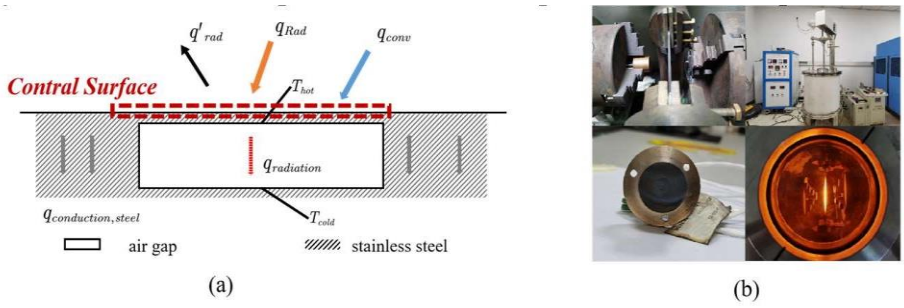

Fig. 1. (a) Heat transfer schematic; (b) Secondary calibration experiments in a vacuum blackbody furnace.

Acknowledgment

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Young’s Modulus of Illitic Clay and $CaCO_3$ Mixtures During Thermal Treatment up to 1200 °C

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Traditional ceramics products have a dominant position in the market. Their production is based mainly on the use of clay minerals. The most common clay mineral in building ceramic materials is illite. It is known that the addition of a CaO source allows the formation of the anorthite mineral. Mainly, this is used in the production of kaolin-based porcelain, where anorthite porcelain can be produced at low temperatures, and in addition with low shrinkage during a firing. This small shrinkage allows higher heating rates to be applied without damaging of the ceramic body. Therefore, anorthite formation can speed up production and help to reduce its costs. The production of ceramic materials based on illitic clays (compared to kaolinitic clays) does not focus on supporting the crystallization of anorthite. However, several studies [1–5] conducted on calcareous illitic clays and their mixtures with CaO-containing waste materials suggest that increasing formation of anorthite leads to improved performance of ceramic materials. The formation of anorthite improves mechanical strength of the material, reduces a porosity, and a shrinkage during a firing [1, 4, 5]. The most commonly used waste materials, which content CaO are: ash, granite dust, marble dust, and waste limestone [1–5]. The idea of adding CaO to ceramic materials is not new. CaO-containing ceramics have been produced for centuries, but CaO was not added intentionally, but as a natural component of illitic clays [1].

The aim of this paper is to study the effect of the amount of calcium carbonate ($CaCO_3$) on the Young’s modulus of illitic samples in the temperature interval from 25 °C to 1200 °C. The samples are made by dry pressing from illitic clay and different $CaCO_3$ contents, namely 17.6 wt.%, 19.6 wt.%, 21.6 wt.%, 23.6 wt.%, and 25.6 wt.%. The measurements of the dimension, mass, and resonance frequency of the samples are measured from which it was possible to calculate the Young’s modulus of the samples. The thermal expansion of samples is studied by thermodilatometry (TDA) using a push-rod horizontal dilatometer Netzsch DIL402. The measurements are performed in nitrogen atmosphere with a flow rate of 50 mL/min. The mass change is measured by thermogravimetry (TG) using Mettler Toledo TGA/SDTA apparatus. The measurements are carried out in air atmosphere with a flow rate of 50 mL/min. The resonant frequency is measured using an apparatus based on the impulse excitation technique (IET) in a static air atmosphere. All measurements are performed from 25 °C to 1200 °C with a heating rate of 5 °C/min.

Obtained Young’s modulus curves had approximately the same trend, because the same reactions and processes occurred in the studied samples. The differences are only at higher temperatures (1000 – 1200 °C). The highest value of the Young’s modulus (17.4 GPa) at the final firing temperature (1200 °C) is recorded in the case of the sample with a $CaCO_3$ content of 17.6%. On the contrary, in the case of the sample with a $CaCO_3$ content of 25.6%, the value of Young’s modulus stabilizes at 13.8 GPa. The values of Young’s modulus of studied samples increase 7-10 times during a firing. Based on the obtained results, it can be concluded that the firing of the samples at 1200 °C has a significant effect on the improvement of the mechanical properties of the studied samples.

Acknowledgment

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Phase diagrams study of choline salicylate derivative –salt-based aqueous biphasic systems for caffeine extraction

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Ionic liquids (ILs) are a modern class of ionic solvents composed of organic cations and organic or inorganic anions whose thermal and physical properties can be tuned for various applications. In this work new Choline salicylate derivatives based ILs such as Choline salicylate, [Ch][SA]; Choline para-methyl salicylic acid, [Ch][pMSA]; Choline para-Amino salicylic acid, [Ch][p-ASA]; and Choline Acetyl salicylic acid, [Ch][AcSA] were synthesized and characterized by various techniques such as NMR, IR, TGA and HPLC-MS. These ILs are used to investigate their ability to form aqueous biphasic systems (ABS) for application in the extraction of caffeine. The choice of various salicylic derivative is to investigate the potential of IL anions in the formation of ABS with the tripotassium phosphate salt (K_3PO_4).

Choline-based biphasic systems (ABS) have a great interest because of their "green" separation processes for the extraction of value-added compounds. The used ABS system "IL+ salt + water" was determined at 298.15 K & atmospheric pressure and the respective solubility curves, tie-lines, and tie-line lengths are reported. The trend in capability of forming ABS was observed in the following order: [Ch][AcSA] > [Ch][p-ASA] > [Ch][p-MSA] > [Ch][SA]. The solubility curve was fitted to the Merchuk equation and tie lines were calculated from mass phase fractions based on the lever arm rule that are described using the Othmer-Tobias and Bancroft equations. Also, the ABS is evaluated through its extraction capability for caffeine using partition coefficient which found to be greater than one in all studied systems indicating an efficient extraction.

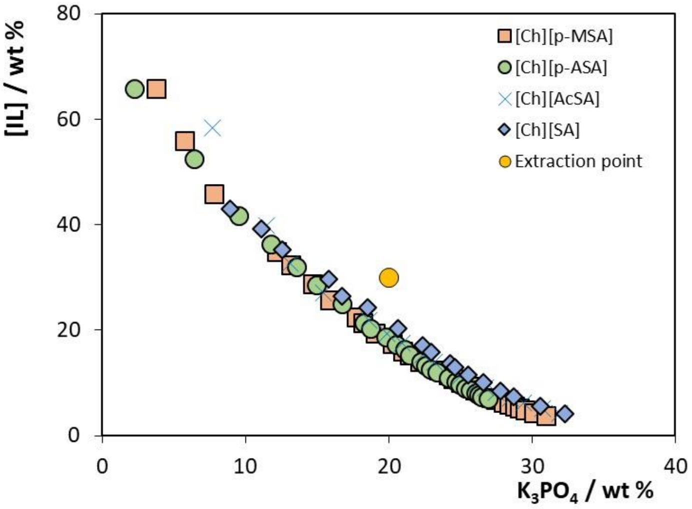

Fig 1: Ternary phase diagrams for systems composed of IL + salt + water at 298.15 K and atmospheric pressure: (■) [Ch][p-MSA], (●)[Ch][p-ASA], (×)[Ch][AcSA], and (♦)[Ch][SA]

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Property Calculation Libraries and Software for Working Fluids in Energy Conversion Processes

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The program libraries for calculating thermophysical properties of water and steam, mixtures with water and steam, and other working fluids are designed for practical use by engineers calculating heat cycles, steam or gas turbines, boilers, heat pumps, refrigerators and other energy conversion processes. Thermodynamic properties, transport properties, thermodynamic derivatives and inverse functions can be calculated.
The following property libraries are presented:

LibIF97 for water and steam, *LibIF97-META* for metastable steam, *LibICE* for ice, *LibSeaWa* for seawater,
LibHuGas for humid combustion-gas mixtures also at high pressures,
LibHuAir for humid air also at high pressures and with high water content,
LibAmWa for ammonia/water mixtures in absorption processes,
LibWaLi for water/lithium bromide mixtures in absorption processes,
LibIdGasMix for 25 ideal gases and their mixtures,
LibRealAir for real dry air,
LibCO2 for carbon dioxide including dry ice, *LibNH3* for ammonia,
LibPropane for propane, *LibButane_Iso* and *LibButane_n* for iso-butane and n-butane,
LibD4, *LibD5*, *LibD6*, *LibMDM*, *LibMD2M*, *LibMD3M*, *LibMD4M*, and *LibMM* for siloxanes used in ORC processes,
LibCH3OH for methanol, *LibC2H5OH* for ethanol,
LibH2 for hydrogen, *LibN2* for nitrogen, *LibHe* for helium, and
LibSecRef for liquid coolants.

In addition, property libraries for a number of refrigerants and hydrocarbons are available.
These libraries contain accurate and fast algorithms currently available for calculating thermodynamic and transport properties.
For extremely fast property computations in CFD or simulations of transient processes, property libraries that use the Spline-Based Table Look-up method (SBTL) are available.
The property libraries can be used in user-specific programs written in Fortran, C/C++, C#, Java, Python, Visual Basic or other programming languages on Windows, Linux or Mac OS.
In addition, add-ons for the use of these property libraries in Excel, MATLAB and Simulink, Mathcad, Engineering Equation Solver (EES), Dymola and SimulationX (Modelica), and LabVIEW are available.

Complex study of the thermophysical properties of a thermal insulation coating for application at elevated temperatures

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The paper deals with the problem of complex investigation of the thermophysical properties of a certain thermal protection – paint layer. The investigated layer was prepared from a Bronya F paint that is a commercially available material for application for both low-temperature protection and up to 260 °C. Available as an aqueous solution paint forms a dispersion composite layer on the protected surface. The matrix of the composite is polymer and the filling is ceramic microspheres filled with gas under reduced pressure. Therefore, the study of this structure should take into account the various phenomena of conjugate and coupled heat transfer.

The aim of the study was to provide reliable data on the values of thermophysical parameters (TP) and their dependence on temperature and thermal history. The measurements were carried out on a free-standing paint flat specimens of thickness about 1.5 mm. The complex complementary study [1-3] includes weight measurements, thermogravimetric analysis, micro-calorimetric analysis [4], and measurement of effective thermal diffusivity using the temperature oscillation technique [5]. While performing measurements the attention was focused on preserving a high temperature resolution in order to directly reproduce the temperature dependence of the parameters under investigation. Typical results obtained in a course of thermal diffusivity investigation are shown in Fig. 1. The findings are complemented with structural test data collected by means of scanning electron microscopy, surface characterization. The parameters determined in the study made it possible to determine the thermal conductivity by recalculating the specific heat, density, and thermal diffusivity data. While analyzing the experimental results, the need to distinguish between parameters in their real form, effective and apparent was taken into account. Analysis of the data allowed the material to be classified as an insulator. The manufacturer's data were also verified, the phase transformation identified around room temperature was characterized and effects of the structure exposition to elevated temperatures were described.

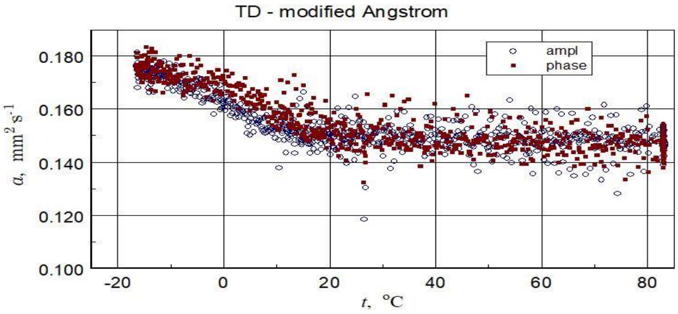

Fig. 1. Thermal diffusivity of the investigated thermal protection layer material at delivery state obtained by modified Ångström method [1, 3] from amplitude attenuation (ampl) and phase shift (phase) analyses.

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Density, speed of sound and viscosity of binary mixtures of {dihydrolevoglucosenone (DLGN) + C_2-C_5 *n*-alcohols}

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New data of density (ρ), viscosity (η) and speed of sound (u) of liquid pure and their mixture have been determined for the binary mixture of {dihydrolevoglucosenone (DLGN) + ethanol, or + 1-propanol, or + 1-butanol, or + 1-pentanol} over the entire composition range at T = (288.15 - 303.15 K) and p = 92.71 kPa. The densities and speeds of sound of pure components and their solutions were measured using a commercial density and speed of sound analyzer, manufactured by Anton Paar (Model DSA 5000), which was calibrated with air and high-purity water. The equipment measures the speed of sound at a frequency of 3 MHz. The combined expanded uncertainty in the density and speed of sound measurements were 1.0 $\text{kg}\cdot\text{m}^{-3}$ and 1.43 $\text{m}\cdot\text{s}^{-1}$, respectively. The temperature was automatically kept constant with an uncertainty of 0.02 K using the Peltier device built in the densimeter. The viscosities of pure components and their solutions were measured using a viscometer manufactured by Anton Paar (Model SVM 3000/G2). The reproducibility was ± 0.35% for the viscosity measurements and ± 0.02 K for the temperature measurements. The results were used to calculate the excess molar volume (V^E_m), the isentropic compressibility deviation ($\Delta\kappa$), viscosity deviation ($\Delta\eta$), and Gibbs excess energy of activation

(ΔG^{E*}), which were correlated by a Redlich-Kister polynomial. The excess molar volumes values were also used in the estimation of the partial molar volumes, excess partial molar volumes and apparent molar volumes. The empirical models developed by Grünberg-Nissan, Heric-Brewer, Katti-Chaudhary and van der Wyk were used to correlate the experimental viscosity data. Furthermore, FT-IR spectroscopy study was performed to explain possible interactions by hydrogen bond between the compounds present in the mixtures. A Fourier Transform Infrared Spectroscopy Thermofisher Nicolet iS50 was used to obtain IR Spectra. The spectroscopic analyzes confirmed specific interactions through hydrogen bonds between the DLGN and the alcohols present in the mixtures.

Acknowledgment

This study was financed by the Fundação Educacional Inaciana Padre Sabóia de Medeiros (FEI), Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP, Process 2009/14556-5) and Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001.

Viscosity modelling of molten salt systems utilized in heat transfer and storage applications in concentrating solar power plants

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In recent years, molten salts have received much attention due to their specific properties in a wide range of applications and are promising candidates as heat transfer fluids in heat transport and storage applications. As a result of high operating temperature ranges and sometimes existence of hazardous species, performing experiments in a wide range of temperatures and compositions is expensive, time and energy-consuming if not impossible. Due to the incomplete available experimental viscosity data in the literature, moreover, since viscosity property is directly related to the designating and pumping costs, viscosity modelling has been employed as a beneficial tool to predict the viscosity of the Na^+, $K^+//NO_3^-$, Cl^-, CO_3^{2-} reciprocal molten salt systems in this work. The modelling methodology is outlined using the system $NaNO_3$-KNO_3. The Arrheniuslike model, which was originally developed for oxide melts and adapted for salt systems in this work, is capable of describing the temperature dependence of viscosity of fully liquid sodium and potassium nitrate salts; furthermore, the composition dependence of viscosity should necessarily be considered for higher order systems since viscosity is strongly affected by the composition-induced change in structure of molten salts, where the structure is described based on the thermodynamic associate species model. By doing this, a good agreement between the experimental and calculated values have been obtained for pure sodium and potassium nitrate salts. However, in binary sodium-potassium nitrate salt systems, a positive deviation for all compositions is observed, possibly due to the network formation of sodium nitrate species (i.e. selfpolymerization effect of $NaNO_3$) since nitrates show different characteristics compared to chlorides and there is the probability of associate ion complex formation. Thus, an additional cluster formation term that takes the $NaNO_3$-induced excess viscosity into account is introduced which could further result in a good agreement between calculated and experimental viscosity values [1- 4].

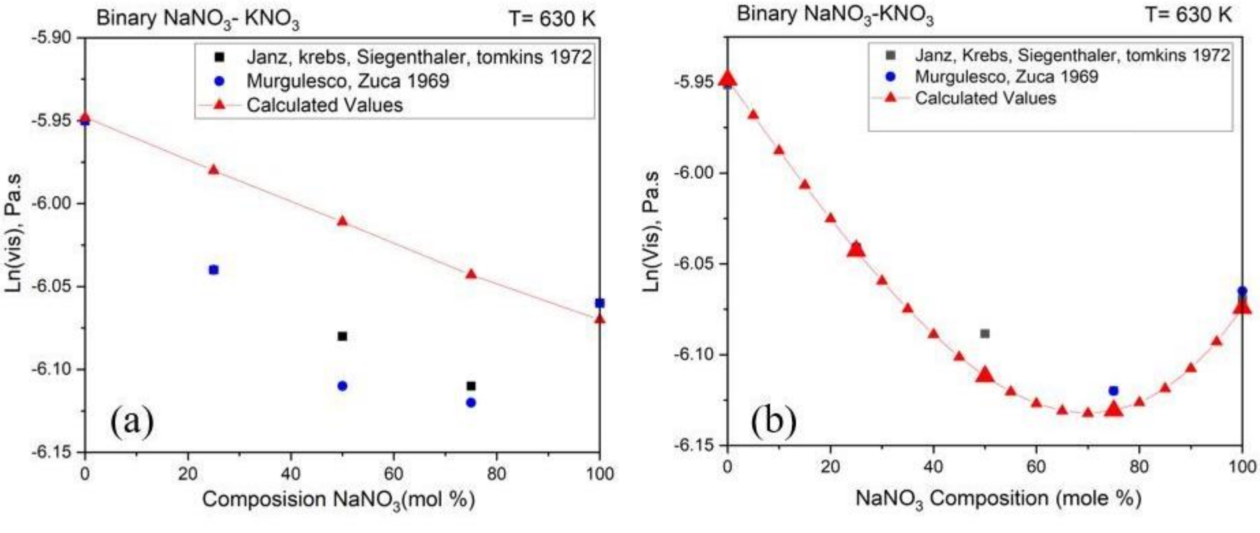

Fig. 1. Viscosity-Composition diagram for binary $NaNO_3$-KNO_3 salts system (a): before (b): after considering cluster formation term.

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Measurement of creatinine through photoacoustic spectroscopy

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Diabetes is threat to global public health. It is estimated that 10.4% of worldwide adults will suffer diabetes in 2024. Diabetic nephropathy is one of the main causes of end stage renal failure [1]. Diagnosis of renal failure is based on ureic albumin and glomerular filtration rate (GFR) [2]. It is necessary to determine creatinine in serum and urine to quantify GFR. Various blood samples are needed to assess the metabolic control and renal function in diabetes. In previous works we used photoacoustic spectroscopy (PAS) to correlate glycaemia with other variables such as oxygenated and glycated haemoglobin, and cytochrome p450 in diabetes [3]. PAS allows the measurement of different variables in the same sample. PAS is a non-destructive technology based on the following phenomena: a sample is confined in a hermetically sealed chamber where it is illuminated with a modulated light beam. The sample absorbs the incident light and heat is produced. The release of heat to the environment is periodic, showing a pressure or an acoustic wave (signal) detected by a microphone. The signal is then integrated as a spectrum in a PC [4]. The goal of the present study was to standardize the methodology to measure creatinine through PAS, using creatinine diluted in isotonic saline solution (1.56 mg/dL to 1600 mg/dL). The peak of absorption was at 263 nm (figure 1). The concentration-absorption curve (log-log) at 263 nm correlated with creatinine concentration (Spearman, $R = 1$, $p<0.0001$, figure 2). Data are shown in the figures as mean ± standard error of mean (SEM). Conclusion: PAS is a sensitive technology to detect creatinine in a small volume of sample. The possibility of measuring different variables in the same sample shows the versatility of the technology and its application not only diabetes but in other diseases.

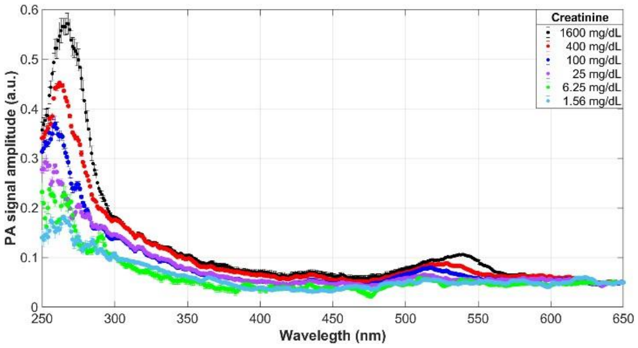

Fig. 1. PA spectra of creatinine. The figure shows the mean ± standard error of the mean (SEM) of five measurements.

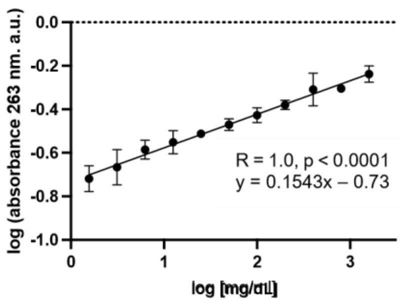

Fig. 2. Creatinine concentration - peak of absorption curve. It is shown the mean ± SEM. R= 1, p < 0.0001. a.u. = arbitrary units.

Acknowledgment

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Data reduction and uncertainty assessment of the direct pulse heating technique with both contact and radiance temperature measurements

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This work presents a data reduction procedure of the direct pulse heating technique with both contact and radiance temperature measurements. The technique is applied for the measurement of thermophysical properties of electroconductive solids over a wide temperature range, i.e., from room temperature up to 2600 K, in standard and up to above 2600 K, in extended temperature range. In the standard range, the absolute temperature of a tested material is measured by contact thermometry, i.e., by using a standard thermocouple, while the radiance temperature of the material is measured by a radiation thermometer. These and other experimental data are then reduced to the values of specific heat, specific electrical resistivity, total hemispherical emissivity and spectral normal emissivity of the tested material. In the extended range, only the radiance temperature is measured and then converted to the absolute temperature by using the extrapolated values of spectral normal emissivity [1]. The related data reduction with corresponding uncertainty assessment for each property is described in detail and discussed. The previous method for the thermocouple signal correction [2] has been improved, which gives more accurate temperature values during the material pulse heating. Also, compensation for finite response time of the applied temperature and radiation sensors has been introduced. An example of uncertainty assessment, including a recent analysis of the temperature nonuniformity along and across the tested material [3], is given for the measurement of specific heat, specific electrical resistivity, total hemispherical emissivity and spectral normal emissivity at 900 nm of a pure polycrystalline specimen of tungsten.

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Complete thermal characterization of glycerol, ethylene glycol, and olive oil by front photopyroelectric configuration by using water as a reference sample

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The thermal properties of glycerol, ethylene glycol and extra virgin olive oil (EVOO) were obtained using the front photopyroelectric (FPPE) configuration [1]. The measurements of the thermal properties of glycerol and ethylene glycol with different water concentrations are carried out. The ratio of the tangent of FPPE phases using $\alpha_s = \alpha_r$ (tan ϕ_s/tan ϕ_r)[2] as a function of frequency (f) (Fig 1a) allowed to obtain the mean value of sample's thermal diffusivity (α_s); with α_r corresponding to the thermal diffusivity of the reference; ϕ_s and ϕ_r are the PPE phase signal data for the sample and reference, respectively. Water measurements were used as reference for all samples. Figure 1b) and c) show the first and second derivatives at low frequencies. The mean value of thermal diffusivity was calculated for frequencies between 14 Hz and 19 Hz, where the values of the derivatives change less than $1\text{x}10^{-4}$ after the discontinuity.

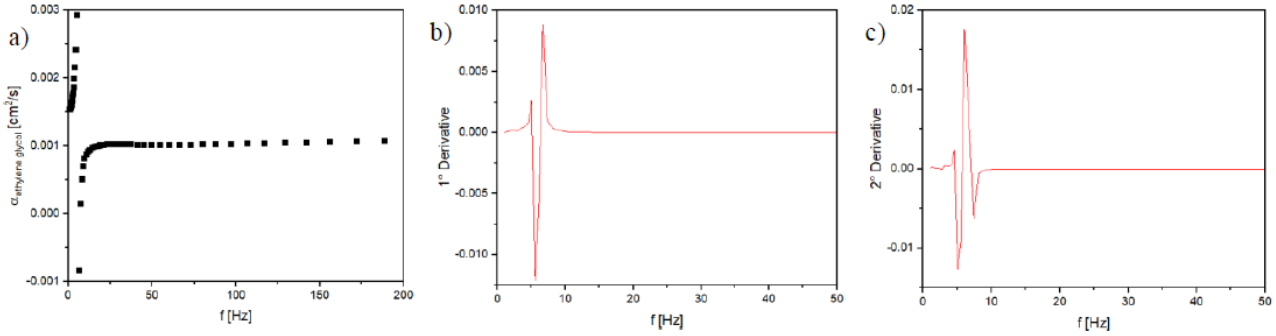

Fig. 1 a) Thermal diffusivity of ethylene glycol sample; b) and c) first and second derivatives of the thermal diffusivity for ethylene glycol, respectively.

Using $k_s = k_r$ ($\alpha_s V_r / \alpha_r V_s$)[2], the thermal conductivity of samples (k_s) were obtained; with k_r the thermal conductivity of the reference, Vr and Vs are the PPE signal amplitude for the reference and samples, respectively. From the FPPE measurements of thermal diffusivity and thermal conductivity; the thermal effusivity, e, and volumetric heat capacity of samples, ρc, were calculated and shown in Table 1.

Table 1. Thermal properties of samples obtained by FPPE

Sample	Water (reference)	Glycerol	Ethylene glycol	EVOO
α_s (x10^{-8}) [m²/s]	14.2	10.4 ± 0.2	9.9 ± 0.2	7.9 ± 0.3
k_s (x10^{-1}) [W/mK]	5.9	3.0 ± 0.1	2.7 ± 0.1	1.9 ± 0.1
e (x10^{2}) [Ws$^{1/2}$/m²K]	15.7	9.4 ± 0.3	8.7 ± 0.3	6.9 ± 0.3
ρc (x10^{6}) [Ws/m³K]	4.18	2.9 ± 0.1	2.8 ± 0.1	2.5 ± 0.1

Acknowledgment

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Study of physicochemical and thermal properties during frying of butter and clarified butter blended with vegetable oils by front photopyroelectric technique

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Butter (BT) and clarified butter (CB) from cow milk fat are commonly used for frying food due to their rich flavor; however, at high temperatures, undesirable components can be formed due to oxidation and thermal decomposition, and also both have a high content of saturated fatty acids (FA), which are considered a health risk. This can be minimized by using BT and CB blends with vegetable oils to obtain a balanced FA profile[1]. The physicochemical and thermal properties changes of BT and CB blends with extra virgin avocado (EVAO), safflower (SFO), and soybean (SBO) oils due to frying were studied. Blends with different FA profiles were analyzed by gas chromatography. Fourier transform infrared spectroscopy was used to evaluate the degradation effect of blends during frying.

Thermal properties of samples were obtained by using the front photopyroelectric (FPPE) configuration [2].

Thermal diffusivity of samples mean values (α_s) were obtained by the ratio of tangents of FPPE phases as $\alpha_s = \alpha_r$ (tan ϕ_s/tan ϕ_r)[3] as a function of frequency (f) (Fig 1a); where α_r is the thermal diffusivity of the reference; ϕ_s and ϕ_r are the PPE phase signal data for the sample and the reference, respectively. Water was used as reference for all samples. Figures 1b) and 1c) show the first and second derivatives, respectively. The mean thermal diffusivity was calculated from 14 Hz to 19 Hz, where the derivative values do not change considerably after the discontinuity.

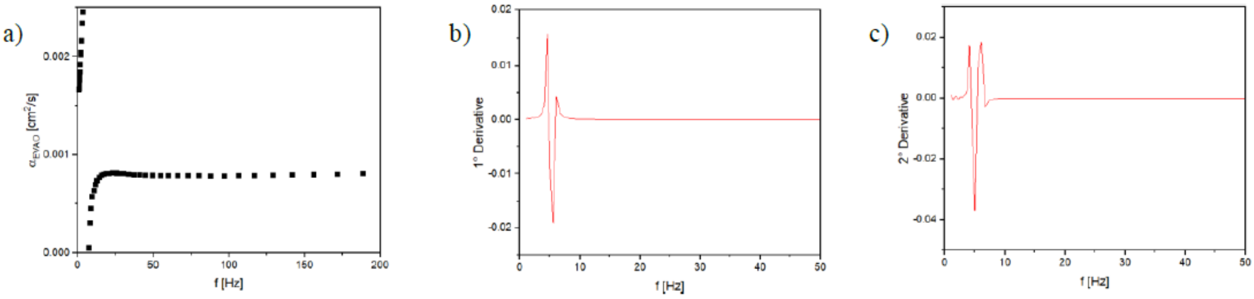

Fig. 1 a) Thermal diffusivity of EVAO sample; b) first and c) second derivatives of thermal diffusivity.

The thermal conductivity of samples (k_s) were obtained using the equation $k_s = k_r\ (\alpha_s V_r/\alpha_r V_s)$[3], where k_r is the thermal conductivity of the reference, Vr and Vs are the PPE signal amplitude for the reference and samples, respectively. The thermal effusivity, e, and volumetric heat capacity, ρc, were calculated using their relationship with thermal diffusivity and thermal conductivity, which were determined by FPPE and presented in Table 1.

Table 1. Thermal properties of reference water sample, butter and vegetable oils

Sample	Water (reference)	BT	EVAO	SFO	SBO
α_s ($\times10^{-8}$) [m²/s]	14.2	8.6 ± 0.3	7.9 ± 0.3	7.8 ± 0.2	7.9 ± 0.3
k_s ($\times10^{-1}$) [W/mK]	5.9	1.8 ± 0.1	1.9 ± 0.1	1.9 ± 0.1	1.7 ± 0.1
e ($\times10^{2}$) [Ws$^{1/2}$/m²K]	15.7	6.2 ± 0.3	6.7 ± 0.4	6.8 ± 0.4	5.9 ± 0.3
ρc ($\times10^{6}$) [Ws/m³K]	4.18	2.1 ± 0.1	2.4 ± 0.2	2.4 ± 0.2	2.1 ± 0.1

Acknowledgment

E. Contreras-Gallegos thanks the financial support from CONACYT through a postdoctoral grant.

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Reference Materials for Differential Scanning Calorimeter Temperature and Enthalpy Calibration

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Differential Scanning Calorimeters (DSCs) are regularly used in numerous applications including, but not limited to, materials characterization, the evaluation of phase diagrams, purity determinations, kinetic investigations, and heat capacity measurements. However, since DSC is not an absolute measurement technique, calibration is essential to ensure the accuracy of measured temperature, enthalpy, and heat capacity. Over the years, NIST has offered several certified Standard Reference Materials® (SRMs®) to support highquality calibrations for DSC. This work will discuss the development of two new calibration materials intended for the temperature and enthalpy calibration of DSCs. The first, designated SRM 2232a, is a replacement for the previously sold out version. SRM 2232a was sourced from a single lot of high-purity (^{3} 99.99999% metals basis, by weight) indium. The second is a new Reference Material (RM), designated RM 8013. The addition of this new RM extends the applicable temperature range of NIST's DSC-related calibration standards down to approximately 209 K and represents an attractive alternative to the previous option, SRM 2225, which was based on mercury and was discontinued in 2017. RM 8103 was sourced from a single lot of high-purity (^{3} 99%) adamantane. For both materials, enthalpy was determined via DSC measurements using an instrument calibrated with certified reference materials obtained from another national metrology institute; the same measurements were used to determine temperature for RM 8103. For SRM 2232a, temperature was determined using a fixed-point cell and direct comparisons with NIST primary temperature standards. This work provides a detailed description of both the measurement and data analysis procedures employed.

Surface tension of low-concentration aqueous mixtures with methanol and ethylene glycol including metastable supercooled state

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New data for the surface tension of water + methanol and water + ethylene glycol measured on a unique apparatus of own design are presented. The apparatus based on a modified capillary rise technique [1,2] enables measurements under the supercooled metastable state, i.e. under the equilibrium freezing point temperature. The data collected in the temperature range from –25 to 30 °C show quite good agreement with the mixture correlation by Wang et al. [3] combined with the IAPWS standard for pure water [4] and the correlations by Mulero et al. [5] for pure methanol and ethylene glycol. Preliminary analysis shows that the correlations can be well extrapolated to the supercooled region. Supporting data for the density of both binary systems were collected using the vibrating tube densimeter over the full composition range at temperatures from 2 to 90 °C. The standard uncertainties of the surface tension and density data are below 0.50 mN·m^{-1} and 0.040 kg·m^{-3}, respectively.

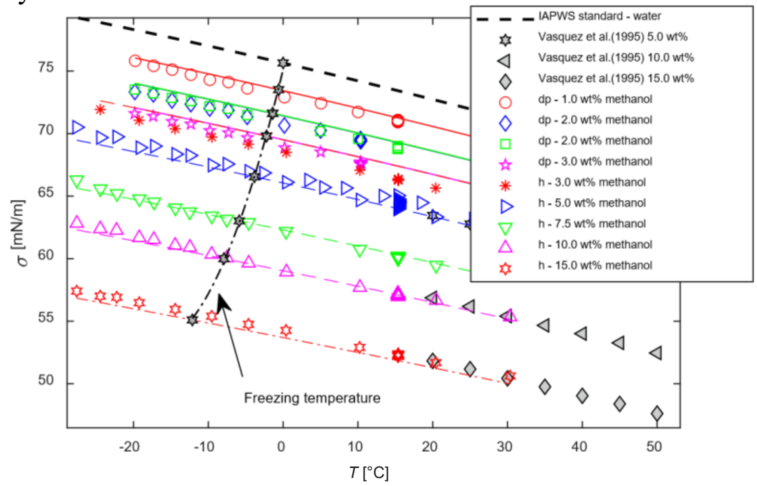

Fig. 1. Preliminary data for surface tension of water + methanol compared to Vasquez et al. [6] data, IAPWS correlation [4] for pure water and Wang et al. [3] mixture model (colored lines); dp marks measurements with helium counterpressure, h denotes the capillary elevation method.

Acknowledgment

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Fueling the future: Density, viscosity, and speed of sound measurements of Oxymethylethers at temperatures from (298 to 423) K and pressures from (0.1 to 100) MPa

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Polyoxymethylene dimethyl ethers, often abbreviated as OMEs, are synthetic fuels that are not based on crude oil or natural gas, but are produced via other sustainable process routes, thus contributing to moving away from fossil fuels. Today, it is already used as a constituent of diesel to reduce emissions and soot formation [13] but can also be a complete replacement of diesel. To fully exploit the potential of OMEs as a sustainable fuel, accurate knowledge of its thermophysical properties is crucial. However, literature reveals a significant lack of data for OMEs (see Fig. 1). Data were only found at ambient pressure, and, e.g., for speed of sound, no data are available at all. Here, we present density, viscosity, and speed of sound measurements of OME 3 and OME 4 over the temperature range from 298 K to 423 K at pressures up to 100 MPa. While density and viscosity are important for example for the flow, spray and consumption characterization, the knowledge of the speed of sound is, e.g, important to study the injection efficiency [4]. For the measurements, commercially available vibrating-tube densimetry as well as in-house fabricated vibrating-wire viscometry and a soundspeed instrument based on the dual-path length pulse-echo technique were employed. Density and sound speed were measured at Chemnitz University of Technology, and density and viscosity were measured at Imperial College London. Advanced models for data analysis were applied. The expanded combined uncertainties ($k = 2$) were estimated to be 1.5 kg/m^3 in density, 0.3 mPa/s in viscosity and approximately 1 m/s in speed of sound. The data will be used to develop accurate correlation equations for OMEs as future test liquids in flow measurements and thus for the assessment of the measurement performance of flow meters. Ideally, these equations will be incorporated into flow-metering standard in the future.

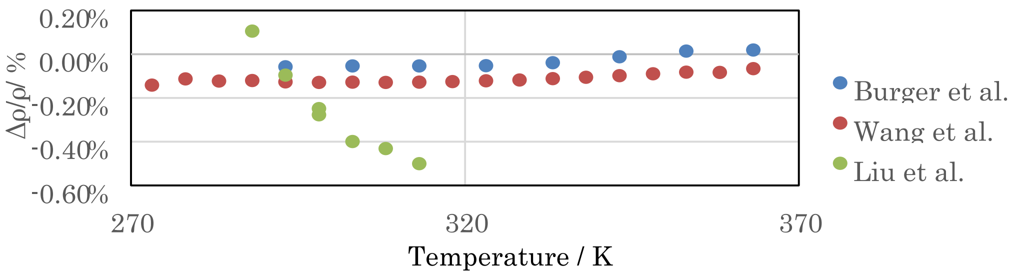

Fig. 1. Relative deviations of experimental density data for OME3 from literature [1-3] from values calculated with the density correlation fitted to the new data measured in the present work.

Acknowledgment

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Physical properties of CO_2 to support carbon capture utilisation and storage (CCUS)

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The European Commission's Green Deal aims to achieve carbon neutrality by 2050, with carbon capture, utilisation and storage (CCUS) named as a priority. CCUS begins by removing CO_2 from emissions before release. However, CCUS is not 100 % emission-free, since some products based on utilised CO_2 will reemit CO_2 over time and CO_2 slip is a relevant factor for all capture and transport technologies. In this context, the MetCCUS project (Metrology Support for Carbon Capture Utilisation and Storge – 21GRD06) within the European Partnership on Metrology will contribute to measurement technologies that are required to meet the targets of the Green Deal.

One workpackage of the MetCCUS Project addresses the metrological infrastructure needed to support the design, the monitoring and the maintenance of industrial infrastructures dedicated to carbon capture, transportation, utilisation and storage. A new capability for the calibration of hygrometers, an innovative sensor concept for the on-line monitoring of phase transitions in CO_2 mixtures, standard test methods for evaluating the susceptibility of pipeline materials to corrosion and improved thermodynamic models, based on original experimental measurements of thermophysical properties, will be delivered. A brief overview of the corresponding activities in MetCCUS is given.

Adsorptive hydrogen storage on porous materials at low temperatures

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The goal of emissions neutrality requires a rethinking of energy supply in all sectors of society and industry. Against this background, a promising approach is the production and efficient storage of hydrogen [1]. In addition to established technologies, the adsorptive storage of hydrogen shows huge potential since the volume related storage density can be significantly increased at comparatively low pressure [2-3]. Furthermore, adsorption is a largely reversible process, i.e., the hydrogen can be easily and loss-free released when needed [4]. While many adsorbents have already been analyzed for their suitability, a detailed investigation of their behavior as a function of temperature, especially in the low-temperature range, is missing. The present work aims to provide a basic overview of the state of art in this research area. Based on an extensive literature research on the adsorption behavior of hydrogen in various porous materials and corresponding data analysis, typical behaviors of respective material groups are identified. These findings, in turn, will be used to select promising materials to systematically investigate the adsorption capacity and desorption behavior of hydrogen at temperatures as low as 77 K (liquid nitrogen) with pressures up to 100 bar. For measurements of adsorption isotherms, a volumetric multi-port sorption analyzer is used for the fast screening of the materials, and a cryogenic gravimetric sorption analyzer based on a magnetic-suspension balance is utilized to study the best-performing adsorbents with higher accuracy. The results of these measurements will give a general indication of the adsorption behavior of hydrogen in porous materials and support fundamental research on this topic. Moreover, the data will provide important information about the reproducibility and accuracy of previously published measurements. Conclusions can also be drawn about the differences in the behavior of the various materials and the technical effort required in terms of temperature and pressure levels.

Acknowledgment

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ISRU-approach for designing and fabricating successfully composite materials

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Space: 1999 is a British science-fiction television programme that ran for two series from 1975 to 1977. In the opening episode, set in the year 1999, nuclear waste stored on the Moon's far side explodes, knocking the Moon out of orbit and sending it, as well as the 311 inhabitants of Moonbase Alpha, hurtling uncontrollably into space. Against all the expectations, the Moonbase crew was surviving and living their Space Odyssey in full sustainability producing energy, propellants, food, breathing air by exploiting the lunar resources.

The Space: 1999 sci-fi fiction seems going to be real in the next future, through the NASA-Artemis program. One of its basic strategy is to make winning the in situ resources utilization approach (ISRU) by focusing on how to process Lunar soil (regolith-a complex mixture of oxides) by using already terrestrial developed advanced technologies for extracting pure elements (i.e. Si, Al, Mg, Fe, Ti, etc.), to produce breathing air, growing plants, and whatever sustain Moon habitants in such extreme conditions, e.g. building materials, screens against radiations, etc. Under a great ISRU-focus is also the “space junk” mainly consisting of light weight alloys based on Al, Si and Ti.

A preliminary investigation on using lunar soil (regolith) properly combined/processed with the abovementioned alloys for fabricating composites feasible as building/functional materials will be introduced in this paper. In particular, as an example, preliminary results obtained by interactions tests between liquid Al7075/DNA-1 regolith simulant will be shown and discussed.

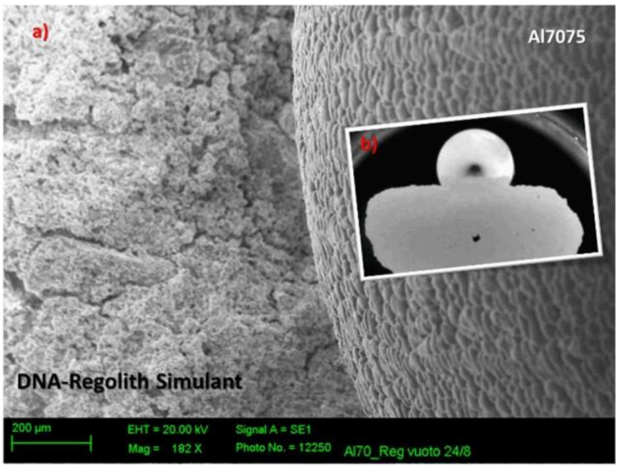

Fig. 1: Al7075/DNA-Regolith sample: a) SEM image after b) wetting experiment performed by the sessile drop method under a vacuum of Ptot = 10-4 Pa at T = 1100°C.

Formation and remediation measurements of solids from impurities in liquefied natural gas

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Liquifying gas and shipping it across vast distances is a common mode of energy transport. A problem all liquefaction facilities face is the freeze-out of impurities present in the gas supply, which deposit on surfaces, reducing flow and lead to unscheduled maintenance or shutdown. Of critical concern are the thin tubing within cryogenic heat exchangers, where plugging can occur rapidly and requires scheduled shutdowns to thaw out solid formations. Understanding the conditions in which solids can form and methods to remediate solids with minimal intervention can reduce downtime in liquefaction facilities.

In this work, we present measurements of solid formation, growth rate, and assessment of remediation strategies using a suite of cryogenic solid detection apparatus designed at UWA. The freeze-out behaviour was quantified for a natural gas sample with water impurity ranging from (10 to 200) ppm. For remediation measurements, a microwave resonator-based sensor previously developed for cryogenic solid detection was used and operated within a liquid nitrogen cooled chamber [1]. Various solvent gases were flowed over the solid at different velocities, temperatures, and pressures to determine the most effective method of remediation. It is anticipated that this sensor will allow industry to develop remediation methods through testing on their own samples.

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Research in fast acoustic gas thermometers: software development of validated physical models

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In November 2018 the international system of units (the SI) was redefined in terms of fundamental constants with defined value. The new definition of the temperature came into force in May 2019, along with the associated *Mise en Pratique* (MeP). From that point on, the kelvin is defined in terms of a fixed and exact value of the Boltzmann constant (k_B). Since that, one of the most accurate instruments useful to disseminate the temperature scale are Primary Acoustic Gas Thermometers (AGTs). AGTs are suitable to determine the thermodynamic temperature of a quasi-ideal gas with accuracy better than one part per million. Their actual capabilities are verified by determining the value of the Boltzmann constant and comparing the obtained experimental results with the fixed reference value. In principle, this procedure aims to verify quantitatively how adopted physical models are suitable to describe the thermodynamic processes occurring in the experimental apparatus. Currently, all these calculations are performed, when measurements cycles are completed, by using unintegrated software tools that can potentially lead to mistakes and errors. This work aimed to implement software libraries suitable to provide the on-line access to the most advanced physical models for calculating the thermodynamic properties of helium-4 from 7 K. Furthermore, new libraries include algorithms needed to correct the experimental measurements obtained using acoustic primary thermometers. The software libraries have been developed in Fortran 2008 and can be linked to any development tool supporting Cformat compiled libraries, in particular, interfaces for LabView are provided.

European Metrology Network for Clean Energy

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One goal of the European Green Deal is to put Europe on a more sustainable path by reducing net emissions of greenhouse gases to zero by 2050. The future European Metrology Network (EMN) on Clean Energy aims to support the objectives of the Green Deal through coordinating actions to provide metrology solutions to industry and societal needs, addressing a broad range of topics related to clean energy not currently addressed by existing EMNs, including energy generation, conversion, efficiency, storage and transport. This contribution shows the work done to support the establishment of this EMN by defining its scope and by focusing on the development of a long term ongoing dialogue between the metrology community and the whole field of clean energy stakeholders.

Metrology for decarbonising the gas grid

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The use of natural gas as a primary energy source represents a major issue to global warming. Decarbonisation of the gas grids is a possible solution, alongside electrification, to meet the climate change targets and reduce carbon dioxide emissions. This project is the first large scale project of its kind that will tackle four measurement challenges that the gas industry needs to solve before the introduction of biomethane, hydrogen-enriched natural gas, 100 % hydrogen, and carbon capture and storage (CCS) can be used for decarbonisation of the gas grid. The objectives of the project cover the priority challenges within flow metering, gas composition, physical properties and safety (including monitoring of gas leaks) required to support these changes. A description of the operative structure of the project, the topics tackled and suggestions on how to stay updated on coming results and their exploitation are provided.

Thermal and tribological characterization of hospital textile fabrics

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Currently, the textile materials used in medical biological areas (MB) [2] require special characteristics for protection with high quality of health for patients and personnel in these areas [2], these materials must have adequate handling and health characteristics without producing an aggressive deterioration when in contact with them [3], in the textile fabrics used by patients and MB operating personnel, this contact of the textiles produces friction with the skin, manifesting degradative and harmful effects of wear leading to physical damage [4] , using photothermal techniques (TF) as they are non-destructive, this type of materials can be characterized in different conditions and by different configurations such as photoacoustic spectroscopy (PAS) [5], calorimetry [6], photopyroelectric technique [7] and modified flash method [8] (MFm), to obtain its optical and thermal properties. In this study, the photoacoustic spectra, the evolution and decay of temperature and coefficient of friction of textile fabrics were obtained. for hospital uses treated with different types of softeners, in the flat cotton textile fabric it presents changes in the evolution and thermal decay when different softeners are used as observed in figures 1a and 1b, where for the textile fabric without softener the decay heat is higher compared to treated with softeners.

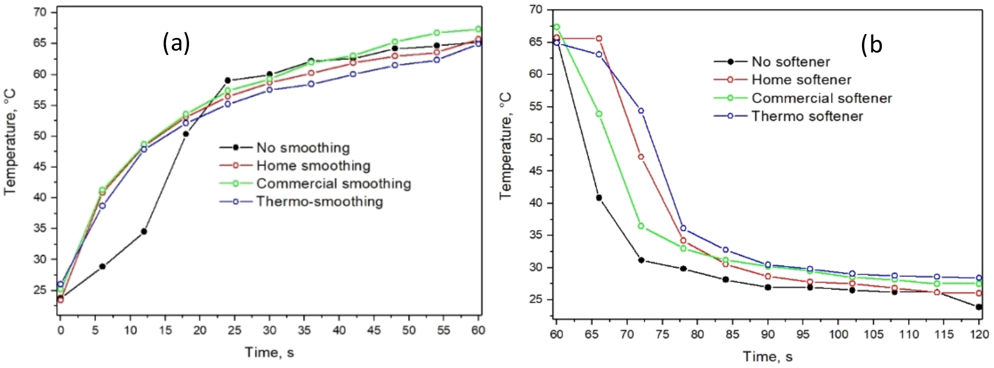

Fig. 1. a) Evolution and b) thermal decay of cotton textile fabric

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Characterization of liver tissues by means of thermal images obtained with photoacoustic microscopy (PAM)

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Determining the characteristics of non-homogeneous biological materials is complex due to the structures of which they are composed in the case of liver tissues of the liver, an organ with a unique role in the metabolism system of living beings, with biochemical functions, synthesis and excretions [1] performs around 500 functions in our body [2], one of them is to produce bile acids which regulates the amount of nutrients glucose and lipids [3]. Housing approximately 450,000 branches of the portal and hepatic vein [4]. This high vascularization of liver tissue limits many techniques to characterize it, photothermal techniques being a viable and non-destructive alternative that can allow obtaining various thermal properties such as diffusivity, optical absorption coefficient, among others [5-7]. In different types of biological and non-homogeneous materials. The objective of this research is to obtain the differences between liver cells of different origins of liver cells (poultry, bovine and fisher rat), by using the photoacoustic microscopy (PAM) technique. Thermal images were obtained to be able to observe the differences between the different liver tissues. In the thermal images, significant changes can be observed in the values of the photoacoustic signals (PA) generated when sampling the different types of liver tissues, as can be seen in figure 1. the heat maps obtained for the liver tissues of the poultry the PA signal has values in the range of 0.1574 to 0.3 mV, for the bovine the PA signal value in the range of 0.170 to 0.41 mV and for the fisher rat the PA signal value in the range of 0.164 at 0.42 mV, where it is observed in the liver tissues of both mammals presented a higher value in the amplitude of the PA signal.

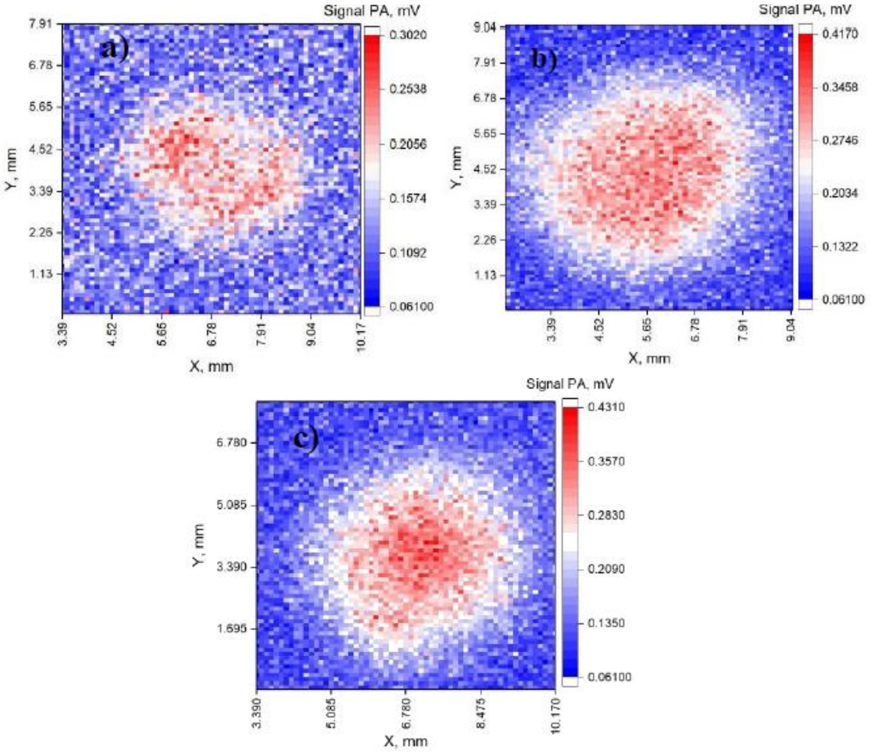

Figure 1. Heat map images of liver tissues of a) poultry, b) bovine and c) fisher rat

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Analysis of the thermal properties of materials to protect the respiratory system (masks) using photothermal techniques

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The importance of the safety cloth material characteristics has a great relevance and impact [1], when they are used in industrial, hospital and research areas, where this type of protective material is of utmost importance and the knowledge of the material characteristics such as textiles must cover optimal conditions and possess high performance qualities to be employed [2]. In this sense, face masks provide a critical barrier, reducing the amount of volatile particles such as dusts, gases, when breathing and exhaling [3], and also their efficient use can contain the spread of biological particles, such as viruses and bacteria [4]. The characterization of this type of materials requires the use of techniques where the material samples are not destroyed or diluted and samples, with different structures, can be characterized, in this sense photothermal (PT) techniques have shown to be an effective tool for material characterizations, being non-destructive and not requiring a sample previous preparation. With different configurations of PT techniques, such as photoacoustic spectroscopy (PAS), photoacoustic microscopy (PAM), photopyroelectric microscopy (PPEM) and open photoacoustic cell (OPC), among others, several optical and thermal properties have been obtained such as the specific heat, thermal effusivity, diffusivity, thermal resistance, etc. [5-7]. In this research we obtained thermal images and thermograms of temperature evolution and decay of face masks by photoacoustic microscopy (PAM) and adapted flash method (FM), both systems implemented for the development of this research. Figure 1.a shows the thermography obtained from the two-layer cotton face mask, where it is observed some areas with higher temperatures, and Figure 1.b shows the temperature evolution of different types of face masks, where the temperature increase is higher in several of the face masks that are made with similar material. By using the temperature rise method under continuous light illumination [8], the ρc of the materials of the face masks was obtained and Table 1 shows these values.

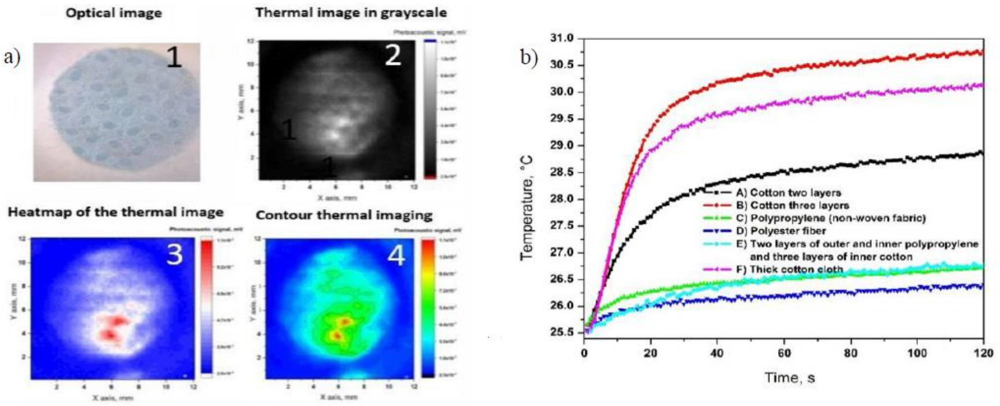

Fig. 1. a) Thermal imaging with photoacoustic microscopy of a two-layer cotton face mask y b) Thermal effects of laser irradiation on face masks (Evolution).

Table 1. heat capacity values (ρc).

Mask type	τ (s)	l (cm)	ρc ($Jcm^{-3}K^{-1}$)
A	3.6359	0.013	1.072
B	5.8625	0.016	1.401
C	5.3915	0.011	1.877
D	7.3888	0.033	0.859
E	10.9127	0.0305	1.368
F	3.0604	0.011	1.067

Acknowledgment

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Phase change materials emulsions for heat exchange applications

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Phase change material emulsions (PCMEs) have risen interest in recent years as potential heat transfer and heat storage fluids. These systems consist of a heat transfer fluid as water and an emulsified or an encapsulated PCM, which should be immiscible with the base fluid [1]. The idea is to exploit the latent heat of melting and crystallization of PCM to increase the thermal energy storage capacity of the base fluid. The continuous phase, the base fluid, confers greater thermal conductivity and lower viscosity with respect to PCM. The emulsified or encapsulated systems have also the advantage to be pumped through a heat transfer circuit independently from the state of PCM.

In this work, a 10 wt% of a commercial PCM material, RT25HC, with nominal phase change temperature at 25 °C, has been successfully emulsified in water by sonication and surfactant addition. The size of PCM droplet as well the emulsion stability was investigated over time.

Since supercooling, that is the crystallization process happening at lower temperature with respect to melting, is a typical issue in PCME development being more pronounced in nano-confined systems, the effect a nucleating agent on crystallization kinetics has been evaluated. In particular, a PCM melting at higher temperature with respect to RT25HC (RT65, melting at 65°C) was added with a concentration of 1 wt%.

The thermal conductivity, the viscosity, the density and the Cp of the emulsion were measured at various temperatures. The heat exchange performance of the PCME was investigated by means of a specifically built apparats for the measurements of the heat transfer coefficient at constant heat flux.

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Behavior of Dielectric Permittivity in Squalane-Graphene Nanofluid at 333.15K

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The global demand for energy is constantly growing, requiring the development of efficient systems to ensure sustainable consumption and prevent long-term environmental damage. Carbon-based materials, such as graphene, have been extensively studied due to their environmentally friendly properties, structure, and availability. Squalene is also a component with significant physiological effects in fields like nutrition, pharmacy, and medicine [1]. However, both graphene and squalene are relatively new in the field of nanofluids. While research on thermal conductivity has been prioritized, studies on permittivity and electrical conductivity remain limited.

In this study, we investigated the dielectric permittivity of graphene nanofluid at temperatures of 333.15K and 343.15K. The nanofluid was prepared using squalane as the base fluid and 0.75% volume concentration of graphene nanoparticles, without the addition of surfactants to avoid interference. The graphene has a thickness ranging from 11-15 nm and a surface area of 50-80 m^2g^{-1}. We analyzed the complex permittivity within the frequency range of 20Hz-30MHz, we also examined the electrical conductivity, static and high frequency permittivity, and the loss tangent.

The experimental values for the real part (ε') and imaginary part (ε'') of the permittivity for this nanofluid, after subtracting the contribution of electrical conductivity, which was extrapolated at low frequencies from the real part of the complex electrical conductivity, are presented in the following figure.

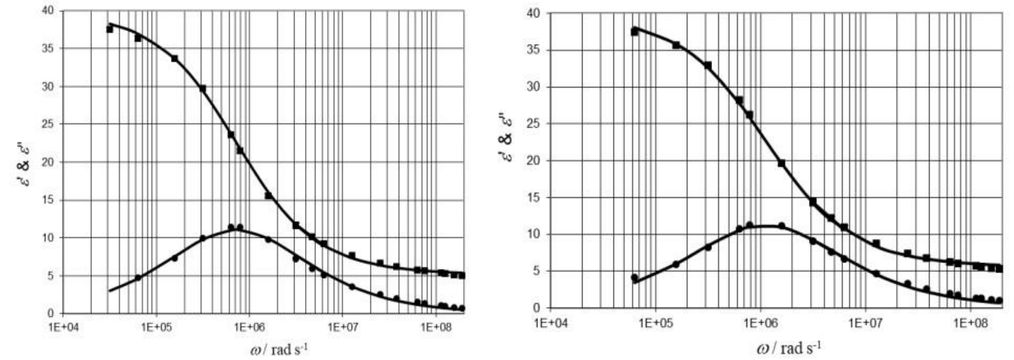

Fig. 1. Dielectric spectra for the nanofluid graphene-in-squalane at 333.15 K and 343.15 K, respectively. Full curve calculated for Cole-Cole [3-4].

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Experimental Study on the Density of Graphene Nanofluid: Effects of Concentration, Temperature

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Thermal and physical properties of fluids are crucial factors in the design and development of heat transfer equipment that operates with high efficiency. These properties determine how effectively a fluid can transfer heat, how it behaves under different temperatures and pressures, and its overall performance in heat exchange processes. By understanding and optimizing these properties, engineers can design more efficient heat exchangers, boilers, cooling systems, and other heat transfer devices that enable effective heat transfer and energy conservation. The density of fluids is an essential physical-chemical property that plays a vital role in mass and heat transfer. Its understanding and control are fundamental to design more efficient processes and maximize the performance of related systems.

The density of the nanofluids was determined by the pycnometric method, for different concentrations and different temperatures at ambient pressure. This summary presents the study of the density of graphene nanofluid, with different volumetric fractions of graphene nanoparticles as a function of temperature. The density measurement was carried out carefully to obtain accurate values, as well as the preparation of the nanofluid in order to be stable during the entire measurement process. The preparation of the nanofluid was carried out by the two-step method [1]. The experimental values of the density compared to the initial models existing in the literature present significant deviations, this difference increasing with the increase of the volume fraction. The most used model for calculating density is the mixture model of Pak and Cho [2], however, this equation is still an approximation. As the density of the nanofluid is affected by numerous factors, one of which is the shape and size of the nanoparticle, we will present the study considering this factor [3].

In the following figure, we present the density of the nanofluid as a function of temperature and density against the concentration of nanoparticles in experimental volume and for the model of mixtures.

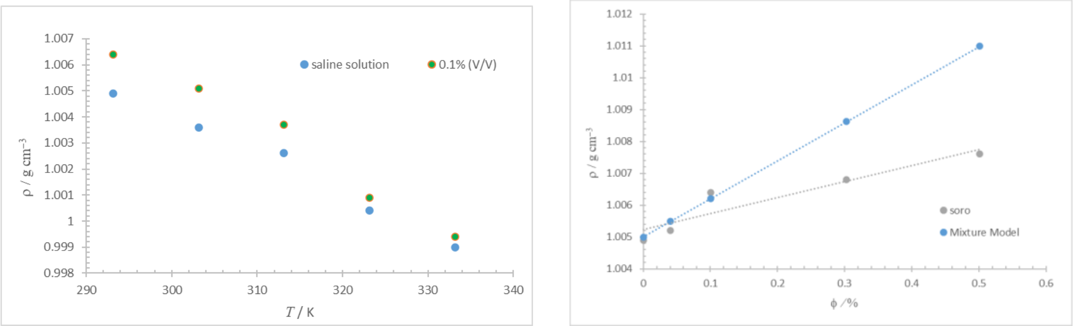

Fig. 1. Density of the nanofluid with 0.1% (V/V) graphene as a function of temperature (a), density as a function of the volume fraction (b)

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Prediction of critical points for carbon dioxide-based binary mixture working fluid by the Heidemann-Khalil approach

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Prediction of critical point is of great interest for CO_2-based binary mixture being the working fluid of power cycle. Compared with empirical correlation, the critical point of mixture can be calculated based on its rigorous thermodynamic criteria, with limited binary interaction parameters and consistency on vapor-liquid equilibrium and other thermodynamic properties. In this study, the critical points of CO_2-based binary mixture, applicable for being working fluid of power cycle, including hydrocarbons, fluorocarbons, dimethyl ether, methanol, water and xenon, were studied based on the Peng-Robinson equation of state (PR EOS) with van der Waals (vdW) mixing rule, according to the Heidemann-Khalil approach. By comparing the predicted results with the experimental data in the literature, the applicability of this method to various mixtures is discussed. The characteristics of critical lines, type of phase behavior, and binary interaction parameters of each mixture were analyzed and discussed, and the results show that the Heidemann-Khalil approach is efficient for critical point calculation and prediction of CO_2-based binary mixture.

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