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on
High Temperature Capillarity

MAY 26–30, 2024, SWEDEN

Book of Abstracts

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PREFACE

Welcome to the collection of abstracts from HTC 2024, held in Stockholm and aboard a cruise ship between Stockholm and Helsinki. It was with great pleasure that we hosted this prestigious conference. I am delighted to present to you the collection of abstracts. This book of abstracts represents the diverse range of research, ideas, and discussions in the fields of high-temperature melts, interfacial phenomena, wetting, and capillarity that were presented and shared during the conference. As an editor, I feel honoured to compile this diverse array of abstracts. It is my hope that this collection will serve as a valuable resource, inspiring further exploration and collaboration in the HTC field and society.

This 11th international conference was supported by many people. I must take a moment to express our sincere gratitude to our esteemed sponsors, Netzsch and Thermo-Calc AB, and to all attendees. Without their generous support and commitment to advancing knowledge and innovation, this conference would not have been possible. Last but not least, I thank the International Scientific Committee and the Local Organizing Committee for their efforts in the preparation of this successful conference.

Taishi Matsushita

Jönköping University

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OVERVIEW

The 11th International Conference on High Temperature Capillarity continues the tradition of the previous conferences organised within this area.

A few of the topics of the conference are:

- Surface/Interfacial phenomena of high-temperature melts (e.g., Marangoni effect)
- Surface/Interfacial tension of high-temperature melts
- Reaction between the high-temperature melts and solid/gas
- Capillary phenomena (penetration behaviour) of high-temperature melts
- Wetting behaviour of high-temperature melts
- Segregation, grain boundary wetting and intergranular films
- Metal/metal, metal/ceramic, metal/glass and ceramic/ceramic interfaces, and surfaces of high-temperature systems

List of the HTC conferences

Conference	**Chairpersons**	**Place**
HTC 1994	Yu. Naidich and N. Eustathopoulos	Smolenice, Slovakia
HTC 1997	N. Sobczak	Kraków, Poland
HTC 2000	K. Nogi	Kurashiki, Japan
HTC 2004	A. Passerone	San Remo, Italy
HTC 2007	E. Louis	Alicante, Spain
HTC 2009	P. Nikolopoulos	Athens, Greece
HTC 2012	W. D. Kaplan	Eilat, Israel
HTC 2015	B. Straumal	Bad Herrenalb, Germany
HTC 2020	G. Kaptay	Hungary
HTC 2022	N. Sobczak	Kraków, Poland
HTC 2024	T. Matsushita	Stockholm, Sweden

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The logo features Viking horned helmets, but it also alludes to the sessile drop, with the contact angle "θ" and arrows representing surface tension incorporated into the design.

The HTC 2024 logo was created by Taishi Matsushita using a copyright-free image as a basis.

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ORAL SESSIONS

01. High-temperature interaction of liquid magnesium with tantalum and 316L stainless steel substrates

Sylwia Terlicka[1], Natalia Sobczak[1,*], Karol Janus[1,2], Jerzy J. Sobczak[2]

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This study is focused on the real-time observation of the high-temperature wetting behavior of liquid Mg drops on two different substrates made of pure tantalum, and 316 L stainless steel. The contact angle values (θ) formed between the liquid metal and the examined substrates were determined using the sessile drop method combined with non-contact heating of the Mg sample and the substrate to the testing temperature accompanied with the deposition of the Mg drop on the substrate by squeezing the liquid metal from the graphite capillary. The applied testing procedure eliminates two factors affecting high-temperature measurements, i.e., the heating history of the Mg/substrate couples and unavoidable presence of the native oxide film on solid Mg samples1-3) . The tests were conducted under isothermal conditions at a temperature of 700 °C in a gaseous mixture of Ar with 5 wt% H_2.

After the sessile drop tests, the structure and chemistry of surfaces and interfaces of solidified Mg/Ta and Mg/steel couples were characterized in detail by scanning electron microscopy combined with energy dispersive X-ray spectroscopy.

Under the used testing conditions, liquid magnesium showed non-wetting behavior ($\theta \gg 90°$) and also did no permanently bond with neither of the investigated substrates. This behaviour can be attributed, according to the available phase diagrams4) for the Mg-Ta and Mg-Fe systems, to the non-reactive and immiscible character of these systems caused by the negligible solubility of Ta and Fe in liquid Mg, as well as the fact that Mg does not form compounds with either Ta or Fe.

The results of this study will be compared to those of other Mg/metal systems obtained using the same experimental facility to demonstrate the role of testing procedure and reactivity between contacting materials.

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Acknowledgment:

This research was supported by the National Science Centre of Poland within OPUS 21 Project, no. 2021/41/B/ST5/02787 in the years 2022–2025.

02. Influence of atmospheric conditions on the high-temperature interaction between molten hypoeutectic Mg-Ca alloy and titanium substrates

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Magnesium (Mg) and titanium (Ti) alloys are promising biodegradable materials for implant applications in orthopedics due to their high mechanical characteristics, including deformation behavior and toughness[1, 2)]. Unfortunately, the Ti alloys have higher elastic modules than the natural bone, and the Mg alloys exhibit insufficient chemical biocompatibility to the internal chemistry of the human body[1, 3)]. Therefore, combining the mentioned alloys by liquid-assisted processes to create biodegradable implants is very promising and requires extensive wettability studies.

This investigation examined the high-temperature interaction between a molten Mg-Ca alloy and a pure titanium substrate. The sessile drop method, combined with the capillary purification procedure and non-contact heating, was employed to eliminate the influence of the native oxide film on an Mg-Ca drop and the heating history of Mg-Ca/Ti couples4[)]. The experiments were conducted under isothermal conditions at 700°C in two protective atmospheres: (i) pure Ar and (ii) a mixture of Ar+5 wt% H_2.

The high-resolution images of Mg-Ca/Ti couples obtained during the experiments, recorded by two high-speed monochromatic CCD cameras with a speed of 57 fps from two observation directions, were utilized to determine the wetting kinetics, i.e., the time-dependent values of the contact angle (θ) arising between the liquid alloy drop and the solid titanium substrate. In the specified experimental conditions, these results indicated that the molten Mg-Ca alloy displayed non-wetting behavior on the titanium substrate in the Ar atmosphere (θ_{av}=91°) while exhibiting wetting behavior in the Ar+5 wt% H_2 environment (θ_{av}=11°) after 180 s of contact time. The detailed analysis by scanning electron microscopy combined with energy-dispersive X-ray spectroscopy revealed a lack of permanent bonding and showed neither new phases nor mass transfer in the Mg-Ca/Ti couples in both applied atmospheres. Hence, the obtained results are discussed in-depth concerning the wetting behavior and reactivity of applied Mg-Ca/Ti couples.

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Acknowledgments: This research was supported by the National Science Centre of Poland within OPUS 21 Project, no. 2021/41/B/ST5/02787 in the years 2022–2025.

03. Penetration equipment and mathematical model for analyzing the penetration of different electric motor impregnation resins

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Due to climate concerns, serious decision of electrifying the transport industry has been made by the European Union. After 2035, no cars with internal combustion power train can be sold in the European Union. As a consequence of this measure, the European car industry has shifted its trend of advancement to the development of hair pin electric motors and their manufacturing technologies. Ever since, a number of new electric motor production lines has been built-up in order to meet the ever increasing demand for new and more efficient electric vehicles, where different types and grades of impregnation resins are used. The impregnation process of electric motors is determined by the wettability and viscosity of impregnating resin, that is a key factor by process design and defining parameters, which determines the end quality of parts.

In order to understand the penetration behavior of different impregnation resins, a penetration equipment has been designed where the penetration of two different impregnating resins were analyzed. One epoxy-based and one unsaturated polyester resin. During the investigation, a mathematical model of penetration has been brought about which describes the penetration times of the applied resin. The obtained modell is in full agreement with the model found in the literature 1).

In the course of the study, Polieter-eter-keton (PEEK) and Poliamid-imid (PAI) coated hair pin copper conductors were used. The resins penetrated between two parallel hair pins.

By comparing the viscosity of the resins, the viscosity of polyester resin is much higher than the epoxy based one owing to its longer molecular length. Initially, the viscosity function was approached with Arrhenius model, however, a more precise fitting has been accomplished with the VFT (Vogel-Fulcher-Tammann) model 2).

From which, it can be concluded that, the resins cure during the viscosity measurement. Temperature dependence of wetting angles of impregnating resins has been also determined on different insulating substrates.

By comparing the penetration times, epoxy resin penetrates much faster due to its lower viscosity, which is more favorable from the point of dwell times and machining costs.

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04. Reactive infiltration. Effect of different parameter

Karla López [1], Leidy Figueroa-Quintero[1], David Villalgordo-Hernández[1], Enrique V. Ramos Fernández[2], Javier Narciso[1,*]

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One of the issues that is still very much in controversy today is who governs the reactive infiltration. Whether the chemical reaction or the fluid dynamics of the process. Although the data showing that the main actor is the reaction 1-3) is overwhelming, at the manufacturing level it is still believed that the decrease of the infiltration rate is due to the reduction of the capillary due to the formation of SiC on the walls. It is important to point out that it is convenient to clarify this fact since the production of materials by this route has multiplied ostensibly in the last years.

The present work is a continuation of a previous one in which the infiltration process was studied in perfect capillaries with different diameters, and with two carbon materials (high density graphite and glassy carbon) 3). The new experimental data show that there are two regimes in the infiltration process, the first one where the infiltration rate is governed by the chemical reaction and the second one where the infiltration rate is governed by the fluid dynamics of the process. This indicates that first a more or less compact layer is being formed and that once formed, infiltration proceeds according to Darcy's law. A detailed theoretical analysis has also been made of how gravity, capillary radius and the reactivity of the carbon material affect the two distinct processes that we have observed.

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05. Surface interactions and compatibility between Al-Si alloy and ceramic materials

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The high latent heat of fusion and high volumetric energy density of some metals and metallic alloys makes them perfect for their use as phase change materials in compact Thermal Energy Storage systems1). However, one of the main challenges for this application is finding a compatible material that can withstand the contact with the metallic alloy when it is molten without degrading.

The interactions on the contact interface between Al-12.6%Si (wt.%) and two different ceramic materials (alumina and cordierite) have been studied as a means to assess the compatibility of both materials when the metallic alloy is molten. Two different purity grades of alumina were analyzed to determine the impact of impurities on the potential interactions.

The existence of a reaction and the formation of new phases occurring at the interface when after holding the system at temperatures above the melting point of the metallic alloy were studied. The type and extent of those phases were also identified with SEM and EDX.

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06. Polarization effect on interface between copper-containing molten iron and slag

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It is difficult to remove Cu from molten iron, the primary technique used in the steelmaking process. It was found to be difficult to be removed by such usual methods as oxidation or evaporation, and Cu may become concentrated in steel products due to repeated recycling. Numerous methods for removing Cu from molten steel scrap have been proposed and studied1), but none of these methods has been commercialized. This study focused on the use of molten oxide electrolysis as a low-cost, clean, continuous separation method suitable for incorporation into actual steelmaking processes. The basic concept of this study is to promote the dissolution of Cu in molten Fe into molten slag via electrolysis. Needless to say, it is usually not possible for Cu, which is more noble than Fe, to be preferentially oxidized by electrolysis. However, it is expected that the use of a liquid metal electrode will cause fluctuations in the interface, including electrocapillarity effect. This acceleration shortens the time to reach thermodynamic equilibrium. As widely known, the electric capillary effect reduces the energy dissipated between the metal phase and the slag, i.e. Cu-rich phase concentrated near the metal-slag interface via electrolysis may contribute preferentially to the dissolution of Cu. The interest in this study is its effectiveness in copper dilute systems.

We investigated the transport behavior of copper-containing molten iron when it was anodically or cathodically polarized in molten oxide slag. Polarization of molten metal (Fe–0.5 wt% Cu–0.1 wt% C) in molten slag (FeO - Al_2O_3 - CaO - SiO_2 - MgO system) using MgO crucible and constant potential electrolysis in 1823 K Ar atmosphere were performed. One hand the change in copper concentration in bulk Cu-containing Fe decreased by 5.2% when no electrodes were installed, on the other hand, the decrease was 11.9% with +1 V electrolysis.

The energies of this systems were obtained by the following methods by first-principles molecular dynamics (MD) calculations (software: VASP, basis: plane wave, exchange-correlation functional: PBE, pseudopotential: PAW), and the dissolution potential of molten Fe containing dilute Cu into slag is −0.44 V (vs. O_2/O^{2-}) for Cu, and −0.68 V for Fe was obtained. Although both values are close compared from pure metals thermodynamic parameter, Cu remains noble than Fe. However, in the energy profile analysis of the Cu dissolution process, there was no energy barrier specifically corresponding to the activation energy, and an energy minimum value existed at the metal-slag interface instead. This suggests the possibility of concentration of Cu in molten Fe near the interface during electrolysis in this system.

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07. Enhanced Iron Impurity Removal in Low Fe Al-Si Alloys by Mn Addition

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Over the past years, the demand for high-purity aluminium has increased in many sectors, like the aerospace and automotive sectors. This is because aluminium has excellent corrosion resistance and a high strength-to-weight ratio. To cope with this significant increase in demand, the production of primary aluminium has increased since the refining processes of secondary aluminium are limited by high impurities, mainly iron. The iron-rich intermetallic compounds (β-Fe phase) in Al-Si aluminium alloy negatively affect the mechanical properties of the aluminium from its sharp-edged coarse plate structure. In order to mitigate this problem and reduce the iron content in the melt, one way is to add Fe-bearing intermetallic particle formers, like Cr, Mn and Sr. This paper aims to investigate the influence of different Mn additions for low Fe composition aluminium melt at a constant cooling rate. Specifically, the impact of using filters, the Fe removal efficiency for different Mn additions, and the Fe-bearing intermetallic particle's Fe removal potential. This was done by running small-scale experiments with 8 kg/experiment of Al-10Si-0.5Fe (wt%) alloy. The main parameter that varied was the amount of Fe-bearing intermetallic particle formers added to the melt. This report concludes that the Fe-bearing intermetallic parties have mostly sedimented from the top surface of the melt since the composition of the filtered and unfiltered samples were similar. Additionally, larger amounts of Mn are required to improve the Fe removal efficiency for low Fe concentration melt since it improves the Fe removal potential and increases both the size and amount of Fe-bearing intermetallic particles in the melt.

08. Some aspects of non-metallic inclusion behaviors at the liquid and solid steels considering interfacial physical parameters

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Inclusion engineering is a comprehensive concept dealing with the control of amount, size distribution, and chemical composition of non-metallic inclusions in advanced steels. Furthermore, it also concerns the correlation between inclusion, microstructure and property on the quality control of the final product. This work aims to summarize the research activities of the authors regarding the inclusion motion and agglomeration at the steel/gas/slag interfaces 1-3), inclusion dissolution in the slag 4-5), and fine inclusion induce intragranular acicular ferrite (IAF) nucleation in solid steels 6). Several aspects of interfacial phenomena, including interfacial energy, contact angle between inclusion and metals, surface tension of metals, viscosity of silicates, etc. are considered in the theoretical models to investigate the mechanism. For instance, the capillary force is found to be the main reason of inclusion agglomeration at the steel/Ar interface, the difference of interfacial energy between inclusion/ferrite and inclusion/austenite affects the nucleation barrier of IAF formation from the inclusion surface. Physical parameters are collected from either open literature as well as measurement 7-8). This work aims to provide insights of the theoretical mechanism considering interfacial phenomena contributing to 'inclusion engineering'.

Key words: Inclusion engineering, interfacial energy, nucleation, capillary force, steels

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09. Wetting and reactivity phenomena occurring due to the interaction between the liquid metals and severely deformed metallic substrates after explosive welding

Joanna Wojewoda-Budka[1, *], Monika Bugajska[1], Sylwia Terlicka[1], Marta Janusz-Skuza[1], Izabella Kwiecien[1], Agnieszka Bigos[1], Anna Wierzbicka-Miernik[1], Magdalena Bieda-Niemiec[1], Fabrizio Valenza[2], Sofia Gambaro[2], Eugen Rabkin[3], Natalia Sobczak[1]

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Liquid-solid interaction plays a crucial role in many joining technologies, surprisingly among them explosive welding can be also named, although being classified as a solid-state welding technology. Literature reports and owns authors research unambiguously evidenced that a thin continuous melted layer (sometimes of nanometers thickness) is being formed at the interface of the colliding metallic plates during explosion. Therefore, the aim of the research was to study the wetting and reactivity phenomena in two systems of large practical importance welded via the mentioned technique: (1) technically pure liquid aluminum and S355J2N steel substrate, (2) liquid magnesium on technically pure A1050 aluminum substrate. Samples for testing were prepared using two methods: the classical sessile drop method and a modified one with capillary purification procedure. Additionally, the wetting test has been carried out in isothermal conditions at the temperatures of 720, 800, 900 and 1000 □C (Al/steel) and non-isothermal conditions of liquid Mg drop (780 °C) on a cold Al substrate. In addition, in the Mg/Al system, the reaction of liquid Mg with Al substrates of varying degrees of deformation allowed verification of the hypothesis that substrate microstructure influences wettability and reactivity in this system.

Acknowledgments

This research was financially supported by the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences within the statutory works ‘‘The interface zone of the welds obtained with the use of explosion energy’’ Z-5/2023. The study has been carried out within the framework of bilateral cooperation agreement between IMMS PAS and Faculty of Foundry Engineering, AGH University of Science and Technology in Krakow, National Research Council – Institute of Condensed Matter Chemistry and Technologies for Energy in Genoa as well as Department of Materials Science and Engineering, Technion – Israel Institute of Technology in Haifa.

10. Simulation and experimental methodology for prediction of laser power and penetration depth in surface cladding/alloying

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Understanding and evaluating the performance of different powder and substrate materials combined in the laser cladding/alloying layer is prioritised by process and material engineers to obtain high-quality durable surfaces. The surface quality is usually determined by the combination of various process parameters, such as laser power, powder feeding rate, and scanning speed, that result in different dilution ratios. Furthermore, process parameter calibration highly depends on the surface geometry and alignment of the deposited tracks. The application of simulation tools for the manufacturing process design tends to reduce experimental efforts. However, laser surface cladding and alloying represents a complex manufacturing process, where powder deposited on the surface of a material solidifies and forms an alloy with the substrate. Full-scale process simulation is often not feasible for parametric studies aiming at tuning the process parameters.

The present work introduces an experimentally validated simulation methodology, including a simplified three-dimensional finite-element heat transfer model of the laser surface cladding/alloying process, Figure 1. Cladding/alloying of a nickel-based superalloy powder on the grey cast iron substrate has been studied. With the help of laser cladding experiments and measurements on cross-section images, it has been shown that the model is capable to predict the actual laser power to obtain the desired penetration depth into the substrate, heat-affected zone size and dilution ratio. It is shown by introducing a laser power scaling factor that the model input and comparison data can be obtained from a single cladding/alloying experiment.

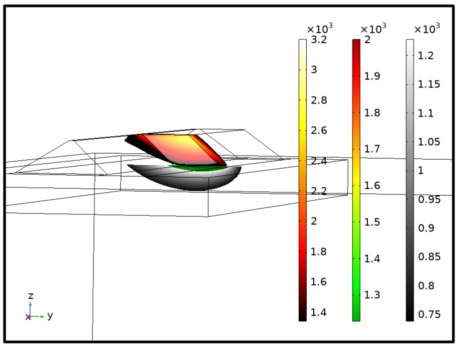

Figure 1. Simulated temperature distribution on the deposited track revealing the melt pool extension, penetration depth and heat-affected zone size.

The financial support from the KK Foundation, Sweden (Grant No. 20200059) is acknowledged.

11. A Systematic study of the origin of pore surface characteristics in cast iron

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Cast iron foundries are often afflicted by porosity defects, resulting in substantial economic, energetic and, by extension, environmental losses. In the current industrial context, where sustainability is key, efforts must be made to avoid these defects. As it is known by metallurgists, the morphological and surface characteristics can hint at the root cause of porosity defects; however, when it comes to the surface characteristics of pores, existing research lacks a systematic assessment concerning the nature of gases involved as well as the timing of pore formation. The present work aims to study the relationship between gases injected at predetermined moments during solidification and the pore surface characteristics. Cast iron cylinders were melted inside alumina crucibles under an inert atmosphere. During the solidification process, known gas mixtures were carefully injected from syringes into the melt with the objective of forming a stable bubble. Once the cooling was complete, the cylinders were sectioned, and the pore surfaces were analyzed with a Scanning Electron Microscope (SEM) and by Energy Dispersive X-ray Spectrometry (EDX). By gaining a deeper understanding of the processes generating various surfaces, foundries are better equipped to identify the root causes of pores and to strategize effectively for decreasing porosity defects in regular production. Future research may expand on this study by exploring a wider range of gases, base melt, and solidification conditions, to broaden the relevance of this study to different industrial contexts.

12. Statistical calculation model for the hydrogen diffusion parameters in metals at high temperatures

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Diffusion of H in metals is very important as it leads to engineering problems associated with creation and using of high strength steels, reactor materials, alloys for transporting and storing hydrogen, etc. Modern ab initio modelling will provide a good description of the diffusion parameters in Me-H systems at the temperatures above 500 K. However, there is no systematic derivation of the diffusion coefficients in which statistical effects are commonly considered at the high temperatures close to the melting temperature.

The purpose of this work is development of the statistical model of hydrogen diffusion in metals and its application for calculating the diffusion coefficients of hydrogen in metals at high temperatures. In the developed statistical calculation model (SCM) the pre-exponential factor of hydrogen diffusion in metals has been calculated from first principle of statistical physics 1). Activation energy of hydrogen diffusion in all metals is determined by correlation with the melting point of these metals. For temperatures below 1000 K, the SCM model uses activation energies calculated from first principles.

Calculated data on the values of the energy activation for metals fall on a straight line relative to the melting point. The experimental values are scattered around this line, especially for the first group of elements, such as Ni, γ-Fe, Pd, Zr, Nb, Mo, W. Second group of metals Al, Ag, Cu, Ti and Hf has experimental activation energies for H diffusion that are greater than the calculated ones. These metals should be used to make alloys for transporting and storing hydrogen.

For the diffusion of H in Fe we obtained good agreement between the values calculated from first principles, the SCM-model and experimental points. In temperature range from 1100 K to 1800 K the SCM-model gives a better description of the experimental points than the basic models using the Arrhenius equation.

For the diffusion of H in Cu, Ti, Mo we obtained very good agreement between the values calculated using the Arrhenius equation and the SCM model over a wide temperature range. For the diffusion of H in Ag, Cr, Ni, W the course of the lines calculated using the Arrhenius equation and the SCM model does not coincide, however, the difference in diffusion coefficients near melting point does not exceed 40%, which is a good indicator for the using SCM-model.

The basic model predicts significantly higher values for the diffusion coefficient of H in Nb and Pd at high temperatures, than SCM-model. However, experimental points on the diffusion of hydrogen in Nb and Pd at these temperatures are unknown.

In our opinion, consideration of the temperature dependence of the activation energy from first principles may well lead to the result predicted by the SCM model for temperatures close to the melting point of Pd and Nb.

The SCM model allows, in contrast to the existing models, to predict the values of hydrogen diffusion parameters for metals in which they are experimentally unknown, as well as for alloys. Thus, for Zr alloys, very good agreement between calculations and experiment was obtained.

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13. Effect of the mixing ratio of Fe and SiO_2 on combustion and melt formation in copper smelting

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The global demand for copper is increasing due to the advancement of electrification technologies. For this purpose, further efficiency improvements of flash smelting that is commonly used in primary smelting of copper are required[1)]. In the flash smelting process, a mixture of sulfide concentrates and silica flux is continuously fed into the reaction shaft with oxygen-enriched air. The oxidation of S and Fe in the concentrates and interfacial reaction with the flux result to form both matte and slag. The ratio of Fe to SiO_2 (Fe/SiO_2 [-]) is one of the parameters that affect the liquidus temperature and is used in the operational guidelines. Nonetheless, the impact of the Fe/SiO_2 in the raw material on the copper concentrate combustion behavior in the shaft remains unclear. This study conducted combustion tests using a small tablet to evaluate the influence of the mass ratio of Fe/SiO_2 on the combustion behavior of the concentrate.

A small tablet of mixed reagents was suspended by an R-type thermocouple and burned in a high-temperature gas with an 80% oxygen concentration at a temperature of 843±5 K [2)]. A high-speed camera was employed to observe the combustion behavior of the sample while recording the sample temperature with the thermocouple. The Fe/SiO_2 was varied from 1.0 to 10 [-] under constant Cu content of 20% or 25% by changing the mixing ratio of $CuFeS_2$, FeS_2 and SiO_2 reagents. The total mass of initially inserted each sample was unified to 25 mg. After combustion, the samples were embedded in resin, cut and polished, then the microstructures and the compositions in their cross-sections were analyzed using an EPMA.

Under the conditions with $Fe/SiO_2 = 1.0$ and 1.5 [-], low-intensity combustions were observed, i.e., the highest temperatures were approximately 1273 K. Local melting of the sample occurred and partially expanded, but some unmelted part remained. Under the $Fe/SiO_2 = 1.0$ [-] condition, numerous unmelted angular SiO_2 particles were retained in the sample after combustion. Under conditions with Fe/SiO_2 higher than 2.0 [-], high-intensity combustions were observed, i.e., the highest temperature exceeded 1673 K and the entire sample melted. Under the conditions of $Fe/SiO_2 = 2.0$ and 3.0 [-], slag was formed over the entire sample, but under the condition of $Fe/SiO_2 = 10.0$ [-], iron oxide, presumably magnetite, was formed near the sample surface instead of slag. This mechanism must be because only a part of the formed FeO became slag due to the low SiO_2 content in the sample, and the unreacted FeO was excessively oxidized in the high-temperature oxidizing atmosphere.

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14. Thin solid state films: the notion of an effective critical radius

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Thin solid state films are widely used in many technologies. They are typically composed of some metallic polycrystalline material which is supported by a ceramic substrate. Reliability in applications requires good control over their stability. Holes may nucleate and then penetrate the film may occur, which may cause in film agglomeration. For some applications, one would like to avoid the appearance of holes; for other applications one would like to maintain an array of small holes which is stable on an appropriate time scale. We approach the question of hole stability by introducing the notion of an effective critical hole radius.

We have been exploring thin film hole stability locally, within the context of special single hole axisymmetric bounded model systems which contain one or two grains which surround a hole. The evolution in our system is nonlinear and focuses on surface diffusion, though various simplifying assumptions are made. Our approach is based on identification and characterization of the steady states, accompanied by investigation of their energetic and dynamic stability properties. We employ a combination of analytic and computational techniques. Our results successfully mimic various experimental phenomena, such as wetting, dewetting, hillock formation, hole closure and thin film break up. Various stability criteria can be identified, including criteria based on the notion of a critical effective hole radius. It appears feasible to incorporate our local models into larger models capable of including various multi-scale effects.

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15. Solid state dewetting of co-sputtered thin Mo-Cu films accompanied by phase separation

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We have fabricated 20 nm-thick Mo-Cu films deposited on basal-plane oriented sapphire substrates employing magnetron co-sputtering method. The films were deposited on the substrates at room temperature, at 300 and 500 °C. The films deposited at the two lower temperatures exhibited Cu-rich particles and hillocks, whereas the films deposited at 500 °C were largely homogeneous and featureless. After partial solid state dewetting in the annealing temperature range of 750-1000 °C in reducing Ar-10 vol.% H_2 atmosphere, Mo-Cu bicontinuous films and Cu-rich particles were obtained, indicating that Cu atoms diffuse much faster than their Mo counterparts. At the final dewetting stages two types of structures were formed: isolated, closely spaced Mo nanoparticles, and large Cu particles decorated with much smaller Mo nanoparticles. The selective etching of Cu with hydrochloric acid has exposed numerous Mo nanoparticles beneath large Cu particles. Copper could also be evaporated during annealing at 1000 °C, leaving Mo particles in the regions previously covered by large Cu particles. However, the Mo nanoparticles exposed by Cu evaporation were much fewer in number and larger in size than their counterparts exposed by etching, indicating that the Mo nanoparticles bound to Cu experience coarsening at 1000 °C. By contrast, no coarsening of the closely spaced Mo nanoparticles outside the Cu regions was observed, meaning a slow Mo diffusion on the sapphire substrate. These morphologies indicated that coarsening of Mo nanoparticles should be related to the Cu evaporation. We formulated a semi-quantitative kinetic model describing the motion of Mo nanoparticles at the edge of a large evaporating Cu particle in terms of Mo self-diffusion from the exposed side of the Mo particle towards the Mo-Cu interface and estimated the effective diffusion coefficient of Mo based on the experimental results. Our results shed new light on the solid state dewetting in the binary immiscible thin films with vastly different diffusivities of the components.

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Invited Lecture

16. High-temperature thermo-physical properties of melts measurement

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High-temperature thermo-physical properties of melts are of critical importance in many applications. This presentation will focus on the measurement of three properties, i.e. the density, the viscosity, and the surface tension. Two techniques were used: The aerodynamic levitation (ADL) coupled with laser heating, which is a contactless method, to measure the three properties and the maximum bubble pressure (MBP), which is a contact method, to measure the density and the surface tension.

The aerodynamic levitator developed at the CEMHTI will be presented and measurements performed on melted alumina will illustrate the measurement technique. Our levitator makes a droplet levitate thanks to a gas flow through a nozzle. Argon and Argon+Oxygen gases were used to study the effect of the gas composition on the properties. The sample was heated with two CO_2 lasers and a high-speed camera recorded the droplet. In addition, speakers controlled with a function generator allowed for to generate an acoustic excitation to make the droplet vibrate at its resonant frequency. Image processing of the video is next used to determine the dimensions of the spheroidal ball and to derive the three properties.

Other results obtained on pure Zirconia will show how we fixed the issue of non-axisymmetric shape of the droplet [1)]. Finally, measurements performed on Al_2O_3-CaO and CaO-SiO_2-Fe_2O_3 slags will be presented to illustrate the relation between the properties, the composition, and the structure of the melts [2, 3)].

In a second part, the maximum bubble pressure device implemented on the PLINIUS platform developed at the LEEAG laboratory of CEA Cadarache and dedicated to nuclear severe accident studies will be presented. The measurement is basically based on the Young-Laplace equation applied to a bubble formed at the extremity of a capillary immersed in the liquid sample. Density and surface tension were measured on different Corium-like samples (U, Zr, O system) [4)]. Effect of temperature, oxidation degree, and U/Zr ratio on the density and the surface tension will be presented.

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17. Thermophysical property measurements of high temperature oxide melts using the Electrostatic Levitation Furnace onboard the International Space Station (ISS)

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JAXA operates Electrostatic Levitation Furnace (ELF) on the International Space Station (ISS). The ELF levitates samples using static electricity on their surface and melts them using high power lasers. ELF can heat the samples to extremely high temperature (ex. 3,000 degree Celsius) without containers. Then, ELF can measure the thermophysical properties (density, surface tension, and viscosity) of high melting point materials. Beside ELF can create metastable phase materials using super cooling. ELF was launched to ISS in 2015. At first, there were many troubles and experiment success rate was low. But now experiment success rate improved drastically by many countermeasures. High speed camera was installed to ELF in 2023, then we can observe samples' solidification behavior during cooling. There are three utilization type for ELF. 1) Domestic basic research, 2) International collaboration, 3) Commercial utilization. JAXA try to expand international collaboration espacially for European users. ELF overview and future prospect will be explained in this presentation.

Figure1 Electrostatic Levitation Furnace on the International Space Station

18. Surface tension model of pure liquid metals: Revisit to Iida's model

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A semi-empirical equation for the surface tension (σ_m) of liquid metals at their melting temperatures proposed by Iida et al. [1)] uses a generalized α value of 0.51 to represent the ratio of the distance required to separate one atomic pair from its equilibrium distance. This study improved the predictability of Iida's equation by refining the α value using the equilibrium interatomic distance (r_e) and the atomic radius (r_a) as proposed by Slater [2)]. The improved α value greatly enhanced the accuracy of the surface tension prediction for elements, such as Bi, Si, Ga, Sn, and Sb, that do not conform to the generalized α value. The present approach yields the R-squared value of 0.939, which is significantly improved from the results of the original Iida's model with the R-squared value of 0.881. Furthermore, the critical temperature (T_c) was calculated using the interatomic distance at the critical temperature (r_c) and the temperature coefficient of density ($d\rho/dT$). The temperature dependence coefficient of surface tension ($d\sigma/dT$) was then determined using T_c and Eötvös law [3)]. The results showed a good linear relationship with experimental data and other theoretically predicted values.

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19. Surface tension of liquid vanadium under the influence of oxygen

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Vanadium is one of the most important alloying elements in the recently upcoming Ti-alloy systems. Despite its high industrial relevance, reliable data on the thermophysical properties, such as surface tension, which is a key property when designing metal manufacturing processes, is still rare. Like titanium, vanadium has a high solubility of oxygen which can significantly impact the surface tension of liquid metals 1). In this work, it is our goal to investigate how oxygen influences the surface tension of liquid vanadium.

In order to process such a highly reactive liquid metal, the non-contact method of electromagnetic levitation was applied. The surface tension is measured by means of the oscillating drop technique. A novel Oxygen Control System (OCS), based on Y_2O_3-stabilized ZrO_2 tubes, was implemented to adjust and measure the oxygen content of the surrounding noble gas atmosphere during the measurement. Accessible oxygen partial pressures range between 10^{-24} an 10^{-3} bar. Vanadium samples were alloyed with different amounts of vanadium(V)-oxide powder, to create samples with oxygen mole fractions x_o between 0.25 at. % and 18 at. %. Afterwards, the surface tension of the samples was measured over a broad temperature range. In a second series of measurements the surface tension of samples of identical composition was measured at a constant temperature while simultaneously increasing the oxygen mole fraction in the process atmosphere. Thereby, the surface tension for liquid vanadium was obtained as a function of oxygen x_O in the bulk as well as oxygen partial pressure in the process atmosphere.

All samples show a linear decline in surface tension with increasing temperature. Simultaneously the surface tension amongst the samples decreases with increasing oxygen mole fraction. Up to a threshold of 1.0 at. % bulk oxygen, only a marginal decrease in surface tension at a constant reference temperature can be observed. Beyond that threshold, a stronger, nearly linear decay in surface tension with $\log(x_o)$ becomes apparent. When repeating the measurements while changing the oxygen partial pressure in the process atmosphere from 10^{-11} to 10^{-3} bar, almost no change in surface tension can be observed for samples with a bulk oxygen mole fraction of up to 3.0 at. %. For samples with bulk oxygen mole fractions larger than 10 at. %, the surface tension slightly decreases when increasing the oxygen partial pressure up to 10^{-4} bar. When further increasing the oxygen partial pressure the surface tension starts to rapidly decrease. It was attempted to link both findings and compare the results with simple, already existing model calculations.

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20. Surface tension of binary and ternary alloys

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More than 70 % of all materials are made directly from the melt. The detailed understanding of the liquid phase is therefore important from both, an economic and a scientific point of view. Accurate thermophysical properties, such as surface tension, play a key role in this context. They are indispensable for any kind of computer aided materials design from the melt. As most materials of technical relevance are multicomponent, the elucidation of their mixing rules is of particular importance.

The present talk is focused on the question how surface tensions of binary and ternary metallic alloys change as functions of their composition and how they are related to each other for chemically similar systems.

In order to answer this question, data is measured for a number of systems by means of the oscillating drop method combined with electromagnetic levitation. The degree of complexity of the alloy material is, hereby, stepwise increased from mono-atomic systems to binary and ternary alloys. Data is obtained as functions of temperature and, in the case of alloys, of their corresponding compositions.

The results are discussed using the following thermodynamic models: The Butler equation 1), the Chatain model 2), both for regular or sub-regular solutions, and the Egry model 3) for compound forming systems. The advantages and shortcomings of each model are discussed in detail and a comprehensive rating of the performances of the different models is presented.

In addition, it is generally found that systems with negative excess free energies exhibit a weak surface segregation while systems with positive excess free energies exhibit a pronounced one. As a rule of thumb, the surface tension of a ternary alloy can be estimated from the properties its constituent binary sub-systems without any assumption or knowledge of potentially existing ternary interaction terms.

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21. Surface oscillation phenomena of compound drop by liquid iron and molten oxide under microgravity conditions with molten oxide surface tension analysis

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Compound droplets' surface oscillation has been a subject of interest for a long time in basic fluid science and materials processing applications. However, experimental investigation of large density difference liquids has been challenging due to weight differences. As a result, investigations have been limited to small-density difference liquids, where it's difficult to observe interface oscillation effects due to small amplitude differences. Under microgravity conditions, two immiscible liquids, even in large-density difference liquids, form a core-shell droplet that is dominated by surface and interfacial free energies 1). This makes surface oscillation phenomena of the core-shell droplet by the large-density difference liquids interesting for new knowledge of drop oscillation phenomena and its application in materials processing. To this end, we have observed the surface oscillation phenomena of compound droplets by immiscible liquids, such as liquid iron and molten oxides, using the electro-static levitation furnace (ELF) installed in the International Space Station (ISS) 2). The shape of the compound droplet by immiscible liquids is determined by the minimum of the total surface free energy of them. The total surface free energy is the sum of the surface tension multiplied by the free area and the interfacial tension multiplied by the interfacial area. By using a core-shell droplet, we can apply the modified drop oscillation analysis by two interface conditions in liquid-liquid and liquid atmospheres. The normal mode of the core-shell droplet has two separate eigenfrequencies in fundamental oscillation. These eigenfrequencies enable us to calculate the interfacial tension value. Our onboard experiments of observing compound drop oscillation phenomena using ELF have been performed since October 2022. We succeeded in observing the surface oscillation phenomena of the compound droplet by molten oxide (SiO_2:CaO:Mn_3O_4:TiO_2: Fe_2O_3=25:7:20:18:30 mass%) and liquid Fe in an Ar atmosphere. We obtained two frequencies from the power spectrum of FFT analysis of the compound drop surface oscillation with the resonant frequency conditions. We could obtain the interfacial tension from two frequencies of the compound droplet's surface oscillation. Furthermore, during the observation of oscillation phenomena, an increase in droplet volume was observed. This may be due to the oxidation of liquid Fe at the interface with the molten oxide. When oxidation occurs, the density and surface tension of the shell molten oxide change due to Fe incorporation into molten oxides. In this presentation, we discuss the surface oscillation of compound droplets under microgravity conditions and their interfacial properties, also we discuss the oxidation of liquid iron at the interface of molten oxides.

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22. The new paradigm of thermodynamics of colloid and interface science

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In the most influential monograph on colloid and interfacial science by Adamson 1) three fundamental equations of "physical chemistry of surfaces" are identified: the Laplace equation, the Kelvin equation and the Gibbs adsorption equation, with a mechanical definition of surface tension by Young as a starting point. In contrary it is shown here that there is only one fundamental equation of the thermodynamics of colloid and interface science (considered a new paradigm here 2)) and all the above (and other) equations of this field follow as its derivatives. This equation is the fundamental equation of Gibbs including the surface term. Positioning the single fundamental equation of Gibbs into the thermodynamic origin of colloid and interface science leads to the following coherent set of equations:

i. the general equation for interfacial forces, leading to the Young equation, to the Bakker equation, to the Laplace equation, etc; although the principally wrong extension of the Laplace equation formally leads to the Kelvin equation, using the new paradigm it becomes clear that the Kelvin equation is incorrect.
ii. the equations that provide equilibrium shapes and positions of phases, including sessile drops of Young, crystals of Wulff, liquids in capillaries, etc.
iii. the size-dependent equations of molar Gibbs energies of nanophases and chemical potentials of their components; from here the corrected versions of the Kelvin equation and its derivatives (the Gibbs-Thomson equation and the Freundlich-Ostwald equation) are derived, including equations for more complex problems.
iv. the nucleation theory of Gibbs that also contradicts the Kelvin equation.
v. the adsorption equation of Gibbs, and also the definition of the partial surface tension, leading to the Butler equation and to its derivatives, including the Langmuir equation and the Szyszkowski equation.

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23. Hybrid solder joints: Thermodynamic and calorimetric studies of the Sn-based Fe-Sn(SAC305) alloys

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The Sn-3.0Ag-0.5Cu (SAC305) alloy is one of the most promising alternatives to the Pb-Sn solder alloys. The alloy is RoHS, REACH, and JEIDA compliant as well as compatible with all commonly used no-clean and water-soluble electronic grade fluxes. However, the next generation of the Pb-free solder alloys is under discussion due to the higher melting point compared to the Pb-Sn solders and the mechanical reliability issues of the solder joints. Minor additions of the nanosized metal inclusions is one of the promising solutions to enhance the microstructure of the solder joints and improve their mechanical reliability.

The present study is focused on the investigations of the thermodynamic properties of the Sn-based Sn-Fe alloys as a part of the quaternary SAC305-Fe alloys. For instance, the enthalpy of mixing was investigated by drop calorimetric technique. During the experiments, the bulk and nanosized Fe was dropped into the liquid Sn. The enthalpy of mixing has been calculated based on the heat effects recorded after each separate drop. The measurements were performed at 1073 K and the results have been compared with the literature.

In addition, differential thermal analysis (DTA) of the Sn-Fe and SAC305-Fe alloys with up to 2.0 wt.% of the nanosized Fe powder was performed to identify phase transformations and the corresponding temperatures. Two heating and cooling cycles were performed for each sample. It is supposed that nanoparticles were completely melted during the first heating up to 1673 K and the samples were transformed from nanocomposite to the bulk quaternary alloys.

The microstructure of the samples after DTA and calorimetric studies was analyzed by scanning electron microscopy (SEM). The experimental results were compared with the literature.

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24. New active brazing alloys based on Ni-Nb-Ta

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Active brazing alloys are widely used for the joining of metals and ceramics. Common active elements that are able to reduce and wet refractory ceramics are titanium, zirconium or hafnium. Alternative active elements could be niobium [1] or tantalum. Recently, we have developed a method to find alloy compositions based on Ni-rich Ni-Nb-Ta alloys that appear as promising candidates for metal ceramic joining. Based on high-throughput thermodynamic calculations of over 20’000 compositions we identified 10-15 promising multicomponent alloys that were arc- melted and homogenized. Their microstructure and melting range were characterized by SEM and DTA, respectively.

Based on the results, the most promising alloy for the production of a solder paste was selected and feasibility studies were conducted. Wetting tests on oxide- and silicide ceramics were done in a pre-evacuated furnace under flowing Ar gas in order to determine the wetting angle and the surface tension by the sessile drop method. The interface of ceramic and metal was evaluated by FIB cross section in the SEM. Powder of the alloy of interest was produced by ultrasonic plasma atomization and the particles were subsequently examined for size distribution. In the future, feasibility studies of the atomization process to industrial level are planned. Furthermore, the exploration of their applicability and the production of brazed parts will be investigated. Finally, long-term stability of the solders will be examined.

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25. High-Entropy Alloys on graphite: wetting and reactivity evaluation by CALPHAD method

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High-Entropy Alloys (HEAs), studied during last years with the aim to replace Ni- and Co-based super-alloys, have been more recently considered for sectors involving liquid-phase processing routes, such as brazing, casting, infiltration to produce composites, production of coatings, etc. Among the broad family of HEAs, AlCoCrFeNi shows appealing mechanical properties, and excellent corrosion and oxidation-resistance; the addition of refractory metals, such as Mo and Ta, was found to enhance the formation of secondary phases (e.g. σ phase or Laves-C14) improving hardness, compressive and yield strength.

In this study, wettability and interfacial reactivity of AlCoCrFeNi, AlCoCrFeNiMo and AlCoCrFeNiTa in contact to graphite were investigated for the first time. An in house-built thermodynamic database, including all HEAs elements and carbon [1, 2)], was used, together with CALPHAD method, to simulate and discuss liquid-solid interfacial phenomena occurring at high temperatures. This combined experimental-theoretical approach, already adopted by the authors [3)], allows to increase the reliability of the database representing, as the same time, a fundamental tool to define the most promising conditions for reaching the desired materials performances.

Specific wettability tests were conducted in an ad-hoc furnace, under inert atmosphere, where the alloy is positioned on a graphite disk. Graphite-HEA couples were extracted from the hot-zone to the cold-zone of the furnace after 10 minutes of contact, then cross-sectioned and their interfacial reactivity studied by means of SEM-EDS and CALPHAD approach. Depending on the system, formation of various kinds of BCC-phase was mainly observed as well as mixed C-rich phases, detected not only at metal-ceramic interface but also inside the bulk of the drops. This investigation constitutes a preliminary study of liquid alloys-graphite interactions that can support further investigation devoted to the production of brazed joints, composites or high entropy ceramics.

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26. Design of new HEAs as brazing fillers: wetting, CALPHAD modelling and mechanical characterization

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The full exploitation of the so-called High Entropy Alloys (HEAs) for high-temperature applications requires to understand the high-temperature interfacial phenomena involving these alloys in the liquid state in contact to solid materials. Applications that foster these studies are joining by brazing, selection of refractories in foundry, and production of metal-reinforced composites or refractory coatings.

In this work, a specific composition, namely $Al_{0.15}CoCrFeNiW_{0.15}$, has been designed by the aid of CALPHAD thermodynamic modeling with the aim to explore the possible use of this alloy as brazing filler for the joining of C- and SiC-based materials for high-temperature applications.

Sessile drop tests were conducted at 1500 °C to test the wetting and the interfacial reactivity of this alloy in contact to graphite, SiC and C_f/SiC composites. Microstructural characterization and thermodynamic calculations were performed to interpret the experimental results and to desume the interfacial interactions occurred during high-temperature wetting tests. Interfacial dissolution and reactivity were observed and, in order to control these deleterious phenomena, an improved alloy was designed by introducing Si into the starting HEA. The most promising composition, $Al_{0.15}CoCrFeNiW_{0.15}$ + 40 at.% Si, was used as a brazing filler to produce SiC-SiC and C_f/SiC-C_f/SiC joints, and the overall strength was determined by shear tests at room temperature.

27. Evaluation of contact angle and wettability of calcium sulfide against molten iron at 1873K

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It is known that wettability is the dominant characteristic in the inclusion removal from molten iron.1) There is plenty of research regarding the contact angles between various oxides and iron melt. However, only one report can be found about the contact angle between calcium sulfide and iron melt, where the contact angle has been reported as 87 degrees.2) Although solid oxides at steelmaking temperatures generally have a contact angle above 90 degrees, calcium sulfide was regareded as a good wettability phase against iron melt, even though it is a solid state. On the other hand, Yoshikawa *et al.*3) suggested that the contact angle of CaS/molten steel is larger than that of CaO/molten steel, whose contact angle is above 90 degrees. These reports show that consensus view on the wettability between calcium sulfide and iron melt has yet to be derived. This study aims to clarify the contact angle between molten iron and calcium sulfide by the sessile drop method and investigate the effect of sulfur content on the contact angle at 1873K. As a result, the contact angle between molten iron and calcium sulfide under Ar atmosphere was measured as 120 degrees, which was as large as those of solid oxides. The contact angle decreased with an increase in sulfur content in the melt. The effect of other content in the iron on the contact angle will be discussed at the conference.

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28. Dynamic wetting behavior of molten Al on α-Al_2O_3 substrate during heating under high magnetic field

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The wetting behavior of molten metals on ceramics holds significant importance across various applications. Fundamentally, the influencing factor for wettability is interfacial energy. Therefore, researchers have explored various approaches to alter interfacial energy, thereby regulating wettability. High magnetic fields exhibit exceptionally powerful magnetic, force, and energy effects, capable of modifying the energy states of substances placed within it. Consequently, it is feasible to modulate wettability using a high magnetic field. We investigated the dynamic wetting behavior of molten Al on C-plane α-Al_2O_3 and R-plane α-Al_2O_3 substrates during heating from 525°C to 750°C under both 0 T and 6 T high magnetic fields. It was observed that, compared to conditions without a high magnetic field, molten Al on both oriented α-Al_2O_3 substrates achieved stability more rapidly under a 6 T high magnetic field. Without a high magnetic field, the spreading process exhibited three stages: three-phase line contraction, three-phase line stagnation, and stabilization. In contrast, under the 6 T high magnetic field, there were two stages: three-phase line spreading and stabilization. During the dynamic wetting process, the driving force originates from the significant reduction in the liquid-solid interfacial energy under high magnetic fields, while the resistance is attributed to the friction between the molten metal and the substrate, as well as the hindrance posed by the oxide film on the molten metal surface. Variances in the relative balance between driving forces and resistance during heating under 0 T and 6 T high magnetic fields resulted in the two distinct spreading processes. Additionally, after achieving stability under the 6 T high magnetic field, the equilibrium contact angle was significantly smaller (below 90°), and the spreading diameter (the diameter of the contact area between the molten metal and the substrate) was significantly larger. These findings present new possibilities for manipulating the wetting behavior of molten metals using high magnetic fields.

29. The wettability of molten metals with different magnetic properties on solid substrates under high magnetic fields

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Wettability is defined as the ability of a liquid to spread out and replace the gas on a solid surface. It is a major parameter in the fields of materials, metallurgy, biology, and manufacturing. Various methods of manipulating wettability fundamentally achieve this by altering the energy states of the system. High magnetic fields exhibit exceptionally powerful magnetic, force, and energy effects, capable of operating at the atomic scale. It is feasible and yields significant effects to regulate wettability through high magnetic fields. We investigated the wetting behavior of molten Al (paramagnetic) and molten Sn (antimagnetic) on the same solid substrate under high magnetic fields. It was observed that the contact angles of the Sn/Al_2O_3 and Al/Al_2O_3 systems both significantly decreased under high magnetic fields. The contact angle of the Sn/Al_2O_3 system exhibited a linear decrease with the increase in magnetic flux density, while the Al/Al_2O_3 system showed a non-linear decrease. With or without a high magnetic field, the contact angles of the Al/Al_2O_3 system both decreased with the increase in temperature. Without a high magnetic field, the contact angles in the Sn/Al_2O_3 system decreased with temperature, whereas under high magnetic fields, they increased with temperature. Under high magnetic fields, the wetting behavior trends of molten Al and molten Sn on an α-Al_2O_3 substrate were found to be similar to those observed on a Al_2O_3 substrate. During the continuous variation of magnetic induction intensity, the contact angle of the Sn/α-Al_2O_3 system gradually decreased to a minimum as the magnetic induction intensity increased, followed by a slow increase as the magnetic induction intensity decreased to zero. In contrast, the Al/α-Al_2O_3 system exhibited a trend of initially decreasing, then remaining unchanged. Additionally, with the increase in magnetic flux density, the contact angles of both Al/SiO_2 and Sn/SiO_2 gradually decreased. Furthermore, high magnetic fields, in conjunction with material characteristics and temperature, couples to influence the wetting behavior of molten metal on a solid substrate. In essence, a high magnetic field affects wetting behavior through Lorentz forces and magnetization energy on interfacial energy.

30. Wettability, high-temperature interaction, and brazing of (Mo–Nb–Ta–V–W)C high entropy carbide by NiTa eutectic alloy

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Abstract

In this work, the wettability and interfacial reactions of molten Ni and NiTa alloys on the (MoNbTaVW)C high entropy carbide (HEC), and the joining of HEC ceramics with NiTa alloy were investigated. The results showed a significant dissolution of the ceramic substrate in Ni alloy when the wetting test was performed at 1480 °C. By adding Ta metal to Ni (an eutectic alloy), the interfacial reaction and dissolution were limited. A low wetting angle was observed for both wetting tests, Afterward, the wetting studies results were transferred to the brazing of HEC ceramics to themselves by NiTa alloy. The pressure-less joining was performed using the field-assisted sintering technique (FAST). The thickness of the interlayer was controlled by the weight of the particles. The interfacial bonding between the HEC ceramics and the interlayer was investigated using scanning electron microscopy (SEM). The phase and microstructure analysis of the joints revealed that due to the formation of an interfacial layer along the interface, a strong joint with a shear strength of 218 MPa was obtained.

Keywords: high entropy ceramics, joining, brazing, wettability, NiTa

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31. Development of optical guiding crystal scintillators for high resolution radiation imaging

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Scintillating fibers were widely used for radiation detection applications such high energy physics, medical imaging, space observation and so on due to its long shape with flexibility, wavelength shifting and light guiding performance like optic fibers. Up to now, our group have developed eutectic scintillators (fig.1-left). The eutectic crystals have a structure in which scintillator crystal fibers of several μm diameter are arranged in a matrix and have excellent position-resolving performance against x-rays and charged particles [1, 2]. However, it was extremely difficult to grow the eutectics enough large or long size, and it was impossible to grow them as a single fiber. In this study we proposed a novel optical-guiding crystal scintillator (OCS). It consists of halide single crystal scintillator core and glass clad. The refractive index of the halide single crystals is higher than the glass in this system. Generated scintillation light above the critical angle is totally reflected at the interface with the glass and optically waveguided like optical fibers and the scintillating fibers (fig.1-right). In OCS, the molding of the cladding and the crystal growth of the scintillator core are performed in the same process. By adjusting the fabrication conditions, the fiber system can be tuned from a few microns to several hundred microns in diameter. The thickness ratio of the core to cladding is also variable by changing the thickness of the glass material. As cladding, quartz glass and borosilicate glass have been tested as available, and halide crystals with melting points below their softening points can be used as the core. Tl:CsI, Eu:SrI$_2$, CeBr$_3$, etc. were examined as scintillator cores, and borosilicate glass were used as cladding to construct OCS fibers. OCS was not limited to single fiber but could also be formed into bundles for high resolution radiation imaging.

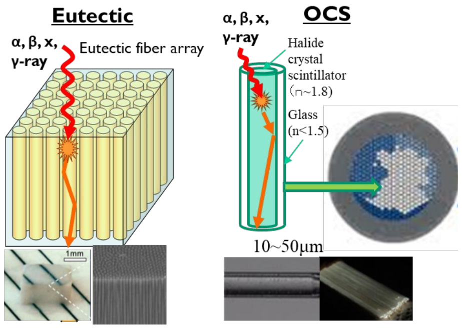

Fig. 1. Schematic of the structures of the reported eutectic scintillator (left) and developed OCS (right)

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32. Reduced-weight ceramic composites for armor applications produced via reactive melt infiltration

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Silicon carbide (SiC)-based composites are lightweight materials with exceptional mechanical performance under impact loading (i.e. high energy absorption upon failure), which makes them suitable for personal armor applications. Production of monolithic SiC is though technologically challenging due to its low sinterability caused by a low self-diffusion coefficient and a high vapor pressure of its native oxide. A more economical approach to manufacturing monolithic specimens is the use of silicon reactive melt infiltration. In this process, carbon-coated SiC powder is consolidated via direct reaction with molten silicon to form new SiC that acts as the bonding phase between the particles. This process requires milder temperatures compared to sintering and is a near net shape process which enables the generation of complex geometries. However, residual silicon is inevitably trapped within the microstructure of the densified material (5-15 vol%). The presence of silicon hinders the mechanical behavior of the material due to its low fracture toughness. It's partial or total replacement with a tougher phase is desired to maximize mechanical performance.

In this work, the infiltration of bimodal particle size SiC/C porous bodies with Si-Me alloys is explored as a means to i) enhance mechanical performance during high strain rate compression (impact) and ii) reduce overall weight of the material. High strain rate compressive strength of produced specimens was measured with the Split Hopkinson Pressure bar system (SHPB). Infiltrated materials were characterized via XRD, SEM and Archimedes balance to determine residual porosity.

33. High-temperature vacuum brazing of a heat-resistant nickel alloy containing 20 % chromium

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For the manufacture of gas turbine engines and power plants, multi-component nickel alloys are used, which contain an increased concentration of chromium (16-20 %). Chromium increases resistance to high-temperature corrosion, which can be caused by the aggressive environment of diesel and gas fuel and salts from seawater. Such alloys are characterized by extremely unsatisfactory weldability. They are prone to the formation of hot cracks, both during brewing and after heat treatment. Therefore, high-temperature brazing is a promising method for obtaining joints from such alloys, which, subject to the correct selection of the temperature regime and the chemical composition of the solder, can help avoid the formation of cracks in soldered joints.

The Ni-Cr-B-Si-(Co-Mo) brazing filler metals are widely used for brazing such materials. When they are used, Cr_xB_y borides are formed, which are observed both along the seam axis and along the grain boundaries of the base metal at a distance of 60-70 microns. They have a negative effect on the mechanical properties during operation of brazed products in conditions of elevated temperature. Long-term heat treatment after the brazing crystallization process does not completely avoid the formation of harmful brittle phases in the brazed joint.

This paper presents the results of metallographic and micro-X-ray spectroscopic studies of brazed joints (of heat-resistant nickel alloy containing 20 % Cr), which were obtained by high-temperature vacuum brazing using nickel brazing filler metals with reduced boron concentration (Ni-Cr-Co-(Me)-B and the effect of silicon on the structure and mechanical properties of joints at room and elevated temperatures is shown.

The results of micro-X-ray spectral studies confirmed the formation of dense defect-free brazed joints with a structure that contains a nickel-based solid solution, needle-like chromium borides, which are released randomly in the brazed joint. In addition, in the presence of silicon (up to 5 % in brazing filler metal) in the brazing seam, nickel silicide and skeletal eutectics (solid solution based on nickel + silicide phase) are formed. By reducing the concentration of silicon (or excluding it from the composition of the brazing filler metal), it was possible to reduce the amount of the silicide phase and avoid the formation of eutectics. Mechanical tests at room and elevated temperatures show the promise of brazing filler metal that does not contain silicon and provides high tensile strength at room temperature and 900 °C, respectively 739 and 423.7 MPa.

34. Effects of alloying element additions on shape-controlled single crystal growth of Ru alloys by the dewetting μ-PD method

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The dewetting micro-pulling-down (μ-PD) method is a continuous casting technique using a ceramic crucible for rapid, shape-controlled growth of metallic single-crystals. It is particularly effective for creating wires from brittle, high-melting-point alloys such as Ir (m.p.: 2446°C) and Ru (m.p.: 2334°C), which are prone to intergranular fracture. This method has potential applications in spark plugs, thermocouples, and high-efficiency resistance heating wires [1-3] .
In traditional μ-PD methods with better melt wettability, increasing the pulling rate from a few to tens of mm/min causes meniscus stretching and rupture. However, recent findings suggest that with certain melt-insulator (die) combinations, pulling rate of several hundred mm/min are feasible, enhancing shape controllability [2].
The variation in crystal diameter with pulling rate can be attributed to the dynamic change in the contact angle between the melt and the die. It's assumed that this dynamic contact angle's dependence on the pulling rate differs from system to system, which accounts for the varied shape controllability across different materials [3]. The behavior around the meniscus in the duetting μ-PD method is still unclear, and detailed investigations have been limited to pure materials. Therefore, in this study, we investigated the effect of alloying elements additions.

In this study, Ru crystals with a diameter of 0.8 mm were grown. Pure Ru and alloys with additive elements (Pt, Mo, W, etc.) were grown to evaluate the achievable pulling rate, the surface condition of the wire, and how the diameter changes. While the crystal growth rate of pure Ru was limited to a few tens of mm/min, faster crystal growth (~300 mm/min) was achieved by the addition of alloying elements. In the presentation, the effect of alloy addition on the velocity dependence of the dynamic contact angle will be discussed further.

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35. Structure evolution in the melting-solidification processes of Fe-based metallic amorphous alloys induced by laser heating

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Amorphous metal alloys, due to the combination of their unique physical and chemical properties, are promising for use in many fields of application including the materials producing technologies. First of all, due to great strength and corrosion resistance, as well as unique magnetic properties, such materials attract the considerable attention. However, the most widely used methods for the synthesis of metal amorphous alloys make it possible to obtain them in the form of powder or thin ribbons. Only some of such materials can be obtained with a size of about a few millimetres in one dimension. It is clear that in order to effectively use all the advantages of amorphous alloys, it is necessary to develop technologies for obtaining them of any size and shape. The most promising technology in this field is the method of selective laser sintering of powder amorphous alloys and its use for obtaining three-dimensional objects by 3D printing methods [1)]. Usually, the most common problem at obtaining amorphous alloys by this method is their crystallization in the process of laser sintering. In order to avoid crystallization processes, it is necessary to select such modes of laser irradiating, which would allow the material to be cooled quickly enough after melting for further amorphization. There is a lot of research in this direction [2)], but up today there are still many unanswered questions regarding the process of forming bulk amorphous alloys using additive technology methods.
In this work, the availability of laser processing of $Fe_{73.5}Nb_3Cu_1Si_{15.5}B_7$ amorphous alloy at low laser radiation powers to control the phase content and structure was investigated. For the study, both a powder amorphous alloy and an alloy in the form of a ribbon were used. Amorphous alloys were irradiated with different power of the laser beam and with different scanning speeds. The methods of electron microscopy and X-ray structural analysis were used to study the microstructure and atomic structure of alloys.

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36. Effect of prolonged aging at sub-zero temperatures on the properties of lead-free solders

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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, such as electrical and thermal conductivity, thermal resistance, fatigue and creep resistance, yield strength, etc. Ternary Sn-Ag-Cu alloys of eutectic or near-eutectic compositions are considered the most promising and are widely used as solder materials. The reliability of solder joints is determined by the intermetallic compound formed at the joint interface. To improve the properties and strengthen the base solder matrix and joints, various metal, bimetal and ceramic micro- and nanosized admixtures, are added. The non-wettable ceramic admixtures are coated by metals to form core-shell structures on their surface and to improve adaptation to the solder matrices. As a result, the metal-coated layer forms a strong “bridge” that reacts 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 subzero temperatures, since, e. g. for applications in aerospace conditions, it is important to know the behavior of mechanical properties of soldered components during operation in extreme thermodynamic conditions. The use of Sn-based solders in microelectronic devices at cryogenic temperatures may not be suitable because they can become brittle due to the allotropic phase transformation of tin, known as "tin pest". Along with the cryogenic temperature range, ensuring the efficient operation of electronic devices for the military, medical, etc. industries is also important at sub-zero temperatures in terrestrial conditions.

The results of the influence of various metal, ceramic, and carbon nanoscale impurities, both uncoated and coated with Au, Au-Pd, Ni and Pt metals, on the structure, thermophysical and mechanical properties of Sn-Ag-Cu solders and solder joints both at room temperature and after several months of aging at sub-zero temperatures are presented and analyzed.

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37. Interfaces in biomaterials

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This presentation outlines the importance of interfaces between the consisting parts (i.e. workpieces of materials) of biomaterials, which are produced by high temperature processing. The first part focuses on the selection of materials that can be eligible for materials aimed at biomedical applications. Alumina and zirconia are considered as the most popular bioceramics; at the other side of the interface, Ti and its alloys are also popular metal materials vastly considered for biomedical uses. Thus, the possible (ceramic/metal) combinations between them and the chemical features and the bonding strength of the resultant interfaces will be presented and discussed in the light of the thermodynamics of the systems. Applications using other types of bioactive glasses produced by modern methods (such as plasma spray) will be presented. Since dental restorations is a special case of biomedicine of hard tissues, the second part will focus on the interfaces developed in materials used in dentistry. Nowadays, very modern techniques are used to prepare them. Besides materials' biocompatibility, the long-term robustness of the produced materials and their good aesthetics are two tough (and still challenging) requirements in modern dentistry. Thus, ISO standards (e.g., the ISO 9693) have been developed and globally established to describe in detail the required features of the modern dental materials. More specifically, there are two types of dental restorative materials for fixed dental prosthetic restorations: the alloy-ceramic systems and the all-ceramic restorations. In the former case, Co-Cr dental alloys are safe materials used as substrates and in the latter case zirconia is nowadays the currently used and most promising ceramic material. The presentation will show the application of the ISO 9693 in the above two cases of dental materials, i.e. their production as well as the results of the bonding strength at the interfaces developed in them (i.e., between the substrates and the veneering porcelains) as far as the satisfaction of the bonding strength required by this ISO standard is concerned.1)2)

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POSTERS SESSION

(P1) Wettability of carbon surface by molten potassium and cesium tetrafluoro aluminate ($KAlF_4$, $CsAlF_4$) complex salts

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The interfacial behavior and the wettability of carbon surface by molten potassium and cesium tetrafluoro aluminate salts were examined in high temperature experiments and also sessile drop method. In the latter case the silhouettes of the molten phase on the carbon substrate were recorded by a CCD camera, and the contact angles were determined by dedicated software.
According to the experiments we found that the melt of the cesium containing complex salt shows better wettability on the carbon surface than the potassium tetrafluoro aluminate. This interfacial behavior is so pronounced that the salt melt penetrates into the micron sized pores of the graphite.
Graphite is also used as the anode material for solid-state lithium-ion batteries, so this phenomenon can also be used in this field as well, e.g. to separate graphite from other materials.

(P2) Analysis of surface oscillation of core-shell droplet by liquid iron and molten oxide under microgravity conditions

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Compound droplets have been of interest for their surface oscillation from a basic fluid science perspective and their use in material processing. However, investigating this phenomenon on the ground is difficult due to the weight difference between liquids of large density differences in compound droplets. Under microgravity conditions, two immiscible liquids can easily form a core-shell droplet, which is dominated by surface and interfacial free energies. By forming the core-shell droplet, we have been studying the interfacial tension between liquid iron and molten oxides, important in steel manufacturing. We can obtain the interfacial tension from the surface oscillation frequencies of the core-shell droplets. The eigenfrequencies of surface oscillation of core-shell droplets can be used to obtain interfacial tension, as derived from the two boundary conditions of the spherical fluids model by Suffern et al.1) To obtain the eigenfrequencies of the core-shell droplet, we have been performing levitation experiments using the electrostatic levitation furnace (ELF) installed in the International Space Station (ISS). In the ELF experiments, to obtain eigenfrequencies of the core-shell droplet we need an estimation of the eigenfrequency valued before experiments to apply resonance oscillation for generation of the surface oscillation of the core-shell droplets. Since the eigenfrequencies are the function of the ratio of surface tension and interfacial tension, the radius ratio of shell and core, and the density difference ratio of core and shell, any solutions exist for these parameters' combinations. Our core-shell droplet sample of liquid iron and molten oxide undergoes oxidation at the interface, requiring us to change the radius ratio of the core and shell with time variation. We need the eigenfrequencies as the function of the radius ratio of the core and shell for the experimental conditions. We put additional boundary conditions from the total surface free energy conditions to the eigenfrequencies derived by Suffern. We obtained the eigenfrequencies as the function of the radius ratio of the core and shell using these boundary conditions. Figure 1 shows the estimated eigenfrequencies of the core-shell droplet by liquid iron and molten oxides as a function of the radius ratio under the constant surface tension value of molten oxide. With this procedure, we succeeded in obtaining the interfacial tension between liquid iron and molten oxide at a temperature of around 1900 K.

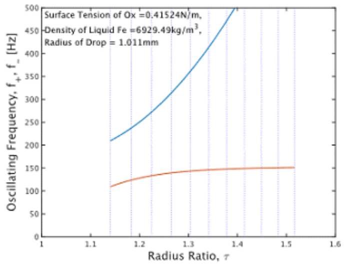

Fig.1 Estimated eigenfrequencies of the core-shell droplet by liquid iron and molten oxides. The boundary condition of total surface free energy is also taken into account.

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(P3) Oxygen potential in cast irons – A thermodynamic discussion on the influence of temperature and Mg treatment

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The activity of dissolved oxygen in cast irons affects the solidification behaviour, for example, the surface/interfacial tension will be influenced, and as a consequence, the graphite morphology and nodularity will be influenced.

In the present study, foundry trials for laminar graphite iron (LGI), compacted graphite iron (CGI) and spheroidal graphite iron (SGI) have been performed for 1 ton of melt. The change in the dissolved oxygen potential was measured during cooling by using a commercially available oxygen sensor, which consists of MgO stabilized ZrO_2 electrolyte and Cr/CrO_2 reference. For the CGI and SGI cases, magnesium treatment has been performed. The influence of the temperature and the amount of added magnesium were thermodynamically discussed.

For the LGI case, it was found that the measured oxygen potential change during the cooling reasonably agrees with the thermodynamic calculation (equilibrium calculation) results when the generation of CO gas is set as “dormant”, i.e., when the CO gas generation is not considered in the calculation.

In general, the oxygen potential becomes lower with the decrease in temperature. In the case of SGI, the decrease of the oxygen potential during cooling becomes moderate as the temperature decreases below a specific temperature (a small kink was observed at the specific temperature in both measurements and calculations). On the other hand, in the case of CGI, the slope of oxygen potential change versus the decrease in temperature becomes steeper below a specific temperature. It was found that this difference is due to the formation of different types of compounds during the cooling. In the case of SGI (i.e., a relatively high amount of Mg addition), MgS will be formed during the cooling. On the other hand, in the case of CGI (i.e., a relatively low amount of Mg addition), $Al_2O_3 \cdot MgO$ will be formed instead of MgS according to the thermodynamic calculation.

The findings in the present study will facilitate a deeper understanding of the oxygen potential change during the solidification and help to determine the required magnesium addition amount. In addition, it will help to estimate the oxygen potential at lower temperatures near the melting point at which the measurement is challenging due to the experimental difficulties.

(P4) Sulfur adsorption on the liquid Fe-Si alloy surface

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Fe-Si alloy is the most representative soft magnetic material. Due to its excellent magnetic properties characterized by low eddy current loss and magnetic anisotropy, it is widely used as a core material for motors and transformers. There is a heightened emphasis on reducing CO_2 emissions by enhancing the energy efficiency of Fe-Si-based electrical steel, particularly by minimizing energy loss. Subsequently, the quality control of the electrical steel is highly anticipated to increase the performance of electric appliances. The surface tension of liquid Fe-Si alloys plays an important role in controlling bubbles and inclusions in the steelmaking and casting processes, affecting the final quality of steel. Sulfur, a representative surface-active element in steel, significantly influences the surface tension and the temperature gradient by surface adsorption of sulfur. However, the sulphur adsorption behavior of the Fe-Si alloy remains unexplored. In the present study, the surface tension values of liquid Fe-3wt.%Si-S alloys measured by the constrained method 1) with varying sulfur concentrations (8 ppm to 360 ppm) and temperatures (1783 K to 1853 K) were applied to analyze the surface adsorption behavior of sufhur. Considering Fe and Si sites on the surface, the two-step adsorption model 2-4) was employed. The present study suggests that the initial step involves sulphur occupancy at both Si and Fe sites. Once the Si sites are completely occupied, adsorption on Fe sites becomes dominant. It is concluded that the presence of silicon promotes sulfur adsorption to the surface of the liquid Fe-Si alloy, which is attributed to the high binding energy between silicon and sulfur.

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(P5) Exploring particle genesis from mold-metal interaction phenomena in compacted graphite iron cast components using automated particle analysis

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A common goal of the automotive industry is to improve fuel efficiency by designing and manufacturing lightweight cast components to meet environmental regulations and recent sustainability targets. To offset the weight benefit of light metal alloys, cast iron components are usually cast with close tolerances and minimum machining, retaining most of their as-cast surface. However, current research has shown that the surface microstructure can differ from the bulk microstructure, primarily due to mold-metal interaction phenomena. This surface deviation, mostly perceived as graphite degeneration for compacted and spheroidal graphite cast irons, stems mostly from Mg depletion at the interface. This phenomenon is mostly associated with the increased S and O concentration in the vicinity of mold and core interfaces due to S-based binders and residual humidity thermal decomposition. Following this, some observations have shown that complex particles - mostly sulfides and oxides - may form at the interface and potentially migrate into the bulk material [1-3)]; S and Mg concentration distribution profiles with increasing distance from the surface have also been measured [4, 5)]; however, the differences in particle populations between the bulk of the material and its interfaces against the mold and cores have not been statistically assessed.

The present work aims to explore the possibility of using the *EDAX Genesis software*, based on automated particle analysis and X-ray spectra acquisition, using Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectrometry (EDS), respectively, to assess and quantify large particle populations. The method combines high-resolution imaging of SEM with semi-quantitative analysis of EDS, focusing on sand mold-metal and core-metal interfaces and comparing them to the bulk material. The compacted graphite iron samples analyzed in this study were sourced from two distinct foundry facilities, each characterized by slightly different chemical compositions and process parameters.

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(P6) Effect of oxidation liquid Fe on density and surface tension of molten oxide in compound droplet using Electrostatic Levitation experiment in ISS

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Interfacial phenomena between immiscible liquids are interesting from scientific and technological viewpoints in many fields. In the field of steel manufacturing, interfacial phenomena between molten oxide and liquid Fe are important for process control. Therefore, the interfacial tension between them is required for using process control simulations. From the background, we have performed interfacial tension measurement using the surface oscillation of compound droplets by molten oxide and liquid Fe in the electrostatic levitation furnace (ELF) in ISS. On ground conditions, it is difficult to form a compound droplet by molten oxide and liquid Fe from the large difference in their density; however, under microgravity without the density difference compound droplets can be easily formed. Using the core-shell droplet, we can apply the modified drop oscillation analysis by two interface conditions in liquid-liquid and liquid atmospheres. From the analytical solution of two interface drop conditions, the normal mode of the core-shell droplet has two separate eigenfrequencies in fundamental oscillation. From the two eigenfrequencies, we can calculate the interfacial tension value 1). Our onboard experiments of observing compound drop oscillation phenomena using ELF have been performed since October 2022. We succeeded in observing the surface oscillation phenomena of compound droplets by molten oxide (SiO_2:CaO:Mn_3O_4:TiO_2:Fe_2O_3= 25:7:20:18:30 mass%) and liquid iron in Ar atmosphere. However, during the observation of oscillation phenomena, an increase in droplet volume was observed (Fig. 1). This may be due to the oxidation of liquid Fe at the interface with the molten oxide. When oxidation occurs, the radius ratio of the shell to the core changes, which affects the interfacial tension measurement. Furthermore, the density and surface tension of the shell molten oxide also change due to oxidation. Therefore, it is necessary to investigate the changes in the density and surface tension of the molten oxide during oxidation. For this reason, we measured the density and surface tension of the molten oxide when liquid iron was dissolved into the molten oxide by oxidation using the aerodynamic levitation method on the ground. From these measurements, we discuss the oxidation of liquid iron at the interface between the molten oxide.

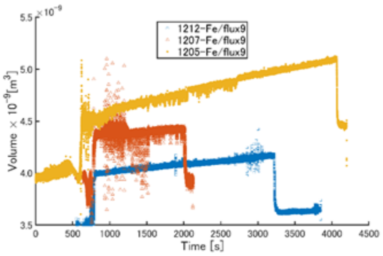

Fig.1 Volume change of levitated core-shell droplet by liquid iron and molten oxide in ELF.

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(P7) Formation of non-metallic inclusions in presence of surface-active elements

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In steel production, the presence of non-metallic inclusions in the melt is an unavoidable occurrence. It is not possible to produce steel completely free of them. However, it is feasible to mitigate their formation, size, and consequently, their impact on steel properties. Non-metallic inclusions enter the melt from surroundings, including slag and refractory linings, or are formed within the molten steel by chemical reactions between dissolved elements. The formation of non-metallic inclusions in the molten steel can be explained by Gibbs free energy. The formation of particles is influenced by the steel melt composition and the system temperature, which affects the solubility of the elements. During production, both the temperature and the composition of the steel melt fluctuate, and exceeding element solubility leads to the formation of non-metallic inclusions.

The formation of non-metallic inclusions in steel is influenced by both the volumetric and interfacial components of Gibbs free energy. The bulk component is directly related to the solubility and activity of the elements in the melt and promotes the nucleation of new phases. Conversely, the interfacial component inhibits the formation of new surfaces and thus the formation of non-metallic particles.

During steel production, aluminium is added to the melt for deoxidation, which leads to the formation of aluminium oxides. These oxides remain solid at the temperatures used in steel production and may accumulate on the walls of submerged entry nozzles during casting, impeding melt flow and potentially causing blockages.

Although sulphur acts as an impurity in molten steel, it can also be beneficial for improving mechanical processing (e.g. milling, turning). Sulphur is a surface-active element, which means that even low concentrations of this element significantly alter the surface tension of the steel melt. In this study, we investigate the effects of sulphur concentration in molten steel on the surface tension of the melt and consequently on the formation of non-metallic aluminium oxide inclusions. As the sulphur concentration in the steel melt increases, the surface tension decreases, which in turn reduces the critical nucleation radius for the formation of non-metallic inclusions.

In this research, we aim to quantify the influence of sulphur on the surface tension of molten steel during casting and gain a deeper understanding of its effects on the nucleation of non-metallic inclusions. These findings may ultimately help us to better control the formation of non-metallic inclusions.

Key words: surface-active elements, non-metallic inclusions, nucleation, sulphur, nozzle clogging

(P8) Thermophysical Properties of Al-Sn system and their effects on solidification related processes

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Metallic Phase Change Materials (PCMs), based on solid-liquid transitions, represents one of the most promising technologies for efficient Thermal Energy Storage (TES), due to their superior thermal conductivity and energy storability per unit volume, but suffer of limited solutions for their handling at the molten state [1)]. As an example, Silicon and Silicon-boron alloys have been recently pointed out as novel ultra-high temperature phase change materials for applications in Latent Heat Thermal Energy Storage (LHTES) and conversion systems. One of the emerging challenges related to the development of such devices is a selection of refractories applicable to build a vessel for storing molten Si-B alloys at high temperatures and under consecutive melting/solidification conditions [2)]. In parallel, some authors propose that the use of eutectic alloys as TES at medium range (≈ 200°C) as feasible materials for industrial processes, as reviewed by [3)]. As suggested by Sugo et al. [4)], an effective solution is the use of Miscibility Gap Alloys (MGAs), as Al-Sn system [5)].

In the present paper, the preliminary results obtained by measuring thermophysical properties (surface tension and density) of liquid Al-Sn system by the pendant drop method, will be presented and discussed in connection with their mixing behavior and developed microstructure [6, 7)].

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(P9) ISRU-approach for designing and fabricating successfully composite materials

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Space: 1999 is a British science-fiction television program running 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. As an example, preliminary results obtained by interactions tests between liquid Al7075/DNA-1 regolith simulant will be shown and discussed.

(P10) Novel Polymer-derived composite materials for latent heat thermal energy storage

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The long-term response to the current situation in Europe must speed up the deployment of renewable energy sources and the development and implementation of energy-efficient solutions. Among them, Thermal Energy Storage (TES) is particularly interesting due to the modularity and the number of different possible applications, such as concentrated solar power systems, electricity storage and co-generation in domestic electrical/heating networks, energy storage in heat recovery systems (energy-intensive high temperature-industrial plants) or sensitive electronics to avoid overheating 1). The temperature at which heat storage occurs represents one of the key performance parameters for material selection and process performance. Metallic Phase Change Materials (PCMs), based on solid-liquid transitions, represents one of the most promising technologies for efficient TES, due to their superior thermal conductivity and energy storability per unit volume 1). As an example, Silicon and Silicon-boron alloys are emerging as novel ultra-high temperature PCMs for applications in Latent Heat Thermal Energy Storage (LHTES) up to 2000°C, and conversion systems. One of the emerging challenges related to the development of such devices is a selection of long-term stable refractories to confine and store molten Si-based alloys at high temperatures and under consecutive melting/solidification conditions 2).

The capability of new UHT-Polymer Derived Ceramics (PDCs) 3), such as SiOC with tailored microstructure as housing/casing system for liquid Si (PCM), has been investigated: wetting experiments performed and 3D-printed SiOC architectures 4) infiltrated. Moreover, their microstructural stability was tested under thermal-cycling conditions. Microstructural characterization of the as obtained Si/SiOC composites was performed by Optical (OM) and SEM/EDS microscope and all the experimental observations, carefully related to the operating conditions, are presented in the paper and discussed.

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(P11) Interfaces between AlN ceramics and metals

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Metalized AlN substrates attract special interest in the field of semiconductors involved in power electronics due to the wide-bandgap (WBG) that is closely related to their high thermal conductivity. Hence, the bonding of AlN ceramics with metals is a key issue for producing reliable AlN-metalized substrates. This work will present two easy and promising methods for producing two different AlN/metal interfaces:

(a) AlN/Al, using CuO interlayer. The method comprises two steps: The surface of AlN is coated with a CuO thick film and sintered at 1100 °C. Then, the Al foil is put on the pretreated AlN surface and bonded to AlN through pre-heating at 400 °C and finally it is heated at 660 °C in N_2-5%H_2 reduction atmosphere. The experimental results, obtained by optical and scanning electron microscope (SEM) observations as well as by X-ray diffraction analysis at the cross-sections of the joints and the fracture surfaces, suggest that the reaction mechanism starts with the reaction of CuO with AlN to form $CuAlO_2$ when the AlN, coated with CuO, is heat-treated at high temperature. A Cu layer is produced by the reduction of CuO and Cu diffuses in the Al foil, forming strong AlN/Al joints. Elongated crystals of Al_2Cu are developed in the reaction zone at the interface between AlN and Al. The peeling-off strength of the Al foil from the surface of the AlN substrate was 15.4 MPa for the AlN/Al couples produced after 30 min of heat treatment at 660 °C. The fracture strength of the produced AlN/Al joints depends on the phases formed at the interface and their microstructure.

(b) AlN/Cu, using porous-copper layer and Ag foil. The method takes place through a film-metallization production process, which involves a porous network of Cu layer and Ag foil. The microstructure and the phases formed at the interface of the AlN/Cu joints produced at various brazing temperatures and times were analyzed by scanning electron microscopy and X-ray diffraction analysis. Strong joints with a shear strength of 48.5 MPa were produced after brazing at 850 °C for 10 min. The typical microstructure at the interfacial reaction zone was Cu / Ag(s)+Cu(s) / $CuAlO_2$+Al_2O_3+Cu(s) / AlN. The experimental results manifest the crucial role of the Ag-Cu eutectic liquid phase, formed by the reaction between the Cu layer and the Ag foil, and of the porous network of the Cu layer deposited on the surface of the AlN ceramic substrate, since both of them effectively favor the reduction of the residual thermal stresses in the joint, resulting in strong ceramic/metal joints.

(P12) Influence of different SiCp additions on the castability of aluminium matrix composites

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The simulation of casting processes is a powerful tool that helps predict the defects in the final component. In practice, material data are often extracted from the literature, and significant deviations can occur between the simulation and real casting. Thermal analysis-driven data are a useful strategy to acquire reliable material data, even more so in the case of aluminium metal matrix composites (MMCs) reinforced with silicon carbide particles (SiCp). High processing costs are a drawback of composites in automotive applications, highlighting the need for simulation techniques based on reliable datasets. The castability and solidification behaviour of aluminium-based composites depend on the reinforcement particle shape, size, and content, among other factors. The calibration of the material dataset for the simulation maximises the defect prediction accuracy and minimises the production costs.

The present study investigates the castability and thermophysical properties of aluminium-based composites reinforced with different SiCp contents ranging from 0 to 30 wt.%. The materials were produced by casting to gather relevant data as input for the material database of the casting simulation of the brake rotor. The simulation model predicted shrinkage defects by dividing the casting into zones with different liquid-phase fractions. The shrinkage porosities were caused by changing the melt and solid phase densities at the temperature change. The laws of heat and mass transfer between the different casting phases and moulds were used to forecast the shrinkage porosity, cold shuts, and hotspots. Material properties, such as thermal diffusivity, thermal expansion, and specific heat capacity, were evaluated as a function of temperature and simulation software with density and thermal conductivity. Computer-aided cooling curves were imported to create a new dataset of aluminium-based composites with different reinforcement additions. A simulation based on the adapted material database was validated in terms of solidification and defect prediction.

(P13) Thermal degradation of Cu/Nb nanomultilayers by formation of nanocomposite structure and hillock growth

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Cu/Nb nanomultilayers (NMLs) have very attractive properties such as high mechanical strength, high conductivity, and radiation-tolerance and show significant potential for applications with elevated operating temperatures. The understanding of microstructure evolution upon annealing is crucial for the use of these materials in such applications.

In this investigation Cu/Nb NMLs with 10 nm single layer thickness were deposited by DC magnetron sputtering on Si(001) substrates with amorphous silicon nitride (90 nm) layer for diffusion protection. The Cu10nm-Nb10nm bi-layer structure was repeated 10 times resulting in a NML total thickness of 200 nm. Subsequently, isothermal annealing of NMLs was carried out at 200 °C, 400 °C, 600 °C, and 800 °C for 100 minutes in a high vacuum (pressure less than 10^{-5} mbar). The microstructural evolution of Cu/Nb NML upon annealing was investigated in detail by Transmission Electron Microscopy (TEM), Scanning Transmission Electron microscopy combined with an Energy Dispersive Spectroscopy (STEM/EDS) and by X-ray diffractometry (XRD).

After annealing of Cu/Nb NML at 600 ºC the growth of a Cu hillock and partial degradation of nanomultilayer structure were observed simultaneously, leading to the formation of Cu-Nb nanocomposite structure. The nanolayer structure was preserved away from the Cu hillock, while the nanocomposite structure was formed directly under the Cu hillock. The subsequent formation of nanocomposite structure occurred with further Cu hillock growth.

This behaviour is significantly different from the widely observed nanomultilayer degradation mechanism by grain coarsening, pinch-off and subsequent spherodization.

(P14) New generation of copper-based coatings with improved antipathogenic efficiency

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Studies were dedicated to the copper-based coatings of enhanced functional properties, which by reducing the number of pathogens infections cases, respond to global trend of illnesses prevention instead of their medical treatment. The innovation objective is the diminishing viability of pathogens on the coated surfaces, obtained in relatively easy to be implemented plating technology with the use of environmentally friendly reagents. The rate of copper's antibacterial activity depends on the percentage of copper in the alloy. Thus, the use of durable and wear-resistant coatings, in which the copper content is as high as possible, is an economical solution. Therefore, a viability of various bacteria was examined on variety of copper and copper-based coatings. Innovative copper-based composite coatings obtained through the addition of TiO_2 nanoparticles during the deposition process, will constitute an even more effective weapon in the fight against the spread of pathogenic organisms. The electroplating and electroless plating deposition technologies were applied for the steel and nickel substrates used as the commonly used surgical tools materials. Next, a comprehensive microstructure characterization of the resulting coatings was performed using scanning and transmission electron microscopy techniques. Biological tests comprising of viability of *Staphylococcus aureus* and *Escherichia coli* and cytotoxic activity of coatings in relation to plating technology parameters, coatings microstructure and chemical composition were also performed.
The adhesion of bacteria to tested materials was analyzed with use of fluorescence microscopy.

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(P15) Metallic honeycombs by additive manufacture

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Nowadays, additive manufacturing is becoming more and more popular, not only in the generation of prototypes, but it has displaced traditional techniques, especially in medicine. Nowadays it is normal to find in our dentist's office a 3D printer where the implants are printed with the perfect colour and shape that is impossible to distinguish them from the original teeth. In surgery it is becoming common to replace a bone with a titanium prosthesis similar to the original bone, which is also porous. However, this type of metallurgical technique does not yet achieve optimal results in terms of microstructure. Its porosity is high and the cost of printing is at least an order of magnitude higher than that of polymers.

In the present work, a study of the additive manufacturing process of three alloys, firstly pure Ti, secondly a tool steel, and finally brass, is carried out. For this purpose, different specimens have been prepared to determine their mechanical, thermomechanical and transport properties. Finally, a series of honeycombs have been prepared with different honeycomb structures with 200 micron walls, which is close to the theoretical limit of this type of printer (selective laser melting). The prototypes show a good finish and the properties are somewhat inferior to those obtained by the traditional methods of casting, forging, etc. However, brass has shown an unusual complexity in the manufacture of parts using this technique, showing in the optimum sample a high porosity and therefore inferior properties to the parts obtained by the traditional method.

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