

EUROMAT 2017

September 17-22, 2017, Thessaloniki, Greece

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Ing. Quim. Uxia Dieguez Salgado¹, Dipl.-Ing. Philipp Dorrer¹, Dipl.-Ing. Dr. mont. Susanne Michelic¹, Ao. Univ.-Prof. Dipl.-Ing. Dr. mont. Christian Bernhard¹

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Mr Gionata Schneider¹, Dr. Ludger Weber¹, Prof. Andreas Mortensen¹

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C5-O-THU-PM2 In-situ apparatus to study gas-metal reactions and wettability at high temperatures

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C5-O-THU-PM2 At the triple line and at the interface of the Ir-Si/C system

Dr Donatella Giuranno¹, Mr Antonio Camarano², Dr Rada Novakovic¹, Dr Enrica Ricci¹, Prof Javier Narciso²
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Phd Candidate Selmar Binder¹, Professor Sophia Haussener¹
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Professor Javier Narciso¹, Doctor Enrica Ricci², Doctor Rada Novakovic², Doctor Donatella Giuranno², Doctor Antonio Camarano¹
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C5-O-FRI-AM2 Wetting and joining of SiC ceramics by Al-Ti alloys

Dr Fabrizio Valenza¹, Dr Valentina Casalegno², M.Sc Sofia Gambaro¹, M.Sc Maria Luigia Muolo¹, Dr Alberto Passerone¹, Prof Milena Salvo²
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PhD Dariusz Jarzabek¹, Msc Cezary Dziekoński¹, PhD Marcin Chmielewski²
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C5-I/K-FRI-AM2 Reactive infiltration: identifying the role of chemical reactions, capillarity, viscosity and gravity

Phd Enrique Louis¹, PhD Juan Antonio Miralles¹, PhD José Miguel Molina¹
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C5-O-FRI-PM1 Strain sensing ability and stress transfer profiles of hierarchical carbon fibers produced via different methods

Material Science Engineer Kyriaki Tsirka¹, Material Science Engineer Giorgos Karalis¹, Professor Alkiviadis Paipetis¹

¹University Of Ioannina, Ioannina, Greece

C5-O-FRI-AM2 Interfacial reactivity in the Al₃Ti-SiC system

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Dr Vladimir Traskine¹, Professor Zoya Skvortsova¹, Dilara Farkhutdinova¹

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Professor George Kaptay¹

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C5-I/K-THU-PM2 Factors affecting wetting and reactivity in Si/ceramic systems at ultrahigh temperatures
T>1450°C

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C5-O-FRI-AM2

Effect of solubility and capillarity driven kinetics on sintered microstructure of WC-Co alloys

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Liquid phase sintering involves a variety of physical processes, including capillarity, neck formation and coalescence, grain growth and coarsening, evaporation-condensation, as well as melting-solidification. The rate at which these processes proceed strongly depends on parameters such as temperature and pressure, grain size and on transport mechanisms such as surface, grain boundary and bulk diffusion, viscous flow and rigid body motion of individual grains. In the present work, we focus on the interplay between dissolution of hard metal component in the liquid phase and the compaction kinetics. A finite solubility is beneficial for rapid compaction. It enhances the grain rearrangement dynamics under the action of capillary forces by dissolving the bonds which form in the early solid sintering stage, prior to the liquid donor melting.

It is shown how the microstructure is influenced by the competition between dissolution kinetics on the one hand and the dynamics of grain rearrangements and compaction on the other hand. Moreover, the effect of the cooling rate and composition on these competing processes is also investigated.

C5-O-FRI-PM1

Experimental investigation of the system non-metallic inclusion-molten steel-refractory at high temperatures

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Clogging in continuous casting is the buildup of particles in the flow control system. Besides other reasons, pre-existing non-metallic inclusions may build up on the refractory wall, interrupting or disturbing the fluid flow conditions. The interfacial properties of the system non-metallic inclusion-molten steel-refractory are believed to play a key role for the clogging tendency of selected steel grades in contact with ceramics.

The combination of a High Temperature Laser Scanning Confocal Microscope (HT-LSCM), for in situ observation of inclusions' behaviour in the liquid steel or at the steel/refractory interface, and a High Temperature Drop Shape Analyser (HT-DSA), for contact angle measurements, enable the investigation of reactions and interactions in the described system at experimental temperatures of about 1600°C.

A relatively new experimental set up is tested in the HT-LSCM: Non-metallic inclusions are observed at a molten steel-ceramic interface and attraction forces are measured. The HT-DSA provides contact angle data between molten steel and different oxides for implementation into a theoretical adhesion model. Finally, observed and calculated forces can be compared and evaluated.

C5-O-FRI-PM1

Some characteristic features of Ni-Re-P coatings electroless deposited on copper

Prof. Joanna Wojewoda-Budka¹, Dr. Anna Wierzbicka-Miernik¹, Dr. Honorata Kazimierczak¹, Izabella Kwiecien¹, Lidia Litynska-Dobrzynska¹, Maciej Szczerba, Pawel Czaja¹, Jerzy Morgiel¹, Fabrizio Valenza²

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Electroless plated nickel layers with a thin layer of gold on the top are widely applied in the electronics industry and known as Electroless Nickel Immersion Gold (ENIG) coatings. The quality of ENIG/solder interconnections is directly related to the nickel solution bath, and the resultant chemical composition (phosphorus content) of the coating. Depending on the amount of phosphorus within the coating, the layer can be either fully amorphous, or with nano Ni crystallites or crystalline. This translates into an increase of the temperature of its thermal stability, which in turn has a direct impact on the processes occurring at elevated temperatures during the soldering process or later during use of the final product (heating/cooling conditions). The subjects of the study were nickel coatings with rhenium addition (Ni-P-Re) deposited on copper in electroless plating process. The main goals for the undertaken work were: to optimize the process of deposition of Ni-P-Re (especially to show the influence of solution pH on the chemical composition of the deposited coating), to verify the real impact of rhenium addition into Ni-P plating on its thermal stability, to expose the relation between the chemical composition of the coating and the phase crystallization and other phenomena occurring during exposure to the higher temperatures. Finally, the wettability of these coatings by liquid Sn, the main constituent of most lead-free solders, was studied in order to investigate the interfacial phenomena occurring when these materials are assembled by soldering.

Acknowledgement

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C5-O-FRI-AM2

Capillarity in pressure infiltration: influence of percolation and wetting angle at high temperature.

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Composite materials are often produced by pressure-infiltration of a liquid matrix into a preform of packed solid fibers or particles. We quantify capillary forces that intervene in this process by measuring saturation curves, which are plots of the fraction pore space filled with the matrix versus the pressure difference between the matrix and the atmosphere initially present in the porous preform to be infiltrated. We do so using an apparatus capable of measuring saturation curves at high temperature ($\approx 1300^\circ\text{C}$) and high pressures (≈ 150 atmospheres), which is suitable for the study of metal matrix composite pressure infiltration. In the work to be presented here we will focus on elevated temperature infiltration, exploring regimes at which flow of the metal under fixed applied pressure does not cease completely, but rather tends towards a low and relatively constant velocity, which is at given temperature a dual function of pressure and saturation. Drawing from an analogy with creep and relaxation experiments that are classically used to characterize elevated temperature plasticity, we explore this dependence to infer by what mechanisms such gradual, fixed-velocity infiltration, might be caused.

C5-I/K-FRI-PM1

Grain boundary wetting by a second solid phase

Prof Boris Straumal^{1,2,3,4}, Dr. Alena Gornakova¹, Dr. Andrey Mazilkin^{2,3}

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The phase transitions can proceed not only in volume but also in grain boundaries (GBs). The GB wetting phase transition can occur in the two-phase area of the bulk phase diagram where the liquid (L) and solid (S) phases are in equilibrium. Above the temperature of the GB wetting phase transition a GB cannot exist in equilibrium contact with the liquid phase. In case if two solid phases are in equilibrium, the GB “wetting” by a second solid phase can occur. In this case the layer of the solid phase 2 has to substitute GBs in solid phase 1. Such GB phase transition occurs if the energy of two interphase boundaries is lower than the GB energy in the phase 1. The experimental data on GB “wetting” by a second solid phase” in systems Zn–Al, Cu–In, Cu–Co, Zr–Nb, Ti–Fe, Ti–Co, Fe–C, W–Ni etc. are analyzed. The GB wetting tie-line can continue in the one-phase area of the bulk phase diagram as a GB solvus (solidus) line. The GB properties change drastically when GB solvus (solidus) line is crossed by a change in the temperature or concentration. Between GB and bulk solidus the few nanometers thin layer of GB phase appear, Such GB phase is unstable in the bulk and stable in the boundary. The experimental data on GB segregation, energy, mobility and diffusivity were obtained in various systems. Between complete and incomplete GB wetting the so-called pseudoincomplete GB wetting can take place.

C5-O-THU-PM2

In-situ apparatus to study gas-metal reactions and wettability at high temperatures

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The understanding of gas-metal reactions and related surface wettability at high temperatures is often limited due to the lack of in-situ surface characterization.

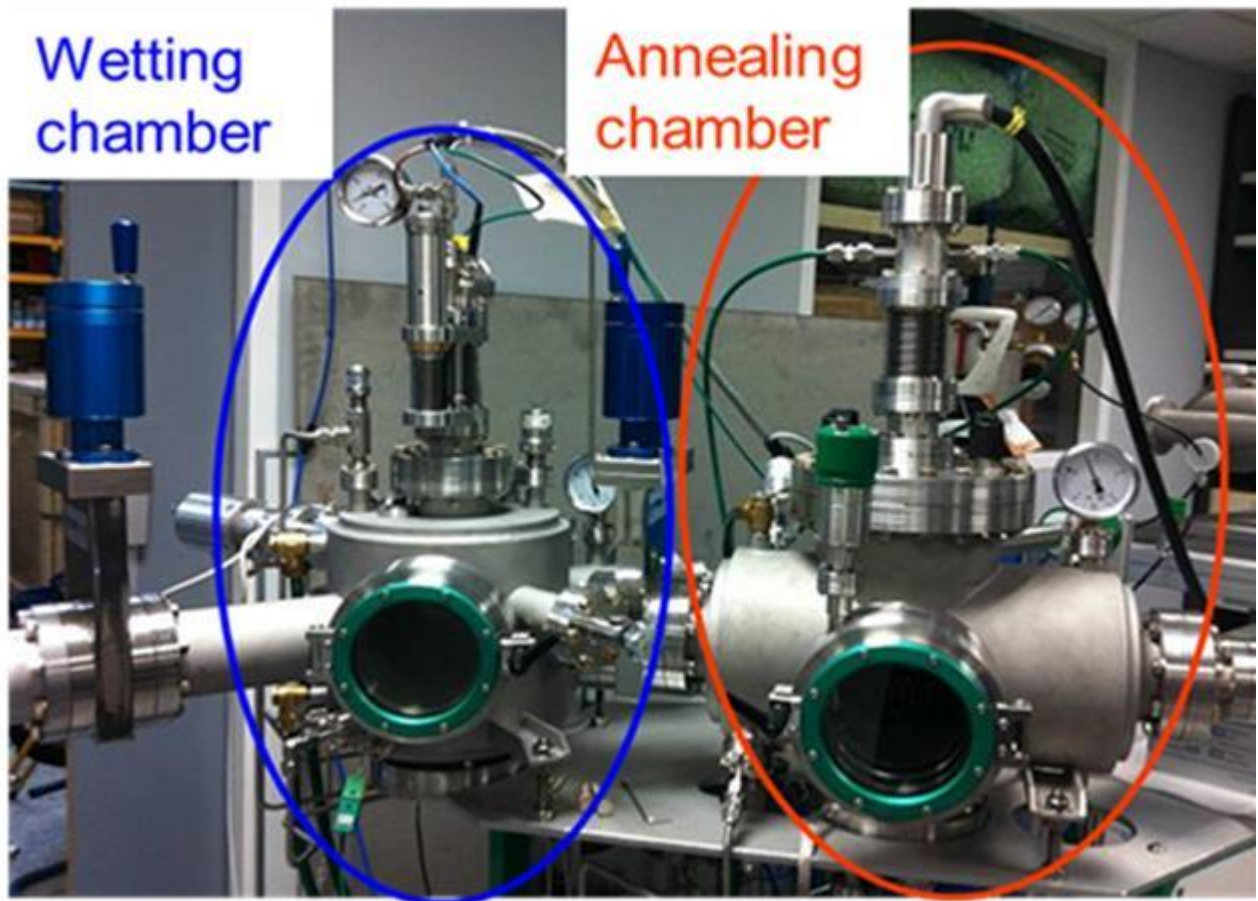
A High Temperature Wettability Device (HTWD) was designed in order to allow the in-situ sample surface characterization by X-rays Photoelectron Spectroscopy (XPS) after gas/metal and liquid metal/solid metal surface reactions. Such characterization gives an access to the real composition of surfaces after heat treatment and chemical reaction without air contact avoiding any contamination and oxidation of surfaces. The device consists of two connected reactors respectively dedicated to the annealing treatments and wettability measurements. The heat treatments are performed in the infra-red lamp furnace in a well-controlled atmosphere conditions designed to reproduce gas-metal reactions occurring during the recrystallization annealing of steels. The wetting experiments are carried out in dispensed drop configuration with the precise control of the deposited droplets kinetic energies. The spreading of drops is followed by high speed CCD video-camera at 500-1000 frames/s in order to reach information at very low contact time.

First trials have started to simulate phenomena occurring during the recrystallization annealing and hot dip galvanizing on polished pure Fe sample. During annealing treatment, substrate was heated to 800°C and hold at this temperature during 2 min. Then during the natural cooling, the sample is rapidly moved to the wetting chamber where the substrate temperature is decreased to about 420-460°C and maintained at the suitable value. The zinc alloy is melted in the crucible above the Fe substrate and the droplet is released at the suitable temperature in the range 420-500°C. The drop spreading kinetics depending steel substrate temperature are discussed. The wetting results are coupled to the characterization of the interfacial layer formed after reaction. The results show the real interest of such in-situ experimental set-up for interfacial chemistry studies.

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C5-O-THU-PM2

At the triple line and at the interface of the Ir-Si/C system

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For industrial, military and space applications, there is an increasing need for structural materials that can withstand oxidizing environments at elevated temperatures: advanced metal-matrix composites, based on refractory silicides compounds could be possible candidates, i.e. Ir-silicides/SiC composites, at least for highly demanding applications.

Wetting characteristics and reactivity are key requirements for manufacturing of composite materials by reactive infiltration method.

Wetting behavior, spreading kinetics and interfacial phenomena of three Si-rich Ir-Si alloys on carbon materials (Glassy Carbon and CVD-Graphite) have been studied using the classical sessile drop technique under a static Ar atmosphere, with a special emphasis on the interface formation and triple line evolution. Wetting behavior and spreading kinetics have been analyzed and governing mechanisms have been discussed. The influence of time, temperature, Si-content, carbon crystallinity have been evaluated.

In order to better understand the interfacial phenomena experimentally observed, the Ir-Si/C system has been analyzed in detail taking into account the thermodynamic and surface properties (surface tension and surface segregation) of the liquid phase as well as the ternary Ir-Si-C phase diagram.

As expected, for a reaction controlled wetting, a strong temperature and time dependence of spreading was observed. In particular, the spreading kinetics and final contact angle values seemed to be affected by the local Si-evaporation/condensation phenomena occurred at the triple line. In addition, carbon crystallinity played the major role in interface formation, where a higher crystallinity yields a thicker and compact SiC-reaction layer.

C5-O-FRI-PM1

Encapsulation strategy for stable metal phase change materials used for high-temperature heat storage applications

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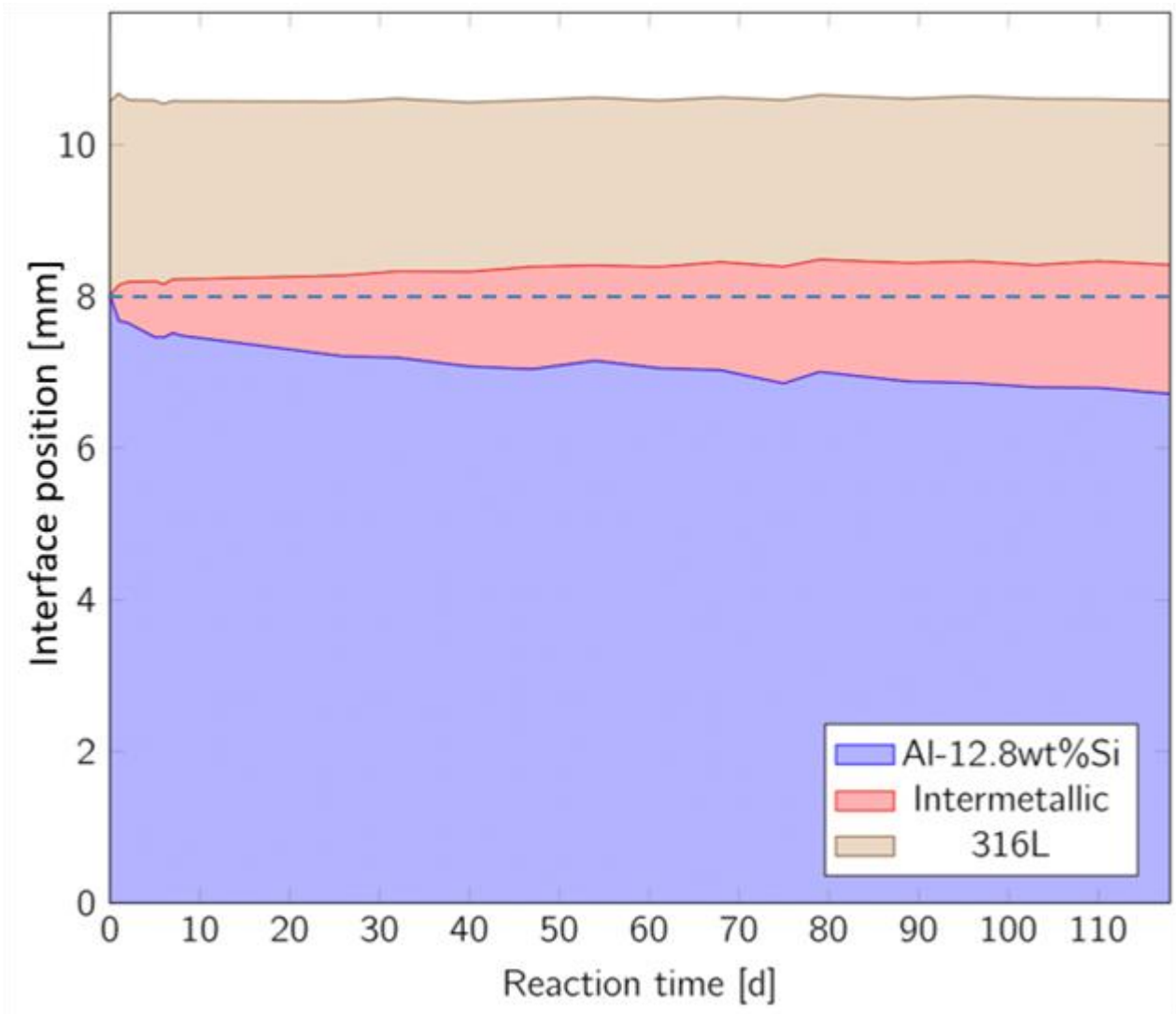
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Metal alloys as phase change materials (PCM) are promising candidates for high-temperature thermal energy storage (TES) applications, but liquid metal corrosion and inter-diffusion poses a challenge to traditional encapsulation materials. We aim at avoiding degradation of storage capacity, heat discharging rate, and mechanical stability by developing and testing a novel encapsulation strategy. The model system is an Al-0.13wtSi as PCM encapsulated in 316L stainless steel cylinders. A diffusion barrier made of hexagonal boron nitride is applied in order to reduce the degradation. Protected and unprotected control cylinders were tested for chemical stability by exposure to 700 °C for up to 100 days in a Nabertherm furnace with constant temperature control. The cross-sectioned samples were inspected by optical microscopy, scanning electron microscopy energy dispersive x-ray analysis (SEM-EDX), differential scanning calorimetry (DSC) and micro indentation (Vickers hardness, HV) in order to quantify corrosion and loss in active PCM material. Unprotected steel units showed up to 25 % loss in PCM volume after 100 days, due to the formation of a high melting intermetallic phase at the interface. DSC studies suggest a lowering of the specific heat of fusion by 15 %. Micro indentation (HV0.3) tests confirm the presence of two distinct, brittle (600/800 HV) intermetallic phases with possibly detrimental impact on mechanical stability and heat transfer. SEM-EDX reveals dissolution of encapsulation material in the PCM. On the other hand, ceramics coated and all-ceramic units are not significantly affected by intermetallic growth and degradation. The provided strategy effectively suppresses degradation and corrosion in the Al-0.13wtSi/steel system. The presented approach may be widely applicable for stable PCM units in high temperature applications since aluminum is the base metal for many high latent heat alloys as well as one of the most corrosive liquid metals.

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C5-O-THU-PM2

Reactivity between SiC single crystals and Ir at high temperature

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Typically, catalytic devices consist of an active phase dispersed in a ceramic matrix. With the aim to increase the efficiency of the process, the increase of combustion temperature is the key requirement, making the design of catalyst properly working at $T > 1000^\circ\text{C}$ as a key issue. One of the candidates is the Ir-SiC system. As a result, understanding the chemical reactions at Ir/SiC interface is crucial: a strong metallurgical bond through solid state reaction is essential in order to manufacture stable Ir/SiC interfaces. In principle, the identity of the reaction products could be predicted from the thermodynamic quantities. However, such a prediction generally fails due to the influence of kinetic and structural effects. For these reasons, a theoretical-experimental method has been applied to investigate the evolution of Ir/SiC interface. Solid state reactions between Ir/SiC have been studied in the temperature range $1200\div 1450^\circ\text{C}$ for a time of 5h under a vacuum. In parallel, with the aim to predict reaction products the thermodynamics of the ternary Ir-Si-C system has been analyzed. Morphology and microstructure of the samples were evaluated by SEM. For the evaluation of the reaction products and their kinetics micro-DRX has been used. As a function of temperature, the Ir/SiC interfaces exhibited first evidences of reaction at $T = 1375^\circ\text{C}$ and the first liquid phase was observed at $T = 1400^\circ\text{C}$ due to the formation of Ir-silicides. Various Ir-silicides and C-precipitates were formed in layered reaction zones: planar layers in the absence of interfacial melting ($T < 1400^\circ\text{C}$), concave layers with interfacial melting occurred ($T \geq 1400^\circ\text{C}$). Free unreacted C was detected in all the reaction zones and its precipitation behaviour was dependent on the metal phase as well as the location with respect to the SiC reaction interface.

C5-O-FRI-AM2

Wetting and joining of SiC ceramics by Al-Ti alloys

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The full exploitation by the aerospace industry of the attractive and unique properties of SiC based composites calls for effective joining technologies that may help to assemble them in complex shapes or to integrate them into existing metallic parts forming hybrid structures.

In view of using liquid-assisted joining processes, such as brazing or transient liquid phase bonding, the wettability of SiC by Al-Ti alloys to be used as brazing material is presented here. These studies show that liquid Al-Ti alloys have a very good wetting behaviour when in contact with SiC. In particular, the Al₃Ti alloy has the most promising interfacial behavior in terms of wetting, adhesion and resulting phases.

Following this line, results are also presented on joining SiC to itself using, in particular, the Al₃Ti alloy as brazing medium. Different brazing routes were pursued, such as capillary infiltration of the liquid alloy between the seam or assembling the joints by sandwiching a paste (Al₃Ti powder + organic binder) and Ti layers between the adjoining materials.

The microstructures and their chemical composition were characterized on cross-sectioned samples by SEM/EDS, while the overall strength of the joints was determined by shear tests at room temperature; the mechanical response coupled with fracture path detected by post mortem analysis was related to the process parameters (e.g. temperature, applied pressure, cooling rates).

C5-O-FRI-PM1

Effect of metal coatings on the interfacial bonding strength of ceramics to copper in sintered Cu-SiC composites.

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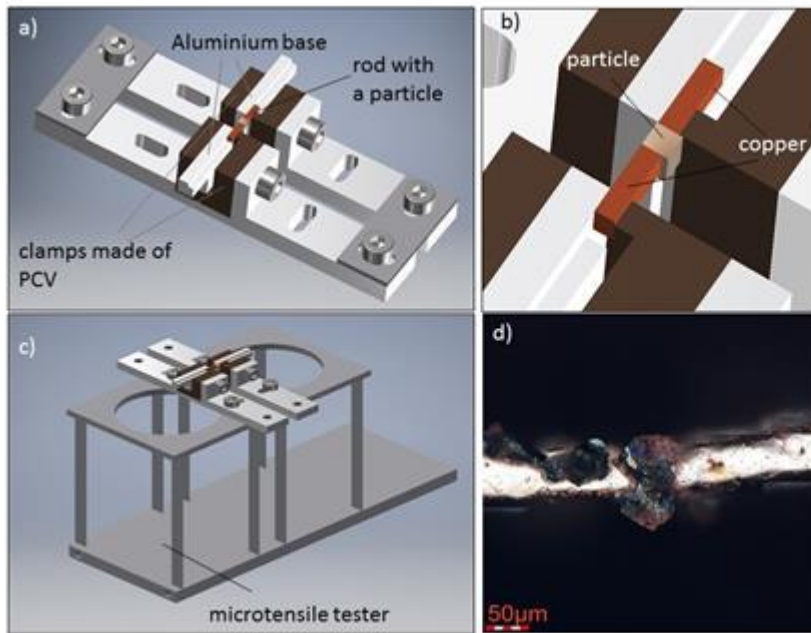
Cu-SiC composites are very promising materials which have high thermal and electrical conductivity and may find many applications. Unfortunately, the main disadvantage of these materials is the dissolution of silicon in copper at elevated temperature, which significantly reduces their properties. In order to overcome this problem particles can be coated with a protective material before sintering. In this paper the influence of three different metallic coatings on bonding strength were investigated. SiC particles were coated with tungsten, chromium or titanium. As reference a material with uncoated particles was prepared. The experiments were carried out with the use of microtensile tester (Fig. 1). The results show that the strongest interface can be produced when chromium coating is applied. In this case the bonding strength is approximately 50% stronger than in the case of uncoated particles. Unfortunately, the porosity of copper which was observed in the vicinity of chromium coating may significantly reduce other parameters such as thermal conductivity of the composite. On the other hand, tungsten coating increases the bonding strength about 30% and it is smooth and regular. This coating seems to be the best choice for applications in which not only mechanical properties of the composites are important. Furthermore, scanning electron and optical microscopes were used to determine the mechanism of debonding.

Figure 1. Microtensile tester scheme.

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C5-I/K-FRI-AM2

Reactive infiltration: identifying the role of chemical reactions, capillarity, viscosity and gravity

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A wealth of experimental data indicate that while capillarity controlled infiltration gives an infiltration length that varies with the square root of time, reactive infiltration is characterised by a linear relationship between the two magnitudes. In addition the infiltration rate in the latter is at least two orders of magnitude greater than in the former. This work is addressed to investigate imbibition of a non-wetting, albeit reactive, liquid into a capillary, within the framework of a simple model that includes capillarity effects, viscosity and gravity. The capillary radius is allowed to vary, due to reaction, with both position and time, according to either an interface or a diffusion law. The model allows for capillary closure when reaction kinetics dominates imbibition. At short times, and depending on whether infiltration is capillarity or gravity controlled, the infiltrated length varies either as the square root or linearly with time. This suggests the following track for reactive infiltration: i) In most cases, the contact angle is initially larger than 90° , ii) after some time, reaction gradually replaces the interface liquid/preform by the liquid/reaction product interface and, concomitantly, the contact angle gets closer to 90° , iii) beyond that time, gravity triggers infiltration (actually the contact angle does not need to be smaller than 90° for the initiation of infiltration due to the metallostatic pressure exerted by the liquid metal on top of the porous preform), iv) thereafter infiltration is controlled by viscosity and gravity, provided that, due to reaction, the contact angle remains close to that at which infiltration was initiated.

C5-O-FRI-PM1

Strain sensing ability and stress transfer profiles of hierarchical carbon fibers produced via different methods

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Strain sensing ability and stress transfer profiles of hierarchical carbon fibers produced via different methods
Tsirka K., Karalis G., Paipetis A. S.

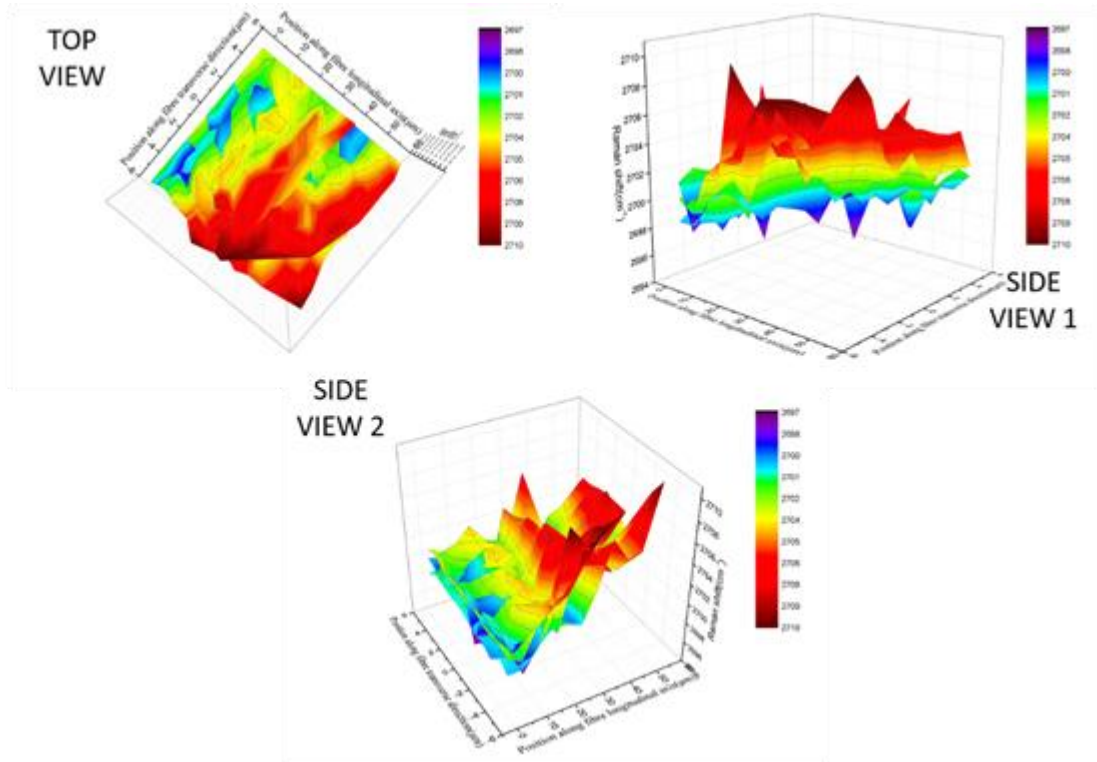
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Hierarchical reinforcements of carbon fibers coated with carbon nanotubes have been produced via two different methodologies, namely chemical vapor deposition and wet chemical treatment. These advanced smart materials are possible candidates for inherent strain sensing when incorporated in the volume of structural fiber reinforced polymer composites. The strain sensing ability of the produced structures was evaluated via Raman spectroscopy at the single fiber level. A comparison of the strain sensitivity of carbon nanotube coated and plain uncoated carbon fibers indicated enhanced values for the coated systems. Then, the stress transfer profiles of the hierarchical fibers as well as the uncoated carbon fibers were produced using Raman spectroscopy. These profiles facilitated the quantification of the amount of stresses acting on the composite interfaces. The random arrangement of carbon nanotubes onto the surface of the carbon fibers led to uneven stress distributions with extended scattering in the stress values accumulated by the nanophase. For a more detailed evaluation of these stresses, maps of the stress distribution along the fibers volume and along the carbon nanotube coatings were acquired which clearly depicted the amounts of stresses undertaken by each structure. The effects of the preparation procedure and the structure and morphology of the carbon nanotube coatings on the stress transfer efficiency were also evaluated. Indicative Raman maps along the surface of carbon nanotube coated carbon fibers are presented in Figure 1. The Raman shift values presented in the maps are subsequently converted to stress values.

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C5-O-FRI-AM2

Interfacial reactivity in the Al₃Ti-SiC system

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SiC based Ceramic Matrix Composites (CMCs) are considered as promising materials for aerospace applications because of their combination of properties such as high thermal, mechanical and chemical stability. In this work, the Al₃Ti alloy is chosen as filler material to obtain convenient metal-ceramic phases such as Ti₃(Al,Si)C₂. In order to define the process parameters for the SiC-SiC joining process, a thermodynamic study of the Al-C-Si-Ti system at high temperature is conducted using an home-made database built by combining and adapting interaction parameters assessed in literature for the binary and ternary subsystems [1-4]. Joining tests at different temperatures (1300,1500°C) have been conducted and the sample sections characterized to determine the metal-ceramic reactivity as well as the sequence of the different layers at the metal-ceramic interface. The reactivity of this metal-ceramic system could be approximately predicted using the CALPHAD approach to analyse the competition between different phenomena inducing an evolution in the liquid composition. The calculated evolution is compared to the experimental results obtained and different pathways are hypothesized and discussed for the system under investigation.

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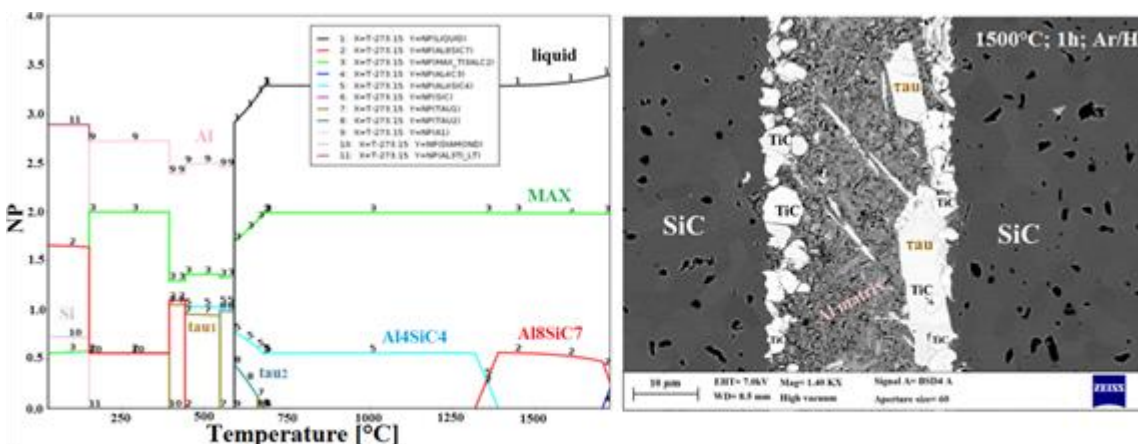


Fig.1- Evolution of the phases in function of the temperature and section image of the SiC-Al₃Ti-SiC joint.

C5-O-FRI-PM1

Rare-earth doping and crystallization behaviour of Bi₄Si₃O₁₂ scintillator

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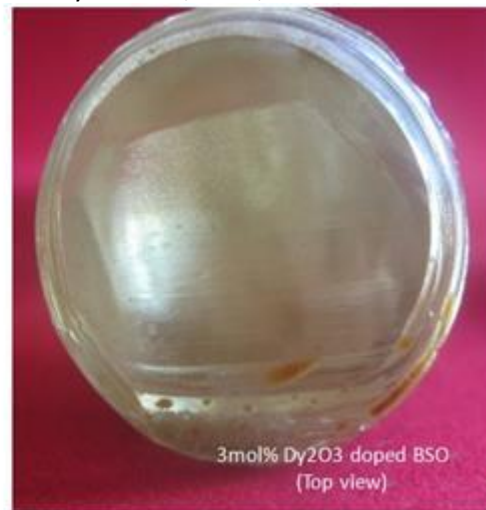
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Scintillation crystals are the excellent materials for conversion of high-energy particles (α , β , γ , X-ray ...) into visible light and have been widely used in high energy physics, nuclear physics, computed tomography and dosimetry. Compared with traditional oxide scintillators, such as Bi₄Ge₃O₁₂ and PbWO₄, bismuth silicate (Bi₄Si₃O₁₂) crystal has some better scintillation properties such as a faster decay time, a greater radiation hardness and lower raw material cost, so it is considered to be the substitute for an alternative to BGO crystals [1-3]. Recently, BSO crystals are regarded as the most attractive candidate for the large experimental programs of the dual-readout method (DREAM) projects in USA and Europe [4]. However, it is difficult to grow large-size and high-quality BSO crystal due to its special crystallization behaviour, including strong growth facet and compositional segregation. The compositional segregation may result in deterioration of crystal quality. In the present work, we reported the growth results of rare-earth doped Bi₄Si₃O₁₂ crystals by the vertical Bridgman method. The segregation was restrained when doping concentration increased up to about 3mol%. The possible mechanism was discussed also.

Keywords: BSO, crystal growth, rare-earth doping, phase segregation

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C5-O-FRI-PM1

Grain boundary wetting in NaCl by molten sodium

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Alkali halides (MeHal) are widely applied in laboratory studies because of their dual importance - both technological (radioactive waste storage) and purely scientific, as solids modeling other materials. In particular, grain boundary (GB) wetting in NaCl has been used to find out a number of general features characterizing this important phenomenon [1].

Adding an excessive amount of alkali metals to their salts is well known to result in formation of coloured units ranging from point defects (F-centers etc) to metal nanocrystalline particles localized along dislocation lines. GB enrichment with metal particles or films has been, to our knowledge, practically not documented. The goal of our work has been to work out an experimental method for introducing an alkali metal into its polycrystalline salts preferentially along GB planes. We have modified for this purpose the technique proposed earlier to decorate dislocations.

Coarse grained polycrystals of NaCl, KCl and KBr were obtained by hot flattening of single crystals and subsequent recrystallization annealing. Polycrystalline samples were heated in sodium vapours. For each salt, a combination of temperature, heating duration, quenching and annealing regimes was used in order to maximize the colour contrast between grains and GBs.

Absorption spectra showed that the diameter range of Na and K inclusions was of 30 to 80 nm. The average alkali metal content in the salts was of 10⁻² to 10⁻¹ %.

The results may be summarized as follows: 1) Only certain GBs are colour-bearing. 2) The colourless GBs are the low energy ones. 3) The average percentage of coloured GBs is in a rough agreement with the known values for the average free energy of MeHal GBs, Na-NaCl interfacial energy [2] and GB energy distribution width.

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C5-I/K-FRI-PM1

Modelling phase equilibria in nano-systems

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The key to model phase equilibria in nano-systems is to model the chemical potentials of components in nano-phases, including the effect of interfaces. Historically the first attempt to describe equilibrium of nano-materials was due to Kelvin (1871), claiming that the nano-effect is due to the increased curvature of the nano-phase. This Kelvin paradigm is the basis of the Kelvin equation, the Gibbs-Thomson equation and the Ostwald-Freundlich equation for the vapor pressure, melting point and solubility of spherical (or quasi-spherical) nano-phases, respectively. The Kelvin paradigm is also successful in the interpretation of more complex phenomena, such as capillary condensation. However, the Kelvin paradigm predicts no nano-effect for not curved nano-phases, such as crystals and thin films, contradicting experimental facts. Moreover, it wrongly predicts that a cubic nano-droplet is more stable than a spherical nano-droplet of the same volume. These and other shortcomings of the Kelvin paradigm are discussed. A new paradigm is worked out in this paper, claiming that the nano-effect is due to the increased specific surface area of the nano-phase. Chemical potentials of components in multi-component free standing nano-phases are derived within this new paradigm. These equations are extended for multi-component and multi-phase nano-systems for the first time. The new paradigm leads to similar results compared to the Kelvin paradigm in all situations when the Kelvin paradigm was successful (such as capillary condensation). Moreover, the new paradigm is able to provide meaningful solutions also for problems, not tractable by the Kelvin equation, such as the case of crystals and thin films having no curvature.

Previous papers on the subject:

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C5-O-THU-PM2

Wetting dynamics of liquid lead and zinc on silica-patterned iron

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Wetting experiments with liquid Pb and Zn-Al (0.2 wt.%) on heterogeneous surfaces were performed at 450°C using the dispensed drop technique. The surfaces were composed of high-purity iron regularly patterned with square silica pillars of different size and spacing (both from 5 to 500 µm). Iron is wetted by liquid Pb and Zn-Al whereas silica is not. The wetting is non-reactive (resp. reactive) between iron and Pb (resp. iron and Zn-Al).

The dynamic wetting was filmed at very short contact time (less than 1 s), in order to measure the evolution of the spreading diameter and the contact angle as a function of time. The contact angle hysteresis was deduced from these measurements. At the end of the spreading, the triple line of the solidified droplet was observed to be pinned on iron and to avoid the silica pillars. In our experimental conditions (droplet impact velocity of 0.6 - 0.7 m.s⁻¹), the dynamic contact angle obtained does not depend on the spreading direction. At constant Cassie fraction (i.e., surface area fraction covered by silica), the dynamic contact angle does not depend on either the size of the pillars or their spacing. At last, the final contact angle increases significantly when the Cassie fraction is increased.

The experimental results obtained, namely the final contact angle, the contact angle hysteresis, the maximal spreading diameter and the dynamic contact angle, will be compared to theoretical estimations from models proposed in the literature.

C5-I/K-THU-PM2

Factors affecting wetting and reactivity in Si/ceramic systems at ultrahigh temperatures $T > 1450^{\circ}\text{C}$

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The work summarizes the research done on real-time observations of high temperature behavior of pure liquid Si and Si-based alloys placed on selected refractory materials during their heating and cooling. The sessile drop method was applied for experimental evaluation of wettability kinetics and reactivity of Si/refractory couples at ultrahigh temperature range of 1450-1800°C.

The research intends to the appropriate choice of proper refractories needed for measurements of thermophysical properties of Si-based melts by container-assisted techniques, as well as to the proper material design of Si-based ultrahigh temperature latent heat thermal energy storage (LHTES) devices. The effects of temperature, type and chemistry of selected refractories, alloying additions, and testing procedures have been examined.

The following phenomena accompanying high temperature interaction in Si/refractory systems will be analyzed: non-wetting-to-wetting transition during heating, wetting-to-non-wetting transition during cooling, substrate dissolution in a melt, liquid metal penetration into a substrate, the formation of solid and gaseous reaction products, liquid metal expansion during solidification.

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