## **KMM-VIN Newsletter**

## Issue 19, December 2018



### **EDITORIAL**

This is the 19<sup>th</sup> issue of Newsletter of the European Virtual Institute on Knowledge-based Multifunctional Materials (KMM-VIN). The Newsletter is published twice a year: in July and December.

KMM-VIN was established in 2007 in Brussels as the key deliverable of the Network of Excellence KMM-NoE project of the EU 6<sup>th</sup> Framework Programme. It is an international non-profit association (AISBL) based on Belgian law. It has two offices: the main one in Brussels and another one at the KMM-VIN Branch in Warsaw, Poland.

KMM-VIN is a self-sustainable European network of universities, research institutes and industrial companies, created to promote and enable cooperative research of its members in the broad area of advanced structural and multifunctional materials. The KMM-VIN R&D activities conducted in Working Groups (WGs) comprise materials development and processing, characterisation of microstructure and properties, modelling and simulation.

The main industries targeted by the KMM-VIN research are Transport, Energy and Healthcare sectors. In addition to R&D activites the KMM-VIN organizes Industrial Workshops and Specialised Courses. Also, it offers research fellowships for young researchers from within the KMM-VIN network.

The KMM-VIN research is focused on:

- metals and alloys (high temperature steels)
- advanced ceramics
- intermetallics and shape memory alloys
- metal-ceramic composites and FGMs
- coatings, layered materials, surface modification
- biomaterials and bioinspired materials
- graphene and 2D materials
- joining of advanced materials
- modelling and simulation of materials

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Besides networking activities for its members KMM-VIN offers services for external customers including: integrated R&D solutions, access to specialised laboratory equipment, database of KMM-VIN materials and members' expertise, customised courses and training opportunities.

This Newsletter presents the main activities of the KMM-VIN Partnership during the past six months as well as actions and events planned for the next future. The December Issue of the 2018 Newsletter commences with the "Latest News" containing up-to-date information on the Partnership, its recent and forthcoming events, such as KMM-VIN Industrial Workshops and alike.

The "News from the Working Groups" reflect the ongoing collaborative research carried out within the Working Groups. At present KMM-VIN consists of five WGs of different sizes and cooperation models: WG1. Materials for Transport, WG2. Materials for Energy, WG3. Biomaterials, WG4. Materials Modelling and Simulation, WG5. Graphene/2D Materials.

In the column "KMM Projects" the Readers can find concise information about selected collaborative projects in which KMM-VIN members are involved.

The column "Cooperation" presents KMM-VIN cooperative links with European organisations and initiatives on materials.

Within the "Research Fellowships, Courses and Trainings" column a pilot information on the next call for KMM-VIN Research Fellowships is provided. The current list of Specialised Courses offered by KMM-VIN Members is given.

The current register of KMM-VIN members and the contact details of the KMM-VIN Office are given at the end of the Newsletter. For more information about the KMM-VIN members, current initiatives and events, please visit the KMM-VIN website <u>http://www.kmm-vin.eu/</u>.

Marek Janas, Editor

## LATEST NEWS

#### PARTNERSHIP

KMM-VIN association is currently composed of 61 core and associate members from 13 European States of whom 56 are institutions (research centres, universities, large companies and SMEs) and 5 are individual members.

The partnership welcomes new members from academia, industry and SMEs interested in collaborative research within the technical scope of five Working Groups: WG1. Materials for Transport, WG2. Materials for Energy, WG3. Biomaterials, WG4. Materials Modelling and Simulation and WG5.Graphene/2D Materials.

New working groups can be formed bottom-up on topics that are not covered by the existing WGs, if requested by a group of minimum 7 members.

Applications to join KMM-VIN are received on a continuous basis; for accession procedure please see: <u>http://kmmvin.eu/network/become\_a\_member/</u>. More information about KMM-VIN can be obtained from <u>Michal.Basista@kmm-vin.eu</u>. The final decision on acceptance of new members is taken by the General Assembly at its annual meeting.

#### **RECENT KMM EVENTS**

**"EUROPE IN MOTION – EuMaT Joint Session**" was held on Sep. 19, 2018 during the EMRS Fall Meeting in Warsaw. A talk on KMM-VIN collaborative research and education activities as an example of effective European networking in the field of advanced materials was given by the KMM-VIN CEO.

8<sup>th</sup> KMM-VIN Industrial Workshop on "Modelling of composite materials and composite coatings" (IW8) was held on 9-10<sup>th</sup> October 2018 at Fraunhofer-IWM in Freiburg, Germany (see "News from WG4").



Participants of KMM-VIN 8<sup>th</sup> Industrial Workshop at Fraunhofer-IWM in Freiburg, Germany.

#### FORTHCOMING EVENTS

The next KMM-VIN General Assembly (GA) will be held on Feb. 26, 2019 at the KMM-VIN registered seat in Brussels.

The GA meeting will be accompanied by the annual meetings of the Working Groups according to a general scheme as follows:

**WG1**. Materials for Transport, Feb. 27, 2019 (Wednesday morning; big room)

**WG2**. Materials for Energy (EMEP), Feb. 25, 2019 (Monday afternoon; big room)

**WG2**. Materials for Energy (non-EMEP), Feb. 26, 2019 (Tuesday morning; big room)

**WG3**. Biomaterials, Feb. 27, 2019 (Wednesday morning; big room)

**WG4**. Materials Modelling and Simulation Feb. 27, 2019 (Wednesday morning; big room)

**WG5**. Graphene/2D Materials, Feb. 25, 2019 (Monday afternoon, small room)

The venue of all meetings will be: rue du Trone 98, 1050 Brussels (big room - 8th floor, small room - 1st floor).

Honorary Colloquium Prof. Bernd Kieback and Colloquium on Powder Metallurgy will be be held on 27-28<sup>th</sup> March 2019 at Fraunhofer-IFAM Branch Lab in Dresden.

**EMRS 2019** The Spring Meeting of the European Materials Research Society (**E-MRS**) will take place on 27-31<sup>st</sup> May 2019 in Nice. The Fall Meeting will be held on 16-19<sup>th</sup> September 2019 in Warsaw.

**7<sup>th</sup> Dresden Nanoanalysis Symposium** on "Nanoscale characterization for cutting-edge materials research and sustainable materials development" will be held on 30<sup>th</sup> August 2019 in Dresden.

**EUROMAT 2019** Bi-annual European Congress and Exhibition on Advanced Materials and Processes will be held on 1-5<sup>th</sup> September 2019 in Stockholm.

**IC-MPPE 2019** International Conference and Workshops on Integrated Computational Materials, Process and Product Engineering together with the celebration of the 20<sup>th</sup> anniversary of Materials Center Leoben Forschung GmbH (**MCL**) will be held on 19-21<sup>st</sup> November 2019 in Leoben.

## WHAT'S NEW IN WORKING GROUPS?

Collaborative research between KMM-VIN members, applications for new projects and organisation of Industrial Workshops are for the time being the key activities of the KMM-VIN network carried out within the Working Groups (WGs). Currently KMM-VIN is composed of five Working Groups:

#### WG1. Materials for Transport

#### Coordinators:

Pedro Egizabal, Fundación Tecnalia, Donostia/SanSebastian, Spain (TECNALIA)

Thomas Weissgärber, Fraunhofer Institute for Manufacturing and Advanced Materials, Dresden, Germany (FRAUNHOFER-IFAM DD)

#### WG2. Materials for Energy / EMEP

Coordinators:

Monica Ferraris, Politecnico di Torino, Italy (POLITO) Christof Sommitsch, Graz University of Technology, Austria (TUG)

#### WG3. Biomaterials

Coordinators:

Aldo R. Boccaccini, Friedrich-Alexander Universität Erlangen-Nürnberg, Germany (FAU)

Christian Hellmich, Technische Universität Wien, Austria (TUW)

#### WG4. Materials Modelling and Simulation

Coordinators:

Andrés Diaz-Lantada, Universidad Politécnica de Madrid, Spain (UPM)

Jerzy Rojek, Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland (IPPT)

#### WG5. Graphene/2D Materials

Coordinator:

Peter Hansen, Haydale Graphene Industries PLC, Ammanford, UK (HGI)

Any member of KMM-VIN (core or associate) can join any WG upon prior consent from WG coordinators, with the exception of the EMEP (subgroup of WG2. Materials for Energy) where special participation rules apply (cf. <u>http://kmm-vin.eu/members\_area/wg2/</u> after login).

### NEWS FROM WG1: MATERIALS FOR TRANSPORT

#### Internal collaborative project:

## Manufacture of joined ceramics using high temperature stable titanium aluminides

Partners: POLITO (Monica Ferraris), CNR-ICMATE (Fabrizio Valenza), University of Hertfordshire (UH, Andreas Chrysanthou), Fraunhofer IKTS (Hans-Peter Martin).

This internal project of WG1 started in May 2017 with the aim to adopt the Ti-Al system as a high temperature braze for ceramic joining. TiAl is established as high temperature stable intermetallic material. Ceramic products are applied at high temperatures but available brazes suffer from limited thermal stability. Ceramics as alumina, silicon carbide, silicon nitride and zirconia were involved in wetting and joining experiments by the project partners. Thermal analysis, wetting behavior, mechanical properties and structural investigations were performed in 2018. Particular interest is directed to high temperature mechanical properties of joined ceramics. A web conference was organized in December 2018. The results and the work plan for 2019 were discussed.

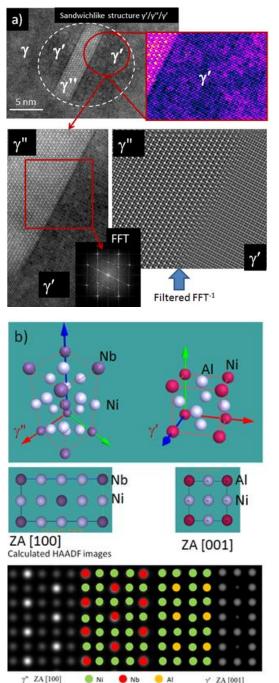
### **News from AGH-UST**

#### Advanced electron microscopy for analysis of the sandwich-like strengthening particles in Inconel 718 superalloy

The Inconel 718 is a nickel base superalloy widely used in aviation and energy industry, especially for turbine disks and also in other applications that require high strength materials for temperature up to 650 C. This alloy exhibits excellent mechanical and corrosion properties in the intermediate temperature range, good weldability and formability. The strength of this alloy comes from coherent precipitates of intermetallic phases: mostly y"-Ni<sub>3</sub>Nb and for a small part y'-Ni<sub>3</sub>(Al,Ti). The  $\gamma'$  and  $\gamma''$  phases precipitate in the temperature range from 600 to 900°C, simultaneously sequentially, depending on the chemical or composition of the alloy, Al/Ti ratio. The sequence of precipitation of  $\gamma'$  and  $\gamma''$  phases is oversaw by the relative concentration of AI, Ti and Nb. The high creep properties, and high-temperature strength of this alloy is attributed to the thermal stability of nanosized precipitates of y' (Ni<sub>3</sub>Al) and y" (Ni<sub>3</sub>Nb). The y" phase is a metastable form of Ni<sub>3</sub>Nb, which tends to transform to stable orthorhombic  $\delta$  phase with the structure D0a. The precipitation process that occurs during thermal ageing is responsible for the evolution of alloy's mechanical properties. Based on our experimental results  $\gamma'$  and  $\gamma''$  often form co-precipitates  $\gamma'/\gamma''$  or sandwich-like structure y'/y''/y' or y''/y'/y''. The ordered

 $\gamma'$  phase has the cubic L1<sub>2</sub> structure, and the  $\gamma''$  is bodycentred tetragonal corresponding to the D0<sub>22</sub> structure. The application of high-resolution scanning transmission electron microscopy (HAADF-HRSTEM) allowed for analysis in the atomic scale of complex precipitates mentioned above. Results of the analysis of sandwich-like  $\gamma' \gamma'' \gamma'$  particles strengthening Inconel 718 alloy are presented in Fig. 1.





**Fig.1.** Results of analysis of the sandwich-like structure of strengthening particles in Inconel 718 alloy. a) HAADF-HRSTEM image of Inconel 718 alloy showing the presence of the sandwich-like structure  $\gamma'/\gamma''/\gamma'$ , filtered HAADF image of interphase  $\gamma''/\gamma'$  region in atomic-scale b) atomic models of phases,  $\gamma''/\gamma'$  interphase calculated of HAADF images for  $\gamma''$  and  $\gamma'$  phases. (Courtesy of AGH-UST)

#### **News from CIDETEC**

As advanced in previous Newsletter (Issue 18), CIDETEC is currently involved in five new transport related projects: Mat4Rail, ECOLAND and AIRPOXY, coordinated by CIDETEC and HARVEST and CHOPIN as a partner.

**A correction** needs to be made from Newsletter 18, to clarify that project Chopin is not coordinated by CIDETEC, but by Materia Nova.

Since Mat4Rail and ECOLAND where presented in KMM-VIN Newsletter 18, this section is focused on the presentation of AIRPOXY, Harvest and Chopin.

AIRPOXY is a 3.5 year large scale collaborative project coordinated by CIDETEC within the Call H2020-MG-2017, in the H2020 framework. The AIRPOXY project aims at reducing production & maintenance costs of carbon composite parts in aeronautics by introducing a new family of enhanced composites. Such composites preserve all the advantages of conventional thermosets, but can also be easily processed and repaired, and even recycled.

This is now possible thanks to a family of groundbreaking thermoset resins recently invented and patented<sup>1</sup> by CIDETEC which presents reversible or "dynamic" bonds. These dynamic chemical bonds enable a series of "smart" properties, creating a new generation of thermoset composites that preserve their high performance, in terms of easy fiber impregnation and overall stability, while showing new unprecedented features once the composite is completely cured, such as Re-processability, Reparability and Recyclability, called "3R" (cf. Fig. 2).

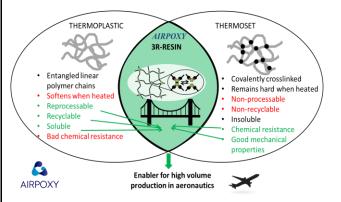


Fig.2. The "3R" resin in AIRPOXY project. (Courtesy of CIDETEC)

AIRPOXY relies on a multidisciplinary consortium of 11 partners from 6 European countries (Belgium, France, Germany, Ireland, Spain and Greece), and will cover the whole value-chain to solve manufacturing process cost-effectiveness problem.

For more information:

www.cidetec.es https://cordis.europa.eu/project/rcn/216013\_fr.html www.airpoxy.eu

<sup>1</sup> WO2015181054-A1

#### HARVEST

HARVEST is a 36 month project financed within the H2020 Call MG-1.4-2017, which started in September 2018. This projects aims at developing multifunctional ThermoElectric Energy Generating (TEG)-enabled structural composite materials for the Aeronautics sector. To do so, HARVEST envisages to combine bioinspired hierarchical TEG-carbon fiber (CF) reinforcements with novel 3R thermoset matrix systems (Repair-Recycle-Reprocess technology), through the combination of the expertise of 11 partners from 6 European countries.

In HARVEST project, CIDETEC, core member of KMM-VIN, leads two WPs dealing with the definition of the roadmap toward the development of hierarchical biomimetic TEG-enabled composite parts, on one hand; and the Dissemination, Exploitation & Communication Activities, on the other hand. Beside this role, CIDETEC is in charge of several key technical tasks and activities regarding the development of 3R epoxy resins with tuned electrical and thermal conductivities, and their use in the fabrication of easilyrepairable aerospace composites with thermoelectric properties. Additionally Università degli studi Di Padova (UNIPAD), associate member of KMM-VIN participates also in HARVEST project with the main role of developing analytical, numerical and hybrid models to study the thermolelastic properties of the novel TG-enabled composites.

For more information: <u>www.harvest-project.eu</u> <u>https://cordis.europa.eu/project/rcn/216003\_en.html</u> <u>www.cidetec.es</u>

#### CHOPIN

Chopin is a 3 year Clean Sky project, coordinated by Materia Nova, which addresses the development of highly durable hydrophobic coatings which can be applied to micro-perforated surfaces typically used for drag reduction. The validation of the technology and the coating process is also part of Chopin project, including tests to clearly assess the mitigation of insect contamination under realistic conditions. CIDETEC leads the WP3 Development of durable coatings.

For more information: <u>www.chopin-project.eu</u> <u>www.cidetec.es</u> <u>https://cordis.europa.eu/project/rcn/213937\_en.html</u>

#### **News from FRAUNHOFER-IKTS**

#### 6<sup>th</sup> Dresden Nanoanalysis Symposium

The 6<sup>th</sup> Dresden Nanoanalysis Symposium, organized by the Dresden Fraunhofer Cluster Nanoanalysis (DFCNA) and supported by the European Materials Research Society (E-MRS) and the European Materials Characterisation Council (EMCC), was held at Fraunhofer IKTS Dresden on August 31, 2018. This year, the symposium stood under the particular motto: "Materials challenges for automotive industry - Microand nanoscale characterization".

The symposium provided highlights in the field of nanoanalysis, represented by three keynote talks, 11 invited talks of world class speakers and a poster session. A report about the conference and the names of the poster prize winners are on the web page: <a href="http://www.nanoanalytik.fraunhofer.de/en/events/6DN">http://www.nanoanalytik.fraunhofer.de/en/events/6DN</a> S.html

The symposium covered the topics of nanoanalysis and materials characterization along the whole value and innovation chain, from fundamental research up to industrial applications. It brought together scientists and engineers from universities, research institutions, equipment manufacturers and industrial end-users. New results in disruptive nanoanalysis techniques were reported in several talks and in the poster sessions, and novel solutions in the field of materials characterization for process and quality control were shown. The discussions and interactions between the stakeholders helped to identify gaps in the fields of advanced nanoanalysis and materials characterization and to propose actions to close them and to support industrial exploitation of innovative materials.

#### CERANEA

Fraunhofer IKTS Dresden has started a funded research project "Multifunctional Ceramic/Graphene Thick Coatings for New Emerging Applications" (CERANEA) to develop novel conductive layered systems and coatings based on ceramic/graphite nanocomposites.

The developed non-oxide ceramic  $(Si_3N_4, SiC)/graphite$  multi-layered system with improved thermal and electrical properties is expected to be applied in energy, automotive and aerospace industry as well as in electronics-related industries, e.g. for integrity sensors, contacts, switches and electrical conductive ceramic parts.

The main challenge is to achieve an efficient transfer of electrical, thermal and mechanical loads in widely used non-oxide engineering ceramics using spark plasma sintering (SPS)/hot isostatic pressing (HIP) techniques. Graphite will be added in the ceramic matrix to form a skeleton-like material with high porosity.

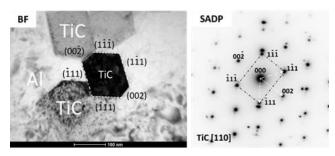
Fraunhofer IKTS will focus on improving the microstructure modulation on the micro/nano scale based on the multiscale characterization using special manipulators for in-situ X-ray microscopy, scanning electron microscopy and transmission electron microscopy.

The consortium has been formed together with two other European partners: Centre for Energy Research Hungarian Academy of Sciences (MTS EK) and Slovak Academy of Sciences (SAS). The project is funded by "Deutsche Forschungsgemeinschaft" (DFG) within the FLAG-ERA Joint Transnational Call 2017.

#### News from IMIM

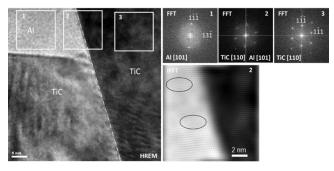
## In-situ cast composites strengthened with ceramic nanoparticles

The latest results of this project supported by Polish National Science Centre (research project no. 2016/21/B/ST8/01181) are concerned with microstructure investigations of aluminium based composite strengthened with the TiC particles being in nanometre size. The composites were fabricated by the casting method combined with in-situ formation of TiC particles. TEM microstructure observations have shown that TiC particles were of average size of 140 nm and faceted shape with walls of particles corresponding to specific crystallographic planes of TiC crystal structure (Fig. 3).



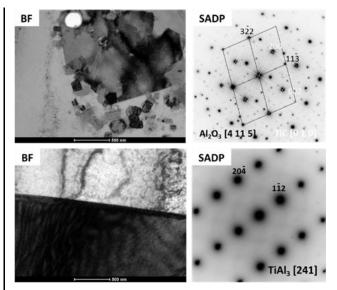
**Fig.3.** TEM BF and corresponding SADP images showing faceted TiC nano particle. (Courtesy of IMIM)

HRTEM investigations of the interface between AI matrix and TiC particles showed the lack of transition zones and reaction products at interface but existence of misfit dislocation located in the AI-matrix about 2 nm from interface boundary (Fig. 4).



**Fig.4**. HREM image and FFT images from the area marked by the white square IFFT obtained after masking procedure of FFT image from area number 2. (Courtesy of IMIM)

Two other, bigger kinds of particles with size of several microns and blocky shape of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and TiAl<sub>3</sub> phases were also identified in investigated composite which can influence both strengthening mechanism and plasticity of composite (Fig. 5).



**Fig.5**. TEM BF and corresponding SADP images showing Al<sub>2</sub>O<sub>3</sub> and TiAl<sub>3</sub> particles. (Courtesy of IMIM)

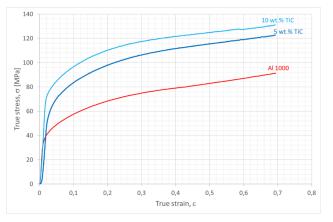
Also, the correlation between microstructure and mechanical properties was determined for these composites differing with technological parameters and composition. Summarizing these activity we find that microstructure investigations and compression tests of in-situ cast AI-1000/TiC composites reinforced with TiC nanoparticles allowed to determine several phenomena:

1. Self-propagation high temperature synthesis (SHS) process occurring during in-situ casting caused formation of TiC particles with average size of 155 nm and characteristic faceted shape. Also, as the reaction product the TiAl<sub>3</sub> particles in a form of blocky, longish plates were formed.

2. Two different kinds of particles containing silicon and iron differing with size and shape also were identified in the microstructure of composite. Origin of these particles is connected with contamination of base 1000 series aluminum alloy.

3. Significant increase of mechanical properties of about 44% were observed for in-situ cast composite as compared to AI 1000 series alloy (Fig. 6), mainly due to coefficient of thermal expansion (CTE) and elastic modulus (EM) mismatch between the reinforcement and the metal matrix, Hall-Petch relation and also in minority the Orowan effect.

4. A slight increase in strength was observed between the composites containing 5 and 10 wt.% TiC, which can be explained by different distribution of particles in the matrix and agglomeration process.



**Fig.6**. True stress-strain curves obtained during compression tests of two investigated composites and 1000 series aluminium alloy. (Courtesy of IMIM)

Wojciech Maziarz, IMIM <u>w.maziarz@imim.pl</u>

#### Development of high-efficiency and wastefree technology for the manufacture of magnetically soft nanocomposites for highfrequency high-power processing

A new research project funded by the National Center for Research and Development in Poland started in October 2018. The main aim is to develop innovative technology to produce Fe-based soft magnetic nanocomposites. The project includes the fabrication and multiscale characterization of structure. microstructure and magnetic properties of materials. The explanation of crystallization process step by step plays a key role in the design of materials with the best soft magnetic properties. Thus, in-situ heating TEM experiments as well as high resolution - HREM observations have been performed together with chemical composition analysis.

Iron based nano-crystalline soft magnetic alloys belong to extensively investigated materials due to their excellent soft magnetic properties and high saturation magnetization. They can be used in motors, transformers, actuators, sensors and electronic communication devices, recently especially in novel electro mobile advanced application [1]. Thus, from practical point of view it is very important to achieve an optimal magnetic properties which are strongly related to structure of alloys which depends on nucleation density. It was reported that the best soft magnetic properties show nanocomposites consisting of nanometric grains located in an amorphous matrix with the thickness of a few nm. This can be achieved by thermal annealing of amorphous alloys [2].

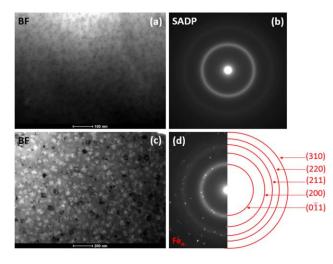
We have investigated the microstructure and structure of both as-spun and heat treated Fe67Co20B12 ribbons. Firstly, Fe67Co20B13 ingot was prepared by induction melting from high purity elements (more than 99.99 wt.%) in an argon atmosphere. Ribbons with the thickness of 20  $\mu$ m and 10 mm of width were manufactured by melt-spinning process. Thermal analysis of as-spun ribbons was carried out by

differential scanning calorimetry (DSC) with heating rate of 20 °C/min. The results allowed to select a temperature of heat treatment and ribbons were annealed at different temperatures for various times to show the dependence of microstructure on the heat treatment temperature. The main part of work was focused on transmission electron microscopy (TEM) observations performed by G2 operating at 200 kV equipped with an energy dispersive X-ray (EDX) microanalyser and high angle annular dark field detector (HAADF). Thin foils were prepared with TenuPol-5 double jet electropolisher using an electrolyte of perchloric acid (20 %) and methanol (80 %) at temperature of about 253 K followed by ion milling using ion polishing system.

The DSC results show two exothermic peaks at 388 and 508 °C corresponding to primary crystallization of α-Fe phase and crystallization of borides from remaining amorphous phase, respectively. Therefore, the temperatures of heat treatment were located between these two peaks (370, 410 and 485 °C). TEM experiments have been performed to investigate microstructure and crystal structure of ribbons. As can be seen in selected area diffraction patterns (SADP) (Fig.7 b), the presence of halo diffraction rings indicate amorphous phase. Nevertheless, HREM an observations reveal the existence of local crystalline clusters with the size of a few nanometers randomly embedded in the amorphous matrix. The lattice spacing of 0.21 nm suits (011) planes of  $\alpha$ -Fe. Thus, the as-spun ribbons show typical hetero-amorphous structure with medium-range order (MRO) in the amorphous phase. Nevertheless, some typical crystalline regions were also found in as-spun ribbons, which were indexed in accordance to  $\alpha$ -Fe structure showing that as-spun ribbon has not been fully amorphous (the results were proven by X-ray diffraction measurements). Fig. 7c demonstrates BF micrograph for Fe67Co20B13 ribbon heat-treated at 410 °C/30s. One can see  $\alpha$ -Fe grains randomly oriented in residual amorphous matrix. Corresponding SADP (Fig. 7d) can be well indexed in accordance with  $\alpha$ -Fe nano-crystallites. The averaged grain size ranged from 10-40 nm. Moreover, we have examined the chemical composition of alloys before and after heat treatment. The results show strong segregation of Co and Fe elements. In summary, the microstructure and crystal structure of both as-spun and heat-treated Fe67Co20B13 have been investigated by TEM method. It was shown that as-spun ribbon was almost fully amorphous with some ordered regions of  $\alpha$ -Fe as well as bigger  $\alpha$ -Fe grains. Whereas, heat treated sample was a composite consisted of α-Fe nanocrystallites embedded in the amorphous matrix.

**Partner search**: A KMM-VIN partner with experience and relevant equipment for Atomic Probe Microscopy for determination of chemical composition of nanocomposites in the atomic scale is sought.

**PhD opportunity**: We are looking for a student interested to make PhD degree in the framework of this research project.



**Fig.7**. BF image (a) and SADP (b) of Fe67Co20B13 asspun ribbon; BF image (c) and SADP (d) of Fe67Co20B13 heat treated at 410 °C/30s. (Courtesy of IMIM)

#### Acknowledgement

This work has been carried out within the frame of project TECHMATSTRATEG/347200/11/NCBR/2017 funded by National Centre for Research and Development (POLAND).

 Herzer G (2013) Acta Mater. 61 718-734
Hou Y, Sellmyer DJ (2014) Magnetic Nanomaterials (ISBN 978-3-527-34134-4)

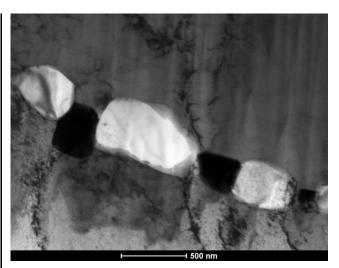
Anna Wójcik, IMIM a.wojcik@imim.pl

#### **IPPT – IMIM cooperation:**

## Improvement of the fracture toughness of NiAI intermetallic

The IPPT and IMIM teams have been cooperating on improvement of the fracture toughness of NiAl intermetallic at room temperature in view of its potential use in aero engines as a lightweight structural material replacing Ni-base superalloys.

The idea pursued in this collaborative work is to enhance the  $K_{IC}$  of NiAl by adding rhenium to NiAl powder and consolidate the NiAl(Re) mixture by hot pressing or SPS technique. Small amounts of rhenium (0.6 at.%; 1.25 at.%; 1.5 at.%) almost doubled the flexural strength of NiAl and improved its fracture toughness by 60%. Microscopic investigations have revealed rhenium particles at the boundaries of NiAl grains resulting in an enhanced fracture toughness (Fig. 8). More information about the processing route and properties of NiAl(Re) material can be found in a recent paper [1].



**Fig.8.** TEM image of NiAl+1.5at%Re. Rhenium particles (bright phase) at the boundaries of NiAl grains are responsible for enhancement of strength and fracture toughness of NiAl, dark particles are aluminum oxide formed during the process. (Courtesy of IPPT and IMIM)

[1] K. Bochenek, W. Węglewski, J. Morgiel, M. Basista Influence of rhenium addition on microstructure, mechanical properties and oxidation resistance of NiAl obtained by powder metallurgy, *Materials Science and Engineering A*, 735, 26, 121-130, 2018.

> Michal Basista, IPPT mbasista@ippt.pan.pl

## NEWS FROM WG2: MATERIALS FOR ENERGY

Collaborative research in WG2 is being conducted within two thematic subgroups: "EMEP" and "non-EMEP". The EMEP partners follow the work model and research programme of the former COST proposal "Engineered Micro- and nanostructures for Enhanced long-term high-temperature materials Performance" (EMEP). The internal work programme EMEP consists of Work Topics (WT) such as: WT1. Advanced Materials Modelling & Design, WT2. Materials Development and Manufacturing (A. Pipework and Tubing, B. Castings, C. Forgings), WT3. Materials Process Development and WT4. Testing and Validation.

#### HOWEFLEX

## Qualification of a new turbine material for improved flexibility and higher efficiency

HOWEFLEX is aiming for the qualification of a new rotor steel applicable in standardized high-tech steam turbines for

- modernization and improvement of existing steam power plants with demand for increased operational flexibility (Europe), - combined cycle power plants with increased exhaust temperatures of the gas turbine to improve the efficiency in combined cycle applications (worldwide),

- base load machines with supercritical steam parameters to improve the overall efficiency in steam power plants (China, India, Japan, Korea, Indonesia).

This activity is intended to maintain European competitiveness in the field of creep resistant steel alloys. Deployment of this material solution reduces the amount of expensive Ni-base alloys required for the highest temperature plant components.

The project is structured into 9 work packages. The 5 German project partners are Siemens Gas and Power (project lead), GE Power, Saarschmiede, University of Darmstadt, Fraunhofer IWM Freiburg.

Forgemaster Saarschmiede will manufacture a rotor part with typical steam turbine dimensions. The real rotor part will be investigated by nondestructive and destructive testing. For this, test specimens will be taken from different positions in the rotor part (surface, center) to determine the microstructure and mechanical properties.

Especially testing with service relevant conditions (e.g. number of cycles, holding times, stress-strain interaction) will be applied to investigate the material behavior under high flexible loading. The involved institutes are qualifying different material models to describe the material characteristics to allow for improved rotor design.

The material models will be explored by industrial partners Siemens Gas and Power and GE Power with the aim to implement them into turbine design and to bring the new material into practice.

The project is funded by German Federal Ministry for Economic Affairs and Energy for a period of four years. The kick-off meeting with all partners was held on September 11<sup>th</sup>, 2018. Next steps for industry and universities have been agreed, and the revised time schedule was discussed. The rotor manufacturing at Saarschmiede will start beginning of year 2019.

> Torsten-Ulf Kern Coordinator KMM-VIN WG2-EMEP WT2. FORGINGS Siemens AG, Power and Gas Division <u>torsten-ulf.kern@siemens.com</u> <u>www.siemens.com/ingenuityforlife</u>

### News from IKTS

## Ceramic components for thermochemical heat storage

Thermochemical heat storage is a promising alternative to sensible or latent storage technologies based on water tanks. Fraunhofer IKTS develops, modifies and processes highly porous ceramics providing interesting approaches for heat storage applications. One approach are cylindrical zeolite pellets which are covered with a good thermal metal conductor (Fig. 9). Thus, their thermal conductivity is increased by a factor of five allowing for faster charging and discharging cycles.

Another technological approach are heat storage bricks enabling a higher heat storage system filling degree and better contact with the heat exchanger than it is achievable with packed beds. The networklike permeation of the zeolite bricks improve the accessibility of the adsorbate to the inner volume of the adsorbent.

Another approach is the development of so-called composite adsorbents. These materials consist of a mesoporous support structure that has a high specific surface area and is impregnated by an inorganic salt. In contact with water, the salt forms different hydrate phases and thereby releases heat. The advantage of these materials is the comparatively low loading temperature (~ 90 °C). Typical support structures are silica gel, attapulgite, and carbons of large surface area.

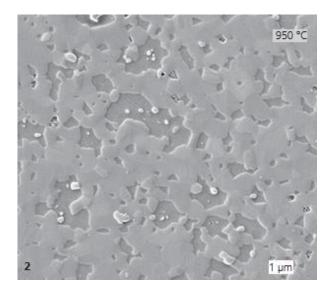


**Fig.9.** Al and copper-coated zeolite pellets to improve thermal conductivity in heat storage applications. (Courtesy of IKTS)

## New materials for lithium-ion conducting solid-state electrolytes

The energy supply requirements of mobile consumer devices, stationary energy storage devices, and electromobility are driving the development of efficient accumulators. Li-ion batteries only attain incremental improvements in power density, operating voltage, and charging and discharging rates. It should be possible to surpass these limits with new battery concepts, such as lithium-sulfur cells or all solid-state batteries. Fraunhofer IKTS develops ceramic materials suitable for solid electrolytes and separators

The system  $Li_{1+x}Al_xTi_{2\cdot x}(PO_4)_3$  (LATP, Fig. 10), for example, shows Li-ion conductivities of up to  $4 \cdot 10^{-4}$ S cm<sup>-1</sup> at 25°C and high thermal a mechanical stability. Preparation of these materials as powders with the desired particle size distributions can be realized by melting a glass frit with subsequent grinding, solidphase synthesis, or the sol-gel process. The selected production route and the stoichiometric composition of the material influence the achievable particle shapes/sizes and sintering activities of the powders. Application of Li<sup>+</sup>-conducting powders as fillers in porous, polymer-bound films allows for hybrid-type separators with significantly higher conductivities than for non-conducting ceramic fillers (e.g. Al<sub>2</sub>O<sub>3</sub>).



**Fig.10.** Fine-grained, dense micro-structure of an LATP solid electrolyte. (Courtesy of IKTS)

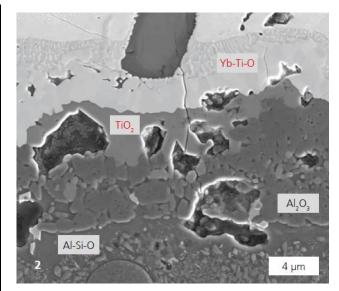
## Corrosion protection coatings for ceramic fiber composite materials

Intensive R&D activities in recent years have brought about several high-temperature-stable ceramic matrix composites (CMC), which offer a high potential for structural applications in advanced energy-producing gas turbines.

Although CMCs are suited for extreme conditions, they show corrosion- and abrasion-related damage, especially in long-term application. Therefore, Fraunhofer IKTS develops and tests coating materials (environmental barrier coatings EBC), which distinctly increase the corrosion stability of CMCs.

Typical current EBC systems with good functionality consist of a bond coat from silicon, and a corrosion-resistant topcoat, such as BSAS, rare-earth silicates, YAG or  $ZrO_2$  and HfO<sub>2</sub> compounds. The Si layer acts as an effective diffusion barrier against O<sub>2</sub> and H<sub>2</sub>O penetrating into the ceramic fiber composite material, but for long-term application it is not sufficient.

In order to improve the lifetime of the EBC system, modifications were made to the layer structure. An  $Al_2O_3$  bond coat was used instead of an Si layer. Furthermore, TiCN particles were incorporated into the bond coat. Thus, it was possible to specifically influence the degradation processes taking place in the hot gas and thus to improve markedly the service life behavior of the entire EBC system (Fig. 11).



**Fig.11**. Layer composition of Al<sub>2</sub>O<sub>3</sub> bond coat with TiCN particles after oxidation (1200°C, 100h). (Courtesy of IKTS)

Hans-Peter Martin, IKTS Hans-Peter.Martin@ikts.fraunhofer.de

#### News from AGH-UST

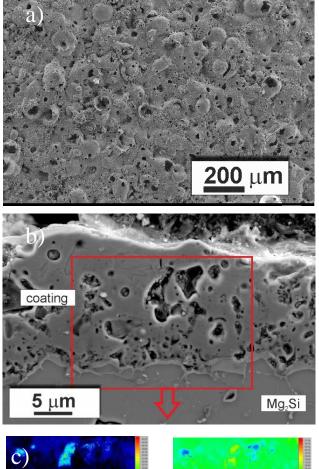
#### Surface modifications of magnesium silicide using Plasma Electrolytic Oxidation technique

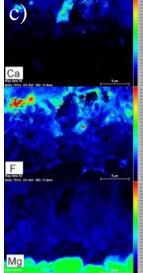
Materials based on Mg<sub>2</sub>Si can be applied in thermoelectric devices, hydrogen storage systems or electrodes in electrochemical cells. The main objective of the study is to answer the question, whether or not and in what conditions the deposition of Plasma Electrolytic Oxidation (*Plasma Electrolytic Oxidation*, PEO) coatings on Mg<sub>2</sub>Si is feasible.

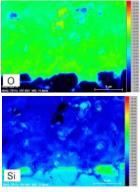
The PEO technique is used for materials surface modifications and is based on electrochemical oxidation enhanced by high voltages, in the rage of several hundred volts, and an alternating current. Because improvement of the corrosion resistance of Mg<sub>2</sub>Si is indicated as an expected result of the study, and the PEO coatings are usually porous, it is important to control porosity by proper selection of the process parameters. The performed studies were focused correlations between on coating microstructure and different process conditions, e.g. current-voltage parameters, duration, electrolyte composition.

Evaluation procedure includes characterization of micro- and nanostructure of the coatings, their chemical and phase composition, assessment of the influence of the coating on the properties of the substrate and examination of the coating/substrate interface. So far the oxide and oxide-fluoride coatings have been successfully deposited on Mg<sub>2</sub>Si materials by means of the PEO technique and the influence of process conditions on coating microstructure was identified, which allowed control of coating porosity.

Surface and cross-section of an exemplary coating are presented in Fig.12. These investigations will contribute to a better understanding of the phenomena accompanying coating growth on Mg<sub>2</sub>Si and will lay foundations for further coating developments involving the PEO technique.







**Fig.12.** SEM images of a coating produced by PEO technique a) sample surface, b) sample cross-section, c) quantitative maps of selected chemical elements. (Courtesy AGH-UST)

Kinga Zawadzka, <u>kinga @agh.edu.pl</u> IC-EM AGH-UST Krakow

### **News from TUG**

## Microstructural modelling of creep deformation in martensitic steels

This study deals with the microstructural modelling of martensitic steels during creep exposure. The applied approach is based on literature models (Ghoniem [1], Basirat [2], Yadav [3]) and has been further improved in significant parts in the course of the project. Huge advantage of the approach is that creep deformation can be simulated in parallel to the microstructural evolution. With this strategy, the model always provides a second reference to the experimental data, which simplifies testing individual parts of the model and improves the reliability of the approach.

Following parts of the model have been combined from the individual sources:

#### Ghoniem:

- Microstructural representation of the martensitic steel.
- Rate equations for the evolution of the microstructural constituents

#### Basirat:

• Damage model

#### Yadav:

• Method for combining the approaches from Ghoniem and Basirat

Recent work at IMAT:

- Interaction of precipitates in subgrains (e.g. VN) with mobile dislocations.
- Revised backstress concept, now compatible to hot deformation.
- Combination of microstructural model with precipitate kinetic simulations carried out with the software MatCalc [4].
- Calculation of creep curves as well as time-torupture diagrams.

Not all parts of the model represent distinctive physical interactions, some parts (mostly the parts dealing with damage evolution) still rely on phenomenological approaches. On the long run, these parts are to be replaced by according mechanistic sub-models.

Currently, the phenomenological parts demand a small number of fit parameters. These parameters are determined by comparing the simulation results (creep deformation and microstructural evolution) with one single creep curve at a specific temperature and stress. Once the overlap of simulated and experimental creep curve has been optimized, the same fit-parameters can be applied to a wide range of stresses at the same temperature. Result of the calculation is a time-to-rupture diagram, which can then be compared to standard literature data. Fig.13 shows an example on the creep resistant martensitic steel P91. This material was chosen for test calculations due to the good availability of literature data.

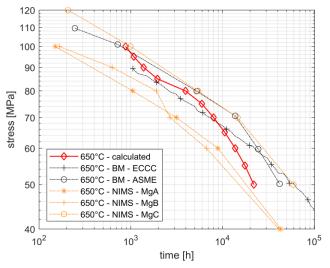


Fig.13. Time-to-rupture diagram of P91 base material, simulated result vs. literature data [5, 6]. (Courtesy of TUG)

Input data for the simulation are the microstructure of the material in as-received state and results from precipitate evolution calculations carried out in the software MatCalc for heat treatment and creep regime. The fit-parameters have been optimized on one single creep curve stemming from experiments at 650°C/80 MPa. All other fracture times from 50-100 MPa are results from the model and simulation routine. Literature data for comparison have been taken from the standard-datasheet from ECCC, ASME and NIMS [5, 6]. Summarizing it can be stated that simulation and experiment show good agreement. Next steps are replacing the phenomenological parts of the model and the fit-parameters with physically sound approaches, and to test the model at different temperatures and materials.

Bernhard Sonderegger, Bernhard Fercher Florian Riedlsperger, Bernd Niederl Institute of Materials Science, Joining and Forming Graz University of Technology (TUG)

#### Acknowledgment

B. Sonderegger and F. Riedlsperger would like to thank for financial support from the FWF Austrian Science Fund within the project P-31374 "Software Development on Dislocation Creep".

[1] N. M. Ghoniem, J. R. Matthews, R. J. Amodeo, Res Mechanica 29 (1990) pp. 197-219

[2] M. Basirat, T. Shrestha, G.P. Potirniche, I. Charit, K. Rink, Int. Journal of Plasticity, Vol. 37 (2012) pp. 95-107

[3] S. D. Yadav, B. Sonderegger, M. Stracey, C. Poletti, Mat. Sci. and Eng. A, Vol. 662 (2016) pp. 330-341

[4] https://www.matcalc.at/

[5] ECCC Data Sheets, Steel Grade 91 (X10CrMoVNb9-1) (2009)

[6] Creep Deformation Properties of 9Cr-1Mo-V-Nb Steel Plates for Boilers and Pressure Vessels, (ASME., SA-387/SA-387 M., Grade 91), Atlas of Creep Deformation Property, NIMS., Creep Data Sheet, N.I.M.S., Tsukuba, No., *D*-2 (2008)

### From WUT to TUG

(KMM-VIN Research Fellowship, call 2018)

## Joining of ultrafine grained aluminium plates using Friction Stir Welding

PhD student: Marta Orlowska (WUT) Host: Norbert Enzinger (TUG)

grained Ultrafine materials reveal enhanced mechanical properties due to the elevated amount of grain boundaries, which act as obstacles for moving dislocations. Nevertheless, the main disadvantage of these materials is their reduced thermal stability. Therefore, methods, in which joining is performed under a solid state might be suitable for such materials. During the research stay Friction Stir Welding (FSW) method was used for materials with different technological history, mainly after severe plastic deformation. The main goal was to evaluate the FSW effectiveness in welding of ultrafine grained materials.



**Fig.14.** Examples of dissimilar welds of commercially pure aluminium after different plastic deformation processes at different degree of deformation. (Courtesy WUT / TUG)

The first stage was devoted to set proper welding parameters to obtain good quality welds of coarse grained commercially pure aluminium AA1050 (99.50 wt.%). The experiments consisted in the selection of a tool (e.g. pin profile), tool depth, rotational and linear speed (800 rpm, 200-800 mm/min). Then, experiments with ultrafine grained commercially pure aluminium were performed. To obtain ultrafine grained structure incremental equal channel angular pressing and hybrid process consisted of multi-turn equal channel angular pressing with subsequent upsetting have been chosen. Both similar and dissimilar welds were manufactured. Similar welds were produced from samples after the same processing technique and with the same deformation degree (i.e. the same amount of applied total true strain). Dissimilar welds (cf. Fig. 14) were performed on samples with different deformation degree but also different aluminium alloy was implemented. Al-Mg-Si (AA6082) alloy has been chosen as an age-hardenable. It has been confirmed that FSW method can be used for ultrafine grained materials. Furthermore, welds were produced for further investigations of the effect of grain size and content of high angles grain boundaries on the microstructure and properties of welds.

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## **NEWS FROM WG3: BIOMATERIALS**

#### POLITO-TUG: Micro and nano structuring in titanium alloys

#### Internal collaborative project

Partners: Sara Ferraris, Silvia Spriano (POLITO), Fernando Warchomicka, Christof Sommitsch (TUG)

During the last two years, Politecnico Torino (POLITO) and Graz University of Technology (TUG) were developing the structuring of micro grooves on the surface of different titanium alloys. Electron beam technique was used to perform grooves patterns between 5 to 30µm to determine the bacteria and cell adhesion (Fig.15). Physicochemical characterization of the surface was performed on the surface of 3 different titanium alloys (Ti Gr2, Ti-6AI-4V and Ti-15Mo), observing a good reproducibility of grooves higher than 10µm, with a depth up to 300nm. The fast cooling obtained during the process provoked a formation of martensite in Ti Gr2 and Ti-6Al-4V that distorts the topography. Concerning to the biological response, the structured surface showed a good adhesion of the fibroblast cells with a strong alignment parallel to the grooves direction by 10µm wide grooves. The bacterial adhesion decreased in the first 48hs for TiGr2 and Ti-6AI-4V. This reduction of the bacteria colonization might be related to the nano features produced by the formation of martensite, independently of the groove dimensions.

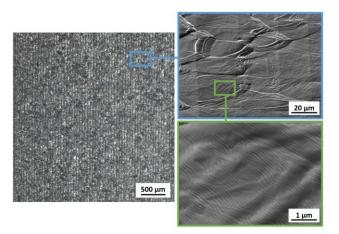


Fig.15. Microstructuring of titanium alloy Ti-6AI-4V by electron beam technique. Details of micro and nano features for structured surface of 30µm wide grooves. (Courtesy of POLITO / TUG)

#### Recent publication:

S. Ferraris, F. Warchomicka, C. Ramskogler, M. Tortello, A. Cochis, A. Scalia, G. Gautier di Confiengo, J. Keckes, L. Rimondini, S. Spriano, Surface structuring by Electron Beam for improved soft tissues adhesion and reduced bacterial contamination on Ti-grade 2, Journal of Materials Processing Technology, 266, 518-529, 2019

https://doi.org/10.1016/j.jmatprotec.2018.11.026

### From WUT to FAU

(KMM-VIN Research Fellowship, call 2018)

PhD student: Agata Sotniczuk (WUT) Host: Aldo R. Boccaccini (FAU)

#### Designing antimicrobial coatings on nanocrystalline titanium

Nanocrystalline titanium due its sufficient mechanical strength, excellent biocompatibility and high corrosion resistance in artificial saliva solutions is a promising material for dental implants. However, the presence of oral bacteria such as S. mutans, and products of their metabolism, may be aggressive for titanium passive layers and accelerate material degradation. Thus, the main idea of this fellowship was to design and fabricate coatings which should enhance the antibacterial properties of nanoscaled titanium surfaces.

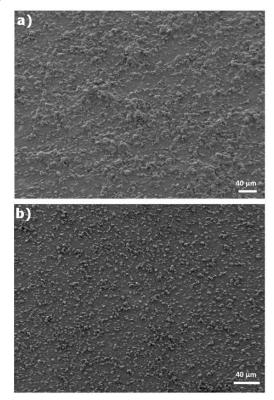


Fig.16. SEM micrographs of chitosan-BG composite coatings obtained by EPD on nanocrystalline titanium. showing the effect of applied voltage on the surface morphology of coatings: a) 40V, b) 20V. (Courtesy of WUT / FAU)

Sufficient adhesion to the nanocrystalline substrate and a positive influence on bioactivity were other requirements for the novel coatings. For this reason, composite chitosan (CG) - bioactive glass (BG) coatings, produced by means of cost-effective electrophoretic deposition (EPD), were selected, following from previous related research at the Institute of Biomaterials (FAU) [1].

The first stage of experiments was to find the appropriate parameters of EPD. Different voltages (50, 45, 40, 35, 30, 25, 20, 15, 12 V) and different times of deposition (1, 2, 3 minutes) were tested. The distance between electrodes was always the same (1cm). After morphology of the surfaces deposition, was

investigated by microscopic techniques (OM, SEM) and the structural quality of coatings and the adhesion to the substrate were examined by the tape test (according to ISO 2409). Based on the results of microscopic observations, two combinations of EPD parameters were selected for further studies (15 V/3 min and 20 V/3 min).

The next step involved optimization of surface roughness in order to provide the best adhesion of the coatings to the nanocrystalline samples. A typical morphology of the final coatings is provided in Fig. 16. During this fellowship EPD coatings were fabricated on 40 round-shaped samples. Further collaboration between FAU and WUT will focus on investigating the bioactivity and antibacterial effect provided by the coatings.

[1] S. Seuss, et al, Alternating current electrophoretic deposition of antibacterial bioactive glass-chitosan composite coatings, *Int. J. Mol. Sci.* 15 (2014) 12231–12242.

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#### FROM FAU to POLITO

(KMM-VIN Research Fellowship, call 2018)

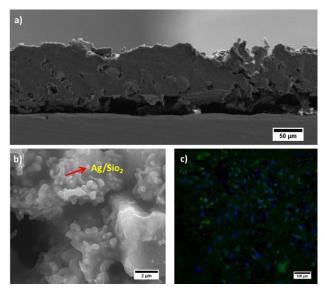
PhD student: Qaisar Nawaz (FAU) Host: Monica Ferraris (POLITO)

#### Multifunctional (Bioactive and Antibacterial) coatings containing silver nanocluster-silica on PEEK/mesoporous bioactive glass nanoparticle substrates by Radio-Frequency (RF) co-sputtering

The development of inorganic (bioceramics)/ organic (biopolymer) composite coatings, as inspired by organic-inorganic composition of natural bone, is gaining increasing interest for bio-functionalization of orthopedic implants [1]. In this study bioactive and antibacterial composite coatings were developed focusing on PEEK based coatings, following a previous joint project between FAU and POLITO [2], and using novel mesoporous bioactive glass nanoparticles (MBGNs) developed at the Institute of Biomaterials (FAU) [3]. A thin layer of Ag nanocluster-silica composite (150 nm) was deposited on PEEK/MBGNs coated substrate (deposited via electrophoretic deposition, EPD) on PEEK/MBGNs coated substrate (deposited via electrophoretic deposition, EPD).

Two sputtering conditions were used by varying the deposition time (20 and 40 min). These coatings were characterized in terms of morphology, composition, in vitro bioactivity, adhesion strength, cytotoxicity, antibacterial activity, and ion release. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX) confirmed the presence of silver nanoclusters, which were homogeneously embedded in the silica matrix (Fig. 17).

The presence of MBGNs particles allows the coatings to form apatite-like crystals upon immersion in simulated body fluid (SBF). The release of Ag ions from the coatings leads to the antibacterial effects against *staphylococcus carnosus* and *Escherichia coli*, which was confirmed from the inhibition hallo test. However, the release of Ag ions did not show any cytotoxic effect against MG-63 (osteoblast cells).



**Fig.17.** a) SEM images of PEEK/MBGNs composite coatings produced using EPD and sintered at 375°C; b) SEM image of Ag/SiO<sub>2</sub> deposited on PEEK/MBGN coating via RF-co sputtering c) fluorescence micrograph showing MG-63 cells on surface of composite coating. (Courtesy of FAU / POLITO)

[1] A. Simchi et al. Recent progress in inorganic and composite coatings with bactericidal capability for orthopedic applications, *Nanomedicine: Nanotechnology, Biology and Medicine*, 7 (2011), 22-39

[2] M. A. Ur Rehman, et al., Antibacterial and Bioactive Coatings Based on Radio Frequency Co-Sputtering of Silver Nanocluster-Silica Coatings on PEEK/Bioactive [Glass Layers Obtained by Electrophoretic, *ACS Applied Materials* & Interfaces 9 (2017) 32489-32497.

[3] Q. Nawaz, et al., Synthesis and characterization of manganese containing mesoporous bioactive glass nanoparticles for biomedical applications, J. Mater. Sci. Mater Med. 29 (2018) 64.

#### From AGH-UST to POLITO

(KMM-VIN Research Fellowship, call 2018)

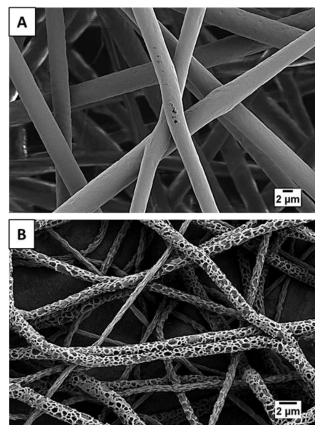
PhD student: Sara Metwally (AGH-UST) Host: Silvia Spriano (POLITO)

#### Zeta potential measurement of electrospun polymer fibers and spin - coated films for biomedical applications

The purpose of the research was to verify surface net charge of PCL samples in simulated biological conditions for tissue engineering applications. The study included measurement of zeta potential on spin-coated poly( $\epsilon$ -caprolactone) (PCL) films, and electrospun fibers. Non-porous and porous PCL fibers were produced applying positive and negative voltage

polarity to the nozzle during electrospinning, see Fig.18.

The zeta potential was determined by streaming potential measured in SBF solution. The study shown higher zeta potential for PCL fibers produced with negative compared to fibers produced with positive voltage polarity. The increase of zeta potential is related to polymer chain reorientation during electrospinning and decreased oxygen content at fibers' surface [1]. The result confirmed that altering voltage polarity is able to change zeta potential on electrospun fibers. during electrospinning and decreased oxygen



**Fig.18**. SEM images of: A) non-porous, B) porous PCL fibers produced applying positive voltage polarity to nozzle during electrospinning. (Courtesy of AGH-UST/ POLITO)

The research stay contributed to development of the lasting collaboration between the International Centre of Electron Microscopy for Materials Science at AGH-UST and Department of Applied Science and Technology in Politecnico di Torino.

[1] S. Metwally, J.E. Karbowniczek, P.K. Szewczyk, M.M. Marzec, A. Bernasik, U. Stachewicz, *Single-Step Approach to Tailor Surface Chemistry and Potential on Electrospun PCL Fibers for Tissue Engineering Application*, Adv. Mater. Interfaces. (2018) 1801211. doi:10.1002/admi.20181211.

Sara Metwally, AGH-UST metwally@agh.edu.pl

## NEWS FROM WG4: MATERIALS MODELLING AND SIMULATION

**8th KMM-VIN Industrial Workshop** on "Modelling of composite materials and composite coatings" was held at the Fraunhofer Institute for Mechanics of Materials IWM in Freiburg, Germany on 9<sup>th</sup>-10<sup>th</sup> October 2018.

The workshop was chaired by Jörg Hohe from Fraunhofer IWM and Michal Basista from KMM-VIN. Jörg Hohe headed the local organizing committee formed by Carla Beckmann, Claudio Findeisen and Achim Neubrand.

Composite materials and coatings are important elements in many fields of modern lightweight construction. Beside the classical field of the aerospace sector, composite materials are nowadays widely used in all transport sectors including the automotive, rail and naval industries, the wind energy sector and other mechanical and civil engineering applications as well. The workshop was dedicated especially to the modelling of composite materials and coatings across all relevant industries and all relevant classes and types of composites.

Two invited keynote lectures were directed to the application of lightweight polymer composites and high temperature ceramic matrix composites in spacecraft launcher and aircraft engine applications, respectively, both using advanced material models. The two industrial keynote lectures were complemented by two keynote lectures from academia, providing an insight into current research activities on modelling and optimization of different types of advanced composite and lightweight cellular materials. A broader overview over the current research and development activities in both, academia and industry, in the field of composite material modelling was given in 22 contributed presentations coming from the group of the participants. The contributions were grouped in four contributed sessions, directed to different topics in modelling of composites. The first session was directed to the definition and application of advanced material models for composite materials, considering both, stiffness and strength as the main aspects in structural assessment. In the second session, special interest was directed to advanced experimental methods for determination of the necessary parameters. Multiscale numerical approaches bridging different levels of structural hierarchy were addressed in the third session. In the fourth session, mean field approaches for numerical determination of effective properties were treated. Both, full field multiscale and mean field approaches together with the corresponding experimental methods for parameter identification form the base for the successful development and application of advanced composite material models.

Jörg Hohe, Fraunhofer-IWM

### **WG4 JOINT ACTIVITIES**

## Special Issue on "Multi-scale modeling of materials and structures"

Jerzy Rojek and Andrés Díaz Lantada are co-editing a special issue for Materials MDPI journal on "Multi-scale modeling of materials and structures". MDPI journals publish on an open-access basis for improved visibility. Until now the special issue has published five papers linked to different multi-scale modeling strategies with applications linked to: the design alloys with enhanced mechanical and thermal performance, the processing of metallic powder using lasers, the modeling of grain growth, the optimization of cementitous composites and the design of active metamaterials.

Submissions will be received until 30th June of 2019.

Should you have any questions, please do not hesitate to contact co-editors. It would be a deep honor to receive submissions from KMM-VIN colleagues. Please visit the special issue website:

https://www.mdpi.com/journal/materials/special\_issue s/Multi\_Model\_Mater\_Struct

## Forthcoming EU calls on materials modeling and simulation:

Several interesting research topics, deeply connected to materials modeling and simulation, will be funded by Horizon 2020 Working Programme the on "Nanotechnologies, Advanced Materials. Biotechnology and Advanced Manufacturing and Processing" through some calls opening along 2019 and 2020. Among the most relevant for the promotion of collaboration among KMM-VIN partners, we would like to highlight and propose joint activities in two specific calls:

- DT-NMBP-10-2019: Adopting materials modelling to challenges in manufacturing processes (RIA). It is a two stage call with January 22<sup>nd</sup> as deadline for the first stage. KMM-VIN members could join forces for supporting the connections between materials producers and product manufacturers through modeling apps for supporting decision making, especially in connection with the field of modeling additive manufacturing processes, which is of interest for many members.

- DT-NMBP-11-2020: Open Innovation Test Beds for Materials Modelling (IA). In connection with other open-innovation test bed projects on materials design and characterization, proposals for such call should establish an open innovation modeling test bed that will create, sustain and drive the use of novel materials modeling techniques to support industrial innovation. In connection with the modeling expertise of KMM-VIN members and the industrial areas of interest (transport, energy, health), KMM-VIN could focus on the implementation of an online infrastructure for supporting EU industries in optimizing materials, processes and products through modeling techniques. For KMM-VIN members interested in collaborating in proposals linked to the aforementioned topics, please contact WG4 coordinators:

Andrés Díaz Lantada: <u>andres.diaz@upm.es</u> Jerzy Rojek: <u>jerzy.rojek@kmm-vin.eu</u>

## NEWS FROM WG5: GRAPHENE / 2D MATERIALS

The main aims of the group are to facilitate the exchange of the latest research in graphene/2D materials and the formation of projects to further this research. We would welcome new members to the group from within KMM-VIN as well as new members to KMM-VIN who may be interested in this topic (Contact: peter.hansen@haydalecs.com).

In this issue of the Newsletter some achievements of two members of WG5 on graphene and graphene containing materials are presented: industrial company (Haydale) and R&D institute (ITME).

### Space box (or CubeSat)

#### Haydale materials went into space

Haydale materials went into space in December 2017 with a CubeSat launch to near space to compare the performance of graphene enhanced and conventional carbon fibre materials. The project was led by the University of Central Lancashire (UCLan) and partfunded by the UK Space Agency (UKSA) with Haydale Composite Solutions providing the prepreg material.

The CubeSat was manufactured by UCLan (using traditional manufacturing methods – no special handling required) from Haydale prepreg with enhanced electrically conductivity. The materials were originally developed in the NATEP project GraCELs for aerospace applications. The graphene enhanced version came out 15% lighter according to partners UCLan.

To ensure accurate results, sensors were attached to each casing to monitor and record key data including how they react to temperatures of up to minus 60 degrees, and the effects of very low pressure. The data is yet to be fully analysed but should provide raw data that will enable decisions to be made on the future uses of these materials.

The mechanical properties of the modified material have been evaluated and it has been shown that the properties are maintained or improved by the addition of nanomaterial, with some improvements of up by 15-20%. The technology has the potential to be used in other fields including:

- Anti-static enclosures for electronics
- Lightning strike protection for wind turbine blades, UAVs, airships
- Lightweight space structures with anti-static requirements



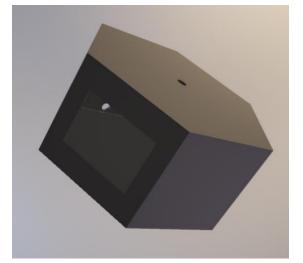


Fig.19. The CubeSat at 130,000 feet. (Courtesy of Haydale)

Haydale have also developed nano-carbon enhanced epoxy adhesives that can be used for bonding together structures and enclosures made from the electrically enhanced prepregs.

## Haydale Supplies Graphene for World's First Graphene Skinned Plane

Haydale has supplied graphene enhanced prepreg material for Juno, a three-metre wide grapheneenhanced composite skinned aircraft, that was revealed as part of the 'Futures Day' at Farnborough Air Show 2018. The prepreg material, developed by Haydale, has potential value for fuselage and wing surfaces in larger scale aero and space applications especially for the rapidly expanding drone market and, in the longer term, the commercial aerospace sector. By incorporating functionalised nanoparticles into epoxy resins, the electrical conductivity of fibrereinforced composites has been significantly improved for lightning-strike protection. The Juno project, led by UCLAN, has been an ideal demonstration for the viability of the prepreg material for structural applications and the ability to manufacture components using traditional composite manufacturing methods.



*Fig.20*. Juno world's first graphene skinned plane. (Courtesy of Haydale)

Further developments are underway to produce the next iteration of lightning strike protection materials based on these nano-carbon enhanced prepregs.

This technology also has performance benefits for a wide range of applications and industries including large offshore wind turbines, marine, oil and gas, and electronics and control systems.

Haydale worked with the aerospace engineering team at University of Central Lancashire, Sheffield Advanced Manufacturing Research Centre and the University of Manchester's National Graphene Institute to develop the unmanned aerial vehicle, that also includes graphene batteries and 3D printed parts.

#### Haydale Receives Funding to Develop Graphene Composite Tooling and Automotive Body Panels

Haydale's lead consortium joined by Briggs Automotive Company (BAC) and Pentaxia has been awarded a research and development grant from the Niche Vehicle Network of a ca. £250,000 to develop graphene enhanced composite tooling and graphene enhanced automotive body panels. Through development in this core strategic area, Haydale is looking to reduce cycle times compared to existing tooling methods, as well as reduce weight and increase performance of component material.

## Haydale's functionalised graphene ink sales to the biomedical sensor market

Haydale Technologies Taiwan ("HTW"), has started to supply commercial quantities of its functionalised graphene ink to a major print house in Taiwan. The graphene ink is used to print test strips in the selfdiagnostic biomedical sensor device market for diabetes monitoring. Haydale's graphene ink is proving to be a high quality, more stable, and consistent product, replacing the established graphite and carbon inks used extensively in producing the test strips for this significant global market. Some customers are now trialling an all-carbon sensor utilising HTW's graphene-enhanced ink that is aimed at removing the need for the expensive silver conductive ink tracks extensively used today. The outcome is a more environmentally friendly (potentially recyclable) product.

#### News from ITME

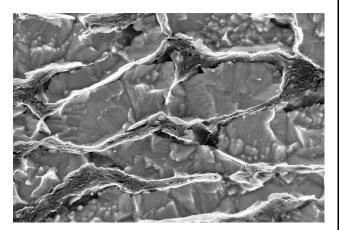
The ongoing activities dedicated to graphene applications have led to breakthrough findings, including the thermally-activated double carrier transport in epitaxial graphene on semi-insulating 6H-SiC(0001), a novel explanation of graphene's nonlinear optical properties and a new approach for an optical-quality graphene doping. Other activity is development of metal matrix composites reinforced with different nanocarbon forms (CNT, RGO) using sintering technique (HP, SPS).

#### Newest research results

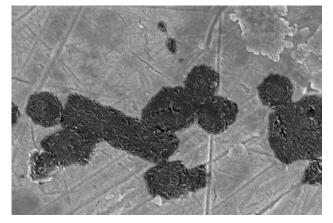
The completion of the long-term research into epitaxial graphene on semi-insulating 4H-SiC(0001) high-temperature properties marks the beginning of the TRL4 stage of ITME's effort to implement graphenebased Hall effect sensors onto the market. Along with the planned scientific publication an industry-supported validation is scheduled for 2019.

#### **Collaboration opportunities for WG5 members**

- Composites based on metal or polymers with graphene
- Heat exchange fluids (and systems) enhanced by graphene



*Fig.21.* Copper-graphene composite microstructure. (Courtesy of ITME)



*Fig.22.* Copper-graphene composite microstructure. (Courtesy of ITME)



Fig.23. Graphene flakes decorated by silver. (Courtesy of ITME)



**Fig.24**. Silver base composite reinforced by carbon nanotubes. (Courtesy of ITME)

#### **Selected papers**

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R. Kalinowski, B. Tomczyk, M. Trzcinska, R. Walkowiak, M. Kazmierczuk, S. Paczkowski, B. Gworek, M. Woluntarski, "Effects of environmental factors on graphene oxide ecotoxicity towards crustacean Daphnia magna", *Desalin. Water Treat.*, 117, 249-256 (2018).

### **KMM PROJECTS**

In several EU projects two or more KMM-VIN members are currently involved as partners, which is a form of integration stemming from their collaboration within KMM-VIN. Some ongoing projects are presented below.

**INTEGRAL** – "Initiative to bring the 2<sup>nd</sup> generation of thermoelectric generators into industrial reality" (2017-20). An innovation action H2020 project coordinated by CEA, with two KMM-VIN partners Fraunhofer IKTS and CIDETEC as project partners.

BIONECA COST Action "Biomaterials and advanced physical techniques for regenerative cardiology and neurology" (2016-2020). This COST action involves three KMM-VIN members: UNIVPM (coordinator), AGH-UST and TUW as project partners.

**MOZART** – "**Mesoporous matrices for localized pHtriggered release of therapeutic ions and drugs**" (2015-2019). This H2020 project is dealing with deployment of nanomaterials in medical applications.

Three KMM-VIN members are involved: POLITO (coordinator), FAU, UCM (project partners).

NICRRE – "Innovative Ni-Cr-Re coatings with enhanced corrosion and erosion resistance for high temperature applications in power generation industry" (2017-2020). This MERA-Net consortium is composed of four KMM-VIN members: ITME (coordinator), IPPT, WUT and IMRSAS as project partners. The first annual meeting of the project was held on 15<sup>th</sup> Nov. 2018 at Warsaw University of Technology (WUT).

## COOPERATION

#### European Technology Platform for Advanced Engineering Materials and Technologies (EuMaT)

KMM-VIN members: Amaya Igartua (TEKNIKER) Pedro Egizabal (TECNALIA), Arnaldo Moreno (ITC) and Michal Basista (IPPT) are members of the EuMaT Steering Committee (SC). Amaya Igartua also serves as the co-secretary of EuMaT ETP. KMM-VIN was represented at the "EUROPE IN MOTION – EuMaT Joint Session" held on 19<sup>th</sup> September 2018 during the EMRS Fall Meeting in Warsaw. A talk on KMM-VIN collaborative research and education activities as an example of effective European networking in the field of advanced materials was given by Michal Basista (KMM-VIN CEO).

## **KMM-VIN RESEARCH FELLOWSHIPS, COURSES and TRAININGS**

One of the ways to boost research integration among KMM-VIN members are the KMM-VIN Research Fellowships offered on a competitive basis for PhD students and early stage researchers (less than 10 years after obtaining the PhD degree). The grants are on average of a 1 month duration supported with a lump sum of 1500 €/month. Both the grantee and the host must be from the KMM-VIN network.

The **11<sup>th</sup> Call** for KMM-VIN Research Fellowships will be opened shortly after the General Assembly 2019 (to be held on February 26, 2019) and will be closed on March 31, 2019. Joint publications by the fellowship holder and the host are expected as a result of the KMM-VIN Research Fellowship within 12 months after the research stay completion. The up-to-date list of published papers resulting from KMM-VIN RF stays is available at http://kmm-vin.eu/fellowships/

# KMM-VIN Specialized Courses offered by Members

KMM-VIN offers customer-tailored Specialized Courses in the fields of materials design, processing technologies, fundamentals of chemical and physical processes, thermodynamics of complex materials, characterization of materials microstructure and properties, modelling of material properties and response to in-service conditions. The courses entail lectures, practices and case studies. They can be delivered at company's premises, at KMM-VIN members' location, or as e-learning.

These courses are designed for experienced staff members, who want to improve their skills in a selected field, but also for non-experienced employees, who would like to gain basic knowledge in the field. The courses are offered on a continuous basis upon individual arrangement with the interested parties. The fees depend on the type and extent of the course and and can be agreed upon with the customers on case by case basis. More information on the courses can be found on KMM-VIN webpage (a detailed booklet to be downloaded) using the following link:

http://kmm-vin.eu/for\_industry/courses\_and\_trainings/

Interested companies can contact the coordinator of KMM-VIN trainings:

Arnaldo Moreno Berto, amoreno@itc.uji.es

#### **Current offer of Specialised Courses**

#### MATERIALS

- Adhesive bonding (LU)
- Biomaterials (FAU)
- Development and applications of microstructured and micro-textured materials (UPM)
- Light alloys and composites (IOD)
- Materials for energy systems and advanced steam power plants (AGH-UST)
- Materials for aeronautics & aerospace (AGH-UST)
- Materials science and technology (POLITO)
- Nanomaterials for biomedical applications (FAU)
- Nickel based superalloys (AGH-UST)
- Sustainable use of materials (LU)

#### **PRODUCTION PROCESSES**

- Automotive body materials (UH)
- Colloidal processing (FAU)
- Electrophoretic deposition (FAU)
- Foundry (TECNALIA)
- Heat treatment of welded joints (ISPL)
- International / European Welding Engineer / Technologist / Specialist (ISPL)
- International welder (ISPL)
- Plastics processing technology (LU)
- Rubber compounding and processing (LU)

#### CHARACTERIZATION TECHNIQUES

- Joining of dissimilar materials and mechanical tests of joints (POLITO)
- Electron microscopy (AGH-UST)
- High-temperature materials characterization in liquid and semi-liquid states (IOD)
- Material characterization via depth sensing indentation tests (IMBAS)
- Microstructural analysis and characterization by microscopy and tomography (AGH-UST and TECNALIA)
- Stress analysis of texturized materials by X-ray diffraction technique (IMIM)
- Testing methods for materials at high temperature and in aggressive environments (IOD)

#### MODELLING TOOLS

- Advanced multiphase and multi-scale material modelling (IMBAS)
- Design and modelling of micro-structured and micro-textured materials (UPM)
- Fracture mechanics of piezoelectric composites (IMBAS)
- Modelling and numerical simulations of multiphase composites (IMBAS)
- Sintering of metal-ceramic composites: modelling of the process, measurement and prediction of residual stresses and properties (IPPT)
- Tissue engineering: biomaterials and cardiovascular systems (BIOIRC)

#### RISK MANAGEMENT

- Risks in Industry (R-TECH)
- Asset/plant Oriented Risk Management (R-TECH)
- Health, Safety, Security and Environment (R-TECH)
- Risk Governance (R-TECH)
- Risk Based Inspection R-TECH)

### PERSONALIA

#### Aldo R. Boccaccini (FAU)

- has been reelected at the Annual General Assembly of the Federation of European Materials Societies (Budapest, July 12, 2018) to the FEMS Executive Committee for another two year term, representing there the German Materials Society (DGM).

- has been listed in the prestigious <u>Highly Cited</u> <u>Researchers 2018</u> list (<u>https://hcr.clarivate.com/</u>), published by Clarivate Analytics. The list contains the world's most influential researchers across 21 scientific disciplines. The 2018 list includes 6000 researchers selected for their exceptional research performance, demonstrated by production of multiple highly cited papers that rank in the top 1% by citations for field and year in Web of Science. Worldwide 208 researchers are included in the materials science category. The methodology used to produce the 2018 list is available on the website of Clarivate Analytics.

- serves as member of the Scientific Committee of the European Congress on Advanced Materials and Processes, EUROMAT 2019, to be held in Stockholm, Sweden, on Sept. 1-5, 2019 and as co-leader of the topic Area "Materials for Healthcare". Amaya Igartua (IK4-TEKNIKER) has been recognised as one of the 21 most outstanding experts working at Basque Technology Centres with the Basque Country Research Prize 2018. Support letter from the KMM-VIN community was acknowledged.

Valentina Casalegno (POLITO), Department of Applied Science and Technology (DISAT), Institute of Materials Physics and Engineering is now Associate Professor at Politecnico di Torino. Her research is focused on joining and mechanical testing of joined components.

**Federico Smeacetto (POLITO)**, Department of Energy (DENERG) is now Associate Professor at Politecnico di Torino. His research is focused on sealants for fuel cells.

Peter Hansen is leaving Haydale at the end of January 2019 to start up his own consultancy business under the name M-Tec Consultants Ltd (plhansen1@outlook.com).

## **KMM-VIN Core Members**

### Institutions

- 1. AGH-UST AGH-University of Science and Technology, Krakow, Poland
- 2. **BioIRC** Bioengineering Research and Developing Centre, Kragujevac, Serbia
- 3. CIDETEC Fundacion CIDETEC, Donostia/SanSebastián, Spain
- 4. CISM Lab Centro Internazionale di Scienze Meccaniche Spin-off, Udine, Italy
- 5. FRAUNHOFER Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V., Germany
- IFAM Fraunhofer Institute for Manufacturing and Advanced Materials, Bremen,
- IFAM-DD Fraunhofer Institute for Manufacturing and Advanced Materials, Dresden,
- IWM Fraunhofer Institute for Mechanics of Materials, Freiburg, Germany
- IKTS Fraunhofer Institute for Ceramic Technologies and Systems, Dresden,
- 6. **HGI** Haydale Graphene Industries PLC, Ammanford, UK
- 7. IK4-TEKNIKER Foundación TEKNIKER, Eibar, Spain
- 8. IMBAS Institute of Mechanics, Bulgarian Academy of Sciences, Sophia, Bulgaria
- 9. IMIM Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Krakow, Poland
- 10. IMRSAS Institute of Materials Research, Slovak Academy of Sciences, Kosice, Slovakia
- 11. INTA Instituto Nacional de Técnica Aeroespacial, Torrejón de Ardoz, Spain
- 12. IOD Foundry Research Institute, Krakow, Poland
- 13. IPM Institute of Physics of Materials, Brno, Czech Republic
- 14. IPPT Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw
- 15. ITC Instituto de Tecnología Cerámica AICE, Castellón, Spain
- 16. **ITME** Institute of Electronic Materials Technology, Warsaw, Poland
- 17. MCL Werkstoff-Kompetenzzentrum-Leoben Forschungsgesellschaft m.b.H. (Materials Centre Leoben), Leoben, Austria
- 18. **POLITO** Politecnico di Torino, Torino, Italy
- 19. R-TECH Steinbeis Advanced Risk Technologies GmbH, Stuttgart, Germany
- 20. TECNALIA Fundación Tecnalia, Donostia-San Sebastian, Spain
- 21. TUD Technische Universität Darmstadt, Darmstadt, Germany
- 22. TUG Graz University of Technology, Graz, Austria
- 23. TUW Technische Universität Wien, Wien, Austria
- 24. UH University of Hertfordshire, Hatfield, Herts, UK
- 25. UNIVPM Università Politecnica delle Marche, Ancona, Italy
- 26. UPM Universidad Politécnica de Madrid, Madrid, Spain
- 27. WRUT Wroclaw University of Technology, Wroclaw, Poland
- 28. WUT Warsaw University of Technology, Warsaw, Poland

#### **Individual members**

- 1. Katarzyna Pietrzak Warsaw, Poland
- 2. Michał Basista Warsaw, Poland
- 3. Krzysztof Doliński Warsaw, Poland
- 4. Michał Urzynicok Koszęcin, Poland

## **KMM-VIN Associate Members**

### Institutions

1.	ALSTOM	Alstom Power Ltd., Rugby, UK
2.	BEG	Böhler Edelstahl GmbH & Co KG, Kapfenberg, Austria
3.	BSGA	Böhler Schweißtechnik Austria GmbH, Kapfenberg, Austria
4.	CSM	Centro Sviluppo Materiali S.p.A., Rome, Italy
5.	UNIPER	Uniper Technologies Limited., Coventry, UK
6.	ETE	Energietechnik Essen GmbH, Essen, Germany
7.	EU-VRi	European Virtual Institute for Integrated Risk Management, Stuttgart, Germany
8.	FAU	Friedrich-Alexander Universität Erlangen-Nürnberg, Germany
9.	GSCLtd	Goodwin Steel Castings Ltd, Hanley, UK
10	IMSETHC	Institute of Metal Science, Equipment and Technologies with HydroAerodynamics Centre of the Bulgarian Academy of Sciences, Sofia, Bulgaria
11	ISPL	Instytut Spawalnictwa, Gliwice, Poland
12	.LU	Loughborough University, Loughborough, UK
13	.MPA	Materialprüfungsanstalt Universität Stuttgart, Germany
14	NOMASICO	Nomasico Ltd, Nikosia, Cyprus
15	NUIG	National University of Ireland, Galway, Ireland
16	.NTUA	National Technical University of Athens, Athens, Greece
17	SIEMENS	Siemens AG, München, Germany
18	.SSF	Saarschmiede GmbH Freiformschmiede, Völklingen, Germany
19	.SWG	Schmiedewerke Gröditz GmbH, Gröditz, Germany
20	TUC	Chemnitz University of Technology, Chemnitz, Germany
21	TUBAF	TU Bergakademie Freiberg, Germany
22	.UCM	Universidad Complutense de Madrid, Spain
23	.UL	University of Limerick, Limerick, Ireland
24	UNIPAD	Università degli Studi di Padova, Padova, Italy
25	VAGL	Voestalpine Giesserei Linz GmbH, Linz, Austria
26	.V&MD	Vallourec & Mannesmann Tubes, V&M Deutschland GmbH, Düsseldorf, Germany
27	.VTT	VTT Technical Research Centre of Finland, Espoo, Finland
28	.VZU	Výzkumný a zkušební ústav Plzeň s.r.o., Plzeň, Czech Republic

### **Individual members**

1. Fabrizio Valenza Genova, Italy

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