

EUROPEAN VIRTUAL INSTITUTE ON KNOWLEDGE-BASED MULTIFUNCTIONAL MATERIALS AISBL

KMM-VIN Newsletter

Issue 25, Winter 2021



EDITORIAL

The European Virtual Institute on Knowledge-based Multifunctional Materials (KMM-VIN) is an international nonprofit association (AISBL) based on Belgian law with the registered seat in Brussels and a branch in Warsaw, Poland. It was established on 13th of March 2007 as the major deliverable of the Network of Excellence project KMM-NoE of the EU 6th Framework Programme.

KMM-VIN is a self-sustainable European network of universities, R&D institutes, and industrial companies, which was created to facilitate cooperative research among members on advanced structural and multifunctional materials. Research activities of the KMM-VIN members development include materials and processing. characterisation, and modelling. These activities are being conducted within five Working Groups (WGs): WG1. Materials for Transport, WG2. Materials for Energy, WG3. Biomaterials, WG4. Materials Modelling and Simulation, WG5. Graphene/2D Materials. The industry sectors targeted primarily by KMM-VIN are Transport, Energy and Healthcare.

Integration of research activities is supported through the KMM-VIN Research Fellowship programme for PhD students and young researchers.

Thematic scope of KMM-VIN R&D encompasses:

- metals and alloys
- advanced ceramics
- intermetallics and shape memory alloys
- composites of metal, ceramic, or polymer matrices
- coatings, layered materials, surface modification
- biomaterials and bioinspired materials
- graphene and 2D materials
- joining of advanced materials
- modelling and simulation of materials.

Besides the networking activities, KMM-VIN offers services for external customers, such as integrated R&D solutions for research problems, access to laboratory equipment, database of KMM-VIN materials and members' expertise, customised Specialised Courses, participation in KMM-VIN Industrial Workshops.

The KMM-VIN Newsletter has a more expanded form than a typical newsletter because it contains detailed information about the research being conducted within KMM-VIN

In this Issue:

Editorial	1
Latest News	2
What's new in Working Groups	3
EU Projects and Cooperation	22
Research Fellowships, Courses and Trainings	23
Personalia	25
KMM-VIN Members	26

Page

Working Groups. Present issue, which is the 25th in the Newsletter history, summarises the main KMM-VIN activities in the second half of 2021 and plans for the near future.

The 2021 was the second year that KMM-VIN networking activities had to be conducted in extraordinary conditions dictated by the COVID-19 pandemic. This had an impact on the KMM-VIN annual meetings in Brussels, Industrial Workshops, and the Research Fellowships programme. Neverthelss, as many other R&D associations the main activites have not been interrupted, the virtual nature of KMM-VIN being of much help in such circumstances.

It should not go unnoticed that March 2022 marks the 15th anniversary of KMM-VIN. Created and being managed by researchers for researchers, KMM-VIN has served its members over all these years offering a stable and flexible framework for cooperation and integration in advanced materials R&D.

The "News from the Working Groups" columns report on the ongoing collaborative research carried out within the WGs. Besides joint research activities, this column contains news from individual members within the WG's thematic scope.

In "European Projects & Cooperation with Platforms" a concise information is given on running EU projects in which two or more KMM-VIN members are involved, Also, recent developments in the European Technology Platform on Advanced Engineering Materials and Technologies (EuMaT ETP) are highlighted.

Within the "Research Fellowships, Courses and Trainings" column the results of the 2021 call for KMM-VIN Research Fellowships are recalled and information about the upcoming call 2022 is provided. Also, a selection of Specialised Courses offered by KMM-VIN members for external clients is presented.

In "Personalia", professional achievements of KMM-VIN members, such as scientific awards, recognitions, and promotion to high positions are reported.

The up-to-date register of the KMM-VIN members and the contact details of the KMM-VIN office are provided at the end of the Newsletter. More information about KMM-VIN can be found on the association's website: <u>http://www.kmm-vin.eu</u>

Katarzyna Kowalczyk-Gajewska, Editor

LATEST NEWS

PARTNERSHIP

Currently, the KMM-VIN association is composed of 57 members, of whom 32 are core and 25 associate members, from 12 European States. Among them 51 are institutions (research centres, universities, large companies and SMEs) and 6 are individual members.

As the content of this Newsletter will show, research collaboration in the second half of 2021 was going on within the Working Groups. New results have been achieved and published jointly with other KMM-VIN partners. To this end, the prominent role of KMM-VIN Research Fellowships for young researches and PhD students in collaborative research is to be highlighted.

KMM-VIN welcomes new members from academia and industry, who are interested in cooperative research within the thematic scopes of the five WGs. Applications for membership are being collected on a continuous basis but the final decision on accession is taken by the General Assembly at the annual meeting in February each year. Information on the accession procedure can be found on <u>http://kmm-vin.eu</u> (under Network). Specific questions can be directed to <u>Michal.Basista@kmm-vin.eu</u>

FORTHCOMING EVENTS

The General Assembly 2022 and the annual meetings of the Working Groups will be held <u>online</u> on February 21-22-23, 2022. Since the statutory three-year term is coming to an end, the General Assembly will, among other items on the agenda, elect the governing bodies and WG coordinators for the next term. The series of annual meetings 2022 to be held using the MS Teams platform, is scheduled as follows:

- WG2. Materials for Energy (EMEP): Feb. 21, 2022 (Monday) 13:00-17:00
- WG2. Materials for Energy (non-EMEP): Feb. 22, 2022 (Tuesday) 9:00-11:00

- KMM-VIN AISBL General Assembly: Feb. 22, 2022 (Tuesday) 13:00-17:30
- WG1. Materials for Transport: Feb. 23, 2022 (Wednesday) 9:00-10:30
- WG3. Biomaterials: Feb. 23, 2022 (Wednesday) 10:30-11:45
- WG4. Materials Modelling and Simulation: Feb. 23, 2022 (Wednesday) 12:00-13:15
- WG5. Graphene/2D materials: Feb. 23, 2022 (Wednesday) 13:15-14:30.

The EMEP Management Committee (**EMEP MC**) will meet online on Feb. 21, 2022 (Monday),10:30-12:00.

The KMM-VIN Governing Committee and Board of Directors (**GC & BoD**) online meeting is scheduled on Feb. 22, 2022 (Tuesday), 10:00-12:00.

The agendas with the links to the online meetings will follow in due time. We do hope that the COVID-19 pandemic will finally be over so that our annual faceto-face meetings will again be possible in Brussels.

9th **KMM-VIN Industrial Workshop (IW9)** on "Design and modeling of innovative biomaterials and bioinspired materials for industrial applications" – scheduled on January 25-26, 2022 in Vienna had to be moved again to another, as yet unkown date because of the new pandemic outbreak. The workshop will be held in a hybrid format, in-person and online (see "News from WG4" for more details).

10th **KMM-VIN Industrial Workshop (IW10)** on "Advanced Materials for Energy: challenges and opportunities" in Turin – scheduled on May 10-11, 2022 will, for the same reason as IW9, quite likely be moved to another date. We shall announce the new date and the workshop format as soon as possible.

WHAT'S NEW IN WORKING GROUPS

The Working Groups (WGs) constitute the internal research structure of KMM-VIN. Collaborative research supported by the KMM-VIN Research Fellowship programme, preparation of project proposals, and organisation of Industrial Workshops are currently the core activities carried out within the Working Groups. Since the internal collaborative projects within WGs receive no funding from KMM-VIN budget they are being formed bottom-up by the WG members without any imposed research agenda or work model from KMM-VIN. Therefore, the results of the collaborative work within WGs are dependent on the members' commitment and the leadership of WG coordinators. In fact, each WG operates in a slightly different way with different intensity.

At present KMM-VIN is composed of five WGs, as listed below.

WG1. Materials for Transport

Coordinators:

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

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

WG2. Materials for Energy

Coordinators:

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

WG3. Biomaterials

Coordinators:

Aldo R. Boccaccini, Friedrich-Alexander Universität (FAU), Erlangen-Nürnberg, Germany Christian Hellmich, Technische Universität Wien (TUW), Austria

WG4. Materials Modelling and Simulation

Coordinators:

Katarzyna Kowalczyk-Gajewska, Institute of Fundamental Technological Research of Polish Academy of Sciences (IPPT), Warsaw, Poland

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

WG5. Graphene/2D Materials

Coordinators:

Peter Hansen, St Albans, United Kingdom Antonios Kanellopoulos, University of Hertfordshire (UH), Hatfield, United Kingdom

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

New Working Groups can be formed on themes not covered by the existing WGs, if requested by a group of minimum 7 members.

NEWS FROM WG1: MATERIALS FOR TRANSPORT

What's new in WG1

During the second part of the year the collaborative work in WG1 has been focused on the continuation of long-standing collaborations and the activity around contacts derived from KMM-VIN research fellowships related to "materials for transport applications". New ideas have been discussed among different groups belonging to WG1 but it is true that the uncertain situation related to the evolution of the COVID-19 persists and there are still limitations for collaboration and exchange of researchers among participants of the group. An extra effort will be made to boost such exchange of ideas in the following months and during the GA meeting 2022 so that they may crystallize into new collaborative projects.

The internal collaborative project led by IPPT to develop aluminium-alumina composites reached its end in 2021 but IPPT intends to start a new collaboration on the evaluation of thermal properties and thermal residual stresses of aluminum-matrix FGMs together with Fraunhofer IFAM Dresden.

Likewise, the collaboration between IMIM and TECNALIA on the development and comparison of the microstructure and properties of aluminium based nanoreinforced samples has finished in its experimental part. Part of the results of the work were presented in the "Materialen zientzia eta teknologia MZTV" congress co-organised by Tecnalia in Bilbao in November 2021 with a presentation entitled "Automobilgintza sektorearen errekerimenduak asetzeko hedatu diren aluminio aleazio indartuen garapena (the congress is entirely held in Basque language) and co-authored by Ane Jimenez, Pedro Egizabal, Wojciech Maziarz and Anna Wojcik (Fig. 1). There is a pending publication where the main results of this project will be summarised and submitted to a scientific journal in 2022.

In the following months the efforts will be made to establish the action plan of WG1 in 2022 and prepare for the General Assembly to be held online in February 2022.

Internal collaborative projects

1. TECNALIA – IMIM collaboration on the development of aluminium based nanocomposites

The last works related to the internal collaborative project have been focused on some final castings of 0.1 wt.% nTiC particulates and AlSi7Mg0.6 alloy into a new investment casting project. Owing to the courtesy of the investment casting company EIPC, they will now thermally treat, machine and test the components and specimens and compare the results with non nanoreinforced samples.



Fig. 1. Slides presented on the MZT congress showing some results obtained in the collaborative project (courtesy of TECNALIA).

Partial conclusions of the work are that nanoparticulates seem to provide an interesting increase in ductility without negatively affecting the rest of properties. Tensile strength and stress also show a slight increase even though the obtained results are far from those reflected in literature by other groups working with nanoreinforced aluminium alloys. It is deemed that the lack of outstanding results in terms of mechanical resistance may be related to Ti and C diffusion related phenomena with the aluminium melt.

From IMRSAS to IMIM

(KMM-VIN Research Fellowship, call 2019)

High-entropy cabrides

In July 2021 International Journal of Refractory Metals and Hard Materials published a study on hardness anisotropy and active slip systems in a (Hf-Ta-Zr-Nb)C high-entropy carbide during nanoindentation [1]. The publication resulted from the cooperation in the framework of the KMM-VIN Research Fellowship program. Tamás Csanádi (IMRSAS), visited the Institute of Metallurgy and Materials Science, Polish Academy of Sciences (IMIM) in Krakow to extend the research in the field of high-entropy cabrides.

High-entropy carbides were between first synthesized by M.J. Reece's group at Queen Mary University of London and their micromechanical properties were investigated in collaboration with Jan Dusza's group at Institute of Materials Research of Slovak Academy of Sciences (IMRSAS) in 2018. The main drive of the research on high-entropy carbides is the potential to find new stable multi-principal element carbide systems that possess higher hardness, strength or improved deformability than the constituent monocarbides. In previous studies, high-entropy carbides subjected to various macro-, micro- and nanomechanical testing, including nanoindentation, micropillar compression, creep and flexural strength measurements both at room and elevated

temperatures. These works have revealed that the hardness and yield strength of high-entropy carbides can go beyond any rules of mixtures for the corresponding carbides and their strengthening is attributed to their enhanced Peierls stresses caused by atomic randomness at the core of their dislocations. To develop novel high-entropy refractory carbides with improved mechanical properties, it is important to understand the factors that control their deformability/plasticity at the micro and nanoscales. Thus, the knowledge of slip systems is essential for the prediction of both hardness/yield strength, their anisotropy and deformability. Active slip systems have been reported for a (Hf-Ta-Zr-Nb)C high-entropy carbide during micropillar compression and hightemperature creep tests. The aim of the collaboration between IMRSAS and IMIM was to extend previous study and determine the active slip during nanoindentation using transmission electron microscopy (TEM) and to correlate the results with the measured hardness anisotropy.

Experiments were conducted on a polycrystalline, single-phase, spark plasma sintered high-entropy carbide (Hf-Ta-Zr-Nb)C sample. To study the anisotropic deformation of grains, nanoindentation was carried out at IMRSAS on a selected area of the EBSD image, covering large grains of {001}, {101} and {111} orientations, Fig. 2. It was found that the hardness anisotropy is small with average values of $H{001} = 35.5 \pm 0.4$ GPa, $H{101} = 37.8 \pm 0.6$ GPa and $H{111} = 37.9 \pm 0.8$ GPa corresponding to the lowindex orientations investigated. The average indentation modulus for all the three low-index orientations was found to be equal, with a value of M = 605 ± 8 GPa.

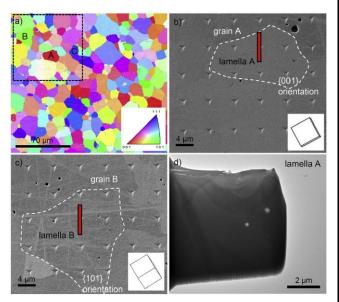


Fig. 2. Microstructure of the (Hf-Ta-Zr-Nb)C high-entropy carbide and the area selected for nanoindentation on the EBSD map, indicating grains A, B and C selected for TEM lamella preparation (courtesy of IMRSAS).

To investigate the deformation zones under the indents, thin lamellas were extracted from these grains using a focused ion beam (FIB) technique and were studied by TEM at IMIM, Fig. 3.

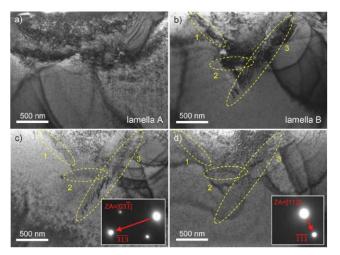


Fig. 3. Bright-field TEM images recorded in zone axes on a) lamella A and b) lamella B, exhibiting different arrangement of dislocations under the indents (courtesy of IMRSAS).

The dislocations were hardly visible because of the heavily deformed zone of the indents. But the careful analyses of the TEM images revealed that the deformation under the indents took place by dislocation slip without the presence of a phase transformation or cracking. A TEM-based trace analysis confirmed that dislocations are active on the $\langle 110 \rangle \{111\}$ type slip systems during nanoindentation of grains of {001} orientation (lamela A). Similar trace analysis was carried out on lamella B, which was extracted from an indented grain of {101} orientation, resulting in the operation of the $\langle 110 \rangle \{110\}$ type slip systems. A transition from the $\langle 110 \rangle \{110\}$ brittle' system to the $\langle 110 \rangle \{111\}$ ductile one was reported for group V transitional metal monocarbides in the literature and was accompanied by a 'softening'of these materials, exhibiting a decrease of their hardness. Based on these reports, it can be inferred that the hardness anisotropy of the (Hf-Ta-Zr-Nb)C high-entropy carbide is attributed to the different slip systems operating.

More details can be found in:

[1] Csanádi, T., Girman, V., Maj, Ł., Morgiel, J., Reece, M. J., Dusza, J. Hardness anisotropy and active slip systems in a (Hf-Ta-Zr-Nb)C high-entropy carbide during nanoindentation, *Int J Refract Hard*, 2021, 100, 105646-1-7.

News from IKTS

Hard, tough, electrically conductive ceramics based on boron carbide

Boron carbide (B₄C) is an advanced non-oxide ceramic and possesses a set of outstanding properties such as high melting point, high mechanical strength, high thermal stability, extreme hardness and high neutron absorption cross section. These outstanding properties make B₄C for various industrial applications attractive, including refractory devices, wear resistance enhancement, hard material polishing and reactor control in nuclear technology. In particular, the

combination of its extraordinary high stiffness (E = 450 GPa) and low specific weight (2.52 g/cm³) produces an outstanding stiffness/weight ratio compared to metals and other ceramic materials like Al₂O₃, SiC and Si₃N₄. Consequently, boron carbide is an ideal ceramic material for minimizing the weight of structural elements in lightweight applications. Nevertheless, one obstacle for its wide application is the low fracture toughness KIC, typically in the range of 2 - 3 MPa m^{1/2}. A strategy increasing K_{IC} of boron carbide is to produce composites by addition of suitable secondary phases to B₄C matrix.

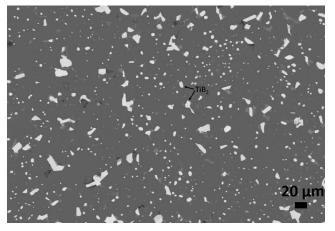


Fig. 4. SEM micrograph of B4C-TiB2 composite (courtesy of Fraunhofer IKTS).

Fraunhofer IKTS fabricated B₄C-TiB₂ composites (Fig. 4) via reactive pressureless sintering of B₄C and TiC powder mixtures. This process is more economic than commonly used hot pressing procedure. But the desired properties can be achieved as well. The effect of in-situ formed TiB₂ phase on sintering behavior, mechanical properties and electrical conductivity of boron carbide were studied. It is shown that dense B₄C-TiB₂ composite with high fracture toughness up to 4.7 MPa m^{1/2} and in combination with high hardness could be achieved. In addition, the electrical conductivity of the materials can be modified from between semiconductive and metallic behavior by the TiB₂ content. B₄C-TiB₂ composites offer opportunities for new applications, like lightweight constructive component, ballistic protection and high temperature electrode with the improved properties. Particularly the combination with light weight metal alloys as aluminium or titanium alloys could help for the development of new lightweight constructional concepts. Cooperation partners for this topic are welcome.

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News from AGH-UST and Ł-KIT

Single-step fabrication of multiphase Mo-Si-B alloys using liquid-phase processing

Boron enhanced multiphase molybdenum silicides are ultra-high temperature materials that can provide a new level of durability and heat-resistance exceeding superalloys. Thus, they are a promising candidate for applications in turbines used in energy systems and aeronautics. Currently, the major drawback of these materials is associated with complex processing routes required to obtain desired microstructure.

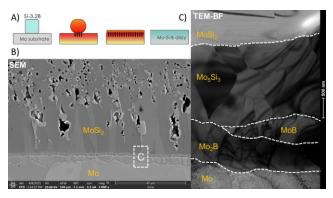


Fig. 5. a) A schematic drawing of the applied single-step processing; b) SEM-SE image of a cross-sectioned Si3.2B/Mo sample; c) TEM-BF image highlighting multiphase interlayer formed between outermost MoSi₂ layer and Mo substrate (courtesy of AGH-UST).

This study explores single-step liquid-phase assisted fabrication of a multiphase material from a Mo-Si-B system under pressure-less conditions. For this purpose, binary silicon-boron alloy (Si-3.2B wt.%) was subjected to contact heating with a polycrystalline molybdenum substrate at a temperature close to 1400°C. A schematic representation of the fabrication process is shown in Fig. 5a.

Further microscopic observations revealed the formation of a layered structure, where the outermost, porous layer of MoSi₂ was about 80 µm thick (Fig. 5b). Between the MoSi₂ layer and Mo substrate, a multiphase interlayer was formed with a thickness of about 5 µm. Detailed phase identification of the interlayer was performed by TEM (Fig. 5c). At least three different Mo-rich phases were identified. First, from the silicon-boron droplet side, the Mo₅Si₃ layer was formed. Next, two B-rich compounds were identified, namely MoB and Mo₂B, adjacent to the Mo substrate. The presence of B-rich layers further from the droplet can be justified by greater diffusion of boron than silicon.

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News from IMRSAS

Recent activities

Since 2015, the Institute of Materials Research of Slovak Academy of Sciences (IMRSAS) is a member of Research centre for progressive materials and technologies PROMATECH

(www.promatech.sk/?lang=en)

The Center was founded by implementing a project (ITMS 26220220186) on the basis of the support from the Operational Program "Research and Development" financed through European Regional Development Fund. The laboratories of the Centre

were equipped with the top-level infrastructure for preparation of advanced materials (spark plasma sintering, electrospinning technology, PVD techniques HiPIMS and HiTUS) microstructure analyses (SEM, TEM, Raman spectroscopy, FIB, AFM) and determination of mechanical properties (hardness, elastic modulus, tribological characteristics at room and elevated temperatures). Mechanical testing can be performed not only at the macro-, but using micropillar and cantilever tests also at the micro- and nano-level.

According to the PROMATECH aims, the IMRSAS' researchers use the modern infrastructure of the Centre for implementation of the cooperative projects. For instance, in previous years, IMRSAS has been involved in realization of three common projects with KMM-VIN core members, from M-ERA.NET Call 2016 project NiCrRE. Innovative Ni-Cr-Re coatings with enhanced corrosion and erosion resistance for high temperature applications in power generation industry, coordinated by Institute of Electronic Materials Technology (now Ł-IMiF), from M-ERA.NET Call 2017 project DURACER, Durable ceramic composites with superhard particles for wear-resistant cutting tools, headed by Łukasiewicz Research Network – Krakow Institute of Technology (Ł-KIT) and FLAG-ERA JTC 2017 project CERANEA, Multifunctional Ceramic/Graphene Thick Coatings for New Emerging Applications, led by Hungarian Academy of Sciences Centre for Energy Research, with the participation of Fraunhofer Institute for Ceramic Technologies and Systems. Currently, IMRSAS collaborates on the preparation of the proposal in M-ERA.NET Call 2021 DuplexCER, High performance duplex ceramics for efficient machining of nickel superalloys, coordinated by Łukasiewicz Research Network – Krakow Institute of Technology.

Selected publication

Micro/nano indentation testing of spark plasma sintered $Al_2O_3 + ZrO_2 + cBN$ ceramics.

The aim of the research presented in the recent paper [1] was the processing of $Al_2O_3 + ZrO_2 + cBN$ composites with an optimized processing route and to investigate the influence of cBN addition on their microstructure characteristics, microhardness and crack-extension resistance. The other aim was to study the nanohardness of the $Al_2O_3 + ZrO_2$ matrix and cBN grains (Fig. 6).

1. Al_2O_3 + 30 vol% ZrO₂ matrix composites with 20 and 30 vol% cBN have been prepared with an optimized processing route, using spark plasma sintering (SPS) at temperatures of 1400°C and 1250°C.

2. The Al₂O₃ + ZrO₂ matrix consists of alumina and zirconia grains with a grain diameter of approximately 220 nm and 160 nm with approximately 1.9 μ m cBN grains in the Al₂O₃ + ZrO2 + cBN composites.

3. The microhardness of the $AI_2O_3 + ZrO_2 + cBN$ composites are slightly increasing with cBN addition

from 16.2 GPa to 17.1 GPa and the crack-extension resistance from 3.72 MPa m^{1/2} to 4.29 MPa m^{1/2} due to the toughening mechanisms in the form of crack deflection, crack branching and crack bridging.

4. The nanohardness and indentation modulus of the matrix are approximately 30 GPa and 420 GPa and the cBN grains 70 GPa and 777 GPa, respectively.

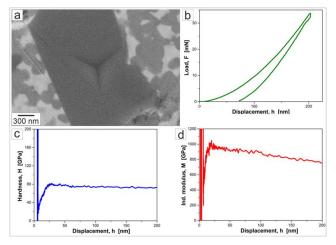


Fig. 6. Characteristics of nanoindentation of cBN grain in $Al_2O_3+ZrO_2+30\%$ cBN composite, imprint (a), load – displacement (b), hardness – displacement (c) and indentation – displacement curves (courtesy of IMRSAS).

[1] Sedlák, R., Ivor, M., Klimczyk, P., Wyzga, P., Podsiadlo, M., Vojtko, M., Dusza, J., Micro/nano indentation testing of spark plasma sintered Al₂O₃+ZrO₂+cBN ceramics; *Ceramics*, 2021, 4, 40–53.

https://doi.org/10.3390/ceramics4010004

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News from IMIM

Development of high-efficiency waste-free technology for the production of soft magnetic nanocomposites for high-frequency high-power conversion.

New possibilities of microstructural investigations at IMIM.

In the middle of 2021, the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences (IMIM) in cooperation with other Polish research groups and companies, completed a project entitled "Development of high-efficiency waste-free technology for the production of soft magnetic nanocomposites for high-frequency high-power conversion" (grant no.: TECHMATSTRATEG/347200/11/NCBR/2017), which was financed by the Polish National Centre for Research and Development (NCBiR). The aim of this project was to develop high-efficiency and waste-free technology for the production of high-inductance and low-loss soft magnetic nanocomposites for highfrequency high-power conversion. These types of materials are intended for electrical devices used in transport applications, mainly for cars and trains. IMIM's activity in the project concerned detailed microstructural studies of rapidly crystalized and heat treated amorphous and nanocrystalline Fe-base ribbons characterized by soft magnetic properties. During the project, a modern, high resolution Titan Themis 200 kV Cs corrected G3 Thermo Fisher Scientific transmission electron microscope S(T)EM (Fig. 7) was installed at IMMS PAS, and employed for analysis of structure, microstructure, magnetic domains and magnetic induction vector distribution through Lorentz microscopy and differential phase contrast (DPC-STEM) imaging.



SPECIFICATIONS (Highlights):

Accelerating voltages: 80 and 200 kV

Ultra stable, high brightness X-FEG (brightness: 1.8 x 10⁹ A/cm²/sr)
X-FEG probe current: 0.9 nA in 0.2 nm spot and 14 nA in 1 nm spot
Cs DCOR probe corrector for sub- Å resolution in STEM mode
TEM point resolution and information limit: < 0.11 nm
STEM resolution: 70 pm (Cs corrected)
EDS energy resolution (Windowless Super-X EDS detector system

•EDS energy resolution (Windowless Super-X EDS detector system with output count rate up to 200 kcps):

- ≤ 136 eV for Mn-Kα and 10 kcps (output)
- ≤ 140 eV for Mn-Kα and 100 kcps (output)

Detectors:

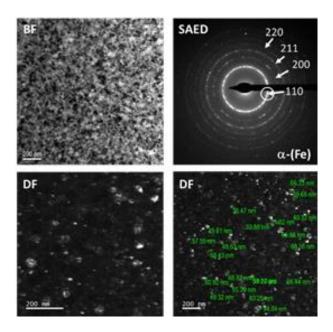
- High angle angular dark field (HAADF) detector for STEM
- 4 on-axis ADF/BF detectors including 4 Quadrant ADF for differential phase contrast (DPC) imaging
- Ceta 16M CMOS camera

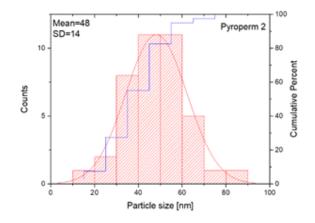
•Specimen Holders:

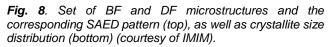
- FEI CompuStage single tilt holder
- FEI CompuStage high-visibility, low-background double tilt holder (±35° tilt range for alpha and ±30° tilt range for beta)
- Fischione tomography holder model 2020 (up to ±70°)

Fig. 7. Titan Themis Cs corrected 200 kV G3 Thermo Fisher Scientific transmission electron microscope S(T)EM installed IMIM in Krakow with the listed specs (courtesy of IMIM).

Exemplary results concerning microstructure analysis and including bright field (BF), dark field (DF) imaging and the selected area diffraction pattern (SAED) (left) al observations, taken at IMIM, on rapidly solidified amorphous Fe_{84.5}Nb₅B_{8.5}P₂ ribbons are shown in Fig. 8. The ribbons were subjected to 30 min heat treatment at 530°C under a magnetic field of 85 kA/m. Crystallite size distribution was evaluated using DF images, taken from the parts of the diffraction ring corresponding to the $(110)_{\alpha-Fe}$ planes. One can see that the heat treatment caused crystallization of amorphous ribbon to nanocrystalline α -Fe solid solution with the average crystallite size of about 48 nm and normal size distribution (Fig. 8, bottom image).







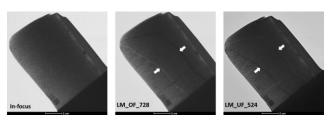


Fig. 9. Result of magnetic domain observations obtained by use of TEM Lorentz microscopy (courtesy of IMIM).

Fig. 9 presents results of magnetic domain observations by TEM Lorentz microscopy. There are three images recorded at different focus values. Defocused imaging was performed according to the classic Fresnel imaging mode. In principle, in Fresnel defocus, it is assumed that when the parallel electron beam passes through the magnetic domain region, the Lorentz force leads to the deflection of the electrons and the diffraction spot splits into two following the right-hand rule. The deflecting electrons are focused in the final image plane and hence no magnetic contrast appears in the in-focus conditions. However, when the Lorentz TEM is in the defocus (over/under) the contrast appears. In over-focus condition, the electron deflection induces a decreased intensity contrast because the electrons are deflected away from the domain wall. It results in the appearance of dark contrast lines in the domain wall regions. Similarly, bright contrast lines appear in under-focused conditions due to the increased electron density caused by the converged electrons. In this sense, the inversion of the magnetic contrast at the domain walls is observed between the over- and under-focused images. In our case, magnetic domain observations were performed on Focused Ion Beam prepared thin lamellae with average thickness of less than 200 nm extracted from nanocrystalline Fe_{84.5}Nb₅B_{8.5}P₂ ribbon after heat treatment at magnetic field. Opposite magnetized 180° domains with a width of about 4 μ m are visible, proving the high magneto-crystalline anisotropy of this ribbon.

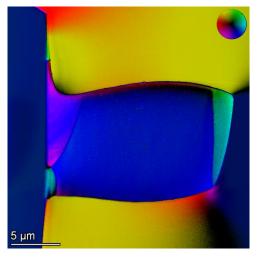


Fig. 10. Example LM STEM DPC image taken from the thin lamella showing magnetic vector distribution according the colour wheel - top inset (courtesy of IMIM).

The results of Lorentz microscopy observation are well correlated with low mag stem LM-STEM DPC (Fig. 10) showing magnetic domain arrangements and magnetic induction vector distribution in the thin lamellae.

Overall, the Titan-Themis S(TEM) permits complex and thorough microstructure analysis and direct correlation between microstructure and magnetic domain distribution. Partners interested in our work or collaboration please do not hesitate to contact any of the below listed researchers from our Electron Microscopy Laboratory group.

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News from IMBAS

Institute of Mechanics (IMBAS) and Institute of Metal Science, Equipment and Technologies (IMSETHC) with Hydro- and Aerodynamics Centre of the Bulgarian Academy of Sciences are participants in project BG05M2OP001-1.001-0008 "National Centre for Mechatronics and clean technology", funded by the Operational Programme Science and Education for Smart Growth and co-financed by the European Union through the European Regional Development Fund. Within the scope of the project joint Laboratory for mechanical tests and express diagnostics has been established. The Laboratory is equipped with Split Hopkinson bar for analysis of material behaviour at high deformation rates (Fig. 11) and Complex for determination of mechanical characteristics and crack resistance in static and dynamic conditions in wide temperature range (Fig. 12) for performing a comprehensive structural analysis that consists of static, dynamic, fatigue and fracture of materials and structures, which must perform a given mission in a fluctuating load environment, including those in the field of transportation and energy.



Fig. 11. Split Hopkinson (Kolsky) Pressure Bar (courtesy of IMBAS).



Fig. 12. Complex for determination of mechanical characteristics and crack resistance in static and dynamic conditions in wide temperature range (courtesy of IMBAS).

The laboratory equipment will be involved in a new project "Fabrication of new type of self-lubricating antifriction metal matrix composite materials with improved mechanical and tribological properties", funded by National Science Fund under "Competition for financial support for basic research projects– 2021", which is in the process of signing. The project will be focused in developing cost-effective and ecofriendly technologies for creating a new type of antifriction metal matrix composite materials, which will be characterized in order to study the influence of technological parameters on the microstructure of metal composites and will establish the relationship between the microstructure of the metal matrix composites and their functional characteristics.

The new type of antifriction metal matrix composite materials will combine the enhanced mechanical properties of aluminium metal matrix composites (AMMCs) and the excellent tribological properties of a babbitt composite. Aluminium alloys will be used as matrix materials and Al₂O₃ and SiC particles will be used as reinforcing phases of the AI composite. The babbitt composite will be reinforced with particles of SiC. The scientific investigations will be focused on the development of technology for creating a highly porous AMMC with open porosity, having low density, high load capacity, high thermal conductivity, and excellent corrosion resistance which will be infiltrated with antifriction composite material in order to reduce the friction coefficient and improve the wear resistance of functional friction elements in engineering facilities. The obtained antifriction metal matrix composite materials will be microstructurally, mechanically, and tribologically characterized.

Since the European Commission adopted its longterm strategy for a prosperous, modern, competitive, climate-neutral economy by 2050, and the development of composite materials has met European targets and values for achieving significant reductions in carbon (CO₂) emissions. The development of highly porous AMMCs with excellent mechanical properties through environmentally friendly technology contributes to the reduction of CO2 emissions thanks to the reduced weight of the composite. The production of a metal antifriction composite with a low friction coefficient and excellent wear-resistant characteristics would also contribute to overcoming various socio-economic problems and financial losses caused by a significant amount of consumed energy due to friction processes, that occur because of the rapid wear of various products and components, especially in the transport and energy sectors.

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News from IPPT

ALU-FGM project

As a follow up of the alumina-aluminum project on graded composites (Al₂O₃/AlSi₁₂ FGM), the IPPT group is currently conducting research on SiC/AlSi₁₂ FGM fabricated by powder metallurgy. The general scientific goal of the ALU-FGM project supported by the National Science Centre is to explore, through a carefully designed experimental program and microstructure-based numerical modeling, the impact of material microstructure on: (i) processing-induced thermal residual stresses, (ii) selected thermal properties, and (iii) mechanical properties of these two AlSi₁₂-matrix graded composites. Besides producing a new knowledge, a comparative analysis of the mechanical and thermal properties of the Al₂O₃/AlSi₁₂ and SiC/AlSi12 FGMs can be of interest for the automotive industry as these two composites are competing materials for modern brake disks. The studied FGMs are composed of several layers with different ceramic volume fractions (a stepwise gradient). The effect of microstructure on the target properties is represented by: (i) using two different types of ceramic reinforcement Al₂O₃ and SiC, (ii) optimizing the process parameters (e.g. milling/pause times and speed), (iii) varying the volume fraction of ceramic reinforcement in composite layers, (iv) using different particle sizes of the ceramic powders, (v) employing two variants of sintering techniques (HP and SPS).

One specific project task is devoted to the evaluation of thermal properties (thermal conductivity, thermal expansion) and thermal residual stresses of the fabricated FGMs, with a particular focus on the effects of microstructure and composition gradient. Since the thickness of the FGM samples produced at IPPT falls well beyond the dimensional limits of thermal conductivity measuring devices based on the Laser Flash method, a collaboration on this topic has recently been started with the Fraunhofer IFAM Institute in Dresden within the KMM-VIN WG1. A common research plan between IPPT and IFAM teams, including inter alia the above mentioned issue of thermal conductivity measurements of thick FGM samples, has recently been discussed and agreed upon, and is about to start in early 2022.

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NEWS FROM WG2: MATERIALS FOR ENERGY

There are two thematic subgroups in WG2 called "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): 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.

The non-EMEP subgroup deals with other topics related to energy materials, especially the low-carbon energy materials. The WG2-EMEP is coordinated by Christof Sommitsch (TUG), whereas the WG2-non-EMEP by Monica Ferraris (POLITO).

From TEKNIKER to POLITO

(KMM-VIN Research Fellowship, call 2021)

Synthesis and characterization of novel catalyst for CO₂ methanation by magnetron sputtering

One of the key points to improve the production of catalyst for industrial processes of interest, like Sabatier reaction which is really useful for the exploitation of CO₂ for energy production, is to find a large-scale catalyst production method which facilitates its industrialisation and avoids the production of harmful by-products for the environment. Taking this into account, PVD by magnetron sputtering arises as a reliable candidate [1], Fig. 13. This technology is considered with a great potential for industrial production of catalyst materials as the catalysts are directly synthesised on the substrate in one step process by a totally automated manufacturing process where parameters are controlled based on desired film morphology and properties.

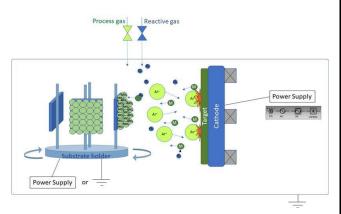


Fig. 13. Schematic representation of magnetron sputtering process (courtesy of TEKNIKER).

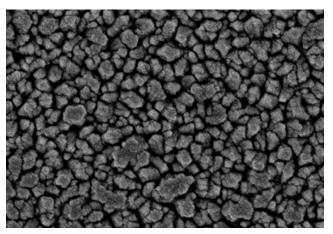


Fig. 14. SEM image of Ni layer over TiO₂ (courtesy of TEKNIKER and POLITO).

The research made at Politecnico di Torino during the fellowship stay, was focused on the development and characterisation of photoactive catalysts for Sabatier reaction using magnetron sputtering technology from two approaches: the development of 10 nm Ni layers deposited on top of columnar TiO₂ thin films and the deposition of embedded Ni into a TiO₂ matrix using RF sputtering technology.

For the first part of the research, several sputtering parameters were modified in order to obtain the desired Ni layer thickness, measured by AFM. As it can be seen in SEM images (Fig. 14), the Ni layers seem to form agglomerates over the TiO_2 surface, which can increase the roughness of the thin film.

For the second approach, TiO_2 (RF) and Ni (DC) were co-sputtered at 10^{-2} mbar pressure in order to obtain coatings with high surface area. The presence of photoactive anatase phase by XRD have not been observed and the presence of Ni was confirmed by EDS. In SEM images it can be seen that the coating has a high roughness (Fig. 15).

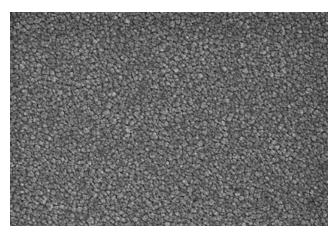


Fig. 15. SEM image of Ni embedded into TiO₂ (courtesy of TEKNIKER and POLITO).

Both developed types of thin films are going to be tested for the photocatalytic degradation of methylene blue in order to see if the layers have photocatalytic properties for UV and visible wavelengths.

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[1] Nalwa, H.S. Handbook of thin Film Materials, Academic Press. 2001, 5, 417.

From TUG to Ł-KIT

(KMM-VIN Research Fellowship, call 2021)

Hot deformation behaviour of low stacking fault energy alloys with a focus on high entropy alloys

The development of high strength and high ductility materials are a vital requirement for most engineering applications to improve energy-efficient improvement and prominent safety factors. The concentrated solidsolution or multi-component are excellent candidates. The field of multi-component alloys has recently emerged. Concentrated solid-solution, multicomponent or multi-principal alloys are familiar names to categorize the novel medium entropy alloys (MEA)s and high entropy alloys (HEA)s. These alloys consist of four or more elements added in notable amounts (typically above 10%), forming a single or multi-phase alloy. The exceptional properties, among others, high corrosion resistance, high resistance to hydrogen embrittlement, high toughness at low and room

temperatures of concentrated solid-solution alloys have been attracting attention in the materials science community and other engineering research fields. Like other metallic parts, thermomechanical processes are typically performed to achieve the desired shape properties and for structural applications. The selection of proper chemical composition for HEA cannot be dissociated from developing a suitable processing route. In this study, two HEAs were produced at Łukasiewicz -Krakow Institute Technology: of Al10Cr17Fe18Co16Ni39 and Al16Cr17Fe17Co14Ni36. The first is an fcc-based alloy, while the second is a bcc+fcc hypo-eutectic alloy, Fig. 16. Those alloys will be further investigated by hot compression tests that will be carried out using the Gleeble system at the Graz University of Technology. Moreover, the hot compression behaviour will be modelled and simulated using physically-based models under development by the Graz University of Technology team.

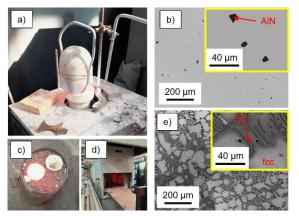


Fig. 16. Production of the high entropy alloys using a) an arcmelt furnace with Ar protective atmosphere poured into c) a ceramic-coated crucible that was heat-treated before melting in another d) furnace. As-cast microstructures of b) Al10Cr17Fe18Co16Ni39, e) Al16Cr17Fe17Co14Ni36 (courtesy of TUG and Ł-KIT).

As part of the modelling of the thermomechanical behaviour of the produced HEAs, other materials were also tested at Ł-KIT. The focus was to collect hot tensile data of low stacking fault energy materials, such as AISI 316L and Inconel 718. The flow stress data and microstructural analysis will be used to model the hot deformation behaviour of low stacking fault energy materials and then applied to the HEAs. The 316L was produced at the TUG using laser powder bed fusion, also known as selective laser melting or SLM.

The tests were carried out in the regime where workhardening is counterbalanced by dynamic recovery, although discontinuous dynamic recrystallization is also expected. The second low stacking fault energy material tested was a commercial Inconel 718 (IN718). The tests were carried out in the range where only carbides, delta (δ) phase and the fcc-matrix are stable. The hot tensile flow curves of the 316L and IN718 are shown in Fig. 17.

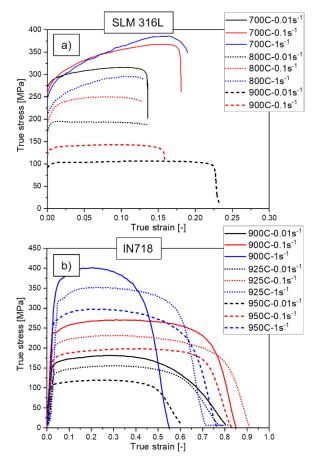


Fig. 17. Measured hot tensile flow curves for a) a laser powder bed fusion (LPBF, or SLM) 316L alloy, tested at 700°C, 800°C and 900°C; b) a commercial IN718 tested at 900°C, 925°C and 950°C (courtesy of TUG and Ł-KIT).

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News from AGH-UST

Microstructural characterization of austenitic steels after steam oxidation

Modern materials designed for superheaters and reheaters in coal fired power plants have to withstand severe conditions of high temperature and increased pressure. Moreover, these elements have to operate in highly corrosive environment (e.g. flue gas or steam). The work of the AGH UST, among others, is focused on characterization of new austenitic steel grades (eg. NF 709, SAVE 25) oxidized in various atmospheres and temperatures.

The advanced test stand (Czylok, Poland) allows for corrosion tests in steam and gas mixtures up to 950° C (Fig. 18). It possess three independent, electronically controlled gas channels. Such test rig, together with modern, high resolution (1 µg) microbalance (Radwag, Poland, Fig. 19) allows for detailed oxidation kinetics characterization of high temperature materials. Oxidized samples underwent very detailed microstructural characterization.



Fig. 18. Digitally controlled, advanced oxidation test stand (courtesy of AGH UST).



Fig. 19. High resolution (1µg) microbalance (courtesy of AGH-UST).

Utilization of advanced microscopy techniques (eg. low voltage SEM-EDX) reveals outstanding results. Due to the accelerating voltage set to 5 kV, the observation of small (~50-60 nm) precipitates (spots in Fig. 20d) was possible.

Investigation of the morphology of corrosion products grown on high temperature materials will allow for better understanding of oxidation processes. Oxide scale of SAVE 25 steel, developed at 700°C for 500 h in steam (Fig. 20) has a thickness of around 3 µm, what is an excellent result. The low-voltage SEM-EDS investigation allows to distinguish three layers of oxides (Fig.20b). Grain boundaries are paths of fast Cr diffusion. Due to the increased grain size of the alloy, the amount of the chromium transported via grain boundaries was lowered, what decreased the oxidation rate. Fe₂O₃ was created as first due to the outward diffusion of Fe. Grain size was not big enough to cause breakaway oxidation, what makes that material suitable for use in energy systems.

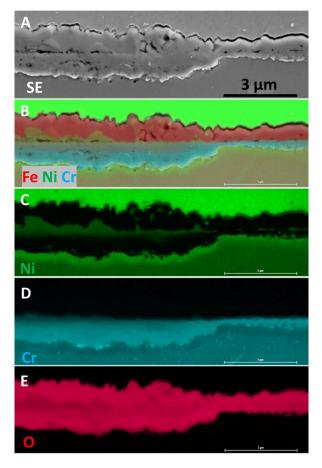


Fig. 20. Oxide scale of SAVE 25 steel: a) cross-section (SEM); b) combined map of Fe, Ni, Cr; c-e) map of selected elements [1] (courtesy of AGH-UST)

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[1] Rutkowski B., Baran K., Błoniarz R., Kozieł T. The morphology and microstructure of oxide scale grown on austenitic steel during steam oxidation at 700°C for 500 h. Materials, 2021, 14, 3821

https://doi.org/10.3390/ma14143821

News from AGH-UST and INTA

Oxidation behavior and oxide scale evolution of 718Plus superalloy in various environments

The ATI 718Plus® is a nickel-based superalloy exhibiting high strength and excellent oxidation resistance in high temperatures. The present study is focused on multiscale 2D and 3D characterization (morphological and chemical) of the scale and the layer beneath formed on the 718Plus superalloy during isothermal oxidation at 850°C up to 4000 h in dry and wet air. Dry air refers to laboratory dry air, so a mixture of 80 vol.% of N2 and 20 vol.% of O2, while wet air refers to laboratory dry air containing 10 vol.% of water vapor. The aim of the study was to establish a correlation between the oxide scale formation on 718Plus superalloy and different oxidation parameters, such as environment and time. In the present study, the evolution of the oxide scale and near-surface microstructure in 718Plus superalloy was

investigated using analytical electron microscopy combined with FIB-SEM tomography.

Longer oxidation duration led to the formation of a thicker external oxide scale and deeper internal oxidation zone of 718Plus superalloy. The outer oxide scale was formed mainly of Cr_2O_3 , with some amount of other oxides, such as TiO₂. The thickness of the oxide scale increased from about 2 µm after oxidation for 120 hours to 10 µm after 4000 hours of oxidation. In the internal oxidation zone, Al_2O_3 were formed mainly at the grain boundaries, the depth of this zone increased with time, from about 5 µm after 120 hours to almost 20 µm after 4000 hours in dry air and 35 µm in wet air.

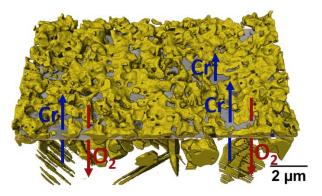


Fig. 21. 3D tomographic reconstruction of the δ phase interlayer formed in 718Plus superalloy after 120 hours oxidation in laboratory dry air (courtesy of AGH-UST).

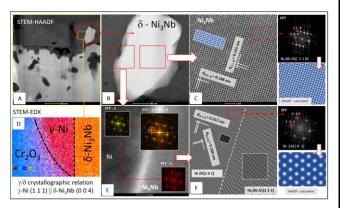


Fig. 22. High-resolution analysis of δ phase observed in the scale layer formed on the 718Plus superalloy after oxidation at 850°C for 120 h in wet air: (A) STEM-HAADF image of the scale and beneath highlighting the presence of δ -Ni₃Nb particle particles in this area Figure (B), (C) HRSTEM-HAADF image of δ phase particle with FFT image and HAADF image simulated using JEMS software, (D) superimposed STEM-EDX maps of selected elements, (E) FFT analysis of selected areas of HRSTEM-HAADF image, and (F) HRSTEM-HAADF image after filtering using an FFT showing the δ-Ni₃Nb/γ interface (courtesy of AGH-UST).

Furthermore, we identified and described formation of intermetallic δ -Ni₃Nb interlayer. It was formed between outermost chromia scale and bulk material of 718Plus superalloy. Due to application of FIB-SEM tomography, we revealed that it is in fact a discontinuous layer, where a significant amount of holes was observed (Fig. 21). In the tomographic reconstruction, this holes were filled with either Cr₂O₃ or Al₂O₃ oxides. Chemical analysis of δ phase by

STEM-EDXS, confirmed low solubility of other elements within it. Thus, it was concluded that holes present within this layer are the only paths to transport chromium, from bulk material into the chromia scale, and for oxygen to diffuse into the bulk material/internal oxidation zone.

Lastly, STEM analysis of the oxide scale revealed some nanometric particles present within it. We found that those were non-oxide, metallic particles containing Ni, Co, and Fe, so γ phase forming elements. Moreover, the particles were precipitated from the chromia instead of getting trapped inside during the grain growth. Figure 22 presents a highresolution STEM analysis of interfaces between δ -Ni₃Nb, y-Ni and Cr₂O₃ phases present in the oxide scale of 718Plus superalloy. Figure 22A shows a STEM-HAADF image of the scale and the area underneath, highlighting the presence of δ phase particles in this area (Figure 22B). The HRSTEM-HAADF imaging revealed columns of δ-Ni₃Nb phase atoms (Figure 22C). The bright and dark spots observed in the δ -Ni₃Nb phase correspond to Nb and Ni, respectively. Figure 14D shows superimposed STEM-EDXS maps of selected chemical elements, and these maps confirm the chemical and crystallographic complexity of the particle shown in Figure 22B. Figure 22E presents FFT images for the y and δ-Ni₃Nb phases as well as their superimposition, from which it follows that the $g_{\gamma} = [1-1-1]$ and $g_{\delta} = [400]$ reflections are very close, indicating that the interplanar distance between the $\{111\}_{\gamma}$ and $\{004\}_{\delta}$ crystallographic planes is small. Figure 22F shows the HRSTEM-HAADF image of the δ-Ni₃Nb precipitate and the y matrix atom columns. The rightmost small figures present a HAADF image of the y phase nanostructure and its image simulated by JEMS (highlighted in blue) as well as an FFT for this phase.

Detailed analysis of the influence of the oxidation time on the oxide scale formation of 718Plus superalloy can be found in the article published in *Corrosion Science* [1], while the description of the relationship between oxide scale formation and the oxidizing environment was published in *Materials* [2]. Formation and role of the δ phase interlayer was most extensively described in articles in *Corrosion Science* [1] and *Scripta Materialia* [3].

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[1] A. Kruk, S. Lech, A. Gil, G. Cempura, A. Agüero, A.M. Wusatowska-Sarnek, A. Czyrska-Filemonowicz, Threedimensional characterization of an oxide scale on ATI 718Plus® superalloy, *Corros. Sci.* 169 (2020) 108634. https://doi.org/10.1016/j.corsci.2020.108634.

[2] A. Kruk, A. Gil, S. Lech, G. Cempura, A. Agüero, A. Czyrska-Filemonowicz, Multiscale characterization of an oxide scale formed on the creep-resistant ATI 718Plus superalloy during high-temperature oxidation, *Materials*, 14, 2021, 6327. <u>https://doi.org/10.3390/ma14216327</u>.

[3] S. Lech, A. Kruk, A. Gil, G. Cempura, A. Agüero, A. Czyrska-Filemonowicz, Three-dimensional imaging and characterization of the oxide scale formed on a polycrystalline nickel-based superalloy, *Scr. Mater.* 167, 2019, 16–20.

https://doi.org/10.1016/j.scriptamat.2019.03.027.

News from TEKNIKER

ECTP Construction

The European Construction Technological Platform organized a conference in Madrid, on 2-3 December 2021, and within the event, there was organized a workshop on Materials on 3rd December 2021. Marco Falzetti, chairman from EUMAT and A4M, gave a talk on Alliance for Materials, EUMAT, Materials Manifiesto, The Role of the Materials in Post Covid Society, addresing materials topics within Horizon Europe Clusters, Partnership, Missions, Bauhaus Initiative, also considering citizens and social engagement. Amaya Igartua from TEKNIKER participated in the organizational team of the materials workshop and participated representing the project Metabuilding Labs.

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NEWS FROM WG3: BIOMATERIALS

From FAU to IPM

(KMM-VIN Research Fellowship, call 2020, completed in 2021)

Evaluation of the mechanical properties of polymer-based coatings, deposited on titanium substrates by electrophoretic deposition (EPD) for biomedical applications

Antibacterial drug-loaded polymer coatings, which are able to release therapeutic agents in a controlled manner with respect to time and dosage, are gaining increasing attention for bio-functionalization of orthopedic and dental implants [1]. The mechanical stability of such coatings plays a crucial role for their long-term survivability in the human body. Therefore, the aim of the current research stay was to evaluate the mechanical and structural properties of drugloaded polymer-based coatings, prepared on titanium substrates by electrophoretic deposition (EPD).

The adhesion strength of the coatings was analyzed at the Faculty of Mechanical Engineering, Brno University of Technology, by using a CSM Instruments scratch tester. A Rockwell indenter having a 200 µm tip radius was moved 5 mm over a coating surface with linearly increasing normal load (from 1 to 10 N) at loading rate of 24 N/min and speed of 5 mm/min. The critical load (Lc), at which the coating was completely removed from the titanium substrate, was obtained by light microscopy observations, acoustic emission and friction force signals. SEM analysis was performed to assess the scratch morphology after testing (Fig.23). In addition, the structural composition of the coatings was analyzed by X-Ray diffraction (XRD). The scratch results showed that the addition of inorganic fillers, such as bioactive glass particles, in the polymer coating increased the critical load of the coatings.

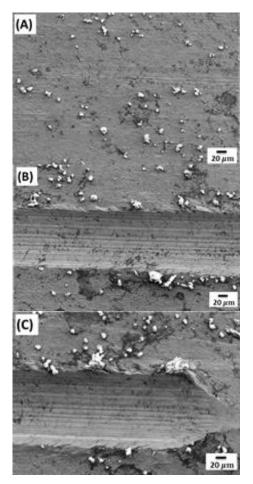


Fig. 23. Representative SEM micrographs of the scratch at (A) low (1 N), (B) middle (3 N) and (C) high (10 N) loads on zein-chondroitin sulphate coatings (courtesy of FAU).

In addition, a chemical pre-treatment of Ti substrates prior to EPD enhances the adhesion strength of the coatings. The XRD analysis confirmed the changes in the structural composition of titanium substrates after chemical pre-treatment. Further collaboration between FAU and IPM will focus on investigating the mechanical performance of novel EPD composite coatings.

> Zoya Hadzhieva, FAU, Aldo R. Boccaccini, FAU Ivo Dlouhý, IPM aldo.boccaccini@fau.de

[1] M. A. Akhtar et al., Electrophoretic deposition and characterization of functional coatings based on an antibacterial gallium (III)-chitosan complex, *Coatings*, 2020, 10(5), 483; <u>https://doi.org/10.3390/coatings10050483</u>

From FAU to POLITO

(KMM-VIN Research Fellowship, call 2021)

Advanced characterization of bioactive glass nanoparticle containing alginate based hydrogels for tissue engineering applications

The aim of the research project was to evaluate the antibacterial properties of alginate-based hydrogels containing phytotherapeutic agent (PA) with antibacterial effect combined with bioactive glass particles. Alginate is often used in biofabrication approaches and in drug delivery systems. Alginate is well known for its high biocompatibility. However, alginate has a high molecular weight and therefore a low degradation rate. Moreover, it does not induce the required cell interaction [1,2]. To overcome these drawbacks, the oxidized form of alginate, alginate dialdehyde (ADA) [2] was used in this work. The oxidation leads to the creation of free aldehyde groups, which enable the covalent bonding to proteins like gelatine (GEL), a well-known material for 3D bioprinting approaches. Plant-based antibacterial agents are being increasingly considered for biomedical application and the combination of phytotherapeutics and engineered biomaterials is a current research field at the FAU Institute of Biomaterials [3]. Mesoporous bioactive glass nanoparticles (MBGN) produced via sol-gel were used in this work. 70S MGBNs are composed of 70% SiO₂ and 30% CaO (mol%).

During the stay at POLITO antibacterial investigation (due to the antibacterial effect of PA) and bioactivity testing (due to the combination with MGBNs) were performed. Preliminary results of the bioactivity test in simulated body fluid (SBF) confirmed the bioactive effect of 70S MBGNs. Bioactivity tests were repeated with the addition of PA at concentrations not having a negative impact on bioactivity.

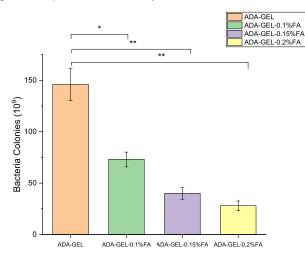


Fig. 24. Counted bacteria colonies on agar blood plates after 9 dilutions. Bacteria colonies for pure ADA-GEL as reference, ADA-GEL-0.1% FA (w/v), ADA-GEL-0.15%FA (w/v) and ADA-GEL-0.2%FA (w/v). Statistical analysis was performed via a one-way ANOVA with the Bonferroni test for $p<0.05=^*$, $p<0.01=^*$ and $p<0.001=^{***}$ (courtesy of FAU).

For antibacterial testing ADA-GEL hydrogels were incorporated with PA in different concentrations; namely 0.1% (w/v), 0.15% (w/v) and 0.2% (w/v). ADA and GEL were mixed at 5% (w/v), respectively, in a 1:1 ratio. For bioactivity testing ADA-GEL with 0.15% (w/v) FA and two different MBGN concentrations (0.1% and 0.5% (w/v)) were used.

Results of antibacterial testing reveled that the addition of PA into ADA-GEL hydrogel increased the antibacterial effect of the hydrogels. The antibacterial effect was significantly different compared to pure ADA-GEL as a reference (see Figure 24). The concentration of PA is limited due to the poor solubility of FA. Moreover, preliminary cell-viability results showed that an increase of PA concentration leads to a decrease of cell viability, which occurs due to a decrease of pH. Therefore, a compromise between antibacterial effect and cell impact needed to be found. In regard to this fact, the middle concentration of PA (0.15% (w/v)) was chosen for further investigation. Results of bioactivity testing reveled that the PA does not have a negative impact on the bioactivity of the MBGN containing ADA-GEL. With both concentrations of MGBN particles (0.1% and 0.5% (w/v)) the formation of a hydroxyapatite (HAp) layer on the surface of hydrogel films was confirmed after 7 days of immersion in SBF. In the case of 0.5% (w/v) MBGN containing samples the HAp formation was more visible. For confirmation, SEM imaging, EDX, XRD and FTIR analysis were used. Further investigations at the FAU Institute of Biomaterials will include a complete cell biology assessment of the novel ADA-GEL-MGBN-PA composite hydrogels.

> Faina Bider, FAU, Aldo R. Boccaccini, FAU Enrica Verne, POLITO <u>aldo.boccaccini@fau.de</u>

[1] M. Xu, M. Qin, Y. Cheng, X. Niu, J. Kong, X. Zhang, D. Huang, H. Wang, Alginate microgels as delivery vehicles for cell-based therapies in tissue engineering and regenerative medicine, *Carbohydr. Polym.* 2021, 266, 118128. https://doi.org/10.1016/j.carbpol.2021.118128.

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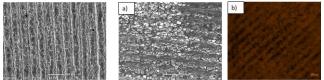
[3] M.A. Akhtar, C.E. Mariotti, B. Conti, A.R. Boccaccini, Electrophoretic deposition of ferulic acid loaded bioactive glass/chitosan as antibacterial and bioactive composite coatings, *Surf. Coatings Technol.*, 2021, 405, 126657. https://doi.org/10.1016/j.surfcoat.2020.126657.

News from WUST and POLITO

Natural coating on Mg alloys to control their degradation in physiological environment

Spatial cast structures for biomedical applications (e.g. bone implants) were elaborated in the cooperation of Politecnico di Torino (POLITO) and Wrocław University of Science and Technology (WUST). Perforated honeycombs were designed via Autodesk Inventor software and printed in PLA with Fused Deposition Modeling (FDM) method. Then these polymer patterns were used for the investment casting process, during which the model was replaced with AZ91 casting alloy, resulting with cellular magnesium elements. Samples were thus prepared at WUST and sent to POLITO for the selection of appropriate anticorrosive coating's deposition technique.

Among the studies to be conducted, the mechanical properties (compressive strength) tests of both coated and uncoated samples are foreseen at WUST. The coatings description is given below (Fig. 25).



Untreated

Treated: reaction layer (a) and fluorescent molecules (b)

Fig. 25. Samples of natural coatings on Mg alloys (courtesy of WUST).

Aim of the work: to modify the surface of AZ91 3D constructs in order to regulate their degradation

- Collaboration in the framework of KMM-VIN
- Master degree thesis of a POLITO student in Biomedical Engineering

Main advantages of the proposed solution:

- Natural molecules: sustainable resources, no toxicity, environmental friendly
- Surface modification/coating by immersion in aqueous solution: possibility to treat complex geometries
- Possibility to modulate process parameters to optimize the coating

Observations:

- No weight loss during the treatment
- Uniform distribution of the molecules on the surface

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News from AGH-UST

Oil transport through the 3D network of electrospun fibers for delivering gamma linoleic acid (GLA) for skin treatment

Atopic dermatitis is one of the most common skin problems affecting people all over the world regardless the age. Different treatments can be applied, oral administration of antibiotics or steroids, but also topical therapy in the form of patches. My team works on the project "Nanofiber – based sponges for atopic skin treatment" funded by Foundation for Polish Science. In this project my PhD student Ewa Sroczyk produced polyimide (PI) electrospun patches with the representative 3D reconstruction of fiber networks showed in Fig. 26.

Besides the biocompatibility testes of the patches, we performed the numerical simulation of blackcurrant seed oil, which is reach in gamma linoleic acid (GLA), passing through the electrospun membrane. The PI patches reaching 95.6 % porosity were filled with blackcurrant seed oil containing 15 % GLA. The 3D reconstruction presented in Fig. 26 was used for modeling GLA mass transport in PI patches. Additionally, PI patches were assessed in vivo with skin hydration tests over 6 h and compared to numerical simulation of GLA release to the skin. The electrospun PI patches allow long-term hydration

showing the potential applicability of our research to treat atopic skin. [1].

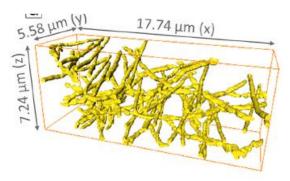


Fig. 26. 3D reconstruction of electrospun PI membrane with voxel size of 20 × 20 × 60 nm. [1] (courtesy of AGH-UST).

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[1] Sroczyk, E. A.; Berniak, K.; Jaszczur, M.; Stachewicz, U. Topical Electrospun Patches Loaded with Oil for Effective Gamma Linoleic Acid Transport and Skin Hydration towards Atopic Dermatitis Skincare. *Chem. Eng. J.*, 2022, 132256. https://doi.org/10.1016/j.cej.2021.13225.

ERC-2020-STG project: *Bioinspired Composites Strategies for Saving Energy (BioCom4SavEn),* 5 years, started 1-01-2021 at AGH UST, Faculty of Metals Engineering and Industrial Computer Science

Nature is an amazing source of inspiration, one of them is thermal insulation structures, such as penguin feathers and polar bear hair. These keratin-based materials are combing the fibers and porosity to obtain the desire thermal and mechanical properties. To investigate natural designs, we use advanced microscopy techniques for high resolutions images and 3D reconstructions based on the scanning electron microscope (SEM) and focused ion beam (FIB) techniques. The microscopy investigations reveal many similarities in the structure of natural materials being a key in constructing novel materials such as new materials constructions for thermal insulations.

Our microscopy studies of polar bear hair and penguin feather [1] have been featured in Materials Today (https://www.materialstoday.com/biomaterials/news/c onnected-pores-keep-polar-bears-and-penguins-

<u>warm</u>). Within the ERC project BioCom4SavEn we construct light and more efficient thermal insulation, develop cooling systems based on fibrous membranes to dissipate effectively heat, and build robust cooling and insulation systems for small devices and smart textiles.

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[1] Metwally, S.; Martínez Comesaña, S.; Zarzyka, M.; Szewczyk, P. K.; Karbowniczek, J. E.; Stachewicz, U. Thermal insulation design bioinspired by microstructure study of penguin feather and polar bear hair. *Acta Biomater.* 2019, 91, 270–283.

https://doi.org/10.1016/j.actbio.2019.04.031.

News from UPM

Multi-scale and multi-material smart implants

Additive manufacturing technologies have reshaped product design in the last couple of decades and are transforming the biomedical industry, by providing innovative and personalized approaches to solving healthcare problems. Tissue engineering and biofabrication are absolutely dependent on additive manufacturing advances, as the extremely complex geometries, micro-environments and biomechanical properties of living tissues cannot be adequately mimicked by using more traditional manufacturing techniques. Current research trends in medical additive manufacturing include the design and manufacture of multi-scale, multi-material and multiphase constructs, for their use as tissue engineering scaffolds capable of providing cells with the correct epigenetic cues for successful repair and regeneration. Besides, shape-morphing structures, in connection with 4D printing principles, are being explored for promoting minimally invasive surgeries and for delivering structures capable of evolving with patients, according to their healing process or along their life. Such smart scaffolds enter the realm of living materials and may lead to a myriad of new biomedical applications, which UPM is researching in two new projects under the lead of Prof. Díaz Lantada, as introduced below.

In the "INKplant" project (funded by Horizon 2020 and led by Profactor GmbH with participation of 20 EU partners). innovative materials, methods and technologies for creating multi-material and multiscale tissue engineering scaffolds are being researched. The main focus is on articular repair and regeneration, including osteochondral defects and meniscal constructs, and on orofacial surgery, including jaw reconstructions and cleft palate recreation. KMM-VIN's partner UPM, under the lead of Andrés Díaz Lantada, contributes with design and computational modeling strategies for improved biomechanical performance of the developed implants and with the simulation of additive manufacturing processes. Rapid prototyping technologies are also being used for the development of biomimetic constructs, as show in Fig. 27.

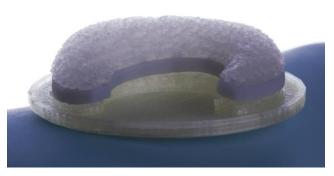


Fig. 27. Example of multi-material scaffolds for meniscus repair (courtesy of Adrián Martínez, UPM).

In the "iMPLANTS-CM" project (funded by Madrid's General Directorate for R&D through its "Synergy Call" inspired by the ERC model), KMM-VIN's partner UPM, under the lead of Andrés Díaz Lantada, and IMDEA Materials, under the lead of Jon Molina-Aldareguia, aim at a new generation of smart implants. These should be able to promote minimally invasive procedures, evolve with patients, enhance personalized approaches and progress towards n-dimensional printing.

A collection of cutom-made cardiovascular, articular and spinal implants will be developed, with a focus on the application of metamaterials to enhance the shape-morphing abilities of smart implants. Polymeric additive manufacturing (see Fig. 28) will be used for conceptual screening purposes. Additive manufacturing employing Ni-Ti alloys will lead to smart implants with superelastic and shape-memory properties, in connection with 4D printing principles and beyond.



Fig. 28. Printed lattices as structures for heart valve reconstruction (courtesy of Adrián Martínez, UPM).

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News from TEKNIKER

TEKNIKER published an article in the Spanish review "Plasticos Modernos" related to Biosmart Project entitled <u>"Smart Biopackaging to improve the food guality conservation</u>".

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NEWS FROM WG4: MATERIALS MODELLING AND SIMULATION

9th KMM-VIN Industrial Workshop on "Design and modeling of innovative biomaterials and bioinspired materials for industrial applications"

Co-chairs: Christian Hellmich (TUW, IW9 local organiser), Katarzyna Kowalczyk-Gajewska (IPPT), Andrés Díaz Lantada (UPM).

Unfortunately, due to a difficult pandemic situation in Austria and the imposed restrictions the IW9 to be held at the Vienna University of Technology (TUW), from 25 to 26 January 2022 had to be postponed. New dates for the workshop will be provided later.

This workshop belongs to the series of industrially oriented workshops organized by KMM-VIN in collaboration with leading research centres and industries. The most recent advances in material science and technology with high industrial potential are presented in this series. These workshops provide a unique opportunity to start and intensify the communication and cooperation between scientists and engineers for reshaping several industrial sectors with the help of knowledge-based multifunctional materials.

The 9th KMM-VIN Industrial Workshop will cover the following topics:

• Design and modeling of biomaterials for medical devices and other biomedical applications.

• Design and modeling of bioinspired materials for industrial applications.

• Multi-scale / multi-physical modeling of the synthesis, processing and application of biomaterials and bioinspired materials.

• Modelling methods of microstructure-property relationship for hierarchical materials.

• Artificial intelligence-aided design of innovative biomaterials and bioinspired materials.

• Manufacturing hierarchical biomaterials and bioinspired materials towards final applications.

• Promotion of knowledge-based biomaterials and bioinspired materials for enhanced industrial performance.

• Cases of success in different industrial sectors: health, energy, transport, space and construction.

At present the following invited keynote speakers have preliminary agreed to present research directions and recent breakthroughs in the aforementioned topics:

Stéphane Avril, Vienna University of Technology, Austria, "Prevention and Treatment of aortic Aneurysms using computational biomechanics"

Tomasz Lipniacki, IPPT, Warsaw, Poland "Mathematical modeling of innate immune response"

Jon Molina-Aldareguia, IMDEA Materials, Madrid, Spain, "Additive manufacturing of advanced alloys for biodegradable and smart implants"

Francesco Moscato, Medical University of Vienna, Austria, "Trends in the personalized design and manufacture of next generation implants"

Stefanie Reese, RWTH Aachen University, Germany, "Stents and heart valves - new aspects in modeling and simulation"

Note that the list of key-note speakers will be reconfirmed once new days of the workshop are known. All participants will be invited to present a poster or oral presentation to show and discuss their work on materials modelling and process simulation.

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News from UPM

Artificial intelligence for metamaterials and metasurfaces

Artificial intelligence (AI) in general, and machine learning (ML) methods in particular, are continuously providing innovative approaches for the design, modeling and discovery of advanced materials. UPM has recently developed studies, in which AI has proven useful for predicting the performance of metasurfaces and metamaterials, as detailed below (Fig. 29).

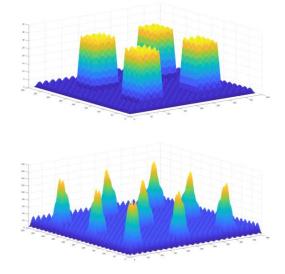


Fig. 29. Examples of metasurfaces, whose wettability performance is predicted by AI tools (courtesy of UPM).

In "Artificial Intelligence Aided Design of Microtextured Surfaces: Application to Controlling Wettability" (Nanomaterials 2020) [1], authors explain the creation of a comprehensive library of microtextured surfaces, with well-known wettability properties. Such library is processed and employed for the generation and training of artificial neural networks, which can predict the actual wetting performance of new design biointerfaces. Present research demonstrates that AI can importantly support the engineering of innovative hierarchical or multi-scale surfaces, when complex-tomodel properties and phenomena, such as wettability and wetting, are involved.

In "Artificial Intelligence Aided Design of Tissue Engineering Scaffolds Employing Virtual Tomography and 3D Convolutional Neural Networks" (Materials 2021) [2], the use of 3D convolutional neural networks (3D CNNs), trained using digital tomographs obtained from the CAD models, is validated as a powerful resource for predicting the mechanical properties of novel tissue engineering scaffolds. This approach stands out for the application of a 3D printing slicing software for the generation of virtual tomographs, as input for training, validation and testing of 3D CNNs (Fig. 30), and can be applied to any kind of 3D metamaterials, mainly for performance prediction purposes. In the near future, these tools may be combined with genetic algorithms for the automated discovery of metamaterials with special properties.

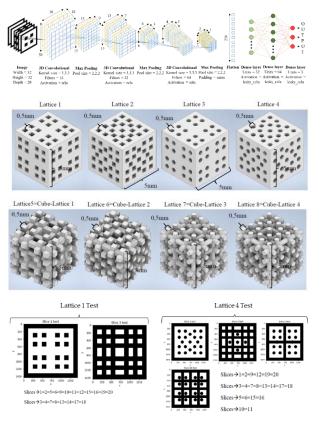


Fig. 30. Proposed structure for the 3D CNNs and CAD models and digital tomographs for testing them (courtesy of UPM).

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[1] Díaz Lantada, A.; Franco-Martínez, F.; Hengsbach, S.; Rupp, F.; Thelen, R.; Bade, K. Artificial Intelligence Aided Design of Microtextured Surfaces: Application to Controlling Wettability. Nanomaterials 2020, 10, 2287. https://doi.org/10.3390/nano10112287.

[2] Bermejillo Barrera, M.D.; Franco-Martínez, F.; Díaz Lantada, A. Artificial Intelligence Aided Design of Tissue Engineering Scaffolds Employing Virtual Tomography and 3D Convolutional Neural Networks. Materials 2021, 14, 5278. <u>https://doi.org/10.3390/ma14185278</u>.

News from IPPT and IMIM

Texture-based optimization of crystal plasticity parameters: application to zinc and its alloy

The collaboration between IPPT and IMIM, both being the members of KMM-VIN, resulted in an interesting research, published recently in *Metallurgical and Materials Transactions A* [1]. Zinc and its alloys are a promising material for bioabsorbable stents. Although the strength and ductility of such materials are often not satisfactory, alloying with magnesium and subjecting the material to hydrostatic extrusion (HE) can solve this problem. Modelling by means of polycrystal plasticity can provide insights into deformation mechanisms active during HE. The crucial issue in such modelling is a proper choice of material parameters which has to be based on experiments. Zinc and its alloys have the hexagonal crystallographic close-packed (HCP) lattice. Therefore, the task is more complicated than in higher symmetry metals due the presence of many slip and twinning systems.

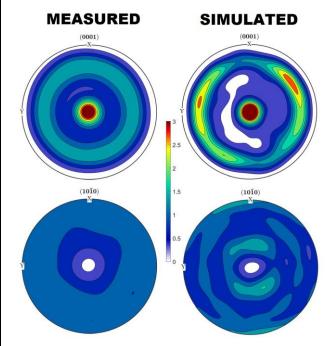


Fig. 31. The automatically obtained matching of the measured and simulated texture for Zn-1.5%Mg alloy subjected to the first pass of hydrostatic extrusion (courtesy of IPPT and IMIM).

One way to deal with the optimization problem is to use a genetic algorithm to automatically search the parameter space. As a fitness function one can use e.g. stress-strain curves obtained in multiaxial mechanical testing, as it was done in [2]. Here, thanks to experience of IPPT team in crystal plasticity modelling, parameter optimization and texture quantification, and the experience of the IMIM team in the experimental evaluation of microstructure, it was possible to propose an entirely novel fitness function based on comparing the simulated and experimental texture in a numerical fashion.

The method enabled to obtain simulated texture in good agreement with the experimental one (Fig. 31). Using this method, two separate EA runs for pure Zn and Zn-Mg alloy were performed which led to two sets of crystal plasticity parameters. The obtained parameters were validated by simulating another large plastic deformation process (rolling) and comparing the texture predicted in this process with the experimental one.

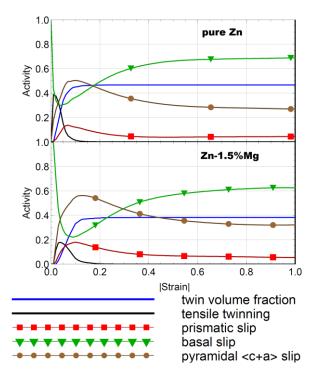


Fig. 32. The relative activities of the plastic deformation mechanisms for pure Zn and Zn-1.5Mg alloy in the first pass of hydrostatic extrusion revelead thanks to the obtained sets of parameters (courtesy of IPPT and IMIM).

The most important outcomes of the research are twofold. First, the new fitness function for the optimization of polycrystal plasticity parameters based on crystallographic texture has been proposed. The function is general and can be applied for any polycrystalline metalic material. Second, the deformation mechanisms active during the HE of zinc and its alloy have been identified. This provides knowledge about the plastic deformation of the studied materials which will be useful in future applications. It also immediately shows what is the result of Mg addition on the active deformation meshanisms and explains what is the source of observed texture evolution differences (Fig. 32).

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[1] Frydrych K., Jarzębska A., Virupakshi S., Kowalczyk-Gajewska K., Bieda M., Chulist R., Skorupska M., Schell N., Sztwiernia K., Texture-based optimization of crystal plasticity parameters: application to zinc and its alloy, *Metal. Mater. Trans. A*, 2021, 52(8), 3257-3273 https://doi.org/10.1007/s11661-021-06285-7

[2] Frydrych K., Maj M., Urbański L., Kowalczyk-Gajewska K., Twinning-induced anisotropy of mechanical response of AZ31B extruded rods, *Mater. Sci. Engng. A*, 2020, 771, 138610-1-14 <u>https://doi.org/10.1016/j.msea.2019.138610</u>.

News from TEKNIKER

OntoCommons International Event

The EU Project <u>OntoCommons</u> organized a <u>Global</u> <u>Workshop: "Ontology Commons addressing</u> <u>challenges of the Industry 5.0 transition".</u> The event included 14 parallel sessions, organized virtually on November 2-5, 2021. The event addressed the needs of standardised data documentation and data sharing valorisation to foster data-driven innovation. Iker Esnaola presented TEKNIKER demonstrator in OntoCommons Project "Characterising Tribological Experiments: Current Status and Impact of OntoCommons". Some videos are available <u>link</u> and the detailed Agenda.

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NEWS FROM WG5: GRAPHENE/2D MATERIALS

Project ASCEND

Peter Hansen of Hive Composites (www.hivecomposites.com) is involved in a UK aerospace and automotive consortium project called ASCEND (Aerospace and Automotive Supply Chain Enabled Development): a 3 year programme which started in January 2021. The project is led by GKN Aerospace with 16 other partners and the aim is to develop and accelerate composite material and process technologies for the next generation of energy efficient aircraft and future mobility.

The £40m consortium, funded by a £20.0M commitment from industry and a £19.6M commitment from the UK Government via Aerospace Technology Institute (ATI - https://www.ati.org.uk/) will focus on greater adoption of composite technologies today, the industrialization of new technologies, as well as accelerating aerospace production rates to meet future high-volume requirements. Part of the HIVE work is to develop multi-functional composite materials using CNT based materials available in several different formats including suspensions, yarns and sheets for electrical conductivity, fire protection and damage detection in composite structures. This includes work on anti-icing technologies, self-heated composite tooling, intelligent structural health monitoring and lightning strike protection.

Peter says about the project: "As a SME organisation the ASCEND programme will offer Hive the opportunity to engage with the supply and end user chain. ASCEND will facilitate our capabilities to develop unique thermally/ electrically conductive polymer composite materials enabling new and more efficient manufacturing processes, multifunctionality in parts, saving mass and emissions. The project will also enable Hive to expand the number of technical staff in the company. Exploitation of the results, technically and commercially, will result in significant increase in turnover and profitability for us over next 3 to 5 years".

ASCEND project information -

https://www.gknaerospace.com/en/newsroom/newsreleases/2021/gkn-aerospace-and-partners-launchascend-to-accelerate-high-volume-compositetechnologies/

News from TEKNIKER

Puzzle Event

The Puzzle X event on Frontier Materials (Quantum, Graphene, 2D materials, metamaterials, biomickmicking, intelligent materials,..) for a better world, has been organized by AMPT Network (Advanced Material Pandemic Taskforce) in collaboration with EUMAT Platform. The event took place in Barcelona, on 16-18 November 2021. PUZZLE X is the first convening forum to bridge the world of Frontier Materials societal impact, entrepreneurship, venture building, corporate innovation, bleeding edge of material science and United Nations Sustainable Development Goals to facilitate the use the materials of the future to help our world today. Amaya Igartua from TEKNIKER participated in the Pannel dedicated to Technology & Global Collaboration towards 2030 Agenda. The presentation highlighted the activities carried out in TEKNIKER in relation to the Sustainable Development Goals. She presented the materials activities within EUMAT and Alliance for Materials (including KMM-VIN). She addressed the role of Tribology for Development Sustainable products and processes and the role of Graphene and Nanomaterials to reduce weight and carbon footprint. The presentations will be available in January.

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News from UH

Antonios Kanellopoulos (University of Hertfordshire) in collaboration with researchers from University of Cambridge and Khalifa University of Science & Technology published their research on the development of an electronic textile for structural health monitoring of cement-based composites. The electronic textile was developed using graphene oxide and embedded into a cementitious matrix. The composites showed mechanical stability and functional durability over long-term cyclic compression tests of 1000 cycles, demonstrating capability of sensing the applied external stress. The work was published in September 2021 in Engineering Reports [1].

> Antonios Kanellopoulos, UH a.kanellopoulos@herts.ac.uk

[1] Irfan, MS, Ali, MA, Khan, KA, Umer, R, Kanellopoulos, A, Abdul Samad, Y. An electronic textile embedded smart cementitious composite. Engineering Reports. 2021;e12468. <u>https://doi.org/10.1002/eng2.12468</u>

EU PROJECTS and COOPERATION with EU ORGANISATIONS

This section presents the ongoing EU projects, MERA-Net and similar international projects with two or more KMM-VIN members involved as partners. The projects which have resulted from partners' networking within KMM-VIN WGs are particularly well suited for this section of the Newsletter.

RePoParts "Additive manufacturing Parts and Coatings using Recycled Powder from waste" (M-ERA.NET Joint Call 2020). The project started in October 2021 for a duration of three years. The consortium: Ł-KIT (project coordinator), Progresja, Gescrap, Delaser, Stern Hidráulica S.A, University de La Rochelle (TEKNIKER acting as a subcontractor).

The main result of the project will be the development of a methodology aligned with the principles of circular economy, to recycle scrap yard waste (100% scrap) and provide high-quality materials in a powder form to generate components for additive manufacturing and thermal spray machines.

This project will develop a new way of producing valuable powder, to be used for additive manufacturing and plasma sprayed coatings that can be applied in advanced components such as hydraulic cylinders.

The project proposes the use of metallic waste of Fe alloys with at least 12 wt.% of Cr in order to generate valuable powder to be used as feedstock materials to produce coatings which enhance the durability of components. Cutting-edge technological steps will allow to produce powder for additive manufacturing (AM) and thermal spray machines. Some of the lab scale powder of 304 steel has been developed and evaluated in a high temperature oxidation process, which demonstrates the feasibility of the approach.

The project proposes two different routes to increase the impact on the final products. First, the milling and crushing of metallic waste to achieve sprayable particle morphologies and fractions is considered. The second process is related to the classic casting process, and the atomisation of the ingots in order to produce a sprayable powder. The powder produced (investigated analytically) will be used in the Laser Metal Deposition process (LMD) and SLM process to fabricate coatings and develop 3D objects. The same powders will also be employed as feedstock for thermal spray processes (i.e. HVOF) in order to produce thick coatings, like with AM processes. Such products (3D printed and thermally sprayed) will be assessed under close-toindustrial conditions and applied to hydraulic cylinder demonstrators. Thus, the tests will include (1) the study of the deposition conditions (power, temperature, flow, nozzle size) as a function of the powder quality. The

performance of the coatings (with and without heat treatment) will be investigated at high temperatures of up to 700°C for up to 1000 hrs in various atmospheres (sulphidation, oxidation, inert, mixed gases). In addition, mechanical testing (tensile, elongation, yield stress, bend test, toughness) will link the resistance to the microstructure. Accelerated tests (erosion and abrasion resistance) will also be performed by tribological tests in neutral and corrosive media. The results will be compared with reference market-based coatings and mechanical samples from the demonstrator. The scientific gain resulting from the project will be especially significant in the growing field of additive materials that show exciting properties but whose mechanisms of fabrication and degradation are yet to be fully understood to forecast increased lifetime in specific applications (e.g. hydraulic cylinders adapted to resist coastal and marine environments as proof of concept of "RePoParts").

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COOPERATION with EuMaT ETP

EuMaT ETP "European Technology Platform for Advanced Engineering Materials and Technologies". KMM-VIN maintains close working contacts with EuMaT. Our representatives are members in the EuMaT Steering Committee: Pedro Egizabal (TECNALIA), Arnaldo Moreno (ITC) and Michal Basista (IPPT). Amaya Igartua serves as the co-secretary of the EuMaT ETP. It is a bottom-up created platform whose main role is to integrate the materials research community and the EU industry in the process of establishing of R&D priorities concerning advanced materials and technologies, and expressing them towards the EU Commission. The readers of this Newsletter are invited to have a look at Latest News on the EuMaT website https://www.eumat.eu

KMM-VIN RESEARCH FELLOWSHIPS, COURSES and TRAININGS

KMM-VIN Research Fellowships 2021 and 2022 Call

The KMM Mobility Programme offers Research Fellowships on a competitive basis for PhD students and early stage researchers from the KMM-VIN members to do research at other KMM-VIN member institutions. Joint publications of the fellowship holder and the host are required as a result of the KMM-VIN Research Fellowship within 12 months after the research stay completion. The up-to-date list of publications from the KMM-VIN Research Fellowships can be viewed at <u>http://kmm-vin.eu/fellowships/</u>.Here we recall the results of the 13th Call in 2021:

Applicant	Host	Duration (months)
F. Bider(FAU)	E. Vernè (POLITO)	1
A. Villamayor (TEKNIKER)	M. Ferraris (POLITO)	1
R. Buzolin (TUG)	T. Dudziak (Ł-KIT)	1
G. Cempura (AGH- UST)	F. Smeacetto (POLITO)	1
H. Mora Sanchez (UCM)	F. Warchomicka (TUG)	1.5
K. Virijevic (BioIRC)	P. Thurner (TUW)	1
H. Kapłon (WUST)	A.R. Boccaccini (FAU)	1
G. Skrabalak (Ł-KIT)	J. Dusza (IMR SAS)	1
E. Rząd (Ł-KIT)	A.R. Boccaccini (FAU)	1.5

The 14th Call of KMM-VIN Research Fellowships will be opened at the end of February 2022 following the budgetary decisions of the KMM-VIN General Assembly which will allocate funds for the 14th Call.

KMM-VIN Specialized Courses

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, KMM-VIN members' locations, or e-learning. These courses are designed for experienced staff members, who want to improve their skills in a selected field, but also for nonexperienced 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 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 link: http://kmm-vin.eu/for_industry/courses_and_trainings/ Interested companies can contact the coordinator of trainings, Arnaldo Moreno at amoreno@itc.uji.es

Current list of Specialised Courses

MATERIALS

- Adhesive bonding (LU)
- Biomaterials (FAU)
- Development and applications of microstructured and micro-textured materials (UPM)
- Light alloys and composites (Ł-KIT)
- Materials for energy systems and advanced steam power plants (AGH-UST)
- Materials for 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 (IS)
- International / European Welding Engineer / Technologist / Specialist (IS)
- International welder (IS)
- 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 (Ł-KIT)
- 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 (Ł-KIT)

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 (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, Erlangen-Nuremberg) has been elected Vice-president of the Federation of European Materials Societies (FEMS). He represents the German Materials Society in the Executive (DGM) Committee of FEMS since 2016. He will start his 2-year tenure as FEMS vice-president in January

2022. Aldo said: 'I am very honoured to have been elected Vice-President of FEMS and am looking forward to working together with President Anna Zervaki and the Management Team to enhance the visibility of FEMS in the materials science community in Europe and worldwide. I thank specially the German Materials Society for the support and trust deposited in me for this position. Materials science plays a key role in tackling today's important challenges in areas ranging from energy, environment, transport, healthcare, and security among others, and FEMS has a central part to play in raising awareness of materials as an essential contributor to modern technologies for a better future".

Aldo R. Boccaccini is listed as one of the highly cited researchers worldwide according to the latest edition of the Stanford List of Highly Cited Researchers <<u>https://elsevier.digitalcommonsdata.com/datasets/bt</u>chxktzyw/3>, published last October. He appears in the top 50 list in the subject "Materials". The "Stanford List" represents a comprehensive analysis of citations including about 100,000 scientists. It provides standardized information on citations, h-index, co-authorship adjusted metrics, citations to papers in different authorship positions resulting in a composite indicator. The list covers 22 scientific fields and 176 sub-fields. The selection is based on the top 100,000 by c-score (with and without self-citations) or a percentile rank of 2% or above.



Jan Dusza (IMR SAS, Košice) was granted the Alexander von Humboldt Research Award 2021. He will conduct his research project at University of Stuttgart, Germany in cooperation with Prof. Siegfried Schmauder

for one year. The Award winner was selected by the international committe of highly qualified experts led by the Nobel Prize winner Prof. Kip Stephen Thorne (see photo).

Ján Dusza received the ESET Science Award 2021. The award is granted to an individual researcher whose work results in the past 10 years have contributed exceptionally to highlighting Slovak science in the European or global science area, and which have a remarkable actual or potential impact on other aspects of the Slovak society.



Peter Mayr (TU Munich) since October 2021 is Head of the Department of Materials Engineering in the TUM School of Engineering and Design. The department includes

8 professorships with a common focus on materials engineering. The materials covered in the department range from structural and functional metals, mineral construction materials, wood to biopolymers. Within the department, a strong focus is put on advanced processing technologies such as additive manufacturing, detailed material characterisation such as computer tomography and advanced materials testing.

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.	FAU	Friedrich-Alexander Universität Erlangen-Nürnberg, Germany
4.	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
	- IKTS	Fraunhofer Institute for Ceramic Technologies and Systems, Dresden
5.	IK4-TEKNIKER	Foundación TEKNIKER, Eibar, Spain
6.	IMBAS	Institute of Mechanics, Bulgarian Academy of Sciences, Sophia, Bulgaria
7.	IMIM	Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Krakow, Poland
8.	IMRSAS	Institute of Materials Research, Slovak Academy of Sciences, Kosice, Slovakia
9.	INTA	Instituto Nacional de Técnica Aeroespacial, Torrejón de Ardoz, Spain
10.	Ł-KIT	Łukasiewicz Research Network - Krakow Institute of Technology, Kraków, Poland
11.	IPM	Institute of Physics of Materials, Brno, Czech Republic
12.	IPPT	Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland
13.	ITC	Instituto de Tecnología Cerámica - AICE, Castellón, Spain
14.	Ł-IMIF	Łukasiewicz Research Network - Institute of Microelectronics and Photonics, Warsaw, Poland
15.	MCL	Werkstoff-Kompetenzzentrum-Leoben Forschungsgesellschaft m.b.H. (Materials Centre Leoben), Leoben, Austria
16.	PG	Gdansk University of Technology, Gdańsk, Poland
17.	POLITO	Politecnico di Torino, Torino, Italy
18.	R-TECH	Steinbeis Advanced Risk Technologies GmbH, Stuttgart, Germany
19.	TECNALIA	Fundación Tecnalia, Donostia-San Sebastian, Spain
20.	TUD	Technische Universität Darmstadt, Darmstadt, Germany
21.	TUG	Graz University of Technology, Graz, Austria
22.	тим	Technische Universität Wien, Wien, Austria
23.	UH	University of Hertfordshire, Hatfield, Herts, UK
24.	UNIVPM	Università Politecnica delle Marche, Ancona, Italy
25.	UPM	Universidad Politécnica de Madrid, Madrid, Spain
26.	WUST	Wroclaw University of Science and Technology, Wroclaw, Poland
27.	WUT	Warsaw University of Technology, Warsaw, Poland

Individual members

1.	Katarzyna Pietrzak	Warsaw, Poland
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- 2. Michał Basista Warsaw, Poland
- 3. Krzysztof Doliński Warsaw, Poland
- 4. Michał Urzynicok Koszęcin, Poland
- 5. **Peter Hansen** St Albans, UK

KMM-VIN Associate Members

Institutions

1.	BEG	Böhler Edelstahl GmbH & Co KG, Kapfenberg, Austria
2.	BSGA	Böhler Schweißtechnik Austria GmbH, Kapfenberg, Austria
3.	CSM	Centro Sviluppo Materiali S.p.A., Rome, Italy
4.	GE Power	General Electric Power Ltd, Rugby, UK
5.	UNIMORE	Università degli Studi di Modena e Reggio Emilia, Italy
6.	UNIPER	Uniper Technologies Limited., Coventry, UK
7.	ENS P-S	Ecole Normale Supérieure Paris-Saclay, France
8.	ETD	European Technology Development Ltd, UK
9.	EU-VRi	European Virtual Institute for Integrated Risk Management, Stuttgart, Germany
10.	GSC Ltd	Goodwin Steel Castings Ltd, Hanley, UK
11.	IMSETHC	Institute of Metal Science, Equipment and Technologies with HydroAerodynamics Centre of the Bulgarian Academy of Sciences, Sofia, Bulgaria
12.	Ł-IS	Łukasiewicz Research Network-Instytut Spawalnictwa, Gliwice, Poland
13.	LU	Loughborough University, Loughborough, UK
14.	MPA	Materialprüfungsanstalt Universität Stuttgart, Germany
15.	NUIG	National University of Ireland, Galway, Ireland
16.	SIEMENS Energy	Siemens Energy Global GmbH & Co. KG, Muelheim/Ruhr, Germany
17.	SSF	Saarschmiede GmbH Freiformschmiede, Völklingen, Germany
18.	SWG	Schmiedewerke Gröditz GmbH, Gröditz, Germany
19.	UCM	Universidad Complutense de Madrid, Spain
20.	UL	University of Limerick, Limerick, Ireland
21.	UNIUD	University of Udine, Udine, Italy
22.	VAGL	Voestalpine Giesserei Linz GmbH, Linz, Austria
23.	VTT	VTT Technical Research Centre of Finland, Espoo, Finland
24.	VZU	Výzkumný a zkušební ústav Plzeň s.r.o., Plzeň, Czech Republic

Individual members

1. Peter Mayr Munich, Germany

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