# 7th Dresden Nanoanalysis Symposium

"Nano-scale characterization for cutting-edge materials research and sustainable materials development"

Abstract booklet

August 30th, 2019
Dresden, Germany

## Symposium Sponsors

# 7th DRESDEN NANOANALYSIS SYMPOSIUM

## "Nano-scale characterization for cutting-edge materials research and sustainable materials development"

The 7th Dresden Nanoanalysis Symposium, organized by the Dresden Fraunhofer Cluster Nanoanalysis (DFCNA), supported by the European Materials Research Society (E-MRS) and the European Materials Characterisation Council (EMCC), will be held at the Fraunhofer Campus Dresden, Maria-Reiche-Strasse 2, on August 30, 2019. It will provide highlights in the field of materials characterization, represented by invited talks and poster sessions. In addition, we will offer to visit institutes. In this year, the symposium will have the particular motto: "Nano-scale characterization for cutting-edge materials research and sustainable materials development".

Based on the positive experience in the previous years, we would like to go ahead with the same format: One-day symposium with **3 keynote talks**, **9 invited talks** in 3 sessions, and a **poster session for contributed papers**. There will be **exhibition space** available for companies and organizations. The symposium will cover the topics of nanoanalysis and materials characterization along the whole value and innovation chain, from fundamental research up to industrial applications. It will bring scientists and engineers together from universities, research institutions, equipment manufacturers and industrial end-users. New results in disruptive nanoanalysis techniques will be reported in several talks and in the poster sessions, and novel solutions in the field of materials characterization for process and quality control will be shown. The discussions and interactions between the stakeholders will help to identify gaps in the fields of advancing nanoanalysis and materials characterization and to propose actions to close them and to support industrial exploitation of innovative materials. The symposium is supposed to reinforce ongoing collaborations and to discuss ideas for new collaborations.

## Venue

August 30th, 2019, Dresden, Germany
Fraunhofer IKTS Dresden, Maria-Reiche-Strasse 2, 01109 Dresden

## Organizational committee

Kristina Kutukova, Fraunhofer IKTS, *kristina.kutukova@ikts.fraunhofer.de*
Wieland Heyn, Fraunhofer IKTS, *wieland.heyn@ikts.fraunhofer.de*
Elke Göring, InnoTec21 GmbH, *elke.goering@innotec21.de*

*Symposium organized by Dresden Fraunhofer Cluster Nanoanalysis and Dresden Center for Nanoanalysis at TU Dresden*

*Symposium management*
*Innotec21 GmbH*

## Symposium Chair:

Ehrenfried Zschech, Fraunhofer IKTS Dresden and Dresden University of Technology, Germany

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- Giovanni Cuniberti, Technische Universität Dresden, Dresden (Germany)
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- Gerd Schneider, Helmholtz-Zentrum, Berlin (Germany)
- Marco Sebastiani, University Roma Tree (Italy)
- Robert Sinclair, Stanford University, California (USA)
- Olivier Thomas, University Marseille (France)
- Wilfried Vandervorst, IMEC, Leuven (Netherlands)
- Oden Warren, Bruker, Minnesota (USA)
- Thomas Weißgärber, Fraunhofer IFAM, Dresden (Germany)

## Invited speakers

- Yury Gogotsi, Director of A.J. Drexel Nanomaterials Institute and Charles T. and Ruth M. Bach Distinguished University Professor, Philadelphia/PA, USA
- Elvira Fortunato, Director of CENIMAT - Centre for Materials Research and Professor at Materials Science Department, FCT, Universidade NOVA de Lisboa, Portugal
- Jaroslav Klima, Chairman of the Board & CEO, TESCAN ORSAY HOLDING, Brno, Czech Republic
- Xinliang Feng, Technische Universität Dresden, Germany
- Georg Haberfehlner, Graz University of Technology, Austria
- Josef Keckes, Montanuniversität Leoben, Austria
- Joanna Wojewoda-Budka, Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Cracow, Poland
- Manon Letiche, Institut Laue-Langevin (ILL) Grenoble, France
- Robert Sinclair, Stanford University, Palo Alto/CA, USA
- Olivier Thomas, Aix Marseille Université, France
- Paula Vilarinho, University of Avairo, Portugal
- Oden Warren, Bruker, Minneapolis/MN, USA

# Program

## Friday, August 30

***Coffee / Registration (8:30 am – 9:00 am)***

***Welcome (9:00 am – 9:10 am)***

Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

***Session 1 (9:10 am – 10:40 am) – Session chair: Ehrenfried Zschech***

**9:10 am – 9:40 am**

**KEYNOTE TALK**

**"Electronic Properties of 2D Transition Metal Carbides and Nitrides (MXenes) "**

Yury Gogotsi, Director of A.J. Drexel Nanomaterials Institute and Charles T. and Ruth M. Bach Distinguished University Professor, Philadelphia/PA, USA

**9:40 am –10:10 am**

**KEYNOTE TALK**

**"Metal oxide materials as a sustainable alternative to low cost and flexible electronics "**

Elvira Fortunato, Director of CENIMAT - Centre for Materials Research and Professor at Materials Science Department, FCT, Universidade NOVA de Lisboa, Portugal

**10:10 am –10:40 am**

**KEYNOTE TALK**

**"Multiscale 3D characterization using X-ray and charged particle based microscopy"**

Jaroslav Klima, Chairman of the Board & CEO, TESCAN ORSAY HOLDING, Brno, Czech Republic

***Coffee break and poster session (10:40 am – 11:10 am)***

***Session 2 (11:10 am – 12:50 pm) – Session chairs: Olivier Thomas***

**11:10 am –11:30 am**

**"Organic 2D materials: Challenges and opportunities for nanoanalysis "**

Xinliang Feng, Technische Universität Dresden, Germany

**11:30 am –11:50 am**

**"Nano-scale characterization of lead free potassium sodium niobate (KNN) single crystals and polycrystals towards sustainable nonlinear dielectrics "**

Paula Vilarinho, University of Avairo, Portugal

**11:50 am –12:10 pm**

**"Advanced nanomechanical characterization for industrial applications "**

Oden Warren, Bruker, Minneapolis/MN, USA

**12:10 pm –12:30 pm**

**"Reactivity close and far from the equilibrium state in various joining processes"**

Joanna Wojewoda-Budka, Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Cracow, Poland

**12:30 pm –12:50 pm**

**"Advanced neutron characterisations techniques apply to micro (nano) technologies"**

Manon Letiche, Institut Laue-Langevin (ILL) Grenoble, France

***Lunch break and poster session (12:50 pm – 2:20 pm)***

***Session 3 (2:20 pm – 3:40 pm) – Session chairs: Malgorzata Lewandowska***

***2:20 pm – 2:40 pm***

**"Correlative studies of surface enhanced Raman spectroscopy with plasmon energies in photolithographically designed nanoparticles as determined by STEM-EELS "**

Robert Sinclair, Stanford University, Palo Alto/CA, USA

***2:40 pm – 3:00 pm***

**"Nanoscale 3D measurements by electron tomography "**

Georg Haberfehlner, Graz University of Technology, Austria

***3:00 pm – 3:20 pm***

**"Stress gradient characterization in thin films using nano X-ray diffraction"**

Josef Keckes, Montanuniversität Leoben, Austria

***3:20 pm – 3:40 pm***

**"The amorphous-crystal transition in GeTe nanostructures: New insights from in situ synchrotron radiation measurements "**

Olivier Thomas, Aix Marseille Université, France

***Best poster award ceremony and closing remarks (3:40 pm – 4:10 pm)***

Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

***Coffee and cake, and poster session (4:10 pm – 5:30 pm)***

Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

***Poster sessions***

***10:40 am – 11:10 am***

***12:50 pm – 2:20 pm***

***4:10 pm – 5:30 pm***

## Poster presentations

# Abstracts
# -Talks-

# KEYNOTE TALK

## Electronic properties of 2D Transition Metal Carbides and Nitrides (MXenes)

Yury Gogotsi*

*A. J. Drexel Nanomaterials Institute and Department of Materials Science and Engineering, Drexel University, Philadelphia, PA, 19104, USA, http://nano.materials.drexel.edu*

**e-mail: googtsi@drexel.edu*

2D transition metal carbides and nitrides (MXenes) are a large family of 2D materials with more than 30 compos itions experimentally synthesized and a few dozens more predicted to be stable and studied computationally. The y benefit from their high electrical conductivity, wide range of optical properties, hydrophilic surface, and high m echanical strength. Because of those properties, they show promise in a wide a variety of applications from energ y storage to photonics. In this talk, an overview of fundamental electronic properties of MXenes are presented an d discussed. Specifically, the roles of intra- and inter-flake electronic conduction on the temperature-dependence of resistivity of MXenes will be discussed in detail. Moreover, systematic investigation of the roles of MXene co mposition, including the transition metals, the X elements (C and/or N), and surface terminations on their electro nic and transport properties will be presented.

Dr. Yury Gogotsi is Charles T. and Ruth M. Bach Chair Professor and Distin guished University Professor of Materials Science and Engineering at Drexel University in Philadelphia, USA. He also serves as Director of the A.J. Drex el Nanomaterials Institute. His research group works on 2D carbides and nitr ides (MXenes), nanostructured carbons, as well as other nanomaterials for en ergy, water and biomedical applications. He has co-authored 2 books, 16 boo k chapters, about 700 journal papers, edited 14 books, and obtained more tha n 50 patents. He was recognized as Highly Cited Researcher in Materials Sci ence and Chemistry (Web of Science) in 2014-2018. He has received numero us national and international awards for his research. He also serves on the M RS Board of Directors and acts as Associate Editor of ACS Nano.

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# KEYNOTE TALK

## Metal oxide materials as a sustainable alternative to low cost and flexible electronics

Elvira Fortunato*

*CENIMAT/i3N Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologia (FCT), Universidade NOVA de Lisboa (UNL), and CEMOP/UNINOVA, 2829-516 Caparica, Portugal*

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Metal oxide semiconductors are a good example as a success story in the area of thin film electronics, since it took less than 10 years after the discovery until the commercialization of the first products mainly in the area of displays. The main advantages of these materials are the low temperature processability, the high mobility and the uniformity over large areas, since they present an amorphous structure.

Nevertheless to decrease costs associated to electronic devices a strategy is using cheap and abundant materials in conjunction with low cost fabrication methods, associated to an overall increase of electrical performance. This is why metal oxide semiconductors are the key materials since they are chemically stable, mostly non-toxic and abundant materials, often manufactured by low cost methods, under ambient conditions. Consequently, devices made of metal oxides are inexpensive, very stable and environmentally safe, the 3 most important requirements for electronics.

Despite being explored for more than a century for electronic applications, from the initial works of Badeker in 1907 with CdO to the cutting edge IGZO available these days in active matrix backplanes of flat panel displays, oxides still present an exceptional and innovative combination of properties not achievable by any other material class. In fact, they are true multifunctional materials, being able to exhibit optical transparency, conducting / semiconducting / insulating behaviour, piezoelectricity and catalytic or self-cleaning properties among many others.

In this presentation we will review some of the most promising new technologies based on oxide conductors, semiconductors, dielectrics as well as electrochromic devices either in the form of nano-films or nanoparticles, and we will summarize the major milestones already achieved with this emerging and very promising technology focused on the work developed in our laboratory.

By using these materials and technologies we are contributing to the evolution of environmentally conscious electronics that is able to add new electronic functionalities onto surfaces, which currently are not used in this manner.

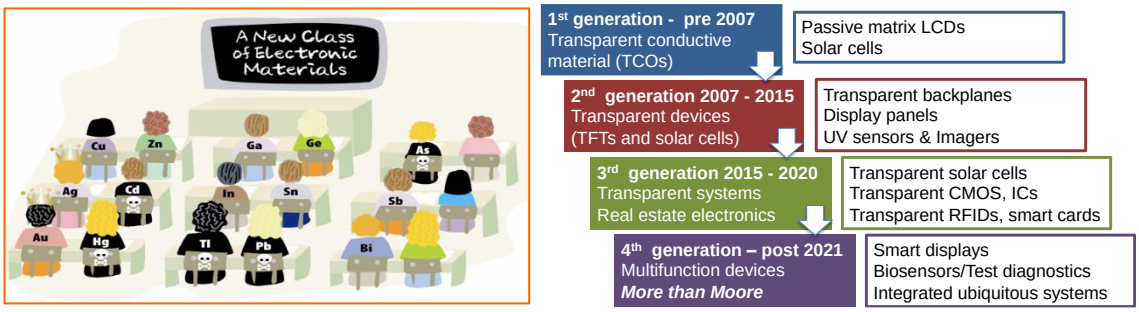


**Figure 1.** A comparison between a classroom and the new class of electronic materials based on metal oxides, by Prof. J. Wager from Oregon State University and the evolution of metal oxide materials..

**Acknowledgments**

This work is funded by ERC AdG project DIGISMART ref. 787410.

**References**

[1] E. Fortunato, P. Barquinha, and R. Martins, "Oxide Semiconductor Thin-Film Transistors: A Review of Recent Advances," Advanced Materials, vol. 24, pp. 2945-2986, Jun 12 2012.

[2] P. Barquinha, R. Martins, L. Pereira and E. Fortunato, Transparent Oxide Electronics: From Materials to Devices. West Sussex: Wiley & Sons (March 2012). ISBN 9780470683736.

# KEYNOTE TALK

## Multiscale 3D characterization using X-ray and charged particle-based microscopy

Jaroslav Klima*

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Users of tools for visualization of micro- and nano- objects require more and more complete/complex information about the studied objects. Besides 2D and 3D as well as time-lapse images for exploration of dynamic processes, also information about the element composition, chemical bonds, crystallinity, electrical and magnetic properties, characterization of proteins, etc. are required. Moreover, the users' interest has been moved to higher lateral and spatial resolution as well as to more precise analytical results. Besides this, the non-destructive imaging and analysis are often required, and high throughput has become one of the most required parameters - not only in industrial and clinical laboratories but also at research institutions. Companies providing high-end scientific instrumentation have been challenged to move from specific HW/SW to dedicated application workflows and even to multiple instruments in one workflow (complete laboratory) over the decade.

This all motivates suppliers to offer wide portfolio of instruments as well as to employ and cultivate strong application teams. It is one important factor contributing to acceleration of the process of R&D and integration of manufacturing capacities as well as to accumulation of capital.

In this contribution a portfolio of instruments and string of operations for failure analysis in semiconductor market segment will be demonstrated. Moreover, some other applications in Materials as well as in Live Sciences will be discussed including the highly demanded cryo-technology.

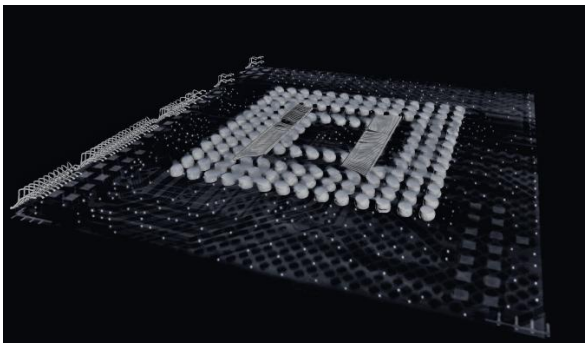

**Figure 1.** Reconstructed 3D CT image of Exynos 8995 chip.

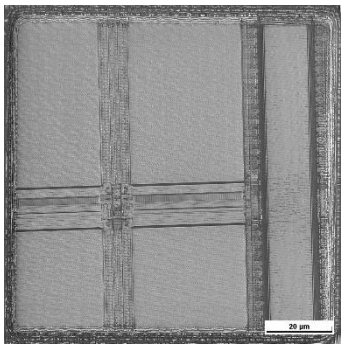


**Figure 2.** 500 eV SEM image of 100 × 100 µm window opened by Xe Plasma FIB-Gas Assisted Etching delayering. (Scale of 20 µm)

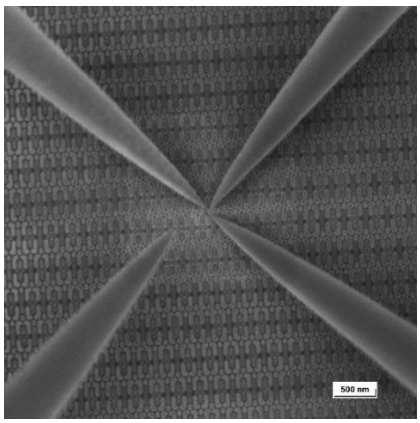


**Figure 3.** 500 eV SEM image of SRAM cells with nanoprobes landed on the bit-cell contacts (Scale of 500 nm)

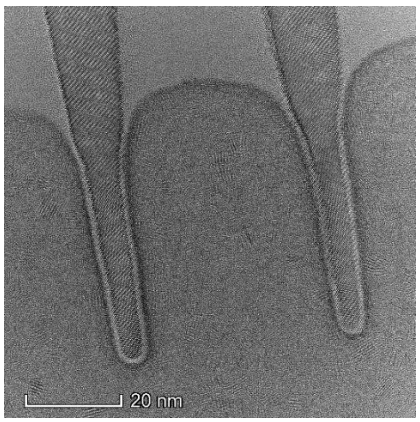


**Figure 4.**TEM image of lamella depicting Fin-FET from 10 nm Exynos chip

# Towards a world of organic 2D materials

Xinliang Feng*

*Center for Advancing Electronics Dresden & Faculty of Chemistry and Food Chemistry, Technische Universitaet Dresden, Germany*

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**Abstract**

In this lecture, we will present our recent efforts on the bottom-up synthetic approaches towards novel organic 2D materials with structural control at the atomic/molecular-level or at the meso-scale. First, we will introduce the latest development on the synthetic 2D conjugated polymers including 2D Schiff-base type covalent polymers and 2D metal-dithienene/diamine coordination polymers at the air-water or liquid-liquid interfaces. The resulting 2D conjugated polymers exhibit single- to multi-layer feature, good local structural ordering and with a large size. The functional exploration of such 2D conjugated (coordination) polymers for the electrical, magnetic and mechanical properties, as well as serving as efficient electrocatalytic water splitting catalysts will be demonstrated. Next, we will introduce the self-assembly of a host-guest enhanced donor-acceptor interaction, toward monolayers of 2D supramolecular polymers at liquid-liquid interface. Third, we will present the supramolecular approaches to synergistically control the multi-component assembly, which results into 2D conducting polymers, such as polypyrrole and polyaniline nanosheets featuring 2D structures and with adjustable mesopores with/without on various functional free-standing surfaces. The unique structure with adjustable pore sizes (5–20 nm) and thickness (35–45 nm), enlarged specific surface area as well as high electrical conductivity make 2D conducting polymers promising for a number of applications. Finally, we will present a controlled synthesis of few-layer 2D polymer crystals on the water surface assisted by soft templates. The future perspective and outlook regarding the goal towards highly crystalline organic 2D materials will be also provided.

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# Nano-scale characterization of lead-free potassium sodium niobate (KNN) single crystals and polycrystals towards sustainable non-linear dielectrics

Paula Maria Vilarinho*

*Department of Materials and Ceramic Engineering, CICECO – Aveiro Materials Institute, University of Aveiro, Campus Universitário, 3810 – 093, Aveiro, Portugal*

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**Abstract**

By non-linear dielectrics we understand, from a simplistic point of view, materials in which the relation between electrical displacement (D) and electric field (E) is not linear. By far the most important non-linear materials are ferroelectrics and piezoelectrics. The piezoelectric devices market is expected to worth USD 31.33 Billion by 2022, growing at a CAGR of 4.88% between 2016 and 2022 [1]. The driving factors for this growth comprise emerging energy harvesting techniques, rising demand in automotive sector, healthcare industry and miniaturization technology [1]. Piezoceramics are the most widely used and commercially accepted piezoelectric materials as they exhibit some of the largest displacements or induce some the largest electric voltages. Due to exceptional figures of merit lead-based piezoelectrics (typified by $PbZr_{1-x}Ti_xO_3$) (PZT) are still the dominant materials family, however strong health restrictions towards poisonous elements as lead, are triggering the search for substitutes. Though not yet fully comparable with the performance of PZT, among lead-free piezoelectrics lies potassium sodium niobate, ($K_{0.5}Na_{0.5}NbO_3$) (KNN) that owing to a high phase transition temperature Tc≈420 ºC, good piezoelectric properties and electromechanical coupling coefficient is a promising lead-free alternative. The electromechanical response is very dependent on the crystalline phase content, crystallographic orientation, microstructure, interfaces, but also on domain configuration at the nanosize level. Piezoforce response microscopy (PFM) is currently one of the most powerful techniques to image and study domains and ferroelectric properties at the nanoscale. In this talk we disclose our systematic investigation of the domain configuration of KNN single crystals and polycrystals at the nanoscale, we establish its relation to the macroscopic electrical properties and review the most relevant advancements on the topic. Based on our insights [2-6] we may expect further improvements of the properties of KNN, contributing to its prospect practical applications.

**References**

[1] Piezoelectric Devices Market - Global Forecast to 2022, by Markets and Markets.

[2] Muhammad Asif Rafiq, M. Elisabete V. Costa, and Paula M. Vilarinho, Establishing the Domain Structure of K0.5Na0.5NbO3 (KNN) Single Crystals by Piezoforce-Response Microscopy, *Science of Advanced Materials*, *6*, 426–433, **2014**.

[3] Muhammad Asif Rafiq, Alexander Tkach, Maria Elisabete Costa, Paula Maria Vilarinho, Defects and charge transport in Mn-doped K0.5Na0.5NbO3 ceramics, Physical Chemistry Chemical Physics, 17, 24403, 2015.

[4] Muhammad Asif Rafiq, Maria Elisabete Costa, Paula Maria Vilarinho, Pairing High Piezoelectric Coefficients, d33, with High Curie Temperature (TC) in Lead-Free (K,Na)NbO3, ACS *Applied Materials and Interfaces*, 8, 33755−33764, **2016**.

[5] Alexander Tkach, André Santos, Sebastian Zlotnik, Ricardo Serrazina, Olena Okhay, Igor Bdikin, Maria Elisabete Costa, Paula M. Vilarinho, Strain-Mediated Substrate Effect on the Dielectric and Ferroelectric Response of Potassium Sodium Niobate Thin Films, *Coatings 8*, 449, **2018**.

[6] Rui Pinho, Sebastian Zlotnik, M. Elisabete Costa, Jacques Noudem, Ian M. Reaney, Paula M. Vilarinho, Spark Plasma Texturing: a strategy to enhance the electro-mechanical properties of lead-free potassium sodium niobate ceramics (KNN), submitted.

# Advanced nanomechanical characterization for industrial applications: High-throughput screening of HEAs by XPM augmented with AI

Douglas D. Stauffer, Oden L. Warren*

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**Abstract**

Nanoindentation and derivative techniques are rapidly gaining usage by industry. Today, Bruker Hysitron nanoindenters are as likely to be procured by companies as by universities and national labs. Given that industry and academic institutions have very different ideas about return on investment, why are we now seeing adoption by the corporate world on par with academia? The most dominant driver for this is the tremendous recent improvement in throughput, not just in test throughout but also in analysis throughput. On the test side, the big leap is accelerated mechanical property mapping (XPM),[1] performing up to 6 indents per second in a grid pattern if using Bruker Hysitron equipment. On the analysis side, the major advancements will likely rely on artificial intelligence, i.e., AI. Our initial AI efforts have reduced the time of analysis for large mapping datasets from hours to minutes with almost no reliance on human input or training algorithms.[2] This presentation will focus on the use of XPM to increase the experimental throughput for high-entropy alloys (HEAs), followed by the exploitation of AI clustering algorithms, to quickly extract reliable screening results from the dense mechanical property maps.[3] The outlook for high-throughput small-scale mechanical property investigations in industrial settings will also be discussed.

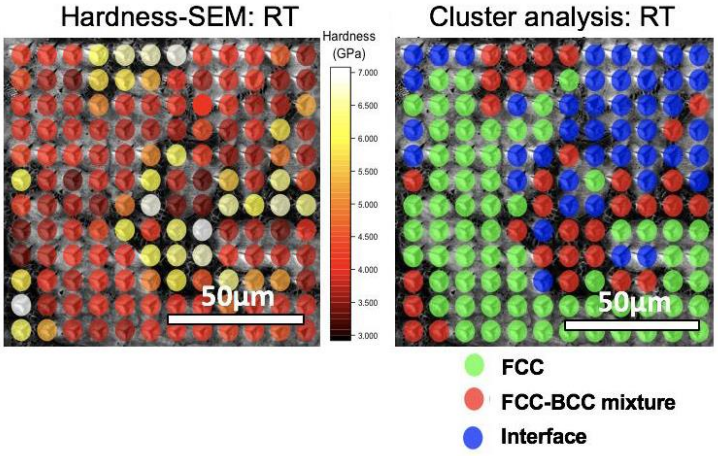


**Figure 1.** Hardness of HEA with Al of x=0.3 overlaid on SE micrograph. Normalized hardness and modulus used as clustering parameters to produce cluster overlay from same dataset.

**Acknowledgments**

We thank Youxing Chen, Nathan A. Mara, Bernard R. Becker, and Eric D. Hintsala for their contributions to this research.

**References**

[1] E.D. Hintsala, U. Hangen, D.D. Stauffer, *JOM* **2018**, *70*, 494.
[2] F. Pedregosa *et al.*, *JMLR* **2011**, *12*, 2825.
[3] Y. Chen *et al.*, "High-throughput nanomechanical screening of phase-specific and temperature-dependent hardness in AlxFeCrNiMn high entropy alloys," *JOM* **2019**, submitted.

# Reactivity close and far from the equilibrium state in the various joining processes

Joanna Wojewoda-Budka*, Anna Wierzbicka-Miernik, Izabella Kwiecień, Paweł Zięba

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**Abstract**

The sequence and morphology of the intermetallic phases growing during the formation of Cu/In-48Sn/Cu interconnections intended to the electronic industry are an example of the effective use of so-called diffusion path. The samples were produced using the diffusion soldering technology being an undemanding and functional tool, when the joint consisted solely of an intermetallic phase(s) is desired to be the final product. The diffusion path drawn at the isothermal cross section of the ternary equilibrium phase diagram represents the average changes of the chemical composition across the interconnection zone. Based on the simple rules the microstructure of such bonded zone can be predicted. However, there are also interesting examples of morphologies created due to the diffusion in the solid state, where the periodic layers consisting of an alternating single-phase or two-phase layers are created. The diffusion path in this case can be visualized as forming many loops crossing the same phase field. Several mechanisms were proposed to explain this peculiar morphology and based on the $Mg/SiO_2$ and $Zn/Co_2Si$ diffusion couples examples they will be discussed. On the other hand, the reaction of widely applied Electroless Nickel Immersion Gold (ENIG) coatings commonly used in the electronics industry with the solder can serve as an example of the reaction going far from the equilibrium state. Two types of the metallization layer: classical Ni-P and with the rhenium addition were studied to reveal how they are transformed after reaction with liquid tin, which constitutes the main component of the currently used lead-free solders. Finally, the explosive welding joining method will be presented, where the extreme conditions of pressure and temperature lead to the formation of the stable and metastable phases of various morphologies within the melted regions located at the interface area. These welds often must go to stress relief annealing, and this in turn causes the growth of the intermetallic phases, which varies strongly from the classical growth observed in diffusion soldering experiment.

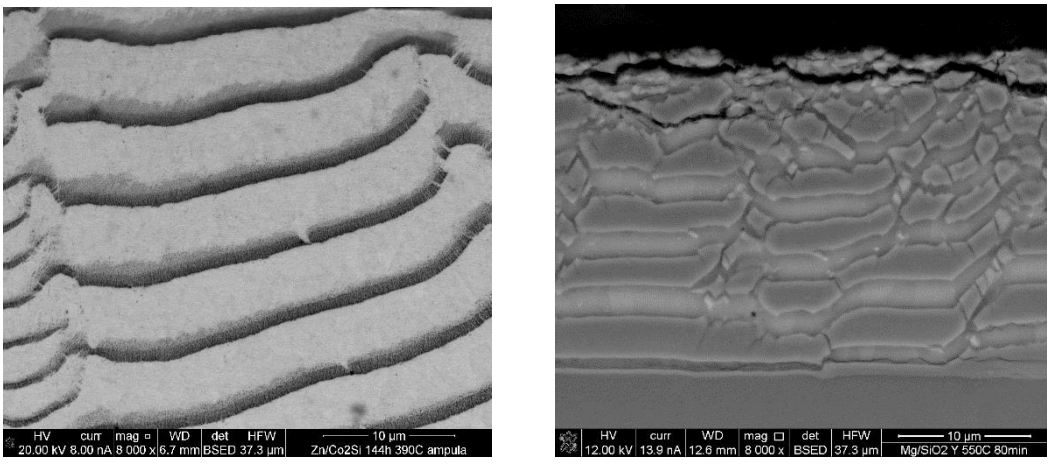


**Figure 1.** The microstructures of the periodic patterns formed in $Zn/Co_2Si$ (left) and $Mg/SiO_2$ (right) diffusion couples.

**Acknowledgments**

The research on the periodic layered morphology was financed by the National Science Centre, Poland under the project No. 2014/15/B/ST8/00195.

# Advanced neutron characterization techniques applied to micro technology

Létiche M.[*1], J. Segura-Ruiz[1], P. Gutfreund[1], R. Cubitt[1], D. Honecker[1], T. Mourier[2], F. Founel [2], E. Hadden[1], R. Varela[1], J. Beaucour[1]

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[2]*CEA LETI, Minatec Campus, F-38054 Grenoble, France*

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**Abstract**

Within the booming of nanotechnologies in the miniaturization process race, semiconductors have to be smaller and exhibit greater performances. These performances are tightly connected with the properties of different materials and their organization in three-dimensional space. Understand the material behavior at every step of the fabrication can be very challenging. Large-scale facilities, including synchrotron and neutron center, can undo technological and scientific locks by their complementarity. The scheme Fig1 shows what technic can be used to solve specific issues. Two examples of the use of neutron technics will be shown and discussed:

1. The use of Neutron Reflectometry (NR) to characterize the presence of Hydrogen at interfaces, which can prevent delamination risks [1].
2. The use of Small Angle Neutron Scattering (SANS) to statistically characterize micro defects in Through Silicon Vias (TSV).

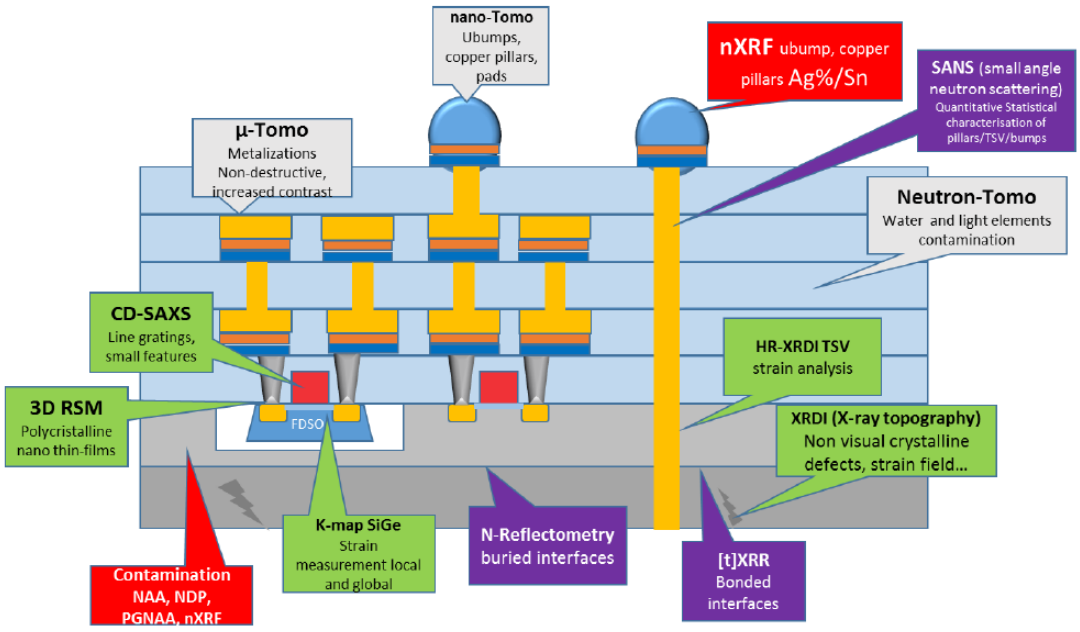


**Figure 1.** Scheme showing which neutron or synchrotron technique can be used to characterize a CMOS at every step of the process.

**Acknowledgments**

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# Correlative studies of surface snhanced raman spectroscopy with plasmon energies in lithographically designed nanoparticles as determined by STEM-EELS

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**Abstract**

There are various ways in which nanotechnology can assist in early cancer detection, oftentimes utilizing sensitive physical properties of nanomaterials to detect them attached to tumors or circulating cancer cells. The group with whom we collaborate has successfully employed gold nanoparticles contained within a silica shell as a triple modality detection agent [1]. One of the properties utilized is surface-enhanced Raman spectroscopy (SERS) whereby the gold nano-spheres significantly enhance the Raman signal from an organic dye when exposed to an illuminating laser beam, which has been incorporated into a working endoscopic system [2]. While this works well, to our knowledge there has never been any systematic study as to the influence of nanomaterial structure parameters such as size, shape, seperation, coating etc. on the strength of the Raman signal, and hence it's utility in detecting small tumors. It is recognized that surface plasmons in noble metal nanoparticles directly contribute to the Raman signal, and that the surface plasmon energy is in turn determined by the nanomaterial properties mentioned above. In this paper, we describe an approach to study the effect of these parameters in order to establish the optimum conditions to generate the highest possible Raman signal.

An array of gold nanoparticles of various size, shape and separation is fabricated from a vapor-deposited gold thin film utilizing standard electron lithographic processes. When a Raman dye is spread over the array, Raman imaging shows the variations of signal and hence the parameters giving rise to maximum signal. Nanoparticle size is seem to be a critical feature. The plasmon resonances and energies are then determined across the array using electron energy loss spectroscopy (EELS) in a scanning TEM (STEM) [3] and the individual spectra are then correlated with the Raman signal from the exact same nanoparticle structures. By this procedure we can establish the critical parameters which yield the highest Raman signal, which leads to the systematic design of the most effective SERS nanoparticles.

**Acknowledgments**

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# Nanoscale 3D measurements by electron tomography

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**Abstract**

Electron tomography extends the capabilities of a (scanning) transmission electron microscopy from two to three spatial dimension, making possible 3D materials characterization at the nano- down to the atomic scale.
ADF STEM tomography provides contrast depending on the atomic number and can thereby be used to distinguish regions with differences in local mass. Here we investigate an Mo/$B_4C$ multilayer system, as used for X-ray optics. With tomography, the buried interfaces between the different materials can be reconstructed throughout the stack and analyzed for their roughness in the sub-nanometer range (Fig. 1a).
Both electron energy-loss spectroscopy (EELS) and energy-dispersive X-ray spectroscopy (EDXS) allow mapping of chemical variations and gradients, approaching the goal of full 3D elemental quantification [1]. This has been used for example to reveal diffusion process forming ordered precipitates in a multicomponent (Al-Mg-Sc-Zr) alloy [2]. In these core/shell precipitates, spectroscopic tomography revealed inhomogeneous aluminum concentration within the shell, with a maximum amount of Al at the core/shell interface (Fig. 1b).
Finally, we will discuss recent developments on reconstruction algorithms, which employ the correlation between different elemental maps and ADF data [3]. This favors similar interface positions for all reconstructions, significantly improving the quality of 3D elemental maps.

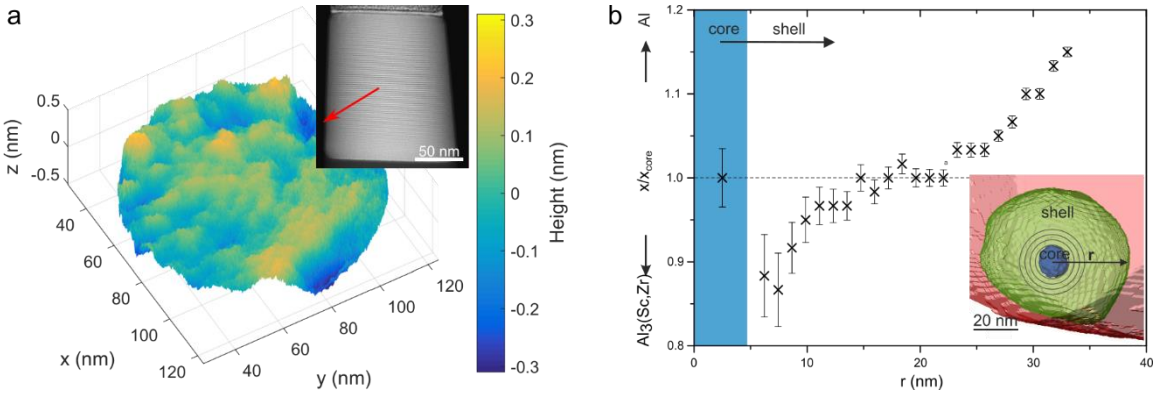


**Figure 1.** (a) ADF STEM projection of a Mo/B4C multilayer (inset) and reconstruction of one interface within the multilayer. (b) Changes of Al content in a core/shell particle within an Al-Mg-Sc-Zr alloy.

**Acknowledgments**

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# Stress gradient characterization in thin films using cross-sectional X-ray nano-diffraction

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**Abstract**

Nanocrystalline thin films may possess complex gradients of residual stresses, which originate (i) from self-organized film growth, (ii) from the intentionally varying deposition conditions and/or (iii) from the inhomogeneous thermal and/or mechanical loads induced during film service. In order to optimize the functional behavior of the thin films, it is necessary to assess the stress gradients at the nanoscale.

Introduced in 2012, cross-sectional synchrotron X-ray nanodiffraction (Figure 1) [1], using monochromatic X-ray beams with diameter down to ~30 nm provides representative depth-resolved data on the evolution of phases, crystallographic texture, grain morphology and strains/stresses [2] across thin film cross-sections. The aim of this contribution is to discuss methodological and instrumental aspects of the approach as well as to present recent achievements from experiments at beamlines ID13 of ESRF [3] and P03 of PETRA III. On the examples of hard nitride, diamond and metallic thin films, it will be demonstrated that the new approach can serve as an effective tool to characterize the inhomogeneous properties of as-deposited and thermally cycled thin films. The observed residual stress gradients can be correlated with the varying film deposition conditions, providing an opportunity to optimize thin film synthesis process. Additionally, results from strain and microstructure characterization in in-situ loaded films will be presented. Finally, an outlook, especially on in-situ experiments as well as an analysis of complex depth gradients of structure-property relationships in nanocrystalline thin films with even smaller X-ray beams, will be shortly discussed.

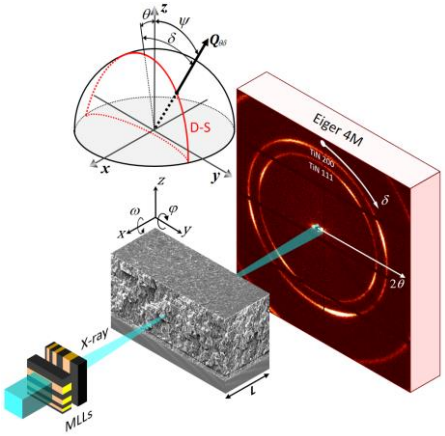


**Figure 1.** A schematic drawing of the CSnanoXRD setup. The experiments are performed in transmission diffraction geometry, where thin film lamella with a width of *L* is exposed to a X-ray nanobeam focused by crossed multilayer Laue lenses (MLLs). The specimen is moved along the *z* axis and diffraction data are collected using the Eiger X 4M 2D detector.

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# The amorphous-crystal transition in GeTe nanostructures: New insights from *in situ* synchrotron radiation measurements

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**Abstract**

Phase-change (PC) memories are now considered to be the most promising technology among emerging non-volatile resistive memories to replace the current Flash memories or to achieve innovative Storage Class Memory. Chalcogenides, such as GeTe and $Ge_2Sb_2Te_5$, can quickly and reversibly switch between an amorphous and a crystalline state with very different optical and electrical properties. These property changes allowed the integration of such chalcogenide materials in optical storage devices (DVD-RAM) and more recently in non-volatile memory technology (NVM) because of the high scalability of PC memories. But, for ultimate miniaturization, energy consumption becomes critical and a promising solution is the geometrical confinement of the memory points. Mastering this with Phase Change Materials at ultimate lateral dimensions (typically 5 nm) is, however, a real challenge, which calls for a fundamental understanding of the interplay between strain (the amorphous-to-crystal transition is accompanied by a density increase of several %) and crystallization kinetics at the nanoscale.

In order to probe the influence of size, stress and confinement on the crystallization behavior of GeTe nanostructures we have developed dedicated setups that allow for a simultaneous *in situ* investigation of substrate curvature or electrical resistance measurement together with x-ray diffraction or x-ray reflectivity during annealing. These measurements are performed at DiffAbs beamline from SOLEIL synchrotron radiation facility. Samples are either capped thin films (thickness ranging between 100 and 5 nm) or arrays of nanopillars (diameter ranging between 200 and 50 nm) embedded in silicon nitride. Clear evolutions in crystallization behavior, crystallization kinetics or stress evolutions are observed as a function of size and confinement and will be discussed.

**Acknowledgments**

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# Abstracts
# -Posters-

# The effect of Co and Cu addition on martensitic transformation temperature and crystal structure in Ni-Mn-Ga Heusler alloys

**P01**

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**Abstract**

The ternary Ni-Mn-Ga Heusler alloys have attracted high interest over past few decades due to their functional properties such as conventional shape memory, magnetocaloric effect and the magnetic-field-induced strain (MFIS). MFIS effect is based on twin variant rearrangement realized by twin boundary motion. The lattice parameters of the martensitic structure define the maximal theoretical MFIS described as 1-c/a. Moreover, by introducing fourth element to ternary system, which modifies the unit cell by decreasing its tetragonality (c/a), it is possible to reduce the twinning stress. Also, the martensitic transformation temperature is very sensitive to the chemical composition of an alloy, in contrast the Curie temperature is not so much composition dependent. Due to chemical modification, it is possible to control material properties necessary for industrial applicationThe two series of polycrystalline alloys were manufactured under argon protective atmosphere from high purity elements using conventional arc melting method. The nominal composition for the first series of alloys were $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ (x=1-10 at.%) and for the second $Ni_{55-y}Mn_{25}Ga_{20}Co_y$ (y=0,5,10 at.%). Subsequently, the buttons were sealed into quartz ampoules at vacuum condition and further annealed at 900°C for 48h in order to obtain homogeneity of chemical composition and then one part was slowly cooled with furnace (FC) to ambient temperature when the other one was followed by water quenching (WQ). X-ray diffraction measurements were used to check the type of crystal structure of alloys at room temperature, in case of samples, where Ga atoms were replaced by Cu atoms, four types of crystal structure were detected: $L2_1$ austenite and 5-layered modulated, 7-layered modulated and non-modulated martensite. Addition of Co instead of Ni did not introduce changes in the type of crystal structure. The non-modulated martensite structure was detected in all of the examined alloys. However, increasing amount of Co modifies the lattice parameters and thus, affected the tetragonality of the martensite unit cell (c/a). The “a “ and “c” lattice parameters changed from 5.40 Å to 5.43 Å and from 6.69 Å to 6.58 Å, and the c/a ratio changed accordingly from 1.24 to 1.21 for 0 and 10 at% of Co, respectively. The microstructure evolution was observed using scanning electron microscope in a backscattered electron mode. The detected changes in morphology were in good agreement with the change of the type of crystal structure. Moreover, in alloys with 9 and 10 at.% of Cu content among the martensitic plates some new phases were observed. During increasing addition of Cu in alloy, the number and size of precipitates also increased. Further investigation of precipitates using energy-dispersive spectrometer map analysis revealed that they are enriched in Cu, which may indicate that the precipitation are so-called γ phase often found in Ni-Mn-Ga alloys. Differential scanning calorimetry were used to determine the characteristic transformation temperatures. In case of first series of alloys, the replacement of Ga by Cu leads to an increase of martensitic transformation temperature, the opposite effect were observed for second series of alloys. This behavior is correlated with the number of valence electron concentration per atom (e/a). To calculate the e/a ratio for alloys, the number of valence electrons of Ni, Mn, Ga, Cu and Co were taken as: 10, 7, 3, 11 and 9, respectively. When Ga atoms were replaced by Cu atoms, the e/a ratio increases, however, substitution of Co by Ni decreases the e/a ratio as well as the martensitic transformation temperature. For both additives the water quenching process shifts the martensitic transformation temperature to lower values.

**Acknowledgments**

The experimental tests were performed in the Accredited Testing Laboratories at the Institute of Metallurgy and M aterials Science of the Polish Academy of Sciences.

# Effect of crystallographic orientation and temperature on superelastic strain of FeNiCoAlTa/ FeNiCoAlTaB single crystals

**P02**

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**Abstract**

Iron-based shape memory alloys such as Fe-28Ni-17Co-11.5Al-2.5Ta-0.05B and Fe-28Ni-17Co-11.5Al-2.5Ta, abbreviated NCATB/NCAT, show a large superelastic strain. However, the effect is strongly dependent on the precipitation hardening which is required to suppressed the deformation of the parent phase. The main strengthening component of these alloys is the *fcc* γ' phase. The size, chemical composition and crystal structure of particles can be efficiently tailored using one-stage or two-stage heat treatments. As a results optimal mechanical properties may be obtained. Both polycrystalline and single crystalline Fe-based alloys show a strong strain anisotropy. Additionally, tension strains exceed the theoretical values which can be predicted based on the lattice crystallography or lattice deformation theory. The unusual high stain could be explain by a subsequent intermartensitic transformation. Therefore, prior to mechanical tests the effect of heat treatment on the precipitation of three different intermetallic phases such as NiAl, Ni3Al and NiAl3 using synchrotron radiation is studied. Then, single crystals with <100> and <111> orientations are compressed at 77 K to provide an insight into the mechanism of superelasticity observed in these alloys. Magnetic studies were also carried out to determine the characteristic transformation temperatures. The obtained superelastic strain is also confront with the so-called stabilization effect shifting the As temperature to higher values. The stabilization is achieved by single variant of martensite. The results are discussed with respect to crystallographic orientation, deformation mode, precipitations and phase transformation.

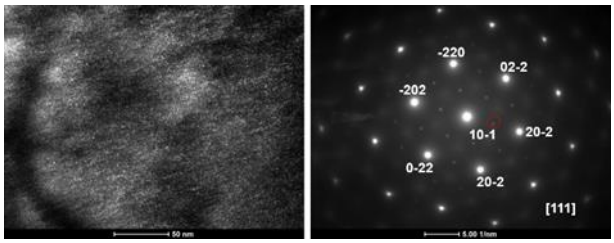


**Figure 1.** TEM/ DF Coherent $Ni_3Al$ type precipitates, annealed for 0.5 h at 973K.

**Acknowledgment**

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# Microstructural characterization and growth kinetics of the intermetallic phases in annealed Ni201/A1050 and A1050/Ni201 explosive clads

P03

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**Abstract**

Explosive welding (EXW) is a technology useful to join metals or alloys of strongly different chemical and physical properties. As a result of explosion, taking place on the top of the flyer plate (fp), it is accelerated with high velocity and collides with the base plate (bp). High pressure and local temperature increase in collision point, therefore, the durable bond consisting of strongly mixed and melted areas forms. Al-Ni is very interesting system due to the possibility of widespread use in industry, mainly in aircraft and electronics. Equilibrium phase diagram indicates the possibility of the formation of five intermetallic phases such as: $Al_3Ni$, $Al_3Ni_2$, AlNi, $Al_3Ni_5$ and $AlNi_3$ in this system. Nevertheless, explosive welding takes place under strongly non-equilibrium conditions, which may cause the creation of metastable phases such as $Al_9Ni_2$ or quasicrystals, due to the rapid cooling causing fast solidification of the reaction zone [1]. This study is dedicated to the microstructure characteristics of the interface zone using various scales of observation. The Ni201/A1050 explosively welded clads obtained in parallel system set-up were examined with both Scanning and Transmission Electron Microscopy techniques. They were involved to follow the microstructure and phase composition of the Ni201/A1050 interface formed in extreme conditions of pressure and locally temperature. Moreover, the neighborhood area after exposure to the joining conditions was compared to the microstructure of the initial materials. As a next step, the transformation of the weld associated with the growth of selected intermetallic phases after annealing was analyzed. As a first step, the morphology of the melted zones accompanied with the identification of the formed phases was conducted based on the chemical composition measured by Energy Dispersive X-ray Spectroscopy in SEM. To fully examine the reaction zones before and after annealing Transmission Electron Microscopy was applied. This approach allowed the nanoscale microstructure observations and local determination of phases existing in the state after welding. Series of samples were annealed at 500°C for various periods of time followed by the thickness measurements of formed intermetallic phases. Moreover, application of Electron Backscattered Diffraction technique revealed any possible crystallographic relations, size and shape of grains in joined materials close to the bonding zone as well as the character of the grain boundaries. It should be also emphasized that two configurations of the flyer plate localization were applied: type I - Ni 201(fp)/A1050(bp) and type II - A1050(fp)/Ni201(bp) , which could expose any possible differences of the joint in the state after explosive welding. This factor is important especially in multilayers, where various mutual localization of the Al and Ni alloys plates takes place in one sample. Explosive welds in the state after joining indicated wavy character of interfaces but the waves were different with respect to used detonation velocities. In the case of 2000 m/s the interface was nearly flat, while the Vd = 2400 m/s resulted in the formation of wavy boundary between joined alloys' plates. The interfaces of all examined clads after EXW consisted of melted areas, where intensive mixing of the formed phases took place. Additionally, within the melted zones, in the case of the highest detonation velocity, in addition to the stable phases, the presence of metastable phases was detected. Annealing at 500 °C caused the simultaneous growth of two rich in Al intermetallic phases, namely $Al_3Ni$ and $Al_3Ni_2$. With time the consumption of $Al_3Ni$ by $Al_3Ni_2$ took place and this was manifested with broadening of the second one. Beside above, the additional phase, enriched in Fe, located between Al and $Al_3Ni$ was formed with the composition of: 4.0 at. % Fe, 13.4 at. % Ni and 82.6 at. % Al.

**Acknowledgments**

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# Deformation and recrystallization behavior of plane strain compressed aluminum bicrystals with {100}<011>/{110}<001> orientations

P04

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**Abstract**

Examination of the characteristic deformation of individual grains and the specific interaction between the grains is useful in order to improve the understanding of their influence on the formation of recrystallization texture in polycrystalline materials. As individual grain affects the texture, the best opportunity for these detailed studies are achieved by examining the bicrystal specimen composed of well-known oriented grains. It enables to study the role of the local lattice rotations occurred in each grain and their influence on the formation of new grains nuclei.

The aim of the present work was to investigate the microstructural and crystallographic aspects of recrystallized grains nucleation and its growth in deformed aluminium bicrystals. The application of high purity aluminium characterized by high stacking fault energy (SFE) enables to discount the problem of mechanical and recrystallization twinning. This makes the analysis simpler and clearer. A bicrystals consisted of two high symmetry orientations, stable Goss{110}<001> and unstable Shear{100}<011> were grown by horizontal solidification technique, using split graphite moulds. The samples were deformed in a channel-die to the logarithmic strain of 0.92 at room and liquid nitrogen (77K) temperatures then lightly annealed in order to obtain different stages of recrystallization. The deformed and annealed samples were examined by scanning electron microscopy using a 3D Quanta FEI, equipped with electron backscattered diffraction facility. A complementary analyses were performed with the use of transmission electron microscopy using Tecnai G2 F20 operating at 200kV.

The bicrystals appears to deform relatively homogeneously at the macroscopic scale. However, detailed micro scale examination reveals different behaviour of particular grains forming bicrystal. The Goss{110}<001>-oriented crystallite is essentially stable, forming only homogeneous dislocation arrangements (Fig.1). This is except very thin layer near the boundary between grains. The Shear{100}<011> orientation has tendency to decompose starting from early stages of deformation *via* transition bands formation as a boundary between areas in which symmetrically situated slip systems operate. It contributes to the formation of two symmetrical variants of {112}<111>-orientations in the range of large deformations (Fig.1). At the initial stages of recrystallization, limited number of new orientation groups is formed in both crystallites. In stable Goss{110}<001> orientation the formation of new grains mostly characterized by elongated shape occurs along the traces of all the {111}-type planes (Fig.2). In unstable Shear{100}<011> orientation new grains are preferentially formed at the transition bands near the boundary between crystallites. It was observed that growing grains demonstrate the tendency to cross the initial boundary between both grains when orientations of recrystallized grain and as-deformed matrix share a common {111} plane. Based on the anisotropy of recrystallized grains growth it is proposed that the thermally activated movement of dislocation groups can lead to the creation of a twist or tilt grain boundary, determining the hinder or rapid movement recrystallization front.

**Ackowledgments**

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# Effect of gas nitriding temperature on microstructure of Ti6Al7Nb alloy

**P05**

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**Abstract**

Titanium show good biocompatibility and corrosion resistance but is relatively soft and therefore subject to fast wear. The most popular Ti6Al4V alloy show much improved strength but aluminum is toxic, while vanadium is strongly cancerogenic. Therefore, recent investigations are focused on finding a new alloys with inert alloying elements such as niobium or zirconium. One of them, proposed as a replacement for Ti6Al4V, could be Ti6Al7Nb alloy [1]. It is characterized by similar mechanical properties to its predecessor being definitely more bio-acceptable. One of main disadvantage in case of using it for implants turned out low wear resistance of Ti6Al7Nb, being a characteristic feature for all titanium based alloy. The latter failing could be removed by additional surface treatment of alloy, like gas [2] or plasma nitriding [3] allowing to cover it with hard wear resistant coatings. Gas nitriding is an industry most widely applied thermochemical method of surface engineering. It allows to obtain the compound surface layer formed by titanium nitrides, while below the compound layer a diffusive zone is created [2,4]. This treatment is usually performed at ~1000ºC, which might affect the surface finish of the treated part as well as soften the core of the element [5]. The latest research on gas nitrided Ti6Al7Nb alloy revealed, that this treatment might be also effectively carried out at lower temperatures, but there are no data on its effect on the microstructure.
Therefore, the aim of the presented work was to describe the effect of processing temperature on thickness of compound layer and the diffusive zone of gas nitrided Ti6Al7Nb alloy as well as they phase composition and microstructure.
The Ti6Al7Nb discs (Ø~ 16 mm, thickness ~3 mm) were mechanically grinded and polished. Then, the samples were placed in the chamber and heated up to 830ºC, 740ºC, 680ºC and 620ºC for 6 hours in pure nitrogen ($5N^+$). After treatment, the samples were cooled in the same atmosphere to 500ºC, and next cooled in air.
The optical microscope (OM) and scanning electron microscope (SEM) observations of sections of the nitrided samples indicated that the specified above decrease in temperature of nitriding resulted in significant thinning of the zone affected by this treatment, i.e. up to 5,5; 2,4; 1,8 and 1,3 µm. Moreover, the hardness of nitrided surface diminished with the processing temperature i.e. 462, 375, 347 and 334 HV5. The observations using transmission electron microscopy helped to establish that nitrided affected zone (NAZ) formed near surface consists of three layers: δ-TiN, α”-Ti martensite and $Ti_3Al$ intermetallic as ascertain using selected area electron diffraction method. The α”-Ti martensite layer was built of relatively coarse grains filled with heavily faulted small platelets, while the $Ti_3Al$ layer consisted of even coarser grains occasionally showing presence of irregular anti phase boundaries (APB) characteristic for ordered lattices. The thinning down of the δ-TiN compound layer with temperature applied in present experiment turned out relatively slow, i.e. from 194 nm for the 830ºC to 67 nm for the lowest applied temperature of 620ºC. What is more important, even the layer obtained at the lowest processing temperature turned-out dens and continuous.
The most of the thinning of nitriding affected zone observed using OM and SEM is connected with the changesin α”-Ti martensite and $Ti_3Al$ intermetallic layers. Additionally, the performed microstructure investigations proved that the compound layer obtained at a gas nitriding temperature as low as ~620ºC is still continuous and dense, i.e. is capable to separate the core from the corrosive environment even as for wear protection their thickness might be too low.

**Acknowledgements**

Investigations were co-financed by the European Union from the European Social Fund (Project No. WND-POWR.03.02.00-00-I043/16).

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# Crystallographic phase and orientation mapping of ferroelectric $HfO_2$ thin films by transmission Kikuchi diffraction



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**Abstract**

Since the discovery of ferroelectricity in $HfO_2$ thin films [1], it obtained great research interest for the implementation into various integrated devices e.g. non-volatile memories or infrared sensors, due to its CMOS compatibility. As the ferroelectricity in $HfO_2$ is assigned to the orthorhombic $Pca2_1$ phase [2], its phase fraction and orientation have a strong influence on the ferroelectric properties of the polycrystalline thin film. Due to the similar x-ray diffraction (XRD) pattern of the tetragonal and cubic phase, XRD analysis of the orthorhombic phase is strongly limited.

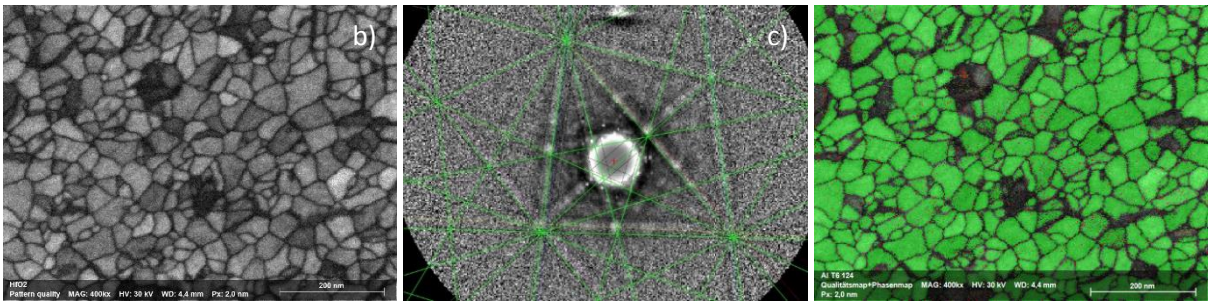


**Figure 1.** Phase identification of $HfO_2$ by TKD. From the EBSD pattern contrast of each point, a quality map (a) can be constructed. By comparing the EBSD patterns with simulated patterns from given phases (b), the phase can be identified and a phase map (c) can be constructed.

Transmission Kikuchi diffraction (TKD) using a scanning electron microscope (SEM) is used for orientation mapping of nanostructured metals and is suggested to become an important technique for the characterization of nanocrystalline structures [3]. Here, we report the TKD analysis of Zr-doped $HfO_2$ thin films. Therefore, the measured electron diffraction of dimpled samples are transformed into the Hough space and compared with the phases of $HfO_2$ (see figure 1). This allows the mapping of the crystallographic phase and grain orientation which indicates a strong out-of-plane texture of the orthorhombic phase.

**Acknowledgments**

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# Microstructure evolution of CrCoNi medium-entropy alloys subjected to uniaxial tension

P07

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**Abstract**

Medium-entropy alloys such as: equiatomic CrCoNi were investigated to have better properties over many high-entropy alloys, e.g. equiatomic CrMnFeCoNi [1]. In this work, we focus on the microstructure evolution of CrCoNi alloy characterized by two different initial states: (i) homogenized for 72h at 1200ºC after casting and (ii) recrystallized for 1h at 900ºC after 50% of thickness reduction performed by cold rolling. In order to obtain microstructure images, samples were observed via EBSD technique.

It is clearly visible that the homogenized state has no recrystallization twins, while pre-deformed state has many of them and were estimated to occupy about 30% of the volume [2]. The grain size is also noticeably different. Mechanical tests were performed at room temperature by uniaxial tension in several ways of processing. At first, uniaxial tension tests until failure of both states were performed in order to obtain maximum longitudinal strain and tensile strength. The mechanical properties of recrystallized sample stay in good agreement with the literature, which indicates that the initial state of material was prepared correctly [1,3].

The main difference in mechanical properties was in the yield strength, which equals about 200MPa and 400MPa for the homogenized and recrystallized state, respectively. The tensile strength turned out to be higher by about 250MPa for the recrystallized state. That range is the result of inhomogeneous microstructure of homogenized sample and is typical for non-recrystallized casted states. Third noticeable difference is much bigger strain in non-recrystallized state, which is connected to much lower hardening rate compared to the recrystallized state.

Afterwards, we have taken EBSD images at difference stages during tension experiments (i.e. at different strain level). Finally, we have also conducted EBSD measurements on recrystallized sample, which has been deformed to a stress level of 900MPa.

Comparing those two states with the initial microstructures, it is clearly visible that recrystallization twins influence microstructure evolution in a significant way. In the non-recrystallized sample, we can observe elongated grains with shear bands located inside the grains and the accompanying lattice rotation with slightly misoriented parts of the grains caused by uniaxial tension without a substantial grain refinement. On the other hand, highly refined grains were observed in the recrystallized sample even before failure but without pronounced elongation.

Final route of investigating was composed of taking EBSD images from specific point on the recrystallized sample and subjecting to uniaxial tension with strain equal to 10%, after reaching that value the sample was unloaded and another EBSD image was taken from the same region of the sample. These measurements were repeated until failure.

Our investigations showed that microstructure evolution of a medium-entropy CrCoNi alloy is strongly connected with the initial microstructure and has higher ultimate yield strength when recrystallization twins were present within the initial microstructure.

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# Thermodynamic properties of alloys from the ternary Ga-In-Li alloys

**P08**

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**Abstract**

Lithium batteries are a new and promising high-energy-density power source. Many new lithium battery systems are being developed, and these batteries are finding many new uses in the consumer market today [1]. In order to prepare the appropriate electrode material, information on the thermodynamic properties, phases equilibria and phases transformations of the elements involved is required. To prepare thermodynamic database, experimental information about a particular system are necessary. In this work we propose the Ga-In-Li alloys as a promising anode material. Unfortunately, there is a lack of thermodynamic data for this systems in the literature. Thus, it was found to be necessary to perform an experimental investigation on the abovementioned system in order to collect the necessary data.

Figures 1-3 present the binary systems which form the ternary system chosen in this study. As it can be seen from the presented figures only the Ga-In system is a simple eutectic system, however the Ga-Li and In-Li systems are more complicated with many intermetallic phases, which poses certain problems when studying the ternary system, as appropriate temperatures have to be chosen in order to avoid those phases.

The cells were prepared using a glovebox chamber with protective argon atmosphere. Eutectic KCl-LiCl and LiF-LiCl salts were used in the preparation of the electrolyte. The construction of the cell is shown in Fig 4.

The obtained results will be used to optimize the thermodynamic properties of the phases present in the ternary Ga-In-Li system, and for calculation of the phase diagrams of binary and ternary systems. The results will be also used to calculate the phase diagrams of the binary and ternary system. Furthermore, they will be introduced into the free of charge Entall database http://www.entall.imim.pl/ [5]. It was determined that the obtained results will allow the use of the mentioned Ga-In-Li system to build new liquid batteries in the future.

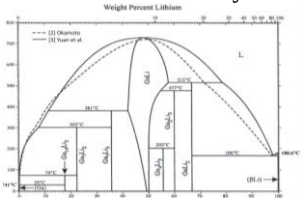


Fig. 1 Phase diagram of the Ga–Li system [2].

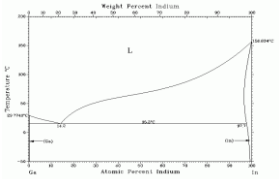


Fig. 2 Phase diagram of the Ga-In system [3].

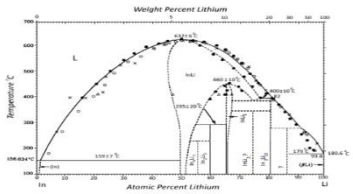


Fig. 3 Phase diagram of the In–Li system [4]

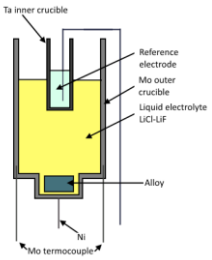


Fig. 4 Illustration of cell for EMF measurements

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# Growth and characterization of Zr doped ZnO structures on femtosecond laser induced periodic structures on different substrates.

P09

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**Abstract**

Doped ZnO nanostructures have been widely investigated for their electrical and optical properties. It is one of the most used transparent conducting oxide (TCO) with low resistivity ($\leq 10-3\Omega cm$), high transparency (>80%) and high carrier concentration ($\geq 1020 cm-3$). Doping with Zr is proposed because of the similar ionic size of Zr4+ and Zn2+. That means the lattice distortions are minimized while the thermal and the chemical stability are improved being a promising alternative for high temperature working devices [1]. Furthermore, ZnO:Zr has shown enhanced photocatalytic properties [2]. In this work, Zr doped ZnO structures have been grown on different oriented Silicon substrates by a vapour-solid method under an Argon flux as is shown in Figure 1a. Mixtures of ZnS and ZrO2 in different percentages were used as precursors. Silicon <100> and <111> substrates have been also irradiated under certain conditions by a femtosecond laser operating at 1030nm with a pulse duration of 340 fs (Figure 1b). The irradiation of the substrates generates laser periodic surface structures (LIPSS) which depending on the irradiation condition could be amorphous or ablative structures [3]. The influence of the processed surface in the growth has been studied according to different parameters as the thermal treatment duration, percentage of doping, type of induced LIPSS and substrate orientation among others. Scanning Electron Microscopy has been used to analyze the morphology of the structures and Photoluminescence measurements provide information about the luminescence properties and the amount of defects in the structure giving a hint about the influence of the different parameters used in the growing process.

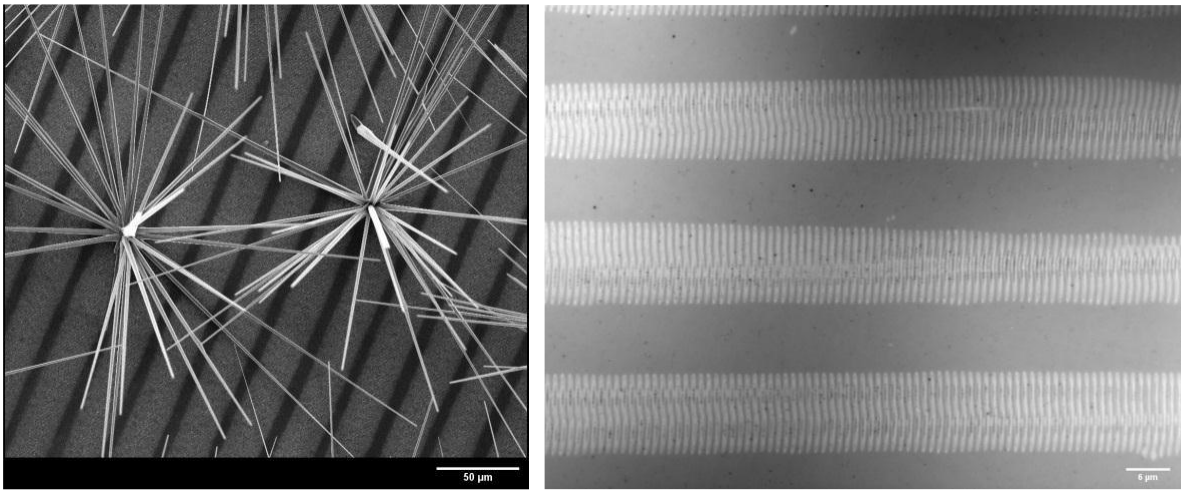


**Figure 1.** a) SEM image of the Zr doped ZnO structures growth on irradiated silicon substrate. b) Optical image of amorphous LIPSS in silicon <100>

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# Microscopic mapping of the full strain tensor, local orientation and composition in an $In_xGa_{1-x}N$ heterostructure via scanning X-ray diffraction

**P10**

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**Abstract**

The growth of single-crystalline thin films is the basis for many modern semiconductor devices, such as LEDs, laser diodes, transistors etc. Lattice strain caused by a mismatch of lattice constants or thermal expansion coefficients between film and substrate is an important consequence of the growth and a major factor to influence the electronic band structure and optical properties of the device. Therefore, strain engineering of the crystal lattice by geometrical relaxation or constraints is an established route to tune these properties. However, an accurate, non-destructive microscopic characterization of strain not done routinely. The "strain microscope" beamline ID01 of the ESRF is aiming to tackle this problem by means of X-ray diffraction microscopy [1].

X-ray diffraction is frequently used to determine the unit cell dimensions of epitaxially grown films which allows to evaluate growth parameters such as lattice perfection, strain, composition, mosaicity and thickness averaged over the X-ray beam footprint. However, microscopic resolution is required to study the origins of deviations in these parameters. While some techniques provide access to such information (e.g. transmission electron microscopy, Raman spectroscopy), scanning diffraction X-ray microscopy (SDXM) proposed here has advantages being non-destructive, model-free and having high strain sensitivity. The spatial resolution is determined by the beam size and can reach values below 100nm at ID01. 3D Reciprocal space maps are obtained for each point on the 2D surface by rocking the sample around the Bragg angle finally resulting in a 5D dataset $I(x, y, q_x, q_y, q_z)$. Combining three of such datasets from non-coplanar reflections yields microscopic maps of local orientation and the full strain tensor $\varepsilon_{ij\,j}$. From the lattice dimensions, the local stoichiometry can be derived on knowledge of the elastic coefficients of the material.

The technique is applied to an $In_xGa_{1-x}N$ heterostructure which is used as a template for growth of a multiple quantum well for LED applications. High In-content is desired as it leads to a red-shift of the emission in $In_xGa_{1-x}N$ LEDs that otherwise perform very well in the blue region of the visible spectrum. This could become one way to close the "green gap" existing in the efficiency of monolithic white light emission devices [2]. Our sample consists of a first $In_xGa_{1-x}N$ layer that has been grown on a Sapphire substrate. Subsequent photolithography patterning is used to produce sub-mm structures and facilitates a stress-release after lifting off the InGaN film from the substrate [3]. This way, another $In_xGa_{1-x}N$ layer of higher (here nominally 5%) In-content and lower mismatch strain can be grown on top. The strain relaxation induced by patterning and the thereby induced variations of In-content are the target of this study. Fig. 1 shows maps of the extracted lattice parameters of the regrown top-layer. These show variations on 1um lengths scales. The lattice tilts and in-plane parameters between lower and top layer correlate. In contrast, out of plane lattice parameter and In-content show rather different distribution.

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# Manufacturing and characterization of oxide dispersion strengthened Ni-free austenitic stainless steel

P11

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**Abstract**

Oxide dispersion strengthened (ODS) austenitic steels are candidates for large scale structural materials in future fusion power plants, owing to their superior phase stability at high-temperatures, as well as excellent oxidation, corrosion and high-temperature creep resistance comparing to their ferritic counterparts. The addition of yttrium oxides (Y2O3) to austenitic matrix is considered to be an effective way to improve both radiation resistance and high-temperature strength. However, traditional austenitic steels contain a large amount of Ni (austenite stabilizer) which is a highly activated element. To eliminate this drawback in the present work Ni-free N-containing ODS austenitic steel was manufactured by mechanical alloying followed by spark plasma sintering. However, its complex microstructure consisting of ultrafine grained matrix and nanoscale precipitates requires multiple techniques to be used to fully characterize the microstructure and understand its properties.

Electron microscopy provided sufficient information to determine matrix grain size, size and spatial distribution of nanoparticles as well as chemical composition of larger particles but from a very small volume. As a consequence, the statistics is rather poor and the precise evaluation of average size and number density of nanoparticles is limited. Therefore, contrast variation small-angle scattering was applied. The complementary techniques are based on X-ray or neutron scattering, where the information comes from a relatively large volume, however, the information is limited to the number density and size distribution. In addition, the stoichiometry of scattering particles can be obtained from an 'alloy contrast variation' (ACV) analysis. This approach guides the determination of the chemical composition, size and number density of the nanoparticles over a wide size range, while probing a large sample volume.

**Acknowledgments**

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# Logic Operations with Oxide Field Effect Transistors on Cellulose Substrates

P12

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**Abstract**

This work demonstrates that a unique set of characteristics can be combined in planar dual-gate oxide–based field effect transistors with a back floating electrode using cellulose simultaneously as substrate and dielectric. The working principle of these transistors relies on the formation of electric double layers at the semiconductor/cellulose and cellulose/back floating electrode interfaces that can be disturbed by a voltage applied at a secondary gate, by the back-floating potential or by the combination of both. This feature allows for the control of the on-voltage of the transistors, from depletion to enhancement mode, for instance. Moreover, this specific characteristic allows the implementation of universal logic gates (NAND and NOR) using only one of these devices, by setting the proper combination of the voltage level applied at each gate.
This way a simple and universal device architecture can be envisaged towards the simplification of the production of low power electronic systems on cellulose based substrates.

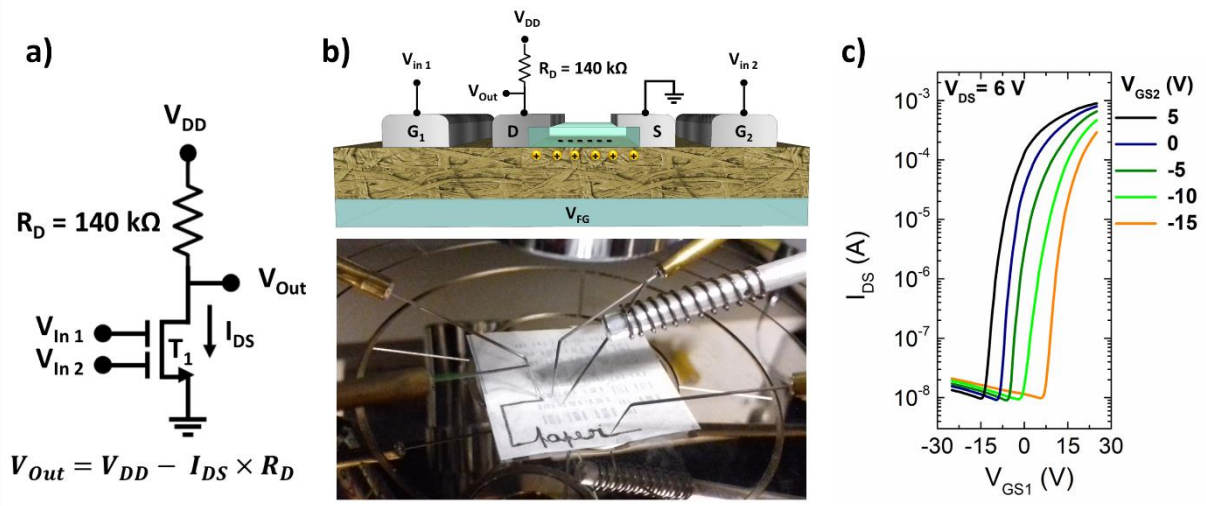


**Figure 1.** Universal logic gates: a) Circuit schematic, b) On-chip implementation, and c) $I_{DS}$-$V_{GS}$ transfer characteristics DG-FGFETs at $V_{DS}$=6 V.

**Acknowledgments**

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# High-temperature oxidation of ferritic steel with and without a spinel coating under thermal cycling conditions

**P13**

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**Abstract**

Sustained development, which has become a priority for many highly industrialized countries at the turn of the 21st century, will not be possible without an efficient solution to the problem of storing surplus electricity. The most suitable technologies designed for this purpose include solid oxide electrolytic cells (SOECs), which utilize electrolysis to efficiently convert surplus electricity into fuel such as hydrogen [1]. One of the most essential components of both SOECs and solid oxide fuel cells (SOFCs) is the interconnect, which allows individual cells to be connected in series and form stacks. The main issue faced when using metallic interconnects based on ferritic steel is the gradual increase in area-specific resistance that occurs due to the high-temperature corrosion of these materials. The main component of the scale formed on ferritic steel in an atmosphere consisting of cathode and anode gases is chromia ($Cr_2O_3$) [2,3]. Unfortunately, this oxide reacts with oxygen and water vapor, subsequently forming volatile compounds of chromium, which adversely affect the catalytic properties of the electrodes [4]. This issue can be mitigated via the use of protective-conducting spinel coatings deposited on the surface of the steel by means of electrophoresis. One of the chromium-rich ferritic stainless steels manufactured specifically for the purpose of application in SOEC/SOFC interconnects is the Crofer 22 H (VDM Metals GmbH). This steel has been studied extensively in high-temperature corrosion conditions, with most studies including the measurements of oxidation kinetics under isothermal conditions and for various reaction media. However, electrolytic cells do not operate solely under isothermal conditions, but are also frequently exposed to thermal shock. The latter occurs during the startup of the device or during an emergency shutdown of the stack. Experiments concerning the oxidation of the Crofer 22 H ferritic steel with a cobalt-manganese coating with a copper addition under conditions that reflect the real-life occurrence of thermal shocks would not only have scientific value, but they would also provide a considerable amount of practical information, allowing a more accurate estimation of the maximum operating time of SOECs.

The aim of the presented study was to deposit a protective-conducting $Mn_{1.45}Co_{1.45}Cu_{0.1}O_4$ spinel coating on the Crofer 22 H ferritic steel by means of electrophoresis and to evaluate its physicochemical properties after high-temperature oxidation with thermal cycling. When the Crofer 22 H steel – whether uncoated or coated with the spinel – was oxidized in 48-hour cycles involving a temperature of 800°C, the kinetics of the process approximately obeyed the parabolic rate law. The oxidation rate observed for uncoated steel was higher than that for the studied coating/steel system. The obtained $Mn_{1.45}Co_{1.45}Cu_{0.1}O_4$ coating was single-phase, compact, and exhibited good adhesion to the metallic substrate. The area-specific resistance values measured for the coating/steel and scale/steel systems indicated that the coating significantly improved the electrical properties of the studied ferritic steel.

The conducted research confirmed the suitability of the $Mn_{1.45}Co_{1.45}Cu_{0.1}O_4$ spinel as a coating on the Crofer 22 H ferritic steel and the viability of using such a system for the fabrication of SOEC interconnects.

**Acknowledgements**

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# Point defect concentration and their diffusivity in $(Co,Cr,Fe,Mn,Ni)_3O_4$ high entropy oxide

**P14**

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**Abstract**

The point defect structure and chemical diffusion in **(**$Co,Cr,Fe,Mn,Ni)_3O_4$ high entropy oxide were studied at 1273 K in the oxygen pressure range $10\text{-}10^5$ Pa. **(**$Co,Cr,Fe,Mn,Ni)_3O_4$ was synthesized by sintering a mixture of starting oxides: $Co_3O_4$, $Cr_2O_3$, $Fe_3O_4$, MnO, NiO. The presence of a single-phase, high entropy solid solution structure was proven using SEM+EDS and XRD methods. On the basis of marker studies, it has been determined that the predominant disorder inside the investigated high entropy oxide is present in the anion sublattice. Non-stoichiometry data was determined by means of thermogravimetry. From this result it can be concluded that oxygen vacancies are the predominant point defects in the studied oxide at low pressures. However, at high pressures the predominant defects become interstitial anions. Consequently, taking into account the deviation from stoichiometry, the chemical formula of this oxide can be written as **(**$Co,Cr,Fe,Mn,Ni)_3O_{4\pm y}$. Reequilibration kinetics were used to determine the chemical diffusion coefficient of defects. The obtained chemical diffusion coefficient values strongly depend on oxygen pressure, indicating the presence of a complex point defect structure in **(**$Co,Cr,Fe,Mn,Ni)_3O_{4\pm y}$.

**Keywords**: high entropy oxide, point defect concentration, chemical diffusion coefficient

# Structural and electrical properties of doped and undoped 3Y-TZP electrolyte

**P15**

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Solid oxide fuel cells are currently the most efficient devices used for the conversion of fuel directly into electrical power. Of the different types of SOFCs, planar cells would likely prove the most beneficial if they were to achieve widespread popularity. In this design, a single cell consists of an oxide electrolyte sandwiched between an anode and a cathode (Fig. 1). One of the strategic goals in this field is to develop materials which allow SOFC devices to operate with high power efficiency at temperatures ranging from 600 to 800°C [1-4].

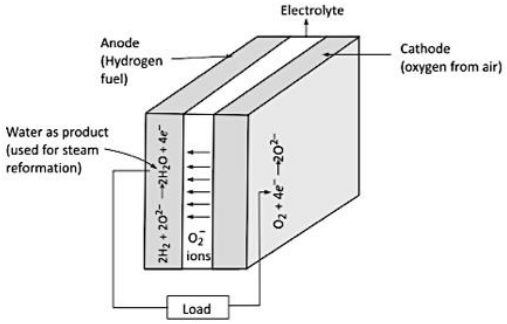


**Fig.1.** A single cell in a SOFC fueled with hydrogen [2].

One of the ways in which this may be achieved is the application of a solid electrolyte composed of tetragonal zirconia polycrystals (TZP) containing a 3 mol% $Y_2O_3$ addition. The arguments in favor of selecting the 3Y-TZP composite as the electrolyte in IT-SOFCs instead of cubic 8-YSZ include its significantly higher mechanical strength and its lower activation energy of electrical conductivity, which is associated with higher electrical conductivity at temperatures below 700°C. It had already been established that adding $Al_2O_3$ in amounts which exceed its solubility limit in 3Y-TZP may in some cases cause the electrical conductivity of the latter to increase, and, in addition, improve its mechanical properties [2-4].

The objective of the presented study was to determine how adding $Al_2O_3$ to the 3Y-TZP material affects its ionic conductivity. Composite 3Y-TZP sinters were obtained from a 3-YSZ powder containing 0, 5 and 15 mol% of $Al_2O_3$, synthesized using the gelation method. Two series of samples with an alumina addition were prepared. For the first type, aluminum was introduced during the gelation of the 3-YSZ powder. To obtain the second type of sample, the 3-YSZ powder was impregnated with an alcohol solution of aluminum nitrate(V) at the appropriate concentration. Both types of powders underwent 2 h of thermal treatment at $400^{O}$C and then cold isostatic pressing to produce green bodies. The green bodies were subsequently sintered for 2 h in air at $1400^{O}$C. The structural and morphological properties of the powder and the microstructure of the obtained sinters were evaluated by means of the XRD and SEM-EDS methods. The ionic conductivity of the composite sinters was determined using electrochemical impedance spectroscopy (EIS).

The conducted research made it possible to establish that the tetragonal form of zirconium in the 3Y-TZP/$Al_2O_3$ system is a promising solid electrolyte for intermediate-temperature solid oxide fuel cells (IT-SOFCs).

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# Cleaving silicene-terminated calcium disilicide in the transmission electron microscope

**P16**

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**Abstract**

The groundbreaking work in 2004 [1] leads an unprecedented boom in interdisciplinary researches for 2D materials. Due to superior properties, 2D materials promise tremendous potential applications in electronics, optoelectronics, membranes, energy storage and generation, catalysis, sensing and so on [2]. Silicene [3], a monolayer of silicon atoms arranged in a honeycomb lattice, is excellently compatible with the materials used in today's semiconductor manufacturing. In this paper, silicene-terminated $CaSi_2$ is cleaved inside a TEM using an in-situ manipulator. HRTEM studies on a standard lift-out lamella performed from several crystallographic orientations confirm the cell parameters of a = 3.7 Å and c = 30.60 Å, and allow to determine its exact orientation in the SEM/FIB system. A FIB procedure with corrected tilting and rotating angles has been developed to ensure that the tensile force applied by the manipulator is perpendicular to the (0 0 1) plane, and that the [1 0 0] pole axis could be used for HRTEM imaging. A sharp and flat cleavage interface with a length of more than 1 µm was observed in one in-situ experiment. HRTEM images from multiple regions confirm that the flat cleavage follows the (0 0 3) plane of the $CaSi_2$ crystal. The current in-situ study demonstrates that a surface sheet with silicene-like atomic arrangement can be mechanically exfoliated from silicide compounds.

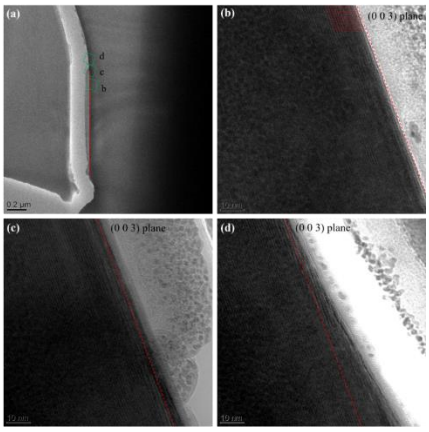


**Figure 1**. (a) Cleaved $CaSi_2$ lamella after in-situ test, (b), (c) and (d) HRTEM image of the area marked by the green square box.

**Acknowledgments**

Funding by the German Research Foundation (project number: 267459210) and Czech Science Foundation (project number 15-08842J) is gratefully acknowledged.

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# The influence of surface roughness on elastic nanoindentation measurements

**P17**

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**Abstract**

The characterization of mechanical properties of layered thin-film structures is important with respect to a better understanding and for improving the design of microelectronic devices. Due to the small investigated volume and the easy implementation of the measurement procedure, nanoindentation is an appropriate method for determination of the mechanical properties of thin films systems. Schwarzer et al. [1] developed an analytical approach that allows to derive values of Young´s modulus of materials from load-displacement curves measured within the elastic range of interaction. This analytical approach together with nanoindentation using spherical indenter geometries is employed in this study. Investigations have been conducted on Fused Silica (FS) standard samples with known values of surface roughness and Young's modulus (E=72 GPa). Surface roughness values of the samples were calibrated considering several etching times with hydrofluoric acid (HF). It could be shown that the roughness has a strong influence on the statistics of the measured load-displacement curves as well as on the derived Young's modulus values (see Figure 1). Therefore, in the current study the influence of surface roughness has been investigated in more detail. This is done by applying a model that was developed for contact stiffness measurements using AFM-based methods. The model takes into account the contact stiffness of the indenter tip and the investigated sample as well as the contact stiffness of the multiasperity contact, arising from the roughness of the sample and the indenter. The aim of the study is to combine the analysis approach used for AFM data with the nanoindentation measurements and thereby to proof the AFM model on a bigger length scale.

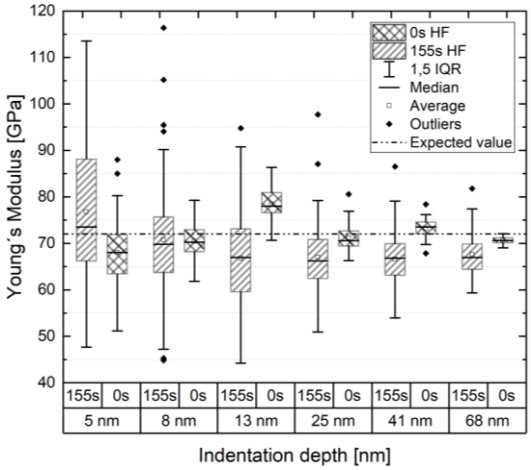


**Figure 1.** *Young's modulus measurement statistics on Fused Silica after HF-etching at several indentation depths. With higher surface roughness and lower indentation depths, the scatter of the data increases. The Young's modulus values of the rough sample (155s HF) tend to be underestimated.*

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# Ga-Sn-Zn liquid metal alloys – a multifaceted functional material

**P18**

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**Abstract**

Liquid metals are defined as metals or alloys that are liquid at room or near-room temperature. The best known liquid metal is mercury, although due to its toxicity it has been withdrawn from use from most applications. A need to find a replacement for mercury arose and the main focus have been turned to gallium and gallium based alloys. The most important gallium based alloy is the commercially available Galinstan, which is based on the Ga-In-$Sn_{eut}$ with additions of elements like Bi or Sb, created in order to replace mercury in thermometers. In this work the Ga-Sn-Zn system has been proposed as an alternative to Ga-In-Sn alloys, as Sn and Zn are less expensive than In. Gallium based alloys exhibit many interesting properties - they are non-toxic, have a high range of working temperatures, have a tendency to supercool and oxidize easily, with a self-limiting oxide layer forming on the liquid metal [1]. They have a potential to be used in many fields of science and technology including as cooling agents in nuclear plants [2], Concentrated Power Cells [3] and computer chips, in medicine, in soft sensors and stretchable electronics [4]. Ga-Sn-Zn alloys can be furthermore used to obtain nanometric oxide layers on virtually any substrate [5,6]. In this work we show how the thermophysical properties of Ga-Sn-Zn ternary alloys can be modified in order to better fit the numerous mentioned applications.

**Acknowledgments**

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# Drop calorimetry method in mixing enthalpy and enthalpy of formation study in the Li-Ag-Sb alloys

**P19**

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**Abstract**

Currently, researchers are looking for materials that would increase the capacity of batteries. High hopes are placed in the alloys containing intermetallic phases that can be applied to new anode materials. According to the work [1], Ag-Li-Sb alloys could be used as anode materials for batteries. At present, no thermodynamic data and complete phase diagram for this ternary system are found in the literature. There is not much information in the literature on the $Li_2AgSb$ phase, which exists in this ternary system. Pauly et al. [2, 3] were the first to synthesize and structurally characterize $Li_2AgSb$. In 2008, Tarasiuk et al. [4] confirmed the structure of this phase. In this work, we experimentally measured enthalpy of mixing of Li-Ag-Sb alloy and enthalpy of formation of $Li_2AgSb$ phase.

Drop calorimetry was applied to determine, at 980 K, partial and integral enthalpies of mixing Ag-Li-Sb liquid alloys. The investigations were performed along five sections by the addition of lithium to mixtures with fixed Ag to Sb molar ratio of 1:1 and 1:4 and by the addition of silver to mixtures with fixed Li to Sb molar ratio equal to 1:9, 3:7 and 1:1. Integral enthalpy of mixing in Ag-Li-Sb alloys strongly depends on composition. Negative enthalpy of mixing is observed throughout the entire range of concentrations studied.

Since there are no thermodynamic data for the $Li_2AgSb$ in literature, we measured its enthalpy of formation. The alloy containing 50 at.% Li, 25 at.% Ag and 25 at.% Sb was prepared by melting in resistance furnace in the glovebox under high purity Ar atmosphere. Next, the X-ray powder diffraction method was used to confirm purity of synthesized $Li_2AgSb$ phase. Measurement of enthalpy of formation of $Li_2AgSb$ phase was carried out in two ways with drop calorimetry. The calorimetric dissolution method involves measuring the enthalpy of dissolution of the studied phase and its components in a liquid bath. In the first case, liquid lithium was used as a bath and the temperature of the measurement was 980 K, In the second case, the enthalpy of formation was measured at 880 K using liquid tin as a bath.

In this work, we discuss the measurement and compare the results of enthalpy of formation of $Li_2AgSb$ obtained using two different baths. Experimental data of enthalpy of mixing were compared and discussed with Muggianu and Toop extrapolation models. In addition, liquidus position was deduced from discontinuities in partial and kinks in integral enthalpies.

**Acknowledgments**

Financial support from National Science Centre Poland grant no 2015/19/B/ST8/01074 is gratefully acknowledged.

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# Kinetics and mechanisms of the electrophoretic co-deposition of PEEK and sulfide or nitride particles

P20

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**Abstract**

Electrophoretic deposition (EPD) is an electrochemical process used to deposit coatings on conductive substrates [1, 2]. This method is relatively simple and does not require the use of expensive equipment, thus the interest in it is fast growing [3]. EPD offers many possibilities of combining various materials and it is versatile to fabricate composite polymer-based coatings [4, 5]. The aim of the present work was to investigate EPD kinetics and mechanisms, to elaborate the EPD route and post-EPD heat treatment parameters to fabricate nanocomposite $MoS_2$/PEEK 708 and $Si_3N_4$/PEEK 708 coatings, as well as to investigate the influence of cooling rate after heating on the coatings microstructure. Stable ethanolic-based suspensions for the EPD of coatings were elaborated based on measurements of the zeta potential of particles depending on the suspension's pH. It was necessary to add cationic chitosan polyelectrolyte, which changed the surface potential of the particles and increased the suspension's stability. Based on zeta potential measurements and TEM studies of particles interaction in the suspensions the mechanisms of interaction between PEEK particles (size in the range of 2 - 15 µm) and $MoS_2$ or $Si_3N_4$ nanoparticles in the suspensions used for EPD were determined. The results indicated electrostatic interactions between both ceramic nanoparticles and the polymeric micro-particles in pure ethanol suspensions. The addition of chitosan polyelectrolyte provide steric stabilization of PEEK 708 and $Si_3N_4$ or $MoS_2$ particles. In order to deposit compact and homogeneous coatings, optimal EPD parameters, such as voltage and deposition time, were experimentally selected. It was found that macroscopically homogeneous coatings were obtained using a constant voltage of 100 V and deposition time of 90 s as well as a constant voltage of 90 V and deposition time of 90 s for $MoS_2$/PEEK 708 and $Si_3N_4$/PEEK 708 coatings, respectively. $MoS_2$ or $Si_3N_4$ separate nanoparticles and their small agglomerates were absorbed and relatively uniformly distributed on large PEEK polymer particles. The investigation of the EPD kinetics showed that the deposition rate is strongly dependent on the deposition time. As-deposited coatings were heated above the polymer melting point (heating temperature of 390 °C) and cooled at different rates (i) with a furnace, 2 °C/min and (ii) in water (RT). Heat treatment caused a change in the morphology of polymer from globular particles into a continuous and dense coating matrix. It was detected that in $Si_3N_4$/PEEK 708 coatings the cooling rate after heating significantly influenced the coating structure, which was semi-crystalline or amorphous, after slow and fast cooling, respectively. Interestingly, $MoS_2$/PEEK 708 heat treated coatings exhibited an amorphous structure, regardless of the cooling rate after heating.

**Acknowledgements**

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# Pore topology of organosilicate glass studied by positron annihilation experiments and positronium migration modeling

P21

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**Abstract**

The pore topology of a self-assembled organosilicate glass (OSG) [1] with a dielectric constant k = 2 (porosity ~40 %, [1, 2]) was investigated using monoenergetic positrons of a positron source at an electron linear accelerator (ELBE, Helmholtz Center Dresden-Rossendorf). The pore diameter was determined to be about 3.5 nm using positron annihilation lifetime spectroscopy (PALS). Furthermore, the ortho-positronium (o-Ps) escape ratio, i.e. the 3γ/2γ ratio of the three-ray decay of o-Ps emitted at the pore surface and the two-ray decay of para-positronium (p-Ps) from the pick-off process and positron annihilation in the material matrix, was determined to be ~50 % for a positron energy of 2 keV, which corresponds to a mean penetration depth of positrons of 100 nm. Since the neck length of the pore interconnection is defined by porosity and pore diameter if spherical pores with a coordination number 6 are assumed (Fig. 1a), the only remaining topology parameter is the neck size, i.e. the width of the interconnection. With positronium migration modeling, the neck size was determined by adjusting it appropriately in such a way that 50 % of Ps atoms escape to the vacuum from a 100 nm penetration depth. The migration of Ps was modeled as bounces off the pore walls, assuming isotropic scattering. The lifetime of the Ps was set to 57 ns, which corresponds to the mean lifetime of Ps in pores with 3.5 nm diameter. If the bouncing path leads to the surface within that time, the Ps atom was counted as escaped. The o-Ps escape ratio is 50 % for a 100 nm penetration depth if the neck size is about 0.3 nm (Fig. 1b), which roughly corresponds to a missing atom in the glass matrix. The conclusion for the topology of the OSG thin film is that the pores (diameter about 3.5 nm) are connected by open volume (i.e. missing atoms) of the glass matrix. The pore topology of the OSG has strong influence on the mechanical properties of the thin films, particularly the Young's modulus. Knowledge of the exact structure provides directions for designing mechanically more robust OSG thin films, i.e. to balance dielectric and mechanical properties of insulating films for BEoL stacks.

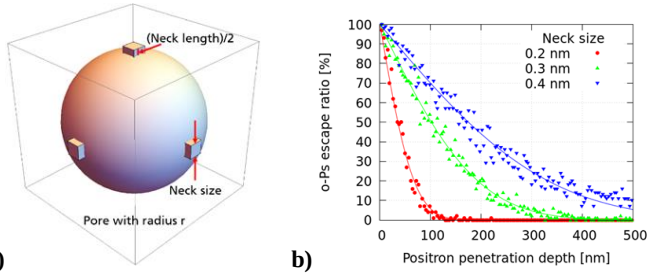


**Figure 1.** a) Pore model, to be repeated periodically in all three dimensions. b) Ps escape ratio for varying neck sizes

**Acknowledgments**

The authors gratefully thank the support staff of the ELBE beamline at the Helmholtz Center Dresden-Rossendorf.

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# Superelastic Ti-Nb alloys fabricated by powder metallurgy route

**P22**

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Ti-Nb alloys obtain using casting technology are characterized by excellent biocompatibility, low elastic modulus and good superelastic properties. Therefore, they are assessed to be one of the possible alternatives to conventional Ni-Ti alloys used for orthopedic implants. Superelastic behavior of Ti-Nb alloys is connected with thermo-elastic martensitic transformation between the body centered cubic (BCC) β parent phase and the orthorhombic α" martensite phase. Addition of biocompatible elements like Nb, W or Ta allows to obtain a high-temperature β phase in room temperature (RT). In the case of Ti-Nb system addition of 1 at.% of Nb to β-Ti decreases *Ms* temperature by 40 K and the addition of 26 at.% allows to obtain the *Ms* slightly below RT, what ensures optimal RT superelastic properties. In the case of the powder metallurgy (PM) processes, the oxygen and nitrogen contamination have a significant impact on *Ms* temperature, as well as on the superelastic properties. Addition of 1 at.% of O to the Ti-Nb alloys causes a decrease of *Ms* by about 200 K. Due to this fact the superelastic effect is not observed in sintered materials even if the Nb content indicates that *Ms* is close to RT . Therefore, the concentration of Nb in Ti-Nb alloys has to be reduced. The aim of the presented work is to study the influence of Nb content on microstructure, mechanical properties and superelastic behavior of Ti-Nb alloys.

Three alloys from the Ti-xNb system, where x = 26, 20, 14 (at.%), were obtained by mechanical alloying (MA) of pure element powders (Ti – 150 mesh, 99,9%, Nb – 325 mesh, 99,8%). The process was conducted in planetary ball mill Fritsch Pulverisette 7 with a rotation speed 150 rpm and cemented tungsten carbide containers and balls. The ball to power weight ratio was 10:1. Time of milling was 30h. In order to avoid extensive oxidation of powders during synthesis, all the operations with powders were conducted under protective argon atmosphere. Powders were consolidated using Spark Plasma Sintering (SPS) method at 1300°C for 30 min under 35 MPa pressure in argon atmosphere.

In the microstructure of as-sintered alloys equiaxed grains of β-phase, with size in range 10-50 μm are observed. It is worth to mention, that porosity of the fabricated materials was low and typically did not excess 0.2 vol.% At the grain boundaries darker precipitations of α-phase, depleted in Nb, can be distinguished. The presence of trace amount of hexagonal α-phase was also confirmed using XRD technique. The existence of this precipitations in Ti-Nb alloys is undesirable, because it can lead to nucleation of cracks at the grain boundaries and as a result decrease the superelastic properties of the alloys. In addition, very bright particles of Nb, undissolved during the sintering, are observed in the microstructure of the as-sintered materials. Their presence causes a reduction of Nb concertation in the matrix. In order to improve the homogeneity of as-sintered samples, additional annealing at temperature 1250°C for 24h was applied.

Applied annealing leads to reduced amount of α-phase, which was confirmed by microstructure observation as well as the XRD phase analysis. Based on metallographic analysis, it was determined that the α phase volume fraction was reduced e.g. for Ti-20Nb alloy from 2.0 vol.% to 0.6 vol.%. In addition, Nb particles are not observable in the microstructure after this process. As a result the concentration of Nb in the matrix was increased and reached the value close to initial composition of powders. XRD patterns for annealed alloys indicate that in the case of Ti-20Nb and Ti-14Nb, during the cooling, martensitic transformation took place, which indicates that *Ms* temperatures of these alloys are near to room temperature.

The compression tests were carried out in order to determine the mechanical properties of obtained alloys. The Yield Strength (YS) of as-sintered alloys decrease with Nb content from 950 MPa for Ti-14Nb to 650 for Ti-26Nb, this dependence is connected with the amount of hexagonal α phase which decrease with Nb content in the alloys. After additional annealing only small difference in mechanical properties of Ti-26Nb and Ti-14Nb was observed, which can be connected with slight grain growth during annealing and the formation of ω phase. In the case of Ti-20Nb alloy YS decrease from 750 MPa to 620 MPa after annealing, which can be connected with martensitic transformation during compression. The superelastic properties of obtained alloys were investigated using cyclic compression tests. The maximum recoverable strain as high as 2,5% was obtained for annealed Ti-14Nb alloy. In the case of Ti-20Nb and Ti-26Nb alloys recoverable strain not exceed 2%.

**Acknowledgements**

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# Analyzing elementary deformation processes during novel in-situ SEM Micro Double Shear experiments in advanced Near-γ Ti-Al alloys

P23

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**Abstract**

Recently, there has been a considerable interest in Ti-Al alloys due to the current implementation of intermetallic Ti-Al turbine blades into the low pressure and low temperature sections of commercial aero engines. The unique combination of low density and good creep strength in the 700°C temperature range makes these materials ideal candidates for such applications. However, Ti-Al alloys feature complex microstructures comprising several phases. The objective of the present work is to obtain basic mechanical data on individual phases present in the Ta-Al system through conducting a series of in-situ scanning electron microscope (SEM) Micro Shear experiments. Until now, this novel technique has been applied primarily to pure model systems [1-3] and most recently to single crystal Ni-based superalloys [4]. Applying this technique to complex enginnering materials like Ti-Al presents a challenging, but nevertheless crucial task for obtaining information on the mechanical properties of specific phases that could potentially serve as an input for crystal plasticity based micromechanical models [5]. The present work outlines our in-situ SEM Micro Shear procedure applied to obtain critical shear stresses for further use in modelling. Microshear samples have been prepared in grains with specific orientations using focused ion beam (FIB) micro machining. Subsequently, in-situ experiments were performed using a micromechanical test system equipped with a flat punch indenter. Transmission electron microscopy (TEM) was conducted to identify activated slip systems as well as to study the elementary deformation processes. Particular emphasis was placed on the mechanical properties of $\alpha_2$ and γ phases as well as on $\alpha_2$ / γ interphases.

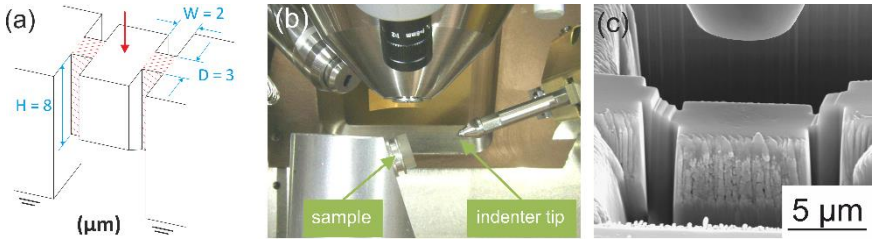


**Figure 1. Experimental setup:** (a) Microshear sample dimensions. (b) Test rig of in situ nanoindenter in a SEM chamber. (c) SEM micrograph after shear deformation of a FIB machined sample in single crystal Cu.

**Acknowledgments**

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# Analysis of the strain dependent acidic etch rate on diamond wire sawn silicon wafer

P24

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**Abstract**

Processing of silicon in microelectronics, photovoltaics and micromechanics includes thermal and mechanical processing that can lead to a change in the silicon lattice, such as phase transitions or lattice deformations which have a crucial impact on the mechanical properties and the chemical resistance of silicon. [1] [2] In this work the correlation between lattice deformations and the etching of silicon using a $HF/HNO_3$ solution is investigated. Here we use Raman microscopy to quantify and qualify strain on mechanically treated silicon, as well as confocal microscopy to measure the topography and to calculate the local etch rate. Additionally, a thermal treatment is used to selectively relax strained silicon for better understanding the effect of a selective kind of strain on the etching mechanism. Our results show that only in tensile strained areas, with a deformation strength of at least 2 $cm^{-1}$, small cracks are formed within the first 10 seconds of etching. After all strained silicon is etched away the etch process mainly depends on the resulting surface texture. The enhanced oxidation rate of tensile strained silicon by nitric acid is also shown exemplary by the surface modification using nitric acid and trichloro(octyl)silane.

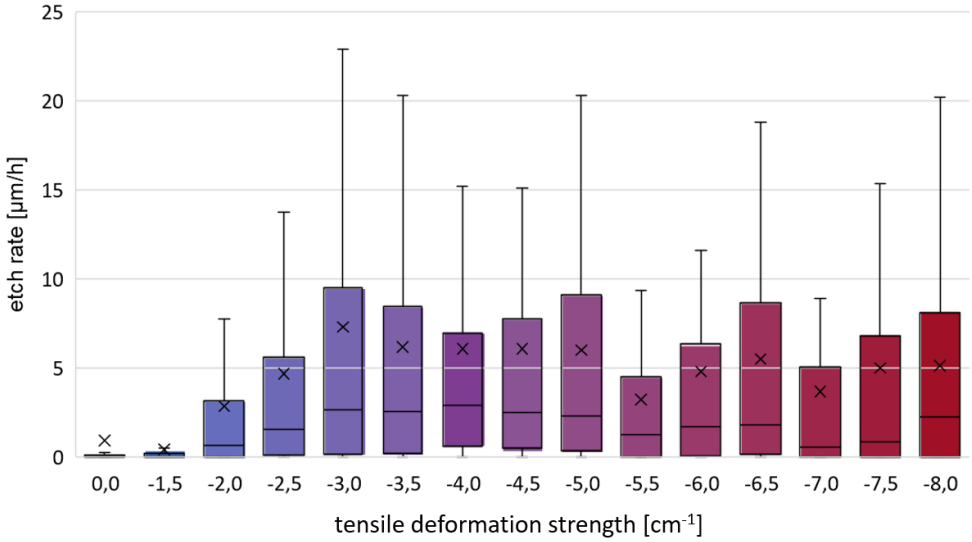


**Figure 1.** The correlation between the acidic etch rate and the deformation strength of tensile strained silicon on mechanical and thermal treated samples.

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# Impact of mechanical strain on 22 nm FDSOI device performance

**P25**

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**Abstract**

In this study, a novel nanoindentation-based approach[1] to induce localized stress/strain fields in device materials with precise lateral positioning and accurate loading is proposed. The selected technique needs less sample preparation efforts and allows to apply consecutive loading profiles compared to a single adjustable stress state achieved by conventional bending experiments. Additionally, the approach enables to investigate mixtures of tensile and compressive stress conditions, and therefore, the simulation of given mission profiles representing use conditions of materials and devices. A better estimation of interaction Chip - Board is approachable due to a packaged test structure that is comparable to a final microelectronic product. Ring oscillator (RO) test structures manufactured in 22 nm FDSOI technology were selected and used to achieve a higher sensitivity for device parameter variations during mechanical loading. The test structure can detect relatively small parameter changes due to the serial connection of the inverter structures and the resulting superposition of parameter deviations. The obtained results correlate the influence of mechanical load on the relative RO frequency shift, and they provide an estimate for package-related stress influencing the device performance.

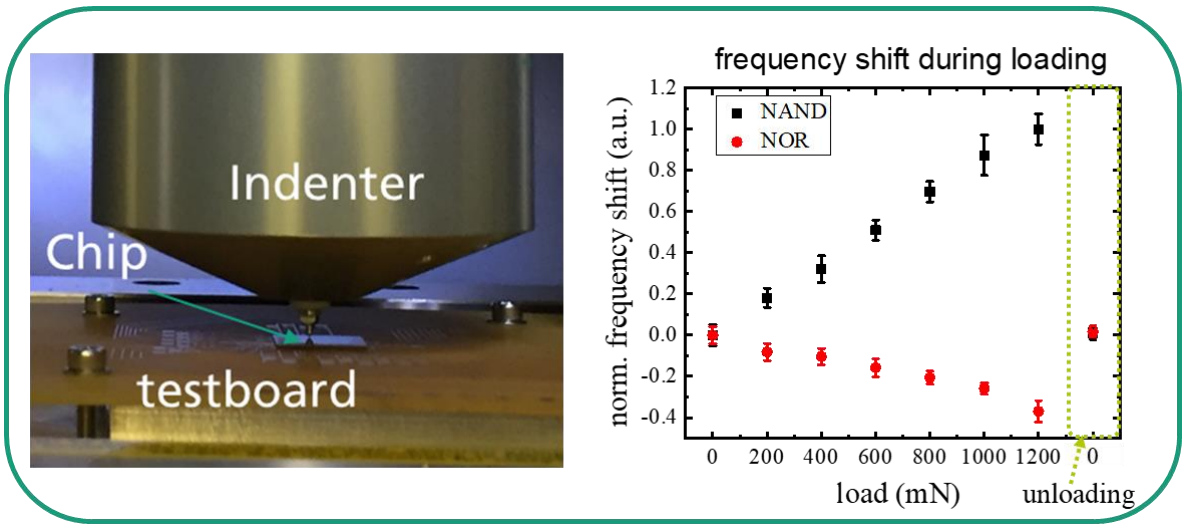


**Figure 1.** Experimental setup used for nanoindentation and obtained frequency shifts for NAND and NOR ring oscillator test structures.

**Acknowledgments**

This work was performed within a collaboration of GLOBALFOUNDRIES Dresden and Fraunhofer IKTS in the project IPCEI/Ringo.

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# Microstructure modelling and simulation of properties of advanced materials

P26

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**Abstract**

The microstructure of the advanced materials has often a great influence to specific materials properties. To simulate those materials properties precise parametric models are necessary. In this paper we present a work flow and tools that were used to develop 3D finite element models for two microstructures. The used material systems include: (a) a ceramic-carbon composite material and (b) a hierarchical structure made of $MoNi_4$ / $MoO_2$ on a Ni foam used for water splitting. The major workflow is to analyse microscopic images (e.g. SEM, TEM and XCT), and to build a 3D model based on the microstructure. The material properties can be simulated and optimized using the built model and allow to change geometric and physical properties of each component individually. The Young's modulus, tensile strength, fracture toughness, electrical and thermal conductivity will be investigated using 3D model for ceramic composite material system. The water splitting property will be predicted / improved by changing tortuosity, active surface area, size distribution, porosity and ellipsoid factor for the hierarchical material system. Mechanical stability for the system will be investigated as well.

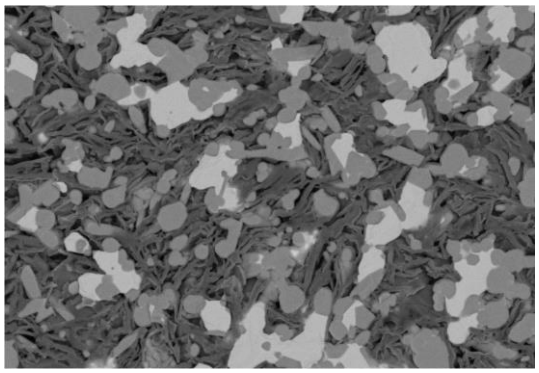

**Figure 1.** SEM image of Microstructure of the Ceramic-Carbon Composite

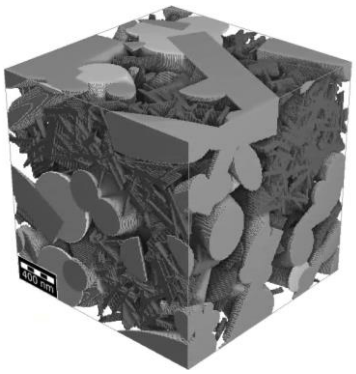

**Figure 2.** 3D model built

**Acknowledgments**

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# Improving ionic conductivity of new energy materials by taylored grain boundary design

P27

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Despite research activities for more than a century [1], the conductivity of the electrolyte material still limits the use of solid oxide fuel cells (SOFCs). The work of Kosacki et al. [2] confirms that the grain boundaries between electrolyte and insulator particles can have even higher ionic conductivities than the electrolyte itself, which indicates a new possibility to increase oxygen ion conductivities of the electrolyte material. Here we describe a novel approach how to make use of this unusual property to decrease the resistance. Due to its high ionic conductivity, Ceria has been chosen as electrolyte candidate and Mg was chosen to be the insulator in this work.

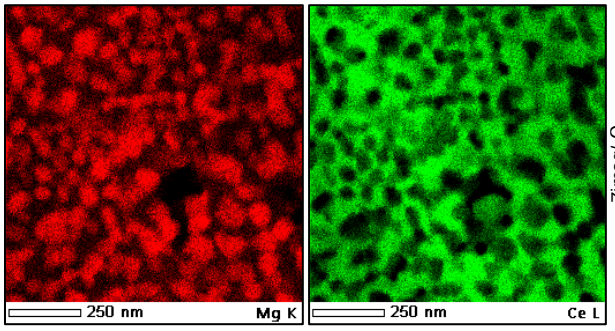


**Figure 1**. High-resolution TEM images of the composite samples.

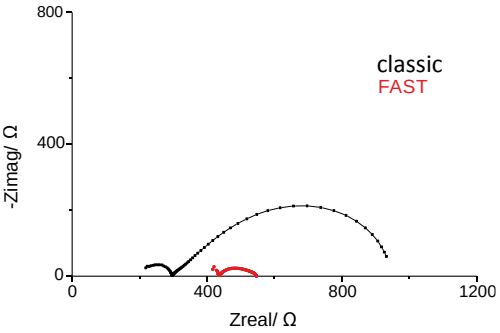


**Figure 2**. Nyquist plots of the impedance spectra of FAST and classically sintered samples at 540 °C.

In order to obtain highly conductive grain boundaries, a well-mixed material with small grain size is required. In this work, GDC and MgO nanoparticles with diameters between 10 and 20 nm were synthesized using a self-propagating high temperature synthesis method. This method do not result in observable particle aggregation [3]. A classical sintering method as well as the Field Assisted Sintering Technology (FAST) were applied to sinter the samples. Figure 1 shows the high-resolution TEM images of the FAST sintered ceramic sample. The domains of GDC and MgO are well mixed and the grain size is around 100 nm, which is obviously much smaller than that of the sample that was sintered using the classical sintering method.
Figure 2 shows the electrochemical analysis result as Nyquist plots, The FAST sintered ceramic has much lower resistance compared to the classically sintered sample with similar sample size.
Therefore, it can be concluded that GDC and MgO nanoparticles with diameters of about 10 nm can be synthesized with narrow particle-size distribution and no aggregation by the self-propagating high temperature synthesis method. The usage of the FAST sintering method can avoid the enlargement of the grain size during the sintering process and leads to a larger grain boundary area, connected with a higher conductivity.

**Acknowledgment**

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# Calculations of micropore carbon materials permeability based on stead-state pore-scale flow in 3D microstructure created based on X-ray computed tomography

P28

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**Abstract**

Pores structure determines many processes in material science: from manufacturing of composite materials to degradation of refractory or building materials. Knowledge of pore morphology allows better designing of materials. Several methods can be used for investigations of pore structure e.g. mercury intrusion porosimetry or scanning electron microscopy. One of the most promising method is x-ray computed tomography (XCT). It is nondestructive and noninvasive 3D imaging technique which can be used for investigations of microstructure of materials. Due to its nature, XCT might be-used to observe exactly the same volume of material before and after degradation processes. Moreover information obtained from XCT might be used in modeling. Observed microstructure can be used to create a geometry for calculations in 2D or 3D. In this work permeability of experimental micropore material is investigated based on the solution of steady-state pore–scale flow of molten metal in geometry representing real material pores in 2D and 3D. Geometry for calculations were created based on XCT measurements. The molten metal flow is described by the stationary Stokes equation for incompressible fluid. Permeability is calculated based solution of pore-scale flow model and Darcy law. Numerical calculation are performed using Comsol Multiphysics software.

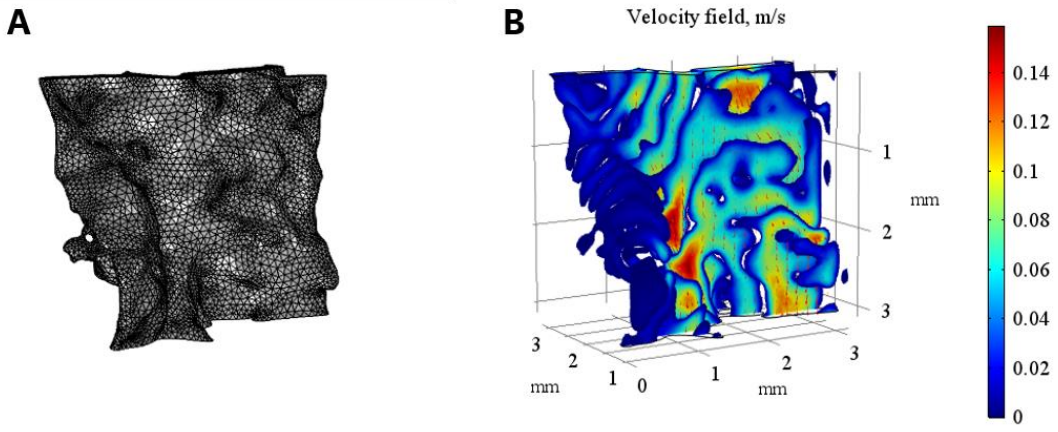


**Fig.1** A) 3D mesh created based on XCT observations. B) Result of the calculations – velocity field

**Acknowledgements**

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# In-situ micro-DCB study of crack propagation in Cu/Low-k BEoL structures

**P29**

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**Abstract**

Low-k and ULK materials with low Young's modulus and low fracture toughness lead to a higher risk of micro-crack propagation in the BEoL stack and failure caused by chip-package interaction (CPI). To prevent fracture and mechanical chip damage, metallic guard ring (GR) structures that are mechanically robust against packaging stress have been designed and integrated into BEoL stacks. The optimization of the GR design for future technology nodes and the evaluation of the risk of mechanical failure require the understanding of the kinetics of crack propagation and the identification of weak structures in the BEoL stack.

Laboratory transmission X-ray microscopy (TXM) and nano-XCT with a spatial resolution of < 100 nm have been applied to image 3D BEoL stacks and failures in microchips during mechanical loading, realized by a novel micro double cantilever beam (micro-DCB) test. The in-situ 3D imaging of the pathways of cracks in fully integrated multilevel interconnect structures allows to localize and to visualize the crack propagation behavior as well as crack stopping and deflection at GR structures nondestructively. Weakest layers and interfaces in the BEoL stack can be identified. With this novel technique it is possible to study the complex failure modes in realistic BEoL stacks, and to discuss the effects of process-induced thermomechanical stress and CPI on chip reliability. Based on the knowledge of how the crack propagation is modulated by the location of crack initiation and the fracture mode mixing, it is possible to draw conclusions for selection of dielectric materials to control the crack path and to ensure the required fracture resistance of BEoL structures for future advanced technology nodes. In addition, these studies deliver information to the effectiveness of particularly designed GR structures to stop or to deflect cracks to guarantee the requested product quality.

**Acknowledgments**

The authors thank Yvonne Standke (Fraunhofer IKTS, Dresden, Germany) in sample preparation. Financial support from SRC under Member-Specific Research Contract P30697 is greatly appreciated.

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# Laboratory-based nano X-ray microscopy for non-destructively visualizing the natural pollen and diatom frustule

**P30**

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**Abstract**

Natural materials can be used to get inspiration for design more reliable, efficient, and environment respecting materials based on their natural features such as miniaturization, hierarchical organizations, and adaptability [1]. That means, it is very significant to understand the hierarchical structures of these natural materials. Here, a laboratory-based X-ray microscope (TXM) is used to investigate the 3D structure of unstained whole pollen grains and diatoms. Pollen grains contain the male gametes among spermatophytes in plant and diatoms are natural single algae cell with cell walls of biogenic silica called frustules, both are abundantly found in nature, have large variations in morphology and hierarchical architecture (from nanometer to micrometers) [2,3]. In the study:

(i) The structural features of the whole unstained pollen (Scots pine) are visualized by TXM with a resolution better than 100 nm. Both surface and internal morphology of the pine pollen are clearly visualized. With the three dimensional (3D) data, the specific volumes of different layer structures are calculated [4].

(ii) Diatoms frustules are visualized nondestructively by TXM and compared with the images from the destructive focused ion beam/scanning electron microscopy (FIB/SEM) [5].

(iii) Two imaging modes, absorption contrast and Zernike phase contrast, are quantitatively discussed particularly for biological objects compared to inorganic objects, in order to reach the best image quality in practice [6].

According to the studies above, TXM provides detailed 3D structural information of pollen grains and diatoms non-invasively, complementary to other imaging techniques. Tomographic reconstruction could reveal detailed structural information as a basis for structure and physiological studies. The Zernike phase contrast mode provides the contrast necessary for imaging the biological samples. Based on these 3D structural data and techniques, the understanding of functionality and potential applications of diatoms and pollen could be improved.

**Acknowledgments**

The authors would like to sincerely thank Dr. Zhongquan Liao for fruitful discussions, and Yvonne Standke for imaging pollen with SEM, both at Fraunhofer IKTS Dresden, Germany.

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# X-ray tomography applied to determine the calcitic prismatic layer in *Pinctada margaritifera* shell

**P31**

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**Abstract**

Mollusk shells are biocomposite materials composed of calcium carbonate crystals in a form of calcite or aragonite which are embedded within organic matrix. $CaCO_3$ crystals may form different spatial configuration or microstructures in several layer. The outer layer of selected bivalves shells is composed of one of such structure. This structure named calcitic columnar prismatic (CCP) [1] is made of long lamellas which elongate according to growth direction. The prisms are simplistically called grains and treated as the single crystals [2] but some studies revealed that they are divided into some smaller units [3,4]. The prisms are separated by thin organic sheaths controlling nucleation process and determining the orientation and growth of calcium carbonate crystals. This construction demonstrates unique mechanical properties like high compression strength and high toughness at relatively low mass [5] that's why shells become natural inspiration to create synthetic, ceramic-based composite materials with superior properties. In the present work *Pinctada margaritifera* shell, in which CCP structure occurs, was submitted to evaluation using X-ray computer tomography.

The shell of bivalve *Pinctada margaritifera* consists of two layers: outer calcitic prismatic and inner aragonitic nacreous. Because the nacreous part was intensively studied the prismatic part was taken under consideration. The investigations were carried out to observe mechanical response of calcitic lamellas building the outer layer of *Pinctada margaritifera* shell. In that case the sample which had 50×50×200µm was stepwise indented using Berkowich and cube corner tip. The load was applied in the middle of the sample. After each loading step the images were provided. After observing some cracks in the material the loadings were performed few more times and the test was stopped.

The observation revealed that CCP layer of *Pinctada margaritifera* shell displays relatively high fracture resistance. The cracks propagate through the lamella until they reach organic phase which acts like a brake. What is more, cracks do not propagate inside the material but facilely.

The microstructural observations are important because they can be a biomimetic basis for the creation of new materials exhibiting unique mechanical and functional properties.

**Acknowledgments**

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# Hemocompatibility of polymer-based coatings dedicated to functionalization of an animal origin heart valve

P32

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**Abstract**

The aim of the work was to develop polymer-based coatings, which were designed to modify animal origin decellurized tissue. Acellularization process was conducted using chemical and enzymatic techniques. Polyelectrolyte Multilayer films (PEMs) were used in two variants: *Chitosan/Chondroitin Sulfate (Chi/CS)* and *Poly-L-lysine/Sodium hyaluronate (PLL/HA)*. Films were deposited using Layer by Layer method, which is based on electrostatic interactions between polyelectrolytes. Process was conducted by automatic dipping machine in two configurations: 24 bilayers and 12 bilayers (Fig.1). The different variants of chemical and physical cross-linking were applied. Chemical stabilization was performed using EDC (1-Ethyl-3-(3-Dimethylaminopropyl)Carbodiimide) and NHS (N-Hydrosulfosuccinimide) solutions mixed in 1:1 ratio. As an alternative for chemical cross-linking process, PEMs were modified with nanoparticles deposited by physical vapor deposition process (PVD) in argon atmosphere. For this reason, amorphous hydrogenated carbon coatings (a-C:H:N) were applied with thickness of 10÷15 nm.

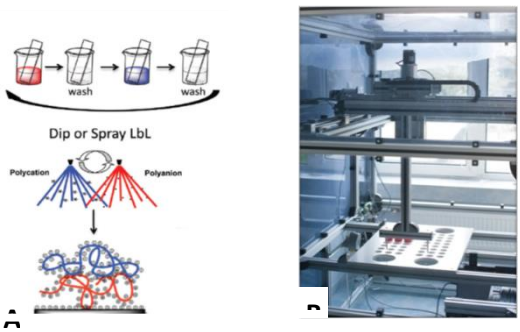


**Figure 1.** Polyelectrolyte Multilayer Film deposition process: A. Basics of the Layer by layer method [1,2], B. Automatic dipping machine, Laboratory of IMIM PAN

Human Umbilical Vein Endothelial Cells (HUVEC) were seeded on standard PEMs and coatings modified with fibronectin, which is one of the extracellular matrix proteins that support endothelial adhesion due to presence of the RGD sequences. Samples were stained with Alexa Fluor 488 for visualizing cytoskeleton structure and DAPI to show number of nuclei. The CLSM analysis showed lack of cell adhesion to materials with no protein and also confirmed that fibronectin stimulates monolayer formation.

Prepared coatings were analysed for hemocompatibility by cone and plate test. Investigation of activation and aggregation allowed to assign type of materials, with the smallest level of activation and formation of monocyte-platelet aggregates circulating in blood taken after test. Based on flow cytometry results, it has been shown that the *Chi/CS 24 bl non cross-linked* coating indicates the lowest activation of platelets in blood.

**Acknowledgments**

The research was financially supported by the European Union from resources of the European Social Fund (Project No.WND-POWR.03.02.00-00-I043/16).

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# Nano-XCT based FEM study of diatom frustule

**P33**

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**Abstract**

Diatoms are unicellular, photosynthetic microalgae with intricate shell morphologies and features. Their unique, three-dimensional (3D) structured silica exoskeletons ranging from the nano and sub-micron to micrometer scales, have drawn attention from a variety of research fields due to their extraordinary properties [1]. They have been proposed to use in a range of applications, including as templates for drug delivery carriers, oil/water separation membrane, optical devices, and metamaterials design. Several studies have found that diatom shells show unique mechanical properties such as high specific strength and resilience and these properties arise from its hierarchically arranged structural components. In this work, in-situ nanoindentation and subsequent X-ray computed tomography (XCT) based Finite Element analysis with the same sample was conducted to shed light on the mechanics of didymosphenia geminata frustule. The method described in this study holds great potential for biomimetics, to explain how morphology is pivotal to the mechanical performance of frustules' hierarchically arranged structures. The results provide information for the design of damage-tolerant lightweight materials and structures.

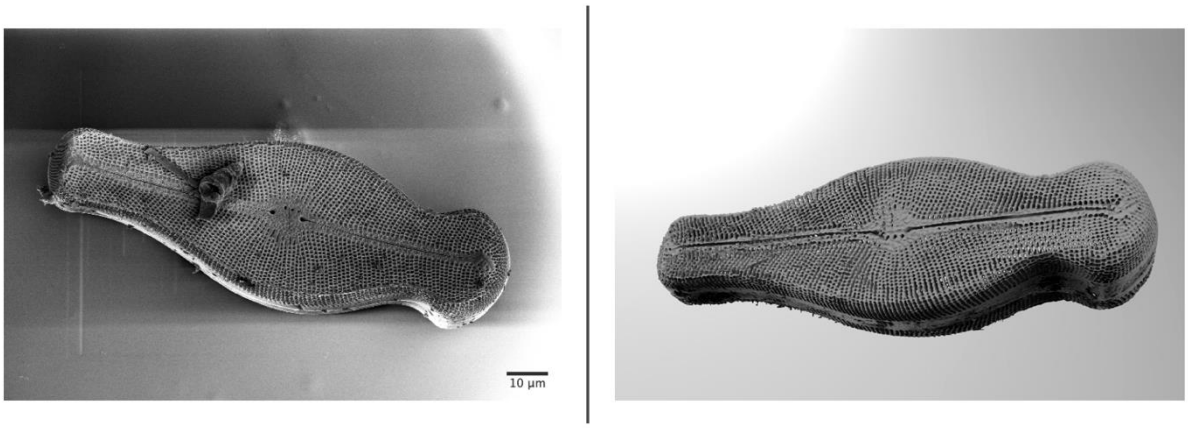


**Figure 1.** SEM image of diatom frustule and its respective FEM model. The FEM model was built based on reconstructed nano-XCT data. It consists of 21.6m tetrahedral, 1.2m hexahedral elements.

**Acknowledgments**

The authors thanks Izabela Zglobicka for providing Didymosphenia geminata frustule samples.

**References**

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# Identification of failure modes in semiconductors caused by mechanical loading using a combined approach of acoustic emission and X-ray-microscopy

**P34**

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**Abstract**

Due to the recent developments in the automobile industry, especially in the field of autonomous driving, the requirements for semiconductor products regarding functionality and performance are continuously increasing. Additionally, the demands regarding reliability are augmented compared to e.g. consumer electronics products. This is due to the harsher operating environments and the resulting loading scenarios. These requirements can only be met by the implementation of semiconductor products manufactured in advanced CMOS technology nodes.

Due to the increasing complexity of these products, non-destructive analytical methods become increasingly important to evaluate the occurrence as well as the type of damages. The selection of adequate methods should enable an analysis of induced damage without any further sample preparation. Considering the size of semiconductor products and their internal structures X-ray microscopy is a promising analytical method to detect and to characterize damages. A specific procedure has been developed for a suitable X-ray microscope to identify different failure modes and evaluate the reliability of specific semiconductor devices. The mechanical Back End of Line (BEoL) stability in advanced packages with Cu-Pillars is of particular interest.

In order to inflict damages to the given semiconductor devices, shear forces are applied by lateral indentation forces [1], [2]. The analysis of the inflicted damage is primarily conducted using X-ray microscopy. Based on numerical calculation of the X-ray attenuation, the applicability and suitability of this method for a specific sample can be estimated prior to the measurement. The calculation is based on the transmission properties of the investigated sample and the energy of the used X-rays. In order to obtain a reliable identification and characterization of different types of damages, X-ray microscopy is complimented by other methods like scanning electron microscopy (SEM), scanning acoustic microscopy (SAM) as well as acoustic emission (AE). Latter is applied *in-situ* during the indentation to determine the moment and severeness of the induced damage. The results combined in a multi method analysis to obtain a better understanding of the origin and the propagation of damages in the BEoL stack. The results of the analyses can be used for the development of specific future applications of semiconductor products in the field of automotive.

**Acknowledgments**
A special thanks to Mareike Stephan, Lars Schubert (Fraunhofer IKTS), Jens Paul (GLOBALFOUNDRIES) as well as Tanja Graf, Knut Schmidt, and Andreas Aal (Volkswagen AG).

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# A novel in-situ 4PB device: Introduction and applications

**P35**

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**Abstract**

In many industries, stress engineering plays an important role for the development of new products and materials. The novel rotational 4-point bending tool (r4PB, Figure 1) uses rotational sample clamps instead of standard fixed grip sets that are displaced towards each other in conventional tools. This enables a low building height for various applications while achieving high loads. Two separate torque and displacement controlled sample mounts with torque feedback control enable automated contact finding, calibration and precise positioning. The tool is designed for use in atmosphere in combination with additional analytical tools and for vacuum use in SEM/FIB applications. Here, the unique working principle (patent pending, [1]) allows various bending modes and alternating compressive and tensile stress on sample surfaces without changing the setup and without handling the sample outside the vacuum chamber, Figure 2.

New applications are enabled by the adjustable surface stress in combination with analytical tools like SEM/FIB, Raman spectroscopy, EBSD, nanoindentation [2], SPM imaging and other techniques, which investigate the sample surface of a given specimen. Some examples of use are shown in this study.

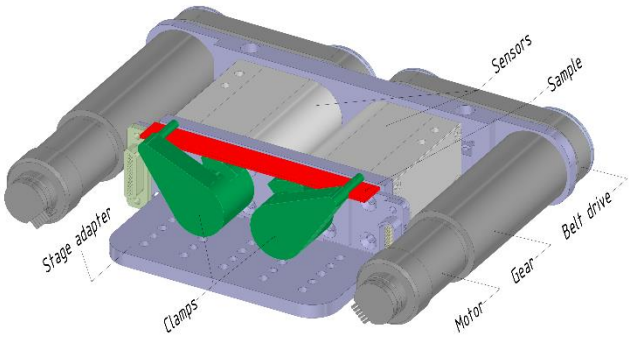


**Figure 1.** CAD drawing of the in-situ 4PB device.

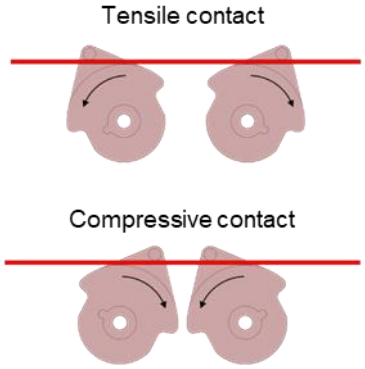


**Figure 2**. Working principle of the rotational sample clamps. Other clamp designs available.

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[2] C. Sander, et al., *7th DNS* **2019**, Poster №36: "Investigations of Oxide Thin Film Fracture by Nanoindentation"

# Investigations of oxide thin film fracture by nanoindentation

**P36**

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**Abstract**

Failure due to fracture in dielectric materials remains a critical risk in semiconductor manufacturing. The main goal of this study is to support the prediction of crack behavior in oxide thin films. To be able to predict the fracture of these films, the fracture toughness has to be evaluated. Here, the crack length of indents with a cube corner tip is examined as input for FEM simulations to compute the fracture toughness while the constraints and intrinsic stress of real deposited films are maintained. A requirement from the applied FEM model is a sufficient crack length to indent base length ratio $a_{crack}/a_{indent} > 1$. During initial indentation experiments, chipping was observed. For better understanding of the indentation process, in-situ indents were performed. Experiments on glued samples did not yield long enough crack length, Figure 1. Therefore, additional tensile stress was applied with a custom-made rotational 4PB system [1, 2] to increase in-plane stresses for longer crack length, Figure 2.

This study includes three different nanoindentation experiments to achieve a better understanding of the oxide fracture behavior:

- Nanoindentation of standard thin films on substrate,
- Nanoindentation of bent sample beams with an in-situ 4PB device,
- in-situ SEM nanoindentation with crack length measurement and observation of delamination events.

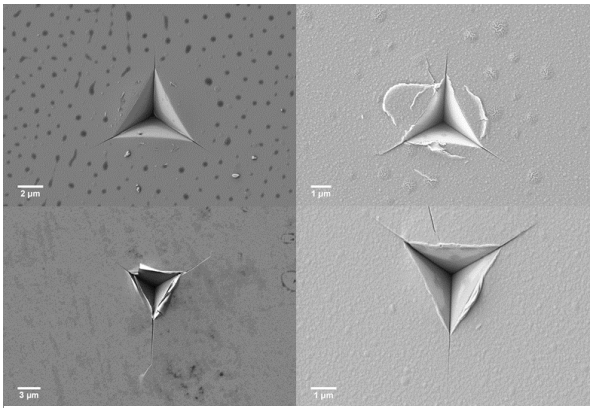


**Figure 1.** SEM micrographs of the indents on sample A (left) and sample B (right): upper images on fixed substrate, lower images with 100 % tensile surface stress applied.

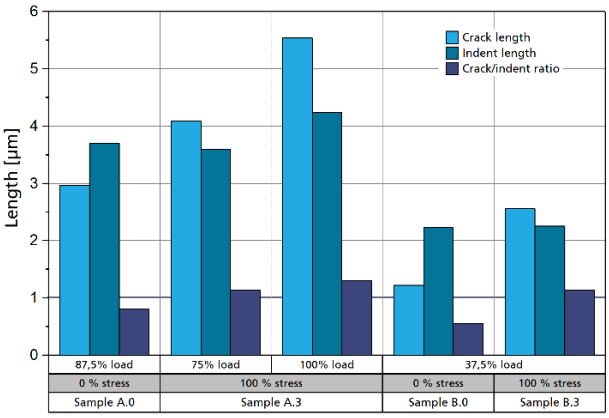


**Figure 2.** Crack length on sample A and B with varying stress and indentation loads.

**Acknowledgments**

The authors thank Matthias Kraatz (FhG, IKTS) for valuable discussions and Philipp Kürschner and Thomas Chudoba (both with ASMEC company) for help with the in-situ SEM indenter system.

**References**

[1] C. Sander, et al., patent pending PCT/EP2018/058190, "Device for carrying out Bending Tests on Panel-Shaped or Beam-Shaped Samples"

[2] C. Sander, et al., *7th DNS* **2019**, Poster №35: "A novel in-situ 4PB device: introduction and applications"

# Tailoring surface potential of electrospun Polyvinylidene fluoride (PVDF) fibers

P37

Piotr K. Szewczyk[1], Sungkyun Kim[2], Sohini Kar-Narayan[2] and Urszula Stachewicz[1]

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**Abstract**

Electrospinning is a versatile technique used to fabricate polymer fibers for many applications area such as bioengineering,[1] through water collection,[2] filtration,[3] nanogenerators[4] or smart textiles.[5] The electrospinning parameters have a significant impact on the properties of obtained fibers.[6] One of them is voltage polarity that has been proven to affect triboelectric properties of nanogenerators in case of electrospun polymethyl methacrylate fibers[4] and improved cell proliferation on PVDF fibers obtained at negative voltage polarity.[1]

In this study, we investigate further the voltage polarity effect on electrospun PVDF in terms of their surface properties for their triboelectric and piezoelectric performance as generators. PVDF fibers morphology was investigated using scanning electron microscope (SEM, Merlin Gemini II, Zeiss, Germany). The topography and surface potential of fibers were measured using an atomic force microscope with Kalvin probe capability (AFM, KPFM, MultiMode8, Bruker, USA).

In conclusion, we confirmed that electrospinning at different voltage polarities for PVDF resulted in similar morphology and average diameter of fibers. However, surface potential measure directly by KPFM showed the possibility of tailoring surface properties of PVDF fibers. The further research is going to include triboelectric and piezoelectric measurements of electrospun PVDF fibers produce with both voltage polarities.

**Acknowledgements**

This study was conducted within the funding from Sonata Bis 5 project granted by National Science Centre in Poland, No 2015/18/E/ST5/00230.The AFM and KPFM measurements were performed during short term scientific mission Grant from COST Action Context CA17107.

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# Controlling of mechanical properties of electrospun PMMA fibers via voltage polarity

P38

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**Abstract**

Electrospun polymer fibers due to their surface-to-volume ratio, mechanical properties and porosity are used extensively in various applications like air filtration, triboelectric generators (TENGs) and tissue engineering [1]. Fibers produced by electrospinning have unique mechanical properties due to the manufacturing method [2]. Many process parameters including humidity, flow rate, distance and the applied voltage polarity have significant influence on properties of polymer fibers [3]–[6]. In electrospinning technique, changing a voltage polarity allows to control of surface potential and moving of polymer chains [3], [7]. In our studies, we aim to verify the effect of voltage polarity on structural and surface changes of poly (metacrylate methyl) (PMMA) fiber in terms of their mechanical properties and crystallinity.

In this work, we used PMMA (Mw=350 000 g·mol-1) dissolved in dimethyloformamide (DMF) to produce aligned and randomly aligned and fibers using EC-DIF apparatus. We produce fibers with positive (PMMA+) and negative voltage polarity (PMMA-). Electrospun fibers morphology and diameters were investigated using Scanning electron microscopy (SEM). The mechanical testing of PMMA was performed using a tensile module equipped with a 1N cell. Surface chemistry of the produced electrospun fibers was analyzed with X-ray photoelectron spectroscopy (XPS) with monochromatic radiation from Al Kα (1486.6 eV). Crystallinity and glass transition temperature was verified using differential scanning calorimetry (DSC).

The fiber morphology and diameter were similar for both type of random PMMA fibers (1.51 ± 0.21 μm for PMMA+ and 1.62 ± 0.29 μm for PMMA-) and aligned fibers (1.54 ± 0.21 μm for PMMA+ and 1.65 ± 0.31 μm for PMMA-). From the mechanical testing of PMMA+ and PMMA- samples, we obtained the maximum stress for random fibers 129 kPa and 230 kPa, respectively. Interestingly, for aligned fibers we observed opposite effect. For PMMA+ the tensile strength was higher, equal to 320 kPa and PMMA- it was 180 kPa. The variations in mechanical strengths indicated clearly that the highest mechanical stress was obtained for aligned fibers.

Within this study, we showed the potential of controlling mechanical properties of electrospun fibers via voltage polarities. In conclusion, electrospun PMMA fibers were successfully produced using positive and negative voltages with similar average fiber diameters and morphology. Random PMMA fibers produced with positive voltage polarity (PMMA+) proved higher mechanical properties than PMMA fibers produced with negative voltage polarity (PMMA-). For aligned PMMA fibers, we observed reveres effect. The further studies include theoretical analysis of charge effect during electrospinning on their structure in relation to mechanical properties of PMMA fibers.

**Acknowledgements**

The study was conducted within the funding from SONATA Bis 5 project granted by National Science Centre in Poland, No 2015/18/E/ST5/00230. This research was conducted with infrastructure of International Centre of Electron Microscopy for Materials Science.

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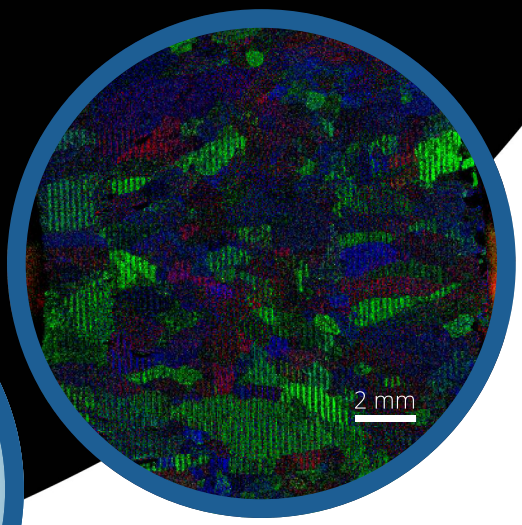


### BATTERIES
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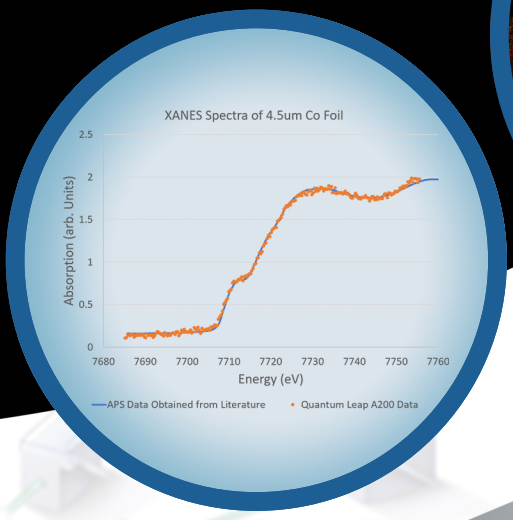
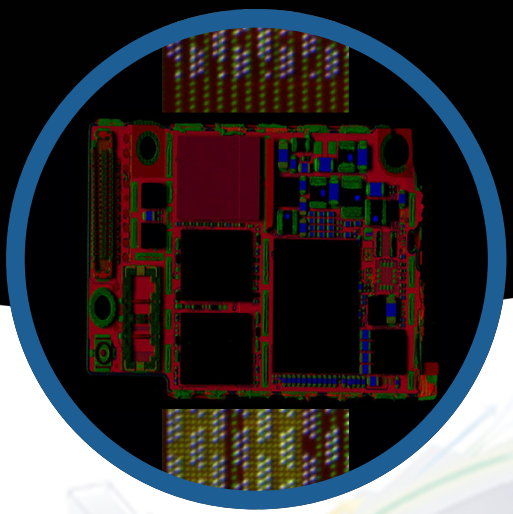
Metal Oxidation States




### ELECTRONICS
Dopants & LIDAR
Thin Film (nm) Measurements



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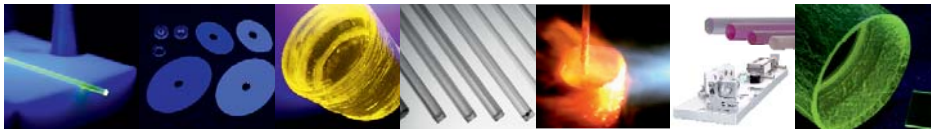

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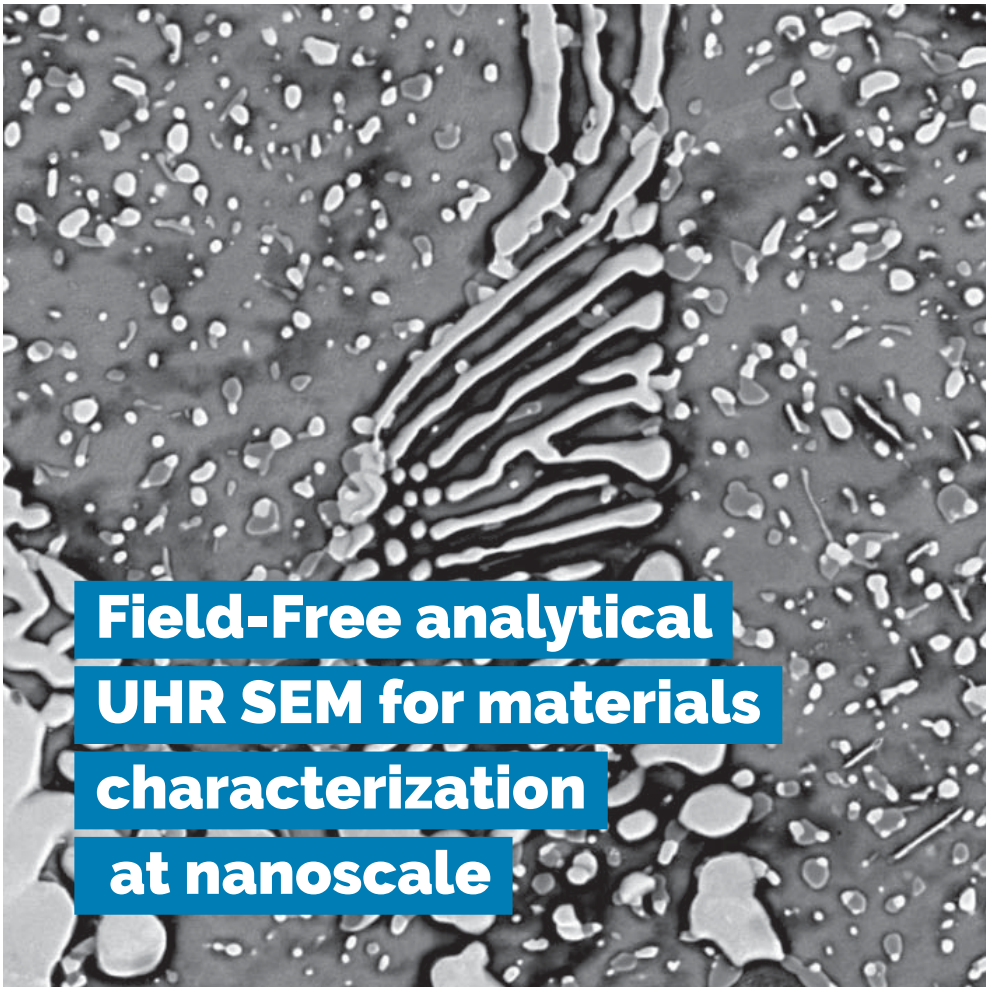


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**By entering the Username and Password you agree to the terms and conditions above.**

*Data privacy:*

Username and connection data (IP-, MAC-Address, times) will be saved and processed for up to 30 days, and used for monitoring errors, performance and possible misuse. This information will be handed over to law enforcement authorities as need be.