Fraunhofer NANOANALYSIS

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5th Dresden Nanoanalysis Symposium

"In-situ Microscopy"

Abstract booklet

September, 1st 2017
Dresden, Germany

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5th DRESDEN NANOANALYSIS SYMPOSIUM

"IN-SITU MICROSCOPY"

The 5th Dresden Nanoanalysis Symposium, jointly organized by the Dresden Fraunhofer Cluster Nanoanalysis (DFCNA) and the Dresden Center for Nanoanalysis (DCN) at TU Dresden and supported by the European Materials Characterization Council (EMCC), will be held at the Fraunhofer Campus Dresden, Winterbergstrasse, on September 1, 2017. 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: "In-situ Microscopy".

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

September 1, 2017, Dresden, Germany
Winterbergstraße 28, 01277 Dresden
Fraunhofer Campus Dresden, Lecture Hall

Organizational committee

Martin Gall, Fraunhofer IKTS, *martin.gall@ikts.fraunhofer.de*
Kristina Kutukova, Fraunhofer IKTS, *kristina.kutukova@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

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Invited speakers

- Jaroslav Klima, Tescan Orsay Holding, Brno, Czech Republic
- Tofail Syed, University of Limerick, Ireland
- Thomas Chudoba, ASMEC Dresden, Germany
- Mathias O. Mosig, Protochips, Morrisville/NC, USA
- Olivier Thomas, University Marseille, France
- Horst Borrmann, Max Planck Institute for Chemical Physics of Solids, Dresden, Germany
- Gisela Schütz, Max-Planck Institute for Intelligent Systems, Stuttgart, Germany
- Gerd Schneider, Helmholtz Center Berlin, Germany
- Wenbing Yun, Sigray, Concord/CA, USA
- Thomas LaGrange, EPFL Lausanne, Switzerland
- Robert Sinclair, Stanford University, Palo Alto/CA, USA
- Andrey Chuvilin, CIC nanoGUNE, San Sebastian, Spain
- Teodor Gotszalk, Wroclaw University of Science and Technology, Poland
- Rafal E. Dunin-Borkowski, Ernst Ruska Center for Microscopy, Jülich, Germany
- Erdmann Spiecker, University Erlangen-Nürnberg, Germany

Program

Friday, September 1

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

Welcome and Introduction (9:00 am – 9:10 am)
Ehrenfried Zschech
Fraunhofer IKTS Dresden and Dresden University of Technology, Germany

Session I (9:10 am – 10:50 am)
Chair: *Ehrenfried Zschech*

9:10 am – 9:40 am
KEYNOTE TALK
Instrumentation for a comprehensive investigation of the nano-world: Multimodal systems for imaging and analysis , **10**
Jaroslav Klíma, Tescan Orsay Holding, Brno, Czech Republic

9:40 am – 10:10 am
KEYNOTE TALK
Infrared (IR) in situ imaging for metrology in advanced manufacturing, **11**
Tofail Syed, University of Limerick, Ireland

10:10 am – 10:30 am
Beyond standard nanoindentation: New methods for the mechanical characterisation of surfaces and coatings , **12**
Thomas Chudoba, ASMEC Dresden, Germany

10:30 am – 10:50 am
Protochips™ in-situ Electron Microscopy Solutions: Capabilities and Applications, **13**
Mathias O. Mosig, Protochips Inc., Morrisville/NC, USA

Coffee break and Poster Session *(10:50 am – 11:20 am)*

Session II (11:20 am – 1:00 pm)
Chair: *Erdmann Spiecker*

11:20 am – 11:40 am
Mechanical properties at the nanoscale: Recent developments using nano-focused synchrotron X-ray diffraction, **14**
Olivier Thomas, University Marseille, France

11:40 am – 12:00 pm
New challenges in high-resolution diffraction, **15**
Horst Borrmann, Max Planck Institute for Chemical Physics of Solids, Dresden, Germany

12:00 pm – 12:20 pm
Scanning X-ray microscopy at MAXYMUS combining chemical, magnetic, depth and temporal sensitivity, **16**
Gisela Schütz, Max-Planck Institute for Intelligent Systems, Stuttgart, Germany

12:20 pm – 12:40 pm
Nanoscale spectroscopy and tomography with the HZB full-field transmission X-ray microscope, **17**
Gerd Schneider, Helmholtz Zentrum Berlin, Germany

12:40 pm – 1:00 pm
Advances in laboratory X-ray technology enabling synchrotron capabilities in home-labs
Wenbing Yun, Sigray, USA

Lunch and Poster Session *(1:00 pm – 2:00 pm)*

Session III (2:00 pm – 4:00 pm)
Chair: *Olivier Thomas*

02:00 pm – 02:20 pm
Aberration-corrected transmission electron microscopy of the structure and chemistry of epitaxial ceria thin films on yttria-stabilised Zirconia substrates, **18**
Robert Sinclair, Stanford University, Palo Alto/CA, USA

02:20 pm – 02:40 pm
Towards atomic resolution magnetic imaging of nanoscale materials in the TEM, **19**
Rafal E. Dunin-Borkowski, Ernst Ruska Center for Microscopy, Jülich, Germany

02:40 pm – 03:00 pm
Perspectives on combined nanosecond imaing and in-situ chemical analysis with EELS using high-speed electron microscopy approaches, **20**
Thomas LaGrange, Interdisciplinary Centre for Electron Microscopy (CIME), École Polytechnique Fédérale de Lausanne (EPFL), Lausanne, CH-1015 Switzerland

03:00 pm – 03:20 pm
Accessing reaction kinetics via direct imaging of atoms – example of graphene, **21**
Andrey Chuvilin, CIC nanoGUNE, San Sebastian, Spain

03:20 pm – 03:40 pm
Investigation of thermal and electrical structure parameters using combined scanning probe microscopy methods, **22**
Teodor Gotszalk, Wroclaw University of Science and Technology, Poland

03:40 pm – 04:00 pm
In situ mechanical testing and manipulation of nanomaterials in FIB-SEM and TEM: New approaches and applications, **23**
Erdmann Spiecker, University Erlangen-Nürnberg, Germany

Best poster award ceremony and closing remarks (3:40 pm – 4:00 pm)
Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

Coffee and cake, and poster session (4:15 pm – 5:30 pm)
Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

Lab tours at Fraunhofer Campus Dresden (4:15 pm – 5:30 pm)
Ehrenfried Zschech, Fraunhofer IKTS Dresden and Technische Universität Dresden, Germany

Option I
Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM

Option II
Fraunhofer Institute for Material and Beam Technology IWS

Option III
Fraunhofer Institute for Ceramic Technologies and Systems IKTS

POSTER SESSION
4:15 pm – 5:30 pm
Chair: Gerd Schneider, Helmholtz Zentrum Berlin, Germany

Poster presentations

Abstracts
-Talks-

KEYNOTE TALK

Instrumentation for a comprehensive investigation of the nano-world: Multimodal systems for imaging and analysis

Ing. Jaroslav Klíma*

TESCAN ORSAY HOLDING, a.s., Libušina třída 1, 623 00 Brno, Czech republic

**jaroslav.klima@tescan.com*

In the last decade, we have been seeing a dramatic need for high resolution instrumentation for nanoscale analysis. The growing demands from semiconductor, life science and material sciences are driven by the continuous trend of miniaturizing the key components to nanoscale. However, in addition to the steadily increasing resolving power of all types of microscopes, obtaining very complex information about the investigated micro- and nano- structures is required.

As a result, we could often see the expression **"Correlative Microscopy",** which typically means imaging of the same objects by various types of microscopes (e.g. light and electron microscope). Although, many features of objects under investigation have been discovered this way, the overall information about the sample is still very limited. Besides morphological information, also the information about elemental composition, chemical bonds, crystallinity, electrical and mechanical features, strain of materials in micro- and nano- scales, etc. is highly needed for better characterization of the particular sample. Therefore, the phrase **„Multimodal Systems for Imaging & Analysis"** seems to be a much more suitable expression for such class of instrumentation.
The origins of such systems can be found in the1970s when electron microscopes started to be routinely coupled with EDS. Nevertheless, those first attempts can hardly be called as "Integrated system". As the first, truly integrated SEM and EDS systems can be considered dedicated systems for gunshot residue analysis (GSR) and especially the systems for the automatic mineralogical analysis (e.g. QEMSCAN, TIMA). Both systems combine morphological and analytical data in one file and the segmentation of objects and their classification is done using both types of data.

In our vision, **the truly integrated systems** shall contain several devices for **imaging** (EM, LOM, AFM) as well as **analytical instruments** (EDS, EBSD, SIMS, SPM, RAMAN), **tools for loading** objects mechanically, with thermal stress, nano-indentors, probing devices, etc.. Further combination of analytical SEM with a focused ion beam (FIB-SEM) will push the limits of material micro- and nano-analysis into the third dimension by sequential FIB slicing followed by analytical imaging. In today's world, the users also strongly emphasize not only high performance but also high throughput and easiness of use and generally prefer the delivery of the "off of the shelf" solution including efficient "work-flows" from a single supplier.

This means that all components shall be **controlled and synchronized by one central control unit**, all components shall produce data in compatible data formats and the operator shall communicate with all components by means of one user-friendly software and GUI.As the system will provide **big data** files – a huge challenge will be to create software for acquisition and/or import, handling, processing, analysis and evaluation of big data. Also communication with databanks, comparing results with archives of spectra and images and proper reporting. **Overall, high level of automation** incl. **elements of artificial intelligence** is required. It is unable to maintain compatibility with all manufacturers of additional accessories - necessity of exchange of confidential information. Thus the targeted integration can be ideally accomplished within one single entity and we must expect next wave of accumulation of R&D and manufacturing capacities from the business point of view accumulation of capital.

Figure 1. SEM (In-chamber SE, BSE, STEM; In-lens SE, BSE) + FIB +GIS + nano-manipulator, TOF SIMS + RAMAN + LOM + EDX + EBS

KEYNOTE TALK

Infrared (IR) *in situ* imaging for metrology in advanced manufacturing

S. A. M. Tofail*[1], A. Mani[1], R. Mouras[1], J. Bauer[2], C. Silien[1]

[1]*Department of Physics and Bernal Institute, University of Limerick, Ireland*
[2]*Department of Biomedical Engineering, Wrocław University of Technology, Poland*

**Tofail.Syed@ul.ie*

Metrology, the science and act of measuring, is important due to the market pull for consistent and reliable performance of manufactured parts, goods and products to establish adequate and standardised measures to ascertain functional properties, shape and dimensional tolerance and performance. It is also important in the monitoring, control and optimization of device fabrication and production process to keep cost low by better utilization of raw materials, trouble-shooting, and increasing production yield and energy efficiency.

Metrology can be carried out at the site of production (*in situ*) or outside (*ex situ* or off line). In the *in situ* in-line metrological approach, metrology tools can be integrated within the production process for investigation at the time coinciding with the production. In the *in situ* on line metrology approach, the manufactured job can be monitored still at the site of the production but not necessarily at a time that coincide with the production. Fast imaging techniques that employ, for example, optical, X-ray, e-beam, ultrasound or scanning probes can be used for such metrological purposes.

In the *ex situ*, at-line metrological approach, a sample can be extracted from an arbitrarily chosen point from the job that is being manufactured. The extracted sample can then be transferred outside the chamber for measurement and analysis. Finally, the completed job or a part of the job can be sent for *ex situ*, off line metrological analysis. This approach does not provide real time process information and may require destructive process in sample preparation but provides a wealth of information regarding process variables that may have deterred desired performance or adequate quality.

Currently available non-destructive testing (NDT) can be a good starting point to detect and characterize defects in metals, polymers, ceramics, composites, hybrid to establish structure-property relationship that determines performance of manufactured objects. Most manufacturing relies some form of dimensional metrology e.g., contact/optical coordinate measurements during making of the object. Contact techniques, while accurate in measuring regular geometric objects, are slow and cannot measure buried features or defects. A switch to non-contact method such as those based on optical, electrical or magnetic measurements can be suitable for both in line and on line metrology *in situ*. Visible light imaging usually provides poor image contrast in polymers, biological materials and transparent ceramics. This can be improved by switching to non-linear optics where the contrast is generated from the materials inherent chemical and structural properties.

We highlight the use of infra-red (IR) sources in particular for *in situ* imaging for metrology in advanced manufacturing. After a brief introduce to the fundamentals and current state of the art of IR thermography and spectro-microscopy we will discuss the scope, current use and opportunity of these IR techniques as metrological tools in advanced manufacturing. We will then identify barriers against enable rapid, real time ambient measurements and technological directions that are required to overcome such barriers.

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Beyond standard nanoindentation: New methods for the mechanical characterisation of surfaces and coatings

Thomas Chudoba*

ASMEC GmbH, Maria-Reiche-Str. 1, 01109 Dresden, Germany

**t.chudoba@asmec.de*

Many different test methods are used for the mechanical characterization of surfaces and coatings. Nevertheless it is often not possible to achieve a good correlation between test results in the laboratory and the behavior of components in an application. The reason for that is the incomplete knowledge of the real application conditions and the difference to the test conditions. In real applications there exists mostly a combination of normal, lateral, torsional and dynamic forces.

Standard nanoindentation technique uses only normal forces for the measurement of hardness and modulus as main mechanical parameters. In the talk it will be explained how dynamic and lateral forces can be measured by modern instruments and how they are considered in the analysis. The difference between quasi-static and dynamic hardness and modulus measurements is explained and the advantages of dynamic tests are illustrated. It will be further discussed how the dynamic capabilities can be used for fatigue tests.

With a lateral force unit as second measuring head for nanoindenters it is possible to simulate lateral forces and displacement with high accuracy additionally to normal forces. This is a step closer to application conditions. Different analysis methods for lateral force-displacement curves will be introduced. This includes the analysis of many reciprocating cycles in micro wear tests, the determination of the lateral contact stiffness and a new method for the determination of the Poisson's ratio.

In a last part several methods for a mapping of mechanical surface properties will be explained. Such a mapping gives better access to the homogeneity of surfaces and to the distribution of components in a composite. The weakest points in a surface can be better found by a mapping than by a single row of indentations. Examples are given for an Al-Si composite and diamond grains in a SiC matrix (see figure 1).

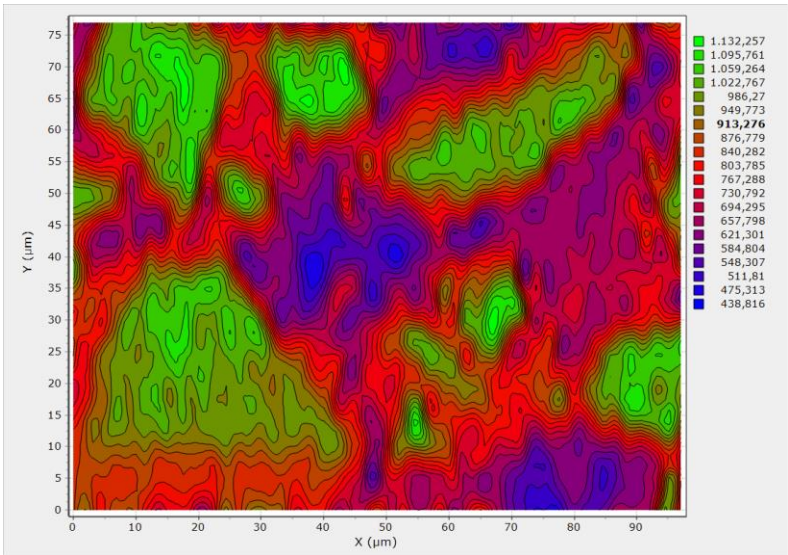

Figure 1. Young's modulus map of diamond grains (green) in a SiC-Si matrix in a field of 98 x 77 µm. The legend depicts modulus numbers.

Protochips™ in-situ Electron Microscopy Solutions: Capabilities and Applications

Mathias O. Mosig

Protochips Inc., 3800 Gateway Centre Blvd Site 306, Morrisville, NC 27560, USA

contact@protochips.com

New innovations are transforming the Transmission Electron Microscope (TEM) from a simple high-resolution image acquisition tool into a nanoscale materials research and development laboratory. Researchers can now better understand material behavior by analyzing samples in real-world gas or liquid environments, at high temperature and with ultra-low noise electrochemical and electrical biasing techniques. With the new in situ tools from Protochips, materials research occurs in highly controlled environments at high resolution without sacrificing the analytical capabilities of the TEM such as EDS. Applications for these tools include heterogeneous catalyst reactions, imaging of living cells, nanostructure nucleation and growth, battery and fuel cell materials, high temperature nanoparticle behavior, soft materials, and semiconductor devices.

In this presentation we show the most recent results using the Protochips Atmosphere™ gas cell, the Protochips Poseidon™ Select flowing liquid and electrochemistry cell, and the Protochips Fusion™ heating and electrical biasing system.

The Protochips Atmosphere™ system combines the Protochips patented silicon carbide MEMS heating technology, closed-cell holder design, and gas handling manifold, with our innovative Clarity workflow driven software allowing for atomic-scale resolution at gas pressures up to 1 atm and sustained temperatures up to 1000°C. The system is also compatible with analysis tools including EDS and EELS. Recent results on gas-phase catalyst reactions will be presented.

The Protochips Poseidon™ Select liquid cell surrounds samples in a self-contained, fully hydrated, hermetically sealed chamber directly within the TEM. Poseidon comes with a wide range of applications, from life science to battery research and corrosion studies. It features in situ electrochemistry capabilities, which enables the observation and characterization of electrochemical reactions in realistic reaction environments in real-time, and now offers liquid heating, for experiments in growth and reaction kinetics and temperature sensitive samples. Poseidon™ Select can also be used to capture high resolution images of living cells and biological processes in their native, hydrated environment.

Protochips Fusion™ offers best in class in-situ TEM heating and electrical biasing performance, simultaneous heating and biasing (electro-thermal mode), and features closed-loop temperature feedback without introducing significant thermal drift. Like Atmosphere, Fusion is based on the Protochips patented silicon carbide heating technology, and the user friendly Clarity workflow software.

Mechanical properties at the nanoscale: Recent developments using nano-focused synchrotron X-ray diffraction

Olivier Thomas*[1], Thomas Cornelius[1], Stéphane Labat [1], Marie-Ingrid Richard [1, 2]

[1]Aix Marseille Univ, Université de Toulon, CNRS, IM2NP, Marseille, France
[2]ESRF, Grenoble, France

**Olivier.thomas@univ-amu.fr*

Diffraction of X-rays by crystals was first evidenced by M. Laue [1] using a white X-ray beam and a photographic film. This white beam diffraction setup now bears the name of its first inventor. Soon after Laue's discovery it was realized that Laue diagrams from deformed crystals show distinct elongated features called asterism [2]. Hence the sensitivity of X-ray diffraction to crystal imperfections was realized even before Taylor, Polanyi and Orowan introduced the concept of edge dislocation independently in 1934. Elastic strains and related stresses can be deduced with great accuracy from the shift of Bragg peaks [3]. This approach still remains very effective in measuring elastic strains in various materials.

Strain and defect imaging with X-rays has made very impressive progress lately. On one hand progress in X-ray focusing optics allows nowadays scanning x-ray diffraction mapping to be performed with a resolution in the 50-100 nm range [4]. Full field X-ray microscopy is improving a lot too with resolutions in the 100 nm range [5]. By far the best spatial resolution is obtained with coherent diffraction imaging, which is a lensless imaging technique, with a typical resolution of 8-10 nm [6, 7].

These recent progress, which have been made possible thanks to the development of brighter and more coherent synchrotron sources are revolutionizing the field of mechanics at small scales. I will present some recent experiments where *in situ* nano X-ray diffraction (Laue diffraction [7] or coherent diffraction imaging) coupled with mechanical testing has been performed. They allow visualizing crystal defects during the first stages of plastic deformation and give interesting insights into the plastic deformation mechanisms at work at the nano-scale.

Acknowledgments

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New challenges in high-resolution diffraction

Horst Borrmann*

Max Planck-Institute for Chemical Physics of Solids,
Nöthnitzer Straße 40, 01187 Dresden, Germany

**borrmann@cpfs.mpg.de*

Reconstruction of electron densities from diffraction data has always been most demanding on data quality and thus been a main driving force on the methodology. Recent development of X-ray sources introducing new technologies in combination with adequate optics solutions provide for various new opportunities. The almost simultaneous advent of pixeldetektors incorporating Cadmiumtelluride sensors finally paved the way towards general use of harder X-rays associated with a very decisive push of resolution limits. Besides allowing to study in the home lab very demanding systems which up to now where considered accessible at Synchrotron sources only, the much more general availability of very high-resolution data allows for deeper and even unexpected insights in many cases where the ultimate goal of a detailed density analysis is not intended or not achievable. Typical examples include interesting new insights into true structural situations clearly deviating from the typically idealized crystal structures. Such observations frequently turn out very significant in terms of understanding particular properties. Another interesting target is the proper coloring of certain atomic arrangements where neighboring elements are considered extremely hard to distinguish by X-ray diffraction methods. It still needs to be proved on a broader basis, but first results are most encouraging, that high-resolution data in general may rival X-ray resonance or neutron diffraction methods on behalf of this problem. The increasing interest in compounds and properties without a center of symmetry puts the task to reliably assign the absolute structure routinely. As a matter of fact, the established methods are not well suited for most of the materials with high space group and local symmetries associated with only a small number of atoms in the asymmetric unit. High resolution data provide for additional information on this issue due to the favorable angular dependence of anomalous scattering. However, new reliable approaches need to be considered for larger single crystals or multi-domain samples. A combination with micro-structuring techniques offers fascinating new opportunities in many respects.

Scanning X-ray microscopy at MAXYMUS combining chemical, magnetic, depth and temporal sensitivity

Gisela Schütz*[1], Markus Weigand[1], Joachim Gräfe[1], Hermann Stoll[1]

[1]*Max Planck Institute for Intelligent Systems, Heisenbergstr. 3, 70569 Stuttgart, Germany*

**schuetz@is.mpg.de*

Since 2011 the Max-Planck Institute for Intelligent Systems has been operating the soft X-ray microscope MAXYMUS (Magnetic X-Ray Microscopy with UHV Spectroscopy) as a dedicated endstation at the UE46 undulator at BESSY II, Helmholtz-Zentrum Berlin. By soft X-rays nearly any element of the periodic table can be addressed by micro- to nanospectroscopy. In addition to the element selectivity the absorption profiles provide a fingerprint of the chemical bonding. Using circular polarized radiation emitted from a helical undulator on the basis of the x-ray circular magnetic dichroism local magnetic moment can detected in a quantitative manner with extremely high sensitivity. By applying sum rules spin and orbital contributions can be well separated. The orbital moment, which cannot be determined by any other method especially in multi-component systems, plays a key role for fundamental and technologically relevant phenomena as spin-orbit effects and torques in magneto-crystalline anisotropy, exchange bias and topological systems.

At MAXYMUS the large variety of sample environments and detection techniques enabled several advanced new developments for X-ray microscopy: The UHV condition in the microscope allows measuring both absorption contrast in transmission and total electron yield. Due to this the unique possibility both bulk and surface information of the sample at the same time. While in typical operation the spatial resolution is limited by the Fresnel Zone Plate (about 18 nm), the recent implementation of a fast x-ray CCD camera and ptychographic imaging allows to resolve even sub-10nm structures.

We have implemented and continuously improved a pump-and-probe setup to allow time resolved microscopy. With this the whole BESSY filling-pattern can be used for imaging, with time resolution only limited by its time structure, which allows to study dynamics with about 10 psec time resolution. These techniques can be combined with a vector magnet system and a recently implemented Helium cryostat, which enables sample temperatures down to 20 K and fields up to 0.3 T in- and out-of- the sample plane. The potential of MAXYMUS will be demonstrated by selected examples in material and environmental science and biology and the planned improvements as tomography, laser excitations and high-temperature studies will be discussed.

Nanoscale spectroscopy and tomography with the HZB full-field transmission X-ray microscope

G. Schneider*[1], S. Rehbein[1], S. Werner[1], B. Kepsutlu[1], J. McNally[1], P. Guttmann[1]

[1]*Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Albert-Einstein-Str. 15, 12489 Berlin, Germany*

**gerd.schneider@helmholtz-berlin.de*

In the nano-age, humans manufacture complex structures atom by atom to design e.g. their specific functionality. Therefore, new tools for the analysis of these structures have to be developed. The HZB microscopy group develops novel methods for X-ray imaging to make use out of the unique interactions of X-rays with matter. For this, X-ray optics for the 10-nm scale characterization of the nanostructure, chemical nature, and composition of materials with high energy resolution are engineered and fabricated. The HZB full-field TXM at the BESSY II U41 undulator beamline allows high spectral resolution of $E/\Delta E=5000$, about 10 nm (half-pitch) spatial resolution and field of views in the range of 10-15 μm [1-4]. With this instrument spatially-resolved NEXAFS studies for material sciences can be performed due to the high energy resolution [5]. Additionally, nano-tomography of cryogenic samples had demonstrated its high potential for life sciences [2].

Conventional spectroscopy methods such as photoemission spectroscopy and X-ray absorption spectroscopy have shown to be particularly well-adapted probes to study electronic properties of nanostructures. However, these conventional spectroscopy techniques typically illuminate areas of 50 μm x 50 μm or larger thus preventing the analysis of a single nanostructure. Spectromicroscopy investigations with nanometer resolution were restricted so far to scanning X-ray microscopes (STXM) or to transmission electron microscopes (TEM) equipped with electron energy loss spectroscopy (EELS). Both methods give no statistical information as they are restricted to small image fields. In contrast, the typical image field in NEXAFS spectroscopy measurements combined with full-field transmission X-ray microscopy (NEXAFS-TXM) is about 10 μm x 10 μm which is large compared to the individual nanoparticle. Therefore, one image stack already contains statistically significant data with nanometer resolution.

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Aberration-corrected transmission electron Microscopy of the structure and chemistry of Epitaxial ceria thin films on yttria-stabilised Zirconia substrates

Robert Sinclair*[1], Sang Chul Lee[1,] Yezhou Shi[1, 2] and William C. Chueh[1, 2]

[1]Department of Materials Science and Engineering, Stanford University, Stanford, CA 94305, USA
[2]Stanford Synchrotron Radiation Lightsource, SLAC National Accelerator Laboratory, Menlo Park, CA 94025

**bobsinc@stanford.edu*

We have applied aberration-corrected transmission electron microscopy (TEM) imaging and electron energy loss spectroscopy (EELS) to study the structure and chemistry of epitaxial ceria thin films, grown by pulsed laser deposition onto (001) yttria-stabilised zirconia (YSZ) substrates [1]. There are few observable defects apart from the expected mismatch interfacial dislocations and so the films would be expected to have good potential for applications. Particular attention is paid to the transition from fully to partially coherent interfacial structures, and this is correlated to x-ray diffraction measurements [2]. Under high electron beam dose rate (above about 6,000 $e^-/Å^2s$) domains of an ordered structure appear and these are interpreted as being created by oxygen vacancy ordering. The ordered structure does not appear at lower lose rates (ca. 2,600 $e^-/Å^2s$) and can be removed by imaging under 1 mbar oxygen gas in an environmental TEM. EELS confirms that there is both oxygen deficiency and the associated increase in Ce^{3+} versus Ce^{4+} cations in the ordered domains. *In situ* high resolution TEM recordings show the formation of the ordered domains as well as atomic migration along the ceria thin film (001) surface. The influence of thin film strain, by using different substrates, will also be considered [3].

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Towards atomic resolution magnetic imaging of nanoscale materials in the TEM

Rafal E. Dunin-Borkowski*[1], Amir H. Tavabi [1], András Kovács [1], Zechao Wang [2], Xiaoyan Zhong [2]

[1]Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons and Peter Grünberg Institute, Forschungszentrum Jülich, 52425 Jülich, Germany
[2]National Center for Electron Microscopy in Beijing, Key Laboratory of Advanced Materials, The State Key Laboratory of New Ceramics and Fine Processing, School of Materials Science and Engineering, Tsinghua University, Beijing 100084, P.R. China

** r.dunin-borkowski@fz-juelich.de*

The development of novel magnetic materials for applications in energy-efficient information and communications technologies requires atomic-level characterization of their magnetic moments, in order to be able to predict and control their physical properties and to obtain a fundamental understanding of the interplay between charge, spin, orbital and lattice degrees of freedom. Techniques for spatially resolved magnetic imaging such as spin polarized scanning tunneling microscopy and magnetic exchange force microscopy can be used to map surface spin structures with atomic spatial resolution, but it is very difficult to use them to obtain magnetic information about bulk or buried materials. X-ray magnetic circular dichroism combined with photoelectron emission microscopy is element-specific, but its spatial resolution does not approach the atomic scale and it falls short of the ability to simultaneously image the local microstructure of the same region of the sample.

In the most recent generation of transmission electron microscopes, chromatic aberration (C_C) correction promises to provide improved spatial resolution and interpretability in recorded images when compared with the use of spherical aberration (C_S) correction correction alone, as a result of the improved temporal damping envelope of the objective lens, especially at lower accelerating voltages. The reduced dependence of image resolution on energy spread in a C_C corrected microscope offers benefits for conventional bright-field and dark-field imaging as a result of the decreased influence of inelastic scattering on spatial resolution, even when using zero-loss energy filtering. For energy-filtered TEM, C_C correction allows large energy windows and large objective aperture sizes to be used without compromising the spatial resolution of energy-loss images.

Here, we assess whether combined C_C and C_S aberration correction of the Lorentz lens of the Titan PICO transmission electron microscope (TEM) in Forschungszentrum Jülich can be used to record atomically resolved real space information about magnetic microstructure in nanoscale materials with the conventional microscope objective lens switched off. We will present an experimental and theoretical assessment of whether C_S and C_C corrected off-axis electron holography can be used to detect in-plane magnetic fields with close-to-atomic spatial resolution and assess the factors that presently limit the spatial resolution of the microscope in Lorentz mode. We will also discuss the use of C_S and C_C aberration correction to perform electron magnetic circular dichroism combined with spatially resolved electron energy-loss spectroscopy, in order to detect out-of-plane magnetization components in materials from individual atomic planes under parallel beam illumination conditions.

Acknowledgments

We are grateful to Juri Barthel, Lei Jin and Jan Rusz for valuable contributions to this work. The research leading to these results has received funding from the European Research Council under the European Union's Seventh Framework Programme (FP7/2007-2013)/ ERC grant agreement number 320832.

Perspectives on combined nanosecond imaing and in-situ chemical analysis with EELS using high-speed electron microscopy approaches

Thomas LaGrange*[1], Kerstin Bücker[2], Matthieu Picher[2], Florian Banhart[2]

[1] *Interdisciplinary Centre for Electron Microscopy (CIME), École Polytechnique Fédérale de Lausanne (EPFL), Lausanne, CH-1015 Switzerland*
[2] *Institut de Physique et Chimie des Matériaux (IPCMS), Université de Strasbourg, 67034 Strasbourg, France*

**thomas.lagrange@epfl.ch:*

Recent developments in time-resolved transmission electron microscopy instrumentation have been motivated by the interest to observe rapid materials dynamics on their relevant length and time scales that are inaccessible with the slow acquisition rates of conventional methods. There are two broad classes of techniques, stroboscopic and single-shot approaches [1-3]. The stroboscopic technique builds-up an image or spectrum by integrating millions of pump-probe events, and investigations by this approach are limited to materials systems that are highly repeatable and completely reset to their initial states between pump-probe events [1, 3]. The single-shot approach can explore irreversible events in materials by taking "snap-shot" images using electron pulses having a bunch length of 5ns to 100s of nanoseconds and containing billions of electrons [1]. With such high peak currents (1-100mA), Boersch effects reduce temporal coherence of the electron beam and limit resolution [2]. With a modified thermionic electron gun, the Wehnelt electrode can be used to the monochromatic beam by filtering out electrons with large energy spreads, reducing chromatic aberrations and improving spatial resolution [3]. Filtering the pulsed electron beam greatly reduces signal, but in optimized setups with high brightness photoemission sources, sufficient numbers of electrons illuminate the camera. In such configurations, spatial resolution is ultimately limited by the electron trajectory changes in the crossovers of the objective lens.

In high current pulses, the Boersch effects near the cathodes can produce energy spreads >500 eV, making spectroscopy with the single-shot approach challenging [2]. Nanometer spatial resolution in the single shot techniques is achieved by reducing energy spreads to <25 eV, though meaningful EELS measurements at those energy spreads are not possible. However, by optimizing the electron optical configuration and using the Wehnelt combined with fixed apertures in the acceleration as a crude monochromator, the energy spread can be greatly reduced while maintaining enough signal to perform core loss EELS, e.g., we have measured the carbon K-edge (See Figure 1). Given these preliminary results and with further optimization of the cathode geometry, it may be possible to improve signal without increasing the energy spread. In this presentation, a synopsis of single-shot imaging and spectroscopy will be given that will conclude with a perspective on future developments to advance these techniques.

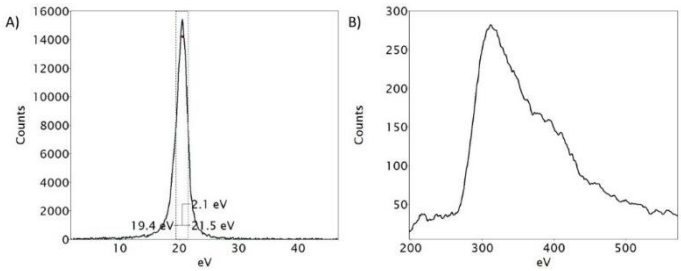

Figure 1. A) Zero loss peak showing an FWHM of ~2 eV measured at the microscope setting optimized for single-shot EELS using 7 ns electron pulses, B) single-shot EELS measurement of the carbon K-edge

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Accessing reaction kinetics via direct imaging of atoms – example of graphene

A.Chuvilin*

CIC nanoGUNE Consolider, Tolosa Hiribidea 76, 20018 Donostia-San Sebastian, Spain
IKERBASQUE Basque Foundation for Science, Maria Diaz de Haro 3, E-48013 Bilbao, Spain

**a.chuvilin@nanogune.eu*

The raise of nanoscience and nanotechnology and necessity to characterize the structure of individual objects consisting of a countable number of atoms determined the shift of structure characterization paradigm from bulk methods like X-ray diffractometry to local high resolution methods like electron microscopy. The similar shift in paradigm is urging now in chemistry – chemical processes defining structure and properties of nanoscale and low dimensional objects often constitute a negligible part of the total volume of the material, and thus their assessment by experimental bulk chemical methods if often impossible. The new concept is provided by the time resolved electron microscopy allowing for direct observation of atomic rearrangements.

We are developing the methodology to apply the formalism and approaches of the classical chemical kinetics for the quantitative description of atomistic processes observable in the microscope. We show that a proper statistical treatment of the data obtained in a range of experimental conditions allows determining the threshold energies for radiation induced reactions. But not only that: we show that true activation energies for thermally activated reaction pathways for individual defects can be estimated as well.

We apply this methodology for reactions of point defects in graphene. The cross-sections and threshold energies of irreversible (atom emission) and reversible (bond rotation, see Fig.1) processes are measured. Observation of statistically significant number of events at variable experimental conditions allows decoupling of radiation induced and thermal reaction pathways and obtaining independent estimations of cross-sections and activation energies for direct and backwards rotations. The cross-sections of direct rotation were found to be in a decent agreement with theoretical estimations. Interestingly the backwards rotation is characterized by very high cross-section exceeding theoretical values by 3-4 orders of magnitude. The values obtained rule out electron-nucleus collision as the main mechanism of energy transfer from electron beam to the sample. We speculate that the energy is transferred through electron-electron interactions via strong coupling of excited electron states with the phonon modes localized around the defect.

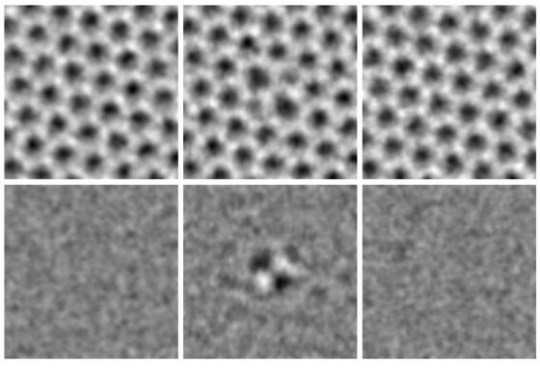

Figure 1. Sequence of electron microscopy images showing the bond rotation reaction in graphene. The upper row – unprocessed images of the graphene lattice separated by time slices of 1s. The lower row – the same images filtered in order to remove the pattern of the lattice.

Acknowledgments

The contribution of my co-workers from Nottingham University, Vienna University, KAUST, Novosibirsk Institute of Inorganic Chemistry and Graphenea Company is greatly appreciated. The work was partially supported by collaboration project with FEI Company and by FP7-PEOPLE-2011-IRSES N295180 (MagNonMag) project.

Investigation of thermal and electrical structure parameters using combined scanning probe microscopy methods

Teodor Gotszalk* [1], Maciej Rudek[1], Paweł Janus[2], Andrzej Sierakowski[2], Berndt Koehler[3], Martin Kuettner[3]

[1]*Wroclaw University of Science and Technology, Faculty of Microsystem Electronics and Photonics, ul. Janiszewskiego 11/17, 50-370 Wrocław, Poland*
[2]*Institute of Electron Technology, Aleja Lotników 32/46, 02-668 Warszawa, Poland*
[3]*Fraunhofer-Institut für Keramische Technologien und Systeme IKTS, Maria-Reiche-Str. 2, 01109 Dresden, Germany*

** teodor.gotszalk@pwr.edu.pl*

Scanning probe microscopy belongs to the technologies, which enable precise and high resolution surface investigations. In this technology interactions between the nanoprobe and the sample are recorded in order to describe various specimen parameters. The SPM methods have been successfully applied in the surface diagnostics but theirs usage in the investigations and especially metrology of technological samples is still limited. In this work we present architecture and application of a SPM system integrating so called piezoresistive cantilevers for measurements of electrical and thermal surface properties. The proposed technology can be easily adopted for the investigations performed with scanning electron microscopy (SEM) machines, in which observations of the interesting details can be defined with high resolution. In this work we will show the results obtained using described system of temperature and electrical properties of biased silicon nanowires. The basic features of the proposed technology will be presented as well.

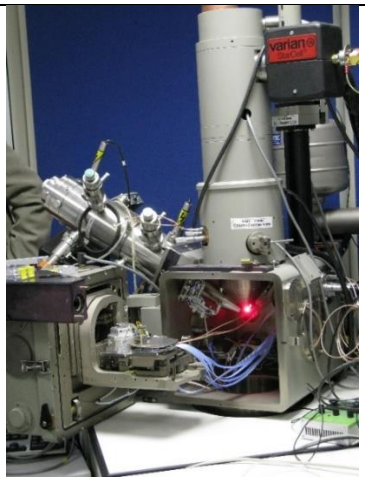

Fig. 1. SPM system utilizing piezoresistive cantilevers integrated with SEM column

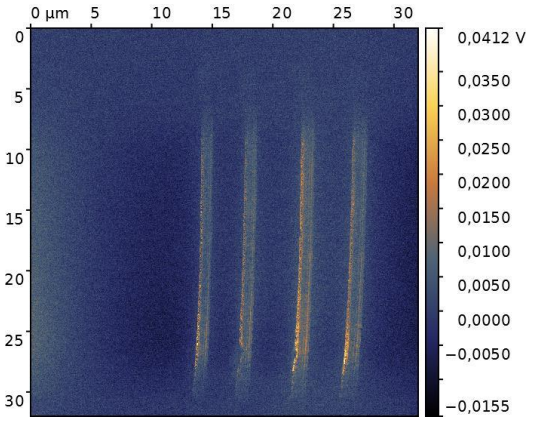

Fig. 2. Distribution of thermal resistance between the microprobe and silicon nanowire (tip temperature 320K, sample temperature 300K)

Acknowledgments

The described research was supported by the Wroclaw University of Science and Technology (WRUST) and statuory grant and FP7 STREP NanoHeat project.

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In situ mechanical testing and manipulation of nanomaterials in FIB-SEM and TEM: New approaches and applications

Erdmann Spiecker

Lehrstuhl für Mikro- und Nanostrukturforschung & Center for Nanoanalysis and Electron Microscopy (CENEM), Friedrich-Alexander Universität Erlangen-Nürnberg, Cauerstr. 6, 91058 Erlangen, Germany

Modern electron microscopes provide an ideal platform for in situ mechanical testing and manipulation of materials at small length scales owing to high flexibility in realizing special sample geometries and loading scenarios without compromising microscope performance. This holds true for both, combined focused ion beam scanning electron microscopes (FIB-SEM) as well as transmission electron microscopes (TEM) with the former providing highest flexibility and the latter highest resolution. In this contribution we report on new approaches of in situ mechanical testing and manipulation in FIB-SEM and TEM that have been developed and applied to various materials systems within Erlangen's DFG Research Training Group GRK1896 “In situ Microscopy with Electrons, X-rays and Scanning Probes”. Examples include recent studies of sliding friction in layered crystals, investigation of the mechanical failure of Ag nanowire networks in flexible transparent electrodes as well as combined mechanical testing and 3D characterization of nanoporous metals. For scale-bridging 3D characterization electron tomography is complemented by high-resolution X-ray tomography.

Abstracts
-Posters-

Combining analytical electron microscopy with cryo-TEM

Petr Formanek*[1], Uta Reuter[1]

[1]*Leibniz-Institut für Polymerforschung Dresden e.V.,*
Institute of Physical Chemistry and Polymer Physics, Hohe Str. 6, 01069 Dresden, Germany

** formanek@ipfdd.de*

Energy filtered transmission electron microscopy (EFTEM) of radiation-stable specimens on the one hand and cryogenic TEM of vitrified ice specimens on the other hand are well established techniques. Combining both of them seems to be impossible due to contradictory requirements: low-dose imaging for cryo-TEM and high dose necessary for core-loss EFTEM. However there are cases where combining EFTEM and cryo-TEM is crucial for analysis of material morphology. Here we present the application of EFTEM for specimens containing ice on two examples.

In studies of the impact of ice crystals on glass transition and mechanical behavior of soft cross-linked elastomers [1], the size and distribution of ice crystals acting as a reinforcement filler were imaged. EFTEM mapping of carbon and oxygen allowed undoubted distinction between elastomer matrix and ice crystal fillers (Fig. 1).

Cryo-TEM often suffers from contamination by condensed air humidity. The condensed ice might be difficult to distinguish from e.g. polymersomes [2] or micelles. In such cases EFTEM is a way to confirm the presence of carbon and thus to identify areas containing polymer (Fig. 2).

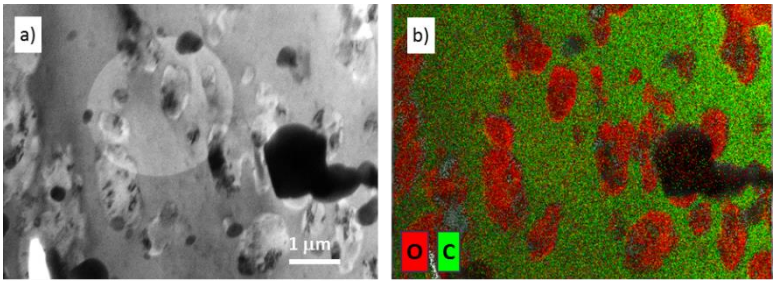

Figure 1. (a) Cryo-TEM image of water-swollen GECO-ice composite (GECO = epichlorohydrin−ethylene oxide−allyl glycidyl ether), (b) carbon (green) and oxygen (red) map overlaid on (a).

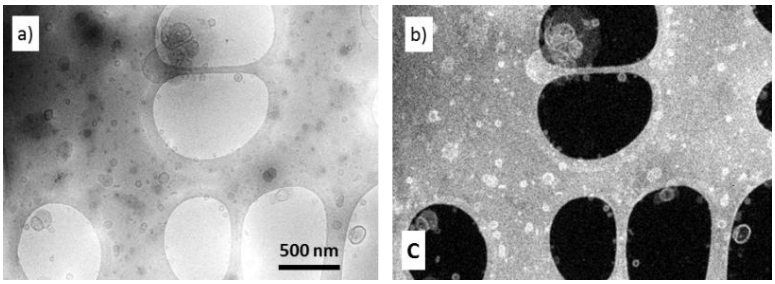

Figure 2. (a) Cryo-TEM image of poly(ethylene glycol)-block-poly[2-(diethylamino) ethyl methacrylate-stat-2-hydroxy-4-(methacryloyloxy) benzophenone] polymersomes in water, (b) carbon map of the area displayed in (a).

Acknowledgments

We acknowledge Dr. Klaus Werner Stöckelhuber and Dr. Dietmar Appelhans from Leibniz-Institut für Polymerforschung Dresden e.V. for providing the specimens.

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Pore topology investigation of organosilicate glass by positron annihilation and positronium migration modeling

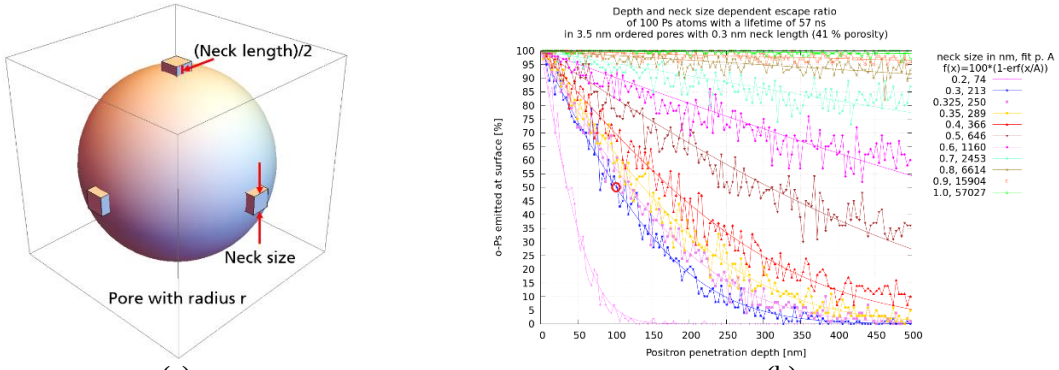
P02

Matthias Kraatz* [1], Christoph Sander[1], André Clausner[1], Maciej Oskar Liedke[2], Maik Butterling[2], Andreas Wagner[2], Wolfgang Anwand[2], Martin Gall[1], Reinhard Krause-Rehberg[3], Ehrenfried Zschech[1]

[1]*Fraunhofer IKTS, Maria-Reiche-Str. 2, 01109 Dresden, Germany*
[2]*Institute of Radiation Physics, Helmholtz-Zentrum, Dresden-Rossendorf, Dresden, Germany*
[3]*Department of Physics, Martin Luther University Halle-Wittenberg, Halle, Germany*

**matthias.kraatz@ikts.fraunhofer.de*

The pore topology of self-assembled organosilicate glass (OSG) was investigated. For the specific case of a dielectric constant k = 2 (porosity ~40 %, [1]), the pore diameter was determined to be 3.5 nm by positron annihilation lifetime spectroscopy (PALS). Further, an ortho-positronium (o-Ps) emission ratio was determined to be ~50 % for a positron energy of 2 keV, which corresponds to a mean penetration depth of 100 nm. The emission ratio equals the 3γ/2γ ratio of the three-ray decay of o-Ps emitted at the surface and the two-ray decay of para-positronium (p-Ps) in the bulk, respectively. Since the neck length of the pore interconnection is defined by porosity and pore diameter when spherical pores with a configuration number 6 are assumed (Fig. 1a), the only remaining topology parameter is the neck size, meaning the width of the interconnection. With positronium migration modeling, the neck size can be determined by adjusting it appropriately in such a way that 50 % of Ps atoms escape to the vacuum from a 100 nm penetration depth, according to the fact that more Ps atoms will escape when the neck size increases. 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 3.5 nm pores. If the bouncing path leads to the surface in that time, the Ps atom was counted as escaped. Fig. 1b shows the simulation results for different neck sizes. It turns out that the appropriate neck size is 0.3 nm. This neck size roughly compares to a missing atom in the glass matrix, such that the conclusion for the topology of the OSG is that it is comprised of 3.5 nm pores, connected by open volume in the glass matrix.



(a) (b)

Figure 2. (a) Pore model. (b) o-Ps emission at surface depending on positron penetration depth and neck size. The marked data area corresponds to 50 % emission from 100 nm depth, and a neck size of 0.3 nm.

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Experimental study of the micro bumps in HBM stacks

P03

Kristina Kutukova*, Jürgen Gluch, Rüdiger Rosenkranz, Ehrenfried Zschech

Fraunhofer Institute for Ceramic Technologies and Systems, Maria-Reiche-Str. 2, 01109 Dresden, Germany

**kristina.kutukova@ikts.fraunhofer.de*

In advanced microelectronic industry there is the demand to increase the performance of integrated systems. One solution are technologies with higher packaging density like multichip stacking based on the 3D through-silicon-via (TSV) and micro solder bumps integration [1]. In stacked products, for example in high- bandwidth memories (HBM), a critical issue is to control the quality of the micro bumps. Soldering process is complex and could introduce physical and chemical changes of the micro bumps, like shape deviation, formation of brittle intermetallic phases in the solder (e.g. SnCu, SnAg) and defects. Micro-voids and micro-cracks could lead to the failure [2, 3]. In this study we characterized critical parameters of the micro solder bumps at HBM stack nondestructively by X-ray computed tomography (nano-XCT). As complementary techniques, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) were applied.

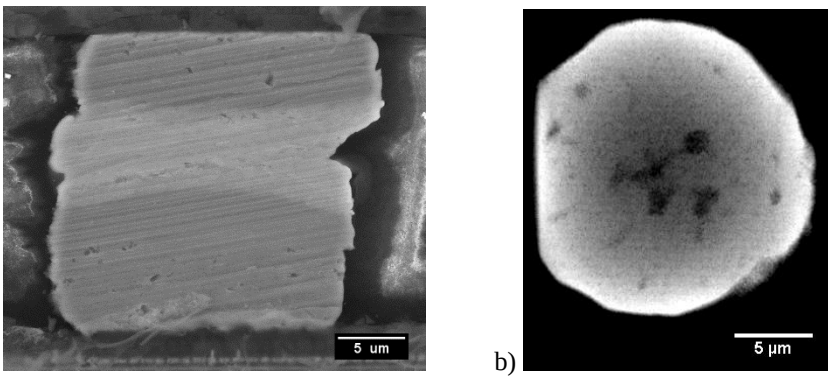

Figure 3 a) SEM cross-section image of the micro solder bump between two dies in the HBM stack represents the geometrical shape deformation; b) Virtual cross-section image based on nano-XCT represents the void distribution in the micro bump volume.

Acknowledgments

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Nanoanalysis of the nitride layers formed on an austenitic stainless steel with different grain structures

P04

Agnieszka Krawczyńska*[1], Ryszard Sitek[1], Malgorzata Lewandowska* [1]

[1]*Warsaw University of Technology, Faculty of Materials Science and Engineering, Woloska 141, 02-507 Warsaw, Poland*

* *malew@inmat.pw.edu.pl*

Nitriding is a well-known surface modification technique to enhance wear resistance of metallic materials. The formation of a nitrided layer is well described for conventional microcrystalline materials. However, with the development of their ultrafine-grained counterparts, the question arises how the microstructure influences the nitriding process. To answer this question, a series of experiments was designed to compare the formation of nitrided layers in an austenitic stainless steel. To refine the microstructure, hot (at 1000°C) and room temperature (RT) hydrostatic extrusion (HE) was applied to austenitic stainless steel 316LVM with a total true strain of 1.4. Subsequently, samples were nitrided using low-temperature plasma-assisted nitriding at 430°C for 5h. Annealed samples with coarse grains of 30 μm in diameter were used as reference samples.

The depth profiles of nitrogen concentration in all samples was measured using Scanning Auger Microprobe-Microlab 350. The results show that although the substrates differed substantially in the microstructure, the thickness of nitrided layers is similar in all of them and varies between 7 and 8 μm. However, the profiles differed considerably in shape. In the case of the HOT_HE_1.4 sample the high nitrogen concentration up to 20 at. % was maintained up to nearly 6μm. In the annealed and HE_1.4 samples, the nitrogen concentration decreased almost linearly with depth.

XRD analysis showed the S-phase formed during nitriding in all samples. Detailed TEM analysis revealed that nitriding process induced not only enrichment in nitrogen but also such defects as microtwins and free dislocations especially in the annealed and HOT_HE_1.4 samples. These defects affected the formation of nitrided layer and are believed to be at least partly responsible for a lack of difference in the layer thickness in the investigated samples.

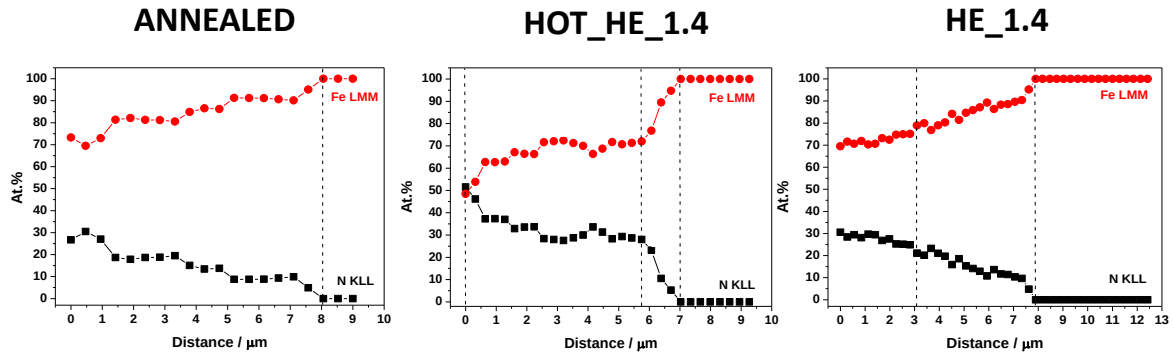

Figure 1. Chemical depth profiles of S-phase layers formed in annealed, HOT_HE_1.4 and HE_1.4 samples.

Acknowledgments

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In-situ TEM studies on nanomaterials for electronics applications

P05

Zhongquan Liao* [1], Martin Gall[1], Uwe Mühle[1], Jürgen Gluch[1], Yvonne Standke[1], Gianaurelio Cuniberti[2], and Ehrenfried Zschech[1]

[1]*Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), 01109 Dresden, Germany*
[2] *Institute for Materials Science, Technische Universität Dresden, 01069 Dresden, Germany*

** Zhongquan.liao@ikts.fraunhofer.de*

In-situ transmission electron microscopy (TEM) is a technique where a TEM tool is used to monitor a sample in real time. It provides kinetic information on a material under certain stimuli. The fast development in the microscopy instrumentation advances in-situ TEM into a fast-growing, increasing important and fascinating technique in various fields ranging from materials science to chemistry, biology, etc. [1]

In our study, several in-situ experimental methodologies in the TEM were developed to investigate several nanomaterials for electronics applications. An in-situ approach was carried out to study the time-dependent dielectric breakdown (TDDB) failure mechanisms and degradation kinetics in on-chip interconnect stacks. An in-situ mechanical testing system was utilized to stretch the patterned graphene nanoribbons (GNRs), aiming to open a bandgap by strain engineering. In-situ strain measurements of silicon close to through silicon via (TSV) in the 3D chip was performed at elevated temperatures. All experiments were accomplished inside the TEM, which provide a unique perspective for fundamental understanding of materials' response under certain stimuli.

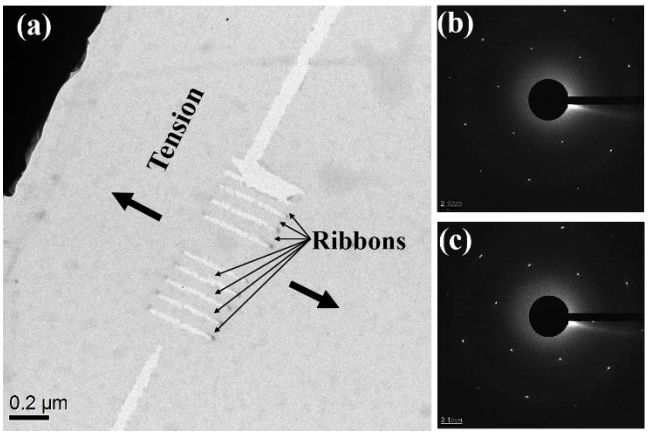

Figure 1. Bright field (BF) TEM image of the patterned GNRs for in-situ mechanical test, (b) selected area electron diffraction (SAED) pattern from the graphene membrane next to the GNRs, (b) SAED pattern from the GNRs.

Acknowledgments

We would like to thank Christoph Sander, Rüdiger Rosenkranz, André Clausner and Matthias Kraatz (Fraunhofer IKTS); Oliver Aubel, Kong Boon Yeap and Norman Vogel (GlOBALFOUNDRIES); Tao Zhang and Arezoo Dianant (TU Dresden); Douglas Stauffer and Oden Warren (Hysitron, now Bruker) for their scientific support and technical assistance.

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Reconstructing thermo-mechanical loads of machining

P06

B. Medina-Clavijo* [1, 2], Pedro. J. Arrazola[2], Andrey Chuvilin[1, 3]

[1]*Faculty of Engineering, Mondragon Unibertsitatea, 20500 Arrasate, Spain*
[2]*Electron-Microscopy Laboratory, CIC nanoGUNE Consolider, 20018 Donostia, Spain*
[3]*Ikerbasque, 48013 Bilbao, Spain*

**b.medina@nanogune.eu*

Understanding the phenomena occurring in the tool-chip interaction is a key to improving the machining process, one of the most used industrial manufacturing techniques. In that process, though the chip is wasted during machining, it storages the stark signatures of the conditions which were present directly during cutting, especially in secondary shear zone (SSZ), which has been in the direct contact to the tool. In this study, samples of AISI 1045 steel were machined and chips subsequently analyzed by electron and ion microscopy. Microstructure pointed to an anisotropic distribution of features along the chip, with special emphasis in the SSZ part. There, ion scans showed that grains tend to extreme refinement and equiaxed shape. In this area, the resulting material is characterized by grains of 70 – 200 nm and cementite partially dissolved into the matrix (figure 1). While this particular area is affected by prominent stress and strain, EBSD data post-processing remarks that material develop certain relaxation. These are evidences for a sort of ultra-fast dynamic recrystallization (DRX) process [1]. While recrystallization depend of atomic diffusion, this process would be extremely accelerated by reduced grain/subgrain size, what minimize the volume of atoms to diffuse and subsequently increase the kinetics of recrystallization. In addition, the area affected by DRX was analyzed mechanically by nanoindentation. The results pointed to a slightly reduction of elastic modulus. This could be explained by a relative large volume of grain boundaries [1] and defects associated with short-term re-solution of carbon (from cementite) into the matrix.

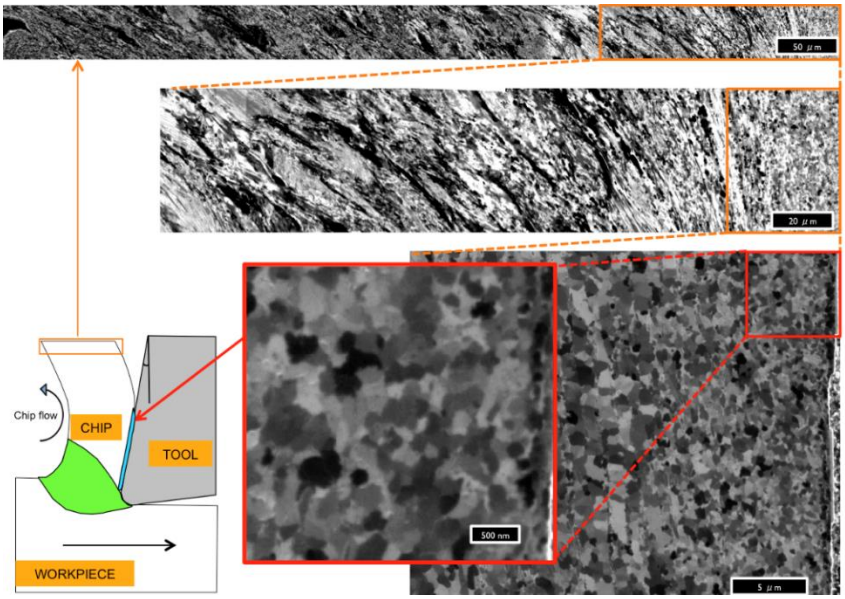

Figure 4. Ion scans of the whole section of machined chip, and successive magnifications on the SSZ are, showing localized equiaxed grains and partially dissolved carbides.

Acknowledgments

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Investigation of a behavior of molten salts in MCFC elements by visualization of kinetic processes with X–ray imaging techniques

P07

Jarosław Milewski* [1], Jürgen Gluch[2], Tomasz Wejrzanowski[1], Ehrenfried Zschech[2]

[1]*Institute of Heat Engineering, Faculty of Power and Aeronautical Engineering, Warsaw Universi ty of Technology, 21/25 Nowowiejska Street, 00-665 Warsaw, Poland*
[2]*Fraunhofer Institute for Ceramic Technologies and Systems (IKTS), 01109 Dresden, Germany*
[3]*Faculty of Material Science Engineering, Warsaw University of Technology, 141 Wołowska Stre et, 02-507 Warsaw, Poland*

**Milewski@itc.pw.edu.pl*

In the past 10 years, there have been studies focusing on the ceramic part of such dual–phase membranes. The influence of the material, microstructure and phase ratio on the flux of the dual–phase membranes was investigated for membranes with alumina, yttria-stabilized zirconia [8], Gd–doped ceria [8, 2], Sm-doped ceria [2], Bi1.5Y0.3Sm0.2O3 [4, 3], or lanthanum-strontium-cobalt-iron oxide frameworks [1]. The effect of pressure and oxygen content in the flue gas on the surface reaction and bulk conductivity have been modeled and studied experimentally [6, 5, 7]. Despite the extensive investigation of these composite membranes, the fundamental transport properties of molten carbonates are not yet fully understood. The aim of this paper is to use the available unique X–ray imaging techniques, X-ray micro-CT and nano-XCT to visualize the 3-D pore structure and morphology of molten salts in a ceramic matrix. The statistic of the pore properties, like pore connectivity, diameter distribution and branch distribution have to be determined and extracted from the CT data in order to understand the main behavior of the salts during penetration of the matrix. Here the dimensions of a miniaturized and the environment supporting the cell (temperature management and gas medium supply etc.) are taken in consideration.

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Precipitation Behavior of Trace Elements in Aluminum

P08

U. Mühle* [1, 2], T. Schubert[3], T. Staab[4], R. Krause-Rehberg[5], B. Kieback[1, 3]

[1]TU Dresden; Institute of Materials Science; Helmholtzstr. 7; D-01069 Dresden
[2]Fraunhofer Inst. for Ceramic Technologies and Systems; M.-Reiche-Str. 2;D-01109 Dresden
[3]Fraunhofer Inst. for Manufact. Techn. and Adv. Mat.; Winterbergstr. 28; D-01277 Dresden
[4]TU Würzburg; Faculty f. Chemistry and Pharmacy; Sanderring 2; D-97070 Würzburg
[5]Martin-Luther-Univ. Halle; Faculty of Nat. Sci. II; von-Danckelmann-Pl. 3 D-06120 Halle

**uwe.muehle@tu-dresden.de*

The main mechanism for the strengthening of aluminum-copper alloys is hardening by the precipitation of copper-rich phases. However, their size, distribution and crystal structure determine the final mechanical properties of the material. It is shown that alloying additionally small amounts of tin influences the precipitation behavior as well as the final strength of Al-Cu-alloys [1]. The binding energy for quenched-in vacancies to trace elements [2] in the aluminum matrix is recognized as an influence on the preferred type of precipitate changes.

A precondition for this influence is the transition of trace element into solid solution. Following thermodynamic calculations a temperature treatment at 520 °C enables less than 0.01 at% of Sn or In to go into solution. For higher concentrations of these elements a temperature above 600 °C is necessary for going fully into solution, which is confirmed by digital scanning calorimetry (DSC).

Considering a Al-0.25 at% Sn alloy, a subsequent ageing treatment at 150°C between 1h and 24h tin precipitates as incoherent particles of about 15 nm size (Fig. 1). This reaction occurs in a homogenous distribution. Due to the well-defined contrast between Al and Sn a quantitative assessment is possible and leads to a nearest neighbor distance distribution with a maximum at about 120 nm.

An equivalent treatment for an Al-0.25 at% In alloy leads to coherent particles with a size between 2 and 4 nm with internal structure, where the indium atoms are arranged parallel to (111) lattice planes in the fcc-Al (Fig. 2). This leads to a strain field around the particle.

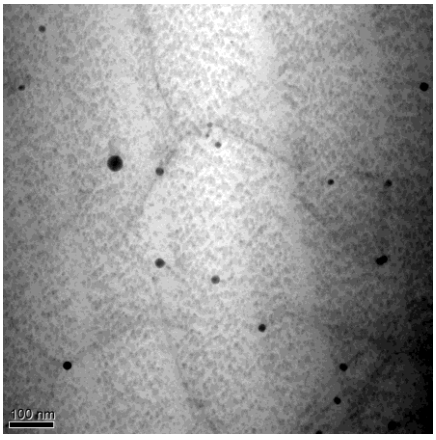

Figure 1. Incoherent precipitations in an Al-0.025at% Sn alloy after solution treatment and ageing for 24h at 150 °C

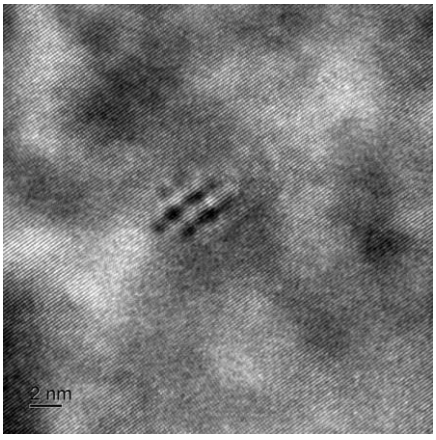

Figure 2. Coherent precipitations in an Al-0.025at% Sn alloy after solution treatment and ageing for 1 h at 150 °C

Acknowledgments

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Traceability of internal length scale in PillarHall thin film conformality test chips

P09

Virpi Korpelainen[1], Feng Gao[1], Mari Laamanen[1], Oili M. E. Ylivaara[1], Jonas Sundqvist[2], Mikko Utriainen[1], R. L. Puurunen[1]

[1] VTT Technical Research Centre of Finland
[2] Fraunhofer IKTS, Germany

The downscaling of future semiconductor devices with increasing 3D character leads to increasing demand of highly conformal thin films. Atomic layer deposition (ALD), based on the use of repeated, self-terminating reactions of typically at least two compatible reactants on a solid substrate, is often the only technique that can meet the conformality specifications. Conformal films made by ALD are also needed in other fields with intrinsic three-dimensionality requirements, such as microelectromechanical systems (MEMS), energy applications, and high-surface-area catalysts.

This work continues on earlier work on conformality analysis [1–3]. All-silicon microscopic lateral high-aspect-ratio (LHAR) structure prototypes have been designed and fabricated with an improved design (PillarHall Prototype 3B). The LHAR structures consist of a lateral gap of typically 500 nm (in some cases, 100 to 2000 nm) in height under a polysilicon silicon membrane, supported by silicon pillars. The gap length varies from 1 to 5000 µm, giving aspect ratios (length vs height) for the typical ~500 nm gap of 2:1 to 10 000:1. In some PillarHall chips, unique distance indicator lines have been etched directly onto the silicon membrane to provide an accurate internal length scale. Each distance indicator (every 100 µm) is unique, providing a means to detect length even when the LHAR structure opening is not visible. Applications are predicted both in non-destructive top-view microscopy analysis and cross-sectional scanning electron microscopy (SEM).

In this work, we analyse the accuracy of the distance indicator lines. Lines are measured using QuickVision optical coordinate measuring machine. The lines near the opening are also measured using atomic force microscope (AFM). The combination of two methods allows lower uncertainty for the positions of the first lines and reduces uncertainty caused by the detection the zero point. i.e. the edge of the membrane. According to preliminary results, standard uncertainty of the positions of the indicator lines is ~0.3 µm. The main uncertainty component comes from detection of the lines in optical measurement. Uncertainty for the AFM measurement is smaller.
VTT MIKES is a national metrology institute (NMI) in Finland, thus the measurements have in-house traceability to the definition of the metre.

Acknowledgements

The authors are grateful to Tuomo Honkaharju for clean room processing, Harri Pohjonen for mask drawing, James Dekker for dry etch specialist advice, Jaakko Saarilahti for valuable hints with process integration, Ville Byman for helping in the measurements and Björn Hemming for discussions about the measurement uncertainty. This work has been partly funded by the Tekes PillarHall project and is linked to the Finnish Centre of Excellence on Atomic Layer Deposition.

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Correlation between nanoscale morphology and the performance of Organic Solar Cell

P10

Mona Sedighi*

Dresden Center for Nano analysis, Technische Universität Dresden, Germany

**mona.sedighi@tu-dresden.de*

One key advantage of organic solar cells is the low weight and the high flexibility of the modules, together with significantly lower manufacturing costs, also due to the possibility of continuous manufacturing processes. The record power conversion efficiencies of organic solar cells were steadily increasing and recently hit the >13% margin [1].

To further push the power conversion efficiency, the role of morphology of the active layer seems to be most vital. Fundamental understanding of the relation between the nanostructure of the active layer and the device efficiency is of paramount importance for further development of efficient organic solar cells. On the one hand, strong intermixing of donor and acceptor molecules is beneficial for efficient dissociation of photogenerated excitons, but too intimate mixing can give rise to strong losses by enhanced recombination of excitons and poor charge-carrier transport [2].

In this work, we visualized the nano-scale morphology and domain sizes in the active layer of small molecule organic solar cells, where the donor phase is ZnPc ($ZnC_{32}H_{18}N_8$) and the acceptor phase is C_{60}, as it is shown in figure 1. Obtaining insights into the morphology of the active layer requires the spatial resolution and a contrast mechanism to discriminate two phases with similar average atomic number. There are only few characterization methods that can be applied to visualize material phases on this length scale (10-20 nm). In order to circumvent this challenge, we use an analytical electron microscopy method to generate contrast between the different material phases in the investigated solar cells.

We combine plan-view and cross-section electron microscopy imaging with different analytical techniques such as energy dispersive X-ray spectroscopy (EDX) in transmission electron microscope (TEM) as well as Energy selective Backscattered (EsB) imaging in scanning electron microscope (SEM) [3].

To analyze the absorber layer morphology of the most efficient ZnPc:C_{60} solar cell, which was created by high substrate temperatures during material deposition, we utilized a STEM equipped with a high-angle annular dark field (HAADF) and EDX detector to map the different phases.

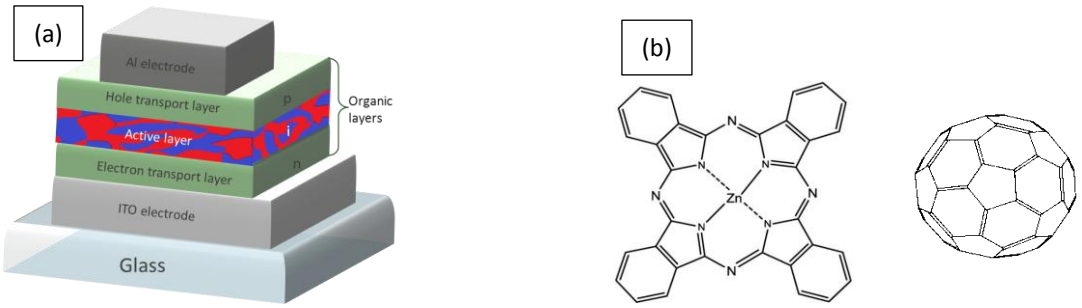

Figure 5. (a) Schematic of the organic solar cell with all electrodes and organic layers. The region of interest is the active layer, which is a blend of Zinc-phthalocyanine (ZnPc) and C_{60}. (b) Chemical structure of ZnPc and C_{60}

Acknowledgments

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An Improved FDK Algorithm for Reconstruction of Misaligned Cone-Beam CT System

P11

Emre Topal*[1], Markus Löffler[1], Ehrenfried Zschech[2]

[1]*Dresden Center for Nanoanalysis, Technische Universität Dresden 01069 Dresden, Germany*
[2]*Fraunhofer-Institut für Keramische Technologien und Systeme 01109 Dresden, Germany*

**emre.topal@tu-dresden.de*

X-ray computed tomography (CT) is a well-known technique for 3D imaging of an object using 2D image data. Commonly, the 2D images, or radiographs are obtained through X-ray imaging taken from different angles around the object. CT has had a huge impact on medical diagnostic and has also provided an efficient method for non-destructive testing in industrial applications. Recently, lab-source CT using cone-beam geometry is getting increasing attention by courtesy of possibility of obtaining a high-resolution 3D reconstruction using a second optical magnification step. In cone-beam CT the measured x-ray data represent 1d line integrals through the 3d object. The object can be reconstructed through 3d inverse Radon transformation from 2d planar integrals, however because of the divergent nature of the cone beam X-rays, the 2d planar integrals cannot be constructed by simply summing up coplanar line integrals as in the parallel beam geometry and hence, advanced mathematics is needed for solving the 3d reconstruction task [1]. The first successful fully three-dimensional algorithm is developed in 1984 and was named after its creators Feldkamp, Davis, and Kress [2]. Ever since, the FDK algorithm continues to dominate the reconstruction task due to its simplicity and computationally less challenging non-iterative nature. The FDK algorithm approximates the line integrals with applying a simple approximate weight to projection value instead of using the exact distances, and for moderate cone-angles, these differences are however small and often acceptable. In addition to this general technical limitation, the standard FDK algorithm requires highly accurate projection geometry of cone-beam CT and it assumes the system is free from any form of misalignment. The key factor on reconstruction process is finding the exact correspondence between the voxels from the object and the pixels from the projection, and when the misalignments exist, the exact correspondence between pixels and voxels cannot be fulfilled and this leads to a deteriorated reconstruction image quality. Based on this point, our in-house written MATLAB (R2015b, Mathworks Inc., Natick, MA) program allows GPU computing and featuring an improved FDK algorithm proposes an efficient method to correct all possible detector related geometrical misalignments and misalignments arise from dynamical motions like wobbling motion, to obtain reconstruction results with excellent accuracy if all the misalignment parameters are known.

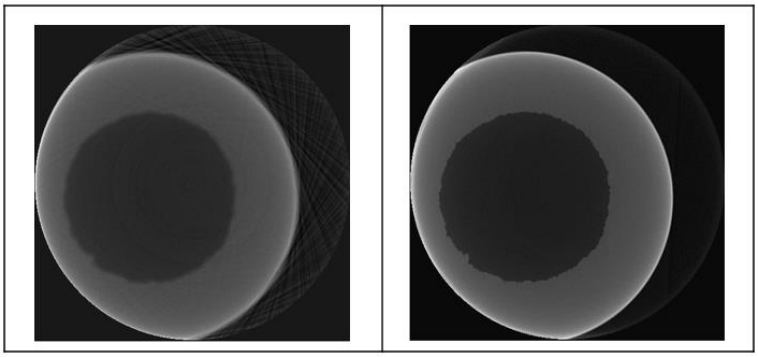

Figure 6. Reconstruction results of calibration phantom data without (left) and with wobbling motion and beam-detector center misalignment correction (right).

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Combined SEM/AFM Nanoprober for Semiconductor Failure Analysis

P12

B. Voigtländer*[1, 2], S. Korte [1, 2], M. Grob [1, 2], R. Spiegelberg [1, 2], V. Cherepanov [1, 2]

[1] *Peter Grünberg Institut (PGI-3), Forschungszentrum Jülich, 52425 Jülich, Germany*
[2] *JARA-Fundamentals of Future Information Technology, 52425 Jülich, Germany*

**b.voigtlaender@fz-juelich.de*

We developed a six-tip combined SEM/AFM nanoprober to contact single transistors at the first contact level. The combination of the two types of nanoprobers in one instrument combines their advantages, while avoiding the disadvantages of the individual instruments. The ultra-compact size of our nanoprober reduces probe drifts to below 0.1 nm/min. This is necessary to create contacts stable enough for electrical measurements on state-of-the-art technology nodes. Fast AFM scanning with non-contact AFM is used to position the tips exactly on the contacts. We will present I/V measurements at single transistors of an SRAM cell.

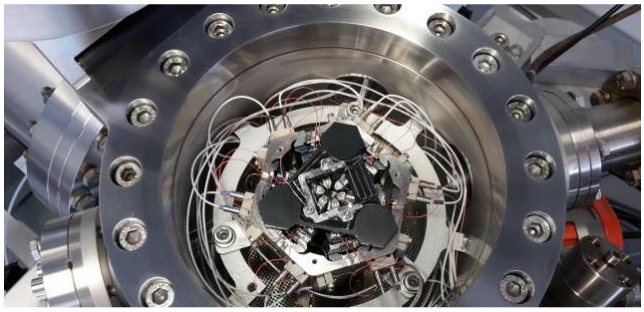

Figure 1. Ultra-compact six-tip AFM/STM/SEM nanoprober

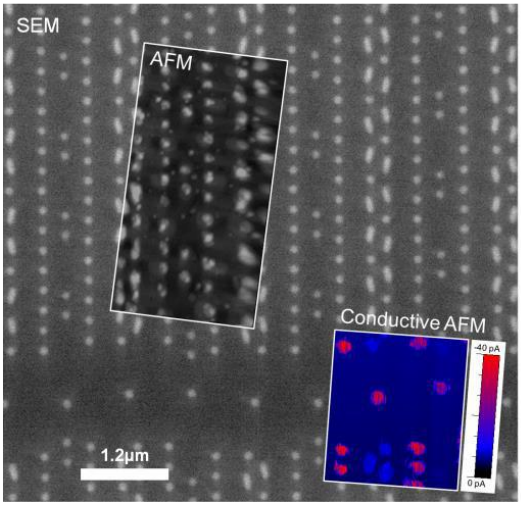

Figure 2. Six transistor SRAM SEM image, upper inset: AFM scan, lower inset: conductive AFM

Advanced materials characterization of strain engineered Germanium by synchrotron-based X-ray diffraction microscopy

P13

M.H. Zoellner*[1], G. Chahine[2], M.-I. Richard[2, 3], P. Zaumseil[1], M. Häberlen[4], G. Capellini[1], F. Montalenti[5], A. Marzegalli[5], P. Storck[4], T.U. Schülli[2], and T. Schroeder[1, 6]

[1] *IHP, Im Technologiepark 25, 15236 Frankfurt (Oder), Germany*
[2] *European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France*
[3] *Aix-Marseille Université, CNRS, IM2NP UMR 7334, 13397 Marseille, France*
[4] *Siltronic AG, Hans-Seidel-Platz 4, 81737 München, Germany*
[5] *Dipartimento di Scienza dei Materiali, Università degli Studi di Milano-Bicocca, Via R. Cozzi 55, 20126 Milano, Italy*
[6] *Brandenburgische Technische Universität Cottbus, Konrad -Zuse-Strasse 1, 03046 Cottbus, Germany*

**zoellner@ihp-microelectronics.com*

SiGe „virtual substrates“ are promising for integrating strain engineered Si, Ge and III/V semiconductors as closed films on a Si(001) platform. Such semiconductor films are of interest in advanced complementary metal-oxide-semiconductors (CMOS) and electronic photonic integrated circuits (EPICs). Although, growth and relaxation processes of SiGe buffer layers on Si(001) are well investigated and major achievements were made, control over the structural homogeneity of SiGe buffers below micro-meter scale is still a challenge for global integration on 300 mm Si(001) wafers.

Modern synchrotrons are at the very heart of fundamental and applied research due to their increased brilliance and the improvement in focusing X-ray optics. A recently developed synchrotron-based scanning x-ray diffraction microscopy technique, called quicK-mapping (K-map), from the beam line ID01 of the European Synchrotron radiation facility (ESRF) is ideally suited to non-destructively image with sub-micron resolution local tilt and lattice constant variations (Δa/a) with a sensitivity down to 10^{-3} degree and 10^{-5}, respectively. Local lattice orientations and constants are extracted by the X-ray Strain Orientation Calculation Software (XSOCS) from 3D reciprocal space maps(q_x, q_y, q_z) at each real space position (x, y).[1] K-map results from SiGe buffer layers and strain engineered Ge layers are demonstrated as examples for the determination of local lattice tilt, strain and composition variation.[2,3]

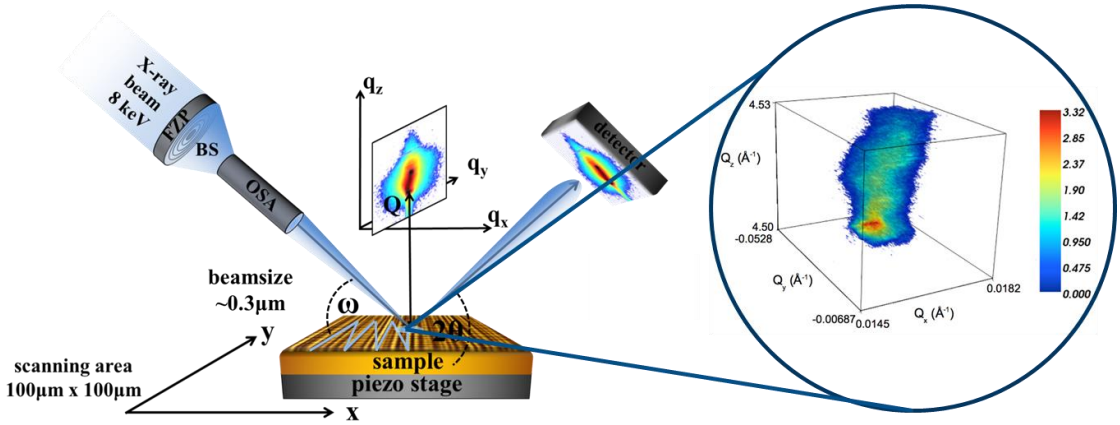

Figure 7. µ-XRD setup with exemplary local reciprocal space map around 004 Bragg reflection of a SiGe buffer layer.

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Characterization of OPV Blends and OSG Thin Films at the Nanoscale with Low Voltage Scanning Electron Microscopy (LVSEM)

P14

Aránzazu Garitagoitia Cid [1, 2], Markus Löffler [2], André Clausner [1], Mona Sedighi [2], Ehrenfried Zschech [1, 2]

[1] *Fraunhofer Institute for Ceramic Technologies and Systems - Materials Diagnostics (IKTS-MD), 01109 Dresden, Germany*
[2] *Dresden Center for Nanoanalysis (DCN), Technical University Dresden, 01062 Dresden, Germany*

**aranzazu.garitagoitia@tu-dresden.de*

Low voltage SEM (LVSEM) combined with Backscattered Electron (BSE) imaging offers new characterization possibilities for beam-sensitive materials. LVSEM reduces the beam-effect named as shrinkage in Organosilicate glass (OSG) thin films, a material widely used as an insulator between on-chip interconnects in microelectronic products due to its reduced permittivity (low dielectric constant (κ)). The energy of the primary beam (E_p) plays a fundamental role in the presence of shrinkage. After optimizing the SEM working conditions, at $E_p \leq 1000$ eV the damage in the OSG film is significantly mitigated. The grid voltage of the Energy selective Backscattered (EsB) electron detector is adjusted to a corresponding value of minimum the half of the primary beam energy, to make sure that all the secondary electrons (SE) are cut off and only BSE images are obtained. In Figure 1, BSE image d) shows no shrinkage in the OSG film [1]. The use of BSE imaging at low E_p values also allows to detect small composition differences between compounds, such as in the case of the blend formed by Zn-Phtalocyanine (ZnPc) and fullerene (C_{60}), used as an active layer in organic photovoltaic (OPV) thin films. The characterization of the specimen morphology is a key step to improve fundamental features of functional materials, e.g. the energy conversion efficiency of solar cells using OPV thin films. BSE imaging (Figure 1e) [2] allows to differentiate the morphology of the ZnPc (bright long rods) and the C_{60} (dark nanoparticles) due to the presence of the material contrast, which is obtained exclusively using BSE imaging at low primary beam energies.

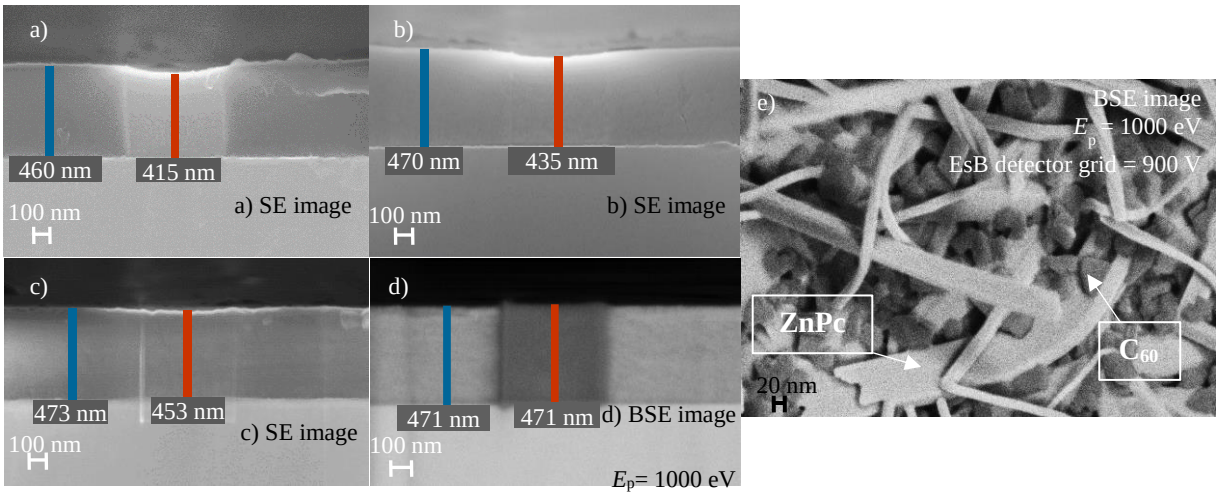

Figure 1. SE images of shrinkage (a), (b), (c) in an OSG thin film on Si substrate at different E_p values. The BSE image (d) shows no shrinkage at E_p = 1000 eV [1]. e) The characterization of the blend morphology (ZnPc-filaments and C_{60} nanoparticles) using BSE imaging allows the identification of the compounds [2].

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Thickness measurements of sputtered Au layers on fine-pitch Cu bump structures using µXRF

P15

S. Bickel* [1], J. Meyer[1], S. H. Lau[2], B. Stripe[2], Z. Liao[3], I. Panchenko[1, 4], E. Zschech[3]

[1]*Institute of Electronic Packaging Technology, Technische Universität Dresden, 01062 Dresden, Germany*
[2]*Sigray Inc., 5750 Imhoff Drive, Suit I, 94520 Concord, CA, United States of America*
[3]*Fraunhofer Institute for Ceramic Technologies and Systems IKTS Maria-Reiche-Str. 2, 01109 Dresden, Germany*
[4]*Fraunhofer Institute for Reliability and Microintegration IZM-ASSID, Ringstr. 12, 01468 Moritzburg, Germany*

**steffen.bickel@tu-dresden.de*

Future advanced packaging requires the development of processing technologies in order to meet the demands for highly-performant, heterogeneous and energy efficient systems. In conjunction with the prospective shrinking of vias and contact pitches the material combinations used become more complex and have to fit specific requirements. In particular, the controlled deposition of thin layers on fine-pitch contacts over an entire wafer is a key issue.

In this work, the layer thickness of sputtered Au films on Cu-microbumps was investigated with micro X-ray-fluorescence (µXRF) [1] analysis using the Sigray AttoMap X-ray analytical microscope [2]. The Au layer thickness was determined from the recorded spectra with fundamental parameter (FP) models and is in good agreement with the desired thickness. For comparison, the Au layer thickness was determined with transmission electron microscopy (TEM) using a Carl Zeiss Libra 200 Cs MC tool after the TEM lift-out lamellae had been prepared with focused ion beam technique (FIB) using a Carl Zeiss NVision40 tool. The cross-sectioned sample revealed Au layers of comparable thickness. A more precise determination of the metal layer thickness using µXRF requires the validation and calibration of the model used on a set of samples with several film thicknesses.

The method used in this work allows time-efficient investigation and quality control of deposited thin metallic layers on micrometer-sized 3D structures.

Acknowledgments

The authors acknowledge Fraunhofer IZM-ASSID for providing the samples. We thank Yvonne Standke (Fraunhofer IKTS Dresden) for FIB-based TEM sample preparation. This study was funded by the development bank of the Federal State of Saxony (SAB project “Functional integration for the micro-/nanoelectronics”, number 100249378) and the Fraunhofer society.

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Laser-induced graphene-based foams for electromechanical transducers

P16

A. F. Carvalho* [1], J. M. Pino[1], A. J. S. Fernandes[1], E. Fortunato[2], F. M. Costa[1]

[1]*I3N-Aveiro, Dep. Física da Universidade de Aveiro, Campus Universitário de Santiago, 3810-193, Aveiro, Portugal*
[2]*I3N-CENIMAT, Faculdade de Ciências e Tecnologia da Universidade Nova de Lisboa, Campus da Caparica, 2829-516 Caparica, Portugal*

* *alexandre.carvalho@ua.pt*

The 2D nature of graphene can sometimes be a hinderer to the full exploitation of its properties. As an example, the high intrinsic surface area of graphene in a 3D context is limited to the substrate area. Additionally, the volume production of pristine graphene is still a challenge, but alternatives exist, although sacrificing some of the extreme properties of graphene [1]. Graphene oxide (GO) and reduced graphene oxide (rGO) have been applied to tasks where a high production volume is required. These materials can be applied to the production of foams where the high intrinsic surface area can be actively explored for applications ranging from electrochemistry to electromechanical transduction.

Laser-Induced Graphene (LIG) can be an alternative to rGO's in this framework, yielding a fast and inexpensive way of producing patternable devices for diverse applications [2]. In this work, we explore electromechanical transduction, namely through the piezoresistive and the thermoacoustic effects.

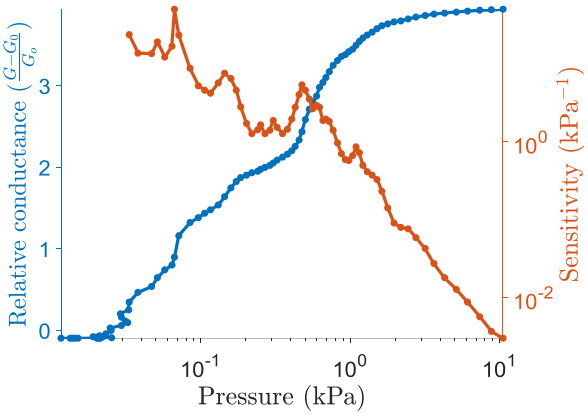

Figure 8. Contact piezoresistive response between two LIG foams.

Acknowledgments

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Zinc-tin Oxide Nanowires: Electrical Characterization inside SEM

P17

Ana Rovisco*, Rita Branquinho, Elvira Fortunato, Rodrigo Martins and Pedro Barquinha**

i3N/CENIMAT, Department of Materials Science, Faculty of Science and Technology, Universidade NOVA de Lisboa and CEMOP/UNINOVA, Campus de Caparica, 2829-516 Caparica, Portugal

**a.rovisco@campus.fct.unl.pt, **pmcb@fct.unl.pt*

Indium-free multicomponent oxide nanowires (NWs) such as Zinc-Tin Oxide (ZTO) are some of the most promising material systems for an upcoming generation of sustainable yet high performing transparent nanoelectronics. In this work we present a simple and low-cost solution-based method to synthesize zinc-tin oxide nanowires, decreasing complexity, cost and high temperature typical of vapor-phase methods [1]. The nanowires were obtained by an hydrothermal process at 200 °C during up to 24 hours in a furnace [2] using two different zinc precursors, Zinc Chloride and Zinc Acetate. Their morphology and crystallinity were characterized by XRD and SEM while FTIR analysis allowed to confirm the nanowires' crystalline phase. Electrical characterization (Figure 1) of $ZnSnO_3$ single-wires was performed with nanoscale Pt electrodes deposited by SEM-assisted deposition and using nanomanipulators inside SEM-FIB, allowing the measurement of the nanowires resistivity, being the obtained values in the desired range for the application as a semiconductor.

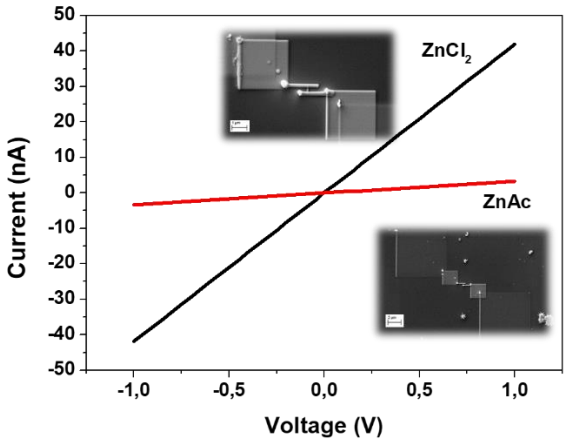

Figure 9. IV-measurements of ZTO single-nanowires produced by hydrothermal process, using Zinc Chloride ($ZnCl_2$) and Zinc Acetate (ZnAc) as Zinc precursor.

Acknowledgments

This work is funded by FEDER funds through the COMPETE 2020 Programme and National Funds throught FCT - Portuguese Foundation for Science and Technology under the project number POCI-01-0145-FEDER-007688, Reference UID/CTM/50025. This work also received funding from the European Community's H2020 program under grant agreement No. 716510 (ERC-2016-STG TREND) and No. 685758 (1D-Neon).

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Cellulose based substrates for application in electronic devices

P18

Diana Gaspar*, Elvira Fortunato, Rodrigo Martins, Luís Pereira*

CENIMAT/I3N, Departamento de Ciência dos Materiais, FCT-UNL and CEMOP-UNINOVA, 2829–516 Caparica, Portugal

** dgaspar@uninova.pt, **lmnp@fct.unl.pt*

Nowadays paper substrates are considered as a potential "electronic" material with growing interest among scientific community, due to the possibility of having low cost, disposable and recyclable electronic devices. Many research groups have being working on the optimization of cellulose based substrates for electronic applications either by using paper as a support for devices, as dielectric layer in field effect transistors (FETs) or by functionalizing it with conductor/semiconductor materials. In this work, we present insights on cellulose-based substrates and device's configuration aiming the application in electronic devices, such as FETs.

FETs using gallium indium zinc oxide (GIZO) (1:2:2 mol%) deposited by rf-magnetron sputtering as semiconductor, were produced on the surface of a different cellulose-based substrates. Unconventional cellulose sources as bacteria-produced biopolymers due to its interesting features can be used in electronic devices presenting similar performance to the devices produced in conventional paper [1].

Highly transparent cotton-based nanocrystalline cellulose films can be used as the gate dielectric layer with FETs reaching a drain–source current on/off modulation ratio higher than 10^5 [2]. Concerning the paper's tailoring, it was observed that the addition of HCl to M-NFC pulp can improve the FETs' performance, achieving saturation mobility up to 16 $cm^2 V^{-1} s^{-1}$, with an I_{ON}/I_{OFF} ratio close to 10^5 [3,4].

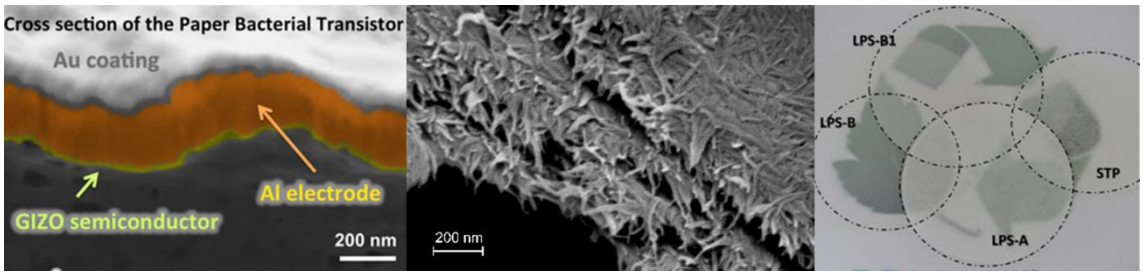

Figure 10. a) Cross section of a FET on bacterial cellulose, b) Nanocrystalline cellulose rods obtained from microcrystalline particles produced from cotton, and c) Paper engineered samples.

Acknowledgments

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Improvement of solution-based high-κ dielectrics at low temperature for electronic structures

P19

E. Carlos*, R. Branquinho, A. Kiazadeh, P. Barquinha, R. Martins, E. 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

**e.carlos@campus.fct.unl.pt; **emf@fct.unl.pt*

In the last decade, significant efforts have been made in the synthesis of a wide range of inorganic dielectrics using chemical solution routes do to their versatility, low cost and scalability for printed and flexible electronics. Solution synthesis provides a wide range of variables that can be tuned to yield the desired material. In solution combustion of oxide materials, the most determinant parameters are the metal source and organic fuel as these in fluences the oxide formation temperature and properties [1].
In the first part of this work the effect of different organic fuels, urea (U) and citric acid (CA), on the properties of solution-processed aluminum oxide thin films was investigated. Then, the influence of far ultraviolet (FUV) irradiation on properties of the insulator on thin film transistors was studied at lower temperatures, in order to have compatibility with flexible substrates [2]. To improve the devices performance at 150 °C, multilayer dielectrics, composed by aluminum and hafnium oxide, were produced, using the alliance between solution combustion synthesis (SCS) and deep ultraviolet (DUV) treatment [3].

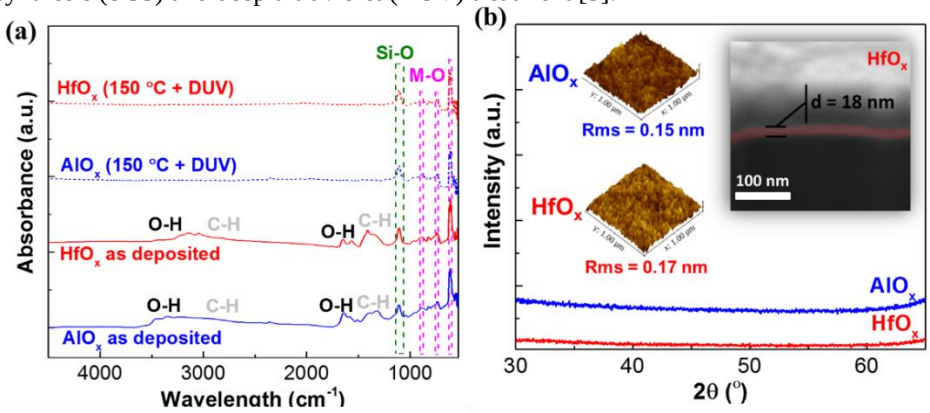

Figure 11. (a) FTIR spectra of AlO_x and HfO_x thin films before and after annealing; (b) XRD diffractograms, AFM deflection images (1×1 μm2) of both dielectric thin films, and high-resolution SEM-FIB cross-section image of HfO_x thin film.

Acknowledgments

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Thermoplastic composite materials obtained via reactive microencapsulation of payloads in polyamide core-shell structures

P20

Filipa M. Oliveira* [1], Nadya Dencheva[1], Senentxu Lanceros-Méndez[2, 3], Zlatan Denchev[1]

[1]*Institute for Polymers and Composites/I3N, University of Minho, 4800-058 Guimarães, Portugal*
[2]*Center/Department of Physics, University of Minho, 4710-057 Braga, Portugal*
[3]*BCMaterials, Parque Científico y Tecnológico de Bizkaia, 48160 Derio, Spain*

* *fmoliveira@dep.uminho.pt*

In this work PAMC were synthesized by activated anionic ring-opening polymerization of ε-caprolactam. The process was carried out in solution, in the presence of metal powders (Al, Cu, Fe, Mg), finely divided carbon allotropes – carbon nanotubes (CNT), carbon black (CB) and their mixtures. A recently developed and patented method was applied for the purpose [1]. All payload-containing PAMC obtained have controlled molecular weight, composition and granulometry, the organic or inorganic load varying in the 10-20% range (according to TGA). After synthesis of PAMC two types of composites were prepared: i) by conventional melt processing PAMC were transformed into thermoplastic polyamide-based hybrid composites; ii) in the second case, the metal-PAMC are compression molded in the presence of carbon fiber textile (CFT) structures to produce polyamide 6 matrix dually reinforced comprising different volume fractions (V_f) of CFT. Previous studies on polyamide powders preparation by AAROP in solution proved the simultaneous introduction into the polyamide matrix of organic fillers without any functionalization in wide concentration ranges and with controlled dispersion [2] and the preparation of polyamide laminate composites based on PA6 matrices reinforced by polyamide 66 (PA66) textile structures and different types of nanoclays [3]. Based on the results obtained it can be concluded that this fabrication concept allows the production of thermoplastic composite materials composites with high content of loads, improved mechanical performance, thermal stability and tailored electrical and dielectric properties.

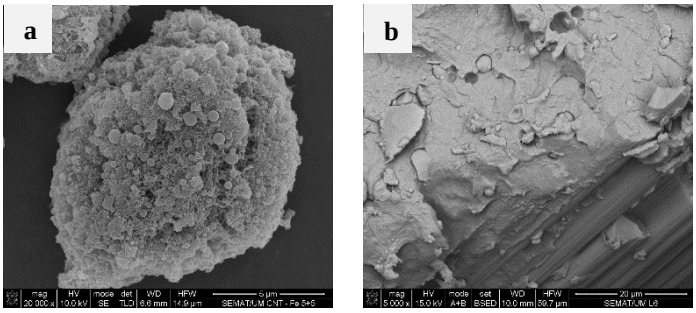

Figure 12. Selected SEM micrographs of a) PA6/CNT-Fe 5+5 wt% and b) polyamide laminate composite prepared with PA6/Fe 10 wt% and CFT structures (V_f = 25%).

Acknowledgments

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Cellulose Ion Ecosticker applied as gate dielectric in paper electrolyte-gated transistors

P21

I. Cunha, R. Barras, P. Grey, D. Gaspar, E. Fortunato, R. Martins, L. Pereira***

CENIMAT/i3N, Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologi, FCT, Universidade Nova de Lisboa, and CEMOP-UNINOVA, Campus da Caparica, 2829-516 Caparica (Portugal)

**i.cunha@campus.fct.unl.pt; ** lmnp@fct.unl.pt*

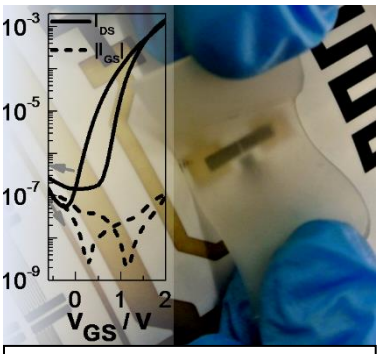

Figure 13. Photography of the developed flexible and transparent Cel-IES and respective cyclic transfer characteristic curve of the bendable EGT on cellulose-based paper substrate.

In this work, we introduce an innovative and versatile generation of reusable and eco-friendly hydrogel electrolytes for iontronics composed of some of the cheapest, renewable and highly abundant materials we can find on Earth, such as cellulose.[1] Besides their environmental benign and economically efficient fabrication process scalable for mass production, outstanding electrochemical properties and stability over time, what stands out in the celulose-based hydrogel electrolytes (CHEs) is their smart, simple, quick and very convenient application strategy which is conformable to a wide range of surfaces when applied on electrochemical devices in the form of a sticker.

The user-friendly concept of implementation of the cellulose ion ecosticker (Cel-IES) consists of cut, transfer, stick and reuse, which allows its successful implementation as gate dielectric in flexible indium-gallium-zinc oxide (IGZO) paper electrolyte-gated transistors (EGTs), which operate at low voltages (< 2 V) with an $I_{on/off}$ above 10^4 and with switching frequencies up to 100 Hz.

Combining the flexibility of the EGTs on paper with the reusability and recyclability of the developed Cel-IES is a breakthrough that opens new horizons in the chemistry of cellulose towards biodegradable advanced functional materials allied with disposable/recyclable and low-cost electronic devices.

Acknowledgments

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Polyaniline and Composite Engineering: A Class of Multifunctional Smart Materials

P22

S. Goswami*, S. Nandy, D. Nunes, S. Pereira, T. R. Calmeiro, R. Martins, E. Fortunato

i3N-CENIMAT, Department of Materials Science, FCT, Universidade Nova de Lisboa, 2829-516 Caparica, Portugal

**sumita.ju@gmail.com/s.goswami@campus.fct.unl.pt*

Polymer material comprises significant advantages over the conventional inorganic material based electronics due to its attractive features including miniaturized dimension and feasible improvisations in physical properties through molecular design and chemical synthesis. In particular, conjugate polymers are of great interest because of their ability to control the energy gap and electro-negativity through molecular design that has made possible the synthesis of conducting polymers with a range of ionization potentials and electron affinities. A central issue in the physics of these π-conjugated polymers is the strong coupling between electronic, geometric and chemical structures, i. e., the bonding pattern of atoms in the molecular system. The excellent properties of π-electron delocalization at the backbone of conjugate polymer can be initiator of charge transfer mechanism with the incorporated nanoparticles resulting tunibility of electronics dynamics within the composites. This unique combination of two types of materials has the ability to expand the class of novel composite materials with controlling their properties through rational chemical synthesis. Polyaniline (PANI) is one of the most investigated conjugated polymers because of its facile synthesis, excellent electronic properties and high environmental stability [1]. The aim of the work is to develop novel strategies for synthesis of polyaniline with different forms (depending on oxidation) and incorporate inorganic nanoparticles (oxides, metals, carbon nano dots) via simple, feasible and cost-effective techniques towards the multiple applications of mechano-electrical devices [2], memory device, PL sensors, electrochromic displays etc. Figure 1 depicts the different nanostructures of PANI and its composites.

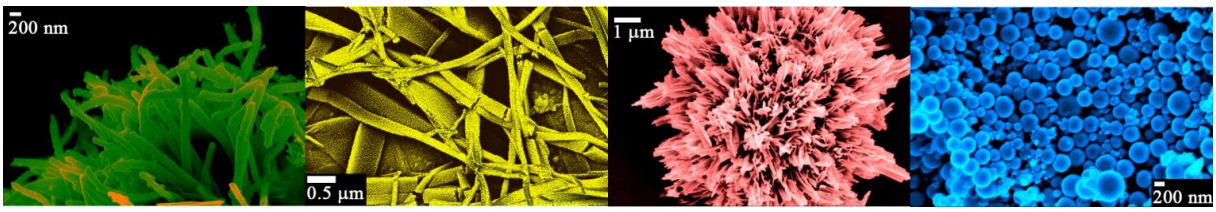

Figure 14. Different nanostructures of polyaniline and polyaniline-composite materials.

Acknowledgments

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TCAD Simulation of Amorphous Indium-Gallium-Zinc Oxide Thin-Film Transistors

P23

Jorge Martins*, Rodrigo Martins, Elvira Fortunato and Pedro Barquinha**

i3N/CENIMAT, Department of Materials Science, Faculty of Science and Technology, Universidade NOVA de Lisboa and CEMOP/UNINOVA, Campus de Caparica, 2829-516 Caparica, Portugal

** jds.martins@campus.fct.unl.pt, **pmcb@fct.unl.pt*

Technology Computer-Aided Design (TCAD) tools allow for simulation of electronic devices at a physical level. Having been a great asset in the past for the development of silicon technologies, it is expected that a similar root can be taken for maturing oxide electronics [1] allowing for an understanding on how to further improve device performance and stability as required to meet the technology's full potential [2]. By giving insight on mechanisms behind device operation and allowing to explore different device configurations (geometries/materials) these tools can lead to a viable process and device development. Currently, TCAD is already enabling us to investigate aspects such as short-channel effects (Figure 1 (a)), trap-related instability mechanisms and carrier distribution for single/double gate devices (Figure 1 (b)).

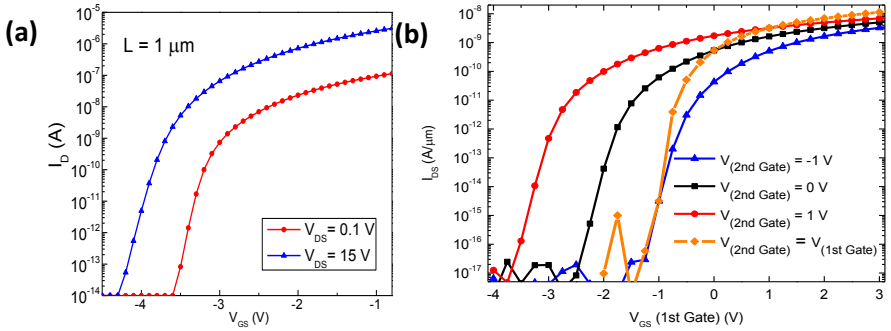

Figure 1. (a) Shifting transfer curve with the drain bias in a short-channel TFT. (B) Transfer characteristics of dual-gate TFT with different second-gate biasing conditions.

Acknowledgments

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Recent Research: Electrolyte and Paper Gated Transistors

P24

P. Grey *, S. Pereira, E. Fortunato, R. Martins and L. Pereira**

CENIMAT/i3N, DCM, FCT-UNL and CEMOP/UNINOVA, Campus da Caparica, 2829-516 Caparica, Portugal

*p.grey@campus.fct.unl.pt; **lmnp@fct.unl.pt

The present work shows recent work in the field of Electrolyte Gated Transistors (EGTs) from the Materials Research Center (CENIMAT) of the Faculty of Science and Technology from the New University of Lisbon (FCT-UNL). EGTs take a very important role in modern society, as electrolytes can be found in a variety of industry branches, reaching form biology to the paper industry, where paper can also be used as an electrolyte. Consequently the development of devices capable of reacting to these electrolytes and responding with a measurable output is crucial [1]. Here we present two different approaches (physical vapor deposition - PVD and printing techniques) of EGT fabrication with two distinct semiconductors: WO_3 and ZnO. Both of these techniques have taken key roles in research and industrial branches, complementing each other. Where PVD has an advantage in device performance and quality, printing techniques blossom due to their low-cost and large area fabrication possibilities. Both techniques are explored in this work in order to produce transistors, UV sensors (ZnO) and electrochromic (WO_3) devices (Figure 1). Two publications resulted from this work [2,3].

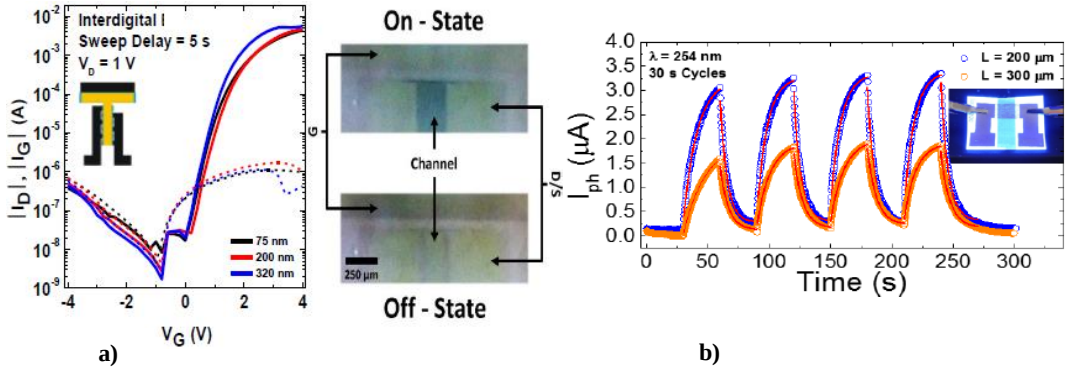

Figure 1. a) Sputtered WO_3 transistors with three different thicknesses exhibits semiconducting behavior and a simultaneous coloration from transparent to blue. b) UV response of a hybrid hand-written/ printed UV sensor on paper with ZnO nanoparticles as the active layer. Two distinct contact distances were used (200 and 300 µm).

Further, a short insight into the ongoing PhD project will be provided where the main goal relies in harvesting electronic and photonic properties of Cellulose Nanocrystals (CNCs) in optoelectronic devices.

Acknowledgments

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Microwave Synthesized ZnO Nanorod Arrays for UV Sensors: A Seed Layer Annealing Temperature Study

P25

A. Pimentel[*], S. H. Ferreira[**], D. Nunes, T. Calmeiro, R. Martins and E. Fortunato[***]

i3N/CENIMAT, Dep. de Ciência dos Materiais, Faculdade de Ciências e Tecnologia – Universidade NOVA de Lisboa and CEMOP-UNINOVA, 2829-516 Caparica, Portugal

[*]*acgp@campus.fct.unl.pt;* [**]*sdl.ferreira@campus.fct.unl.pt;* [**]*emf@fct.unl.pt*

Zinc oxide (ZnO) is an n-type semiconductor that has been extensively studied due to its unique properties, namely its direct and wide band gap (3.37 eV) and high exciton binding energy (60 meV) at room temperature. These exceptional properties allow ZnO to behave as an efficient semiconductor and, therefore, to be applied in several functional devices, such as thin film transistors [1], light emitting diodes (LEDs), UV/ozone sensors [2], biosensors piezoelectric devices, among others.

Among all the ZnO nanostructures that can be synthetized, ZnO nanorods (NRs) have attracted much attention due to their high surface-to-volume ratio and high sensitivity under ambient conditions, which make them highly appealing for sensing applications [3].

The present work reports the influence of zinc oxide (ZnO) seed layer annealing temperature on structural, optical and electrical properties of ZnO nanorod arrays, synthesized by hydrothermal method assisted by microwave radiation, to be used as UV sensors. The ZnO seed layer was produced using the spin-coating method and several annealing temperatures, ranging from 100 to 500 °C, have been tested. X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM) and spectrophotometry measurements have been used to investigate the structure, morphology, and optical properties variations of the produced ZnO nanorod arrays regarding the seed layer annealing temperatures employed. After the growth of ZnO nanorod arrays, the whole structure was tested as UV sensors, showing an increase in the sensitivity with the increase of seed layer annealing temperature. The UV sensor response of ZnO nanorod arrays produced with the seed layer annealed temperature of 500 °C was 50 times superior to the ones produced with a seed layer annealed at 100 °C.

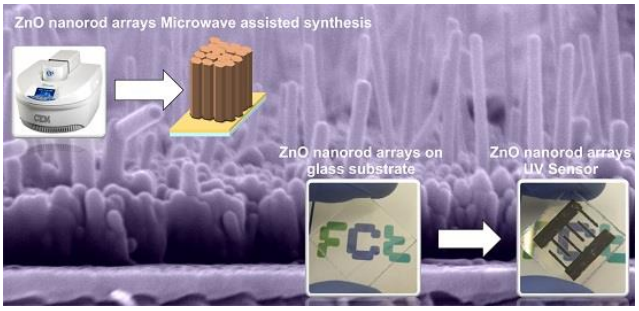

Figure 16. Graphical abstract that summarizes the presented work.

Acknowledgments

This work was partially financed by FEDER funds through the COMPETE 2020 Programme and National Funds through FCT (Portuguese Foundation for Science and Technology) through BPD/76992/2011 and under the projects UID/CTM/50025/2013 and EXCL/CTM-NAN/0201/2012. S. H. Ferreira acknowledges the support from the Portuguese Foundation for Science and Technology through the AdvaMTech PhD program scholarship PD/BD/114086/2015.

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Non-destructive Characterization of Surfaces and Coatings Using Laser-Induced Surface Acoustic Waves

P26

Martin Zawischa*, Dieter Schneider, Michael Leonhardt, Stefan Makowski, Volker Weihnacht

Fraunhofer Institute for Material and Beam Technology, Dresden, Germany

martin.zawischa@iws.fraunhofer.de

The measurement of mechanical coating properties is essential for understanding and development of modern coatings systems as well as for quality control of coating processes.
For more than ten years laser induced surface acoustic wave (SAW) spectroscopy has been made commercially available is used for fast and non-destructive measurement of coating and surface properties.
The SAW phase velocity depends on several material parameters like elastic modulus, density, coating thickness, which can be influences from secondary parameters like porosity, cracks, delamination and hardness gradients. Since the penetration depth of SAW depends on their wave length, depth-resolved material information can be collected by evaluation of different frequencies.

After the so called dispersion curve (phase velocity over frequency) has been measured, material properties can be calculated by fitting the curve to a theoretical material model.
Application of this method is wide-spread and includes R&D and quality control for PVD and CVD coatings, thermal-sprayed coatings, semiconductors and bulk materials.
The current material model was limited to either single or alternating double layers. In this work a new material model, featuring up to five individual layers, has been implemented. Its application is demonstrated on a tetrahedral amorphous carbon coating (ta-C) with a Chromium interlayer on a Silicon wafer. Depending on the curvature of the measured dispersion curve up to three independent properties of the material system can be determined, including the thickness of the covered Chromium interlayer (Figure 1).
These results extend the capabilities for direct measurement of coating and surface properties and open up new possibilities for more advanced measurement strategies.

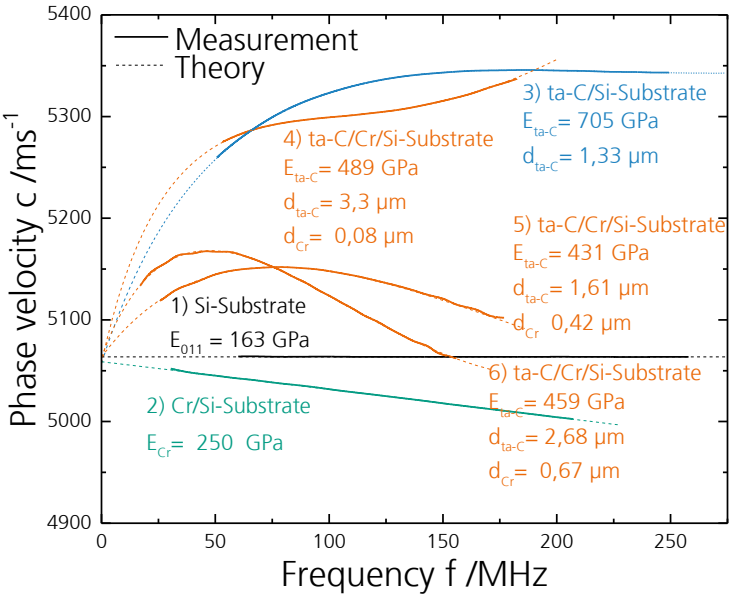

Figure 1: Measured (solid line) and calculated (dashed line) dispersion curves for an uncoated silicon wafer (black), Chromium coated silicon wafer (green), ta-C coated silicon wafer (blue), and ta-C/Cr-coated silicon wafers (red). Measured material properties are shown in the diagram next to the corresponding dispersion curves.

In-Situ Study of High-Temperature Mechanical Properties of Aluminide Bond Coatings

P27

Sanjit Bhowmick, Ude Hangen*, S. A. Syed Asif

Bruker Nano Surfaces, Minneapolis, MN 55344, USA

* *Ude.Hangen@bruker.com*

Aluminide bond coatings are compositionally and microstructurally graded materials with significant variation in engineered mechanical properties across the cross-section. An in-situ SEM nanomechanical study was conducted to understand the variation in mechanical properties and deformation mechanisms of such coating as a function of temperature. The results indicate that plasticity is characterized by major strain hardening at room temperature and limited strain hardening at higher temperature. Transgranular fracture appears on the pillar surface at room temperature whereas grain boundary sliding and intergranular fracture dominates at higher temperature.

Mechanical Property Distribution by Nanoindentation Mapping

P28

Dr. Ude Dirk Hangen[1], Dr. Junhe Lian[2]

[1]*Bruker BNS, 52068 Aachen*

[2] *IEHK-RWTH Aachen*

Microstructures in metals are composed from grains of various phases and precipitates ranging from tens of micron of down to the nm scale with a large variation in yield strength and Young´s Modulus. Micro hardness tests typically determine an average materials behavior. Nanohardness testing allows to probe volumes of the order of 100nm^3. Recent developments in nanoindentation instrumentation allow performing hardness tests at rates of several indents per second. The increased amount of precise nanoindentation experiments is used to investigate the spatial distribution of mechanical properties within the microstructure and compare it to complementary information like EBSD maps.

A good match is found between the micro hardness experiment and the average hardness found from the nanoindentation maps with the added information of the hardness distribution. These findings are promising and can be used for studying local changes in microstructures originating from welding processes or mechanical surface treatments.

DNA-based plasmonic nanoantennas

P29

Mathias Lakatos*[1], Hanna Brunner[1], Andreas Heerwig[2], Darius Pohl[3], Bernd Rellinghaus[3], Toni Haugwitz[4], Dirk Plettemeier[4], Michael Mertig[1, 2]

[1]Technische Universität Dresden, Physikalische Chemie, Mess- und Sensortechnik, 01062, Dresden, Germany
[2]Kurt-Schwabe-Institut für Mess- und Sensortechnik e.V. Meinsberg, 04736 Waldheim, Germany
[3]Leibniz IFW, Institut für Metallische Materialien, 01171 Dresden
[4]Technische Universität Dresden, Nachrichtentechnik, Hochfrequenztechnik, 01062, Dresden, Germany

** Mathias.Lakatos@tu-dresden.de*

Artificially designed DNA structures can be used for the site-specific arrangement of functional heteroelements into optical or electronic nanodevices, e.g. for optical data processing and signal transmission. Here, we present the bottom-up assembly of optical nanoantennas by arranging plasmonic nanostructures operating at optical frequencies with thw help of DNA origami templates. As templates, we have used rectangular, two-dimensional origami structures with a basic dimension of 40 nm x 150 nm- They specifically form dimers, so that DNA templates with sizes of either 40 nm x 300 nm or 80 nm x 150 nm were obtained. The incorporation of specific binding sites enables the local positioning of functional elements. For the construction of plasmonic active nanoantennas, complementary functionalized gold nanoparticles of different shape (spheres, rods), size (e.g. aspect ratio of the rods) and material (Au/Ag) were used. Depending on the design of the template structures and the size of the immobilized nanoelements, different designs of nanoantennas of about 100 nm up to 300 nm size have been synthesized.

We present results of the structural and optical characterization of these nanoantennas. This includes bulk absorption measurements by UV-vis spectroscopy as well as characterization of individual antenna structures by low-loss electron energy loss spectroscopy (EELS) using monochromated scanning transmission electron microscopy (STEM) to gain insight into near-field coupling effects and the localization of bonding and antibonding plasmonic modes. Additional FEM simulations show that these experimental results are in good agreement with theory.

Antimicrobial Polyelectrolyte Multilayer Films in regulation of cell response

P30

Aldona Mzyk*[1], Michał Wojasiński[2], Justyna Straub[1], Martijn Riool[3], Leonie de Boer[3], Roman Major[1], Sebastian Jan Zaat[3]

[1]*Institute of Metallurgy and Materials Science, Polish Academy of Sciences, Reymonta 25, 30-059 Cracow, Poland*
[2]*Department of Biotechnology and Bioprocess Engineering, Warsaw University of Technology, Waryńskiego 1, 00-645 Warsaw, Poland*
[3]*Academic Medical Center, Meibergdreef 9, 1105 AZ Amsterdam, The Netherlands*

**aldonamzyk@gmail.com*

Biomaterials engineering gives a great promise for the reconstruction of damaged myocardium. An effective cell transfer into human body takes place only with application of a biomaterial carrier. For this purpose, it is necessary to design materials regulating niche-specific cellular response. This is not possible without knowledge on fundamental relations between scaffold parameters and mechanism of adhesion, differentiation, as well as exosomal activity of cells. Moreover, newly designed scaffolds should prevent biomaterial associated infections. Therefore our studies are focused on delivery system for cardiac progenitor cells, which will provide with cells response regulation and antimicrobial activity. Scaffolds functionalized by Polyelectrolyte Multilayer Films (PEMs) facilitate control over surface properties such as stiffness, roughness, surface wettability and thus proteins adsorption as well as cellular response. Properties of PEMs were controlled by structural changes through the chemical cross-linking process and nanoparticles incorporation. Selected coatings have been subjected to the JIS contact/release antimicrobial activity test against Gram-positive (*S. epidermidis, S. aureus)* and Gram-negative (*E. coli*) bacterial strains. Significant contact/release antimicrobial activity has been found for the coatings with incorporated silver nanoparticles, as well as cross-linked by genipin. Results of cardiac progenitor cells – scaffold interaction indicate that PEMs modification has improved cell adhesion and proliferation as well as changed cell morphology and activity.

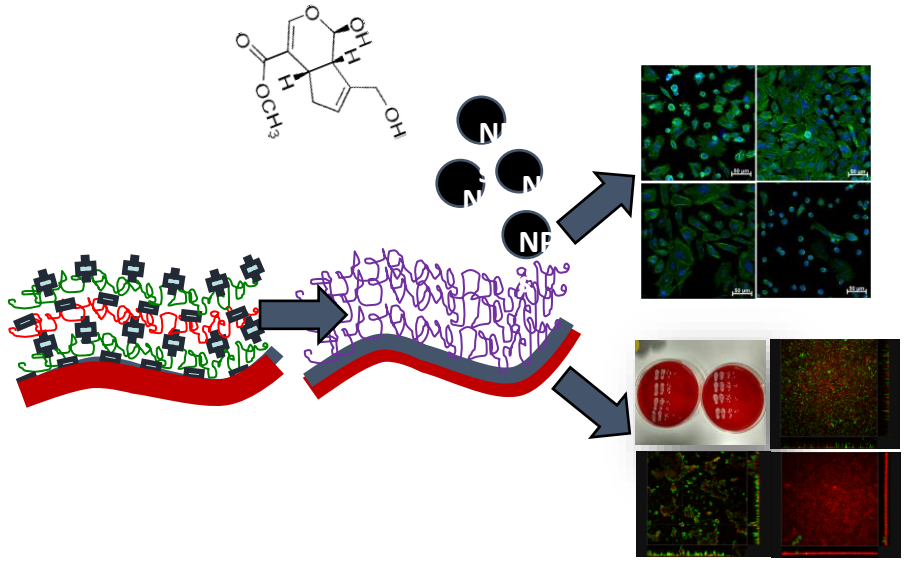

Figure 17. The concept of Polyelectrolyte Multilayer Films with multifunctional properties – human cells and bacteria response control.

Acknowledgments

The research was financially supported by the grant No. 2014/15/N/ST8/02601 of the Polish National Center of Science and partly by the statutory funding of the IMM PAS (Z-2) as well as the COST Action TD1305.

X-ray nano-diffraction analysis of core-shell semiconductor nanowires

P31

Ali Al Hassan[1], Arman Daytyan[1], R. B. Lewis[2], H. Küpers[2], D. Bahrami[1], L. Geelhaar[2]; U. Pietsch*[1]

[1]*Naturwissenschaftlich- Technische Fakultät der Universität Siegen, 57068 Siegen, Germany*
[2]*Paul-Drude-Institut für Festkörperelektronik, Hausvogteiplatz 5-7, 10117 Berlin, Germany*

**pietsch@physik.uni-siegen.de*

Semiconductor core-shell nanowires are attractive for future application in nano-optoelectronic devices. A(III)-B(V) zinc-blende and wurzite type materials can be grown along the [111] and [0001], resp., crystallographic direction onto silicon substrate by MBE or other growth techniques. Because of the low difference in formation energy nanowires are typically composed by sequential stacking of zinc-blende and wurtzite units. In addition, the formation of a core-shell structure creates an anisotropic epitaxial strain distribution within the nanowire.

In this work, we report on the evaluation of radial structure of core-shell nanowires. using synchrotron radiation. In particular we investigated individual GaAs/$In_xGa_{1-x}As$,(GaAs) core-shell-(shell) nanowire (NW) heterostructures with variable nominal indium contents and shell thickness grown by MBE onto silicon [111]. Composition analysis is performed by nano-XRD using nanobeams of less than 1µm² spot size. Along the growth directions all components grow pseudomorph, i.e. we find a single lattice parameter along the growth direction inducing compressive strain of $In_xGa_{1-x}As$ shell and tensile strain of the GaAs components. Subsequently the respective in-plane lattice parameter is expanded (compressed) towards the nanowire [1-10] side planes and along the [11-2] edges. Both become accessible via separated Brag peaks or core and shell allowing for determination of the full strain tensor.

We acknowledge support by DFG Pi217/38 and Ge2224/2 and beamline teams of ESRF (ID1) and PETRA III (P08).

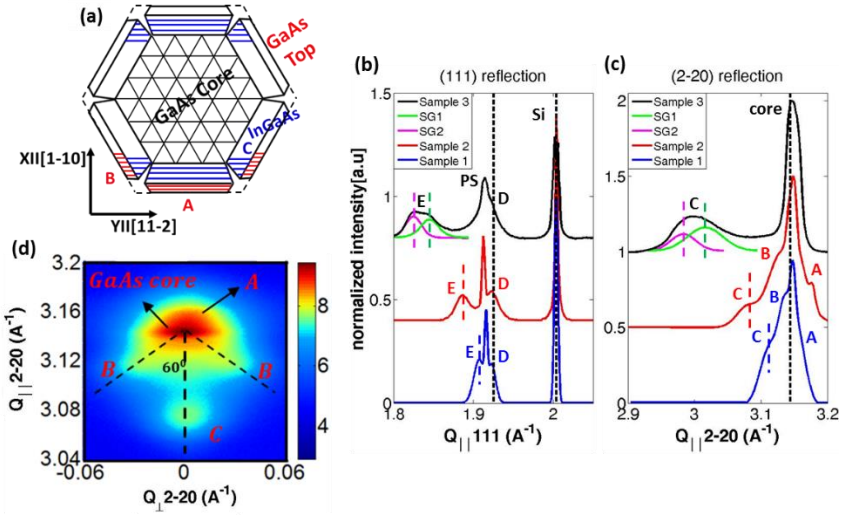

***Figure 1**: (a)Strain state along [2-20] for single GaAs/InGaAs/GaAs core-shell NW hetero-structure. (b) and (c) show XRD line scans of all samples plotted along scattering vectors* $Q_{\parallel}(111)$ and $Q_{\parallel}(2-20)$, *respectively. For better visibility, the curves are plotted with vertical offsets. The position expected for unstained GaAs in* $Q_{\parallel}(111)$ *is marked by "D". Positions expected for unstrained* $In_{0.15}Ga_{0.85}As$ *(blue),* $In_{0.27}Ga_{0.73}As$ *(red),* $In_{0.6}Ga_{0.4}As$ *(green) and* $In_{0.75}Ga_{0.25}As$ *(pink) are marked by vertically cut dotted lines and named "E"and "C", respectively. Graphical explanation of peaks A, B and C is shown (a). (d) 2D RSM of the (2-20) reflection for sample 2.*

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New composite ceramics based on gadolinium-doped ceria and magnesia nanoparticles

P32

J. Yao*[1], M. Schelter[1], J. Zosel[1], W. Oelßner[1] M. Mertig[1,2]

[1] *Kurt-Schwabe-Institut für Mess- und Sensortechnik e.V. Meinsberg, 04736 Waldheim, Germany*
[2] *Physikalische Chemie, Mess- und Sensortechnik, Technische Universität Dresden, 01062 Dresden, Germany*

**yao@ksi-meinsberg.de*

Composites of gadolinium-doped ceria (GDC) and magnesia (MgO) are being developed to achieve solid electrolyte materials for electrochemical energy storage devices with very high oxygen ion conductivity. A mixture of these two components was prepared and sintered in this study. The structures of the nanometer-sized powders and ceramic have been characterized by X-ray diffraction and transmission electron microscopy.

Introduction

Despite they have been investigated for more than a century [1], electrolyte materials limit the broad use of solid oxide fuel cells (SOFCs). A new possibility to obtain materials with high oxygen ion conductivities offers the work of Kosacki et al. [2]. They found that the grain boundaries between electrolyte and isolator particles can have even higher ionic conductivities than the electrolyte. Here we describe a novel approach how to make use of this unusual high boundary layer conductance. Due to its high ionic conductivity, GDC has been chosen as the electrolyte candidate.

Results and Discussion

A self-propagating high temperature synthesis method [3] is used for the synthesis of both GDC and MgO nanoparticles. The nanoparticles have diameters between 10-20 nm and show a narrow particle-size distribution. No aggregation was observed.

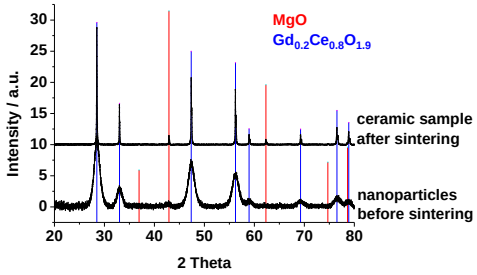

Figure 2. XRD pattern of the mixture of GDC and MgO sample before and after sintering

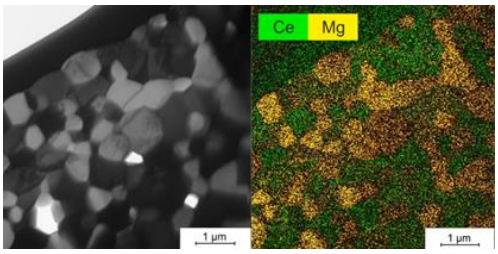

Figure 2. TEM and TEM-EDX images of the composite ceramic sample after sintering

The sample was then pressed and sintered. The XRD patterns in Figure 1 show two pure phases (MgO and GDC) of the samples both before and after sintering. Compared to the wide peaks of the nanoparticles, the narrowing of the peaks of the ceramic sample indicates the growth of crystallites during the sintering. The TEM and a TEM-EDX image of the composite ceramic shown in Figure 2 also manifest larger domains than the disperse nanoparticles before sintering.

Conclusions

Using the self-propagating high temperature synthesis method, GDC and MgO nanoparticles with diameters of about 10 nm can be synthesized with narrow particle-size distribution and no aggregation. Although the particle domains in the sintered ceramic are enlarged, they are still in the nanometer range.

Acknowledgments

The presented work is based on a project supported by the German Federal Ministry of Education and Research (support code: 03EK3029C). The authors are responsible for the content of the paper and thank gratefully for financial support.

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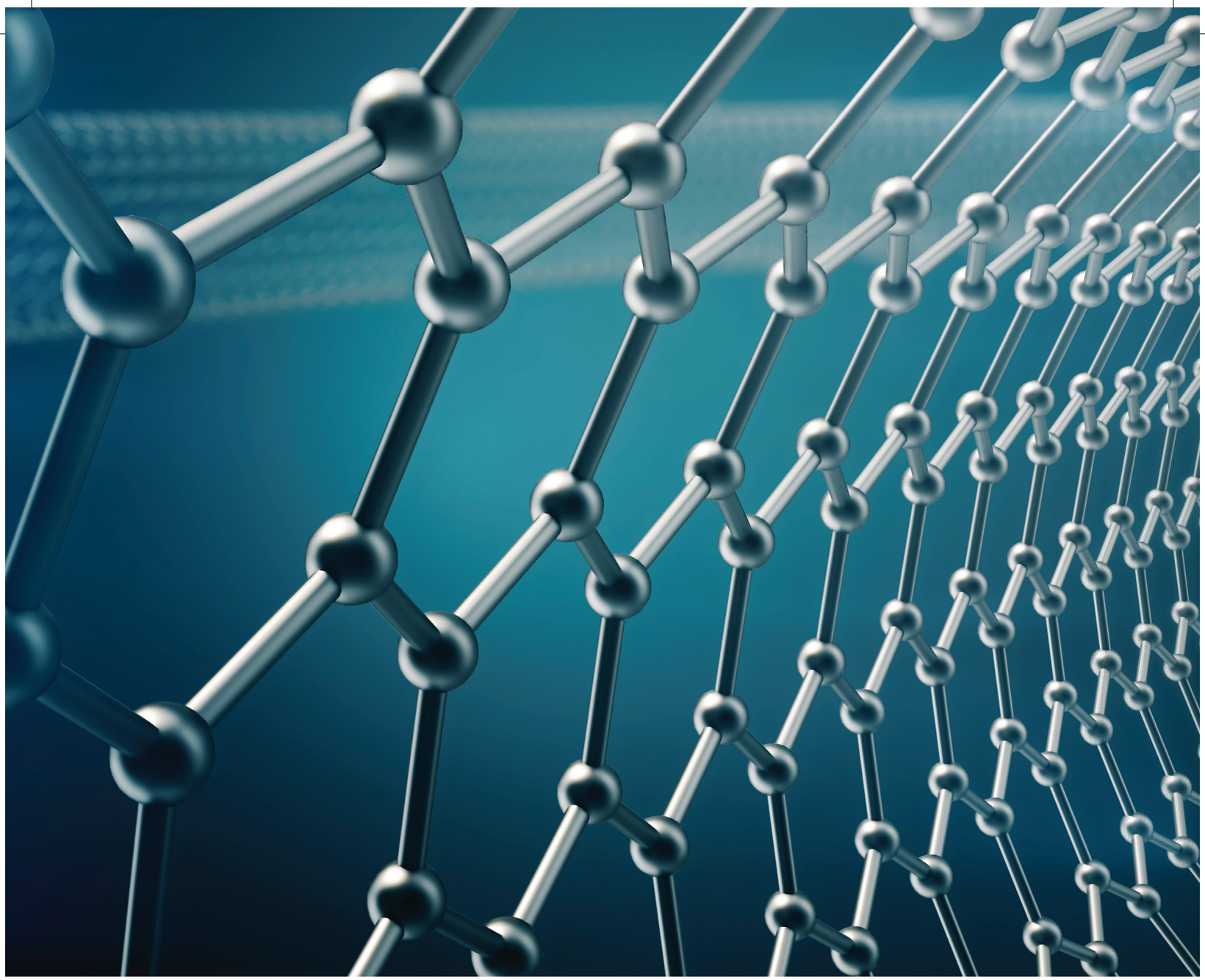

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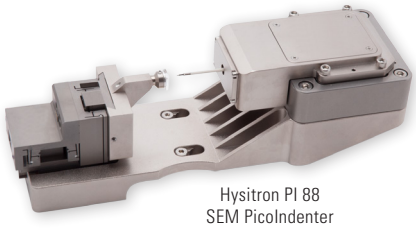

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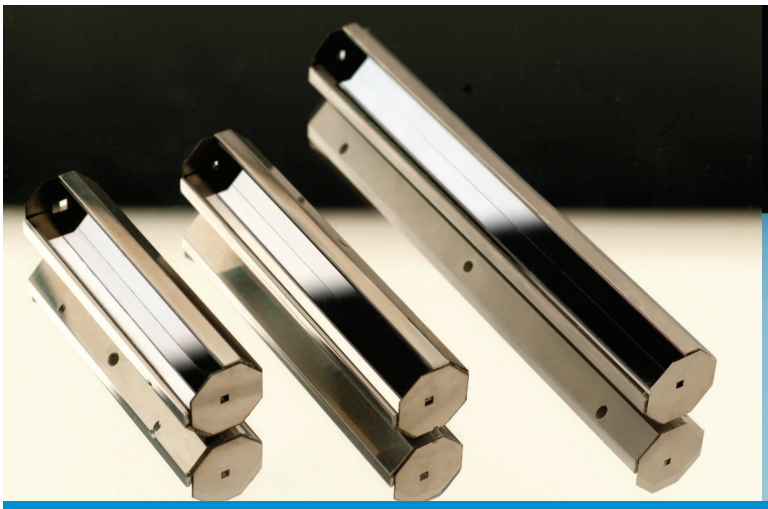

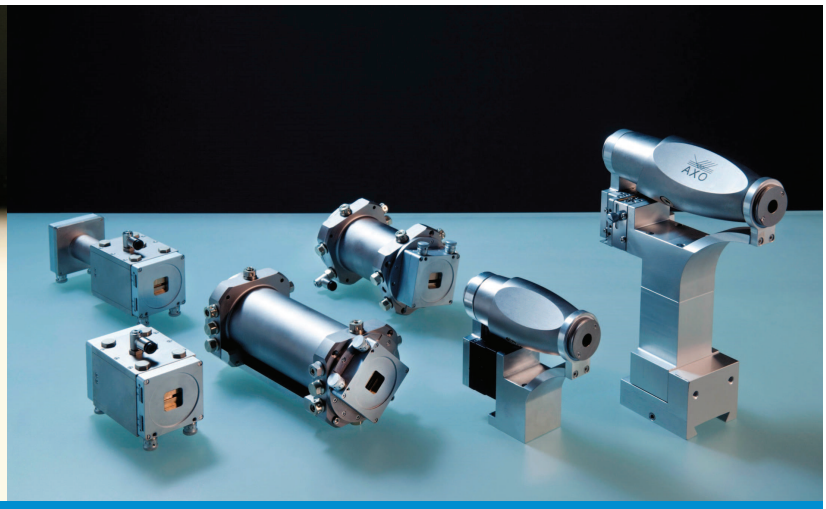

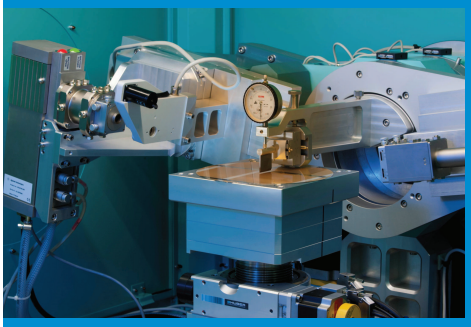

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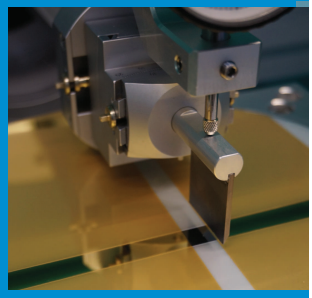

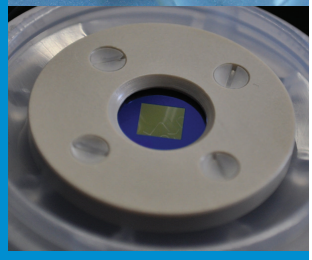

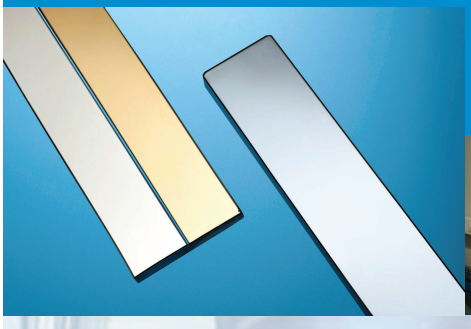

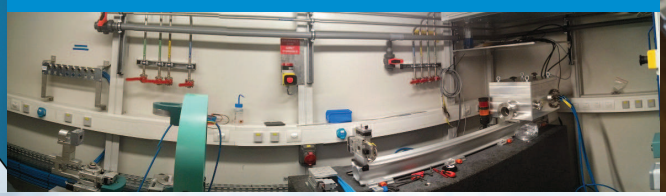

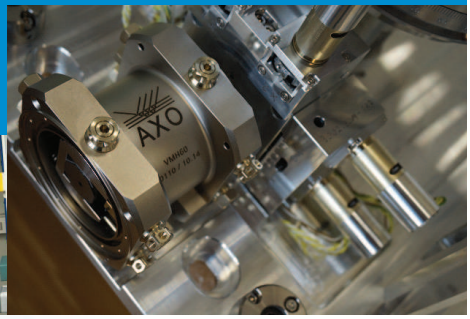

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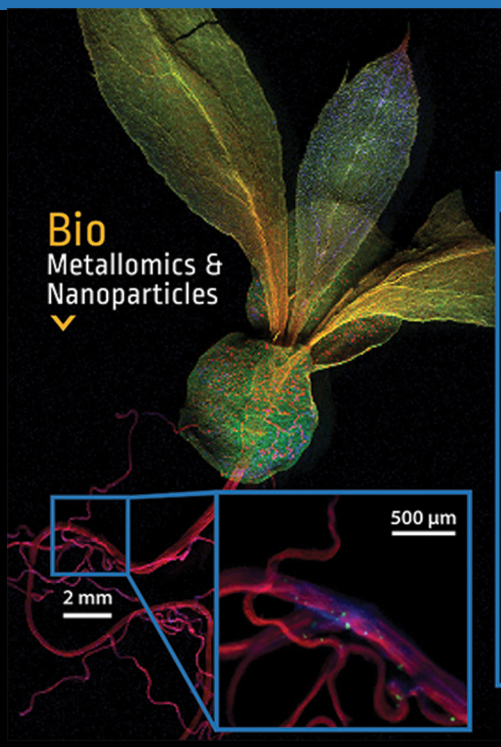
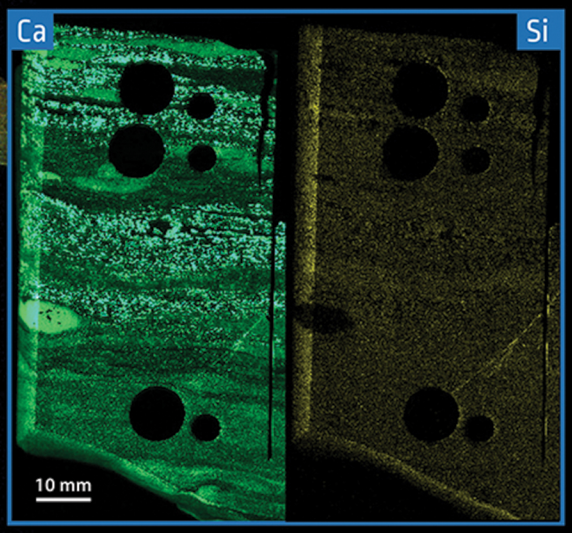
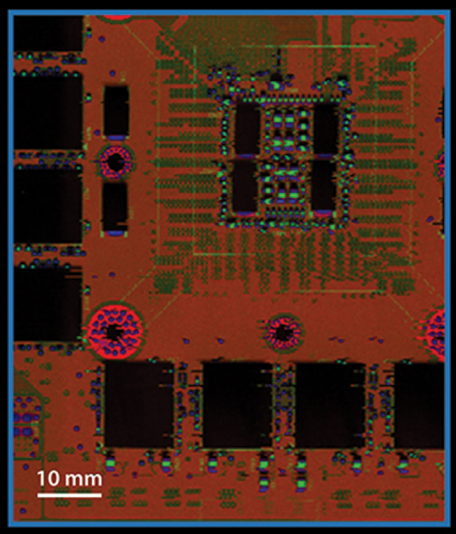
Bringing the **Power of Synchrotron Beamlines** to the Laboratory With **Patented** X-ray Source and Optic Innovations

Synchrotron-like throughput, resolution, and performance are finally possible in the laboratory







Techniques including:

- Micro X-ray Fluorescence for Elemental Distribution
- X-ray Absorption Spectroscopy for Chemical and Electronic Structure Determination
- X-ray Microscopy for Structural Determination

All enabled by patented x-ray source and optic technology

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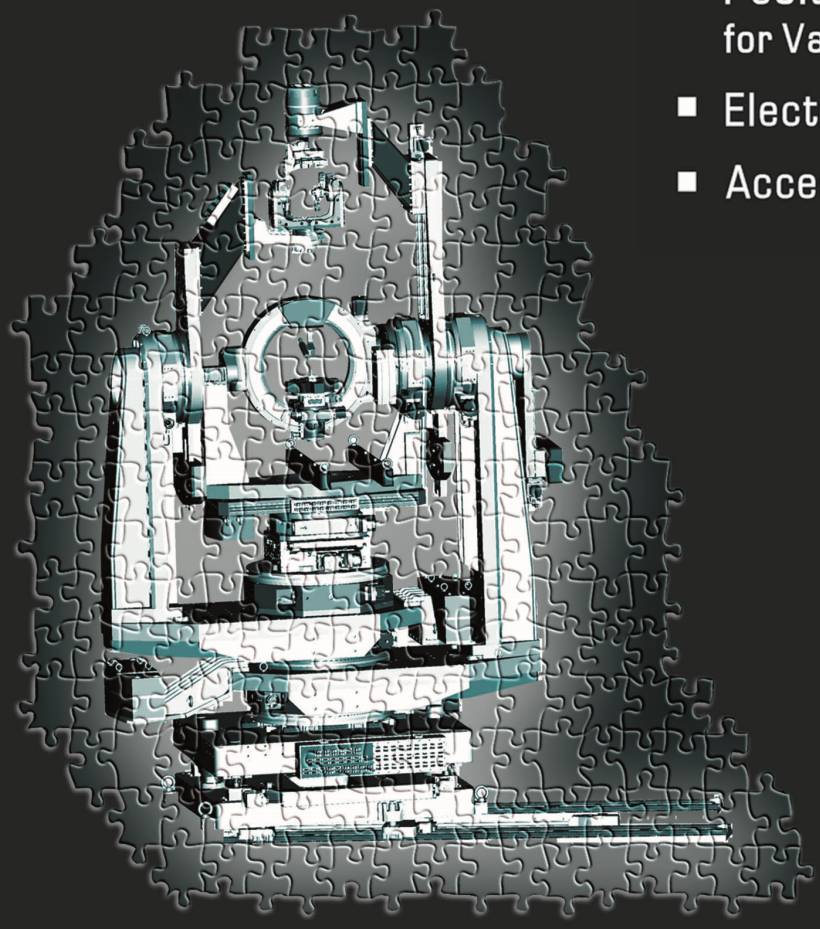
HUBER
Diffraction and Positioning Equipment

It fits

- X-Ray Diffractometers and Cameras
- Multiaxis Goniometers for X-Ray-, Synchrotron- and Neutron Facilities
- Monochromators
- Positioning Devices for Various Environments
- Electronics
- Accessories



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