Fraunhofer NANOANALYSIS

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6th Dresden Nanoanalysis Symposium

"Materials challenges for automotive industry – Micro- and nanoscale characterization"

Abstract booklet

August 31st, 2018
Dresden, Germany

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6th DRESDEN NANOANALYSIS SYMPOSIUM

"Materials challenges for automotive industry – Micro- and nanoscale characterization"

The 6th Dresden Nanoanalysis Symposium, organized by the Dresden Fraunhofer Cluster Nanoanalysis (DFCNA), supported by the European Materials Research Society (E-MRS) and the European Materials Characterisation Council (EMCC), will be held at the Fraunhofer Campus Dresden, Maria-Reiche-Strasse, on August 31, 2018. 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: "Materials challenges for automotive industry – Micro- and nanoscale characterization".

Based on the positive experience in the previous years, we would like to go ahead with the same format: One-day symposium with 3 keynote talks, 11 invited talks in 3 sessions, and a poster session for contributed papers. There will be exhibition space available for companies and organizations. The symposium will cover the topics of nanoanalysis and materials characterization along the whole value and innovation chain, from fundamental research up to industrial applications. It will bring scientists and engineers together from universities, research institutions, equipment manufacturers and industrial end-users. New results in disruptive nanoanalysis techniques will be reported in several talks and in the poster sessions, and novel solutions in the field of materials characterization for process and quality control will be shown. The discussions and interactions between the stakeholders will help to identify gaps in the fields of advancing nanoanalysis and materials characterization and to propose actions to close them and to support industrial exploitation of innovative materials. The symposium is supposed to reinforce ongoing collaborations and to discuss ideas for new collaborations.

Venue

August 31st, 2018, Dresden, Germany
Fraunhofer IKTS Dresden, Maria-Reiche-Strasse 2, 01109 Dresden

Organizational committee

Kristina Kutukova, Fraunhofer IKTS, *kristina.kutukova@ikts.fraunhofer.de*
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Symposium organized by Dresden Fraunhofer Cluster Nanoanalysis and Dresden Center for Nanoanalysis at TU Dresden

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

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- Robert Sinclair, Stanford University, California (USA)
- Olivier Thomas, University Marseille (France)
- Wilfried Vandervorst, IMEC, Leuven (Netherlands)
- Oden Warren, Bruker, Minnesota (USA)
- Thomas Weißgärber, Fraunhofer IFAM, Dresden (Germany)

Invited speakers

- Lars Dittert, Volkswagen Saxony, Germany
- Zhiyong Ma, Intel, Hillsboro/OR, USA
- Rodrigo Martins, UniNova Lisbon, Portugal
- Tobias Schulli, ESRF Grenoble, France
- Xijie Wang, SLAC, Stanford University, Palo Alto/CA, USA
- Luiz Tizei, Universite Paris Sud, Orsay, France
- Christoph Bäumer, Forschungszentrum Jülich, Germany
- Eckhard Langer, GLOBALFOUNDRIES, Dresden, Germany
- Wenbing Yun, Sigray, Concord/CA, USA
- Robert Sinclair, Stanford University, Palo Alto/CA, USA
- Tanja Graf, Volkswagen, Wolfsburg, Germany
- Sabrina Sartori, University of Oslo, Norway
- Marco Sebastiani, Universita Roma Tre, Italy
- Randi Holmestad, NTNU Trondheim, Norway

Program

Friday, August 31

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 – 11:00 am)
Chair: *Ehrenfried Zschech*

9:10 am – 9:40 am
KEYNOTE TALK
Volkswagen: Right on schedule for E-mobility
Lars Dittert, Head of Transparent Factory, Volkswagen Saxony, Germany

9:40 am – 10:10 am
KEYNOTE TALK
Materials innovation for technology scaling and the implications to metrology
Zhiyong Ma, VP Process Control, Intel, Hillsboro/OR, USA

10:10 am – 10:40 am
KEYNOTE TALK
Multifunctional paper electronics - Sustainable materials applied to flexible electronics
Rodrigo Martins, UniNova Lisbon, Portugal, President of E-MRS

10:40 am – 11:00 am
High resolution imaging of strain, crystal structure and defects by 2D and 3D X-ray diffraction
Tobias Schulli, ESRF Grenoble, France

Coffee break and Poster Session *(11:00 am – 11:30 am)*

Session II (11:30 am – 1:10 pm)
Chairs: *Robert Sinclair / Marco Sebastiani*

11:30 am – 11:50 am
Imaging structure transition and energy flow in 2-D and nano-scale materials with MeV electrons X-ray diffraction
Xijie Wang, SLAC, Stanford University, Palo Alto/CA, USA

11:50 am – 12:10 pm
Material science applications of cathodoluminescence in a STEM
Luiz Tizei, Universite Paris Sud, Orsay, France

12:10 pm – 12:30 pm
Switching mechanisms and reliability optimization of memristive devices derived from micro- and nanospectroscopy
Christoph Bäumer, Forschungszentrum Jülich, Germany

12:30 pm – 12:50 pm
Process control and physical failure analysis of FDSOI technology
Eckhard Langer, GLOBALFOUNDRIES, Dresden, Germany

12:50 pm – 1:10 pm
Bring synchrotron capabilities to individual labs: Results and possibilities
Wenbing Yun, Sigray, Concord/CA, USA

Lunch and Poster Session *(1:10 pm – 2:20 pm)*

Session III (2:20 pm – 4:00 pm)
Chairs: *Rodrigo Martins / Xijie Wang*

2:20 pm – 2:40 pm
ETEM study of the reduction of amorphous Molybdenum Sulphide catalysts
Robert Sinclair, Stanford University, Palo Alto/CA, USA

2:40 pm – 3:00 pm
Advanced materials modelling for batteries
Tanja Graf, Volkswagen, Wolfsburg, Germany

3:00 pm – 3:20 pm
Unravelling nanoscale phenomena with in-situ and operando small-angle scattering measurements
Sabrina Sartori, University of Oslo, Norway

3:20 pm – 3:40 pm
Nano-scale residual stress depth profiling by FIB-DIC ring-core method: Recent advances and applications to thin films
Marco Sebastiani, Universita Roma Tre, Italy

3:40 pm – 4:00 pm
Using TEM to study precipitates in age hardenable aluminium alloys
Randi Holmestad, NTNU Trondheim, Norway

Best poster award ceremony and closing remarks (4:00 pm – 4:15 pm)
Ehrenfried Zschech, Fraunhofer IKTS Dresden and Dresden University of Technology, Germany

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

Lab tours at Fraunhofer IKTS (4:15 pm – 5:30 pm)
Ehrenfried Zschech, Fraunhofer IKTS Dresden and Dresden University of Technology, Germany

POSTER SESSION
11:00 am – 11:30 am, 1:10 pm – 2:20 pm, 4:15 pm – 5:30 pm
Chair: Marco Sebastiani, Universita Roma Tre, Italy

Poster presentations

Abstracts
-Talks-

KEYNOTE TALK

Volkswagen: Right on schedule for E-mobility

Lars Dittert*

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We see a massive change in the automotive landscape, esp. at the Volkswagen Transparent Factory with the production of the e-Golf. The change in Dresden is just a first steps of a comprehensive shift. Over the next five years, the brand Volkswagen will be investing about €4 billion in the new MEB electric architecture at its plants around the world. An additional amount of about €2 billion is to be invested in development. The Zwickau plant – also in Saxony - is to be developed into the largest European e-mobility center. Initially, the series production of all vehicles based on the new modular electric drive kit (MEB) will be concentrated at the plant. The first vehicle in the new generation of electric cars is to be the I.D., which will be launched in 2020.

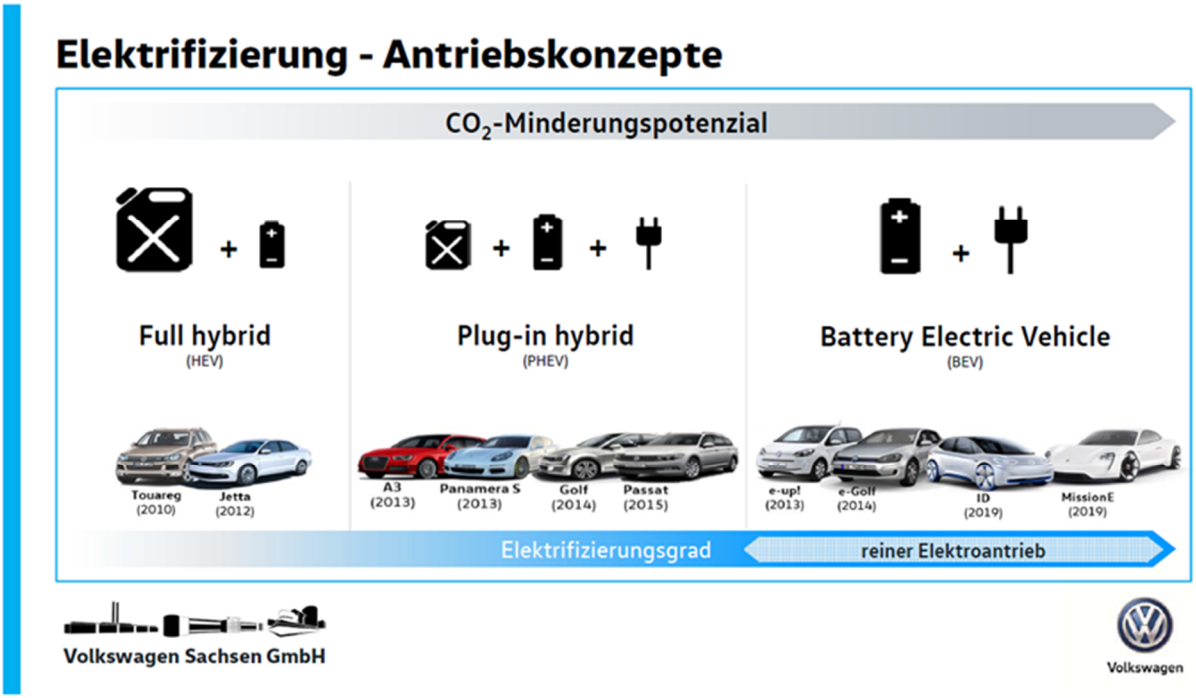

Figure 1. Development of E-mobility concepts at VW.

KEYNOTE TALK

Materials innovation for technology scaling and its implication to metrology

Zhiyong Ma*

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Continued technology scaling and miniaturization of low power semiconductor chips with ever increasing functionality in the past decade have been relentlessly driving research and development of new devices, materials, and process capabilities to meet performance, power and cost requirement. The evaluation and development of these new technology capabilities are alternately challenging and pushing the limits of existing characterization and metrology techniques and are fueling numerous innovations and advances across the metrology industry, measurement community, and academia in this field. As CMOS transistor scaling and interconnect RC scaling reach their respective physics and/or material limitations, some fundamental changes in alternative materials, device architecture, and process integration schemes are being considered and explored to extend CMOS technology to its ultimate limits. To overcome these limits, the emerging nano-scale technology beyond CMOS is also being actively researched as possible alternatives to continue technology scaling. Characterization and metrology are usually the first technology area to work routinely in the area of nanoelectronics and nanostructures. This is because a variation in feature size one tenth of the nominal dimension often results in significant variations in device properties. This will in turn significantly impact product level performance and reliability. In this respect, metrology provides indispensable measurement capability necessary for exploratory research, technology development, manufacturing control, and product improvement.

There are a few major technology trends placing unprecedented demand on metrology capability needs. Continued pitch scaling to improve transistor packing density requires measurement and control of dimensions of very thin films and interfaces down to atomic resolution. A major shift from 2D planar CMOS transistors to 3D FinFETs in the semiconductor industry for device performance and power improvement presents considerable challenges in all aspects of metrology. The complex 3D geometry of these nano-scale Fin structures already made some well-known analytical techniques inadequate as stand-alone techniques for measurement and quantification. These gaps are then amplified with the shrinking dimensions at each successive technology node. A hybrid metrology approach is often being sought after to extend these methods in combination with modeling and simulation to extract the actual 3D dimensional information.

This talk will discuss the technology scaling challenges and various considerations. It will also address the pressing needs for advancing metrology capabilities and their pivotal roles in enabling technology breakthrough and materials innovations.

KEYNOTE TALK

Multifunctional paper electronics

Rodrigo Martins*, Luís Pereira and Elvira Fortunato

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

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The use of disposable recyclable, eco-friendly, sustainable and low-cost devices with multiple functions is becoming a demand in the emerging area of the Internet of Things as a way to decrease the degree of complexity of the electronic circuits required to serve a plethora of applications. Moreover, for low-cost disposable applications, it is relevant the systems to be recyclable.

Printable electronics and flexible electronics are key areas of development worldwide, once offer the potential to add functionality to everyday objects at very low costs that would be difficult with conventional technologies. This was pushed by the large success of organic electronics over the past few decades due to their attractive features such as low process temperatures, good mechanical flexibility, light weight and the possibility to use a wide range of substrates and being recyclable. Besides that, we can prepare these devices using inexpensive solution processes over large areas. These benefits offered by printable and embedded electronics have being recognized in many sectors. Nevertheless, the bottleneck here is the low electronics performances so far achieved.

The idea beyond the present study concerns to exploit our imagination with simple questions such as: What happens if it was possible to have a simple and universal device architecture, easy to implement on paper substrates, but capable to provide different multiple functionalities? It would be possible to have a common template for electronic systems on paper that would be then easily customized depending on the final application? The present study answers to these challenges by reporting a multigate paper transistor where paper is simultaneously the substrate and the dielectric, while a metal-oxide-semiconductor (IGZO) is used as the active channel, being the same device able to present logic functionalities simply by varying the amplitude and frequency of the input gate signals. These transistors operate at drain voltages of 1 V with low power, exhibiting ION/IOFF > 10^4 and a mobility ≈ 2 $cm^2V^{-1}s^{-1}$, serving the specifications for a broad range of smart disposable low power electronics. To sustain all this study, an analytical compact model was developed able to precisely reproduce the response of paper dual-gate FETs and provide full understanding of their unique and innovative characteristics.

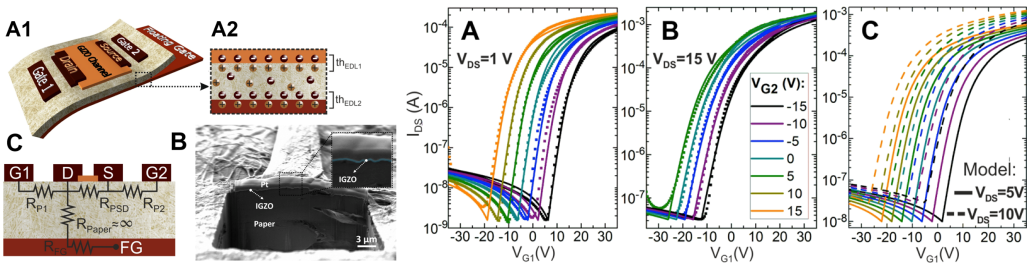

Figure 1. Device configuration, structure and current-voltage transfer characteristics of the PDG/BFG-FET.

Acknowledgments

This work was funded by European Projects NewFun (ERC-StG-2014, grant GA 640598); BET-EU (H2020-TWINN-2015.

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High resolution imaging of strain, crystal structure and defects by 2D and 3D X-ray diffraction

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X-ray diffraction and X-ray imaging have for one century mostly been regarded as two distinct applications of the same type of radiation. Traditionally X-ray diffraction is considered as a method with poor spatial resolution yielding only spatial averages as useful results. Very recent developments in the use of highly focused beams produced on the most advanced synchrotron sources show however a great and rapidly developing potential of diffraction imaging techniques. These are much improving the resolution of traditional X-ray imaging and topography but are as well combined with X-ray diffraction. In this way a new portfolio of techniques emerges, coupling the information of strain and texture with spatial information. As so far most of these new imaging techniques are brilliance limited they are naturally developed at synchrotrons. With the rapid development of the availability of synchrotron and in interaction with the very active user community in this field, new imaging techniques rapidly gain practically all fields of materials science and biomedicine. While X-ray optics typically limit today's practical resolution to about 50 nm, technological progress in this field, as well as the use of reconstruction techniques pave already the way towards nanometric resolution in space while preserving the structural information available through diffraction. With new source projects at the horizon these exciting imaging techniques will be established on a growing number of beamlines.

In the future, the field of diffraction imaging will supply unique information on the atomic structure of samples while preserving the operando capacity of X-rays with their penetration power and tolerance of sample environments. This is demonstrated by various projects that use these tools today to selectively image buried layers in devices or nanoparticles during catalytic reactions.

The Talk will present the state of the art of scanning and full field diffraction imaging tools with examples of recent application in the imaging of semiconductor devices and applied materials.

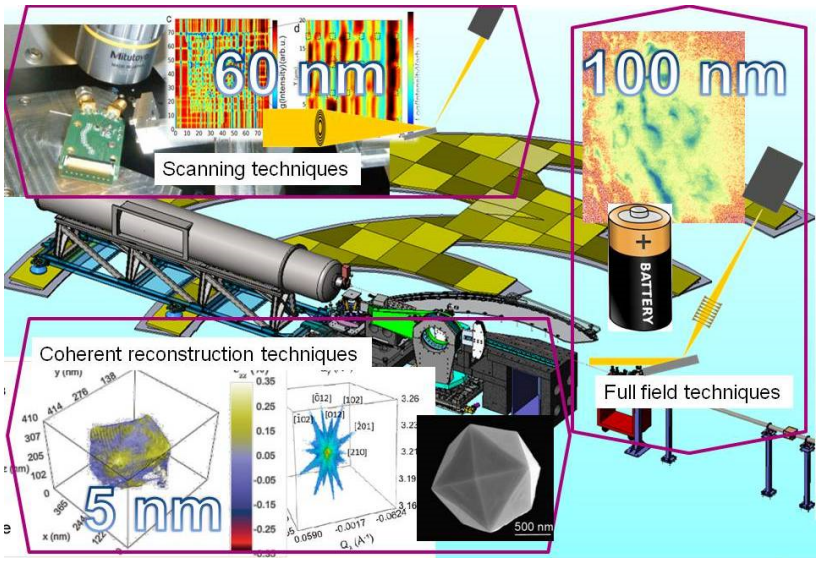

Figure 1: Bragg diffraction combined with imaging supplies techniques with different spatial and temporal resolution and can tolerate sample packaging or complex sample environments.

Imaging structure transition and energy flow in 2-D and Nano-Scale materials with MeV electrons

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Future electronic devices will be 3-dimensional heterostructures with 2-D or nano-scale materials where new and exciting properties emerge, such as superior mechanical strength and an extraordinary ability to conduct electricity and heat. Understanding these 2-D and nano-scale materials how to acquire their unique characteristics and the energy flow in the heterostructures pose severe challenges for the device design. Therefore, understanding of the fundamental interactions that determine non-equilibrium energy flow, such as energy relaxation and dissipation in such systems is of great importance. Ultrafast MeV Electron diffraction (MeV-UED) [1, 2] provides unique capabilities for imaging structure transition and energy flow in 2-D and nano-scale materials. The functionality of 2-D materials critically depends on how their atoms move, and the atomic spatial-temporal resolution demonstrated by MeV-UED, made it a reality [3, 4]. The longer mean free path of MeV-UED greatly simplified the experimental studied of 3-dimensional heterostructures, such as through precise measurements of the transient Debye-Waller-factor, the mean-square atomic displacement is directly determined, which allows to quantitatively follow the temporal evolution of the lattice temperature after short pulse laser excitation [5, 6].

References

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Material science applications of Cathodoluminescence in a STEM

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Optical spectroscopy is traditionally performed using photon excitation (with a laser beam, for example). Typical emission experiments allow one to gather information of the different energy levels available in the system by measuring the intensity of the emitted light as a function of wavelength/energy. In a confocal optical microscopy setup, the ultimate spatial resolution is limited by the size of the detection volume (of the order of half a micrometer).

In this contribution, we will discuss the benefits and difficulties of performing optical spectroscopy using fast electrons as the excitation source (either in emission, using cathodoluminescence, [1, 2], or in absorption, by measuring electron energy loss, [3]). Much higher spatial resolution (from tens of nanometers to below a nanometer) is the evident attraction. Indeed, advances in electron optics (aberration correctors, monochromators and spectrometers) and improvements in electron microscope instrumentation (stages with cryogenic temperatures, including light collection systems) have brought electron spectroscopies closer to the energy resolution and detection sensitivity of standard optical spectroscopies. To start with, we will describe cathodoluminescence (light emission from a material excited by electrons) experiments aimed at understanding exciton physics in two different systems: GaN quantum disks in AlN nanowires and hBN flakes. In Figure 1, an example of what is currently possible for AlN/GaN nanowires is presented. In addition to standard spectral analysis, cathodoluminescence coupled with light intensity interferometry allows the detection of single photon sources [4]. This capability allowed the detection of single photon sources in hBN thin layers [5].

Finally, we will present our latest results from monochromated electron energy loss experiments using the ChromaTEM microscope [6] for different materials.

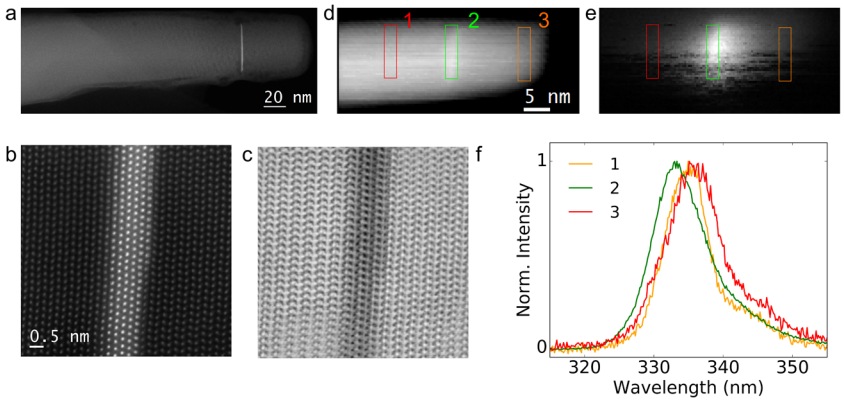

Figure 1. a-c) High angle annular dark field and annular bright field images of a 1.5 nm wide GaN quantum disk in a AlN nanowire. d) Annular dark field image of the same nanowire acquired at the same time as a spectrum image acquisition. e) Total light intensity emitted integrated around the emission peak. f) Normalized spectra averaged from the spectrum image in the regions marked 1, 2 and 3. A wavelength shift of 2 nm (22 meV) is observed due to the quantum confined Stark effect.

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Switching mechanisms and reliability optimization of memristive devices derived from micro- and nanospectroscopy

Christoph Bäumer[*1,2,3], David Cooper[4], Carsten Funck[3], Stephan Menzel[1], Claus M. Schneider[1], Rainer Waser[1,3] and Regina Dittmann[1]

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Memristive devices based on resistive switching in transition metal oxides are attractive candidates for next-generation non-volatile memory applications. Direct observation and quantification of switching and failure mechanisms, however, remain challenging because the suspected net changes of structure and stoichiometry during switching are very small and occur primarily at electrode interfaces or within nanoscale filaments.

Here we will present local changes in the chemical and electronic structure of memristive devices utilizing *operando* characterization tools like transmission electron microscopy (TEM) and photoemission electron microscopy (PEEM). To overcome the surface sensitivity typically limiting PEEM investigations of memristive devices, we used photoelectron-transparent graphene top electrodes to attain spectroscopic information from the buried active layer [1]. Combining TEM, PEEM, and single band transport theory, we can derive a complete and quantitative description of the nanoscale switching processes based on a reversible change of the oxygen vacancy concentration in the conductive filament bridging the active layer (Figure 1) [1, 2].

Similarly, PEEM investigation reveals the most important failure mechanisms in memristive devices: The so-called cycle-to-cycle variability results from changes of the fine structure of the conductive filament or even a change in its location [3]. The retention failure, i.e., the loss of information over time, is caused by reoxidation of the conductive filament [4]. Based on these insights, we can derive a rational design rule to prevent retention failure through the incorporation of retention-stabilization layers with slow oxygen transport properties.

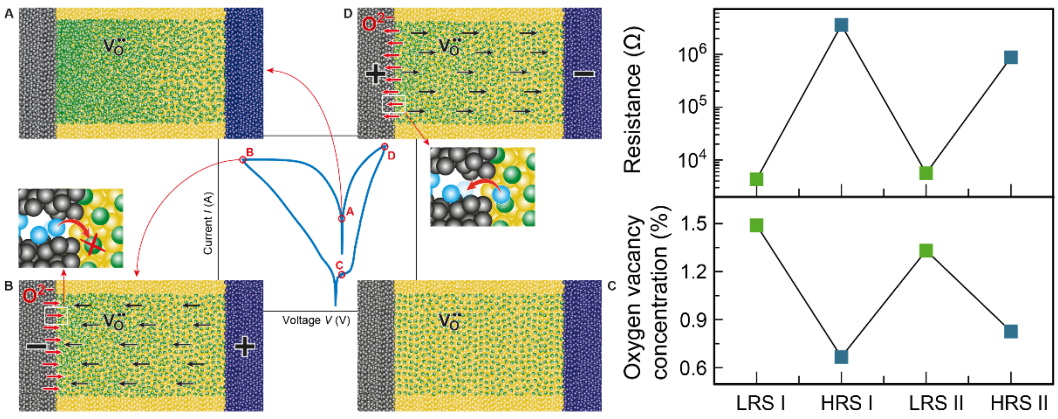

Figure 1. Left: Proposed mechanism for resistive switching through oxygen evolution and reincorporation of oxygen. Oxygen vacancies (green circles) in a matrix of stoichiometric $SrTiO_3$ (yellow circles) sandwiched between a Pt top electrode (gray circles) and a Nb:$SrTiO_3$ bottom electrode (blue circles). Right: Resistance variation correlated to the oxygen vacancy concentration in the conductive filament. Modified from [1, 2].

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Process control and physical failure analysis of FDSOI technology

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With the development and productization of FDSOI technology at the 12 and 22nm node process control and failure analysis at Globalfoundries are facing new challenges, since especially FEOL processing steps differ substantially from conventional technology integration schemes. FDSOI offers an excellent combination of performance, power consumption and cost for IoT, mainstream mobile, RF connectivity and networking applications. This is realized by FinFET-like performance and much improved energy-efficiency at a cost comparable to 28nm planar technologies.

From process control perspective several process steps need either new approaches or adapted methods to measure critical parameters. As the performance of the FDSOI transistors is boosted by strain engineering, it is crucial to measure the local strain state in the transistor channel in correlation to local layout features. The transistor channel region itself has thickness of some nm only. Examples on how to measure the local strain state in single transistors with TEM precession diffraction technique and how to measure overall strain state in transistor arrays with High Resolution XRD will be shown. It is inherent to FDSOI technologies that S/D and halo implant processes are skipped. Instead, the S/D dopant species are introduced by in situ-doped epitaxial deposition processes, The S/D connection to the transistor channel is still crucial for performance tuning. So far inline measurements and modelling cannot cover all parameters of interest reliably surface analysis methods have to be applied in out-of-FAB laboratories in order to get correct data about film thicknesses and dopant concentrations. Methods like TOF-SIMS, VASE and XRD are used for this purpose and their applications will be shown.

From the perspective of failure analysis, the specifics of FDSOI imply some technical challenges, especially for electrical measurements by Nanoprobing. The active transistors are completely isolated from the bulk material. Hence, SEM-based in-situ probing needs to be carried out with great caution since SEM charging leads to parameter drift of transistor. Best practices on how to minimize the effects will be presented and examples for root cause analyses for FDSOI specific failure modes will be shown.

Bringing synchrotron capabilities to individual laboratories: Results and possibilities

Wenbing Yun*, Benjamin Stripe, SH Lau, Srivatsan Seshadri, Alan Lyon, David Vine, and Sylvia Lewis

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X-ray techniques have grown in popularity over the past decade. Owing to the high penetrating power and non-destructive capabilities of X-ray radiation, these techniques have opened new frontiers for material characterization. In spite of these advantages, however, conventional laboratory X-ray instrumentation is inherently limited by the brightness of the illuminating x-rays, which, in turn, limits the detection sensitivity and spatial resolution of all x-ray analytical equipment, including x-ray absorption spectrometry (XAS), micro x-ray fluorescence (microXRF), and nano 3D x-ray microscopy (XRM).

Recent advances to x-ray source and x-ray optic technology are now enabling capabilities that were previously exclusively available to synchrotron beamlines. We will first discuss several innovations, most notably a novel microstructured x-ray source that enables substantially higher brightness and energy tunability. Furthermore, new advances in x-ray mirror lens fabrication processes are now capable of producing optics with substantially higher efficiency and resolution.

Several powerful laboratory x-ray systems have been enabled by the source and optics developments, including: the Sigray QuantumLeap: an XAS system that enables high throughput determination of chemical state information for element(s) of interest (e.g. oxidation state, local atomic geometry, bond lengths, etc.) and furthermore provides micro-XANES capabilities at 100 um resolution; the Sigray AttoMap: a microXRF system with the ability to measure nm-thick thin films and sub-ppm sensitivity for chemical migration/contamination and compositional imaging; and the Sigray NanoXRM: the highest resolution 3D x-ray microscope with multi-energy capabilities and 40 nm resolution.

Possible applications of such techniques relevant to the automotive research community include: visualization of the nanostructure of polymers such as carbon black with 3D nano x-ray microscopy, microXRF analysis of dopants and thin films in LIDAR technology for self-driving vehicles and inclusions in PET foils, XAS oxidation state determination of transition metals for batteries *in operando*, chemical analysis of welds, and more.

ETEM study of the reduction of amorphous molybdenum sulphide catalysts

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Amorphous MoS_X is a highly active, earth abundant catalyst for the electrochemical hydrogen evolution reaction (HER). Previous studies have revealed that this material initially has a composition of MoS_3, but after electrochemical activation, the surface is reduced to form an active phase resembling MoS_2 in composition and chemical state. However, structural changes in the MoS_X catalyst and the mechanism of the activation process remain poorly understood. In this study, we employ aberration-corrected transmission electron microscopy (TEM) to image amorphous MoS_X catalysts activated under two hydrogen-rich conditions: ex situ in an electrochemical cell and in situ in an environmental TEM (ETEM). For the first time, we directly observe the formation of crystalline domains in the MoS_X catalyst after both activation procedures as well as spatially-localized changes in the chemical state detected via electron energy loss spectroscopy (EELS). It is found that the presence of hydrogen is critical for enabling the restructuring process. Our results suggest that the surface of the amorphous MoS_X catalyst is dynamic: while the initial catalyst activation forms the primary active surface of amorphous MoS_2, continued transformation to the crystalline phase during electrochemical operation could contribute to catalyst deactivation. These results have important implications for the application of this highly active electrocatalyst for sustainable H_2 generation. [1] The role and utility of the environmental TEM will also be discussed.

Acknowledgments

Financial support from the Toyota Research Institute – Accelerated Materials Design and Discovery program (Stanford University) is much appreciated.

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Advanced materials modelling for batteries

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The automotive industry is facing major challenges. Sustainable mobility will strongly rely on the successful design and implementation of advanced energy materials. Furthermore, future mobility concepts, such as automated driving, car-to-car communication and digitalization will require the use of high-performance, energy efficient and reliable materials. Therefore, making smart materials choices will be essential to meet these challenges.

Modern computational methods combined with experimental analytics can accelerate materials development by reducing trial-and-error and allow for the design of materials with targeted properties for a specific application.

Here, a fundamental understanding of the potential and the limitations of existing materials as well as the possibility to design new materials will be the keys to accelerate material innovation and guarantee competitiveness of future products.

Unravelling nanoscale phenomena with in-situ and operando small-angle scattering measurements

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The synthesis of nano-sized particles infiltrated in porous carbon scaffolds has being investigated as a way to improve the thermodynamic and kinetic properties of metal hydrides for hydrogen storage applications.

With small-angle neutron scattering (SANS) we demonstrated the successful wet or melt infiltration of $Mg(^{11}BD_4)_2$, $NaAlD_4$, MgD_2 and a mixture of $Li^{11}BD_4$-$Mg(^{11}BD_4)_2$ into nano-carbon templates [1-4]. Depending on the hydride and/or the scaffold used, the particle sizes were found to range from 1 to 6 nm. Combining SANS and *in situ* small-angle X-ray scattering (SAXS), it was possible to underline important differences in the morphology and surface area of the hydride particles during heating when they are confined in the nano-porous scaffolds, compared to their values in the bulk state. Kinetic and thermodynamic effects due to the nano-confinement will be discussed in view of the application of these materials for vehicular hydrogen storage.

We will also show that small-angle scattering can highlight new mechanisms of reactions otherwise undetected for the investigation of batteries. We report an innovative *operando* measurements with the simultaneous acquisition of small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) on a high-capacity amorphous anode for Na-ion batteries during charge/discharge cycles [5]. The advantage of this study lies in the possibility to monitor both particle nanoscale effects and phase transformation over a large range, without disassembling the battery.

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Nano-scale residual stress depth profiling by FIB-DIC ring-core method: R ecent advances and applications to thin films

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Analysis and control of residual stresses in advanced engineering materials are important issues for reliability assessment at small scales, e.g. for micro-electromechanical systems (MEMS) and nano-crystalline and amorphous bulk and thin film materials. This presentation gives an overview of the recent advances in the field of sub-micron scale residual stress assessment by the use of focused ion beam (FIB)-controlled material removal techniques.

Materials and The two step method consists of incremental FIB ring-core milling combined with high-resolution in-situ SEMFEG imaging of the relaxing surface and a full field strain analysis by digital image correlation (DIC).

In this presentation, we present and validate a novel non-integral method for residual stress depth profiling in coatings, which overcomes many limitations of the current state-of-the-art and possesses several important advantages. It obviates the need for matrix calculation, equipping the user with efficient closed form solution.

In addition, we will review the most recent advances in the field of FIB-DIC methods for residual stress assessment at the micro and nano scales, with focus on recent efforts for development of automated procedures for local residual stress analysis of thin films and coatings.

Practical applications of the method on several systems will be described and discussed. In particular, the issues of residual stress assessment on very thin films and micro-devices, stress depth profiling, stress measurement on amorphous materials and the effects of ion induced damage and elastic anisotropy on the relaxation strains will be reviewed.

Using TEM to study precipitates in age hardenable aluminium alloys

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Age hardenable aluminium alloys are important structural materials for automotive applications due to their high strength/weight ratio and good formability, often combined with good corrosion resistance. These properties are given by numerous nano-sized metastable precipitates, which form in the Al matrix during heat treatment and mechanical processing. Our research group at NTNU and SINTEF in Trondheim has over a long period worked together with Norwegian light metal industry on the studies of precipitation in Al-Mg-Si(-Cu) alloys. Our objective is to acquire a deeper understanding of the fundamental physics governing at the atomic scale, which controls nucleation, phase stability and precipitation development. By understanding this evolvement, 'alloy design' can be used to tailor materials to desired properties.

We are using advanced transmission electron microscopy (TEM) based techniques such as scanning precession electron diffraction (SPED) [1, 2], aberration-corrected scanning transmission electron microscopy (STEM) [1, 3, 4], image distortion correction for strain analysis [4] and atomically resolved electron dispersive spectroscopy (EDS) [5]. Recently, we have also studied deformation behavior of these materials, focusing on precipitation free zones [2]. In this presentation, I will show some recent examples of work in the group illustrating how advanced TEM methods are used to acquire information about precipitates and how this connects to properties like hardness, strength and corrosion.

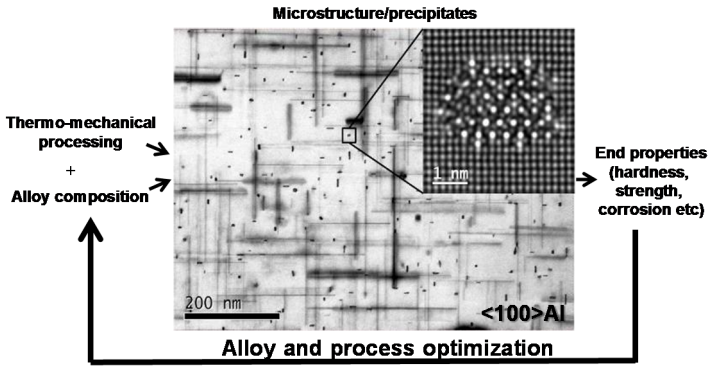

Figure 1. Alloy composition, heat treatment and mechanical processing determines the microstructure giving the alloys properties.

Acknowledgments

We want to acknowledge the Norwegian Research council and Norwegian aluminum companies for support through the projects CASA (no 237885), FICAL (no 247598) and AMPERE (no 247783). TEM work has been carried out at the NORTEM infrastructure (no 197405) in Trondheim, Norway.

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Abstracts
-Posters-

Structure and properties of iron based amorphous ribbon after laser interference irradiation

P01

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Laser interference heating applied to induce crystallization iron based amorphous ribbon was investigated. Such treatment allowed to form two-Dimensional (D) micro-islands of laser-affected material periodically distributed in amorphous matrix. Comparing structure with sample after annealing at 600°C indicates to different type of crystallization during laser heating and annealing. Scanning and electron microscopy indicate to occurrence crystalline structure in amorphous matrix after laser interference irradiation but after annealing were observed dendritic structure. Magnetic properties were determined by magnetic hysteresis loop measurement and magnetic force microscopy. As-cast ribbon and laser heated sample are magnetically soft materials [1-3].

Acknowledgments

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III-V nanostructures for optoelectronic applications: a spectroscopic study

P02

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Since the successful development of quantum well lasers in the 1970s, one of the richest areas of application of semiconductor nanostructures is the area of optoelectronics, such as lasers and detectors [1]. The most widely used semiconductors for optoelectronic applications are the compounds formed by group III and group V elements. For instance, GaAs and related compounds are mostly used for optical fiber communications, near infra-red and visible light emitting diodes (LEDs) as well as laser diodes. While, GaN and AlGaN are used for LEDs, solid-state lasers and color displays [2], in the short wavelength range. Furthermore, optoelectronic devices based on novel semiconductor nanostructures are foreseen to revolutionize nowadays technology in terms of superior performance and efficiency, as well as the reduction of costs and material consumption.

The use of semiconductor nanostructures integrated in devices is governed by the realization and control of p-n junctions, obtained by p- and n-type doping of the base materials. In addition to n and p-type doping, the semiconductor nanostructure emission can be tuned by incorporating rare earth ions by implantation and post-growth annealing. Doping can be performed by intentionally adding dopants during the growth (*in-situ*) or after the growth (*ex-situ*) by diffusion and ion implantation. However, several issues and controversies still need to be investigated in order to access/control semiconductor properties at the nanoscale level.

In this work, we present our recent research on semiconductor nanostructures such as: *i)* AlxGa1-xN $(0 \leq x \leq 1)$ NWs grown by molecular beam epitaxy on Si (111) substrate implanted with europium (Eu) ions and subject to rapid thermal annealing, *ii)* AlGaN/GaN superlattice-based diode structures grown by metalorganic chemical vapor deposition implanted with two Eu fluences and subject to high temperature and high pressure annealing [3], and *iii)* silicon-doped GaAs NWs with four nominal silicon doping levels [4]. The optical and vibrational properties of these nanostructures, of utmost importance for optoelectronic applications, will be assessed mainly using contactless spectroscopy techniques such as: photoluminescence, photoluminescence excitation and micro-Raman spectroscopy.

Acknowledgments

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Optically active borate glasses doped with cerium oxide

P03

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Cerium doped borate glasses have been prepared from the BaO-B2O_3 system. The effect of CeO_2 admixture on thermal stability of the glass was investigated. Thermoluminescence (TL) and optically stimulated luminescence (OSL) properties, such as glow curves shape and linearity of dose-response are presented. Results showed that the increase of cerium dopant concentration from 1 to 5 mol% caused the efficiency enhancement of TL by more than ten times and OSL by more than one hundred times. Based on the XRD measurements we can state that a new phase of $BaCeB_9O_{16}$ has been obtained which is not present in the structural databases. This phase is isostructural to $BaLaB_9O_{16}$. Controlled crystallization of cerium doped borate glasses revealed that appropriate process of heat-treatment can enhance the intensity of TL process.

Acknowledgments

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CVD graphene for broadband third-harmonic generation

P04

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Among the many fascinating physical properties of graphene, its third-order optical susceptibility is particularly interesting, being 10^8 times larger than in a regular insulator. This allows for broadband third-harmonic generation [1], which in turn enables new exciting applications, such as the use of graphene coatings (single or multi-layer) for characterization of ultrashort laser pulses [2]. Thus, this work explores graphene grown by different synthesis techniques towards the aforementioned application, focusing on their characterization in order to better understand the influence of its morphology on third-harmonic generation.

The first type of graphene was grown by thermal CVD (TCVD). It was confirmed, by Raman spectroscopy, SEM and optical microscopy, to be a single-layer graphene film with few-layer islands (non-coalesced domains of secondary layers). The growth parameters were varied in order achieve different sizes and distributions of these islands in order to study their impact on third-harmonic generation. In particular, pulsed introduction of methane into the CVD chamber was explored. The effects of different methane flow rates and growth times were also studied. Lastly, the influence of the copper substrates used in the synthesis of graphene on the size and distribution of the few-layer islands was investigated.

The second type of graphene was grown by microwave plasma CVD (MPCVD). These samples consist of up to 8 layers, as revealed by HRTEM. The graphene displayed Raman spectra characteristic of single-layer graphene, due to a weak interaction between the constituting layers, offering an opportunity to ascertain the importance of interlayer interaction for third-harmonic generation. Different growth times were evaluated with regards to the influence of this parameter on the resulting films' structure and, consequently, on the third-harmonic generation.

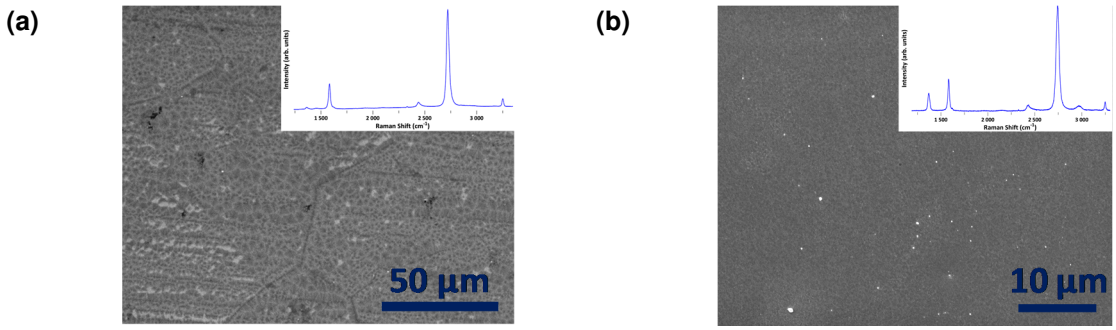

Figure 1. (a) SEM image and a Raman spectrum of TCVD graphene. (b) SEM image and Raman spectrum of MPCVD graphene.

Acknowledgments

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Influence of Gd_2O_3 nanoparticles on the oxidation resistance of Fe-16Cr steel

P05

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In recent years, there has been a trend towards the development of technologies that allow the storage of surplus electricity. The tool that seems to be the most suitable for this purpose is the solid oxide electrolytic cell (SOEC), which utilizes electrolysis to effectively convert a surplus of electricity into fuel. One of the key components of the SOEC is the interconnect. It allows single cells to be connected in series to form stacks, and also supports the entire structure, giving it the necessary rigidity, while the channels located on both of its sides allow gas reagents to be transported to the cathode and the anode. Metallic materials that meet most requirements set for SOEC interconnects include ferritic stainless steels (FSS) with a Cr content of 22-25 wt%. It should, however, be emphasized that all metallic interconnects have a common disadvantage, which is susceptibility to high-temperature oxidation. This process leads to the formation of a chromium (III) oxide scale on the surface of the ferritic steel. The thickness of this scale grows with the operating time of the electrolyzer. As a result, the area-specific resistance of the interconnect (ASR) continuously increases. Another undesirable effect associated with the formation of the Cr_2O_3 scale is its tendency to react with the reagents that surround the system during operation. This leads to the formation of volatile compounds of chromium, which can subsequently react with the anode and cathode materials, causing their catalytic properties to deteriorate. In order to reduce the impact of these adverse phenomena, the physicochemical properties of ferritic steels undergo certain modifications that can generally be divided into two types. One approach is to develop new ferritic steels with improved oxidation resistance and the second is the deposition of protective-conducting coatings on the surface of FSSs. Regardless of the way in which a steel is modified, the costs of manufacturing a steel/coating layered system that is suitable for the construction of a SOEC stack remain high due to the persistently high prices of the steel substrates. An answer to this problem is the application of significantly less expensive commercially available ferritic steels with a chromium content of up to 17 wt%. However, such steels undergo very rapid high-temperature oxidation in oxidizing and reducing conditions. One of the ways in which the growth rate of the chromia layer can be reduced is inducing the so-called reactive element effect (REE). The addition of a small amount of elements such as Y, La, Gd, or their oxides, improves the adhesion of the Cr_2O_3 scale to the metallic core and, in addition, reduces its growth rate.

Consequently, the objective of the presented work was to investigate the influence of coating the Nirosta 4010/1.4010 ferritic steel with a 16.3 wt% Cr content with gadolinium nanoparticles on its oxidation resistance. Two series of samples were prepared. In the case of the first one, Gd_2O_3 nanoparticles were deposited via electrolysis, while for the other one they were deposited by means of dip-coating. In the electrolytic method, the surface of the samples was modified using an electrolyte consisting of a gadolinium nitrate solution with a concentration of 0.01 M. The deposition of coatings via dip-coating involved the immersion of the samples in a gadolinium nitrate bath with the same concentration as in the case of electrolysis for 30 s. Gadolinium deposition via dip-coating was performed three times in total. Both series of samples were heated at 400°C for 30 min in order to thermally decompose the hydroxides and nitrates. During this process, a layer consisting of gadolinium oxide nanoparticles formed on the surface of the material. An oxidation study was performed for 100 hours under isothermal conditions in air at the following temperatures: 700, 750, 800 and 850°C. Furthermore, morphological observations and chemical and phase composition analyses of the oxidation products were performed using X-ray diffraction (XRD) and scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDS).

The conducted oxidation studies of the unmodified steel and the steel samples modified with Gd_2O_3 revealed a reduction in the mass gain observed after modifying the investigated steel and improved adhesion of the scale to the metallic substrate. They also demonstrated that both applied methods are suitable ways of modifying ferritic stainless steel materials intended for the manufacture of interconnects applied in planar SOEC devices.

Effect of copper addition on microstructure and martensitic transformation temperature in Ni-Mn-Ga Heusler alloys

P06

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The Ni-Mn-Ga alloys have attracted considerable attention during the past few decades due, to their functional properties such as: giant magnetic field-induce strain effect. These alloys are very promising candidates for actuator and sensor applications. It is well-known, that the martensitic transformation temperature is very sensitive to the chemical composition [1]. The main drawbacks for industrial application of this alloy are brittleness and low operating temperature. This has led to the search for new alloy compositions with enhanced functional properties. For instance, addition of a fourth ductile element, such as Cu, into ternary Ni-Mn-Ga system may improve ductility and simultaneously bring the martensitic transformation temperature closer to room temperature. Therefore, this contribution investigates the effect of substitution of Cu for Ga in $Ni_{50}Mn_{25}Ga_{25-x}Cu_x$ (x = 1 - 10 at.%) alloy on martenisitic and magnetic transformation temperatures and the crystal structure. Polycrystalline samples were produced by arc-melting from high purity elements and thereafter re-melted four times under argon atmosphere to encourage chemical homogeneity. The buttons were encapsulated under vacuum in quartz ampoules homogenization for 24h at 1173 K and then slowely cooled to ambient temperature. Then, the samples were cut into two piece. One part was again encapsulated in quartz ampoules heated at 1173 K for 30 minutes and then water quenched. The final chemical composition of alloys were checked by an energy-dispersive spectrometer attached to a scanning electron microscope. The crystal structures were analyzed by x-ray diffraction technique at ambient temperature. Four types of crystal structure were detected: L21 austenite and 5-layered modulated, 7-layered modulated and non-modulated martensite. The substitution of Ga for Cu in the Ni50Mn25Ga25-xCux system results in an increase of martensitic transformation temperature (TM) which in turn is proportional to the e/a ratio. Electron microscopy observations revealed that the Cu addition significantly affects the martensite microstructure, as shown in Figure 1. At higher Cu concentration, i.e. 9 and 10 at.%, a so-called γ phase precipitates of face-centred cubic crystal structure form being substantially enriched with Cu.

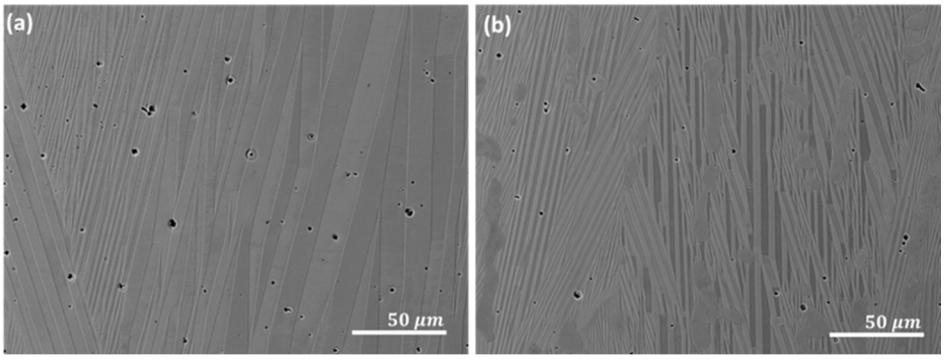

Figure 1. SEM images of furnace cooled samples: (a) microstructure of single martensite phase of $Ni_{50}Mn_{25}Ga_{17}Cu_8$ alloy and (b) a dual-phase of $Ni_{50}Mn_{25}Ga_{16}Cu_9$ alloy with the second phase as a precipitate.

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Stress induced mechano-electrical writing/reading of a polymer film through atomic force microscopy based contact electrification

P07

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Mechano-electrical writing and reading in polyaniline (PANI) thin films are demonstrated via a metal-polymer contact electrification mechanism (CEM). We present an innovative conception for a non-destructive self-powered writable-readable data sheet, which can pave the way towards new types of stress induced current harvesting devices. A localized force deformation of the interface has been produced by pressing atomic force microscope probes against the polymer surface, allowing for charge transfer between the materials' interface. The process yields a well-defined charge pattern by transmuting mechanical stress into readable information. The average output current rises from 0.5 nA to 15 nA as the applied force - not electrical bias - is raised from 2 nN up to 14 nN, and is also correlated with the adhesive force of different PANI film regions, as demonstrated through conductive-force spectroscopy mapping. These results underscore the importance of stress-induced current harvesting mechanisms and charge patterning of polymer surfaces, which can possibly be scaled up towards writable-readable data sheets. A time evolution current distribution study (TECD) of the stress-induced patterned PANI surface shows the response and readability of the recorded data with time.

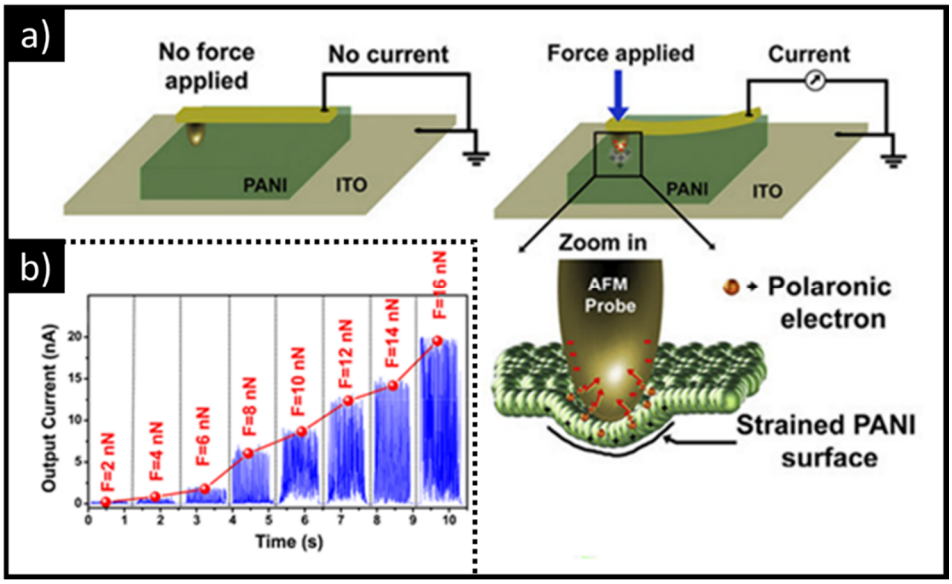

Figure 1. a) Schematic of the experiments based on CEM, demonstrating charge generation in the presence of an applied force and illustrating localized electron transfer at the metal-polymer (Probe-PANI) interface. b) Progressively applying higher forces at the metal-polymer interface produces increasing current.

Acknowledgments

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Piezoresponse Force Microscopy and Electron Backscattering Diffraction of 90° Ferroelectric Twins in $BaTiO_3$ Positive Temperature Co-efficient (PTC) Thermistors

P08

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Donor doped barium titanate ($BaTiO_3$) is known as a smart material that possesses unique thermo-sensitive electrical properties with huge applications in over-voltage protection, automobiles, hair-dryers and self-regulating heaters. $BaTiO_3$ is a ferroelectric insulator at room temperature; however, the materials can become a ferroelectric semiconductor by suitable doping with ions La^{+3}, Sm^{+3}, Ho^{+3} or Nb^{+5}. The semiconducting property in ferroelectric $BaTiO_3$ gives rise to a huge resistivity change at the ferroelectric-paraelectric transition temperature with a positive temperature coefficient of the resistivity (PTCR) [1, 2]. Here, we combine two surface analysis tools, electron backscatter diffraction (EBSD) and piezoresponse force microscopy (PFM) to investigate the crystallographic orientation, topography and intergranular polarization in polycrystalline PTC $BaTiO_3$ ceramic that leads to PTC effect. EBSD of $BaTiO_3$ reveals individual grains of $BaTiO_3$ possess a preferred orientation. Ferroelectric domains and twinning is evident in both electron back scattered images and PFM images. In individual grains, the domains mostly appear in a single twin pair set rather than random pairs of all available phase variants. While the EBSD on these thermally etched samples showed 90° domains it could not discern the type between a-a and a-c domains. PFM on the other hand clearly distinguishes a-a and a-c type domains using vertical and lateral piezoresponse. The work reported here will contribute to grain boundary control of PTC effect in semiconducting ferroelectric barium titanate ceramics.

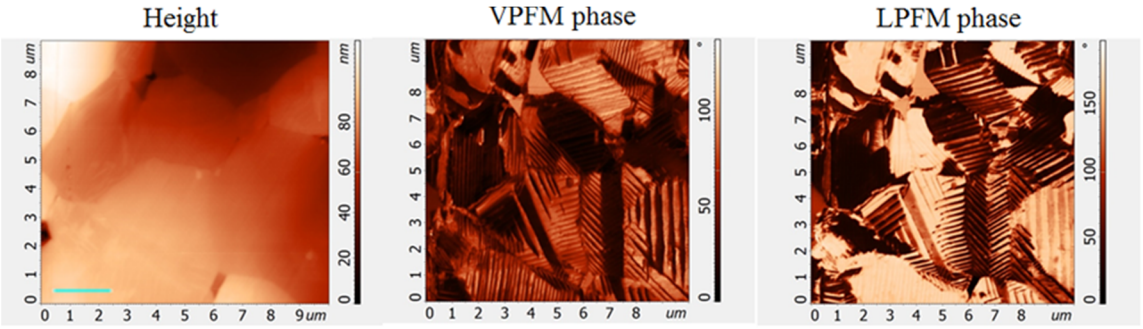

Figure1: PFM images of $BaTiO_3$ showing (a) height, (b) vertical piezoresponse VPFM (phase) and (c) lateral piezoresponse LPFM (phase) of the same area. A clear herringbone pattern is evident on most of the grains (b, c), indicating that the domains are ferroelectric in nature.

Acknowledgments

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The influence of crystallographic orientation and precipitates on the superelasticity of Fe-based shape memory alloys

P09

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Shape memory alloys are one of the most important groups of functional materials. Particularly the thermoelastic martensitic transformation in FeNiCoAlTaB alloy, discovered by Tanaka et al. [1, 2] is of significant importance. In this work the global and local orientation measurements were performed by diffraction of high-energy synchrotron radiation and electron backscatter diffraction. In order to obtain optimal mechanical properties the effect of heat treatment on the precipitation hardening in multicomponent single crystalline materials was studied. Heat treatment with variable annealing time (0.5h, 1h, 5h, 10h, 24h) at 973 K was performed on single crystals with <100> and <111> orientations. Employing the synchrotron diffraction three different intermetallic phases such as NiAl, Ni_3Al and $NiAl_3$ were analyzed. Subsequently, single crystals with <100>, <110>, <111> and <112> orientations were compressed at different temperatures (77, 123, and 295 K) to provide an insight into the mechanism of superelasticity observed in these alloys. Elastic, elasto-plastic and plastic response, has been observed depending on the orientation and deformation temperature. The results are discussed with respect to crystallographic orientation, deformation mode, precipitations and phase transformation.

Acknowledgments

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Office paper decorated with silver nanostars - an alternative cost-effective platform for trace analyte detection by SERS

P10

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Surface Enhanced Raman Spectroscopy (SERS) is a technique based on light scattering by analytes near plasmonic nanostructures able to perform highly sensitive detection. For analytical point-of-need applications portable sensors such as low-cost SERS substrates using paper as a base, are an alternative. In this work, SERS substrates were produced on two different types of paper: a high porosity paper (filter paper); and a low porosity paper (office paper). Solutions containing spherical silver nanoparticles (AgNPs) and silver nanostars (AgNSs) were separately drop-casted on hydrophilic wells patterned on the papers. The AgNP and AgNS distribution along the paper fibres was conditionate by the porosity of the paper, with most of the nanoparticles being retained at the illuminated surface of the office paper substrate. The paper-derived fluorescence on both papers was concealed when treated with NPs. Also, to obtain in a simple manner a highly sensitive SERS-active substrate, paper-induced aggregation of AgNPs was found to be a viable alternative to the classical salt-induced aggregation. A limit of detection for rhodamine-6G as low as 11.4±0.2pg could be achieved, with an analytical enhancement factor of $\approx 10^7$ on the office paper substrate with deposition of AgNS. Not only this well patterning technique allowed a good uniformity of signal inside the wells, but also contributed to a good reproducibility when different AgNSs synthesis batches were tested (RSD of 1.7%). Besides, these SERS substrates remained highly stable after 5 weeks of storage (RSD of 7.3%) without any type of encapsulation/protection [1].

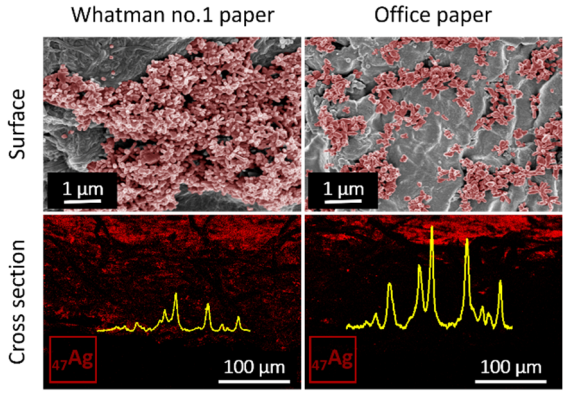

Figure 1. SEM images of AgNSs in Whatman no.1 and office paper. AgNSs distribution on Whatman no.1 and office paper leads to higher signals from office paper SERS substrates.

Acknowledgments

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Measurement of the temperature dependence of lattice deformations in silicon using Raman microscopy

P11

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Processing of silicon in microelectronics, photovoltaics and micromechanics includes thermal and mechanical processing that can lead to a change in the silicon lattice, such as phase transitions or lattice deformations which have a crucial impact on the mechanical properties and the chemical resistance of silicon and on the failures of finally processed devices [1, 2]. In particular thermal treatment is used to relax lattice deformations and to recrystallize silicon phases [3]. However, these are complex processes and proceeds, according to our study presented here, individually for different components.
We used a mechanical treated silicon wafer (as-cut diamond wire sawn, multicrystalline) and studied the state of the silicon lattice before, during and after different annealing programs with temperatures up to 900°C with Raman microscopy. Special emphasize was given to the local identification of tensile and compressive lattice deformations and its deformation strength next to high pressure phases extracted from the deconvolution of Raman spectra.

Diamond structured silicon lattices with compressive deformations relaxes at around 400 to 500°C during the heating phase, while lattices with tensile deformation were partially relaxed during the cooling phase using low cooling rates. Only by repeated heating and cooling cycles tensile deformed, hexagonal or nanocrystalline silicon were transformed into diamond structured silicon. As a result, with a single thermal treatment compressive deformation can be relaxed while tensile deformation is mainly retained which opens the path to influence the chemical reactivity as shown exemplary shown by a modified etching behaviour.

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Atomic force microscopy: Impact on nanoscale charge-transport dynamics

P12

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Nanotechnology is now most enabling technology worldwide. Every section of material research, which is passing through this part to achieve ultimate goal of applications in nanoscale engineering, can be named as nanometrology. With a tremendous progress in nanoelectronics, the controlling operation with knowledge in localized charge-transport dynamics of nanometer-sized device remains both critical and challenging. Though several researches are going on this area through all over the world, even a high impact research is still demanding in nanometrology using nanoscale probing microscopy. Therefore, nanotechnology does not demand only the improvisation through device fabrication program, the reality lies on the true characterization of the materials in nanoscale region. The most unavoidable primary requirement to any device performance depends on the interface dynamics of the material. Study on the localized charge-transport mechanism at the interface of material plays a big role in improvisation of device engineering.

In that context, we are using atomic force microscope to visualize charge propagation through inorganic-organic nanostructure materials through nanoprobe induced charge injection method. Our experimental finding will open up a conceptually new design for next generation mechano-electrical miniature nanoelectronics [1-3].

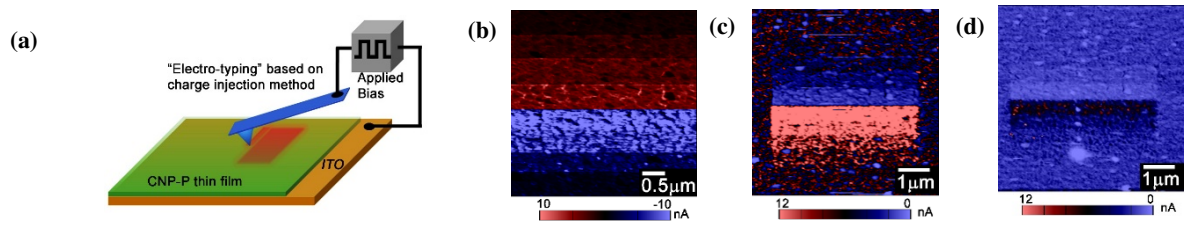

Figure 1. (a) Schematic of charge injection through AFM probe. (b) Visualization of current mapping during charge injection with different bias voltage (from ±1 to ± 7V). Visualization of current mapping after charge injection using (c) +5V and (d) + 1V bias voltage.

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Decomposition of zirconia-mullite material in contact with copper slag

P13

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The process of copper obtaining is one of the most effective in the non-ferrous industry. From the ore which purity reaches a few percent almost 100% purity product can be obtained. To achieve it raw material has to go through several steps which will increase the purity of copper by removing impurities: melting in the kiln, two-stage converter process, fire and electrolytic refining and at the end have to be formed [1, 2].

The highest temperatures during these processes do not exceed 1400ºC so the worst step for durability of refractory linings is the formation of copper slag. The main phase of this slag is fayalite with low melting temperature (1205ºC) but it consists also different even lower melting components such as PbO, CuO, and As_2O_3. The silica which is added as a flux also can form harmful chemical compound both with the components of the slag and the components of the refractory material [1, 2]. Refractory materials used in this field of the industry should characterize by a densified structure which will be resistant to the corrosive influence of copper slag. Magnesia-chromite refractories are still a suitable solution for application in the copper industry but a lot of research nowadays are carried out to reduce the amount of harmful chromium in refractory linings.

Zirconia-mullite material thanks to high mechanical and thermomechanical properties but also high corrosion resistance of mullite and the high refractoriness of zirconium oxide which can improve the properties of mullite seemed to be a promising candidate for application as a replacement of magnesia-chromite refractory during copper obtaining process [3].

The main purpose of the work was to investigate the influence of copper slag on the corrosion resistance of the zirconia-mullite material. The research was conducted on powder samples of copper slag and zirconia-mullite material mixed together and heated up to 1400ºC. Characteristic temperatures were determined by using hot stage microscopy. Open porosity and apparent density were determined by means of Archimedes principle. Phase composition of the samples after firing was investigated using XRD method. Observation of microstructure of the fired samples was achieved using scanning electron microscopy with the analysis in micro-areas (SEM/EDS). Also the total shrinkage was determined. Based on the above results the copper slag influence on the corrosion resistance of the zirconia-mullite material was determined.

Acknowledgments

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Microstructure and mechanical properties of Ti-6Al-4V titanium alloy fabricated by Laser Engineered Net Shaping (LENS)

P14

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Laser Engineered Net Shaping (LENS) is an additive manufacturing process in which it is possible to obtain near net and geometrically complicated parts in a stepwise strategy. It is a powder-feed technique in which the powder is injected into a molten pool created on the previously deposited layers by a focused, high-powered laser beam. In the present study LENS technique was used to build up a thin wall from gas-atomized Ti-6Al-4V powder. The deposition was performed using a LENS MR7 system with a 500 W fiber laser, which has a minimum beam diameter of 200 μm at a central emission wavelength of 1070 nm. The process was performed in purified argon atmosphere.

Primary β grains are observed in the microstructure of the as-deposited material. Those grains, in the centreal region of the wall, take the form of columnar grains, perpendicular to the substrate. In addition, smaller and equiaxed grains are visible at the edges of the wall. Only small amount of spherical pores are present in the microstructure of the material. The measured density reaches 99, 6 %. During deposition, the cooling of molten pools occurs through the substrate and by the surrounding atmosphere. Much higher heat loss through the substrate, leads to the directional growth and subsequently to the formation of elongated towards the substrate columnar grains. Their length typically exceeds several millimeters, while the width is in the range 100 – 300 μm. Inside the prior β grains, as a result of a high cooling rate, fine acicular α' is observed. The XRD analysis confirms the absence of the β phase. The α' martensite has a hexagonal lattice structure with lattice parameters similar to the α phase, therefore it was concluded that it does not play a significant role in the mechanical properties of the deposited material [1]. The measured hardness 371±4 HV5 is higher in comparison to the cast material (330- 360 HV), but lower than for the solution treated and aged material (380-420 HV) [2].

Acknowledgments

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Indirectly extruded biodegradable ZnAg alloys with improved strength and ductility

P15

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In recent years biodegradable materials with potential application for short-term implants like small plates, screws or stents have drawn increasing attention. Among metallic materials such as magnesium or iron alloys investigated for these kinds of applications, zinc alloys stand out a good combination of biocompatibility, reasonable corrosion rate, lack of toxic effects in contact with the human body and also ease of manufacturing [1–3]. However low mechanical properties of zinc limit its application in pure form. As-cast pure zinc is extremely brittle with low elongation usually lower than few percents [4]. This creates a necessity of new alloys system with biocompatible alloying elements. Silver ions and nanoparticles exhibit germicidal properties, as well as ability to protect surfaces against the adhesion of various compounds. Silver is used in the treatment of slow-healing wounds, burns and as implant component in dentistry [5]. For those reasons, silver is considered as an alloying addition for biodegradable implants. Silver is a peritectic-forming addition in zinc that causes solution strengthening within the solubility range, and also forms the intermetallic ε phase responsible for precipitation hardening effect. As reported in the literature this results in improvement of mechanical properties of zinc alloys [6, 7]. As a part of the research, the simultaneous effect of silver additions and hot indirect extrusion process was investigated. Based on the Hall-Petch relation the main goal is to obtain effective grain refinement so as to increase the yield point and strength of zinc. Three different newly designed Zn-Ag alloys were prepared and analyzed. The microstructure characterization was performed using light microscope and scanning electron microscope equipped with EDS and EBSD detectors in the initial and as-extruded state. Mechanical properties of Zn-Ag alloys were evaluated based on the Vickers hardness test, static uniaxial tensile and compression tests. In addition in vitro corrosion tests conducted in Hank's solution that simulates the physiological environment of the human body allow to assess the biodegradation of Zn-Ag alloys. As an effect of dynamic recrystallization during hot plastic deformation process, the grain size is reduced resulting in improvement of strength and ductility. Moreover a slight increase of corrosion rate in Zn-Ag alloys was observed. Presented research deliver complex information about designed zinc alloys, and provide insight into their applicability as a biodegradable material used for short-term implants.

Acknowledgments

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Liquid metal alloys – a study of the physicochemical properties of Ga-Zn alloys with Sn additions

P16

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Liquid metal alloys are a new and exciting group of materials, gaining more and more importance in various fields of science and technology. Non-toxic alloys with low melting points are heavily researched for a number of different applications, including lead-free solders [1], soft and wearable sensors [2], stretchable electronics [3] or as heat transfer fluids in Concentrated Solar Power Technologies [4]. Due to the outstanding thermal conductivity and the fact that those alloys remain liquid in the range of operating temperatures, alloys with low melting temperatures are suitable candidates for thermal management in different systems, including cooling computer chips [5], Li-ion batteries [6], high power LEDs [7] or in thermal management of nuclear reactors [8]. In order to successfully apply a material in any of the listed technologies, the basic properties of the alloy should be assed. In this study, the density, surface tension and viscosity of liquid eutectic Ga-Zn alloys with Sn additions were measured using the discharge crucible method in the range of 323-823 K. Across the analyzed temperature range, the viscosity and density of the eutectic Ga-Zn alloys increased with increasing Sn content, while the surface tension decreased. The obtained experimental values of the physicochemical properties were compared with the values determined using appropriate models, including the Egry model for density, Sato, Kucharski, Melywen-Hughes, Kozlov, Romanov and Petrov, Schick and Gasior models in the case of viscosity and Kohler, Toop, Muggiano and Butler models for the surface tension.

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In-situ mechanically cleave silicene-terminated $CaSi_2$ phase inside TEM

P17

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The groundbreaking work in 2004 [1] leads an unprecedented boom in interdisciplinary researches for 2D materials. Due to superior properties, 2D materials promise tremendous potential applications in electronics, optoelectronics, membranes, energy storage and generation, catalysis, sensing and so on [2]. Silicene, i.e., one-atom thick silicon sheet arranged in a honeycomb lattice, draws a considerable attention since it shows potential compatibility with current mainstream semiconductor manufacture, and current silicon-based semiconductor approaches the physical limit. However, lack of controlled ways to obtain these corrugated, sp^3 bonded silicon atoms in one sheet hinders its concrete application in devices. And the silicon atoms tend to be oxidized once they are exposed in the air for a very short time. Therefore, much effort has to be spent exploring methods to produce high-quality silicene. Here, we demonstrate an approach inside TEM to cleave $CaSi_2$ phase containing silicene sheet. It seems that it is not practical to produce a large sheet of silicene using mechanical exfoliation method due to strong sp^3 bonding with foreign atoms.

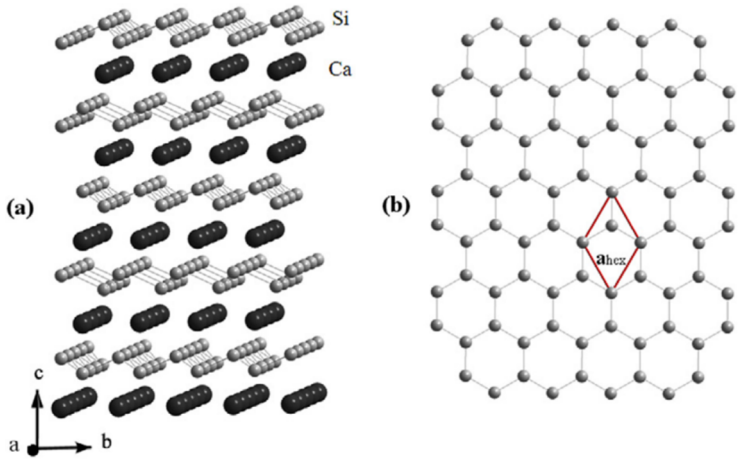

Figure 1. (a) Crystal structure of $CaSi_2$, (b) silicon sheet in $CaSi_2$ crystal structure [3].

Acknowledgments

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Growth kinetics of the selected intermetallic phases in Ni/Al/Ni system with various nickel substrate microstructure

P18

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Diffusion soldering (DS) is a useful method for joining different elements and it can be applied in many industrial branches e.g.: electronics, automotive or aerospace. The basis of the DS technology is an isothermal solidification taking place due to the reaction between solid substrates of high melting point with liquid solder (thin interlayer of low melting point). The microstructure and phase composition of the interconnection zone strongly depend on the applied temperature and time of annealing. For proper determination of the growth kinetics of selected intermetallic phases, accurate control of soldering conditions is required regarding not only the temperature and time but also atmosphere and pressure. As a result of DS, the diffusion zone is composed of one or several intermetallic phases making it thermally stable up to the melting temperature of the intermetallics that are filling the joined area. The Ni-Al equilibrium phase diagram indicates on the possibility to create several different intermetallic phases, among which one can exist in a wide range of chemical composition: from 40 up to 55 at. %. They are formed in a sequence starting from the lowest melting component (Al) to the highest melting one (Ni): Al_3Ni, Al_3Ni2, AlNi, Al_3Ni_5 and $AlNi_3$.

The subject of this work is the analysis of the microstructure of reaction products in Ni/Al/Ni system which is annealed at 720°C related to the grain size and number of boundaries of nickel substrates. Although Ni/Al/Ni system has been studied before [1], there is no data on such relation. The substrates cut from the nickel rod along two orientations were prepared: perpendicular (A) and along (B) the direction of the rod. Samples were annealed within a special holder in vacuum atmosphere for various periods of time. The observations of the morphology of the microstructure of both types of samples were accomplished using Scanning Electron Microscope (SEM). The Energy Dispersive X-ray Spectroscopy (EDS) analysis showed that continuous layers of Al_xNi_y phases have been formed in all samples. Initial studies performed for the samples annealed for 5 h at 720°C, have shown that the intermetallics location sequence was the same in both two types of joints and were as follows: $Ni_{substrate}/Ni_3Al/Ni_5Al_3/AlNi/Al_{solder}$. For the substrates of the B type, the coexistence of two types of NiAl phase was observed. The large difference in composition (50 at. % and 45 at. %) caused that these phases possessed various contrast in backscattered electrons mode. Although, in general, the phase composition and sequence of layers were the same in both types of samples, their thickness and overall thickness of the joined area was different. This fact points that the grain size, orientation and therefore, the grain boundaries amount and type had an influence on the joining process kinetics. Broadening of the joined area is a phenomenon that takes place before isothermal solidification in diffusion soldering process. Next, the shrinkage of the joined zone is observed. It can be concluded, therefore, that Ni_{needy} NiAl will be consumed by the growth of the neighboring phases. Additionally, Electron Backscattered Diffraction (EBSD) technique in SEM was employed in order to obtain the quantitative information on the crystallographic orientations of grains, their size and character of grain boundaries of both types of substrates. These preliminary results evidenced the validity of research on the growth kinetics related to the substrate's grain size and their boundaries.

Acknowledgments

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

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Thermodynamic properties of Ga-Ge-Li liquid alloys

P19

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Lithium batteries are a new and promising high-energy-density power source. Many new lithium battery systems are being developed, and these batteries are finding many new uses in the consumer market today [1]. In order to prepare the appropriate electrode material, information on the thermodynamic properties, phases equilibria and phases transformations of the elements involved is required. To prepare thermodynamic database, experimental information about a particular system are necessary. The Ga-Ge-Li alloys could become a promising anode material. Unfortunately, there is a lack of thermodynamic data for this systems in the literature. Thus, it was found to be necessary to perform an experimental investigation on the abovementioned system. This work presents thermodynamic information about Ga-Ge-Li phase diagram. The different temperatures were measured using the DTA method during thermal process. Moreover electromotive force measurements were conducted for the mentioned system. The cells were prepared using a glovebox chamber with protective argon atmosphere. Eutectic KCl-LiCl and LiF-LiCl salts were used in the preparation of the electrolyte. The construction of the cell is shown in Figure 1.

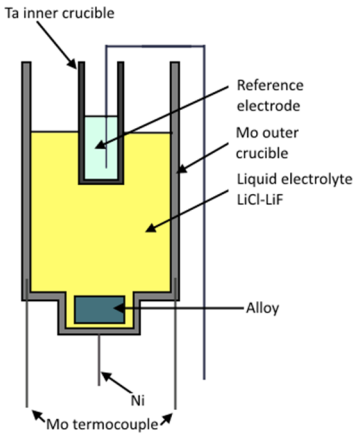

Figure 1. Illustration of cell for EMF measurements

The obtained results will be used to optimize the thermodynamic properties of the phases present in the ternary Ga-Ge-Li system, and for calculation of the phase diagrams of binary and ternary systems. Furthermore, they will be introduced into the free of charge Entall database http://www.entall.imim.pl/ [2].

Acknowledgments

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An approach to improve the electrical properties of oxide thin films by embedding metallic nanowires

P20

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The emergent growth of flexible and transparent electronic circuits requires thin-film transistors (TFTs) to operate at high speed and provide high current density without the need of high processing temperatures and exotic processing. While channel length miniaturization towards nm-scale is a natural route to accomplish this, it typically demands expensive patterning techniques not compatible with large area fabrication and can result in undesirable short-channel effects. Solution-based materials are an attractive alternative however their performance is still short of their standard high vacuum counterparts.

One possible workaround is presented here is, the use of oxide solution-based thin films with embedded metallic nanostructures as a replacement of traditional oxide semiconductor thin films. One important advantage of this approach is that owing to the remarkable unidirectional electronic transport properties of 1D nanostructures such as nanowires (NWs), these can greatly enhance the electron movement inside thin films. This is particularly interesting when considering low-temperature solution-based oxide semiconductors, where electrical properties are typically degraded when compared to physically processed films. Hence, one can expect fully solution based oxide materials at temperatures compatible with polymeric substrates without compromising their electrical performance. The fact that the NWs are embedded in a thin film is also beneficial to stabilize these nanostructures, as it is well-known that the interaction of their surface with a surrounding environment rules their properties. In other words, the thin film can act as a passivation of the NWs and increase adhesion to the substrate.

The approach followed in the present work is based on sustainable solution-based indium-free zinc-tin oxide (ZTO) thin films, solution that already demonstrated promising results in TFTs [1]. At this initial stage embedded Ag NWs were used owing to the simple solution-based route to obtain long (≈10 μm) nanostructures with this material. More sustainable NWs (e.g. Cu or Zn-based) can be used afterwards. The Ag solution was firstly transferred to the substrate and then the ZTO thin film was spin coated on the surface and annealed. The influence of annealing temperature and different Ag NW contents on the electrical and optical properties of the ZTO thin films will be discussed.

These optimized transfer processes offer the possibility of high performance solution-based TFTs for applications requiring low-cost and low-temperature nanofabrication on transparent and flexible substrates.

Acknowledgments

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Using solution-processed AlO_x as dielectric and resistive switching active material towards system-on-panel applications

P21

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Solution-based high-*k* dielectrics are suitable as resistive switching materials which can be applied in transparent and flexible resistive random-access memory (RRAM) for system-on-panel (SOP) applications. Intrinsic defect states and the reversible dielectric breakdown motivate many research groups to study resistive switching properties of this type. Furthermore, major advantage is that high *k* materials are widely used as gate dielectric of thin film transistors (TFTs) which facilitates tremendously the circuit integration for advanced transparent SOP applications [1, 2].Towards low cost fabrication approaches, TFTs and RRAM devices are developed in this work from a solution-based aluminum oxide (AlO_x). In principle, to obtain the resistive switching device from an insulating active material, an electroforming process (soft breakdown) is required to establish the resistive switching template. The migration of oxygen ions induces localized oxygen deficient conductive paths with respect to the pristine state and set the device to low resistance state. However, if during fabrication process these defect states are created, then the electroforming occurs at low voltages which can be understood as forming-free resistive switching.

Here we show an optimized solution processed bilayer AlO_x with different defect states which results in low-power resistive switching properties [3]. In addition, we demonstrate that by changing the parameters of solution combustion synthesis and controlling the annealing method a stable amorphous Al_2O_3 dielectric can be developed. Both RRAMs and TFTs exhibit excellent electrical performance and are strong candidates towards low cost integration for SOP applications.

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3D analysis of thin layers by ToF-SIMS

P22

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Numerous usages of thin film deposition techniques such as atomic layer deposition (ALD) for deep microscopic structures and development of three-dimensional large-scale integrated structures like trenches and cavities introduce a true challenge for film characterization. These 3D-micro-structured substrates are typically vertical oriented high aspect ratio (HAR) structures.

Study or characterization of specific nano-domains like local defects, doping concentration, conformality, and interfaces, rely predominantly on cross-sectional sample preparation and characterization by electron microscopy. This approach faces the challenge of single lamella preparation of each interested trench part, low spatial resolution to characterize interfacial diffusion and monitoring light elements (e.g. lithium), and cleavage plane inaccuracy.

To overcome this challenge, we used the capability of time-of flight ion mass spectrometry analysis (ToF-SIMS). This approach enables access to more detailed compositional information on the trench walls.

Two test structures were used to proof the method feasibility. First study investigates the non-plated area at the bottom of large through-silicon-vias (TSVs). One of the main challenges of TSV metallization is the elemental characterization of seed layer through TSVs. ToF-SIMS is capable of checking this seed (cobalt) layer and its corrosion under the copper electrolyte influence.

Second study monitors the uniformity of Silicon dopant concentration in ALD deposited HfO_2 thin films in high-aspect-ratio structures (Pillar-Hall[1]). The test structure itself enables analysis of plane surfaces after lifting off the roof, see Figure 1.

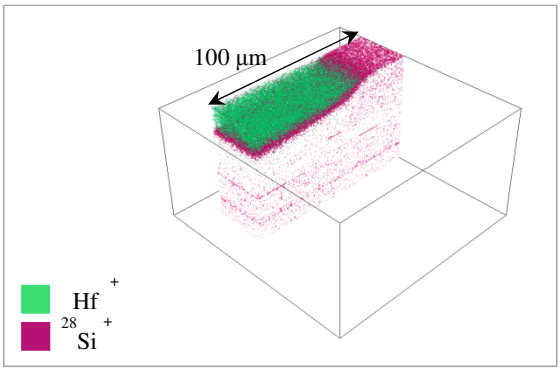

Figure 1. 3D ToF-SIMS analysis from deposited Si doped HfO_2 in high-aspect ratio structure (Pillar-Hall®).

[1] PillarHall® silicon wafers and chips enable easy analysis of thin film conformality using well-defined, record-demanding microscopic 3-D structures. Typical usage areas are atomic layer deposition and chemical vapor deposition R&D.

3D localization of spinel and sodium contamination in alumina by TOF-SIMS

P23

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Phase and chemical compositions are crucial for properties of advanced ceramic materials. Study of the phase and chemical composition is nowadays limited to localized 2-dimensional methods and its sensitivity to local changes. Alumina as the most used ceramic materials is often doped by MgO to prevent abnormal grain growth to allow annihilation of pores pinned at grain boundaries. The phase equilibria of Al_2O_3-MgO has been widely studied and discussed. However, chemical composition in three dimensions of spinel ($MgAl_2O_4$) has never been described. 2D & 3D TOF-SIMS analysis of the spinel in an alumina matrix and its chemical composition will be presented (Figure 1 and 2).

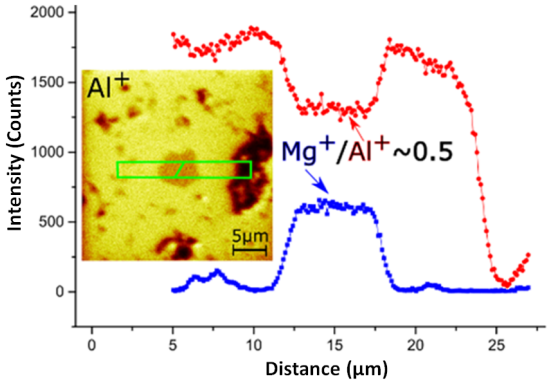

Figure 1: TOF-SIMS image and lateral profile of Mg and Al through a spinel grain.

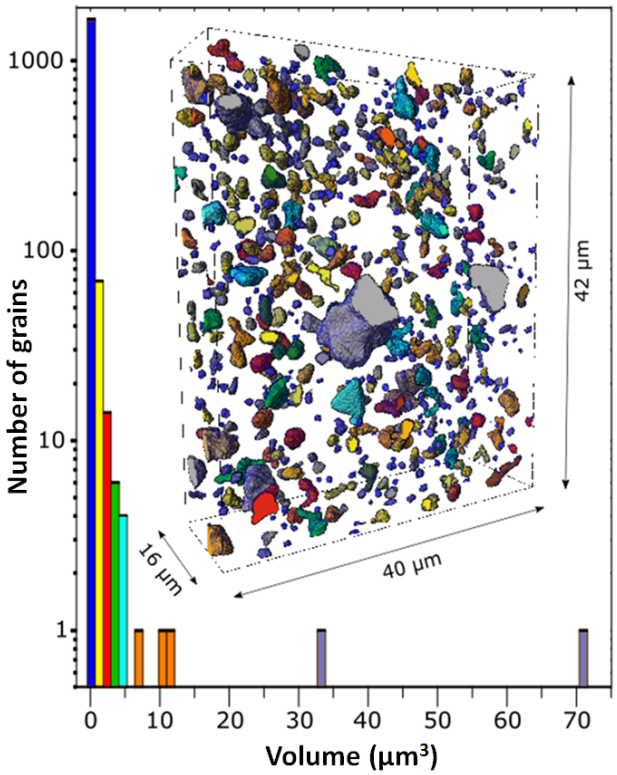

Figure 2: Spinel grain size distribution in sintered alumina measured by TOF-SIMS. 3D render was colorized according to the volume of grains.

Microstructure of nitride diffusive zone formed on Ti_6Al_7Nb alloy

P24

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The Ti_6Al_7Nb alloy was developed as a bio- compatible substitute to $\alpha + \beta$ phase Ti_6Al_4V alloy, which has been the most widely used titanium alloy since its development at XX century. The Ti_6Al_4V soon found its way into such products as implants and prosthetics. However, long time tests showed that the first of alloying addition causes aluminosis, while the second one is strongly cancerogenous. Research in that area showed, that the two phase microstructure and resulting high strength could be obtained with a Ti_6Al_7Nb alloy. The aluminium has not been so easy to substitute as the vanadium, a partial solution should be sought in nitriding of this alloy. Such protection works as long as the protective layer lasts, but its high hardness improves material wear resistance and secures a reasonable lifetime even for medical application.

Nitriding of titanium alloys could be performed through gas (GN) [1] or plasma nitriding (PN) [2, 3]. First of them, carried out in nitrogen filled chamber at ~ 1000°C is relatively simple to apply, but high temperature usually negatively affects both alloys microstructure and surface roughness. The PN calls for vacuum chamber and high-voltage power supply, but carries the advantage of being executed at much lower temperatures. In case of Ti_6Al_4V both type of treatments needs roughly same time-frame to produce diffusive zones of comparable thickness. The long established knowledge of the phase composition of the latter acquired with optical microscopy, SEM and XRD investigation was only recently contested by TEM investigations [2]. They showed, that in case of Ti_6Al_4V, the nitrided diffusive zone is built not of three δ-TiN/ ε-Ti_2N/ α-Ti(N) sub layers as previously presumed, but of four δ-TiN/ δ'-Ti_2N α"-Ti martensite/ Ti_3Al intermetallic/ α-Ti(N). The problem is significant as in originally accepted sequence most of diffusive layer was formed of ceramic phases, while in the following one ceramic layer is only a small fraction of it. The data on phase composition of nitrided diffusive layer on Ti_6Al_7Nb is much more sparse than Ti_6Al_4V, as only Siyahjani et al [4] published SEM observations and XRD spectra confirming presence of TiN on gas nitrided samples.

The TEM microstructure investigation of plasma nitrided diffusive zone formed on Ti_6Al_7Nb alloy at 680°C / 6 h and at 740 °C / 6 h helped to establish that it consists of three distinct layers. However, the first one formed by δ-TiN differs in defect density, i.e. the part near surface is porous nano-crystalline, while the one located below it is non-porous and slightly coarser. The TiN is backed by α"-Ti martensite and Ti_3Al layers, respectively. It indicates that the ceramic barrier between human body and the metallic core is again quite thin. Fortunately, analysis of chemical composition indicated, that both the aluminium and niobium are pushed away from the surface and out from the next martensite layer, what significantly improves protection from the former.

Acknowledgments

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Visualization of nanocrystalline CuO in the grain boundaries of Cu_2O thin films and the role in bipolar resistive switching

P25

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Direct evidence for the presence of a CuO structure in the grain boundaries of Cu_2O thin films is provided by high resolution automated phase and orientation mapping (ASTAR), which was not detectable by classical transmission electron microscopy techniques. Conductive atomic force microscopy (C-AFM) revealed that the CuO causes a local loss of current rectification at the Schottky barrier between the C-AFM tip and Cu_2O. The suppression of CuO formation at the Cu_2O grain boundaries is identified as the key strategy for optimization of devices, which rely on the intrinsic properties of Cu_2O.

Since electrochemical resistive switching involves redox reactions and filamentary conduction, secondary phases can offer advantages over homogeneous switching matrixes. The remarkably stable multilevel cell (MLC) operation of an Al_2O_3/Cu_2O bilayer memory is related to the highly conductive grain boundaries, which present an as-fabricated pattern of preferential current paths. With increasing current compliances, a transition from a thermally activated behavior to metallic conduction was observed as different regions of the sample dominate the current transport, depending on the extent of the copper filament into the copper oxide switching matrix.

Transparent molybdenum oxide thin films grown on organic and inorganic substrates

P26

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In recent times, relevant investments have been done to promote the implementation of electrochromic (EC) materials in displays or light modulation systems. Transition metal oxides (TMOs) in the form of thin-films have high potential for these applications. TMOs nature, "green" production processes, low consumption of raw materials and higher device durability when compared with organic electrochromic devices are strong motivations to focus on their research and development [1]. Among TMOs, molybdenum oxides (MoO_x) are very interesting for electrochromic proposes showing an intense electrochromic colour change and relevant electrical and dielectric properties in a wide range of temperatures (220 K and 330 K).

In this study, two series of MoO_x thin films were deposited in different substrates (glass and PET, both with ITO conductive layer and silicon) by reactive DC magnetron sputtering. A pure Mo target was sputtered by applying a current density of 100 A/m^2 in a plasma composed by argon (working gas), introduced using a fixed flow, and a reactive gas which was oxygen (O_2). For the first series of films, different O_2 flows were used to prepare a set of films with different Mo oxidation states. In the second series, the O_2 flow was fixed and the deposition time was decreased from 60 min (used in the first series) to 30 min and to 15 min, to prepare MoO_x with different thicknesses. No external heating was used during the deposition and the substrates were grounded.

It was found the existence of a structural threshold, from crystalline to amorphous nature, between 8 and 16 sccm of O_2 flow. This structural modification is responsible for an opaque to transparent transition. Optical measurements showed transmission higher than 80% in the visible range. Transparent films presented thicknesses between 230 and 900 nm and a compact/dense and featureless morphology. XPS and RBS revealed different oxidation states and different stoichiometries. The electrical measurements showed that the dielectric losses are almost independent of the temperature but dependent on the sample microstructure. Figure 1 shows an optimized MoO_x thin film in a coloured and bleached state. The cyclic voltammograms clearly showed good reversibility and reproducibility in the electrochromic performance.

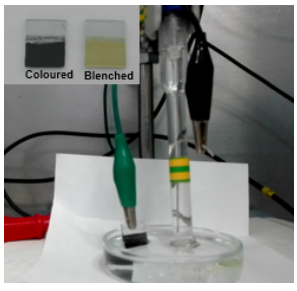

Figure 1. Thin film of molybdenum oxide in coloured and blenched state.

Acknowledgments

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Enhanced UV flexible photodetectors and photocatalysts based on TiO_2 nanoplatforms

P27

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Titanium dioxide (TiO_2) has elevated stability and photoactivity, moreover it is non-toxic, and earth-abundant. It has been extensively studied for applications ranging from photocatalysis, solar cells to sensors [1, 2]. In the present study, TiO_2 nanostructured films were grown on bacterial nanocellulose, polyester and tracing paper substrates using a hydrothermal method assisted by microwave irradiation without any seed layer [2]. The selected substrates are inexpensive, reliable, recyclable, flexible, lightweight, and when associated to low temperature synthesis and absence of seed layer, they become suitable for several low-cost applications. Structural and morphological characterization was carried out by scanning electron microscopy (SEM) coupled with X-ray energy dispersive spectroscopy (EDS), X-ray diffraction (XRD) and by Raman spectroscopy. The microwave synthesis totally covered the substrates, forming uniform nanostructured films while maintaining the substrates flexibility. Fine nanorod aggregates forming TiO_2 flower-like structures were observed and regarding the substrate used, different nanostructured films were obtained (Figure 1). The photodetection behaviour of each material was studied by Kelvin probe force microscopy experiments with a clear relation between contact potential difference shift and their photosensitivity/photocatalytic activities. The material photocatalytic activities were evaluated from rhodamine B degradation under solar radiation.

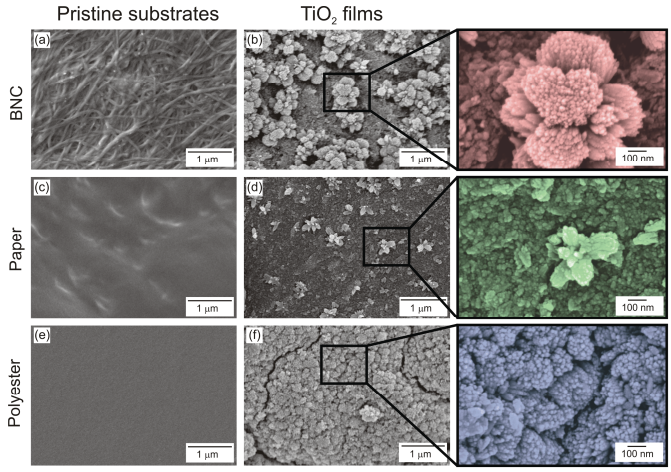

Figure 1. SEM images showing the BNC (a), paper (c) and polyester (e) pristine substrates together with the TiO_2 films grown on BNC (b), tracing paper (d) and polyester (f) substrates [2].

Acknowledgments

The work was supported by the FCT - Portuguese Foundation for Science and Technology, through the scholarship BPD/84215/2012. This work is also funded by FEDER funds through the COMPETE 2020 Program and National Funds through FCT - Portuguese Foundation for Science and Technology under the project number POCI-01-0145-FEDER-007688, Reference UID/CTM/50025/2013.

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Degradation of $Li(Ni_{0.33}Mn_{0.33}Co_{0.33})O_2$ in the recycling of lithium battery cathodes

P28

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The compound $Li(Ni_{0.33}Mn_{0.33}Co_{0.33})O_2$ (NMC) is the state-of-the-art lithium-ion battery cathode material. Due to the increasing demand NMC is of crucial economically importance for the worldwide emerging market of electromobility. Recycling of end-of-life lithium-ion batteries to recover NMC, in particular of batteries from automotive vehicles, is one future strategy to save costs and to become more independent from the supply of the essential elements Co and Mn. Several concepts for NMC recycling from lithium-ion batteries are based on wet-chemical process steps, in particular, to separate the NMC containing cathode layer from the underlying metal foil. However, NMC is very sensitive against the attack by water and reagents that are added to promote the separation process.

The present study deals with the wet-chemical recycling of NMC using aqueous reagent solutions in a under varying process conditions. The recovered NMC samples are characterized in order to study the ongoing degradation at the surface of the NMC particles. In particular, two major degradation pathways are identified: (i) a preferential loss of lithium and nickel and (ii) the formation of passivation layers due to unwanted side reactions. DRIFT measurements are performed to study the NMC surface species after the recovery processes. SEM/EDX mappings are used to detect changes in the chemical composition in the surface region of the chemically treated NMC particles. Finally, a detailed study of the changes in the chemical state at the NMC particle surface is done by Raman microscopy by means of the deconvolution of the recorded spectra into their A_{1G} component (representing the metal-oxide phonons) and into the E_g component (representing the oxide-metal-oxide phonons).

As result of this study, the consequences of different wet-chemical process conditions on the quality of the recovered NMC material are discussed.

Detection of ultra-small amounts of exchanged oxygen by oxygen solid electrolyte coulometry

P29

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For the characterization of metal oxide based new materials for electrochemical applications, e.g. cathode materials, it is crucial to know the changes of its oxygen stoichiometry at high temperatures and varying oxygen partial pressures ($p(O_2)$). For this purpose, often oxygen solid electrolyte coulometry (OSEC) [1, 2] is used. State-of-the-art systems contain galvanostatically controlled zirconia-based pump cells up- and downstream of a heated sample reactor. With such a setup, however, it is not possible to quantify small amounts of exchanged oxygen in the $p(O_2)$ range between 10^{-5}…0.1 Pa.

To enable experiments at the above-mentioned conditions, a new setup was designed and tested using a modified measuring circuit, recently described by Schelter et al. [3]. The setup is shown in Figure 1.

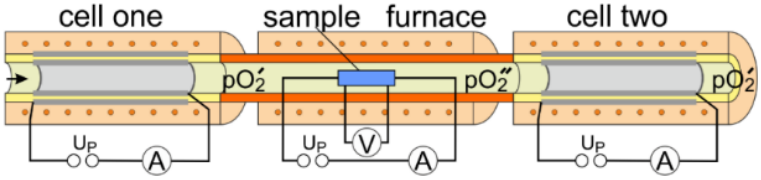

Figure 1. Setup for coulometric measurement of oxygen exchange of heated samples.

A gas mixing station (not shown) for adjusting the measuring gas flow and the incoming oxygen partial pressure $p(O_2)$ during the experiment is followed by a first oxygen titration cell (cell one, O_2-DF-28.0, Zirox Sensoren und Elektronik GmbH, Greifswald, Germany) for precise adjustment of the $p(O_2)$ in the gas atmosphere around the sample. The cell is connected to a tube furnace with a sample holder for four-point AC and DC conductivity measurements at elevated temperatures (300…800 °C). From the gas outlet of the furnace, the gas is guided to the inlet of the second titration cell (cell two) for measuring coulometrically the oxygen or other oxidizable gases exchanged with the sample.

Due to the modification of the oxygen titration cell and the optimized sealing concept of the sample furnace, the tightness of the system could be considerably increased. This improvement and the usage of an integrated control unit enabled a significant diminishment of the limit of detection for this setup down to amounts < 100 pmol of exchanged oxygen. Fixing an oxygen slip during titration of 1 % as the maximum tolerable error, the upper limit of the measurable titration current ranges above 5 mA at a flow rate of 50 ml/min through the setup. Assuming a titration time of 30 min, the upper limit of detectable exchanged oxygen amounts accordingly to 25 µmol. This newly developed arrangement enables the precise measurement of oxygen and of oxidizable gases from the pico-mol up to the micro-mol range within a temperature range between 300 and 800 °C.

Acknowledgments

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Functional oxide based nanocomposites for energy harvesting systems

P30

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United Nation recognizes the relevance in assuring access to affordable energy for all and has adopted this as a resolution for 2030. Off the grid standalone power harvesting is for sure an approach to consider when targeting this goal [1]. Among all the sustainable sources, conversion of kinetic energy may be considered as widely available. Within this context, and considering the last, piezoelectric wearable devices that follows body motion and convert it into to electric energy presents huge potential to scavenge and make use of this source of energy [2].

In this PhD project we aim to develop nanocomposite based fibers, functionalized to be either electric conductor and piezoelectric. They will be combined and integrated in textile, paper like cellulose matrices and biocompatible elastomers by embroidering or weaving to construct energy harvesting systems and sensors for wearables, flexible electronics, opening also the route for their exploration in e-skin devices. Selection of materials and methods will have in account economic aspects and relative availability of compounds, as well as ecological impact and life cycle upon final applications. Having this in mind, carbon fibers are a promising materials for this application since they combine high conductivity and mechanical strength and can be produced from textile wastes material [3]. In this work, carbon fibers and carbon fiber based yarn will be used as flexible electrodes for PENGs. Regarding piezoelectric materials, lead free ceramics like ZnO, perovskites, $BaTiO_3$ and KNN have good piezoelectric properties but their brittle nature and poor mechanical properties limits their application on flexible and stretchable PENGs like wearables and such. To withstand this issue, they can be used in the form of nanostructures and used as coatings, blended or doping polymers [2]. NFC antennas, touch/pressure sensor arrays and piezoelectric nanogenerators will be made by means of integrating conductive, piezoelectric and fibers that combine both functionalities in textiles, cellulose and biocompatible polymers templates by either weaving, embroidering or sewing.

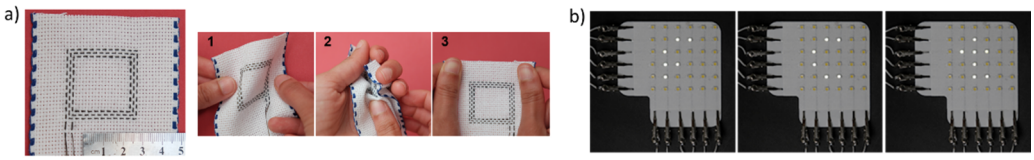

Figure 1. a) NFC antenna produced by sewing using carbon fiber yarn and b) 6x6 LED display on paper using carbon fibers as rows and columns of the addressing matrix here displaying "FCT".

Acknowledgments

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New composite materials based on ceria and magnesia

P31

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In spite of investigations for more than a century [1], the broad use of solid oxide fuel cells (SOFCs) is still limited by electrolyte materials. The group of Kosacki et al. [2] found out that the grain boundaries between electrolyte and isolator particles can have even higher ionic conductivities than the electrolyte itself, which offers a new possibility to obtain materials with high oxygen ion conductivities. Here we describe a novel approach how to make use of this unusual high boundary layer conductance. Gadolinium doped ceria (GDC) has been chosen as the electrolyte candidate due to its high ionic conductivity.

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 [4].

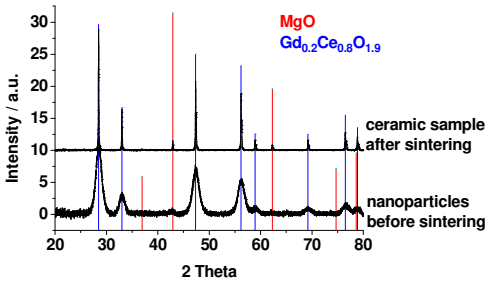

Figure 1. XRD pattern of the mixture of GDC and MgO sample before and after sintering

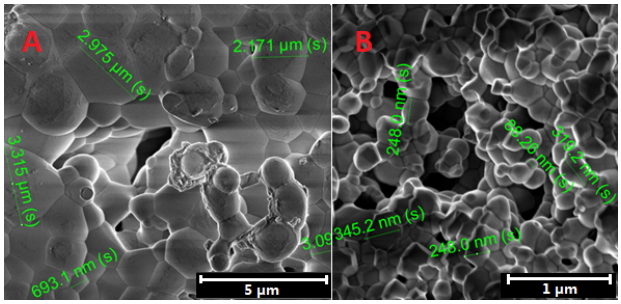

Figure 2. TEM images of the composite ceramic samples (A: normal sintering method, B: FAST/SPS sintering)

The sample was pressed and sintered using normal sintering method. 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 image of the sintered ceramic shown in Figure 2A also manifest larger domains than the disperse nanoparticles before sintering. A new sintering method Field Assisted Sintering Technology (FAST)/Spark Plasma Sintering (SPS) was then used to avoid the grain growth during the sintering. Figure 2B shows the sample sintered using FAST/SPS. The grain size is obviously much smaller than that of the sample, which was sintered using the normal sintering method.

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. The particle domains in the sintered ceramic are enlarged, but the enlargement can be avoided using the FAST/SPS sintering method.

Acknowledgments

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Multi-length scale characterization of the commercial aluminium alloys used in automotive industry

P32

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Modern aluminium alloys used in the automotive industry are characterized by a complex microstructures obtained by special heat treatments of alloys with finely tuned chemical compositions. On the other hand the parts for automotive are produced in large quantities and there are instances of faulty products appearing in various stages of the supply chains. Elimination of the imperfections in question requires tracing back the to their origins; frequently in the extrusion of the bars, in machining/forming of the parts, their subsequent heat treatment and joining. In this situation, for solving problems with quality and reliability of products, modern methods of characterization of the macro-, micro and nano-structure need to be used. In this context we present results demonstrating the benefits of the "holistic" approach, which starts with on non-destructive screening of the parts, X-ray tomography of pre-selected in NDT, testing of the mechanical properties with use of ultra-small samples, investigations of the microstructure with standard light and electron microscopy and ends-up with high resolution electron techniques. The examples provided include aluminium strain and age hardened aluminium alloys used in for production of air-conditioning units.

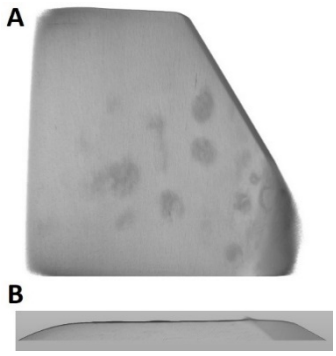

Figure 1. X-ray tomography of the defects in the parts made of AA series 6081: (A) parallel, (B) perpendicular to the external surface.

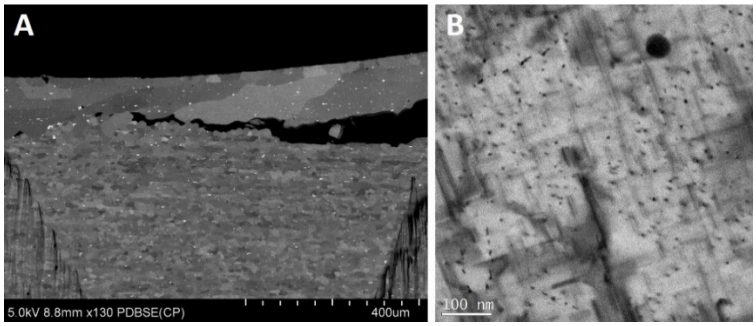

Figure 2. SEM (A) and TEM (B) images of the structure of the parts with the defects shown in Figure 1.

Reliability of micro- and nano-electronics under mechanical load for automotive applications

P33

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The car of the future can be described as a computer on wheels with central processing units controlling high-end applications like infotainment, car-to-car communication, electric and especially automated driving. To ensure the functionality of these applications as well as the safety of the passengers, a big amount of automotive-ready micro- and nanoelectronic devices has to be implemented. Due to the harsher use conditions (mechanical, thermal and combined), the reliability requirements for micro- and nanoelectronic devices are higher for automotive applications than for consumer applications like smartphones. To cope with these extended requirements, specific standards and guidelines have been established e.g. by the AEC (Automotive Electronic Council, e.g. AEC-Q 100 [1]) but also by original equipment manufacturers (OEMs). The content of these documents differ from the established JEDEC (Joint Electron Device Engineering Council) standards (e.g. JEP001 [2]). However, the guidelines are not holistic and exhibit several gaps especially when it comes to mechanical stress and high-end technologies like advanced packaging.

To determine the severity of these gaps, the focus of this work is on the Back End of Line (BEoL) reliability as well as Chip Package Interaction (CPI) under mechanical load. In the context of the European project TRACE (Technology Readiness Process for Consumer Electronics), test boards are developed together with partners to determine the reliability of solder joints of different package types under thermomechanical loading conditions occurring in automotive applications. To apply a controlled mechanical load (Vibration, Board Warpage, local constant as well as pulsed load on package), a flexible probe card holder is developed for a nanoindentation system. The test vehicles will be provided by GLOBALFOUNDRIES and TRACE partners. The investigations should mainly focus on advanced packaging solutions like 3-D stacking, CSP (Chip Scale Package), and FCBGA (Flip Chip Ball Grid Array) with copper pillars [3].

To determine the effect of the applied load and the induced damage, different metrology techniques are deployed. When possible, the damages should be quantified electrically. However, also the nondestructive technique of X-Ray microscopy is applied. The long-term objective is to perform *in-situ* nanoindentation experiments in a novel X-Ray microscope. This approach should provide a better understanding of crack propagation in the back-end of line of the test vehicles. The overall objective of the project is to develop a framework, which can be used to determine the automotive readiness for high-end micro- and nanoelectronic technologies to ensure the functionality of applications used in cars as well as the safety of the passengers.

Acknowledgments

Without the support of Tanja Graf, Andreas Aal, and Knut Schmidt from Volkswagen AG as well as Jens Paul, Maciej Wiatr, and Matthias Klude from Globalfoundries this research project would not be feasible.

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Sustainable functionalized fiber-based structures for application in electronic and electrochemical systems

P34

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Wearable electronics is nowadays a reality in enabling monitoring, sensing and storing/harvesting energy systems. Nevertheless, the challenge is to develop sustainable energy-storage devices with stable cycling performances using environmental friendly and abundant materials, not harmful, able to result in ergonomic, flexible, stretchable and skin or body mounted and sustainable.[1] Fiber-based structures owing to the high specific surface area combined with suitable inorganic nanostructures, organic or polymeric materials have unique potentialities to respond to whether mechanical, thermal, chemical, electrical, or even optical stimulus. [2] When integrating functionality and wearability, these devices are capable of reaching high capacity and enable unique characteristics in electrochemical devices such as electrochromics flexible lithium-ion batteries and supercapacitors. [3].

This PhD project aims to promote and combine such goals, exploring the potential of fiber-based structures combined with abundant and environmental friendly conductive (for both electrons or ions), semi conductive and insulating materials. If integrating them in single or multifiber core-shell architectures, one may get superior performances regarding the high surface area and the tuned porosity of the intended hybrid structures, of high relevance for electrochemical and energy-storage applications. Therefore, it will contribute to achieve the goals proposed for 2030 by the United Nations, namely the goal number 7: Ensure access to affordable, reliable, sustainable and modern energy for all.

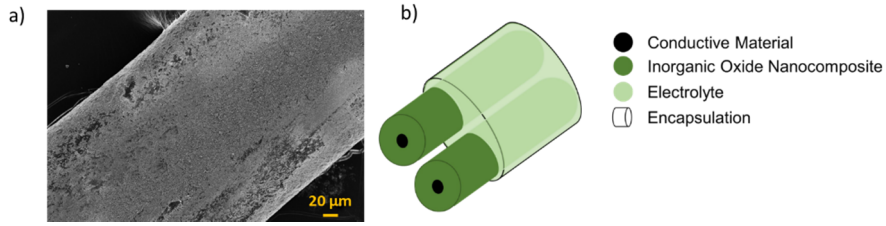

Figure 1. a) Ethylcellulose fiber functionalized with ZnO nanoparticles by sol-gel method, and b) Schematic representation of the symmetric supercapacitor, where two equal conductive fibers can be functionalized with inorganic oxide materials and thereafter an electrolyte may be used there between and working, at the same time, as the encapsulation layer.

Acknowledgments

The authors kindly thank the IDS-FunMat international doctoral school program for funding of this PhD. The authors would also like to acknowledge European Commission under project NewFun (ERC-StG-2014, GA 640598), BET-EU (H2020-TWINN-2015, GA 692373), 1D Neon (H2020-NMP-2015-IA, grant 685758-21D). This work was also supported by FEDER funds through the COMPETE 2020 Programme and National Funds through FCT – Portuguese Foundation for Science and Technology under the project number POCI-01-0145-FEDER-007688, Reference UID/CTM/50025 and project PapEl, reference PTDC/CTM- NAN/5172/2014.

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Combination of soft X-ray microscopy with in-situ mechanical testing to image crack propagation in microchips

P35

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The combination of high-resolution X-ray imaging with in-situ mechanical testing, particularly the application of a specially designed indenter manipulator in the full-field transmission X-ray microscope at the U41-PGM1-XM beamline of the synchrotron radiation source BESSY II [1] is used to study the fracture behavior of microchips. The X-ray microscope was operated at a photon energy of 1200 eV for the experiments described here. The achieved spatial resolution of about 25 nm allows to image the metal interconnect structures in microchips manufactured in advanced technology nodes. The mechanical manipulation of the samples was performed using a Picoindenter PI95 (Bruker) equipped with a tungsten wedge indenter [3].

Thermomechanical stress in microchips increases the risk of failure in on-chip interconnect stacks, also called backend-of-line (BEoL) stack [2], caused by delamination along Cu/dielectrics interfaces (adhesive failure) and/or fracture in dielectrics (cohesive failure). In this paper, crack propagation in the BEoL stack of a microprocessor manufactured in an advanced technology node is presented. It consists of 12 layers of Cu interconnects which are insulated by thin film materials with low dielectric permittivity (low-k materials) [2]. Particularly designed dense metal structures, so-called crack stop structures, are supposed to dissipate energy and to slow down or even stop the crack propagation.
The example demonstrates that the pathway of cracks, and consequently the weakest structures for cracking, can be identified in fully integrated, realistic multilevel Cu/low-k interconnect structures of microchips manufactured in advanced technology nodes.

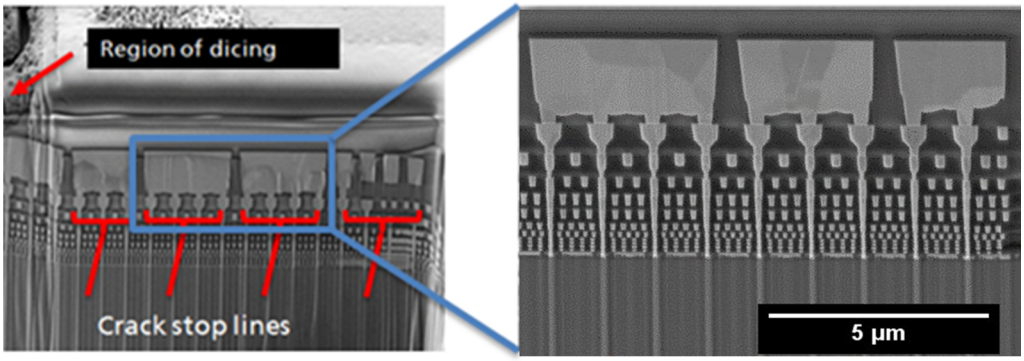

Figure 1. SEM cross-section images with crack stop structures of a leading edge microprocessor with 12 layers of Cu interconnects [3].

Acknowledgments

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In-situ mechanical measurements on M1-M5 BEoL cantilever beams

P36

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For advanced packaging and particularly for 3D stacked microchips, managing the effects of mechanical stress is a key task to maintain the performance of microelectronic products and to avoid unintentional effects caused by chip-package interaction (CPI). A dedicated stress engineering for such complex 3D systems requires accurate data of materials properties as input for FEM simulations [1]. Taking into account the nonuniform character of the effective mechanical properties, i.e. average values for a "composite" consisting of a part of the BEoL stack, mechanical properties can be determined experimentally for small beams extracted from the BEoL stack. It was shown that the copper density and the dominating copper line direction in the BEoL stack significantly affect the CTE of the metallization layers [2]. The elastic properties have been widely studied for homogeneous thin films using indenter tools [3], however, whole BEoL layers with a "composite" nature have not been investigated so far. In this study, a novel methodology for the measurement of the Young's modulus (E) for "composite" layers is demonstrated and applied for the BEoL stack using free-standing silicon and M1-M5 BEoL cantilevers which were prepared by FIB milling. The experiments were conducted in-situ in an SEM applying the Bruker/Hysitron PI87 nanoindenter. Load and displacement were measured while the beams were manipulated with a spherical indenter tip (Figure 1). For the verification of the beam bending, high resolution SEM images were recorded before and after the manipulation and at the largest deflection of the beam. The images were then processed with an image analysis routine for robust and reproducible measurements of the beam length. The experimental results verify the FEM simulations (Figure 2). FEM simulations demonstrated that the simple Bernoulli beam theory is not sufficient to calculate E with just the deflection measured by the indenter tool. The support itself is deflected and the indenter deforms the sample surface plastically (Figure 2). Therefore, the beam displacement was corrected which is especially important for short beams. The effective E and CTE [2] values for the BEoL stack are the input data needed for accurate FEM simulations of 3D-stacked ICs, without the need to consider the individual metal and dielectric materials data of the BEoL structures.

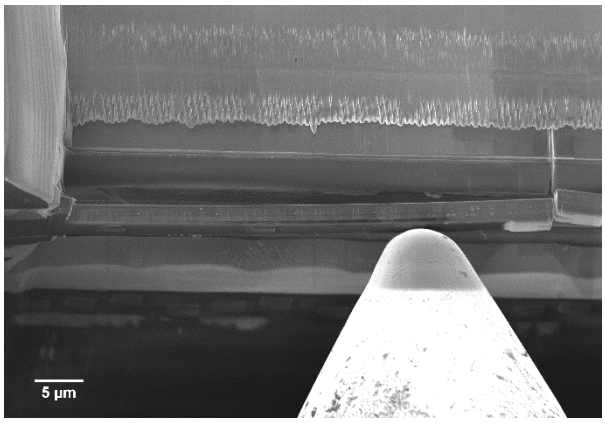

Figure 1. SEM image of a BEoL beam and the indenter tip in contact.

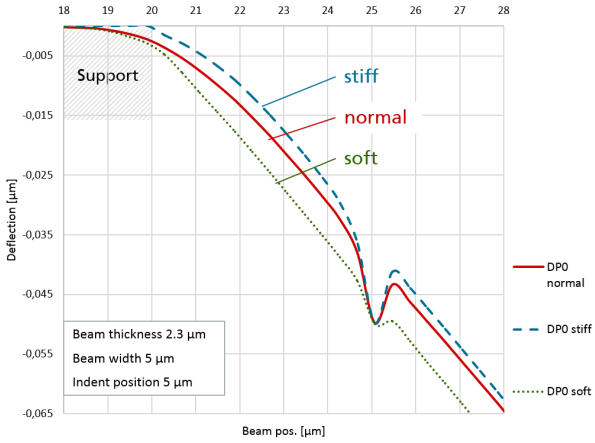

Figure 2. Deflection of the sample from the FEM simulation. The support and the indent position show high displacements. These effects have to be eliminated for precise calculation of the Young's modulus.

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Influence of mechanical stress on leading edge technologies – FEM-modeling for a novel reliability investigation

P37

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Microelectronic devices for automotive applications need a stringent reliability assessment due to a high stress environment, expected long lifetimes, and continuous scaling of new technologies. Thermo-mechanical stress caused by Chip-Package-Interaction (CPI) can lead to catastrophic failures but also affect general transistor and device parameters. To investigate the reliability behavior of the 22 nm FDSOI GLOBALFOUNDRIES technology, ring oscillator test structures are used for a faster analysis of several load conditions and material parameters. Comparable to a previous investigation method developed for the 28 nm technology [1], a mechanical test method using nanoindentation, an electrical test setup and FEM simulation to obtain the critical stress level at the transistor level will be combined. Besides an indentation approach from the rear side after a thinning process of the silicon layer, direct indentation of the Cu-Pillars and additional electrical testing of ring oscillator parameters will be evaluated [2].

Finite element method (FEM) simulations are conducted using the chip layout and semiconductor stack parameters to yield stress/strain fields at the indented area. The model consists of the actual chip device with a thinned Si-layer, Cu-pillars, a finely layered BEOL structure and a spherical tip for the indentation. The obtained stress/strain data is used to define suitable loads for upcoming indentation experiments. The used finite element solver is a deformation-based solver, which means that the deformations are calculated, and then the stress tensor is obtained using the standard constitutive models.

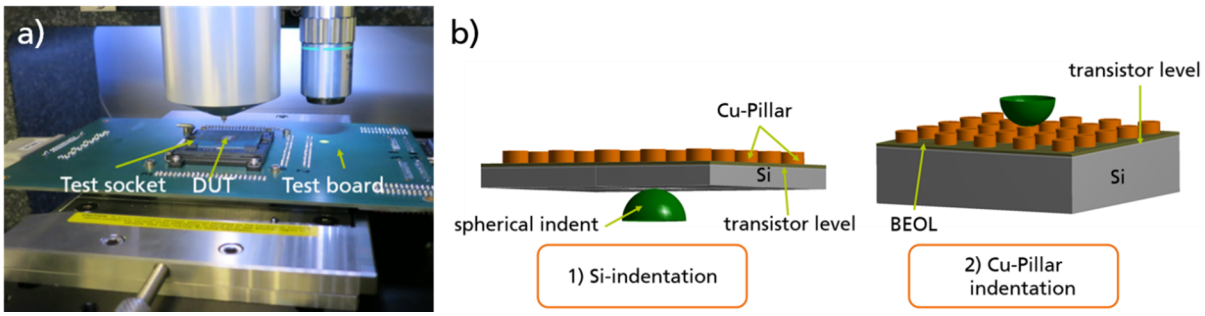

Figure 1. a) Experimental setup for nanoindentation b) FEM–models of Si-chip with Cu-Pillars.

Acknowledgments

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

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Orientation mapping with manometer spatial resolution using "On-Axis" TKD in SEM

P38

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Introduced just a few years ago [1], TKD in SEM, a.k.a. t-EBSD, has already become an established technique due to its much better spatial resolution as compared to standard EBSD [2-3] and to the ever increasing need of analytical tools for characterizing nanomaterials. The original sample-detector configuration using the standard EBSD detector a.k.a "off-axis" TKD had certain limitations resulting in a drop in data quality and/or measurement efficiency. The two most important drawbacks of "off-axis" TKD were the strong gnomonic projection distortions in the patterns and the fact that the patterns were produced by high angle scattering electrons, i.e. very weak signal. These two limitations have removed by the introduction of "on-axis" TKD [4] which uses a modified head of the EBSD detector. The new TKD configuration features a horizontal phosphor screen that is placed under the electron transparent sample so that the SEM' optical axis intersects the center of the screen. This sample-detector geometry allows capturing Kikuchi patterns where signal yield is strongest and with minimized gnomonic projection induced distortions. The improvement in these two acquisition parameters has led to significant gains in spatial resolution, data acquisition speed and data integrity [5].

In this context, the talk will present the latest developments in terms of TKD technique optimization with a focus on its spatial resolution. The most important factors influencing the spatial resolution of TKD in SEM will also be discussed as well as its integration with other techniques like Energy Dispersive X-Ray Spectroscopy (EDS).

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Development and optimization of point-of-care nanobiosensing platforms P39

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The use of inexpensive materials and cost-effective manufacturing processes for mass production of disposable platforms is very attractive and has spurred a variety of approaches. Paper and other polymeric substrates are attractive candidates to be used as low-cost disposable materials for healthcare diagnostic platforms, with great emphasis in point-of-need settings, particularly in resource-poor countries. In here, several nanobiosensing platforms will be presented and discussed:

- **Colorimetric paper-based biosensors:** Lab-on-paper technology is used for the fabrication of colorimetric paper-based biosensors for a multitude of applications, ranging from glucose level control for diabetics to the detection of several diseases, in a very simple, low cost and fast approach. The production method is based in a simple printing process with a wax printer, that renders hydrophobic barriers onto paper, to confine reagents to specific areas and to transport the biologic samples to different reaction zones, by capillarity. This simple process can thus be applied to several biosensors with different designs and purposes putting in evidence the multifunctionalities of this technology [1].
- **Electrochromic devices:** Electrochromic devices are increasing its interest in the last decades due to the wide range of applications, from smart windows to biosensors. In here, a new planar electrochromic device based on tungsten oxide nanoparticles is presented. The mentioned was produced using laser technology for electrodes patterning and with a paper pad inserted in the sensor area for hydration on time of usage, thus replacing the electrolyte material of a typical multi-stack electrochromic structure, eliminating leakage problems, easy integration with other devices and enhancing the shelf life of the devices to several months.
- **MIP-SERS platform for cancer screening:** A new sensor strategy that integrates Molecularly Imprinted Polymers (MIP) for increasing the selectivity, combined with Surface-Enhanced Raman Scattering (SERS) for a higher sensitivity is under development, using low-cost substrates and technologies, such as Laser Direct Writing. It shall provide a simple, reliable, inexpensive, robust, specific and sensitive approach for detection/follow-up of breast cancer disease.

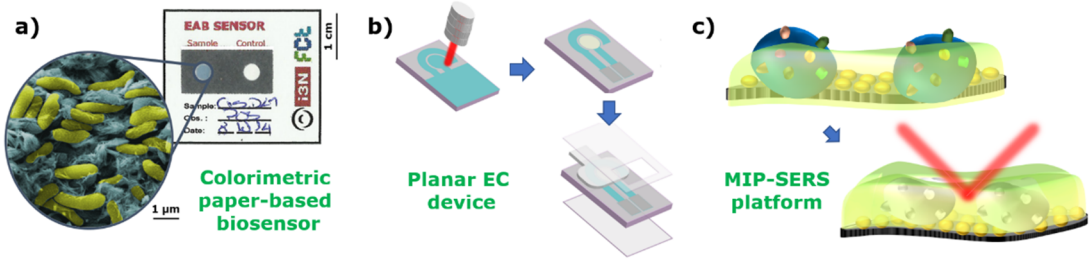

Figure 1. Different examples of nanobiosensing platforms: a) Colorimetric paper-based biosensor for bacteria detection; b) Workflow for the production of planar electrochromic devices; c) MIP-SERS platform for cancer-screening.

Acknowledgments

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Digging deeper into high resolution computed tomography reconstruction

P40

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X-ray computed tomography (XCT) is a non-destructive high resolution imaging technique to acquire the internal structures of an object using a series of X-ray measurements taken from several views around the object. The major challenge of the reconstruction process based on obtained projections (radiographs) is to find the exact correspondence between the voxels from the 3D object and the pixels from the 2D projection. This relation cannot be correctly determined during the scanning step due to existing mechanical instabilities of the XCT system and motions of object. In order to eliminate or at least to mitigate the errors in the reconstruction results, we developed a fully parallel reconstruction software package which offers 7 different iterative and non-iterative reconstruction algorithms for cone-beam and parallel-beam imaging geometries. Artefacts caused by mechanical tool instabilities, misalignment and sample motion are efficiently compensated with the novel 3D machine-vision based correction algorithm. For reconstruction approaches based on an incomplete data set, e.g. in the case of the limited angle tomography, we employed a convolutional neural network for artefact reduction and detail recovery. The software package also includes center of rotation correction and beam hardening correction. The quantitative comparison of the experimental data with simulations proofs that the proposed advanced correction methodology is able to provide accurate and artifact-free reconstruction results.

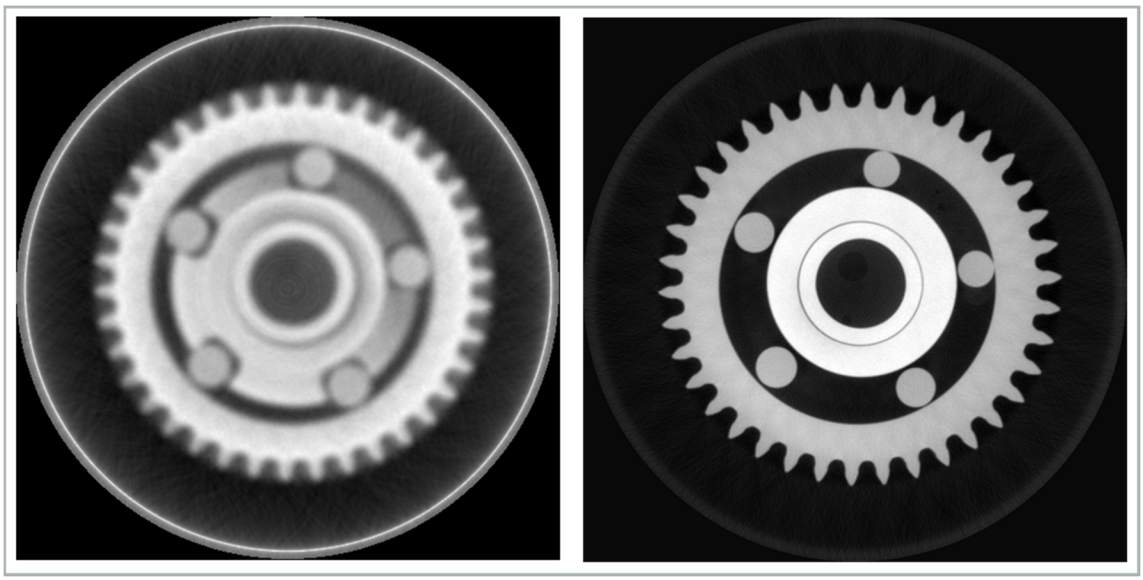

Figure 1. The comparison between uncorrected (left) and corrected (right) reconstruction results of ball bearing sample. For corrected reconstruction, we applied motion compensation, center of rotation correction, offset of detector correction and beam-hardening correction. Both are reconstructed by FDK with 3200 projections and the voxel size is 7.3 µm.

In-situ SEM material characterization of Cu-Sn solder joint system using the Bruker nano-mechanical test platform – PI 87

P41

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In the field of material science, a multi-physics property mapping of materials would vastly improve the understanding of its behavior and performance. Hence an advanced and a semi-automated approach for micro-scale characterization of materials with respect to spatially correlating several physical and chemical properties of the sample is significant. For example the figure shows a two-phase, ferritic (α) and austenitic (γ) stainless steel sample [1]. When these steels are exposed to high temperatures, they further form damaging intermetallic phases like Sigma (σ) and Chi (χ). The grain orientation spread map (right) indicates that the plastic strain field developed by the indent does not cross from the Ferrite grain into the much harder Sigma phase grain. To relate these property maps directly to mechanical characteristics, an in-situ nano indentation device, the Bruker Hysitron PI 87 [2], is used simultaneously within the same vacuum recipient of a SEM-FIB system. In order to achieve the coordination of the two individual stages (i.e. the PI 87 with five degrees of freedom and the SEM stage) with respect to the electron column, EDX and EBSD, several calibration techniques are evaluated. One such technique is the conception of a calibration grid within the sample stub along which the sample to be tested can be mounted. The main purpose of the grid lines is to serve as points of origin upon which the relative distance to the region of interest within the sample could be calculated using the SEM stage input. Further coordinate transformation leads to the necessary indenter positioning with the tested experiments within the SEM vacuum chamber. The tests and methods are further optimized to find a reliable and reproducible stage coordination technique which was then adopted in analyzing and characterization of a complex Cu-Sn solder joint system where high melting component copper forms intermetallics (such as Cu_3Sn and Cu_6Sn_5) with low melting component tin [3].

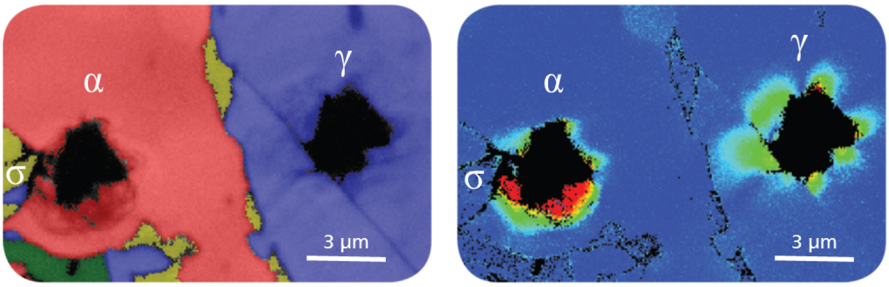

Figure 1. EBSD phase map (left); Grain orientation spread map (right) of a Duplex stainless steel sample [1].

Acknowledgments

This work was done in collaboration with Hysitron/Bruker Corporation.

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The use of mass spectrometry to investigate NPs in biological system

P42

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There is no denying that nanoparticles (NPs) and nanotechnology nowadays become one of the most important issues in both terms of advantages and limitations. NPs are widely applied in a range of fields. Unfortunately, this can lead to a viable threat since NPs can ubiquitously disperse into an ecosystem. The release of a high amount of engineered nanomaterials (ENMs) into soil and water can be unbeneficial for the growth of terrestrial plants, especially for agricultural crops. Recently, the effect of NPs on plants has been intensively studied. NPs interaction with plants depends on various factors such as: their concentration, type, size, shape, specific surface area, and stability. Moreover NPs can change their behaviors via dissolving and agglomerating. Their transportation from manufacture to our hands, their exposure to light, and temperature or to culture medium cannot guarantee that their original characteristics such as chemical composition and particles size will be constant overtime. Therefore, the characterization and detection of NPs before and after being applied to plants is equally important. The information obtained from the NPs characterization is really helpful in order to explain the reasons behind their phytotoxicity in plants.

The aim of our work is to extend the capabilities of the methodology used for the characterization of nanomaterials beyond the present state of the art. Various features of mass spectrometry will supplement each other towards raising complementary information which extends the capability to characterize nanoparticles (NPs), especially in a view of their interaction within biological systems. In our experimental perspective, a model plants with well-characterized processes of biotransformation will be exposed to NPs of various composition and characteristics. A measurement procedure for characterization of NPs are as follows: (1) inductively coupled plasma mass spectrometry (ICP-MS): single particle ICP-MS and single cell ICP-MS to study the distribution and the uptake of NPs by plants; (2) laser ablation coupled with ICP-MS (LA-ICP-MS) to investigate the distribution of NPs deposited on solid support, nanomaterials aggregation processes, and NPs uptake and their tissue level distribution in plants; (3) multicollector-ICP-MS (MC-ICP-MS) to study isotopic ratio of selected elements in individual NPs and their isotopic fractionation in plants; (4) gel electrophoresis (GE) coupled with LA-ICP-MS to investigate possible NPs-bounding proteins in plants exposed to NPs.

In order to check the performance of such a novel methodology of single particle ICP-MS, preliminary studies were conducted in our laboratory. For this purpose, various suspension of Au and Ag nanoparticles were examined. The intensity of the signal pulse is directly proportional to the mass of the detected nanoparticle, and thereby to the nanoparticle's diameter.

Characterization of crystallographic relationships at the interfaces of biocomposite mollusk shells

P43

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Mollusk shells have several superposed layers consist of an organic, soft matrix reinforced by an inorganic and hard-mineral phase which is mineralized calcium carbonate in two polymorphic variants: trigonal calcite and orthorhombic aragonite. Despite the small volume representation, approximately, the organic fraction plays a key role in the shell growth process and later in dynamic or static loading conditions. This fraction, mainly composed of proteins, polysaccharides and lipids, determines the atomic structure of the mineral - hard part which constitutes 95-99,9% of shells mass. The organic phase decides about the size, shape, and polymorph form of $CaCO_3$. It is a matrix in which calcium carbonate crystallites locate [1-3]. Mollusk shells possess high strength and resistance to brittle fracture at a relatively low mas. Hence, the shell becomes a natural inspiration for the design and manufacture synthetic composites with desirable mechanical properties.

The structure of these biocomposites is well-ordered, implemented from the nano to macro level, in accordance with a certain idea. The 90% of all mollusks prefer crossed-lamellar structure. This structure is stacked by the 1st-order lamellae, which are further composed of laths of parallel mineral fibers. The fibers are parallel within a given 1st-order lamella but almost perpendicular to those in the neighboring 1st-order lamellae. The unique construction of the crossed-lamellar structure provides multiply and complex interfaces at different levels of lamellae, which have great influence on mechanical properties of these materials [4].

The investigation of mollusk shells microstructures was performed using electron backscatter diffraction (EBSD) in scanning electron microscope and electron diffraction in transmission electron microscope. It came out that the selected species of mollusks prefer a strictly defined set of misorientation. Among misorientations with high frequencies, there are twins observed in synthetic calcite, as well as others of which has not been shown in the literature so far.

Acknowledgments

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Animal origin functional tissue elaborated for human substitutes P44

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Tissue engineering is one of the most promising methods to create therapeutic solutions for the heart valve substitutes. Currently available prosthetic heart valves have been successfully used clinically; however, they have several limitations. As an alternative for the creation of heart valve bioprostheses, tissue engineering techniques can be used. These modern methods use biological scaffolds subjected to an acellularization procedure followed by cell seeding.

The aim of this study was to determine the effect of different acellularization process on morphological properties of pericardium and aortic valve prepared using tissue engineering techniques to achieve optimal conditions to obtain stable heart valve prostheses. Evaluation of these parameters can determine the success of the clinical application of tissue-engineered heart valves. Pericardium was collected from adult porcinis. Properly crafted tissue was subjected by the acellularization process, which means the removal of cells from the tissue in order to obtain pure, non-degraded extracellular matrix (ECM). Elimination of cells from the tissue was carried out by Trypsin/EDTA (Ethylenediaminetetraacetic acid) followed by SDS (Sodium dodecyl sulfate) and some trials were executed associated with the development of the unconventional methods of tissue decellurization involving a laser ablation with different power and beam configuration and acoustic waves with different frequencies.

After reaching the final form of the surface functionalization the tests were carried out in direct contact with human blood. The protocol of pericardium acquisition from pig's heart has been developed. The decellular tissue was tested using scanning electron microscopy (FEI Quanta 3D FEGSEM), confocal laser scanning microscopy (LSM Exciter 5) and scanning acoustic microscopy (SAM). The effectiveness of the acellularization process was analyzed using histological staining. Acellular tissue was examined for the generation of shear forces in a direct, dynamic test with full human blood. The tests of the surface after blood tests were completed using confocal microscopy and flow cytometry. The results showed significant efficiency of detergent method, there was no nucei. The analysis was carried out under the full control of the histological and molecular analysis. The issue of the cell elimination from tissue, and then formation of new tissue on the basis of ECM meets strong clinical interest, but at the moment states a complex issue from a scientific point of view.

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

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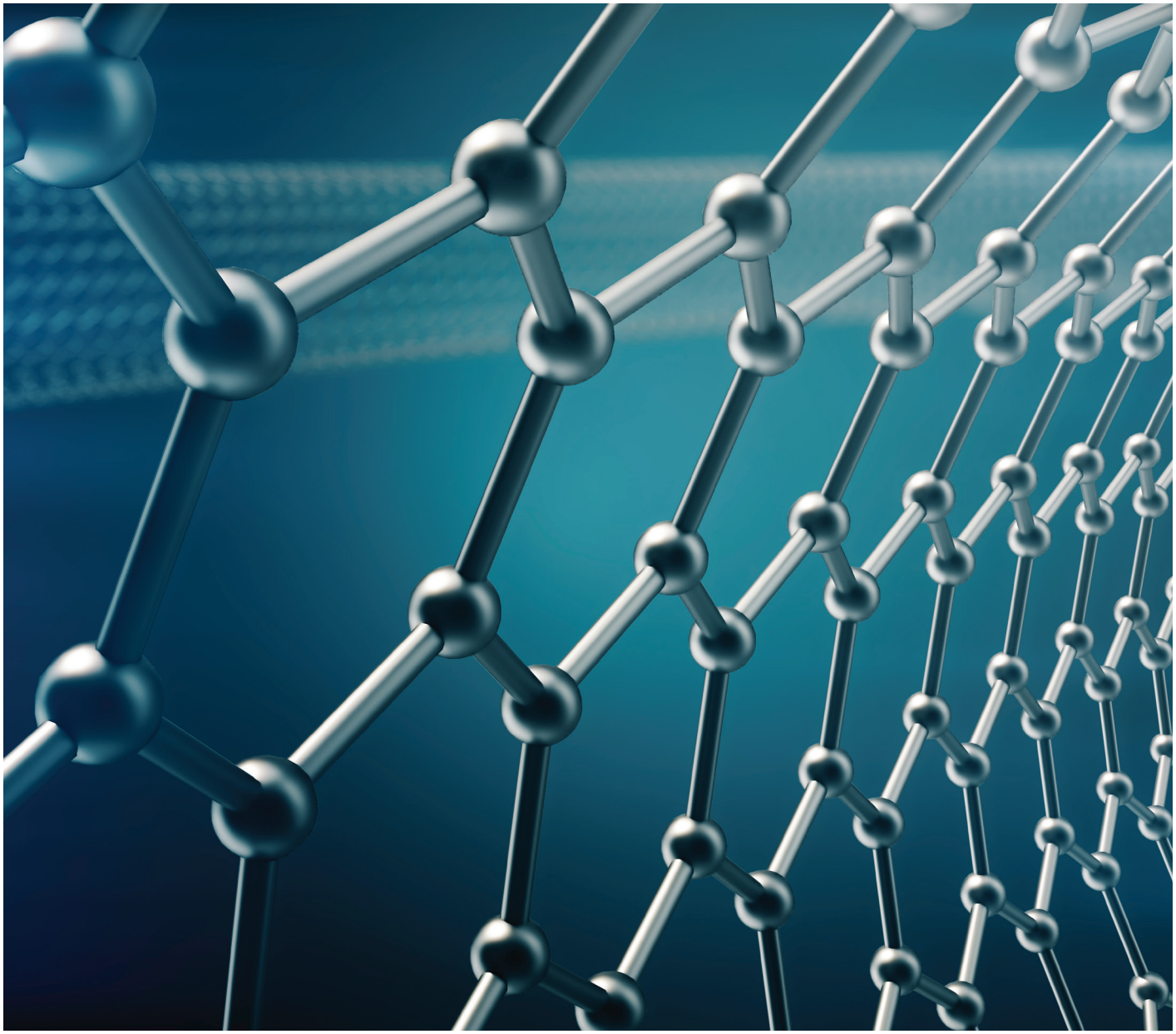

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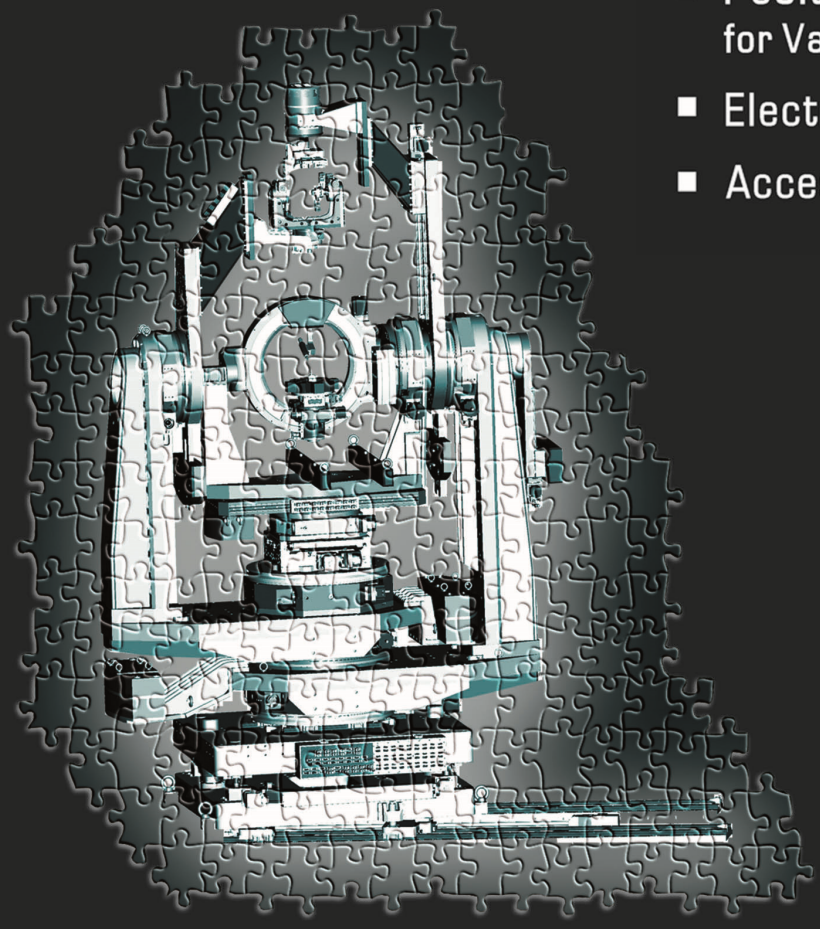

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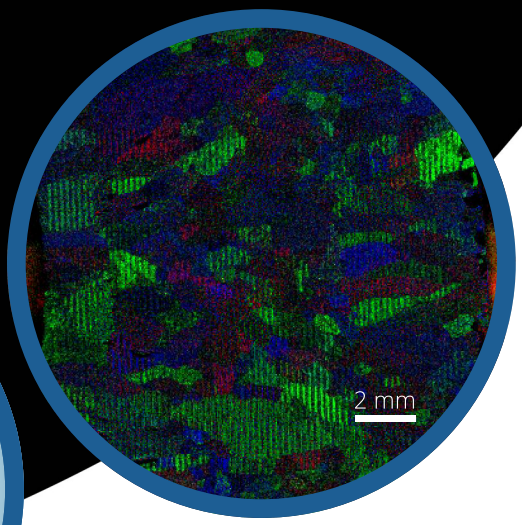

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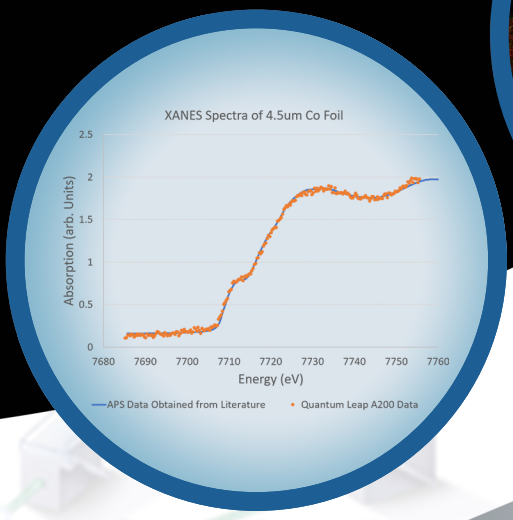
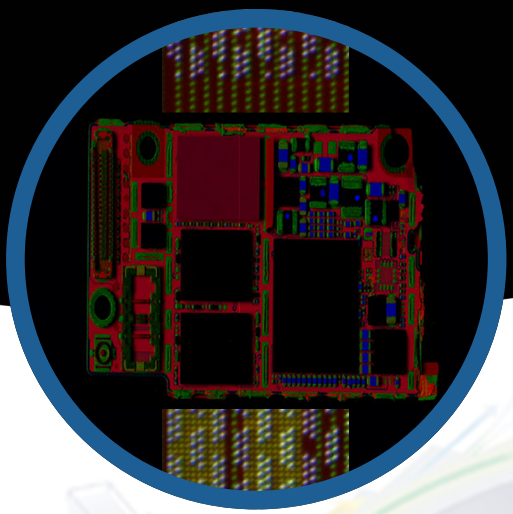
Metal Oxidation States



ELECTRONICS
Dopants & LIDAR
Thin Film (nm) Measurements



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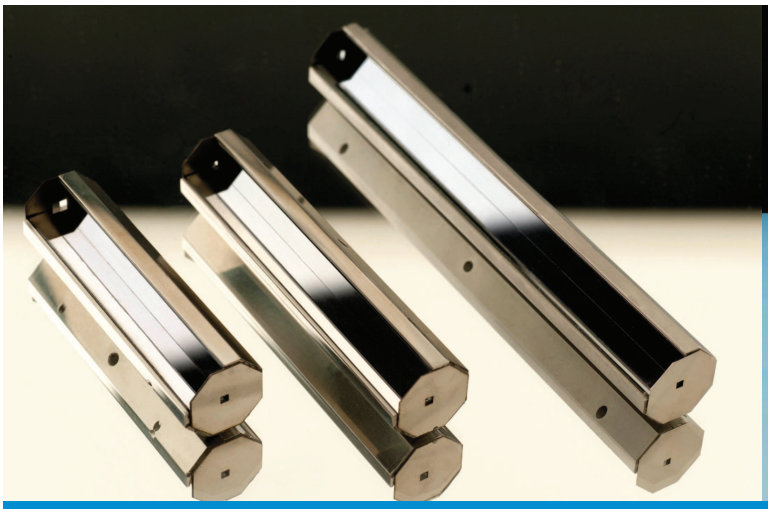
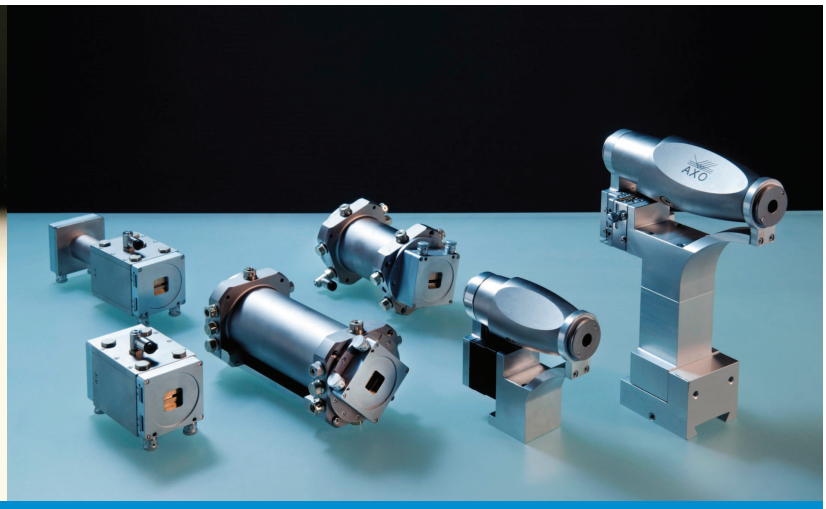
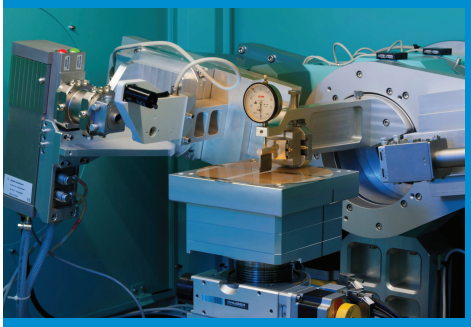
APPLIED X-RAY OPTICS

AXO

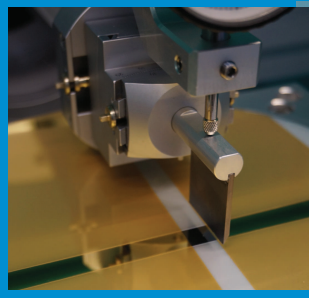
Multilayer X-ray Optics
High Precision Deposition

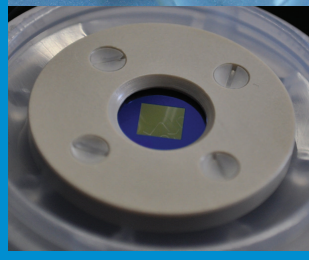


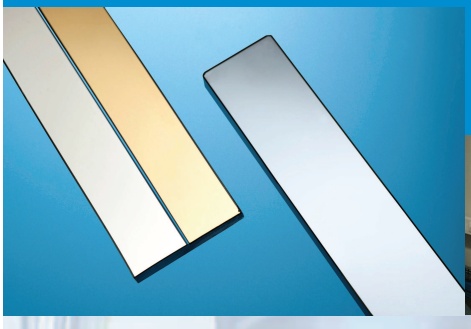


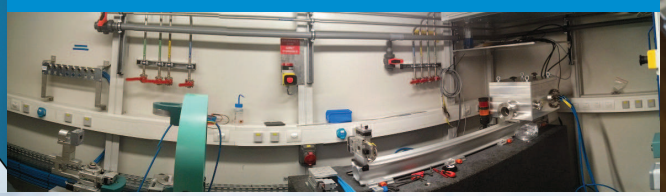


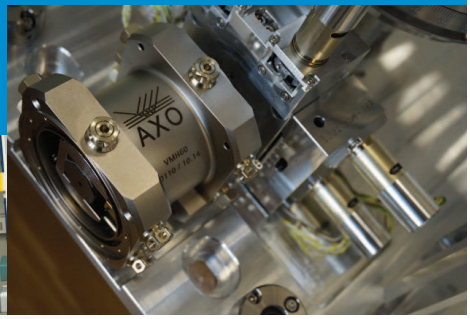
- One- and two-dimensional focusing and parallel beam multilayer X-ray optics for X-ray reflectometry, high resolution and single crystal diffraction
- Up-grade of all common X-ray systems
- Multilayer synchrotron optics from EUV to the hard X-ray region up to a substrate length of 1000 mm
- Tailored micro-focus X-ray source systems with adapted 1D or 2D optics
- Sub-nanometer precision large area deposition
- Thin film multi-element reference samples for XRF











AXO DRESDEN GmbH
APPLIED X-RAY OPTICS
MULTILAYER X-RAY OPTICS
HIGH PRECISION DEPOSITION
MORE THAN 20 YEARS EXPERIENCE IN X-RAY OPTICS AND HIGH PRECISION DEPOSITION

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