

AN INITIATIVE IN NANOTECHNOLOGY AND NANOMATERIALS



CONTENTS

- 1 Preface
- 2 DFCNA – A recognized partner for industry
- 4 Nanoanalysis for progress in nanotechnology and nanomaterials

- Case studies**
- 6 Copper/diamond composites for heat sink applications
- 8 Improved transistor degradation models to predict circuit lifetime
- 10 Analytical TEM for product-related material development
- 12 Crack imaging in composite materials using high-resolution nano-XCT
- 13 Measurement of micromechanical properties with the FIBDAC method

- 14 Microscopy and analysis – tradition in Saxony
- 15 A center for research and innovation – integral part of the DRESDEN-concept with European orientation

- 16 Members, partners, steering committee, management
- 17 Publication data

Front cover

Analytical transmission electron microscope for materials characterization, TEM.

Back cover

High-resolution X-ray laminography.



PREFACE

The importance of nanoscience and nanotechnology in all aspects of everyday life is becoming widely acknowledged. Health, medical technologies, energy, textiles, automotive, chemistry and/or processing are some of the sectors where nanotechnology is having a tremendous impact on people.

Fully aware of its potential, the European Union invested more than 896 million euros in nanotechnologies under its 7th framework programme for research (FP7) through the dedicated nanoscience, nanotechnologies, materials and new production technologies programme (NMP). The objective was to support knowledge creation in nanotechnology and nanoscience to transform technologies and make them smarter, more efficient and environmentally friendly.

Horizon 2020 draws on the research foundations of FP7 and pursues the ambition to turn Europe's intellectual capital into commercial products for growth and job creation. The main challenge was to bridge the gap between nanotechnology research and market applications, paying close attention to match safety requirements for citizens' health and environmental protection. Building on FP7, Horizon 2020 – the 8th EU research and innovation programme – is addressing the European shortfall in technology transfer, putting the EU in the driving seat for the commercialization of emerging nanotechnologies across different industry sectors and successfully tackling grand societal challenges.

The recently launched initiative of open innovation test beds in nanotechnologies and advanced materials goes one step further and lays the foundations for the creation of a European innovation ecosystem for the design, development, testing, and upscaling of advanced materials and nanotechnologies.

This should enable a vast array of applications and facilitate innovators to bring disruptive ideas to the market. Success will take the form of an effective and functioning eco-system allowing innovators to respect the EU regulatory framework while having the means to develop innovative and safe products incorporating new materials and nanotechnologies.

Regional initiatives such as the Dresden Fraunhofer Cluster Nanoanalysis DFCNA in Saxony are key contributors to the creation and the success of such a European materials and nanotechnologies innovation ecosystem. Seven Fraunhofer Institutes, Technische Universit at Dresden and Helmholtz-Zentrum Berlin have created an internationally visible competence centre with an important range of key competences in nanoanalysis, metrology, and related modelling as a fully recognized and indispensable partner for industry.

Such an initiative can be considered a prime example of the process towards the development of an open innovation test bed at EU level that can, with a single entry point, offer facilities, knowledge and services across Europe and beyond for the benefit of economy and society.

H el ene Chraye

Head of Unit Advanced Materials and Nanotechnologies
European Commission | DG RTD - Unit D3 | Directorate for
Industrial Technologies

DFCNA – A RECOGNIZED PARTNER FOR INDUSTRY

Consolidated nanoanalysis competence in Dresden

The Dresden Fraunhofer Cluster Nanoanalysis DFCNA, an internationally visible competence center for nanoanalysis and a recognized partner for industry, performs applied research and development in the field of nanoanalysis to discover suitable technical and conceptual solutions, including:

- Development of new analysis techniques and advancement of existing ones
- Development of components and systems for new analysis techniques
- Development of application strategies for advanced analysis techniques and systems
- Consulting and services in the field of analysis for high-tech companies
- Hosting specialized working groups in e.g. micro-/nanomechanics and X-ray techniques.

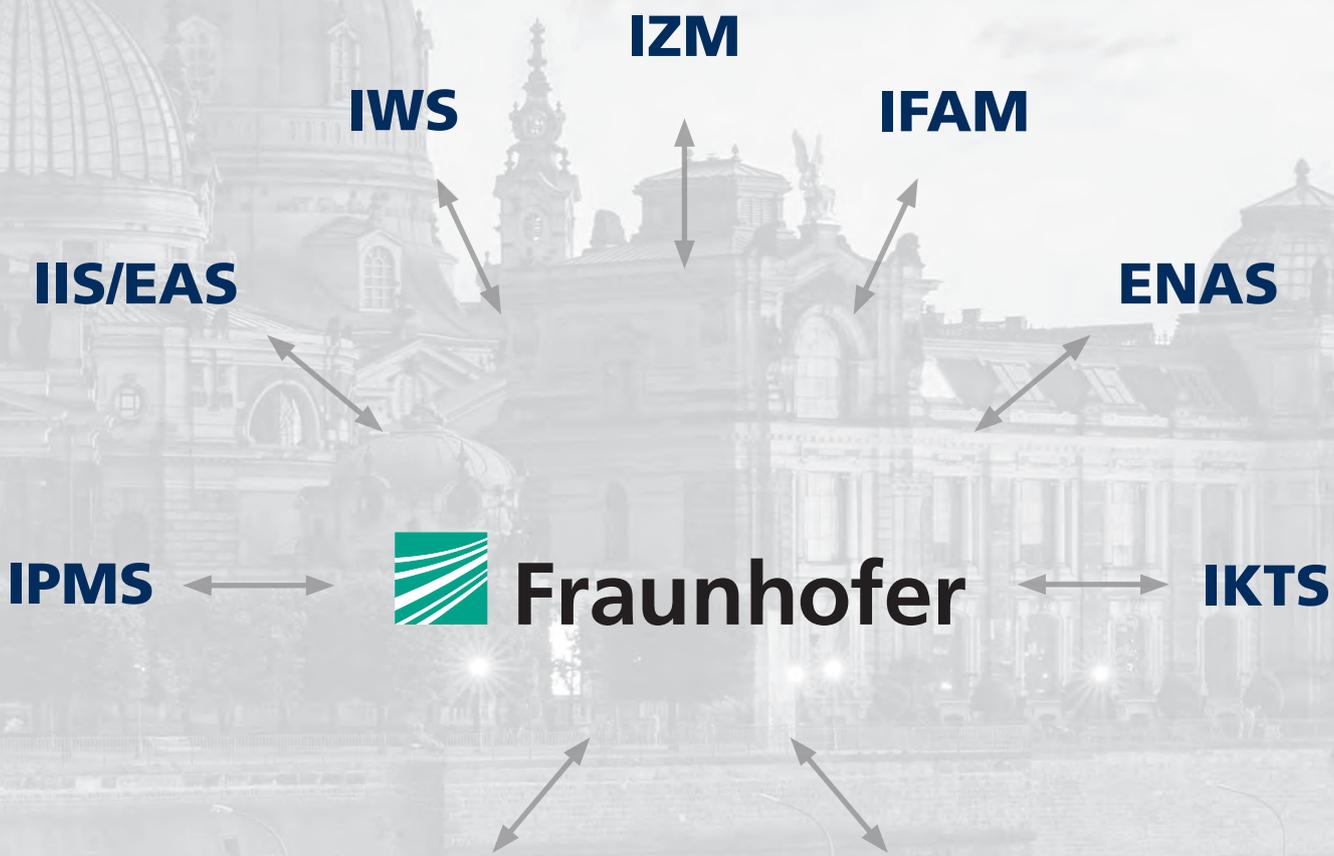
The application areas covered are micro-, nano- and optoelectronics, energy storage and conversion as well as lightweight construction and functional materials. Seven Fraunhofer Institutes and the Technische Universität Dresden as well as the Helmholtz-Zentrum Berlin bring together their competences and cover the complete range of topics in the field of nanoanalysis. The institutes are flexibly linked and cope with even the most complex project requirements.

Process and quality control – a need for modern manufacturing and high-tech products

Nanoscience and nanotechnology will provide important basic approaches for improved functionalities and performance of products, particularly in high-tech industries. In this scenario, materials will play a key role. It is expected that nanoscale

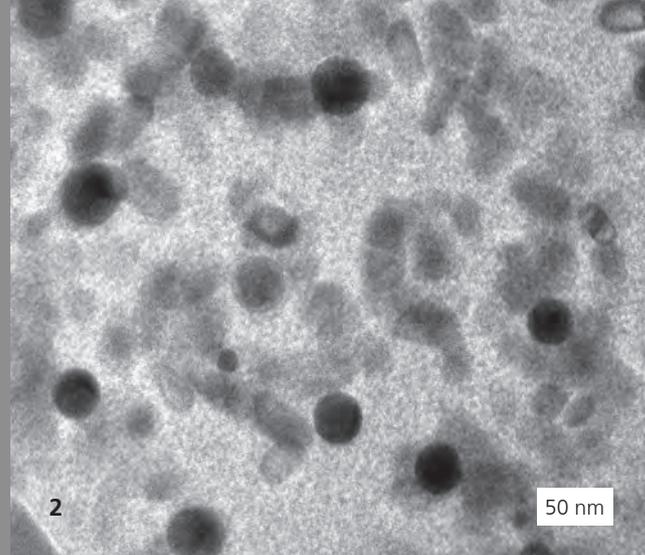
materials will fundamentally change products and manufacturing processes over the next two or three decades. Nanoscale materials are at the cutting edge of virtually every innovation in key technologies and high-tech products necessary to meet the demands of the market. Therefore, materials research and development, including nanoscale characterization of novel materials, are competitive enablers for a wide range of key technologies. Our network of Fraunhofer Institutes, together with Technische Universität Dresden and Helmholtz-Zentrum Berlin, provides an excellent platform to reinforce science-industry dialogue and to offer new attractive scenarios for fruitful collaborations between academia and industry.

For both process and quality control as well as for reliability engineering, novel analytical techniques must be developed and existing ones must be enhanced and adapted, with regard to specific needs. Furthermore, in high-volume manufacturing industries, time-to-data must be minimized for efficient process and quality control. These requirements become increasingly complex, as often more than one analytical technique has to be applied. Hybrid metrology is a novel approach in industry associated with the need to manage big data. With the integration of new materials into products, often novel processes have to be studied and evaluated for their reliability. In such cases, only a dedicated team of scientists and engineers is able to provide appropriate solutions. The Dresden Fraunhofer Cluster Nanoanalysis brings together a strong team of motivated staff, with the express goal of providing application-specific solutions in the field of nanoanalysis. More than 100 scientists, engineers, students and technicians contribute to research and development projects developing new equipment, and providing analytical services.



 TECHNISCHE
UNIVERSITÄT
DRESDEN

 **HZB** Helmholtz
Zentrum Berlin



NANOANALYSIS FOR PROGRESS IN NANOTECHNOLOGY AND NANOMATERIALS

Highly sophisticated products need nanotechnology and nanomaterials

An increasing number of new products and services rely on new design concepts, new technologies, and new materials. Future manufacturing based on advanced nanotechnology at very small length scales, new device structures for nanoelectronics, and advanced materials are proving to be challenges for process development and control as well as quality assurance. Advanced nanoanalysis techniques will play a key role in the characterization of thin films, nanostructures, surfaces, and interfaces. It is necessary therefore, to sustain developments in nanoscale characterization as well as in nanotechnology and materials science and engineering, with a particular focus on advanced thin film and nanoscale materials.

Nanotechnology – a modern approach, but with a long history

Nanotechnology has deep historic roots. It is fascinating that nanoparticles were used by artists to generate brilliant colors in glass artifacts and artwork long before scientists recognized the unique optical properties of metal nanostructures. The Lycurgus cup, dating back to the Byzantine Empire in the 4th century, is an example of Roman glass work. The cup appears green or purple, depending on whether it is observed in reflecting or transmitting light. The brilliant colors of medieval stained glass, visible in numerous churches in Europe, and the beautiful design of Meissen china are other such examples. Meissen china contains gold particles of sub-100 nm size. In all these cases, gold nanoparticles of different size were embedded in glass. Today, these gold nanoparticles are visualized and their diameters determined in a transmission electron microscope. Based on Maxwell's theory, these optical

phenomena can be explained by surface plasmons, i.e., electromagnetic excitations that propagate along the interface between a metal and a dielectric medium.

Scientific interest in the electromagnetic properties of metal-dielectric interfaces is steadily increasing, motivated by improved nano-fabrication techniques, such as extreme ultraviolet and electron lithography or ion beam milling, and by advanced characterization techniques, such as near-field microscopy. Metal nanowires, ordered arrays of metal nanoparticles, and grooves in metal films can serve as light guides for manipulating the propagation of light at the nanoscale. Current applications of surface plasmonics include nanoscale plasmon waveguides, antennas for novel biochemical sensors, and efficient solar cells. Superlenses proposed by Pendry are a promising development which will allow imaging of objects with sub-wavelength dimensions. Synthesis, manipulation, and characterization of metallic nanoparticles and nanostructures are key tasks for plasmonics – from research to industrial applications.

Plasmonics – New approaches in nanoanalysis

In recent decades, developments in the field of nonlinear optical experiments and plasmon optics have become possible due to the ability to fabricate nanoscale metallic structures in a controlled fashion. The presence of gold or silver nanoparticles results in a Raman scattering signal that is enhanced by many orders of magnitude due to the resonant excitation of plasmons. This surface-enhanced Raman effect is used, for instance, in tip-enhanced Raman spectroscopy to determine strain with sub-wavelength resolution in silicon channels of leading-edge field-effect transistors.

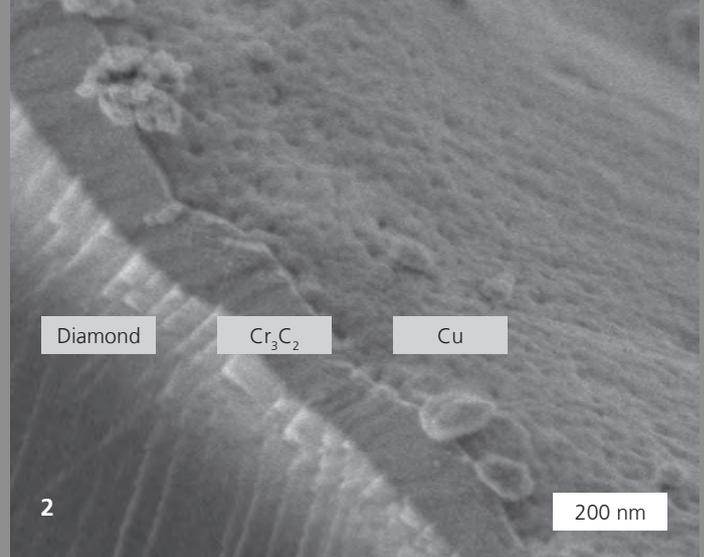
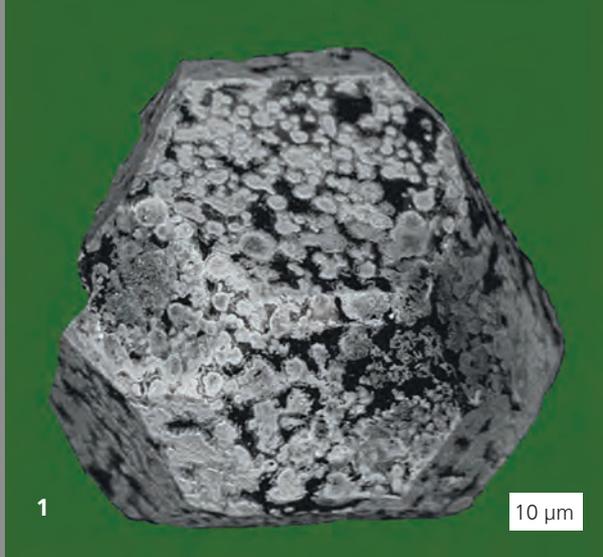


1 A plate of Meissen china painted with Rosenpurpur (Rose Purple) color.

2 A bright-field TEM image showing metallic gold nanoparticles in a glass matrix that give Meissen china its unique Rosenpurpur color. The circular features show a large distribution of spherical gold particles, with an

average diameter of 27 nm. The discovery of the Rosenpurpur overglaze color can be traced back to the chemist and long-standing director of the Meissen porcelain factory Johann Gregorius Höroldt (1696–1775).

3 Stained glass (window in the Notre-Dame de Chartres cathedral).



COPPER/DIAMOND COMPOSITES FOR HEAT SINK APPLICATIONS

Thermal aspects are becoming increasingly important for the reliability of electronic components driven by continuous progress of the electronics industry. Effective thermal management is therefore a key issue in the packaging of high performance semiconductors. The ideal material for a heat sink and heat spreader should have a coefficient of thermal expansion (CTE) of $4\text{-}8 \times 10^{-6}/\text{K}$ and high thermal conductivity. Metal matrix composites (MMCs) offer the possibility to tailor the properties of a metal by adding an appropriate reinforcement phase thus meeting the demands for thermal management.

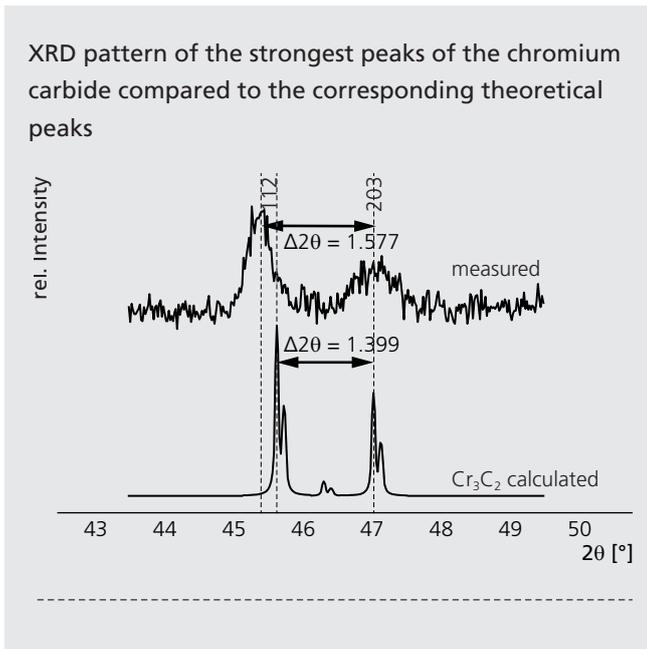
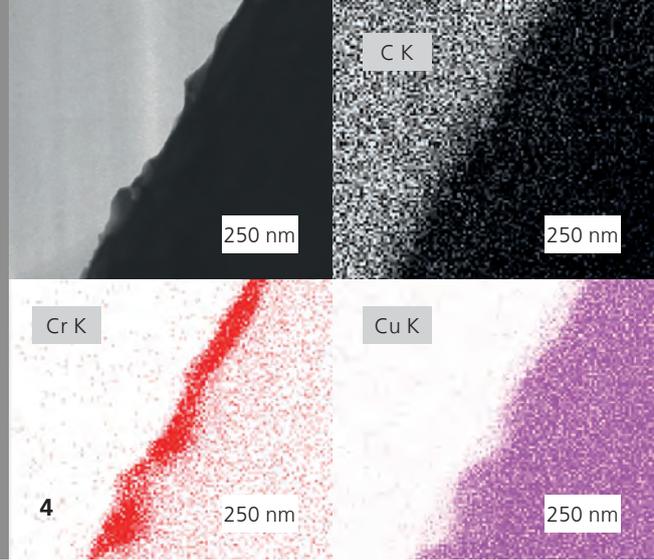
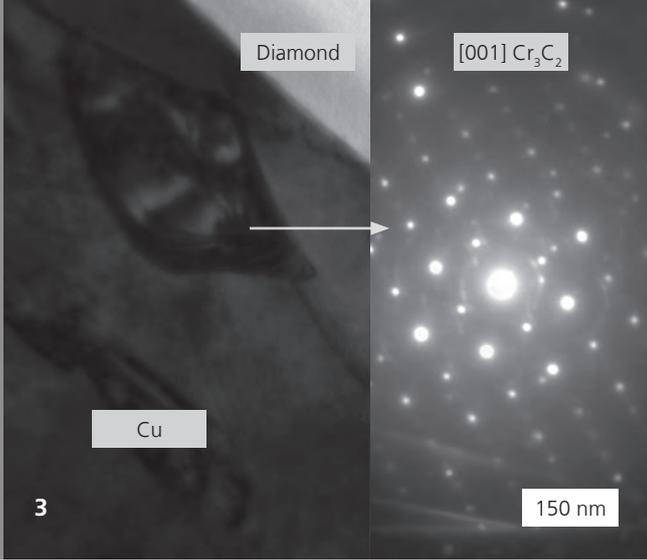
When diamond particles are embedded in a copper matrix, the interface plays a crucial role in determining thermal conductivity, CTE and mechanical properties of the composite. The ideal interface should provide good adhesion and minimum thermal barrier resistance. Pure liquid copper (Cu) does not wet diamond and pure Cu/diamond composites have been shown to feature weak interfacial bonding; debonding occurs upon thermal cycling. It is well known that alloying of copper with a strong carbide-forming element like Ti, Cr, B or Zr promotes wetting and bonding of diamond. Electrons dominate heat conduction in copper, whereas phonons dominate that in diamond. Hence, for heat conductivity of the metal matrix composite (MMC), energy transfer must occur between electrons and phonons. A very thin interface layer of a carbide phase can presumably aid the necessary electron-phonon coupling.

In order to solve the interface problem between copper and diamond, the use of different carbide formers as alloying elements to the copper matrix is being investigated. High thermal conductivities were achieved with diamond reinforced

CuCr matrix composites. Fast pressure-assisted sintering of the corresponding powder mixtures with heating/cooling rates of 100-150 K/min and holding times up to a few minutes result in the most promising thermal conductivity values compared to conventional hot pressing with heating/cooling rates of about 10 K/min. Experimental results confirm that alloying the copper matrix with the carbide-forming element chromium delivers better thermal properties in CuCr/60% diamond composites compared to a pure copper matrix. Without alloying, rather low thermal conductivities of the composite (~ 200 W/mK) were measured indicating high thermal boundary resistance. This is because there is no chemical affinity between copper and diamond, and therefore it is difficult to produce a bond of low thermal resistance and high mechanical strength between the matrix and the reinforcement. A high thermal conductivity of about 640 W/mK along with a CTE of about $7 \times 10^{-6}/\text{K}$ was achieved in the CuCr/diamond composites.

A detailed study of the interfaces was done on diamonds released by simple chemical etching with nitric acid from the composites. X-ray analyses reveal the formation of Cr_3C_2 as the main reaction product in CuCr/diamond composites. Cr_3C_2 is the thermodynamically stable reaction product that forms between chromium and carbon. The XRD patterns also reveal a shift of the (112) peak of Cr_3C_2 indicating a larger lattice parameter b compared to the equilibrium orthorhombic carbide. Using the half peak width a crystal size between 20 and 40 nm in the interfacial carbide was estimated.

SEM pictures using the BSE signal of the interfaces in the released diamonds show carbide formation on all diamond faces – {100} and {111} (Fig. 1). This carbide interlayer with



a thickness of about 100 nm is clearly visible in the SE mode using HR-SEM (Fig. 2). TEM observations, performed on FIB processed thin foils, revealed the nanocrystalline structure of the interlayer. Electron diffraction analysis revealed the presence of chromium carbide (Cr_3C_2) at the Cu/diamond interface (Fig. 3).

EDS element mapping of the interface area of a FIB processed sample shows high chromium content in the carbide interlayer (Fig. 4). These analyses confirmed the continuous nature of the thin carbide layer also observed at the fracture surface.

This work demonstrates that high thermal conductivities are possible with diamond reinforced CuCr matrix composites. Controlling the interfacial reaction results in the formation of a thin Cr_3C_2 layer, and this is necessary to enable the manufacturing of Cu/diamond heat sinks with high thermal conductivities. Obviously, rapid heating (during directly heated hot pressing) can cause a smaller critical radius and a higher number of nuclei for carbide formation resulting in finer and smoother interfacial structures compared to conventional hot pressing. This presumably correlates with the achieved higher thermal conductivity of the composites.

- 1 SEM image showing formation of chromium carbide at the surface of a diamond particle.
- 2 HR-SEM image of the fracture surface of hot-pressed CuCr/60% diamond composite using SE signal.
- 3 TEM image of the interface, exposed by FIB milling in the CuCr/60% diamond composite and the electron diffraction pattern of the interfacial carbide.
- 4 EDS element mapping at the interface – FIB milled specimen of a CuCr/60% diamond composite.

IMPROVED TRANSISTOR DEGRADATION MODELS TO PREDICT CIRCUIT LIFETIME

Ongoing developments in manufacturing processes of integrated circuits have seen a progressive shrinking of the individual features. Ever smaller component sizes as well as new materials create physical effects that can actually hinder circuit operation. Process variations and parameter degradation are two significant examples of such effects. While variations lead to differences in the operation of individual circuits right after production, parameter degradations modify circuit properties over their functional life. Moreover, external environmental conditions like thermal or mechanical stress affect the electrical behavior of semiconductors.

As a consequence, there is a danger that integrated circuits can deviate significantly from several specified characteristics over their functional lifetime. This usually leads to failure of the electronic components during operation. This needs to be prevented, particularly for safety-critical systems, in order to avoid fatal failures in the field. Appropriate verification processes are required to cope with these effects already at the design stage.

Today's design tools and environments need to be improved to ensure that extended lifetime targets can be verified for each circuit under the conditions of its intended application.

In order to avoid failures of circuits during operation, it is important to understand the physical cause behind the parameter deviations. Furthermore, it is essential to analyze their influences on device and circuit behavior. This enables circuit designers to optimize the circuits to meet the specifications under the given application conditions.

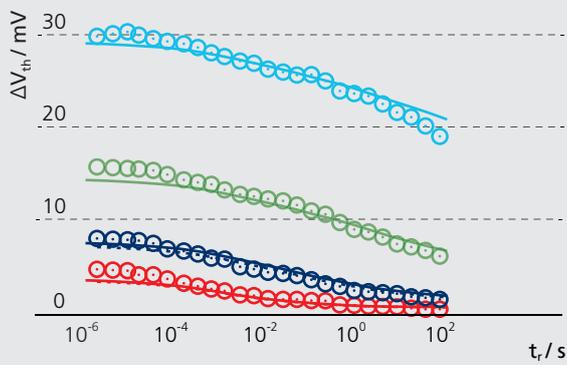
The EU FP7 research project MoRV ("Modelling reliability under Variability", grant agreement no. 619234) investigates in detail the physics of semiconductor degradation mechanisms and employs this knowledge into circuit level models.

The project contribution of Fraunhofer IIS/EAS was the development of an abstraction process to establish efficient compact models of real device behavior. This abstraction process was based on the whole spectrum of semiconductor physics including quantum effects. Extensive computations from a project partner provided a detailed understanding and statistical confidence. Proceeding from these analyses, the scientists of Fraunhofer IIS/EAS derived abstract models to represent the important features of device physics. The main challenge was to generate accurate transistor-level aging models that are fast enough to be used in simulations of full-chips comprising thousands of devices.

Technology-aware degradation model

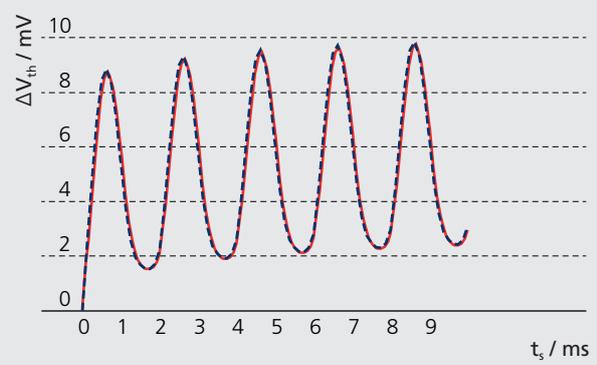
In the analysis, we concentrated on one major degradation mechanism in advanced semiconductor technologies: negative bias temperature instability (NBTI). The origin of deviations from the device characteristics are defects in the gate oxide as well as in the interface to the silicon channel. These traps can be either charged or discharged, which affects the device threshold voltage and thereby overall circuit functionality. Charging and discharging occurs on a statistical basis and depends on device bias, device geometry and junction temperature. Extensive characterization of devices using a typical analog/mixed signal technology was done by a project partner for different values of the influencing factors. Statistical confidence was reached by performing a large number of

Relaxation of threshold voltage shift after stress



2
Sine 125°C, 2 kHz
 $t_s = 10^4s, t_s = 10^2s, t_s = 10^0s, t_s = 10^{-2}s$

Threshold voltage shift for sine wave stress signal



3
— compact - - TCAD
Sine 125°C, 500 Hz

TCAD (Technology CAD) simulations, which were calibrated with measured device characteristics. Unfortunately, stress measurements can only be executed for single devices, as electrical overstress is impossible for a functional circuit. TCAD simulations are highly time consuming and therefore allow analyzing device behavior only at short time scales - of the order of seconds. To determine the behavior of entire circuits or systems-on-chip, completely new approaches are needed that accelerate the simulation by several orders of magnitude, while maintaining comparable levels of accuracy.

Circuit level compact model

Based on detailed physical behavior, a mathematical model was established that represents the statistical probabilities of the charging and discharging processes including their voltage dependencies. For this mathematical representation in the form of an ODE (Ordinary Differential Equation) a very efficient solution was found using numerical integration. Depending on the size of the device in the technology at hand, the model takes a subset of defects from a large database and computes the change of the characteristics for the whole device. Due to its numerical efficiency the developed algorithm is able to predict the behavior of a circuit over several years of operation.

The current purely digital NBTI models account for only two stress levels. In contrast, the approach of Fraunhofer IIS/EAS is capable of taking truly analog signals (e.g. sine wave) into account and therefore applies to analog circuit design as well.

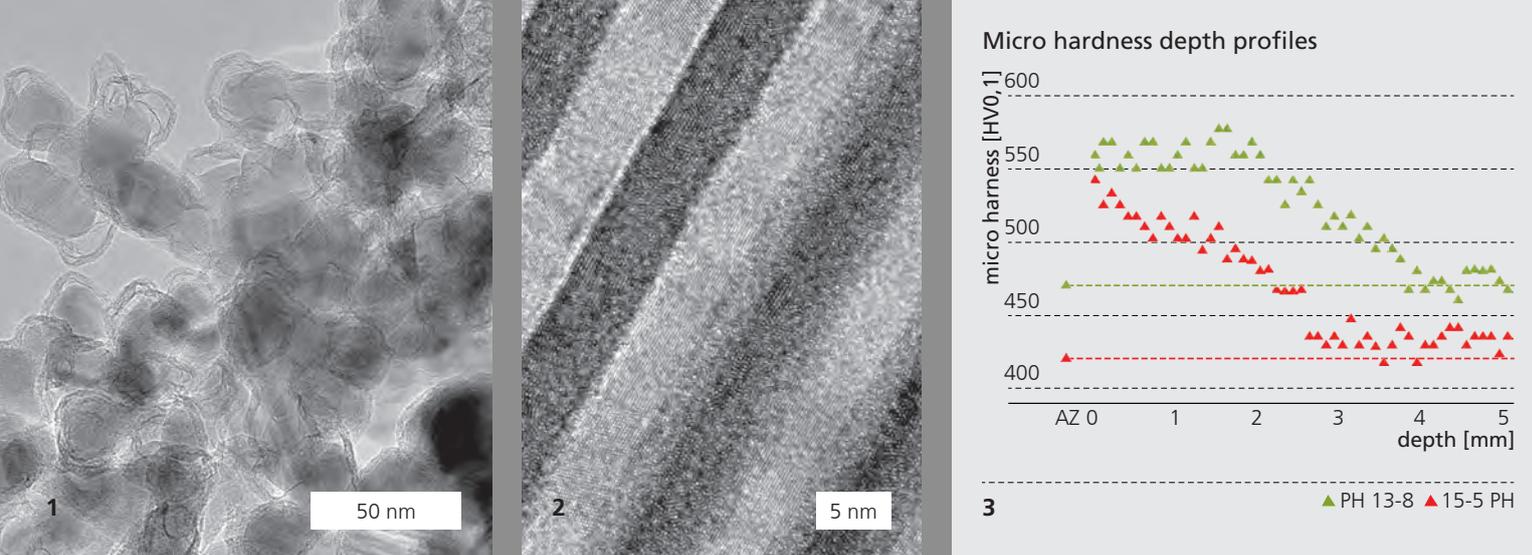
Results

As a result of model development and efficient implementation, we demonstrated that detailed TCAD simulation out-

comes as well as measurements can be accurately reproduced at the transistor level. As opposed to previous approaches, the model of Fraunhofer IIS/EAS allows simulations of several years of operation. This analysis can be done within milliseconds – as well as extensive statistical analysis for different defect configurations and device geometries.

This fast and efficient compact model for the NBTI degradation effect enables circuit designers to analyze the long-term behavior of a circuit under realistic load conditions during operation. Circuit optimization can be performed by assessing and reducing reliability margins and thereby avoiding unnecessary overdesign. At the same time functional verification assures reliable operation over the entire lifetime.

- 1 *Distribution map of time constants for gate oxide traps.*
- 2 *The developed compact model (solid lines) fits the voltage shift relaxation characterized by measurements (circles) as well as detailed TCAD results (dashes) at various stress times.*
- 3 *Comparison of our fast compact model to a detailed TCAD model when a sine wave signal is applied.*



ANALYTICAL TEM FOR PRODUCT-RELATED MATERIAL DEVELOPMENT

Introduction

Materials and material parameters form the background for many technological innovations. The desired material properties depend, to a large extent, on their microscopic structure, making micro analytical tools a necessity. Analytical transmission electron microscopy (TEM) plays a key role as it is the only method that provides complete structural characterization down to the atomic level. TEM combines the three fundamental analytical methods of imaging, diffraction and spectroscopy in the same tool, allowing for comprehensive analyses.

The analytical TEM has decisively contributed to tailoring the structure of materials with regard to their application-relevant properties. The method is not only advantageous to address basic research questions but can also be used:

- for improving product related material properties (e.g. process technologies used to design surface-near microstructures),
- to further develop material-dependant manufacturing processes (e.g. joining),
- for failure analysis and
- to evaluate the quality of manufacturing processes.

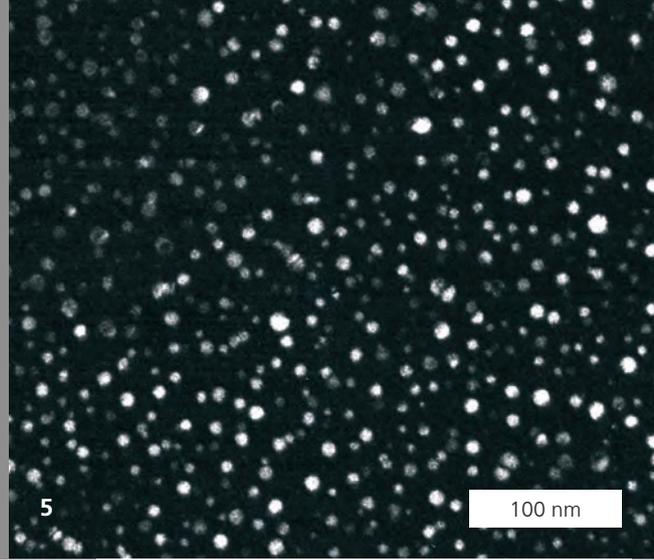
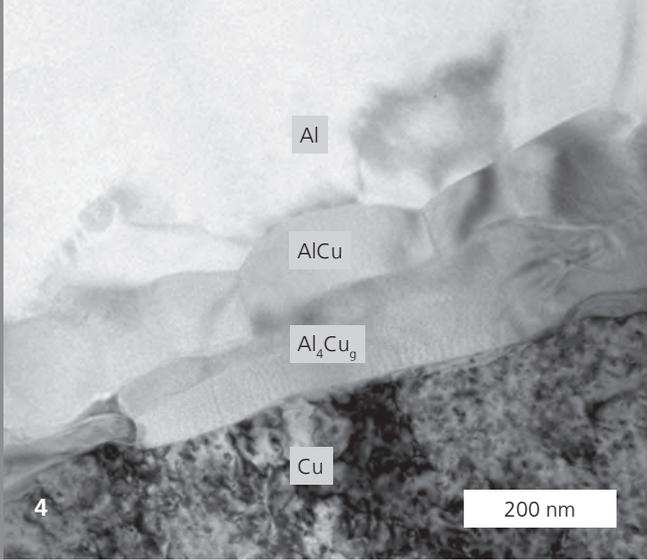
In this respect it is the goal of Fraunhofer IWS engineers to utilize the technique for product-related materials development and also to offer this particular expertise to its customers and their process-driven research issues. At Fraunhofer IWS the analytical TEM is combined with comprehensive metallographic capacities, scanning electron microscopy (SEM), focused ion beam facilities and materials testing capabilities. The following results present an overview of Fraunhofer IWS research activities in the field of material-related process and product design.

Results

Current research is devoted to the synthesis of silicon carbon nanoparticles in a so-called “core and shell” arrangement. This material is designated as an electrode material in lithium ion batteries. TEM investigations provide information such as the structure, size and distribution of the nanoparticles and information about the condition of the carbon shell as a function of the synthesis conditions (Fig. 1). This information in return provides the opportunity to optimize synthesis parameters.

Another research topic at Fraunhofer IWS is the development of reactive multilayer coatings (RMC), which are applied in low heat impact joining processes for various material combinations by delivering heat energy precisely and reproducibly to the contact zone. RMCs consist of nanometer multilayers with hundreds or thousands of individual films. The exact knowledge of the periodic thickness allows for a precise property design of the RMCs. Another important aspect of the development is to avoid diffusion within the coating stack during fabrication. TEM analysis demonstrated that specific barrier coatings help avoid this undesired diffusion effect (Fig. 2).

New materials and material combinations require efficient methods for the realization of dissimilar joints. In this respect IWS focuses on such promising new technologies as laser welding, electromagnetic pulse welding, friction stir welding and laser induction roll plating. TEM analysis is once again one of the key methods to study the microstructural changes as the process parameters are changed and hence analyzing the development of undesired phase seams that may form at the interface between two materials during such joining processes. Friction stir welding, laser induction roll plating

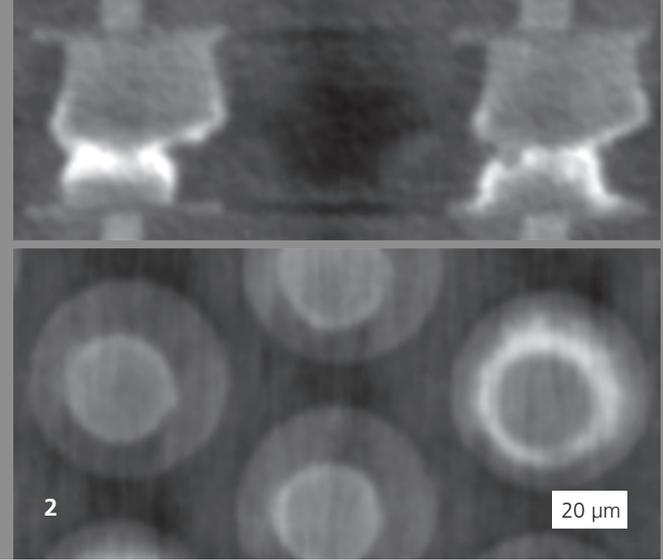
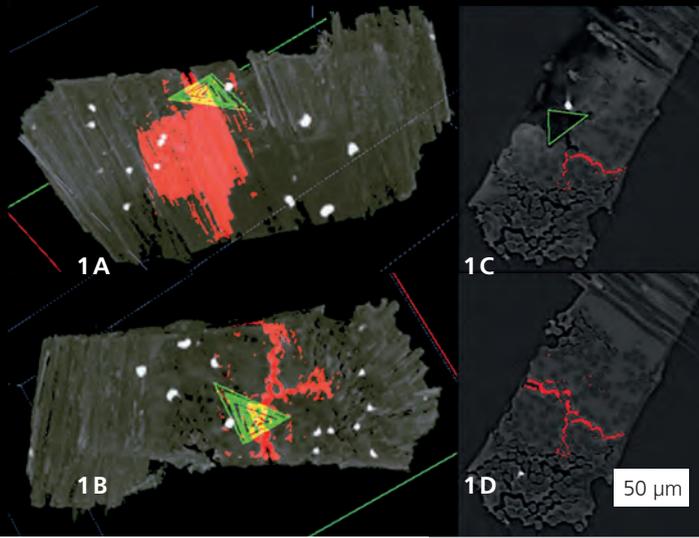


and electromagnetic pulse welding all achieve subcritical phase seam thicknesses of less than 1 μm . In the case where aluminum alloys are the base material, mechanical tests revealed that failure always occurred in the base metal and not in the joint. The low phase seam thickness also reduces contact resistance, which was much lower for these three techniques when compared to laser welding and conventional screw connections. TEM analysis provided additional valuable information about the growth process and the nature of the forming phases (Fig. 4).

Precipitation-hardened martensitic stainless steels offer a good combination of strength, ductility and toughness, good fabrication characteristics and corrosion resistance as well as superior fatigue performance. Therefore these steels are extensively used for structural components in aircraft, chemical, naval, nuclear and power generation industries. However, if the application requires high wear resistance, common surface heat treatments are not applicable for this kind of material. To overcome this drawback an effective surface modification technique has been developed at Fraunhofer IWS Dresden, which allows the selective generation of wear resistant surface regions up to several millimeters in depth without altering mechanical properties in the bulk of the material. This technique consists of selective short-time laser surface solution annealing (austenitization) at unusually high temperatures to completely dissolve the precipitations in the near surface region and maintain a solid solution condition upon rapid self-quenching and phase transformation to lath martensite and an aging treatment at relatively low temperatures to optimally strengthen the solution-annealed surface regions. By choosing the appropriate laser and aging treatment, the

hardness of precipitation-hardened steels (15-5 PH, 17-4 PH and PH 13-8) can be increased by more than 150 HV up to a depth of 4 mm (Fig. 3). SEM and TEM analyses verified that the improved properties of the age hardened surface are due to a more homogeneous and finer precipitation arrangement. The property-determining Cu and Ni_3Al precipitations in the age-hardened surface are generally smaller than 10 nm and are thus much smaller than in the bulk (Fig. 5). Further positive effects of the laser surface annealing are the dissolution of coarse carbides and a substantially higher thermal stability of the precipitations.

- 1 *SiC nanoparticles, encapsulated with amorphous and graphitic carbon (TEM).*
- 2 *High resolution TEM image of a ZrAl RMC with (upper left) and without (lower right) diffusion barrier.*
- 3 *Micro hardness depth profiles of different precipitation-hardened steels after surface age hardening.*
- 4 *TEM image of the phase seam formed during laser induction roll plating of Al and Cu.*
- 5 *TEM dark field image of Ni_3Al precipitations in the surface age hardened region of PH 13-8 steel.*



CRACK IMAGING IN COMPOSITE MATERIALS USING HIGH-RESOLUTION NANO-XCT

High-resolution X-ray microscopy and nano X-ray computed tomography are excellent techniques for three-dimensional investigation of nanostructured materials. In combination with mechanical loading devices, e.g. an in-situ nano-indentation stage, the technique combines the advantages of high-resolution three-dimensional visualization of microstructure with the observation of microstructural changes under loading. Phenomena such as crack initiation, crack propagation and, with composite materials, also delamination can thus be studied. Therefore, the set-up can provide fundamental understanding about the deformation behavior and failure mechanism of advanced materials.

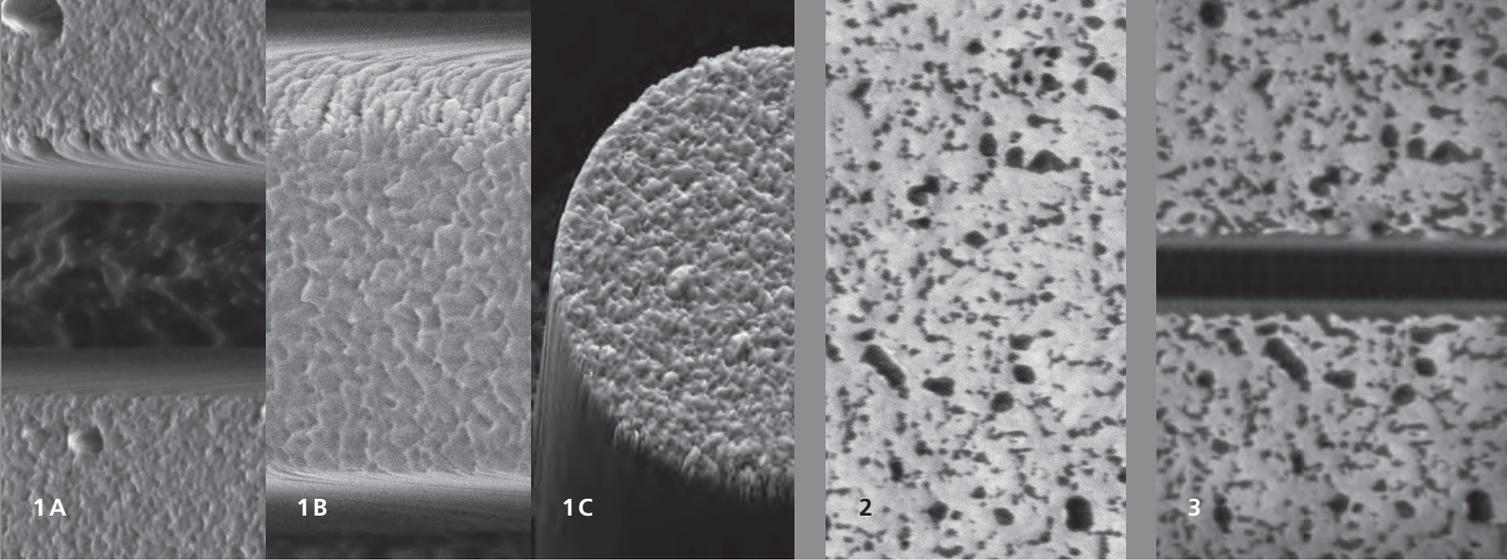
A newly designed mechanical load stage for in-situ mechanical loading was developed for the laboratory-based X-ray microscope at Fraunhofer IKTS (Xradia nanoXCT-100). The special design of the stage allows the use several types of indenters and force sensors depending on the mode and type of material. The indenter geometry and the samples are prepared to fit within the field of view of the X-ray microscope (FOV width: 65 μm or 16 μm). With an operating range of up to 1000 mN and a force resolution better than 0.25 mN, it is well suited to perform mechanical tests with various composite materials like carbon fiber composites and composites with ceramic or metal matrices while enabling the simultaneous non-destructive 3D imaging of the components/micro-structure of the composite materials with sub-100 nm resolution. This can be used, for instance, to monitor crack propagation and delamination under mechanical loading at the microscale.

The apparatus has the following properties:

- Experimental set-up with miniaturized micro-indentation using diamond tip with Berkovich geometry to initiate damage in composites
- High-resolution transmission X-ray microscopy (TXM) and nano X-ray computed tomography (nano-XCT) for non-destructive 3D imaging with sub-100 nm resolution.

1 XCT based virtual cross-sections through a carbon fiber reinforced Aluminum matrix composite. 1A and 1B display the 3D reconstructed data set with the indenter tip (green) and the crack along the carbon fiber/Aluminum matrix interface (red), 1C and 1D show virtual cross-sections of the crack grown along the interfaces.

2 Virtual cross sections from a 3D nanoXCT data set showing micro solder bumps in a 3D stacked microchip.

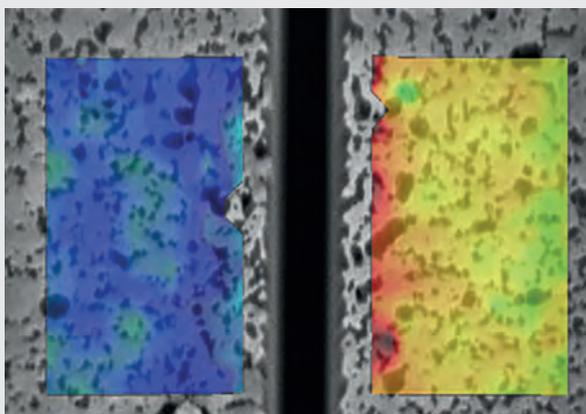


MEASUREMENT OF MICROMECHANICAL PROPERTIES WITH THE FIBDAC METHOD

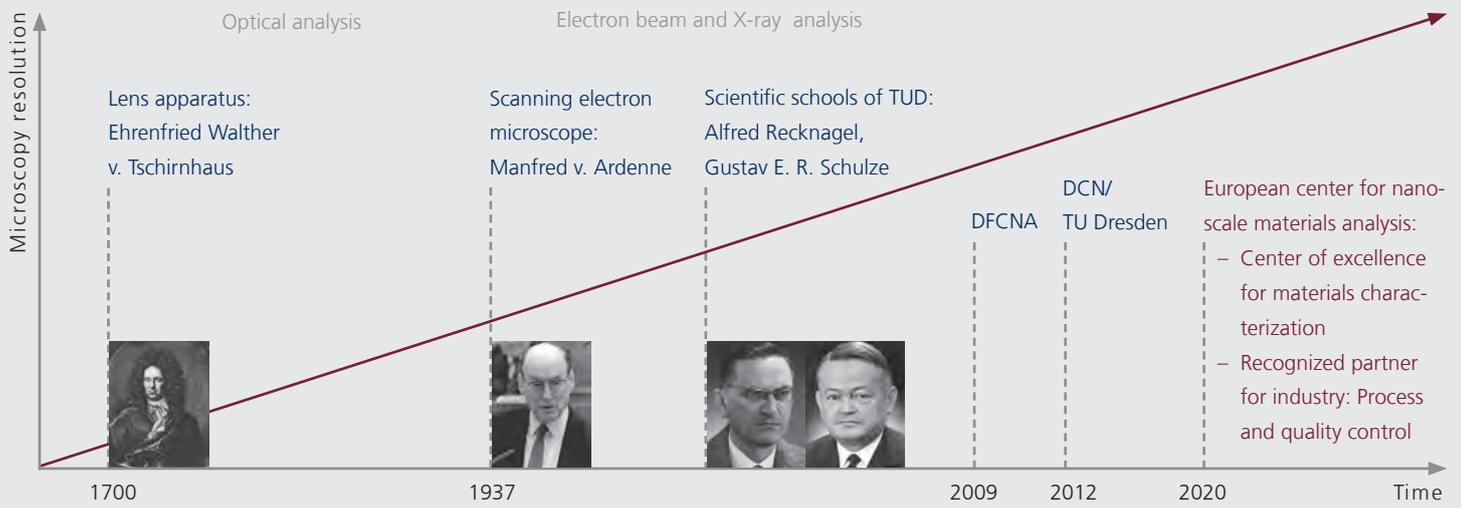
The determination of micro-mechanical properties is an important field in material science as well as microelectronics research and development. Beside basic material parameters like Young's modulus or Poisson's ratio, the stress-strain state of a material is one of the key parameters to describe its overall mechanical behavior. Beside well-established methods for micro-mechanical parameter determination like nanoindentation, also methods for the characterization of the microscopic stress-strain state exist at Fraunhofer IKTS. One of these methods is the FIBDAC method which is here described stepwise:

- Creating a stable, unique, high contrast pattern on top of the Region Of Interest (ROI) by gold sputtering and successive focused ion beam (FIB) milling
- Taking a high quality image of the ROI with a Scanning Electron Microscope (SEM)
- Milling a slit into the region of interest with the FIB, slit dimensions: width 1–2 μm , length approx. 10 μm , relaxing in-plane stress results in stress-strain relief
- Taking a second high quality image of the ROI
- Calculation of the resulting strain field with the help of image processing software (Digital Auto Correlation – DAC) x-shift in pixel
- Feeding the strain data into a finite-element model or into a semianalytical model in order to calculate the stress on the basis of pre-determined elastical properties (Young's modulus)

Determined strain field with subpixel resolution



- 1 (A-C) Different FIB patterns on a microelectronic sample in order to relieve in-plane stress (left-to-right: single slot, double slot, pillar).
- 2 SEM image of the high contrast pattern on top of the ROI.
- 3 SEM image of the ROI after FIB milling.



MICROSCOPY AND ANALYSIS – TRADITION IN SAXONY

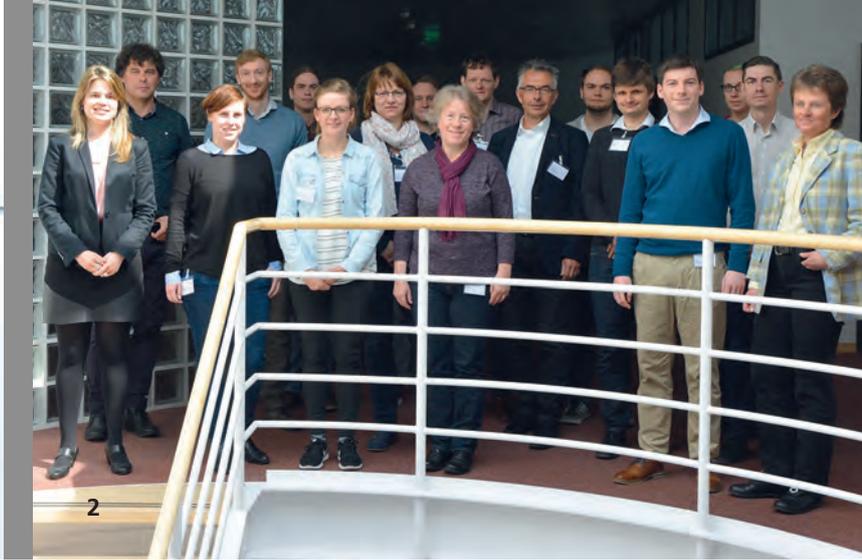
Three centuries of technological breakthroughs

During the past three centuries, several technological innovations in the field of materials as well as in microscopy and high-resolution analysis of materials and structures have originated in Saxony, particularly in Dresden. The contributions from Ehrenfried Walther von Tschirnhaus in the field of light optics and Manfred von Ardenne's developments in the field of electron microscopy are typical examples. Ehrenfried Walther von Tschirnhaus, known as the co-inventor of Meissen china, together with Johann Böttger, developed a lens apparatus in the 17th century, an important invention in optical equipment manufacturing. The focusing mirrors and the lenses used outperformed all existing optical systems in precision, size, and quality. One important feature was the precise surface treatment of the lenses. Ehrenfried Walther von Tschirnhaus's essential contribution to the development of microscopy was the fundamental mathematical study of the light paths in focusing lenses. Fundamental physical inventions at the end of the 19th and the beginning of the 20th century (e. g. by Thomson, Röntgen, de Broglie, and Laue), and a diversification of physics in the first half of the 20th century, brought about significant advancement in the electron and X-ray analysis of materials and structures. Alfred Recknagel and G. E. R. Schulze created scientific schools in the fields of electron physics and X-ray physics at Dresden (TU) after the second world war. Manfred von Ardenne, inventor of the scanning electron microscope, moved to Dresden in 1955 and headed a research institute at Dresden Weißer Hirsch for several decades. In the second half of the 20th century, significant developments at the interface between physics and electrical engineering were accomplished in Dresden. These developments led to the formation of profile-determining

fields of research in Dresden and Saxony, which have since become sustainable. Just like 300 years ago, analytical know-how is essential today for inventions and innovations in the field of materials, for materials research and development, and for the integration of new materials into products. Like ores and minerals were once investigated as raw materials for metal and porcelain manufacturing, today's targets are nanoscale materials for applications in nanoelectronics, thin-film photovoltaics and other high-tech branches.

Current scientific profile

The scientific profile line of high-resolution analysis is alive in the Dresden region, with core competencies in nanoanalysis and materials characterization at TU Dresden and at the Fraunhofer Institutes located in Saxony, and it has found significant international repute. Today, basic research in the fields of nanoscale materials and structure analysis at TU Dresden is performed in the new Dresden Center for Nanoanalysis. In addition, fundamental research and applied developments in the field of nanoanalysis are performed at several Max Planck, Helmholtz, Leibniz, and Fraunhofer Institutes, all included in the DRESDEN-concept – a network of local scientific expertise that is unique in Germany. The Dresden Fraunhofer Cluster Nanoanalysis DFCNA is in line with historic developments in Saxony. This network strengthens research and development in this field as well as the efficient application of analytical techniques. With its mission to bridge the so-called "innovation gap", the DFCNA provides a relevant response to the current need for the rapid transformation of knowledge into products. Therefore, the network has become an attractive partner for industry in the field of nanoanalysis, with a particular focus on process and quality control.



A CENTER FOR RESEARCH AND INNOVATION – INTEGRAL PART OF THE DRESDEN-concept WITH EUROPEAN ORIENTATION

One key element of the success of the Dresden Fraunhofer Cluster Nanoanalysis (DFCNA) is the close collaboration with scientists and engineers in Europe. Researchers of the Fraunhofer cluster contribute to the activities of the European Materials Characterization Council (EMCC), a European initiative that supports the process of developing and improving characterisation tools to bring the development of nanomaterials and advanced materials in Europe into end products more successfully. Fraunhofer researchers from Dresden provided essential input to the Roadmap for Materials Characterization, established by the EMCC, in particular.

The Dresden Nanoanalysis Symposium (DNS), held in Dresden once in the year since 2013, is already a marked time slot in the schedules of many researchers from Europe and from the United States. This annual event is supported by the European Materials Research Society (E-MRS) and by the European Materials Characterization Council (EMCC). The symposium covers topics of nanoanalysis and materials characterization along the whole value and innovation chain, from fundamental research up to industrial applications. It brings together scientists and engineers from universities, research institutions, equipment manufacturers and industrial end-users. Highlights in disruptive nanoanalysis techniques are reported in invited talks and during the poster sessions, and novel solutions in the field of materials characterization for process and quality control are shown. The discussions and interactions between the stakeholders 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 Dresden Nanoanalysis Symposium is a "place to meet"!

Every year, an European Advanced Training Course "Nanoscale Materials – Characterization Techniques and Applications" is hosted by the Dresden Fraunhofer Cluster Nanoanalysis. This course, organized by Deutsche Gesellschaft für Materialkunde (DGM) and supported by the Federation of European Materials Societies (FEMS), is intended for individuals who wish to expand their knowledge in the field of nanoscale materials and nanoanalysis. The subjects covered in this course extend from fundamentals of materials science and analysis to the current nanotechnologies and challenges in industry. Scientists, engineers and technicians working in industry, research and education, who are interested to extend their knowledge in nanoanalysis, benefit from this course. The course considers the fact that nanoscale materials are enablers for high-tech products. High-resolution analytical techniques are essential for both development and introduction of new nanotechnologies and thin-film technologies as well as for the integration of advanced materials into high-tech products. Nanoanalysis is more and more needed for process and materials characterization during manufacturing of nanostructured systems and devices as well as for the understanding of nanoscale microstructure in materials. Therefore, research and development in the field of physical analysis is increasingly focused on the study of thin films and nanostructures.

1 Prof. E. Zschech with the poster award winners at the 5th Dresden Nanoanalysis Symposium 2017.

2 Participants of the FEMS-supported DGM course "Nanoscale materials" 2017.



MEMBERS

Fraunhofer ENAS
www.enas.fraunhofer.de

Fraunhofer IFAM
www.ifam-dd.fraunhofer.de

Fraunhofer IIS/EAS
www.eas.iis.fraunhofer.de

Fraunhofer IKTS
www.ikts.fraunhofer.de

Fraunhofer IPMS
www.ipms.fraunhofer.de

Fraunhofer IWS
www.iws.fraunhofer.de

Fraunhofer IZM
www.izm.fraunhofer.de

PARTNERS

Technische Universität Dresden
www.tu-dresden.de

Helmholtz-Zentrum Berlin für Materialien und Energie
www.helmholtz-berlin.de

STEERING COMMITTEE

Ehrenfried Zschech, Head of steering committee
ehrenfried.zschech@ikts.fraunhofer.de

Andreas Leson, Committee member
andreas.leson@iws.fraunhofer.de

Thomas Weißgärber, Committee member
thomas.weissgaerber@ifam-dd.fraunhofer.de

MANAGEMENT

Simon Schlipf, Office management
simon.schlipf@ikts.fraunhofer.de

Katja Herklotz, Project coordination
katja.herklotz@ikts.fraunhofer.de



PUBLICATION DATA

Editor

Dresden Fraunhofer Cluster Nanoanalysis DFCNA
c/o Fraunhofer IKTS
Maria-Reiche-Strasse 2
01109 Dresden | Germany
Phone +49 351 8 8815-609
Fax +49 351 8 8815-509

info@nanoanalysis.fraunhofer.de
www.nanoanalysis.fraunhofer.de
Twitter: @nanoanalysis

Technical assistance

Fraunhofer IKTS, Press and Public Relations

© Photos

p.3: ddpix.de | DML BY
All others: Members and partners of Dresden Fraunhofer
Cluster Nanoanalysis DFCNA

The Dresden Fraunhofer Cluster Nanoanalysis DFCNA is a constituent entity of the Fraunhofer-Gesellschaft and as such has no separate legal status.

© 2018

All rights reserved. Reproduction of any material is subject to editorial authorization.

- 1 Ehrenfried Zschech
- 2 Andreas Leson
- 3 Thomas Weißgärber
- 4 Simon Schlipf
- 5 Katja Herklotz

