

Nanotechnology Competence Center "Ultrathin functional films"

Activities and actual projects



nanotechnology

CC "Ultrathin functional films"

**Nanotechnology Competence Center
"Ultrathin functional films"**

**Office at Fraunhofer Institute for
Material and Beam Technology IWS**

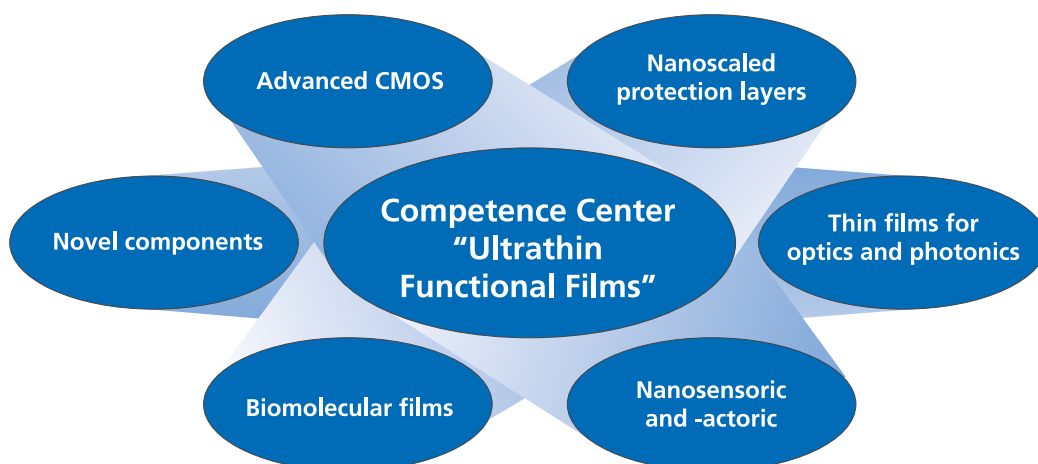
Winterbergstr. 28
01277 Dresden, Germany

Contact partner: Dr. Ralf Jäckel
Phone: +49 (0) 351 / 25 83 444
Fax: +49 (0) 351 / 25 83 300
E-mail: ralf.jaeckel@iws.fraunhofer.de

Internet: <http://www.nanotechnology.de>

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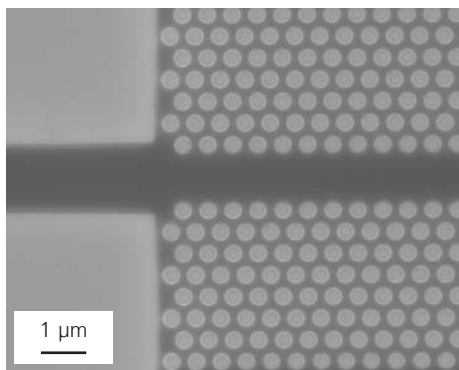


Meanwhile the nanotechnology competence center has been existing already for five years. After a short start-up period in 1998 the following years have been characterized by an active public relation, participations in fairs, numerous workshops as well as joint realizations of many research projects. During the first years the discussion centered in new project ideas and the preparation of mutual R+D projects. Here the members were successful in such a way that many proposals were accepted and funded by the German Federal Ministry of Research and Technology. These projects and feasibility studies build an excellent focus for future activities and further possibilities within the scope of nanotechnology.

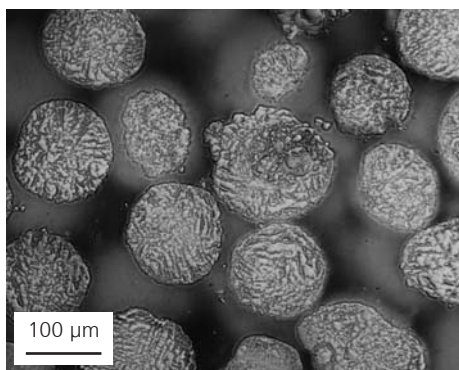
The continuously increasing access numbers reveal the high interest in the nanotechnology and the center of competence. It was possible to transfer competent contacts within the member's circle to numerous external inquiries with respect to problems within the field of nanotechnology.

This is the reason why the competence center regards the support of technologically-orientated foundations as one of its main tasks. It cooperates, e.g., with the project: "Demand-oriented starts-up from the university (Dresden exists)". First, already existing foundations let hope for the future and give reason to the idea that the nanotechnology will show even economic success apart from the scientific one. The founding of competence centers, such as ours for example, represents a new way of research funding, which will be attached to much importance, because it will contribute to a quick realization of innovations.

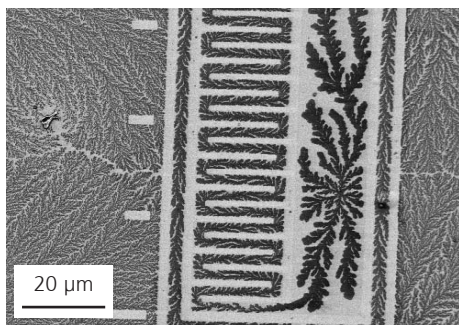
This is proved by the increasing number of competence centers and networks which have come into existence, for example, in the field of optical technologies. Not only because of the numerous activities within the centers, we are strongly convinced that this new funding device will prove itself as a very successful one. The evaluation of nanotechnology competence centers in which our center came of very well confirms this idea. This is the reason why the ministry has decided to fund the competence center for



Waveguide and photonic crystal obtained by electron beam lithography (Fig.: Raith GmbH Dortmund)



Freeze-dried sprayed granulate of nano-scale TiO_2 , brightfield image (Fig.: Fraunhofer Institute for Ceramic Technologies and Sintered Materials IKTS Dresden)



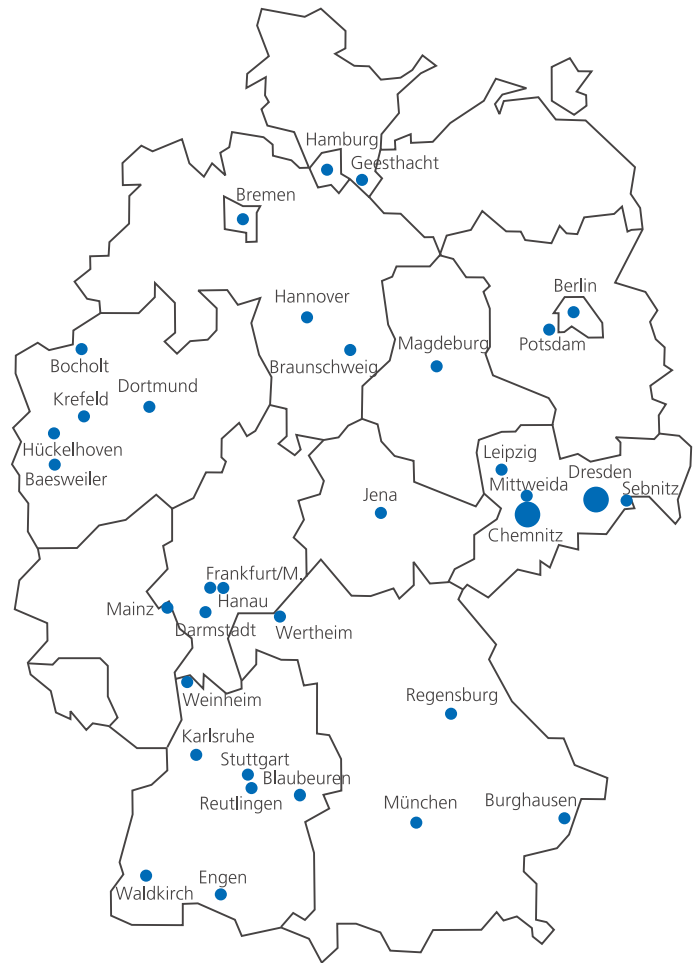
Diffusion controlled crystallization in metastable micro-structured PEO-layers (Fig.: Leibniz Institute of Polymer Research Dresden)

The numerous workshops, organized for many subjects, developed themselves to an essential activity focus point. They form a stage for the exchange of new ideas and the arrangement of mutual activities. Furthermore, they fulfil an essential function within the nanotechnology center with respect to the agreement on strategically important R+D development directions. With the internet presentation (www.nanotechnology.de) the members have an essential medium for an effective communication at their disposal. It comprises an internal part with current information to the center's activities as well as a part with information to press releases or topically main subjects, which is available for the general public.

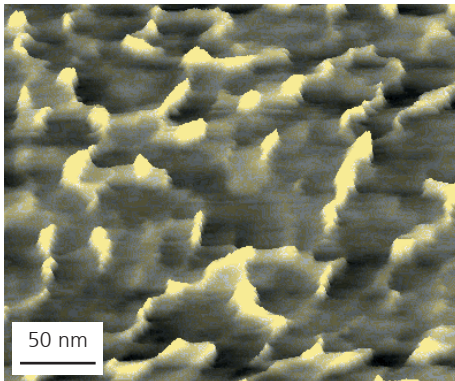
3 further years. In the coming years it will be important to strengthen the contacts and connections in such a way that these will endure even after the end of the ministry's support. We would like to thank especially those who contributed to the hitherto success by their activities and support. We will do our best to maintain this success.

On behalf of the executive committee

Dr. Andreas Leson



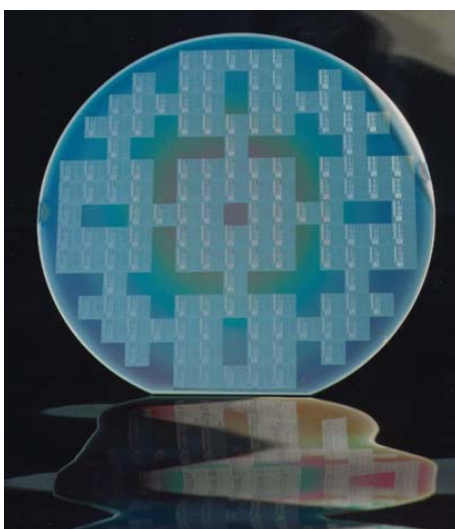
Location of the members of the Nanotechnology competence center



AFM-picture of the surface of a titan-platin-film, tempered at 700 °C
(Fig.: Fraunhofer Institute for Ceramic Technologies and Sintered Materials IKTS Dresden)



Electron beam lithography system for the production of nanostructures
(Fig.: Raith GmbH Dortmund)



6 inch silicon wafer, microstructured with chrome and oxide films
(Fig.: University of Technology Chemnitz)

Objective of the competence center

Nanotechnology belongs to the key technologies of the 21st century. Already now we have first applicable products at our disposal: hard disks and reading heads for the data storage, coated with few nanometer thin films, or scanning microscopes, which allow an insight into the world of atoms and molecules. Other examples are high precise x-ray optics and ultra thin films for the microelectronic, which are the presupposition for a further increasing of the integration density and the clock rate.

Ultra thin films are the gist of nanotechnology. Their application range covers the fields of microelectronics and optics to medical science and sensor technology up to wear protection films. In order to enable a consequent industrial development 52 enterprises, 10 institutes of universities, 22 research establishments and 6 associations have focused their knowledge and joined to a network. The competence center for "Ultrathin functional films" is funded by the Federal Ministry for Education and Research.

Tasks

The intensive cooperation between the single members within the competence center secures an efficient usage of present resources and enables quick solutions.

To fulfil this task we employ especially:

- workshops and seminars,
- presentations of R+D projects,
- arrangement of competent partners,
- support of education and further education,
- support of founders of new business,
- support of standardization and technical rule sets,
- public relation.

Interested customers will find a wide variety of equipment and an outstanding know-how which will be made available by the center's agency.

We are pleased to be able to offer the following services:

- consultation,
- feasibility studies,
- expert opinions,
- execution of R+D activities,
- process tests,
- cooperative system development.

Organization

The competence system is represented by the board of management:

Prof. Dr. Thomas Geßner
University of Technology Chemnitz,
Center for Microtechnologies
Phone: +49 (0) 371 / 531 31 30
Fax: +49 (0) 371 / 531 31 31

Prof. Dr. Karl Hofmann
University Hannover,
Institute for Semiconductor Devices
and Electronic Materials
Phone: +49 (0) 511 / 762 42 12
Fax: +49 (0) 511 / 762 42 29

Dr. Andreas Leson (chairman)
Fraunhofer Institute for Material and
Beam Technology IWS Dresden
Phone: +49 (0) 351 / 25 83 317
Fax: +49 (0) 351 / 25 83 314

Prof. Dr. Wolfgang Pompe
University of Technology Dresden,
Institute for Material Science
Phone: +49 (0) 351 / 463 314 20
Fax: +49 (0) 351 / 463 314 22

Research groups in the nanotechnology competence center

Research group 1: Advanced CMOS

Head of the research group:

Prof. Dr. Thomas Geßner
University of Technology Chemnitz,
Center for Microtechnologies
Reichenhainer Straße 70
09126 Chemnitz, Germany

Phone: +49 (0) 371 / 531 31 30

Fax: +49 (0) 371 / 531 31 31

Research group 2: Novel components

Head of the research group:

Prof. Dr. Christian Radehaus
University of Technology Chemnitz,
Center for Microtechnologies
Reichenhainer Straße 70
09126 Chemnitz, Germany

Phone: +49 (0) 371 / 531 30 86

Fax: +49 (0) 371 / 531 30 04

Research group 3: Biomolecular films for medical science and technology

Head of the research group:

Prof. Dr. Wolfgang Pompe
University of Technology Dresden,
Institute for Material Science,
Mommsenstraße 13
01069 Dresden, Germany

Phone: +49 (0) 351 / 463 314 20

Fax: +49 (0) 351 / 463 314 22

Research group 4: Nanoscaled protection layers

Head of the research group:

Dr. Bernd Schultrich
Fraunhofer Institute for Material and
Beam Technology IWS Dresden
Winterbergstraße 28
01277 Dresden, Germany

Phone: +49 (0) 351 / 25 83 403

Fax: +49 (0) 351 / 25 83 300

Research group 5: Thin films for optics and photonics

Head of the research group:

Prof. Dr. Karl Leo
University of Technology Dresden,
Institute for Applied Physics
Mommsenstraße 13
01069 Dresden, Germany

Phone: +49 (0) 351 / 463 343 89

Fax: +49 (0) 351 / 463 370 65

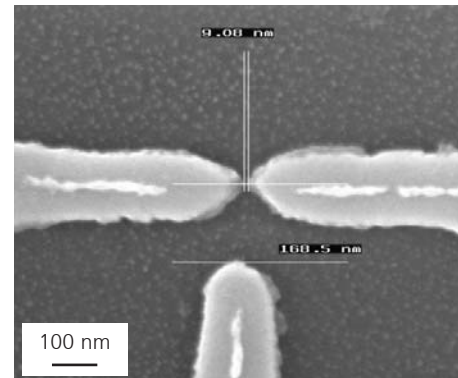
Research group 6: Nanosensoric and nanoactinic / nanosystems

Head of the research group:

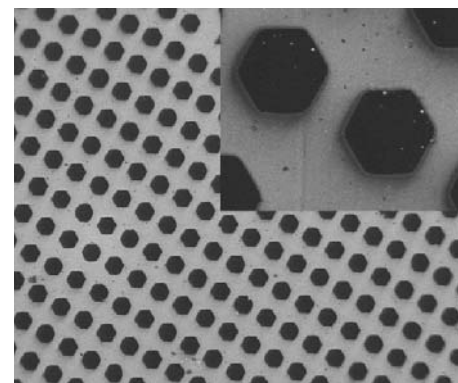
Dr. Andreas Schönecker
Fraunhofer Institute for Ceramic Technologies and Sintered Materials IKTS
Dresden
Winterbergstraße 28
01277 Dresden, Germany

Phone: +49 (0) 351 / 25 53 508

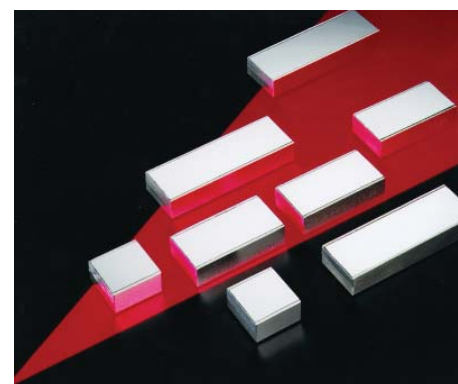
Fax: +49 (0) 351 / 25 53 605



Single electron transistor structure with contacts for the testing of electrical properties (Fig.: University of Technology Chemnitz)



Ultra thin structured bio polymer films (35 nm), produced by surface polymerisation (Fig.: Leibniz Institute of Polymer Research Dresden)



Different precision x-ray mirrors for x-ray reflectometry, produced by pulse laser deposition (Fig.: Fraunhofer Institute for Material and Beam Technology IWS Dresden)

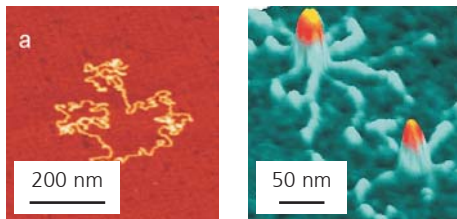


Fig. 1: Linear (left) and star-shaped (right) molecules as components for the future electronical technology (Fig.: Leibniz Institute of Polymer Research Dresden)



Fig. 2: Laser acoustic measuring device LAwave® for the non-destructive testing of thin films (Fig.: Fraunhofer IWS Dresden)

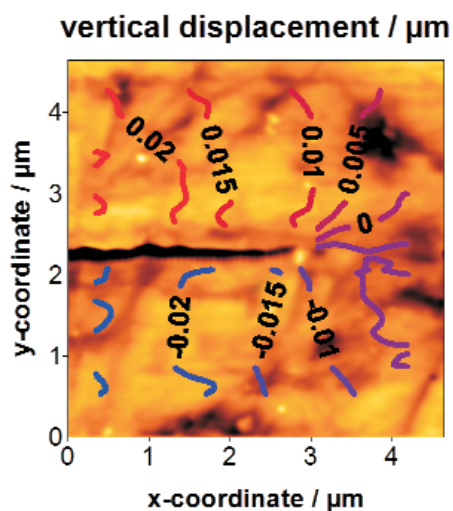


Fig. 3: Displacement field at a crack tip of a thermoset polymer (The crack opening field is shown as contour lines of equal displacement in y-direction and the scanned surface is shown in the background of the image.) (Fig.: Fraunhofer IZM Berlin)

Nanoparticles from single poly-electrolytes

Nanoparticles are of great interest due to their potential applications as building blocks for modern nanoelectronics or for catalysis. One of the most promising route to such particles, so-called "bottom-up" approach, have been applied by researchers from the Institute of Polymer Research Dresden.

Using Atomic Force Microscopy they were successful to observe polymer molecules of various architecture and composition as individual entities. Applying special external stimuli they are able to deposit polymer molecules onto a solid support in desired conformation. Finally, functional materials could be selectively attached to active sites of molecules. Importantly, shape, size, and location of thus fabricated particles are predefined by the conformation of the polymer precursor whereas physical properties are controlled by the properties of deposited materials. As an example, Figure 1 shows linear (a) and star-shaped (b) molecules which can be used for fabrication of palladium nanowires or semi-conductive Prussian Blue nanoparticles - important building blocks for future molecule-based electronics.

Contact: Leibniz Institute of Polymer Research Dresden
 Prof. Dr. Manfred Stamm
 Mail stamm@ipfdd.de
 Dr. Anton Kiriy
 Mail kiriy@ipfdd.de

Non-destructive quality inspection of ultrathin films

Scientists of the Fraunhofer Institute for Material and Beam Technology IWS Dresden have developed a laser-acoustic technique for testing thin films and surfaces (fig. 2), having the trade name LAwave®. The technique

has found manifold uses, determining the elastic modulus, density and / or thickness of thin films and coatings. The elastic film modulus is accepted as fundamental mechanical parameter, determining the mechanical response and the stress distribution in coated materials and sensitively depending on the nature of the chemical bonding and the defect density. For hard-coatings, it correlates with the hardness. For defective layers, it indicates enhanced defect density and porosity, what has given rise to the special useful application of determining the damage depth in semi-conductor materials. A special version for 300 mm wafers has been designed for a semi-conductor producer.

Films can be tested, being soft as polymers or super-hard as diamond. On silicon, films of a thickness down to few nano-meters can be investigated, advantageously applied to characterize top-coats of computer hard-discs. On polycrystalline substrate, the minimum film thickness is about 50 nm. The laser-acoustic test is standardized in accordance to DIN EN 50992-1. The LAwave technique was granted an R&D 100 Award 2001.

Contact: Fraunhofer Institute for Material and Beam Technology IWS Dresden
 Dr. Dieter Schneider
 Mail dieter.schneider@iws.fraunhofer.de

NanoDAC - A new method of deformation measurement on the micro- and nanoscale

NanoDAC is a new method for displacement and in-plane strain field measurement based on scanning probe microscopy images (nanoDAC, nano Deformation Analysis by Correlation). It was developed by the Micro Materials Center of the Fraunhofer Institute for Reliability and Microintegration IZM Chemnitz. The structures

to be analyzed (micro components, nanomaterials) are scanned in-situ in different thermo-mechanical load states in the AFM (atomic force microscope).

By means of these images it is possible to determine local displacements fields. The underlying measurement principle is locally applied digital image correlation. Thus it is possible to track image patterns and to obtain qualitative and quantitative information on the deformation behavior.

The results of the nanoDAC technique are in-plane displacement fields $u_x(x, y)$ and $u_y(x, y)$ (fig. 3). Applications of nanoDAC are thermo-mechanical reliability analysis of MEMS and NEMS and the mechanical qualification of newly developed nanomaterials.

Further application areas are focused on:

- micro- and nanoscale in-situ deformation measurements,
- verification of nanomechanical material models,
- determination of fracture properties of micro- and nanomaterials,
- measurement of coefficient of thermal expansion (CTE),
- tracking of nanoparticles and nano-manipulated objects,
- evaluation of fracture behavior at material interfaces.

Contact: Micro Materials Center
Chemnitz
Prof. Dr. Bernd Michel
Mail michel@izm.fraunhofer.de

New elastomer materials on the basis of nano-structured multigraft-copolymers

Nano-structured block copolymers have already been subject to several researches during the last years. Now scientists of the Institute of Polymer Research together with colleagues,

coming from the USA and Greece, investigate, how new molecular architectures influence the tensile strength of polymer materials and which new possibilities for the material development will result. The developed multi-graft-copolymers, made of polystyrene and polyisoprene, showed quite astonishing properties. The elongation is with 2000 % about the twice that of common thermoplastic elastomers. Furthermore, the new multigraft copolymers are superior with respect to the flexibility. Up to an elongation of 500 % a complete re-deformation will take place. Decisive reasons for these desired properties are the morphology in the nanometer range and molecular architecture. The morphology can be directly tailored by the number of branches and the kind of the inserted functionalities.

Contact: Leibniz Institute of Polymer Research Dresden
Priv.-Doz. Dr. Roland Weidisch
Mail weidisch@ipfdd.de

Addition of carbon nanotubes: an efficient way to make conventional polymers conductive

Due to their exceptional properties, carbon nanotubes become subject of quite a number of research projects in materials science. Only very small amounts of these tubes of inner diameters of 3 to 5 nm and lengths of 1 to 10 micrometers are necessary to form a percolation network in a polymer matrix and, consequently, to obtain electrostatic or conductive properties of the intrinsically isolating polymer materials. In addition, they may serve to improve material properties like mechanical strength and fire resistance.

Researchers at the Institute of Polymer Research Dresden have tackled the task to adapt conventional plastics processing technologies in extruders to

the manufacture of composites of polymers and carbon nanotubes. By now, such composites have been produced only in very small amounts in laboratory - their manufacture by conventional machinery can make them highly interesting for industrial applications. The researchers have successfully optimized the mixing conditions and the use of additives. As a result, the addition of only 1 wt% of multiwalled carbon nanotubes leads to antistatic properties, addition of 2 wt% yields to conductivity. The effects have been demonstrated for polycarbonate (fig. 4). To bring about the same effects by filling with carbon black, about 10 to 20 wt% of filler would be necessary, and other material properties would considerably deteriorate.

Contact: Leibniz Institute of Polymer Research Dresden
Dr. Petra Pötschke
Mail poe@ipfdd.de

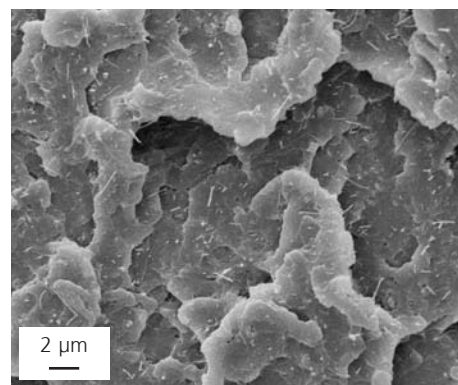


Fig. 4: Cryofracture of a composite of polycarbonate with 1.5wt% multiwalled carbon nanotubes (MWNT). The MWNT were synthesized in the Leibniz Institute for Solid State and Materials Research Dresden in the group of Dr. Leonhardt. (Fig.: Leibniz Institute of Polymer Research Dresden)

Examples of current R+D projects

Development of a nanostructure assembler with motor protein control

Motivation: parallel processing in nanoelectronics

To move and position single atoms or molecules with an atomic force microscope, impressive as it is, has contributed to a high extent to the increasing public interest in nanotechnology.

It has been demonstrated in general how structures can be developed in a size range too small to be accessible by standard photolithographic methods. The size reduction to the nanometer scale, with its inherent drastically changed physical and chemical properties, is the most interesting aspect of this new technology.

The use of just a few new structure elements, however, will not be sufficient for an application on an industrial scale. As extremely high integration is needed, thousands of well-organized nano-parts will need to be processed to achieve effective results, thus manipulations of just single pieces is not an option. In the field of nanoelectronics and nanomechanics, however, suitable tools are still lacking to accurately and reproducibly integrate sufficiently large numbers of nanoscale functional elements into a macroscopic or microscopic world.

As often before, nature can serve as a model system. The living cell with its polymorphic structure can be regarded as a highly complex microscopical factory consisting of highly specialized functional nano-units. If it could be demonstrated that cellular machines can do their job in vitro, one might imagine to look for solutions for the manipulation of nano-sized functional elements by these cellular machines.

The "Namos" project represents a basically new approach to this idea, because microfluidic techniques are employed in the first place for the generation of structures on the nanoscale. In particular, cellular machines are directly arranged on a substrate. By using molecular selection mechanisms, electrical and nanomechanical networks could be generated.

Biological principles as solution to nanowiring

The starting point for the construction of electrically conducting networks is DNA. Long-chain DNA molecules are first spread on a substrate and then metallized by a special procedure developed at the Institute for Material Science of the University of Technology Dresden, thus allowing the production of nanowires consisting of different metals.

For simple patterns, the DNA molecules are attached at certain spots on the surface (e.g., gold pads), either covalently or by using the biotin-streptavidin interaction, and then the positioning of the DNA is achieved by applying fluid flows in different directions. The addressing of special positions follows the key-hole principle. Furthermore, controlled cutting of the DNA by restriction endonucleases can modify already formed structures, e.g., to eliminate unwanted connections.

An even more effective transport mechanism can be achieved by molecular motors which transport attached DNA molecules until they reach their desired place. This technique was developed at the Max-Planck-Institute for Molecular Cell Bio-

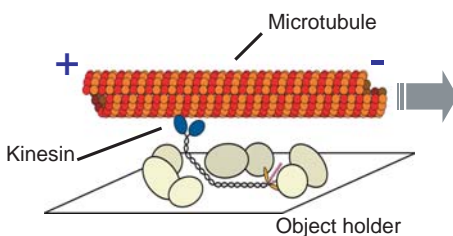


Fig. 1: Moving of microtubule by motor protein (Kinesin) on a surface (Fig.: Max-Planck-Institute for Molecular Cell Biology and Genetics Dresden)

Project partners:

- Namos GmbH Bannewitz
- University of Technology Dresden, Institute for Material Science
- Max-Planck-Institute for Molecular Cell Biology and Genetics Dresden
- GeSiM mbH Rossendorf

logy and Genetics in Dresden and requires a motor protein, kinesin, that is immobilized on a glass support, and microtubules, i.e. elongated proteins of the cytoskeleton on which kinesin migrates *in vivo*, which is the basis of the axonal transport in nerve cells. The microtubules attached to the immobilized kinesin motors are dragged by the action of kinesin across the glass surface. DNA molecules are attached to these microtubules (also between two different microtubules) using different methods and can be transported to certain places on purpose.

The control of the movement can be enhanced by applying hydrodynamic flow fields, such that the microtubules can only migrate in the direction of the flow, or by using nanostructured surfaces such that the microtubules can only move inside predefined channels. These channels can not only define the track, but also the direction of the movement.

The nano-assembler: first applications

Our long-run aim is to use motor proteins to generate nanoscaled structures and thus to obtain electrical and nanomechanical networks by molecular mechanisms.

The structuring of metallic conducting surfaces by the use of transport systems opens up completely new fields of application in the microelectronics. Hence a paradigm change takes place by replacing the "top down" by a "bottom up" approach. The construction of nanoscaled devices could be automated in a microfluidic flowthrough cell, which is fed by different reservoirs and thus can switch

on and off biochemical reactions, such as the microtubule movement, and also controls the direction of the movement. All in all, this set of technologies make up the "nano-assembler".

The project partner GeSiM has developed a system for fluid handling, which performs a whole set of functional flow cycles under complete microscopic control. This new system enables us to run the entire structuring process in a controlled way. The project partner Namos develops a nano-scaled strain gauge on the basis of metallized DNA as an application of the nanoassembler. The advantage of the resulting nanostructure is its well-defined material property which leads to reproducible measurement results. This is the first step towards a simple and cost-effective measuring device and displays a resolution hitherto unknown except in very expensive electromechanical methods.

The aim of the involved working groups of the University of Technology Dresden is the construction of simple electrical circuit structures made of nanowires, which are integrated into a microelectronic contact structure. The goal of the group at the MPI for Molecular Cell Biology and Genetics is to convert a kinesin-microtubule-based assay into a fine-tunable cellular machinery on a glass carrier.

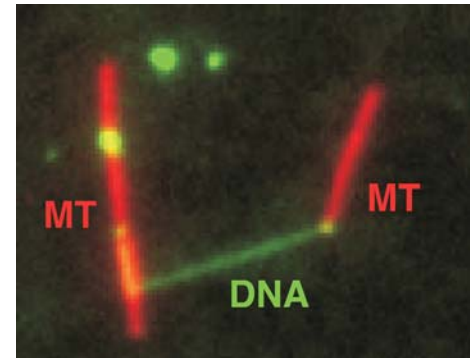


Fig. 2: Positioning of nano elements
A DNA is transported and stretched by two microtubules
(Fig.: Max-Planck-Institute for Molecular Cell Biology and Genetics Dresden and University of Technology Dresden, Institute for Material Science)

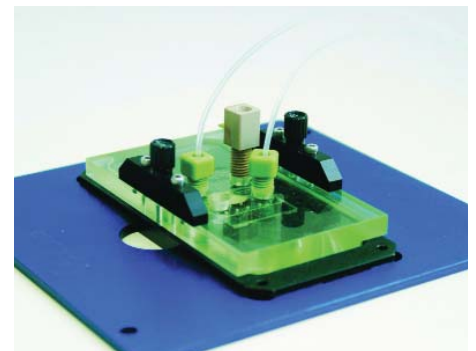


Fig. 3: Microscope cell as a basis for a nano assembler
(Fig.: GeSiM mbH Rossendorf)

Contacts for further information:

Namos GmbH Bannewitz
Windbergstr. 54
01728 Bannewitz, Germany
Dr. Jürgen Hofinger
Phone +49 (0) 351 / 4790 610
Fax +49 (0) 351 / 4790 611
Mail juergen.hofinger@namos.de

Quality control of EUV optics at-wavelength by a laboratory reflectometer

Tasks

Multilayers for EUV optics such as those applied for the next generation lithography have to fulfill highest demands with respect to reflectivity, reproducibility, homogeneity concerning the precision of film thickness distribution, resolution ability and temperature stability. In particular, it is important that these properties can be realized and controlled also on curved optics surfaces.

For film development and production the control of the achieved film parameters at wavelength is an essential time factor.

Thus multilayers are mainly controlled by the evaluation of reflectometry measurements. Up to now we were dependent on the use of the synchrotron radiation (e. g. BESSY II, Berlin). The distance, however, always caused a certain delay in the development and production process.

Solution

Thus, the development of a laboratory reflectometer with the wavelength in the EUV range has been a declared aim of scientists all over the world. In a close cooperation with the PTB (Physikalisch-Technische Bundesanstalt), Max-Born-Institute (MBI), Bestec, AIS Automation and Carl Zeiss SMT AG the Fraunhofer Institute for Material and Beam Technology IWS has developed such a laboratory reflectometer. The project is supported by the BMBF.

Four main tasks were to be solved:

- the development of a suitable beam source which guarantees a constant high photon flux in the EUV range,

- the monochromatisation of the generated EUV radiation,
- the realization of a suitable sample and detector manipulation for reflectometry measurements of mirrors and masks, which are developed for the semiconducting industry,
- a control and measuring technology, which realizes the required operations.

Results

In figure 1 you can see this laboratory EUV reflectometer. Due to the absorption of the EUV radiation in air, the EUV beam has to be routed in vacuum.

The EUV source works on the basis of a commercial Nd:YAG laser (wavelength: 532 nm, repetition rate: 10 Hz, pulse duration: 10 ns, laser beam focus: 10 μ m), with a series-connected laser beam focus system. The focused beam on a gold target surface generates a plasma, which supplies a quasi-continuous spectrum in the EUV range (per shot a new target surface, spot size: approx. 50 μ m). Using a laser power of 650 mJ the spectral photon flux of the EUV source amounts to $11 \cdot 10^{13}$ photons / (s sr 0.1 nm bw) at a wavelength of 13.4 nm.

The monochromator consists of aperture, plan-elliptic premirror, spherical grating with 1200 lines / mm, exit slit, spherical refocusing mirror, 2 scattered light apertures and a beam splitter to monitor the EUV beam. The monochromator provides EUV radiation, tunable in the range of 10 nm to 16 nm with a wavelength resolution of 0.03 nm. Using a laser power of 250 mJ a photon number of $3 \cdot 10^6$ photons per shot is sufficient for standard measuring operations. The



Fig. 1: Photograph of the laboratory EUV reflectometer (Fig.: Fraunhofer IWS Dresden)

Project partners:

- Fraunhofer Institute for Material and Beam Technology IWS Dresden
- Physikalisch-Technische Bundesanstalt Berlin
- Max-Born-Institute Berlin
- Bestec GmbH Berlin
- AIS Automation GmbH Dresden
- Carl Zeiss SMT AG Oberkochen

measuring spot on the sample is smaller than 2 mm.

The sample manipulation system of the reflectometer is located in the goniometer chamber (fig. 2). Mirrors and masks can be measured, with a diameter of up to 50 cm, a thickness of up to 20 cm and a weight of up to 30 kg. The incidence angle of the EUV beam can be varied from 3° to 60°.

A complete control system controls the operation of the reflectometer.

With this laboratory reflectometer it is now possible to measure flat as well as curved samples. Here the relative deviation of the peak reflectance are below 0.5 % and of the wavelength below 0.013 %. During a long-term test over 3 months the relative deviation for the peak reflectivity was only 0.13 %. The relative deviation of the wavelength was unchanged 0.013 %.

By the help of such an excellent measuring device it is also possible to optimize quickly the coatings of convex and concave mirrors. These surfaces can be applied for Schwarzschild optics used in EUV microscopy or EUV spectroscopy applications. Even strongly curved surfaces have to be coated with precise gradients. This coating requires the usage of special masks with lateral varying transmissions of the coating material. The exact design of the mask has been determined by an iterative process of coating and measuring.

In the case of optic systems, like Schwarzschild optic, the center wavelength of the single mirrors have to be matched exactly. The coating can be optimized effectively for a special optic design by using an own laboratory reflectometer.

Besides the usual measuring operation, the laboratory reflectometer provides the opportunity to measure with grazing incidence of the EUV beam for smaller samples. Thus, film properties can be determined by measuring of the total reflection edge.

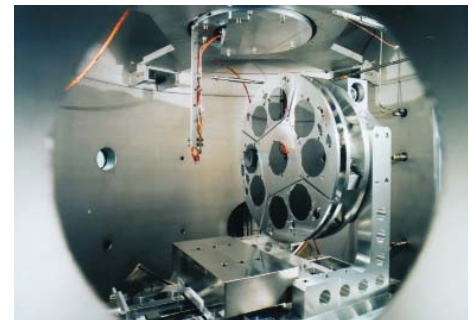


Fig. 2: Sample manipulation system within the goniometer chamber (Fig.: Fraunhofer IWS Dresden)



Fig. 3: Precision drive for the grating movements for the wavelength adjustment at the monochromator exit (Fig.: Fraunhofer IWS Dresden)

Contacts for further information:

Fraunhofer Institute for Material and Beam Technology IWS Dresden
Winterbergstr. 28

01277 Dresden, Germany

Dr. Ludwig van Loyen

Phone +49 (0) 351 / 2583 422

Fax +49 (0) 351 / 2583 300

Mail ludwig.vanloyen@

iws.fraunhofer.de

Oxidic nano composite films

Nano composites

Composites can be explained as materials which consists at least of two different materials (phases). These different materials are not mixed on a nuclear level and do not build up a chemical compound. A common form of composites is the one in which parts of the one material are evenly distributed within the other material (matrix). Composites often have properties, which cannot be achieved by single-phased material (e. g. high stability). Well-known composite materials are: e. g. concrete (gravel particles in concrete matrix) or carbide (tungsten carbide particles in cobalt matrix).

As the measurable hardness of crystalline hard material is often clearly beneath its theoretical hardness, which can be figured out of the bond force. This is caused by crystal defects which cannot be avoided during the production. If the size of hard material is reduced to some few nanometers the defects can move to the particle surface and thus they will become effect-less. If composites are constructed by such particles extreme macroscopical hardness values will emerge. Important is here that the matrix share is very small (few per cents) and that the matrix will be present in an amorphous mode (that means non-crystalline). Thus the track formation will be constrained. Such nano composites have been recently produced in the shape of thin films, e. g. of a titanium nitride particle in a silicium nitride matrix and circonium nitride particles in a copper matrix (fig. 1). In these films extreme hardnesses of 50 resp. 64 GPa have been measured (the hardness of the correspondent single-phase films amounts 25 resp. 16 GPa).

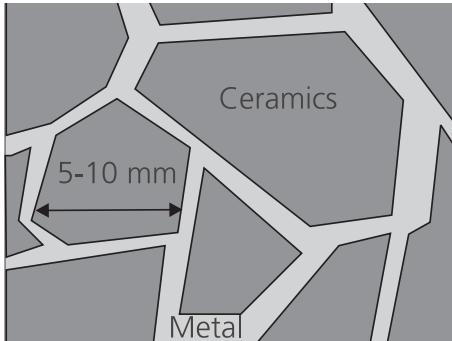


Fig. 1: Structure of a nano composite (schematic)
(Fig.: Fraunhofer IST Braunschweig)

Project partners:

- Fraunhofer Institute for Surface Engineering and Thin Films IST Braunschweig
- Robert Bosch GmbH Stuttgart
- Volkswagen AG Wolfsburg

In the case of the minimization of abrasion the hardness of the film is the most important factor. Other factors, however, are of high importance, too, such as the friction behaviour, the ductility and chemical and thermal stability. Especially with respect to the chemical and thermal stability certain oxides are superior to the nitrides. Thus the aim of the described project was the production of nano composites with extremely high wear resistance under the influence of multiple stress.

Film production

The field of nano composite films is quite new and up to now we have only few information about suitable material combinations and about the production process. Thus we have chosen materials, which are high melting and especially hard, namely aluminium oxide (corundum) and titanium dioxide. Especially copper, but also tungsten, zinc and tin have been investigated as matrix.

The films were produced by the help of gas flux sputtering, a PVD procedure, which is based on hollow cathode glow discharge and a gas flux controlled material transport. Due to the operating powers (0.5 mbar) and the specific transport mechanism, the agglomerated nano particles can be produced already in the vapour phase by gas flow sputtering (fig. 2). The film deposition takes place either via two independent sources for particle and matrix (co-deposition) or via one source with a composed target.

Film structure

The hardest oxide films were produced on the basis of TiO_2 . Results gained by TEM and XRD surprisingly revealed that copper is not stored temporarily in the particle spans, but is discarded as crystalline pillars (system TiO_2 / Cu).

The emerging nano composite, however, is constructed by crystalline TiO_2 in rutile structure and by amorphous TiO_2 as matrix (fig. 3). Films of this structure, even without copper, reveal hardnesses of up to 25 GPa, a value that exceeds the hardness of common titanium dioxide films (11 - 15 GPa).

Film characteristics

The wear characteristics of films have been investigated by a pin-disk test. Here an antibody is moved on a film during stress on a round stress mark. As antibody we used a hard, fixed ceramic ball (Al_2O_3) with 4 mm diameter. The load was 2 N, the resulting surface pressing 900 N mm^{-2} . Under these circumstances we achieved a low wear of only $0.3 \cdot 10^{-6} \text{ mm}^3 / \text{Nm}$.

In the case of lubricated friction the wear was not measurable (limit $0.1 \cdot 10^{-6} \text{ mm}^3 / \text{Nm}$). Surprisingly the wear of the antibody was hardly noticeable. In the case of the unlubricated friction the value increased slightly with the friction way and arrived at a value 0.4. In the case of lubrication we observed a slight decrease and the values were around 0.1. In a salt atomised spray test (DIN 50021 SS) the films showed a very good corrosion

result. After 240 hours we could not notice any changes, a fact that is not typical for PVD films. Comprisingly, we could state that it is possible to gain excellent material characteristics for thin oxide films by well-directed structure adjustments in the nanometer range. As, up to now, only few investigations have been done, we can count on some surprises.

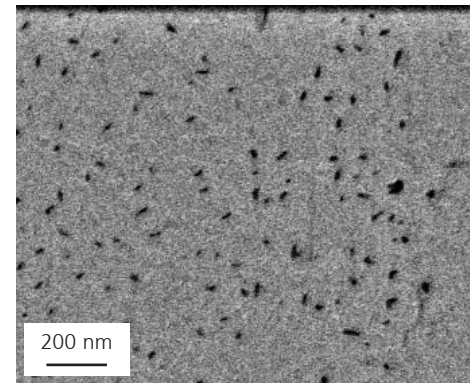


Fig. 2: TEM picture of tungsten nano particle (the size distribution has its focal point at 18.7 nm and a width of 11.0 nm) (Fig.: Fraunhofer IST Braunschweig)

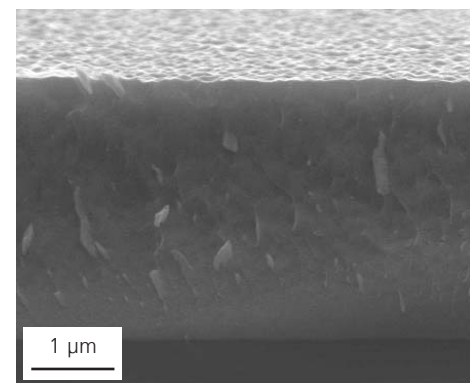


Fig. 3: SEM picture of the fraction edge of a TiO_2 nano composite film (Fig.: Fraunhofer IST Braunschweig)

Contacts for further information:

Fraunhofer Institute for Surface Engineering and Thin Films IST
 Braunschweig
 Bienroder Weg 54e
 38108 Braunschweig, Germany
 Dr. Thomas Jung
 Phone +49 (0) 531 / 2155 616
 Fax +49 (0) 531 / 2155 900
 Mail thomas.jung@ist.fraunhofer.de

Innovative reactors and in-situ analytics for nano protective films

Background

The continuous growth of the computer industry and the departure into the digital information age produce gigantic, continuously increasing demands to storage capabilities. If you rely on the annual transferred storage amount it becomes clear that, up to now, magnetic hard disk storage dominates in the storage technology. This leading position will be preserved during the next years. The advantage of the magnetical data storage results in the very high growth rate of the storage density on the data media.

With the introduction of thin film storage media and the first magneto-resistive reading heads the annual increase of the storage density goes up to 60 %. By the development of GMR reading heads it could even be increased to up 90 - 100 % per year.

The demand to increasingly higher area storage densities (product of bit and track density) on the applied thin film disks (hard disks) implies a reduction of the magnetic distance between head and disk.

A potential possibility to reduce the magnetic distance is besides the reduction of the altitude of the reading head over the magnetic storage disk, the reduction of the thickness of the magnetically inactive carbon-based protective films. The protective films e. g. have to be reduced from more than 100 Gbits / in² to 1 - 2 nm thickness, nevertheless, the functionality has to be maintained. During the coating of head and disk with amorphous carbon the surface-near part of the magnetic functional elements is destroyed by the penetrating of the film-building carbon species. This

so-called dead layer (magnetically inactive area) may be of a thickness of no more than 0.5 - 1.0 nm for the desired storage density. Furthermore, RMS roughness values of 0.1 - 0.2 nm are demanded for protective films on head and disk.

Presently different film types for head and disk are applied. For the protection of the head's writing - reading elements hydrogenous carbon films (a-C:H- or CH_x-films) are deposited by an ion beam process (IBCD - Ion Beam Chemical Vapor Deposition) in an argon - methane atmosphere. The magnetic storage disk, however, is protected by a film of amorphous carbon nitride (a-C:N or CN_x). This film is applied to an alloy, lying under the magnetical alloy, by reactive magnetron sputtering of a graphite target in an argon-nitrogen plasma. According to present insights it is not clear that the used protective films (< 3 nm thick) guarantee a sufficient functionality, especially with respect to the anti-corrosive effect.

Objectives

The following aims were defined for this project:

- systematical development, optimization and scaling of innovative plasma coating reactors, which might be suitable for the industrial production of functional ultra-thin protective films (2 nm),
- provision of suitable ex- and in-situ analysis procedures to characterize film and plasma parameters,
- functional film characterization by tests, which guarantee the suitability of the reactors and films.



Fig. 1: IBM-microdrive, one of the smallest commercial drives presently available, capacity 1 GByte, weight 16 g, size 42.8 · 36.4 · 5 mm³, storage density 15.2 Gbit per square inch (Fig.: IBM Deutschland E&TS GmbH Mainz)

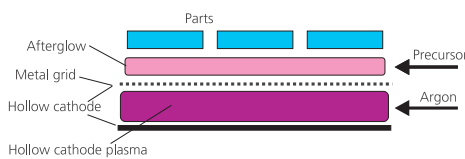


Fig. 2: PACVD-deposition with MHC-source (schematic) (Fig.: Fraunhofer IST Braunschweig)

Project partners:

- CCR GmbH Rheinbreitbach
- Fraunhofer Institute for Surface Engineering and Thin Films IST Braunschweig
- Fraunhofer Institute for Material and Beam Technology IWS Dresden
- IBM Deutschland E&TS GmbH Mainz
- IFU GmbH Lichtenau
- Sentech Instruments GmbH Berlin
- University of Technology Chemnitz, Institute of Physics
- Unaxis Deutschland GmbH
- University Cambridge, Department of Engineering
- University Mainz, Physics

These tasks have been worked out by an association of industrial partners, research institutes and universities:

- CCR GmbH Rheinbreitbach
- Fraunhofer Institute for Surface Engineering and Thin Films IST Braunschweig
- Fraunhofer Institute for Material and Beam Technology IWS Dresden
- IBM Deutschland E&TS GmbH Mainz
- IFU GmbH Lichtenau
- Sentech Instruments GmbH Berlin
- University of Technology Chemnitz, Institute of Physics
- Unaxis Deutschland GmbH
- University Cambridge, Engineering
- University Mainz, Physics.

As a casual partner we could welcome TFH Wildau (Advanced Technical College Wildau, Department Engineering).

Results and progress

Within this project we investigated different reactor types for the production of functional, ultra thin carbon protective films. These reactor types have been gradually optimized in cooperation with the producers:

- Copra-source (CCR technology)
- Carbon-Gun (Unaxis)
- high current arc source (Fraunhofer IWS)
- hollow cathode reactor (Fraunhofer IST)
- Penning-magnetron (University of Technology Chemnitz).

Scientists of the IWS developed a pulsed high current arc (HCA), which manifested itself as a very promising device to produce functional ultra thin carbon protective films. By this high current arc it is possible to produce anticorrosive carbon films of a thickness of only 1.7 nm. A prototype of

this high current arc source has been developed for the industrial disk production to produce ultra thin carbon protective films. Subsequently it has been investigated and optimized for a permanent application.

The RMS surface roughness (AFM) of carbon films, produced by the presented sources, is between 1.5 and 0.15 nm (exception Penning-magnetron). The density of the DLC films, deposited at room temperature varies between 1.8 g / cm³ (Copra-source) and 2.7 g / cm³ (HCA-source).

In general it can be said that all here presented sources are suitable for the production of ultra thin carbon films. With respect to the wear and corrosion parameter the HCA films seem to be especially suitable, followed by those films deposited by a CVD procedure.

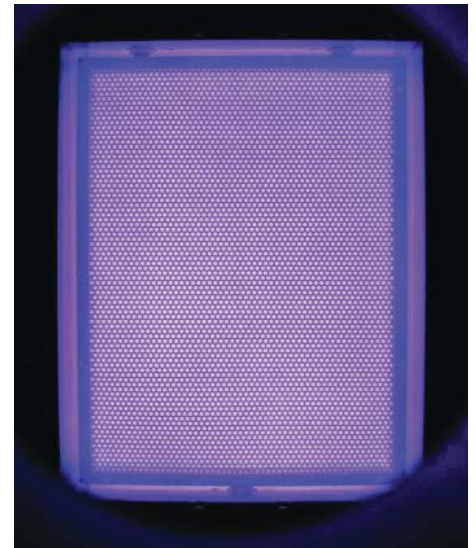


Fig. 3: MHC plasma source in operation (active surface: 150 · 200 mm², power: 1000 W, pressure: 0.2 mbar)
(Fig.: Fraunhofer IST Braunschweig)

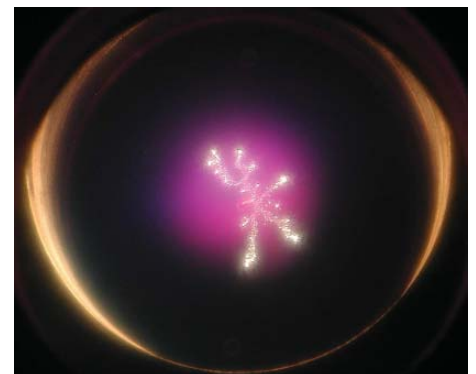


Fig. 4: Pulsed arc discharge on carbon as source of highly activated plasma for the deposition of super hard diamond-like protective films on hard disks and writing and reading heads
(Fig.: Fraunhofer IWS Dresden)

Contacts for further information:

IBM Deutschland E&TS GmbH
Mainz
Hechtsheimer Str. 2
55131 Mainz, Germany
Dr. Heinz Hilgers
Phone +49 (0) 6131 / 842 041
Fax +49 (0) 6131 / 846 810
Mail hilgers@de.ibm.com

Thin functional polymer films for nanoanalysis and the reaction technique

Motivation

Thin ($10 \text{ nm} < d < 100 \text{ nm}$) and ultra thin ($d < 10 \text{ nm}$) polymer film systems open numerous possible applications in the nanoanalysis, in the control of nuclear growth processes as well as in the nano reaction technique. These microstructured film systems should adhere on the surface, should be modifiable by bio-functional units (e. g. enzymes, antibodies, Oligo-DNA) and they should be used as transport systems of aqueous reactive partners.

State of the art

On the one hand the microstructured surface functionalization can be performed by the transfer of suitable low-molecular units by a micro stamp process (soft lithography). On the other hand it is possible to functionalize chemically thin molecular films in the sense of molecular resist-systems by electron beam lithography.

Recently scientists described the application of the electron beam lithography for the surface connection and linking of a suitable polymer system, for the first time.

Solution

The electron beam lithography structuring represents a very suitable method for the production of ultra thin PEO hydro gel films, structurable up to the submicrometer range (fig. 1). The degree of cross-linking and the source ability can be varied by the selection rate. As presented in fig. 2 a, b the different thickness increase of different strongly linked PEO-films heads to different changes of interference colours in reflecting light.

The reactivity in the film, necessary for a further functionalizing, can be achieved by final groups of PEO molecules. Thus the presence of reactive amino final groups could definitely be proved in the structured hydro gel film by the amino-specific fluorescence colouring. (Koop. Dr. Opitz / Max-Bergmann-Zentrum) (fig. 3). Thus we have a further requirement for the functionalization of gel films. A further chemical realization of the film with aldehydic groups enables the reductive silver deposition within the PEO gel film.

The applied chemical basic reaction is the long-known aldehyde reduction of an ammonia silver nitrate dilution, applied in the silver mirror production. Differently from the case of large area deposition of a continuous silver film, isolated nanoscopic silver particles are produced during a silver reduction in amino-terminated PEO films, which are immobilized in a hydro gel phase.

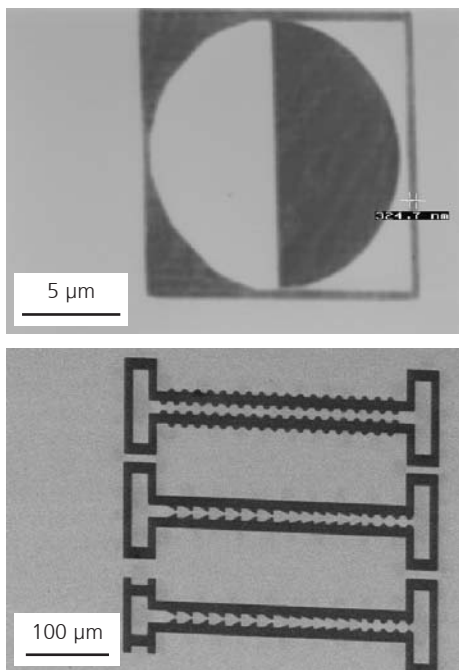


Fig. 1: SEM images of electron beam cured PEO structures ($d < 400 \text{ nm}$) (Fig.: Max-Bergmann-Zentrum für Biomaterialien Dresden)

Project partners:

- Max-Bergmann-Zentrum für Biomaterialien Dresden

Aims

After we could demonstrate the structuring, the surface bonding and reactivity of the PEO hydro gel film, we will deal with the integration of bio-functional molecules into the film. Special emphasis, in the sense of a reactive film design as part of a nano reaction system, is given to enzymes, e. g. peroxidase. The activity of the enzymes in the films will be made visible by the analytical detection by chemical luminescence of the film.

The biotinylation of the film is considered as an often used modification to an immobilization of bio-functional units. Oligo-DNA sequences similar to avidin labelled antibodies, serving as basis elements of molecular recognition processes belong to this group. Future aims will be the charging of micro fluid gel films at pre-defined places, the diffusive material transfer

into the films and the production of components interesting for material science, (controlled particle generation, diffusion-controlled structure generation).

The combination of structured film systems with electrode systems, which might enable an active control of the transport process will be of huge technological relevance. This may as well be applied to the linking of novel micro fluid transport principles such as the fluid transport by surface acoustic waves.

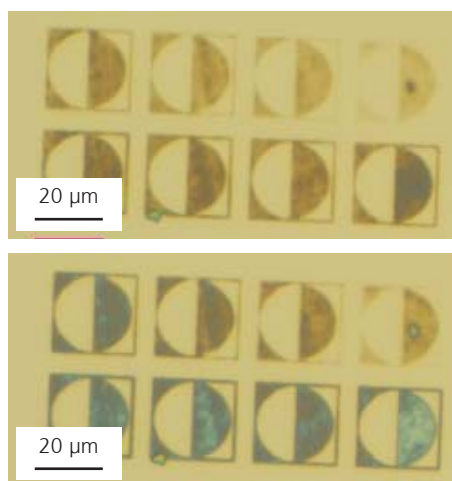


Fig. 2: Film thickness changes (colour changes) of structured PEO films of different linkings at welling with water (Fig.: Max-Bergmann-Zentrum für Biomaterialien Dresden)

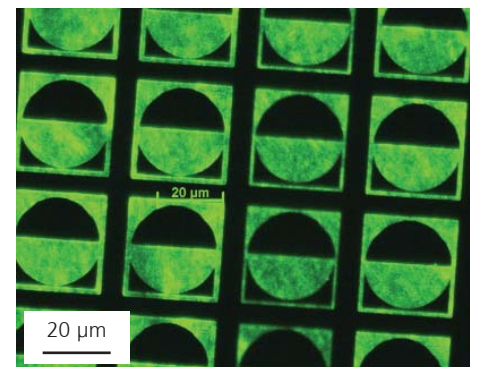


Fig. 3: Specific attachment of fluorescence-marked DNA-molecules on amino-terminal groups of the structured PEO hydrogel (Fig.: Max-Bergmann-Zentrum für Biomaterialien Dresden)

Contacts for further information:

Max-Bergmann-Zentrum für Biomaterialien Dresden
Hohe Str. 6
01069 Dresden, Germany
Dr. Hans-Georg Braun
Microstructuring Group
Phone +49 (0) 351 / 4658 548
Fax +49 (0) 351 / 4658 533
Mail braun@ipfdd.de

Nanotemplating from block copolymer supramolecular assemblies

Motivation: nanostructured materials

The current trend of the modern high-tech industry toward minimization and increase of surface/bulk feature density dictates the search of novel principles and routes. Although photolithography is currently able to produce feature sizes down towards 100 nm, the associated cost of their manufacturing increases rapidly as the process shifts to shorter wavelengths (X-ray or electron beam). Therefore, self-organization by means of a chemical or a physical driving force has attracted considerable interest as an alternative way to build periodic nanostructures with a feature size ranging from few nm up to sub-micrometers. Self-assembled nanostructured inorganic and soft materials open a venue to fabricate periodic arrays which are to be explored as templates for further processing.

Though self-assembly in block copolymers is a well-known phenomenon since 60ths, the needs of modern technology opens the "second breath" in this field. Since 1995, the interest in self-organization in ultrathin (10 - 100 nm) block copolymers films resulted in significant growth of investigations in this field.

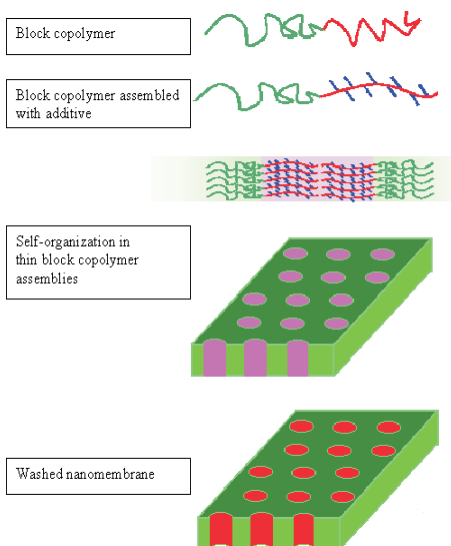


Fig. 1: Self-organization in block copolymers (Fig.: Leibniz Institute of Polymer Research Dresden)

Project partners:

- Leibniz Institute of Polymer Research Dresden
- Brandenburg University of Technology Cottbus

Self-organization and molecular assembly

In the Institute of Polymer Research Dresden, Department of Nanostructured Materials, we explore a new approach for fabrication of nanopatterned surface using self-organization in block copolymers. It is based on the molecular assembly of one of the blocks with active additives. The molecules of the additive are relatively mobile in the system because they are attached to the monomers by weak hydrogen bonds. Two important consequences come from this fact.

First, the self-assembly improves the order and well ordered samples can be obtained in several minutes. Secondly, the additive can be easily removed by washing in the suitable solvent leaving caves or tubes packed hexagonally in arrays with periodicity in the range of 10 to 100 nm. The typical examples of the obtained patterns are shown on the figures below.

Subsequently, the cylinders are to be filled with metals, electroconductive polymers etc. to obtain the arrays of columns of nanostructured materials. For example, we performed sputtering of Cr and Au to demonstrate the possibility of fabrication of metal nanodots or nanocolumns. Also, Ni ordered nanorods were electrodeposited through nanomembranes as an example of new nanostructured magnetic materials. It is worth to

compare the ideal information storage capacity of such material with the known data storage devices. Thus, our Ni sample contains 1.2 tera per cm^2 . If one considers every dot as a bit of information and compares the density with the information data density of a CD disk (700 MBytes placed on 97.9 cm^2 which gives ca. 57,000,000 bit per cm^2), he obtains 21 thousands times higher density of the new "nanoCD".

In collaboration with Prof. D. Schmeisser (Brandenburg University of Technology, Cottbus), we performed exciting experiments with X-ray photoemission electron microscopy (XPEEM) using synchrotron radiation at BESSY. The sample preparation procedure combines photolithography and micro-phase separation in self-assembled block-copolymer thin films. While photolithography pattern of insulating covering has periodicity of $12 \mu\text{m}$, the block copolymer film provides pattern with periodicity of 24 nm. Finally, electrodeposition of Ni results in two-scale hierarchy structure of Ni nanodots.

Future plans and perspectives

The prospective research directions of our group include, but are not limited by, fabrication of ordered arrays of nanowires, nanorods and nanotubes of magnetic and electrooptic materials. Also, we investigate fundamental properties of molecular assemblies of

block copolymers in order to control the morphology of the nanotemplates and to increase the ordered crystal-like domain size up to millimeter scale. We expect to obtain new nanostructured materials ordered on the macroscale.

The use of new self-organized materials based on the block copolymer assemblies opens new horizons in the modern technology. Of course, many fundamental and technical problems are to be solved on the long way from laboratory experiments to applications. Our group in the Department of Nanostructured Materials of Institute of Polymer Research Dresden performs application oriented fundamental investigations in this and another exciting fields of polymer physics, chemistry and materials science.

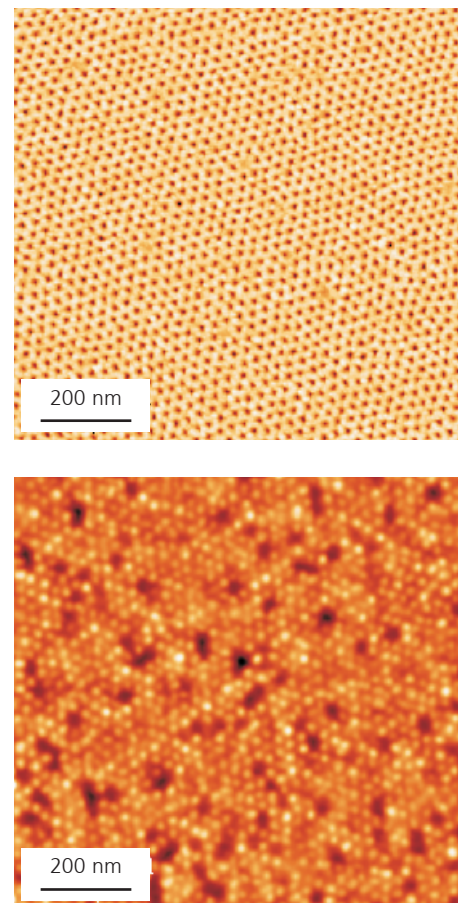


Fig. 2: Nano-template (above) and nano-structured material (Ni nano-rods), produced by a self-organized block copolymer (Fig.: Leibniz Institute of Polymer Research Dresden)

Contacts for further information:

Leibniz Institute of Polymer Research
Dresden
Hohe Str. 6
01069 Dresden, Germany
Dr. Alexander Sydorenko
Phone +49 (0) 351 / 4658 271
Fax +49 (0) 351 / 4658 281
Mail sydorenko@ipfdd.de

Fabrication and characterization of ultra-thin nano-structured diffusion barriers on Ta- and W-basis for metallization in the < 0.15 μm technology

Aim

The aim of this project is the evaluation of potential ultra-thin (≤ 10 nm), electrically conducting barrier films based on Ta and W for the application in future high integrated micro-electronic circuits (≤ 0.15 μm technology), produced by PVD and CVD processes.

The scaling down of the film thickness of diffusion barriers close to the atomic range is a challenge for the material and production choice as well as for the barrier film characterization. These tasks are new and partly not solved until now. Currently, we have not the material and procedures, which allow to fabricate reproducible only few monolayers thick and thermodynamically stable metallic thin films. Within the scope of this research project we will consider possible solutions related material science and process engineering. The technical and industrial realization will be shown as well. An alloying of elements in addition to the basis material Ta and W, should enable a nano-structure with crystallite sizes of only few nanometres down to an amorphous material with a preferably low defect density. The advantages and disadvantages of PVD and CVD deposition for these special thin films will be balanced. Here it is necessary to characterize the barrier films from the structural as well as the functional point of view.

Current results

Ternary diffusion barriers on Ta-basis

The structure and thermal stability of copper-covered barrier films with a film thickness of 10 nm on SiO_2 are investigated by x-ray, spectroscopical and chemical methods. PVD Ta-Si and

Ta-Si-N with a different nitrogen content were used as barrier material.

After the deposition in the initial state all investigated barrier films were amorphous. Depending on the barrier structure and heat treatment a generation of crystalline phases might be induced. The grain growth caused Cu-diffusion into or through the barrier. If the nitrogen content is increased, the thermal stability and the electrical resistance of the ultra-thin diffusion barriers is increased too. Table 1 shows typical results.

For an industrial application in Cu-multilayer metallization we favour TaSiN (2 sccm N_2) because here we found an optimum condition between barrier stability, specific electrical resistance of nearly $200 \mu\Omega\text{cm}$, intrinsic compression stress of $< (-400 \text{ MPa})$ and very low film roughness of $R_{\text{nms}} \sim 0.15$ nm. In figure 1 this barrier material on SiO_2 with copper as final film after an annealing at $450^\circ\text{C} / 1$ h, resp. $600^\circ\text{C} / 1$ h is shown. In both cases the amorphous structure of the diffusion barrier remains stable. If the temperature stress at $600^\circ\text{C} / 8$ h is continued, first reflexes of crystalline Ta_2N -areas will be noticed by x-ray diffraction. However, an initial copper diffusion could not be proved.

Diffusion barrier on W-basis

To deposit WN_x -films we used an PECVD procedure (plasma-enhanced CVD) with a WF_6 (Ar) / H_2 / N_2 gas composition. By varying the WF_6 / N_2 gas ratio and change of the total gas flow we produced 3 different films (with respect to W / N ratio [15 - 25 at % N]). In the initial state the WN_x -films have a film resistance in the range of $200 - 220 \mu\Omega\text{cm}$ and they are x-ray amorphous. An annealing at 600°C in a vacuum was necessary to unveil the differences

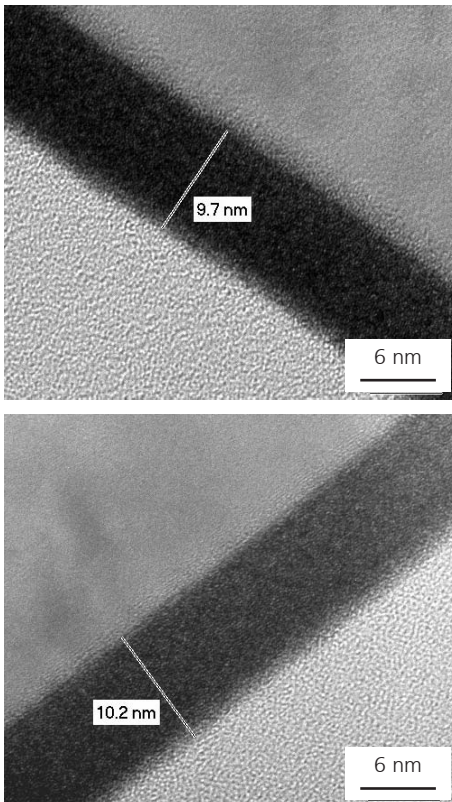


Fig. 1: 10 nm TaSiN-diffusion barrier on SiO_2 after different thermal stresses
above: $450^\circ\text{C} / 1$ h
below: $600^\circ\text{C} / 1$ h
(Fig.: AMD Saxony LLC & Co. KG)

Project partners:

- AMD Saxony LLC & Co. KG
- University of Technology Dresden, Institute for Semiconductor and Microsystems Technology
- University of Technology Chemnitz, Center for Microtechnologies
- IFW Dresden, Institute for Solid State Analysis and Structural Research

barrier films	investigated samples	structure of the barrier	Cu-diffusion into the barrier / SiO ₂ -film
Ta	600 °C / 16 h	crystalline / amorphous	not measured
	600 °C / 100 h	crystalline / amorphous	yes
TaSi	500 °C / 1 h	crystalline / amorphous	yes
TaSiN (1 sccm N ₂)	600 °C / 16 h	amorphous	not measured
	600 °C / 100 h	crystalline / amorphous	yes
TaSiN (2 sccm N ₂)	600 °C / 100 h	crystalline	no
TaSiN (3 sccm N ₂)	600 °C / 100 h	crystalline	no
TaSiN (4 sccm N ₂)	600 °C / 100 h	amorphous	no
	900 °C / 1 h	amorphous	no
TaN	600 °C / 100 h	amorphous	no
	900 °C / 1 h	crystalline	no

Tab. 1: Experimental results of ternary diffusion barriers of Ta-basis

between these films. WN_x1 (x ~ 0.18) and WN_x2 (x ~ 0.33) crystallize at 600 °C / 1 h in α-W and β-W₂N. WN_x3 (x = 0.25) shows not before 600 °C / 4 h small peaks of (100) levels of α-W and β-W₂N can be assigned to a higher amorphous degree of the films. This can be achieved by an extreme increase of the total Argon flow. Structural investigations of the stability in the initial state of amorphous WN_x-barriers show that no crystallization emerges up to a heat treatment of 550 °C / 1 h.

Figure 2 shows the film WN_x3 after annealing at 400 °C / 100 h in vacuum. No structural changes are visible in comparison to the initial state. At 600 °C β-W₂N and b.c.c.-W can be noticed without a detectable Cu-diffusion.

Further depositions of W-Si-N films were performed. Here the gas composition Silane (SiH₄ / WF₆ = 0.5 up 9) was added. X-ray investigation re-

vealed that these samples in their initial state were x-ray amorphous, too. After a heat treatment (600 °C in vacuum) these samples crystallize with a ratio 0.5; 1 and 1.5, mainly in α-W. There is only low evidence for β-W₂N, plainest for SiH₄ / WF₆ = 0.5. Higher ratios lead to a two film system, in which the SiON film can be formed during the long incubation period of the WN_x process. And thus the film becomes thicker the higher the Silane flow is.

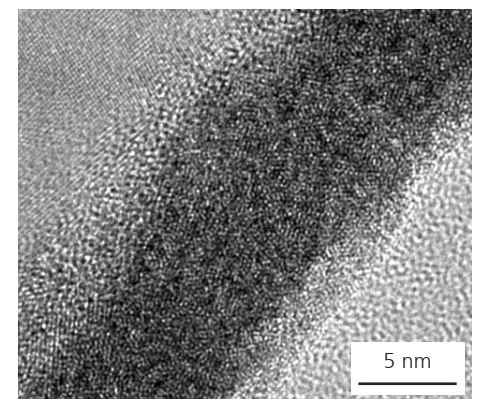
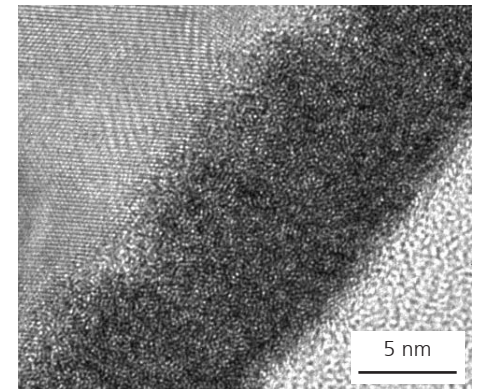


Fig. 2: Amorphous structure of the barrier in the initial state (above) and after the tempering 400 °C / 100 h (below) (Fig.: AMD Saxony LLC & Co. KG)

Contacts for further information:

University of Technology Dresden,
Institute for Semiconductor and Micro-
systems Technology
PO Box
01062 Dresden, Germany
Dr. Christian Wenzel
Phone +49 (0) 351 / 463 364 13
Fax +49 (0) 351 / 463 370 21
Mail wenzel@ihm.et.tu-dresden.de

A new certified reference material for the analysis of surface structures in the nanometre range

Aim

The development of nanotechnology is connected with growing demands on methods of nanoscale analysis in industry. Information on surface topography, chemical composition and physical properties of nanoscaled structures are of high importance for stable production processes. Accuracy and reproducibility of measuring results must be guaranteed and the corresponding measuring methods must be integrated into quality assurance systems. For these tasks suitable reference materials are needed.

State of the art

The lateral resolution of the measuring instruments is a key point for the analysis of nanoscaled structures. Thus reference materials are needed for the determination and the continuous control of lateral resolution and as a tool for the adjustment of instrument settings. Up to now there are no reference materials with regular structures in the range between the dimensions of the crystal lattice ($d < 1 \text{ nm}$) and lithographic structures ($d > 150 \text{ nm}$). Just in this gap you will find the lateral resolution of most devices, working with electron beams or ion beams to analyse the surface.

Solution

Within the project "Quality Assurance in Nanoscale Analysis" a new type of reference material has been developed and certified at the Federal Institute for Materials Research and Testing (BAM).

The certified reference material BAM-L002 "Nanoscale strip pattern for length calibration and testing of lateral resolution" is a cross sectioned multilayer stack of different semiconductors. The $\text{Al}_x\text{Ga}_{1-x}\text{As} - \text{In}_x\text{Ga}_{1-x}\text{As} - \text{GaAs}$ -layers were grown by Metal-organic Chemical Vapour Deposition (MOCVD) at the Institute for Solid State Physics at the University of Technology Berlin. The cross section of the multilayer stack (fig. 1) shows strip widths between 0.4 and 500 nm.

Results

The reference material enables the calibration of length scales and the determination of different instrument parameters such as lateral resolution, beam profile and smallest detectable structure. For this purpose the strip pattern includes a calibration distance, gratings with different strips widths, very narrow strips (δ -strips) and wide strips providing step transitions (fig. 1).

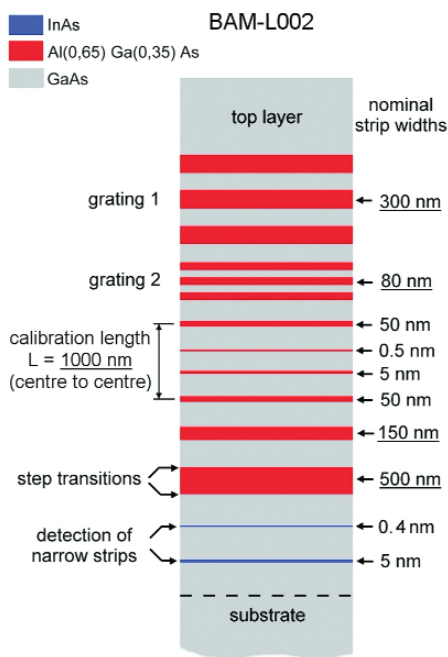


Fig. 1: Scheme of the strip pattern in the surface of the reference material BAM-L002 (Fig.: Federal Institute for Materials Research and Testing (BAM) [Bundesanstalt für Materialforschung und -prüfung])

Project partners:

- Federal Institute for Materials Research and Testing (BAM) (Bundesanstalt für Materialforschung und -prüfung (BAM))

The certification of strip widths and distances (cf. underlined values in figure 1) was performed by transmission electron microscopy (TEM). TEM-lamellae were prepared from different positions at the semiconductor wafer by focussed ion beam (FIB) and subsequently measured by a calibrated TEM. Finally the quality of the sample's surface after embedding, grinding and polishing was controlled by SEM (cf. fig. 2).

The certified reference material BAM-L002 has been successfully used in an interlaboratory test for secondary ion mass spectrometry (SIMS). Here the lateral resolution of the instruments was determined and their length calibration was checked. The image of a strip pattern taken with a Time of Flight-Secondary Ion Mass Spectrometer is presented in figure 3. The profile of the aluminium distribution shows that a grating with 80 nm strips is at the limit of resolution. The indium distribution shows a clear prove of an InAs strip with a width of approximately 0.4 nm.

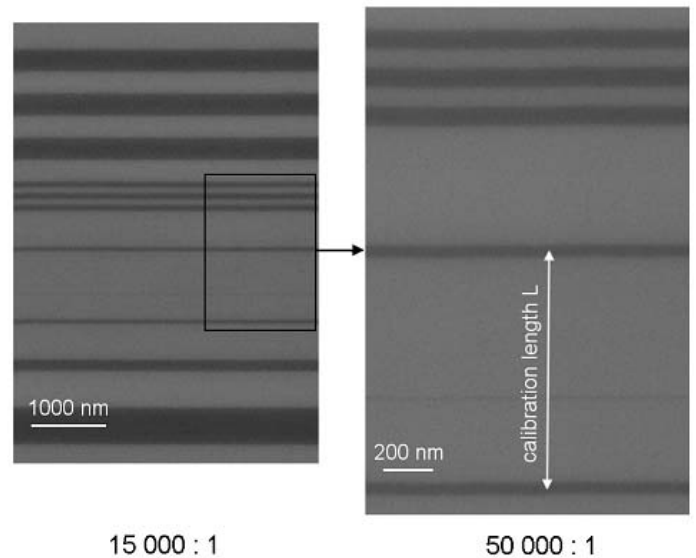


Fig. 2: Images of BAM-L002 taken by a scanning electron microscope at two different magnifications
(Fig.: Federal Institute for Materials Research and Testing (BAM) [Bundesanstalt für Materialforschung und -prüfung])

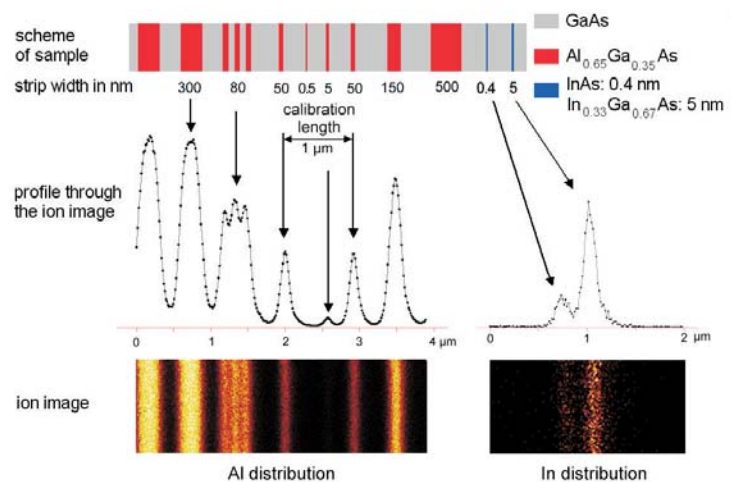


Fig. 3: ToF-SIMS analysis of BAM-L002
(Fig.: Federal Institute for Materials Research and Testing (BAM) [Bundesanstalt für Materialforschung und -prüfung])

Contacts for further information:

Federal Institute for Materials Research and Testing (BAM) [Bundesanstalt für Materialforschung und -prüfung]
Postfach
12200 Berlin, Germany
Dr. Mathias Senoner
Labor VIII.23
Phone +49 (0) 30 / 810 435 64
Fax +49 (0) 30 / 810 418 27
Mail mathias.senoner@bam.de



01: Agfa-Gevaert N.V.

Septestraat 27
B - 2640 Mortsel, Belgien

**02: ALOTec Angewandte Laser- und
Oberflächensystemtechnik GmbH**

Winterbergstraße 28
01277 Dresden

03: AMD Saxony LLC & Co. KG

PO Box 11 01 10
01330 Dresden

04: AMTEC GmbH

Anwendungszentrum für Mikrotech-
nologien Chemnitz
Annaberger Straße 240
09125 Chemnitz

**05: AMTEC Analysenmeßtechnik
GmbH**

Wachsmuthstraße 3
04229 Leipzig

**06: Applied Materials Europe
GmbH**

Buchenstraße 16
01097 Dresden

**07: AXO Dresden GmbH - Applied
X-Ray Optics**

Siegfried-Rädel-Straße 31
01809 Heidenau

08: Bestec GmbH

Carl-Scheele-Straße 14
12489 Berlin

**09: Biomet Merck BioMaterials
GmbH**

Frankfurter Straße 250
64271 Darmstadt

10: Bruker AXS GmbH

Östliche Rheinbrückenstraße 49
76187 Karlsruhe

**11: Federal Institute for Materials
Research and Testing (BAM)
(Bundesanstalt für Material-
forschung und -prüfung (BAM))**

PO Box
12200 Berlin

12: Capital Stage Nanotech GmbH

Domstraße 17
20095 Hamburg

**13: Carbo Tec - Gesellschaft für
nano- und biotechnische Produkte
mbH**

Arnold-Sommerfeld-Ring 2
52499 Baesweiler

**14: Centrotherm Elektrische Anla-
gen GmbH + Co. KG**

14a: Head office
Johannes-Schmid-Straße 3
89143 Blaubeuren

14b: Branch Dresden

Manfred-von-Ardenne-Ring 20
01099 Dresden

15: CeWOTec gGmbH

Lassallestraße 14
09117 Chemnitz

16: CreaPhys GmbH

Schmiedeberger Straße 38
01768 Reinhardtsgrμμα

**17: DAS Dünnschicht-Anlagen-
Systeme GmbH**

Gostritzer Straße 61 - 63
01217 Dresden

18: 3D-Micromac AG

Max-Planck-Straße 22b
09114 Chemnitz

**19: Dresdner Transferstelle für
Vakuumtechnik e.V.**

Löbtauer Straße 71
01159 Dresden

**20: European Society of Thin Films
(Europäische Forschungsgesell-
schaft "Dünne Schichten" e.V.)**

Gostritzer Straße 61 - 63
01217 Dresden

21: Feinchemie AG Sebnitz

Höhenweg 9
01855 Sebnitz

22: Feinmess Dresden GmbH

Am Promigberg 22
01108 Dresden

23: Fidura Capital Consult GmbH

Sendlingerstraße 24
80331 München

**24: Research Center Rossendorf
(Forschungszentrum Rossendorf
e.V.)**

PO Box 51 01 19
01314 Dresden

**25: Fraunhofer Institute for Manu-
facturing Technology and Applied
Materials Research IFAM**

(Fraunhofer-Institut für Fertigungs-
technik und Angewandte Material-
forschung IFAM)

Wiener Straße 12
28359 Bremen

**26: Fraunhofer Institute for Cera-
mic Technologies and Sintered
Materials IKTS**

(Fraunhofer-Institut für Keramische
Technologien und Sinterwerkstoffe
IKTS)

Winterbergstraße 28
01277 Dresden

**27: Fraunhofer Institute for
Photonic Microsystems IPMS**

(Fraunhofer-Institut für Photo-
nische Mikrosysteme IPMS)

Grenzstraße 28
01109 Dresden

28: Fraunhofer Institute for Surface Engineering and Thin Films IST (Fraunhofer-Institut für Schicht- und Oberflächentechnik IST)
 Bienroder Weg 54e
 38108 Braunschweig

29: Fraunhofer Institute for Material and Beam Technology IWS (Fraunhofer-Institut für Werkstoff- und Strahltechnik IWS)
 Winterbergstraße 28
 01277 Dresden

30: Fraunhofer Institute for Non-Destructive Testing IZFP (Fraunhofer-Institut für Zerstörungsfreie Prüfverfahren IZFP Saarbrücken)
 Branchlab for Acoustic Diagnosis and Quality Assurance EADQ Dresden
 Krügerstraße 22
 01326 Dresden

31: Fraunhofer Institute for Reliability and Microintegration IZM Berlin (Fraunhofer-Institut für Zuverlässigkeit und Mikrointegration IZM Berlin)
 31a: Branchlab Chemnitz, Department Micro Devices and Equipment
 Reichenhainer Straße 88
 09126 Chemnitz

31b: Micro Materials Center Chemnitz
 Otto-Schmerbach-Straße 19
 09117 Chemnitz

32: Fraunhofer Institute for Reliability and Microintegration IZM Berlin (Fraunhofer-Institut für Zuverlässigkeit und Mikrointegration IZM Berlin)
 Munich Branch of the Institute
 Hansastraße 27d
 80686 München

33: Freudenberg Forschungsdienste KG
 Höhnerweg 2 - 4
 69465 Weinheim

34: Gesellschaft für Wissens- und Technologietransfer mbH
 Reichenhainer Straße 88
 09126 Chemnitz

35: Gesellschaft zur Förderung von Medizin-, Bio- und Umwelttechnologien GMBU e.V.
 Arbeitsgruppe Funktionelle Schichten
 PO Box 52 01 65
 01317 Dresden

36: GeSiM GmbH
 Bautzner Landstraße 45
 OT Rossendorf
 01454 Radeberg

37: Hitachi High-Technologies Europe GmbH
 Europark Fichtenhain A12
 47807 Krefeld

38: Hochschule für Technik und Wirtschaft Mittweida
 Technikumplatz 17
 09648 Mittweida

39: IBM Deutschland E&TS GmbH
 Hechtsheimer Straße 2
 55131 Mainz

40: iLF Research and Development Company for Lacquers and Paints Ltd. (iLF Forschungs- und Entwicklungsgesellschaft Lacke und Farben mbH)
 Fichtestraße 29
 39112 Magdeburg

41: Incoatec - Innovative Coating Technologies GmbH
 Max-Planck-Straße 2
 21502 Geesthacht

42: Infineon Technologies AG München
 42a: Department Research
 Otto-Hahn-Ring 6
 81730 München

42b: Department Marketing
 St.-Martin-Straße 76
 81609 München

43: Infineon Technologies SC300 GmbH & Co. KG Dresden
 Königsbrücker Straße 180
 01099 Dresden

44: Institute for Lacquers and Paints (Institut für Lacke und Farben e.V. Magdeburg)
 Fichtestraße 29
 39112 Magdeburg

45: Interessenverband Chemnitzer Maschinenbau e.V.
 Annaberger Straße 240
 09125 Chemnitz

46: Kayser-Threde GmbH
 Wolfratshauser Straße 48
 81379 München

47: Laserinstitut Mittelsachsen e.V.
 Technikumplatz 17
 09648 Mittweida

48: Laser-Zentrum Hannover e.V.
 Hollerithallee 8
 30419 Hannover

49: Leibniz Institute for Solid State and Materials Research Dresden (Leibniz-Institut für Festkörper- und Werkstoffforschung e.V. Dresden)
 PO Box 27 01 16
 01171 Dresden

50: Leibniz Institute for Surface Modification Leipzig
(Leibniz-Institut für Oberflächenmodifizierung e.V. Leipzig)
Permoserstraße 15
04318 Leipzig

51: Leibniz Institute of Polymer Research Dresden
(Leibniz-Institut für Polymerforschung e.V. Dresden)
Hohe Straße 6
01069 Dresden

52: Leica Microsystems Semiconductor GmbH
Ernst-Leitz-Straße 17 - 37
35578 Wetzlar

53: Leybold Systems GmbH
Wilhelm-Rohn-Straße 25
63450 Hanau

54: Meier Vakuumtechnik GmbH
Vennweg 18
46395 Bocholt

55: Namos GmbH
Windbergstraße 54
01728 Bannewitz

56: Nanocraft
Innovationszentrum Engen
78234 Engen

57: Nanoparc GmbH
Bautzner Landstraße 45
OT Rossendorf
01454 Radeberg

58: Natural and Medical Sciences Institute at the University Tuebingen
(Naturwissenschaftliches und Medizinisches Institut an der Universität Tübingen)
Markwiesenstraße 55
72770 Reutlingen

59: Papiertechnische Stiftung
Heßstraße 134
80797 München

60: Photronics MZD GmbH & Co. KG
Grenzstraße 28
01109 Dresden

61: Physikalisch-Technische Bundesanstalt Braunschweig
PO Box 33 45
38023 Braunschweig

62: PicoRapid Technologie GmbH
Fahrenheitstraße 1
28359 Bremen

63: PINK GmbH Vakuumtechnik
PO Box 15 09
97865 Wertheim

64: Raith GmbH
Hauert 18, Technologiepark
44227 Dortmund

65: Robert Bosch GmbH
PO Box 10 60 50
70049 Stuttgart

66: ROTECH GmbH
Bautzner Landstraße 45
OT Rossendorf
01454 Radeberg

67: Roth & Rau AG
Gewerbering 3
OT Wüstenbrand
09337 Hohenstein-Ernstthal

68: Sachsen LB - Corporate Finance Holding GmbH
PO Box 10 02 06
04002 Leipzig

69: SENTECH Instruments GmbH
Carl-Scheele-Straße 16
12489 Berlin

70: Sick AG
Sebastian-Kneipp-Straße 1
79183 Waldkirch

71: Siemens AG Berlin
Siemensdamm 50
13629 Berlin

72: SURFACE
Rheinstraße 7
41836 Hückelhoven

73: SUSS MicroTec Testsystems GmbH
Süss-Straße 1
01561 Sacka

74: University of Technology Chemnitz, Institute of Physics (Institut für Physik)
PO Box
09107 Chemnitz

75: University of Technology Chemnitz, Institute of Chemistry (Institut für Chemie)
PO Box
09107 Chemnitz

76: University of Technology Chemnitz, Center for Microtechnologies (Zentrum für Mikrotechnologien)
PO Box
09107 Chemnitz

77: University of Technology Dresden, Institute for Material Science (Institut für Werkstoffwissenschaft)
PO Box
01062 Dresden

78: University of Technology Dresden, Institute for Semiconductor and Microsystems Technology (Institut für Halbleiter- und Mikrosystemtechnik)
PO Box
01062 Dresden

79: University of Technology Dresden, Institute for Applied Physics (Institut für Angewandte Physik)
PO Box
01062 Dresden

80: University of Technology Dresden, Institute of Physical Chemistry and Electrochemistry
(Institut für Physikalische Chemie und Elektrochemie)
PO Box
01062 Dresden

81: University of Technology Dresden, Institute for Macromolecular and Textile Chemistry
(Institut für Makromolekulare Chemie und Textilchemie)
PO Box
01062 Dresden

82: Triple-O Microscopy GmbH
Behlertstraße 26
14469 Potsdam

83: Unaxis AG
PO Box 1000
FL - 9496 Balzers, Liechtenstein

84: University Hannover, Institute for Semiconductor Devices and Electronic Materials
(Institut für Halbleiterbauelemente und Werkstoffe)
Appelstraße 11a
30167 Hannover

85: University Hannover, Institute for Solid State Physics
(Institut für Festkörperphysik)
Surface Science Section
Appelstraße 2
30167 Hannover

86: Verband Deutscher Maschinen- und Anlagenbau e.V.
PO Box 71 08 64
60528 Frankfurt

87: Wacker Siltronic AG
PO Box 11 40
84479 Burghausen

88: Zentrum Mikroelektronik Dresden AG
PO Box 80 01 34
01101 Dresden

new members in 2004:

89: German Textile Finishing Association
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Frankfurter Straße 10 - 14
65760 Eschborn

90: Arc Precision - Sources, Coatings and Analysis GmbH
Winterbergstraße 28
01277 Dresden

The Internet addresses of all members you can find under <http://www.nanotechnology.de/..../ger/s04/s04-ext.cgi> .

Contributors

Editorial staff: Dr. Ralf Jäckel

Coordination
/ Editing: Dr. Ralf Jäckel

Part of the results presented in this issue has been obtained within the frame of projects supported by Bundesministerium für Bildung und Forschung BMBF or Deutsche Forschungsgemeinschaft DFG.

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