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ISSN: 1864-225X

INM
Leibniz-Institut für
Neue Materialien

Jahresbericht 2008 | INM

Leibniz Gemeinschaft

INM
Leibniz-Institut für
Neue Materialien

Jahresbericht 2008

Annual Report

Jahresbericht

2008

INM – Leibniz-Institut für Neue Materialien

Ein Institut der Leibniz-Gemeinschaft

Saarbrücken

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Liebe Freunde des INM,

noch stärker als die vergangenen Jahre war das Jahr 2008 von der Neuorientierung unseres Instituts geprägt. Weitere neue, forschungsintensive Programmbereiche, und mit ihnen eine große Anzahl an neuen Mitarbeiterinnen und Mitarbeitern, haben ihre Arbeit aufgenommen:

- Zum 1. Januar 2008 wurde der Programmbereich Biomineralisation unter der Leitung von Frau PD Dr. Ingrid Weiss eingerichtet. Der Programmbereich erforscht Konzepte der Natur zur Synthese biologischer Materialien, die als Vorbild für das Design neuer Materialien dienen sollen.
- Die Juniorforschungsgruppe Nanotoxizität mit ihrer Leiterin Frau Dr. Annette Kraegeloh hat zum 1. Mai 2008 ihre Arbeit aufgenommen. Ziel der neuen Gruppe ist die Erforschung der Risiken nanopartikulärer Materialien für lebende Zellen und Gewebe.
- Der Programmbereich Nanotribologie wurde zum 1. Juni 2008 etabliert, Leiter ist Prof. Dr. Roland Bennewitz (vorher McGill University, Montreal, Kanada). Seine Gruppe befasst sich mit grundlegenden Fragestellungen zu Reibung und Verschleiß.
- Seit Juni 2008 unterstützt Prof. Dr. Robert McMeeking von der University of California in Santa Barbara das INM beim Aufbau des Programmbereichs Modellierung/Simulation.

Ein wesentlicher Schritt wird nun sein, dass die neu eingerichteten Gruppen mit den technologisch ausgerichteten Bereichen des INM gemeinsam neue Themen aufgreifen. Unser Ziel für die nächsten Jahre ist es, ein Gleichgewicht aus Grundlagenforschung und Anwendung am Institut zu schaffen und den Austausch von Ideen weiter zu intensivieren.

Auch als Wissensdrehscheibe hat sich das INM im vergangenen Jahr gut weiterentwickelt. Im Sommersemester 2008 hat das Institut eine neue Kolloquiumsreihe mit Vorträgen von hochkarätigen Wissenschaftlern aus dem In- und Ausland gestartet. Als Zuhörer werden hierzu neben den INM-Mitarbeitern auch die Mitarbeiter der Universität des Saarlandes (UdS) und ihrer An-Institute, sowie die Ausgründer des INM eingeladen. Die Kooperation mit der UdS wurde durch weitere Schritte intensiviert, etwa die Einrichtung eines gemeinsamen Studierendenaustausches mit der University of California in Santa Barbara (USA). Dieser fand im Sommer 2008 zum ersten Mal statt.

Ein besonderes Ereignis im Jahr 2008 war die Auszeichnung des INM als „Ausgewählter Ort“ im Rahmen des Wettbewerbs „365 Orte im Land der Ideen“. Hierfür öffnete das Institut seine Türen zu einer „Langen Nacht der Wissenschaften“. Fast 200 Schülerinnen und Schüler aus dem gesamten Saarland wurden mit einem Labor- und Versuchsprogramm mit den Chancen der Nanotechnologie vertraut gemacht.

Wir danken allen, die im Jahr 2008 zur erfolgreichen Arbeit des INM beigetra-

Prof. Dr. Eduard Arzt

Prof. Dr. Dr. h. c. Michael Veith

gen haben, herzlich, besonders unseren Mitarbeiterinnen und Mitarbeitern, den Zuwendungsgebern, den Kuratoriums- und Beiratsmitgliedern sowie den Kooperationspartnern aus Wissenschaft und Wirtschaft.

E. Arzt

Prof. Dr. Eduard Arzt
(Wissenschaftlicher Geschäftsführer und Vorsitzender der Geschäftsführung)

M. Veith

Prof. Dr. Dr. h. c. Michael Veith
(Wissenschaftlicher Geschäftsführer)

Dear friends of INM,

This past year has been primarily focussed on the re-organization of the institute. New research-intensive program divisions were established and a number of new employees joined us over the year.

- On January 1, the program division "Biomineralization", headed by Dr. Ingrid Weiss, was established. This program division explores biomimetic concepts for the synthesis of biological materials that will act as a model for the design of new materials.
- The junior research group "Nanotoxicity", headed by Dr. Annette Kraegeloh, was formed on May 1, 2008. The purpose of this new research group is to investigate the risks of nanoparticle interactions with living cells and tissues.
- The program division "Nanotribology" was established on June 1, 2008, headed by Prof. Dr. Roland Bennewitz (formerly of McGill University, Montreal, Canada). This group is engaged in fundamental questions concerning friction and plasticity.
- Since June 2008, Prof. Dr. Robert McMeeking, from the University of California in Santa Barbara, supports INM in the establishment of the program division "Modelling/Simulation".

For the newly established program divisions, an essential step will be to promote new ideas, in collaboration with the technologically orientated groups of INM. Our main purpose for the next few years is to balance fundamental research and application at INM and to continue to foster the exchange of ideas between the new and old program divisions.

With the creation of these new program divisions in the last year, INM has been steadily developing a scientific platform. In the summer term of 2008, INM launched a lecture series with outstanding national and international scientists. Participants of this lecture series include employees of INM, students and professors from the Saarland University and its associated institutes, as well as personnel from spin-off companies of the INM. Cooperation with the Saarland University has been further intensified by the establishment of a student exchange program with the University of California in Santa Barbara, USA, which took place for the first time in summer 2008.

A special event in 2008 was the recognition as "Landmark" within the contest "365 Landmarks in the Land of Ideas". As part of this special event, INM opened its doors for a "Long Night of Science". Almost 200 students from throughout Saarland came to learn about nanotechnology. The activities included lab tours and presentations, as well as a session where they had the chance to conduct their own experiments.

Our special thanks go to all who contributed to the successful work of INM in 2008, especially to our employees and our sponsors, to the members of the Board of Directors and of the Scientific Board, as well as to the partners from industry and research.

E. Arzt

Prof. Dr. Eduard Arzt
(Scientific Director and Chairman)

M. Veith

Prof. Dr. Dr. h. c. Michael Veith
(Scientific Director)

Scientific Articles/
Wissenschaftliche Artikel

Research on the synthesis and applicability of surface modified nanoscaled metal oxide particles as curing catalysers for UV-print-colours and lacquers

C. Becker-Willinger, S. Schmitz-Stöwe and D. Bentz

Abstract

In the BMBF joint research project – NANOCURE – New Curing Methods for Print-Colours, Glues and Lacquers (prom. ref. : 13N9115) - a new class of nanoscaled photo initiators for radical polymerization processes in the printing industry is to be investigated. A possible mechanism for the reaction of TiO_2 with acrylates, which is already described in literature is presented. Different methods of manufacturing TiO_2 nanoparticles in the lower nanometric size range and their subsequent processing are described, the method is of special interest as it may lead to a possible integrated production process of UV-printing inks. The TEM analysis shows the homogeneous arrangement of TiO_2 nanoparticles in an acrylic matrix, which is a necessary requirement for successful polymerization process. IR-spectroscopy is used to show the effect of TiO_2-nanoparticles as UV photocatalytic polymerization initiators.

Introduction

UV radiation curing polymerization methods are allowing a rapid upward trend in printing industry. The average growth ratio per year was about 5-8 % in the recent past. Besides the many advantages of the coating materials used for this application there is one problem which has attracted attention - especially in the field of food packaging - the migration of sometimes toxic byproducts of organic photo-catalysts [1, 2] into food.

Since October 2006 a new class of nanoscaled photo initiators for radical polymerization processes in the printing industry has been investigated.

Within the scope of the project's total objectives INM deals with several work packages. These include the improvement of material's basic properties according to crystalline and amorphous modified or unmodified metal oxides (especially TiO_2), the tailor-made surface modification of these particles regarding their compatibility to the binder matrix (especially acrylates), as well as studies on the curing kinetics and conversion rate of polymerization in the laboratory scale. Characterization methods of these materials and coatings include material science methods and application oriented tests. Up-scaling of material synthesis in the pilot plant stage is also planned.

The technical objectives are that the new photoinitiators exhibit a good radiation energy conversion performance, that they are harmless towards the environment and human health and that they can be produced in a cost efficient way.

Photocatalytic process and polymerisation

The formal photocatalytic process of an inorganic photo catalyst [3] such as TiO_2 [4] and an acrylate monomer can be described as follows: UV-radiation exposure leads to excited states of Ti^{4+} in TiO_2, thereby promoting an electron of the valence band (VB) to the conduction band (CB) leaving an electron defect ("hole") in the VB. If the HOMO (= Highest Occupied Molecular Orbital) of the acrylate monomer is located above the VB of the excited TiO_2, an acrylate electron is pumped into the defect centre created. Formally the acrylate is oxidized due to the electron loss and forms an acrylic radical, whereas the Ti^{4+} becomes reduced to Ti^{3+} (colour: dark blue / grey) due to the electron assimilation. This can be optically tracked by a temporarily grey colour of the acrylate coating. The acrylate radical itself can start the intended free radical polymerization process. In order to propagate the reaction, Ti^{3+} again has to be oxidized to Ti^{4+}. This aforementioned process is electrochemically favoured because the standard redox potential of the reaction $Ti^{4+} + e^- \rightarrow Ti^{3+}$ is $E_0 = +0.1$ V, as Ti^{3+} is a strong reductive species which is easily oxidized. Therefore an electron acceptor must be offered. This task can be optionally fulfilled by doping the TiO_2 basic structure or modifying the TiO_2 particle's surface.

At a critical diameter size which, for TiO_2 is 10 nm, the quantum dot effect becomes decisive for the particle's charge carrier dynamics. With decreasing particle size, the band gap of a semiconductor is broadened and its absorption is shifted to shorter wavelengths (blue shift). The shift of the absorption edge corresponds to higher redox potentials, as the VB is shifted to lower and the CB to higher energies.

In order to enable the TiO_2 particles to work as a photo initiator of a polymerization reaction, recombination of the charge carriers (excited electron and hole) must be delayed. In large particles, volume recombination prevails over the surface recombination thus leading to a low photocatalytic activity. Surface recombination is intensified by decreasing the particle size. However at a certain size recombination of electrons and holes occurs faster than the desired charge transfer with species which are adsorbed at the particle's surface. Finally the quantum efficiency of a photocatalytic reaction is given by the ratio of simultaneously running competition reactions: On the one hand the competition between charge carrier recombination and trapping (stabilization) of charge carriers and on the other hand the competition between recombination of trapped charge carriers and boundary surface-charge transfer can occur [5]. Known methods to increase the quantum efficiency are the doping of the TiO_2 with metal ions [6, 7] or double semiconductor systems and sensitizing with dyes [8, 9]. These methods lead to an electron injection into the TiO_2-particle.

In addition to this, the compatibility of TiO_2-nanoparticles with the acrylic matrix is of prime importance for the project's success in order to get a homogeneous distribution of particles in the system leading to a homogeneous distribution of polymerization initiating radicals.

TiO_2 synthesis and modification – TiO_2 dispersion in print acrylate mixtures and print process

Four different methods, based on refluxing under water separator technique for the synthesis and modification of TiO_2 were investigated. A number of Ti-precursors, solvents and hydrolyzing educts were examined. The surface modifiers were added during the synthesis process or applied after the manufacturing of TiO_2. A third method used an autoclave to produce surface modified TiO_2-nanoparticles. Modifiers used included aliphatic alcohols, UV-dye sensitizers, molecules with cumulative double-bondings or aromatic parts and silanes with organic network forming groups like acrylates or epoxides. Besides modification with N or S containing surface modifiers, structure doping with pentavalent ions was also tested.

In all three manufacturing processes the end product is a powder. For application in the printing process the powders have to be dispersed in an acrylate monomer mixture, consisting of a highly viscous, fast reacting acrylate combined with a low viscosity, but slowly reacting acrylate for reactive dilution in order to get a printable ink. A number of methods were attempted to disperse the dry nano-powder homogeneously in the acrylate matrix (different stirring systems, triple roller, disc mill), but the best results have been observed by using solvents such as toluene to pre-disperse the particles. Due to the application under radiation conditions in printing industry, the use of a solvent as a dilution aid has to be avoided. So the solvent must be removed from the acrylate monomer matrix under vacuum. In the production process this would be an additional time and cost factor.

As a result, a forth synthesis method, in which the particles were synthesized in an acrylic thinner matrix, was developed. In this method a master batch of particles was produced in a thinner acrylate which easily can be mixed with the fast curing highly viscous acrylates. The thinner acrylate matrix has to be compatible with the hydrolyzing component, while the reaction rate of the Ti-precursor and optionally the modifier has to be adapted to the changed reaction medium. The acrylic thinner matrix also should not gel or polymerize during the production process of the nanoparticles. Most of the produced particles were very small or in some cases amorphous. Nevertheless they show improved performance compared to the TiO_2-particles produced ex-situ. For amorphous particles this is not obvious at first sight, but it can be explained by the Anderson localization of electrons in amorphous materials [10, 11]. Figure 1 shows a TEM micrograph of crystalline TiO_2-particles produced in a thinner acrylate matrix.

Application of the print acrylate mixtures including the nanoscaled initiators was performed with a manual roller printing unit ("Little Joe - Model H") on PVC foil provided by a project partner. Curing was carried out with an IST-UV-Minicure lab machine ("Type M-25-1-Tr-SS"). The irradiated intensity (100 mW/cm² and 200 mW/cm²) as well as the conveyor belt speed (20 m/min up to 70 m/min) were varied. The dry-coat thickness measured after the curing process averaged between 2 and 8 µm (measured with TENCOR P-10, surface profiler), depending on the

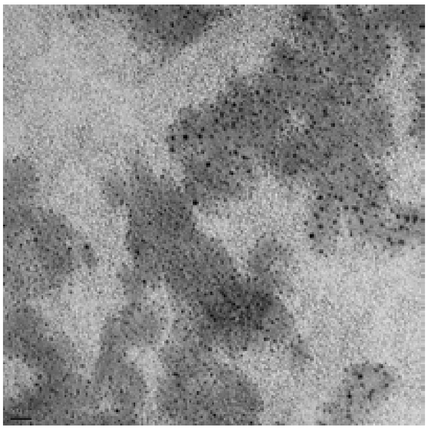

Figure 1: TEM micrograph of crystalline TiO_2-particles prepared in acrylic matrix.

- Light grey = background noise
- Dark grey patterns = dried acrylic matrix on TEM net
- Black structures = TiO_2-nanoparticles of about 1 to 4 nm diameter homogeneously distributed in the acrylic matrix

composition and viscosity of the applied acrylic matrix.

Performance of the particles produced in thinner acrylate

The curing properties of the used acrylates and their mixtures with and without photopolymerization catalytic particles were investigated by IR-spectroscopy to assess the efficiency of the produced TiO_2. For this purpose the diminishment of the C=C-double bonding peak area (several peaks from 1650 to 1550 cm^{-1}) is related to the fixed C=O bonding peak area (at 1730 cm^{-1}). The ratio [C=C : C=O] therefore should become smaller for increasing C=C-double bond conversion.

For application in the printing industry 65 to 75 % polymerization is required fingerprint dry, in order to avoid the gluing of printed papers or foils in a stack. This can be calculated from the IR-spectra, using the start and end ratios of the peak areas. Figure 2 shows the required numbers of curing cycles in order to get a fingerprint dry coating using an acrylate without initiator, and one with TiO_2, manufactured in an autoclave and one with TiO_2 manufactured in acrylate. Finally it was shown, that particles which were prepared directly in thinner acrylates have the most significant acceleration effect on the polymerization process.

The polymerization behaviour can also be observed if a mixture of different acrylates is used.

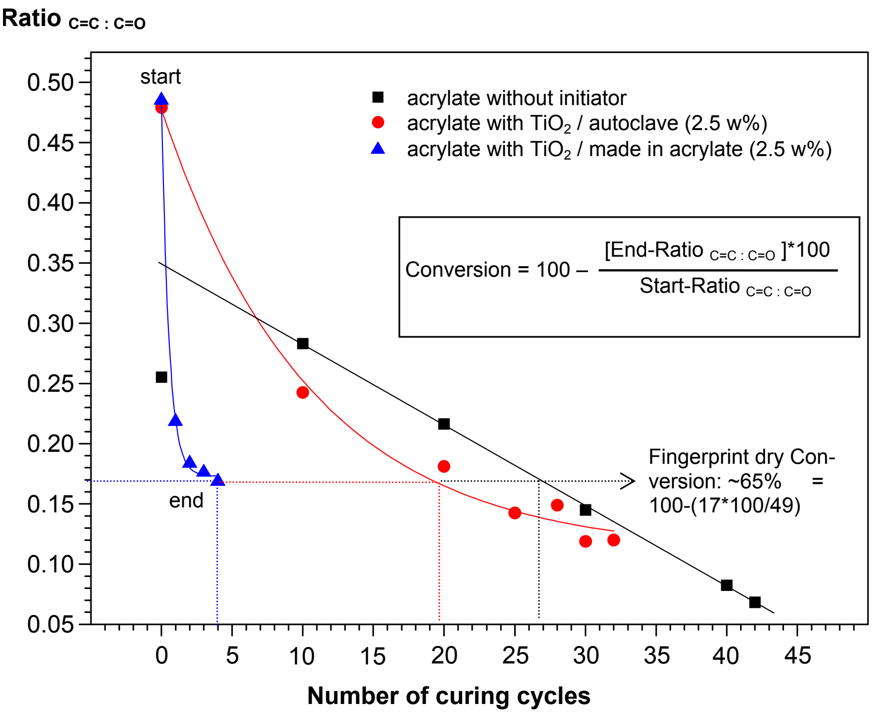

Figure 2: Needed number of cycles to get a fingerprint dry coating on PVC-foil.

Cross cut / tape test on PVC foil showed an excellent adhesion of the coating material leading to the conclusion that any possible bad influence of the nanoparticles on the adhesion is too weak to be noticed. The acrylate mixtures with TiO_2 are transparent and most of them show a slight yellow color.

Outlook

Further investigations into upscaling of the synthesis process are well underway and are being optimized. In cooperation with the other project partners, pigmented acrylates will be tested in the next step, because the acrylates will be finally used as printing colours. The long term stability of the material also must be investigated and optimized.

References

[1] N. Arsu, J. Eng. Nat. Sci., 2006, 1, 1-20.

[2] J. Bieleman, T. Bolle, A. Braig, J. Glaser, M. Köhler, R. Spang, In: J. Bieleman, Ed., Lackadditive, Wiley VCH: Weinheim [u. a.], 1998, 359-374.

[3] D. Beydoun, R. Amal, G. Low, S. McEvoy, J. Nanopart. Res., 1999, 1, 439-458.

[4] C. Dong, X. Ni, J. Macromol. Sci., Part A, 2004, A41, 547-563.

[5] J. Ye, X. Ni, C. Dong, J. Macromol. Sci., Part A, 2005, A42, 1451-1461.

[6] C. Damm, J. Photoch. Photobio. A, 2006, 181, 297-305.

[7] W. Choi, A. Termin, M. R. Hoffmann, J. Phys. Chem., 1994, 98, 13669-13679.

[8] A. L. Stroyuk, V. M. Granchak, A. V. Korzhak, S. Ya. Kuchmii, J. Photoch. Photobio. A, 2004, 162, 339-351.

[9] B. O'Regan, D. T. Schwartz, Chem. Mater., 1995, 7, 1349-1354.

[10] R. I. Bickley, T. Gonzalez-Carreno, J. S. Lees, L. Palmisano, R. J. D. Tilley, J. Solid State Chem., 1991, 92, 178-190.

[11] B. Ohtani, Y. Ogawa, S. Nishimoto, J. Phys. Chem. B, 1997, 101, 3746-3752.

Work on non photocatalytically active titania particles

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Abstract

Titanium dioxide has photocatalytic properties, i.e. under UV irradiation it develops an oxidative potential. In photocatalysis this is very desirable, but not when nano particulate titania is embedded into organic polymer matrices in order to increase the refractive index. UV irradiation would in this case destroy the material in the long run. For deactivation in general the titania is coated by e.g. silica or alumina which leads to other undesired effects like growth of the particle size and enhanced light scattering. The current work focuses on the application of techniques for doping during synthesis or crystallization of nano particulate TiO_2. The photocatalytic activity was determined by degradation experiments of 4-chlorophenol using dip coated glass plates under artificial sunlight, where decreases of the photocatalytic effect of up to 90 % were found.

Background

In order to increase the refractive index of a composite material, it is often necessary to embed high refractive index oxides into organic polymeric matrices. For optical applications – like reflective or anti reflective coatings – the use of highly transparent composite materials is a must. Therefore light scattering has to be avoided. For that reason the particle size of the embedded material needs to be smaller than about 1/20 of the shortest wavelength under use. With 400 nm as the low wavelength limit for the human vision, this translates to a size limit of 20 nm that may not be surpassed for the application fields.

Titania, both in the anatase as well as in the rutile modification, has a sufficiently high refractive index for the previously described application. Furthermore there are established preparation techniques [1] to obtain nano particles of anatase with mean particle sizes of 10 nm or less. These can be re-dispersed in suitable solvents or matrices at the level of the primary particles without any significant aggregation or agglomeration. However anatase shows a very high photocatalytic potential. This means that under UV irradiation, it develops a significant oxidative property. This can be explained by a look at the band structure of the semiconductor titania. The gap between the edges of the conductive (CB) and the valence band (VB) is 3.05 eV in the case of rutile and 3.23 eV in the case of anatase. This means that UV-photons can promote an electron from the VB to the CB. There are two reaction systems starting from water and oxygen respectively which can be initiated by the promoted electron or the hole remaining in the VB after the promotion. They lead to the formation of hydroxyl radicals being responsible for the oxidative attack on organics.

Although photocatalysis is desirable in many applications, it leads to problems when the material is embedded in organic matrices, where the UV irradiation leads to several undesired effects starting from yellowing of the matrix material and destruction of the synthesized material in the long term.

Rutile compared to anatase has a significantly lower photocatalytic activity, but being the high temperature modification, the formation of rutile from amorphous or anatase material in general leads to much bigger primary particles which are not suitable for the incorporation into transparent polymers for optical applications.

To elude the photocatalytic activity, methods are described in literature which are mainly based on covering the titania with shells of inert, isolating layers such as silica or alumina [2]. In general, this procedure leads to an increased particle size and thus to increased scattering of light. This is good for pigment applications, but not suitable for the described optical composite materials mentioned before.

Certain metals are known to "poison" the photocatalytic effect, by forming recombination sites in the titania, where exciton pairs are annihilated, preventing the oxidative effects. Attempts have been described utilizing the doping of the titania with ions of manganese or chromium [3]. The incorporation of these ions leads to a change in the band structure of the titania and a reduction of the band gap. The before mentioned reaction systems can no longer be activated diminishing the activity. But due to the reduction of the band gap, also blue light is absorbed by such a material, leading to a strong yellowing effect in comparison to undoped titania.

The ongoing work is targeted on the application of doping techniques using sodium ions to nanoscaled titania during the synthesis or crystallisation phase. Variations of the concentration of the dopant material and studies of the crystallization under lyothermal conditions were performed. This variation was necessary in order to minimize the deteriorating effects, especially a tendency towards particle growth.

Experimental

Preparation of the titania nanoparticles

The titania particles were prepared by a precipitation-crystallization technique starting form organic titania precursors such as titanium tetraisopropylate, $Ti(O^iPr)_4$. These precursors were diluted in suitable solvents, and were mixed with – soluable or partially soluable – sodium compounds (see below in chapter results) either before, during, or after the hydrolyzation step. For this, the mixture was treated with acids such as hydrochloric acid and a stoichiometric or sub-stoichiometric amount of water. This treatment was executed in such a manner, that in no point of time large, visible precipitates of gel or flocculates were formed in the sol. If such coarse-particles were formed, it was impossible to obtain nano particulate titania as the final product.

The sol was treated in a lyothermal process in order to obtain crystalline titania particles of anatase. Therefore, the sol was transferred into stainless steel pressure vessels with Teflon liners, and was treated at temperatures between 150 and 250 °C under autogenously formed pressure. After a successful treatment, the vessels contained two phases, an organic liquid which was almost void of titanium and a precipitate in the form of a white slurry.

This white slurry could be dried under reduced pressure and gave a white paste or powder, which was readily dispersible in deionized water or diluted acids, e.g. hydrochloric acid with c=1 mole/l.

When dispersed in deionized water, the acidity of the dispersion obtained was around pH 2-3.

Preparation of thin films

For the evaluation of the photocatalytic behaviour of the materials, thin films on glass substrates were produced. A clear TiO_2 sol was used for this application. 1 g of nanoparticles were dispersed together with 1 g of 3,6,9-Trioxa-decanoic acid (TODA) in 19 g of an ethanol / water mixture of 50:50 by weight. For the application of the thin film a dip coating apparatus was utilized. The films were drawn at a speed of 2 mm/s and dried at 200 °C.

Photocatalytic degradation experiments

Photo degradation experiments were performed for the evaluation of the photo catalytic activity of the doped TiO_2 nanoparticles. 4-Chorophenole (4-CP) was used as the model substance. Sample solutions with a concentration of 4-CP of c = 50 µmoles/l were prepared. For each experiment a microscope slide was used; the coated surface of the silde was 9.25 cm^2, the volume of the 4-CP solution used in each experiment was 50 mL. The degradation experiments were performed under artificial solar light using an Atlas Suntester CPS+ equipped with a 750 W Xenon lamp. The concentration of the 4-CP was determined by UV-Vis spectroscopy. As a reference, films prepared with undoped anatase obtained from INM's standard process [4] were used.

Results

The effect of deactivation of titania by utilizing sodium dopant was reached fast, but the optimisation towards particles with suitable sizes took significant experimental work.

This work originally started with the idea of using sodium methylate as the hydrolysis catalyst for the formation of titaniumoxide / titaniumhydroxide from titanalcoholate precursors. When mixtures of Na-methanolate with an alcohol used as a dilutor were combined with titaniumalcoholates, only very big amorphous particles were obtained, delivering milky white suspensions. As so often, hydrolysis with strong bases leads to a fast particle growth.

When mixtures of Na-methanolate with an alcohol were adjusted with hydrochloric acid to acidic conditions, most of the sodium was removed from the mixture by the precipitation of sodiumchloride crystals. Under these conditions, as well

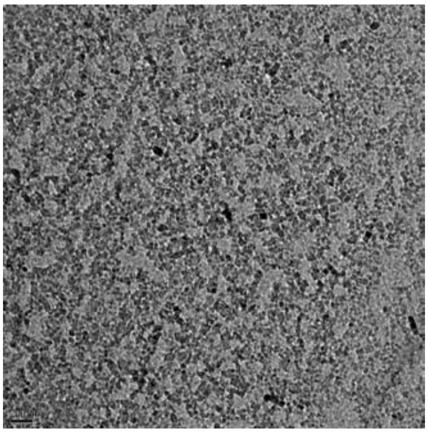


Figure 1: Transmission electron micrograph of a titania particle sample doped with 2 atom % sodium.

Figure 2: 10 % by weight particle suspensions of Na-doped particles with 1 % and 2 % sodium.

as in the case when sodium chloride was used as the direct dopant, titania particles were obtained which were comparable in their photocatalytic properties to undoped material. It can be concluded that under these conditions a lattice doping of the titania did not occur.

Smaller particles with significantly reduced photocatalytic activity were only prepared upon changing the dopant to sodium carboxylates such as sodium acetate or sodium oleate. In first experiments particles with sizes of 20 to 50 nm were obtained, a significant improvement compared to the first attempts but still much to large for transparent composite materials. Additional work resulted in samples of Na-doped anatase with small particle sizes (< 12 nm) and strongly diminished photocatalytic properties. In Figure 1, a transmission electron micrograph (TEM) of a sample of Titania doped with 2 wt% sodium is shown. Figure 2 depicts two samples of dispersions of sodium doped titania with 1 and 2 atom % sodium, respectively. In order to prepare these 10 wt% aqueous dispersions, no additional detergent or dispersing agent was needed. In Figure 3, the XRD data from a particle sample doped with 1 atom % sodium is plotted. The material obtained has anatase crystal structure with a mean crystallite size of 6 nm. Furthermore additional signals from halite were found; these sodium chloride crystals are due to the sample preparation, as no cleaning steps of the particles were executed. As in some of the TEM pictures also NaCl-crystals were found, it was tested whether thin films prepared from mixtures of photocatalytically active anatase with sodium chloride showed a reduction of the photocatalytic activity: the results were negative.

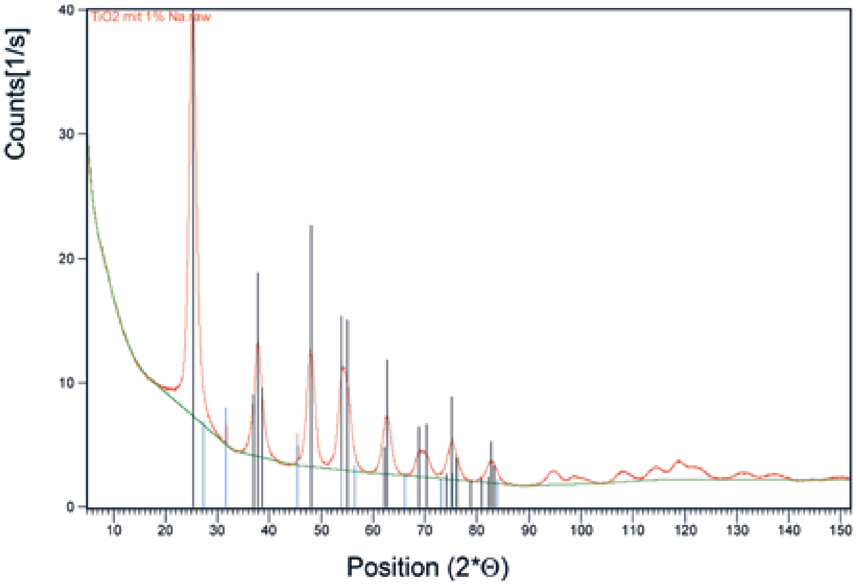


Figure 3: XRD Data from titania doped with 1 atom % sodium. Depicted are the measured signal as well as the signal positions of anatase and halite.

Figure 4 shows a typical degradation experiment with two sample films drawn from dispersions doped with 1 and 2 atom % Na in comparison to an undoped material. It can be seen, that in the 60 hours experiment with the undoped material, the test substance 4-CP was completely degraded, whereas in the 60 hours experiments involving the doped materials about 85 % of the 4-CP still remained unchanged after this time.

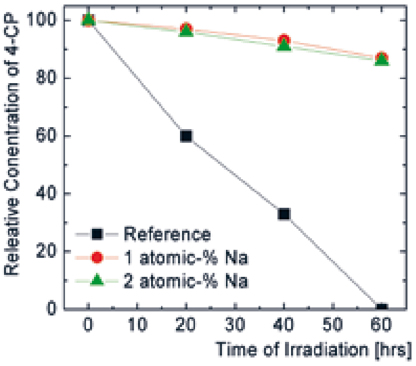


Figure 4: Degradation experiment applying films coated with Na-doped titania in comparison with undoped titania obtained from INM standard procedure.

Outlook

By further optimization of the doping process, e.g. by variations of doping timing or precursor substance, it is intended to further decrease the photocatalytic properties as well as to enhance the particulate properties like particle size.

Acknowledgement

The TEM picture was taken by A. Haettich, the XRD measurement was executed by R. Karos.

References

[1] M. Akarsu, E. Arpac, H. Schmidt, EP Patent 1 681 370 A2, 2006.

[2] F. Pflücker, J. Beck, B. Hirthe, S. John, N. Schulze, DE Patent 102 005 007 482 A1, 2006.

[3] J. S. Knowland, P. J. Dobson, G. Wakefield, WO Patent 99/060 994 A1, 1999.

[4] H. Schmidt, M. Akarsu, M. Naumann, Th. S. Müller, Trans. Mater. Res. Soc. Jpn., 2004, 29, 2717-2724.

Translucent UV light absorbant coatings for wood

A. Ullrich, B. Reinhard, M. Wittmar

Abstract

Efficient protection of fast growing domestic woods against the effects of weathering and UV irradiation is of increased significance to obtain improved dimensional stability and durability comparable to tropical wood. To advance the properties of these woods, a combined treatment process was developed. First, samples of pine sapwood were impregnated by project partners in the University of Göttingen to improve the dimensional stability and to avoid fungal infection of the wood, then a coating containing zinc oxide nanoparticles was applied on the impregnated wood samples at INM. Due to the UV light absorption of zinc oxide nanoparticles, the coated wood substrates are distinguished by enhanced colour stability. Together with the impregnation the coating prevents wood from deterioration by UV-irradiation and changing climates while retaining optical appearance.

The water-repellent properties of the coatings interfered with the impregnation and reduced the ingress of water. This resulted in a stabilising reduction of the swelling and shrinking and avoided the formation of cracks in both wood and coating. The improved characteristics were demonstrated by artificial weathering. A prototype of an impregnated and coated window has been manufactured and is currently exposed to outdoor weathering.

Introduction

European wood species show considerably poorer durability and dimensional stability in applications in outskirt areas compared to tropical wood species.

Therefore, the aim of this project was the development of a novel combined treatment for woods used for window constructions and in the structural-facings sector. The first step in this two-stage process was pressure impregnation to chemically modify the wood and lead to enhanced solidness and stability. The nanoparticulate composite coating was applied in the second step. It should ensure improved surface characteristics such as hydrophobic properties and absorption of UV light.

The impregnations were performed at the Department for Wood Biology and Wood Technology at the University of Göttingen [1-3]. The main task of the INM was the development of a functional coating providing improved water repellent properties and UV resistance.

Hydrophobic properties lead to decreased moisture uptake and consequently improved dimensional stability. The absorption of UV light should prevent photo-induced degradation of wood due to the decay of the lignin and the elution of its decomposition products. Further requirements for the coating included good adhesion on impregnated wood, permeability to water vapour, and additionally, boehmite particles should result in improved abrasiveness.

Experimental

High quality samples of pine sapwood with a size of 75 x 40 x 10 mm^3 were used for the experiments. The samples were impregnated at the University of Göttingen.

The coating consisted of an inorganic-organic hybrid matrix structure with embedded nanoparticles, including nano zinc oxide as an UV light absorbing component. The matrix was built of phenyltriethoxysilane, tetraethoxysilane and (3-glycidoxypropyl)-trialkoxysilanes, crosslinked via bisphenol A. The zinc oxide nanoparticles (ethanolic suspension with ZnO-content of 32 % by weight) were provided by Bühler-Partec GmbH, Saarbrücken. The coatings were applied using dip-, roll- or spray-techniques and cured at 120 °C in a furnace.

The properties of the coatings were characterized using artificial weathering [4] in a UV cabinet (alternating condensation atmosphere and UV irradiation) and a suntest chamber. Evaluation of the samples and especially the surfaces by crack characterisation was performed at regular intervals. The surface colour and brightness was determined with a Micro Color device according to the Commision International de l'Eclairage (CIE) based on the three dimensional LAB colour space [5]. Brightness is represented by the L axis from black (0 %) to white (100 %). Chromaticity is characterized by the two coordinates a (representing the red-green-axis) and b (representing the blue-yellow-axis). Chroma can be calculated according to the following equation:

$$C^* = \sqrt{(a^*)^2 + (b^*)^2}$$

Volume changes due to absorption of moisture were determined using a climate chamber with standard climatic conditions (20 °C, 65 % relative humidity) and humid climatic conditions (20 °C, 90 % relative humidity) [6]. Weight and

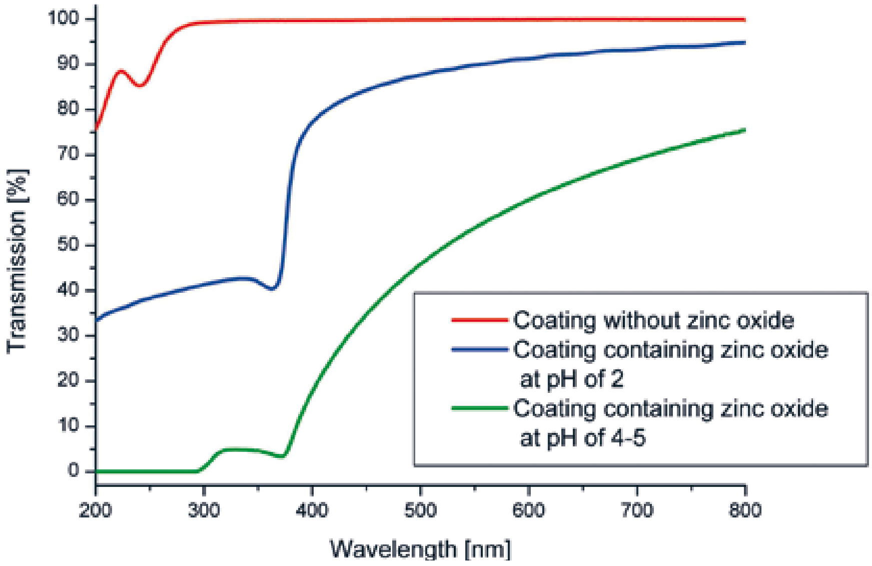


Figure 1: Transmission spectra of coatings on quartz glass sides.

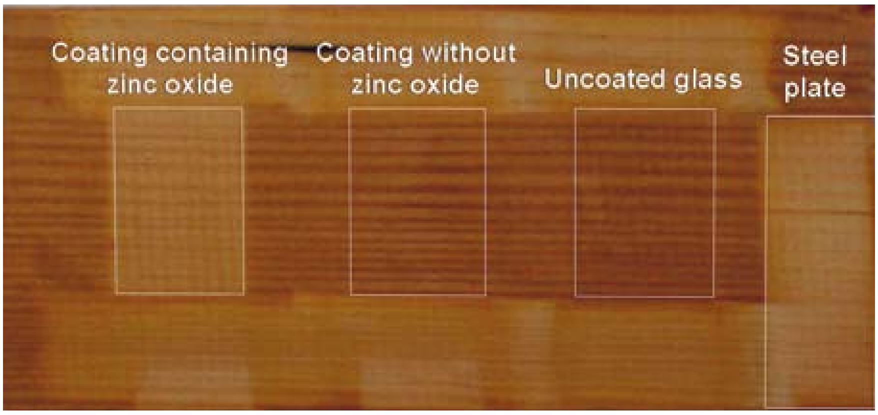


Figure 2: Wood sample after sun test; the areas were covered with different plates and glass sides during the test.

dimension gains were measured after reaching constant weight.

The abrasion resistance was determined using taber abrader tests [7]. The coating was applied to 10 x 10 cm glass plates and subjected to the wearing stress of two abrading arms each loaded with 500 g pressure. The loss of weight was measured after 1000 cycles.

Results and discussion

UV light absorption by the zinc oxide nanoparticles was tested by UV spectroscopy and using a suntest chamber. A 20 micron thick coating was applied on quartz glass sides. The transmission of UV light was measured with an UV spectrometer (Figure 1).

Within a wavelength range of 800 to 200 nm, 100 % transmission of light was observed without any UV absorbing agent. Using a sol containing zinc oxide particles, at a pH value of 2 due to the acidic reaction catalyst, a loss of transmission beginning at approximately 400 nm was observed. However transmission of UV light remained at almost 40 %. This reduction was due to the low pH value partly dissolving the zinc oxide. Setting the pH between 5 and 6 resulted in slowly decreasing absorption down to 5 % at 400 nm and finally to non measurable transmission at 300 nm.

In further experiments in the sun test chamber only slight browning was observed on areas covered with coated quartz glass. Figure 2 shows a sample after 8 weeks of irradiation. Two further areas were covered, one with uncoated glass and the other with a coating without zinc oxide particles. As expected, these areas showed browning similar to uncovered areas.

Lightness values (Table 1) confirmed the observations. Areas covered with zinc oxide containing coatings had darkened less compared to the areas without such a coating. Although, compared to the areas covered with steel a slight darkening was observed.

Covering	L	a	b	C
Initial values	83.6	5.7	21.0	21.8
Steel plate	78.4	9.3	26.8	28.4
Glass without coating	57.0	18.9	33.7	38.6
Coating without Zinc oxide nanoparticles	56.9	17.8	31.2	35.9
Coating including zinc oxide nanoparticles	67.7	13.9	32.0	34.9

Table 1: Colour values after 8 weeks of UV irradiation in sun test chamber.

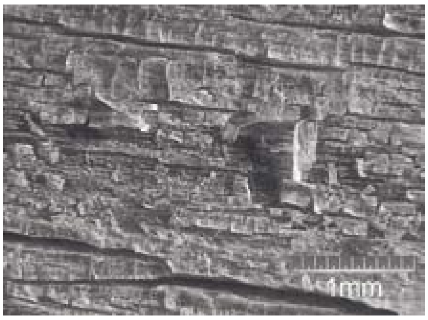


Figure 3: Light microscopic image of an inflexible coating after four weeks of artificial weathering.

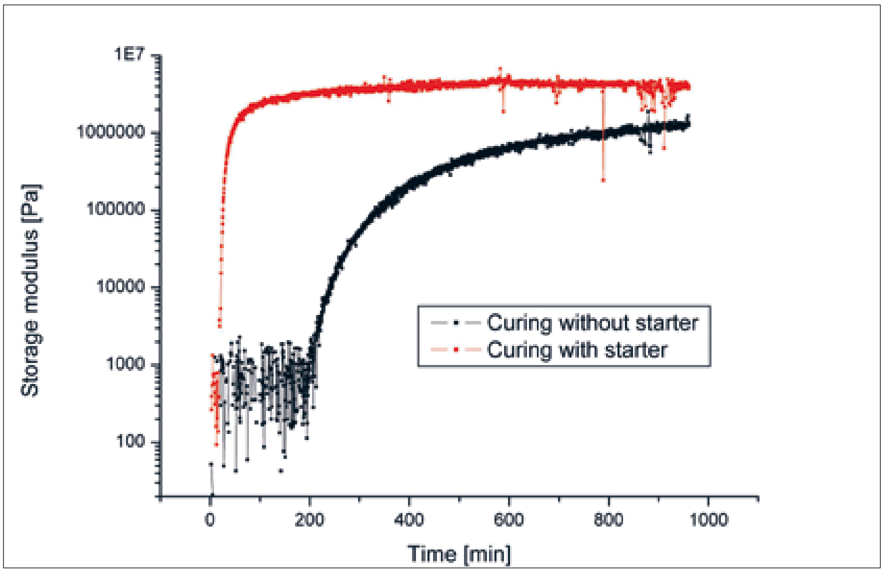


Figure 4: Dynamic mechanical thermal analysis of the coating sol with and without the addition of catalyst.

The investigation of treated and untreated wood specimens in a climate chamber using standard climate followed by humid climate showed a remarkable volume change even when using impregnated specimens (Table 2). The observed values scattered over a relatively wide range what can be attributed to the fact that the arboreal growth and the density have remarkable influence on the characteristics of wood.

	Volume gain [%]	Water uptake [mg/mm^3]
Untreated wood	5.0 – 6.7	41.5 – 46.1
Impregnated wood	2.9 – 3.1	27.9 – 34.3

Table 2: Volume gain and water uptake of wood caused by the change from standard climate conditions to humid climate conditions; all values are related to the volume at standard climate.

Therefore, it was necessary to develop flexible coatings to avoid crack formation and flaking of the coating as it was observed for coatings with a pure inorganic network (Figure 3).

Silanes with reactive organic groups in their side chains should allow organic crosslinking to improve flexibility. To this end, (3-glycidoxypropyl)-trialkoxysilanes and bisphenol A were used to crosslink the silanes via polymerisation of epoxide rings.

The reaction was initiated with a catalyst. In Figure 4, the curing behaviour with and without the addition of starter was measured by isothermal dynamic mechanical thermal analysis (DMTA) at 120 °C. The DMTA allows the characterization of the curing progress and the estimation of the necessary curing time. Without the catalyst the reaction did not start within three hours whereas the reaction with the catalyst was initiated immediately and reached a stable state after a short time indicating the completion of the reaction.

The coatings were manufactured by dip-, roll- or spray-techniques. To receive homogeneous and plane layers, the coatings were slightly abraded after curing followed by another coating step. The procedure was repeated several times if necessary.

The coatings showed excellent abrasion resistance as shown in Table 3.

	Weight loss [mg]
Coating without boehmite particles	21
Coating including boehmite particles	8

Table 3: Average values of weight loss as consequence of taber abrader tests.

Without the use of boehmite particles the coating was partly worn after 1000 test cycles (Figure 5, top), whereas boehmite containing coatings still showed complete surfaces without crack formation (Figure 5, bottom).

The coated specimens as well as some uncoated and non-impregnated samples were tested by artificial weathering using a Quick-UV-test (QUV) cabinet.

As a consequence of the treatment with silanes and adjacent heat curing, lightning values of the impregnated wood displayed a darker surface colour than untreated specimens before the weathering test. After one week of artificial weather-

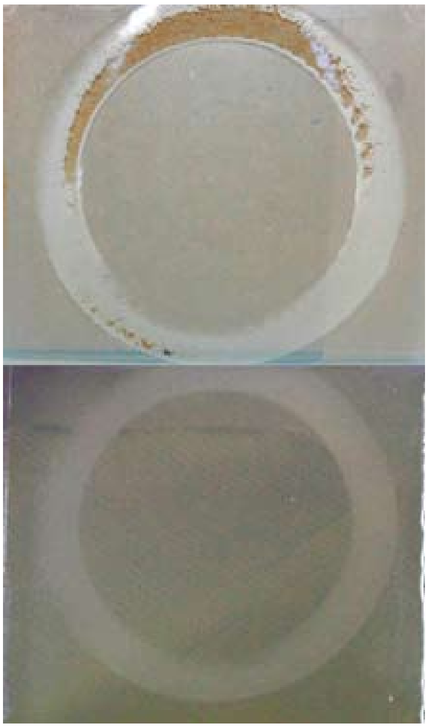


Figure 5: Coating without boehmite particles (top) and with boehmite particles (bottom) after 1000 cycles taber abrader test.

ing, completely untreated controls had darkened due to lignin oxidation and formation of chromophoric degradation products. Continuation of the experiment led to colour lightening caused by the wash-out of these degradation products (Figure 6 left).

After four weeks of artificial weathering impregnated wood without any coating showed internal cracks due to water infiltration. The colour of the wood lightened according to the wash-out of degraded lignin as already observed in the test of untreated wood when continuing the test with uncoated wood for up to 12 weeks.

Wood with inflexible coatings without organic crosslinking showed cracks and flaking after just 4 weeks of weathering as shown in Figure 3. However, no surface crack formation could be observed after 12 weeks using flexible coatings containing zinc oxide nanoparticles. Furthermore, enhanced colour stability was observed as no lignin had been washed out (Figure 6 right). An impregnated and coated window has been built and outdoor weathering experiments are currently underway.

Conclusions

In summary, a translucent coating which provides permanent protection against weathering and UV light degradation was developed. Integrated nano zinc oxide particles provided nearly complete UV protection.

The coating showed excellent adhesion on untreated and on impregnated wood, optical appearance, high abrasion resistance, UV light absorption and no crack formation due to weathering.

To compensate for the dimensional instabilities of weather exposed wood, it was necessary to build up a more flexible organic-inorganic hybrid matrix using (3-glycidoxypropyl)-trialkoxysilanes. This flexibility was adequate to equalize the dimensional fluctuations due to moisture absorption and to avoid crack formation. No lightening of the surface colour caused by the wash-out of degraded lignin could be observed after 12 weeks of artificial weathering.

Acknowledgments

The German Federal Ministry of Economics and Technology (BMWi) and the “Arbeitsgemeinschaft industrieller Forschungsvereinigungen ‘Otto von Guericke’ e.V. (AiF)” is gratefully acknowledged for financial support within the program for the promotion of “Industrial Joint Research (IGF)” (Research project V-Nr. 14897 N of the German Society for Wood Research (DGfH e. V.)).

We express our gratitude to Bühler Partec GmbH, Saarbrücken, for providing suspensions with ZnO nanoparticles.

References

[1] S. Donath, H. Militz, C. Mai, Holz Roh. Werkst., 2007, 65, 35-42.

[2] C. Mai, H. Militz, Wood Sci. Technol., 2004, 37, 339-348.

Figure 6: Untreated wood (left side) and impregnated and coated wood (right side) after 12 weeks of artificial weathering.

[3] C. Mai, H. Militz, Wood Sci. Technol., 2004, 37, 453-461.

[4] modified DIN EN 927-6: Exposure of wood coatings to artificial weathering using fluorescent UV lamps and water.

[5] DIN 6174: Colorimetric evaluation of colour coordinates and colour differences according to the approximately CIELAB colour space.

[6] DIN 52184: Determination of swelling and shrinkage.

[7] DIN 53754: Determination of abrasion, abrasive disk method.

Functional surfaces for controlled adhesion

E. Arzt and A. del Campo

Abstract

Adhesive joining with molecular (van der Waals) interactions without chemical glue is presently receiving much attention because of many potential applications. Research on how insects, spiders and geckos stick to surfaces has inspired a new paradigm: fibrillar surfaces with appropriate design can show much higher adhesion performance than flat surfaces. The insight gained in studying biological systems can be transferred to the development of optimized artificial attachment devices. By systematic variations of fiber diameter, aspect ratio and contact shape, we have produced, on a laboratory scale, artificial structures with adhesion strengths similar to the gecko. Further advances with switchable adhesion ("smart adhesives") have been demonstrated and may lead to interesting applications in medical products, sports equipment, construction materials and microfabrication.

Introduction

A new paradigm has emerged over the last years in adhesion: enhanced sticking of patterned *versus* planar surfaces. This concept of "contact splitting" [1] arises from the observation of flies, spiders and geckos, which exhibit attachment organs with long micro to nano-sized hairs, e.g. [2, 3]. The complex design of these hairs seems to be the key to their smart adhesion behavior, which enables repeated attachment and easy release from almost any kind of surface.

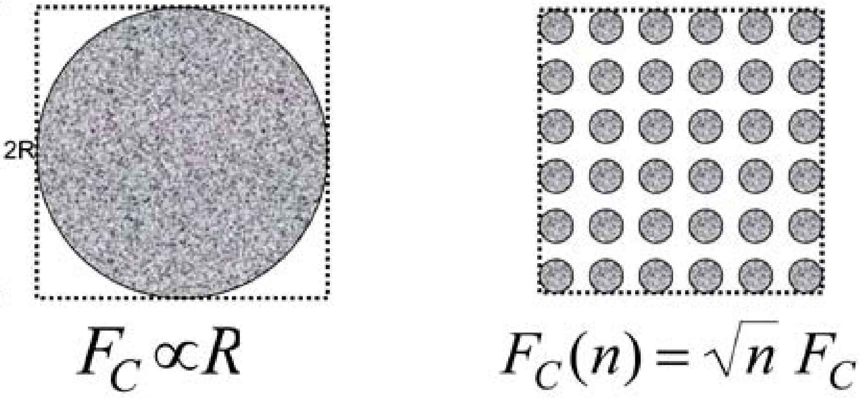


Figure 1: The principle of contact splitting: splitting of a reversible van der Waals contact (initially of radius R) into many fine contacts (new radii r) increases the adhesion force. This principle suggests the application of miniaturization technologies to achieve artificial adhesives.

Inspired by this principle, theory and experiment of reversible contact mechanics in small structures have made much progress in recent years, e.g. [4-16]. Yet attempts to produce such surfaces with significantly enhanced adhesion have so far found mixed success. While individual fibers exhibited the expected attachment, larger surfaces often did not demonstrate superior adhesion performance. The reason for this failure very likely lies in the complexity of the problem: the design parameters and underlying adhesion mechanisms need to be understood and optimized on the nano, micro and macro scale. For this purpose, singular experiments, as are frequently published in the literature, fail to advance the subject; rather, systematic model studies backed by suitable modelling efforts are required. The present contribution describes some of our recent progress in this area.

First systematic studies into effects of fiber size and shape

We have fabricated model fibrillar structures by soft-molding elastomeric precursors on photolithographic molds [17]. Typical structures, with 20 µm diameter, are shown in Figure 2. Adhesion studies on regular arrays of Poly(dimethylsiloxane) (PDMS) micropillars (pillar radii were varied between 2.5 and 25 µm and aspect ratio between 1 and 4) have proved the enhanced adhesion of structured surfaces against flat controls. Systematic studies of the influence of the contact radius and aspect ratio of the pillars on the final adhesion performance have demonstrated that decreasing the contact radius and increasing the aspect ratio of the pillars enhances adhesion [18].

New methods for the fabrication of structured polymer surfaces possessing pillars with controlled 3D tip geometries were developed: spherical, spatula-like and suction cups [19, 20]. The fabrication strategies exploit the filling mechanism of lithographic templates by viscous polymers, combined with inking and printing steps using elastomeric precursors with various viscosities and crosslinking kinetics. Homogeneously structured areas were obtained which allow reproducible and reliable testing of adhesion behaviour and can be scaled-up to prototypes. These fabrication routes do not require sophisticated 3D structuring equipment and can be extended to other materials, dimensions and geometries.

Our adhesion experiments show significant variation of the adhesion performance of the surfaces depending on the tip geometry and testing conditions [21]. The highest pull-off forces were found for mushroom-like pillars, which showed an increase of up to 30 times over the flat controls (see Figure 2, bottom and Figure 3). This corresponds roughly to the adhesion strength of a gecko toe. Flat and spherical shapes showed lower adhesion, while concave shapes resulted in the poorest performance, coupled with an unusual preload dependence.

The fiber radius effect studied previously augments the shape effect, creating vastly different “splitting efficiencies” [5] for the different shapes. Within the size range tested, mushroom and spatular shapes hold the greatest promise for further adhesion improvement through size reduction.

Towards switchable adhesion: “smart” surfaces

We have recently demonstrated that patterning technologies can be combined with responsive polymer materials to create microstructured surfaces with switchable adherence [22]. Application of an external field (e.g. temperature) causes changes in the topographical design, and this influences the final adhesion performance. A shape memory polymer was selected for this purpose. Arrays of microfibers, with diameters between 0.5 and 50 µm and lengths between 10 and 100 µm, were patterned by soft molding. Mechanical deformation at the shape-memory transition temperature, followed

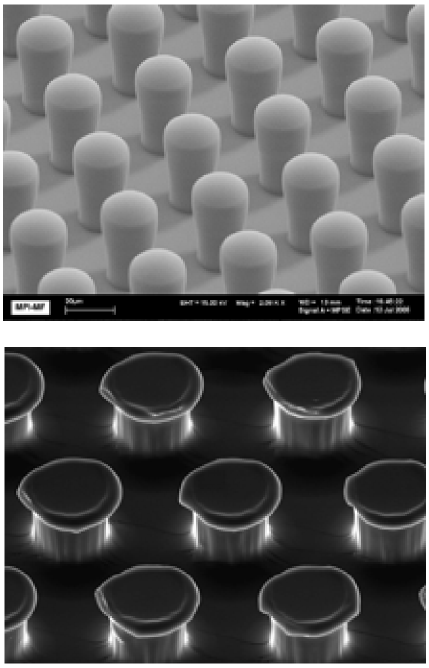


Figure 2: Micropatterned fibrillar PDMS surfaces: spherical tips (top) vs. mushroom-shaped tips (bottom). The diameter of both types of fibers is 20 µm. Adhesion is strongly enhanced by the presence of mushroom tips, which reach the adhesion strength of real gecko toes (after del Campo et al. [21]).

by cooling to room temperature in the deformed position yielded a temporary non-adhesive surface consisting of pillars in a tilted position. By reheating above the transition temperature, the patterned surface switches from the temporary non-adhesive state to a permanent adhesive surface with at least a 200-fold increase in adherence. Such active structures may have interesting applications in responsive systems where adhesion or friction management is required.

Outlook

While some of the ingredients of biological attachment systems have been successfully emulated in artificial systems, several challenges still lie ahead. Currently, a new adhesion test system is being developed, which will allow more advanced measurements (Figure 4). Obviously, biological devices are much more complex from a geometrical and materials point of view. These peculiarities still need to be analyzed theoretically; for example, the superiority of mushroom-type fibers has recently been explained by an improvement of the near-perimeter stress distribution by numerical methods [23]. The next challenge is to imitate the hierarchical structure of biological systems in order to achieve good adhesion to rough surfaces; a first attempt has been made [14], but the resulting adhesion strengths need to be improved to be of practical use. Biological fibers are most likely composed of gradient materials, e.g. to allow small fiber radii without the propensity to clumping [9]. Also asymmetric and anisotropic contact shapes, as they are present in flies and geckos, will need to be reproduced to combine reliable adhesion with easy detachment. Because of the large parameter space, experimental work needs to be accompanied by suitable modelling activities. For example, the concept of “adhesion design maps” [9], which are useful graphical displays of the trade-offs in producing efficient attachment structures, need to be extended from simple spherical to more complex shapes [24]. Finally, of course, practical applicability will depend on cost-effective fabrication routes for large areas; such developments are currently underway.

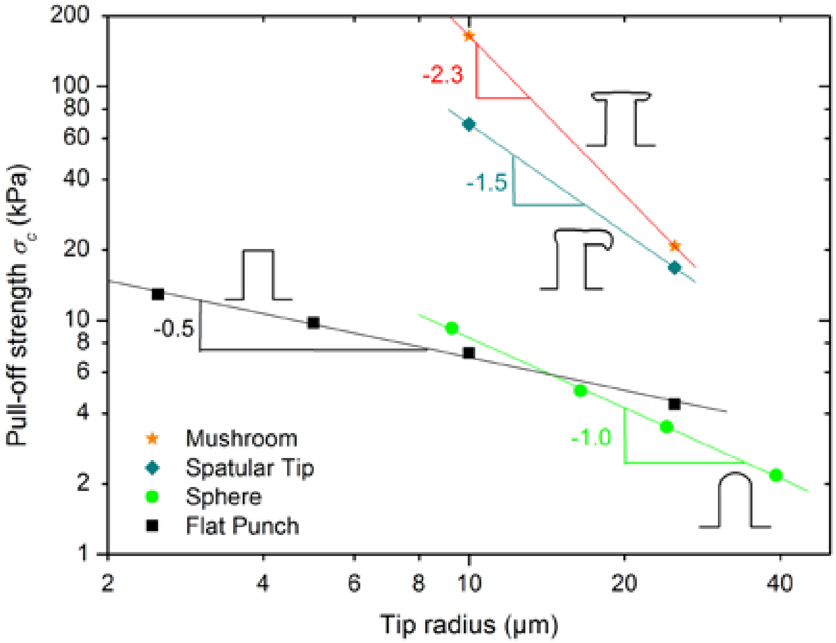


Figure 3: Adhesion strength (pull-off force divided by apparent contact area) of different contact shapes as a function of fiber radius. Mushroom and spatular tips scale most favorably to smaller dimensions (after [21]).

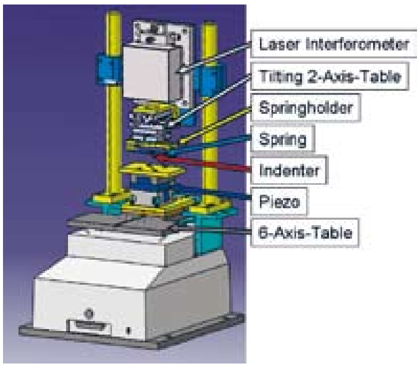


Figure 4: Schematic of a new adhesion tester for patterned surfaces, developed at INM [25].

References

[1] E. Arzt, S. Gorb, R. Spolenak, PNAS, 2003, 100, 10603-10606.

[2] S. Gorb, Attachment Devices of Insect Cuticle, Kluwer Academic Publishers: Dordrecht [u.a.], 2001.

[3] K. Autumn, Y. A. Liang, S. T. Hsieh, W. Zesch, W. P. Chan, T. W. Kenny, R. Fearing, R. J. Full, Nature, 2000, 405, 681-685.

[4] E. Arzt, Mater. Sci. Eng. C, 2006, 26, 1245-1250.

[5] R. Spolenak, S. Gorb, H. Gao, E. Arzt, P. Roy. Soc. A, 2005, 461, 305-319.

[6] B. N. J. Persson, J. Chem. Phys., 2003, 118, 7614-7621.

[7] C. Y. Hui, N. J. Glassmaker, A. Jagota, J. Adhesion 2005, 81, 699-721.

[8] L. Ma, R. McMeeking, E. Arzt, Int. J. Mats. Res., 2007, 98, 1156-1162.

[9] R. Spolenak, S. Gorb, E. Arzt, Acta Biomater., 2005, 1, 5-13.

[10] H. Yao, H. Gao, J. Mech. Phys. Solids, 2006, 54, 1120-1146.

[11] A. K. Geim, S. V. Dubonos, I. V. Grigorieva, K. S. Novoselov, A. A. Zhukov, S. Yu. Shapoval, Nature Mat., 2003, 2, 461-463.

[12] A. Ghatak, L. Mahadevan, J. Y. Chung, M. K. Chaudhury, V. Shenoy, P. Roy. Soc. A, 2004, 460, 2725-2735.

[13] J. Y. Chung, M. K. Chaudhury, J. R. Soc. Interface, 2005, 2, 55-61.

[14] M. T. Northen, K. L. Turner, Nanotechnology, 2005, 16, 1159-1166.

[15] M. Sitti, R. S. Fearing, J. Adh. Sci. Tech., 2003, 17, 1055-1073.

[16] A. J. Crosby, M. Hageman, A. Duncan, Langmuir, 2005, 21, 11738-11743.

[17] A. del Campo, E. Arzt, Macromol. Biosci., 2007, 7, 118-127.

[18] C. Greiner, A. del Campo, E. Arzt, Langmuir, 2007, 23, 3495-3502.

[19] A. del Campo, I. Alvarez, S. Filipe, M. Wilhelm, Adv. Funct. Mater., 2007, 17, 3590-3597.

[20] A. del Campo, C. Greiner, I. Alvarez, E. Arzt, Adv. Mater., 2007, 19, 1973-1977.

[21] A. del Campo, C. Greiner, E. Arzt, Langmuir, 2007, 23, 10235-10243.

[22] S. Reddy, E. Arzt, A. del Campo, Adv. Mater., 2007, 19, 3833-3837.

[23] A. V. Spuskanyuk, R. M. McMeeking, V. S. Deshpande, E. Arzt, Acta Biomater., 2008, 4, 1669-1676.

[24] C. Greiner, R. Spolenak, E. Arzt, Design Maps for Non-Spherical Contact Shapes, in preparation.

[25] E. Kroner, A. del Campo, E. Arzt, unpublished.

Kelvin Probe Force Microscopy of charged indentation-induced dislocation structures in KBr

P. Egberts and R. Bennewitz

Abstract

The incipient stages of plasticity in KBr single crystals have been examined in ultrahigh vacuum by means of Atomic Force Microscopy and Kelvin Probe Force Microscopy (KPFM). Conducting diamond-coated tips have been used to both indent the crystals and image the resulting plastic deformation. KPFM reveals that edge dislocations intersecting the surface carry a negative charge similar to kinks in surface steps, while screw dislocations show no contrast. Weak topographic features extending in <110> direction from the indentation are identified by atomic-resolution imaging to be pairs of edge dislocations of opposite sign, separated by a distance similar to the indenter radius. They indicate the glide of two parallel {110} planes perpendicular to the surface, a process that allows for a slice of KBr to be pushed away from the indentation site.

Introduction

The fundamental study of the initial stages of plasticity is challenging as there are few instruments that combine the ability to quantify plastic deformation with accurate depth and force measurement, as well as acquire high resolution images of the deformed structure. A recent development is the increased use of Scanning Probe Microscopy (SPM) in the study of plasticity. It is supported by the wide distribution of instruments, the outstanding resolution of surface imaging [1-3], and additionally, the capability of Atomic Force Microscopy (AFM) to measure forces with single-bond sensitivity [4-9]. The small contact radius of the probes used in SPM allows for the application of large stresses at small applied loads. Furthermore, the use of SPM probes as indenters can produce localized indentations, where the plastic zone can be confined between a few micrometers down to a few nanometers. Such small areas can be imaged beforehand to identify regions where the indented volume can be considered to be of perfect crystallinity, free from pre-existing dislocations. In these regions, plasticity must be accommodated by homogeneous nucleation of dislocations rather than activation of Frank-Read sources [10]. Incipient plasticity has now been studied using SPM for indentation and imaging on a number of surfaces, including MgO(100) [5], Au(100) [2-4], Au (111) [4, 6], KBr (100) [7], Cu (100) [8], and stepped Au surfaces [9], in both ambient and ultrahigh vacuum conditions.

Plastic deformation of ionic crystals is often closely connected with charge movement within the crystals and a build-up of charge on their surfaces. For example, the application of a large electric field to KCl(100) during indentation was found to affect the movement of edge dislocations in a way that allowed assignment of a negative charge to edge dislocations [11]. In contrast, the movement of screw dislocations was not affected by the electric field, indicating that they carry no charge. However, when alkali halides are doped with divalent cations, edge dislocations were found to be negatively charged [12]. Charge compensation of divalent impurities has also been studied by Barth and Henry. They found that kink sites on the surface produced in the cleavage process trap cation vacancies. The result is a negatively charged surface and a positive space charge of divalent cations below the surface that together forms the surface double layer [13]. Barth and Henry identified kink sites as charge traps by means of Kelvin Probe Force Microscopy (KPFM). In the present study, we use KPFM to study the topography and the charges on surfaces plastically deformed in indentation experiments, using the same tip for indentation and imaging. An extended version of this article will appear as Ref. [14].

Experimental Techniques

For all experiments, a home-built AFM with optical beam deflection method for force detection [15] was used in both contact and non-contact modes. The experiments were performed in ultra-high vacuum (UHV) at room temperature and at a pressure of < 2×10^{-10} mbar. Single crystals of KBr were cleaved along the (100) surface in air and introduced into the UHV chamber immediately. The crystals were then heated for 1 hour at 120 °C to remove charges and surface contaminants. The main impurities in the crystal were found by mass spectrometry to be 93 mg/kg of Na and 6 mg/kg of Ca. The KBr(100) surface was imaged by non-contact AFM [16] and by frequency-modulated KPFM [17]. Once a suitable area for indentation was found, the oscillation of the cantilever was stopped and the surface indented until dislocation activity was detected. Following indentation, the surface topography and charges were imaged again.

Kelvin Probe Force Microscopy of Indentations

Figure 1 shows typical topographic and KPFM maps of a surface area selected for an indentation experiment. The topography includes a defect-free atomically flat

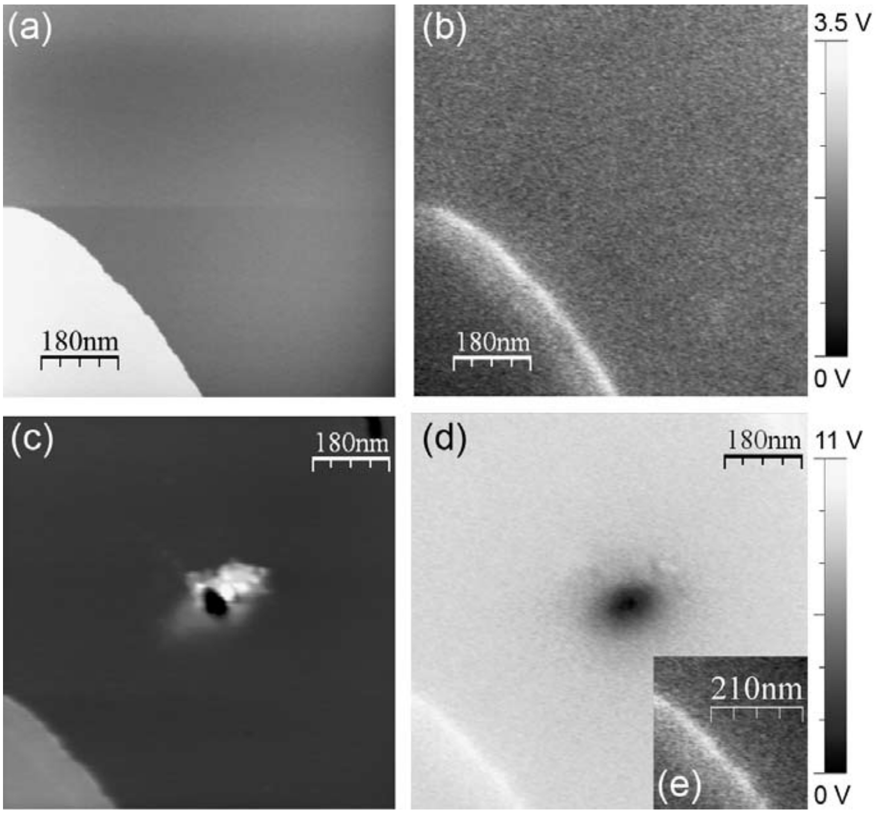


Figure 1: Topographic (a) and KPFM map (b) of an atomically flat terrace and a monatomic step on KBr(001) before indentation. Topographic (c) and KPFM map (d) after indentation of the same area. The KPFM map in inset (e) is a detail of (d) using the same colour scale as in (b), demonstrating that polarity and magnitude of the KPFM signal over the curved step edge remains the same.

terrace and a monatomic curved cleavage step. This variation in the KPFM signal over the curved step edge, which shows a positive contrast compared to the surrounding terraces, is used as a reference of the surface charge before and after indentation. The positive contrast indicates that there is a high density of negatively charged kink sites at this step edge. The surface area after indentation is shown in Figure 1 (c-e). The topography exhibits a depression at the site of indention, some elevated material around the indentation, and a string of weak features extending from the indentation toward the upper left corner of the frame. The KPFM map shows several common features observed after indentations in KBr(100). The indentation site itself shows a strong negative KPFM signal while the KPFM signal over the curved step edge next to the indent maintains the same magnitude and polarity as was observed before indentation, as demonstrated by Figure 1(e) which uses the same colour scale as Figure 1(b). This result confirms that the conducting diamond-coated tip does not change its properties with respect to the KPFM signal during the indentation. All indents studied by KPFM using different diamond tips were observed to require a negative applied bias of several volts at the site of indentation, revealing significant positive charges in the indents.

Edge Dislocations in KBr

The stress caused by the indentation has a component parallel to the surface and the KBr crystal has an active glide system parallel to the surface, namely in the {110}<110> glide system. If glide parallel to the surface is activated, then no new surface steps are created and the glide of edge dislocations, whose Burgers vectors have no component normal to the surface, is initiated. Consequently, glide planes and edge dislocations have no topographic characteristic at the surface and are difficult to detect by non-contact AFM. In this section we will show that the string of weak features observed in Figure 1(c) are edge dislocations.

Figure 2(a) gives a magnified topographic view of the features from Figure 1(c). The height of an individual hillock is 0.051 nm, clearly less that one atomic step. Figure 2(c) reveals the atomic configuration of one of the hillocks from Figure 2(a). We find two edge dislocations, one at the upper right end of the hillock, and one at the lower left.

The topographic image at higher magnification of an edge dislocation in Figure 2(d) shows how the Burgers vector of an edge dislocation was determined. Following a square of equal side length in units of distinctly visible atoms, the Burgers vector is given as the vector closing the square. For this dislocation, the Burgers vector has a magnitude of $^{\sqrt{2}a}/_{2}$ in the [1-10] direction, as expected for an edge dislocation intersecting the (100) surface. Using the same method, the two Burgers vectors can be determined for the hillock in Figure 2(c). At the end of the hillock labelled *I*, the Burgers vector points in the [1-10] direction, and at the end of the hillock labelled *II* the Burgers vector points in the [-110] direction, both having the same magnitude of $^{\sqrt{2a}}/_{2}$. The distance between the dislocations is 28 atomic protrusions, or 9.28 nm. Note the close agreement between the width of the hillocks and the tip radius estimated earlier to be 11 nm.

Figure 2(b) shows the KPFM signal recorded together with Figure 2(a). The individual hillocks are not resolved, although there is a path of positive KPFM signal along the <110> direction from the indentation site in the direction where hillocks are observed in the topography. This result is in contrast to the lack of KPFM contrast for screw dislocations and indicates that the edge dislocations carry a negative charge similar to the kink sites in step edges.

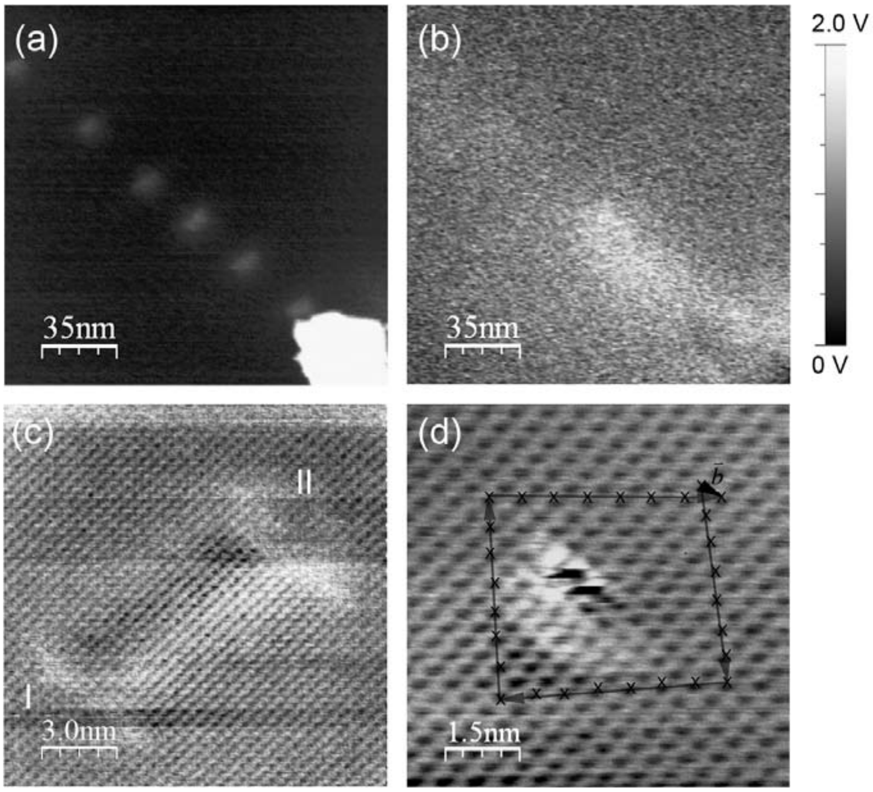


Figure 2: Higher magnification topographic (a) and KPFM map (b) of the string of shallow hillocks emerging from the surface during the indentation shown in Figure 1. Atomic resolution of an entire hillock (c) and of the edge dislocation at one end of a hillock (d). The Burgers circuit drawn on (d) illustrates the direction and magnitude of the Burgers vector for this edge dislocation.

Conclusion

In summary, we have demonstrated that Kelvin Probe Force Microscopy is a suitable tool to detect charges of dislocations at surfaces of insulating crystals. Diamond-coated tips have been used to nucleate few dislocations and analyze the resulting surface structure including atomic resolution of pairs of edge dislocations. Edge dislocations in KBr containing traces of divalent impurities carry negative charge similar to kinks in monatomic steps, while screw dislocation show no contrast in KPFM. Two mechanisms of material transfer away from the indentation site are observed: glide initiated normal to the surface followed by multiple cross-slips creates a complex structure of monatomic islands in the vicinity of the indentation and initiation of glide parallel to the surface allowing for movement of slices of material towards the <110> direction, resembling the classical rosette pattern.

This study has been carried out at McGill University, Montreal (Canada) and the INM. The work was supported by the Canada Foundation of Innovation and Natural Sciences and Engineering Research Council (NSERC). The chemical analysis of the crystals by Claudia Fink-

Straube and Andrea Jung at the INM are gratefully acknowledged.

References

[1] J. de la Figuera, M. A. González, R. García-Martínez, J. M. Rojo, O. S. Hernán, A. L. Vázquez de Parga, R. Miranda, Phys. Rev. B, 1998, 58, 1169-1172.

[2] E. Carrasco, O. Rodríguez de la Fuente, M. A. González, J. M. Rojo, Phys. Rev. B, 2003, 68, 180102.

[3] E. Carrasco, M. A. González, O. Rodríguez de la Fuente, J. M. Rojo, Surf. Sci., 2004, 572, 467-475.

[4] J. D. Kiely, J. E. Houston. Phys. Rev. B, 1998, 57, 12588-12594.

[5] P. F. M. Terán Arce, G. Andreu Riera, P. Gorostiza, F. Sanz, Appl. Phys. Lett., 2000, 77, 839-841.

[6] A. Asenjo, M. Jaafar, E. Carrasco, J. M. Rojo, Phys. Rev. B, 2006, 73, 75431.

[7] T. Filleter, S. Maier, R. Bennewitz, Phys. Rev. B, 2006, 73, 155433.

[8] T. Filleter, R. Bennewitz, Nanotechnology, 2007, 18, 044004.

[9] V. Navarro, O. Rodríguez de la Fuente, A. Mascaraque, J. M. Rojo, Phys. Rev. Lett., 2008, 100, 105504.

[10] D. Lorenz, A. Zeckzer, U. Hilpert, P. Grau, H. Johansen, H. S. Leipner, Phys. Rev. B, 2003, 67, 172101.

[11] L. Colombo, T. Kataoka, J. C. M. Li, Phil. Mag. A, 1982, 46, 211-215.

[12] M. R. Dalmau, A. Paz, J. A. Gorri, Phys. Stat. Sol. A, 1986, 96, 533-540.

[13] C. Barth, C. R. Henry, Phys. Rev. Lett., 2007, 98, 136804.

[14] P. Egberts, T. Filleter, R. Bennewitz, Nanotechnology, 2009, in print.

[15] L. Howald, E. Meyer, R. Lüthi, H. Haefke, R. Overney, H. Rudin, H.-J. Güntherodt, Appl. Phys. Lett., 1993, 63, 117-119.

[16] F. J. Giessibl, In: S. Morita, R. Wiesendanger, E. Meyer, Eds., Noncontact Atomic Force Microscopy, Springer: Berlin, 2002, 11-46.

[17] M. Nonnenmacher, M. P. O'Boyle, H. K. Wickramasinghe, Appl. Phys. Lett., 1991, 58, 2921-2923.

The scale-up of material microstructuring: a perspective

T. Kraus

The microstructure of materials is crucial for their behaviour. High-resolution microscopy, advanced spectroscopy and improved modelling have enabled researchers to analyse the microstructure's influence in detail. In addition, methods derived from semiconductor technology now allow researchers to prepare rationally structured materials. Microstructures were thus adapted from living organisms or *de novo* from physical insight in order to evoke specific behaviour in synthetic materials. Biomimetic materials exhibit the properties of their natural counterpart, for example, the adhesion of Gecko feet [1] or the photonic band gap of opals [2]. Designed materials can show previously unknown behaviour, e.g. a negative refractive index, as in artificial electromagnetic metamaterials [3].

To synthesize such "engineered materials", structural information is derived from nature or modelling and then physically realized in the microstructure of the material. This requires a synthesis process very unlike traditional approaches from metallurgy or polymer technology; those enable the engineer only to bias microstructure. Information input is limited, and the resulting structure is a complex function of the process parameters. The free parameters only implicitly define average structural features. Individual elements (for example, the grains in a piece of steel) are stochastically distributed and have a random size distribution. This is insufficient for rational microstructure design, but often sufficient for the economical production of a material with a certain property.

In contrast, microfabrication technology provides full control of structure. The technology was originally developed for the production of microelectronic circuitry, where each functional element is unique. Comprehensive information (in the form of a mask design) enters the fabrication process and is explicitly translated into structure. In the ideal case, the fabrication process is a conformal mapping of the design data on the target substrate. This is sufficient for rational microstructure design even with extreme complexity, but is usually an extreme waste for the synthesis of designed materials.

Typically, the structures in engineered materials are arranged in small unit cells that repeat to form a macroscopic material (Figure 1). Thus, replication is a first step towards the scale-up of designed materials. The expensive primary patterning is performed only once and on a limited area; large areas are structured by transferring this master to the substrate multiple times. Common transfer methods include photolithography, where light intensity is modulated by the master, and imprint or printing [4], where mechanical contact leads to deformation of the substrate by the master (Figure 2). Many functional structures are now routinely fabricated by replication, including mass products such as holograms. Replication is expected to become one of the key technologies in emerging fields like flexible electronics.

Replication limits fabrication to materials that can be photopatterned or imprinted. It does not solve the problem of three-dimensional structures, either: while two-dimensional replicas can be stacked

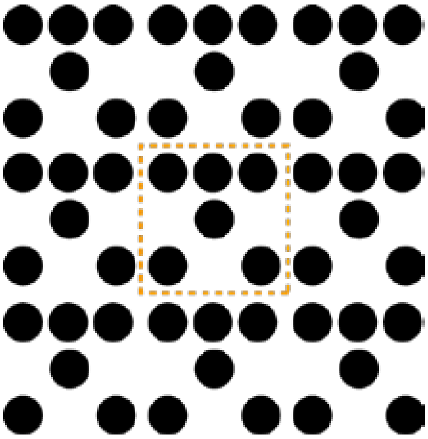


Figure 1: Many structured materials are composed of relatively small unit cells (marked in orange) that are repeated to form the bulk material.

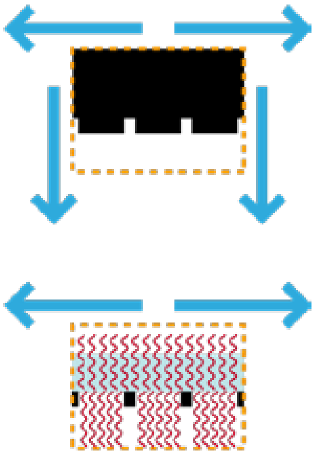


Figure 2: Replication methods, such as printing or imprinting (top) or photolithography (bottom) can be used to create large numbers or repeating unit cells.

on top of each other, this is far too slow to make macroscopic bodies and not suitable for any but the simplest three-dimensional structures. An alternative is to multiplex the patterning probe.

In a multiplexed approach, multiple parts of a material are addressed concurrently. The multiplexed probes can be the maxima of an interference pattern as in interference lithography (Figure 3) [5], or the synchronously moving tips of an Atomic Force Microscope (AFM) array (Figure 4) [6]. Interference patterns extend in three dimensions, thus enabling 3D fabrication. However, their geometry can only be modulated in a limited range. AFM arrays depend on many small tips that all have to work properly, are expensive and still rather slow. Such problems often plague multiplexed patterning. It is tempting to get rid of the probe altogether.

When parting with a central source of order, information has to enter the system on a different route. Because interaction lengths are limited, information has to be distributed throughout the ordering system. An elegant solution it to encode small building blocks so that they arrange through specific interactions. This is how viruses assemble in an infected cell: individual components are synthesized by the hijacked cell nuclei and assemble into the functional virus. In the inanimate world, supramolecular chemists synthesize molecules that interact to form complex superstructures. Similarly, colloidal particles have been found to interact with each other depending on their geometry and chemical nature, thus forming complex nanostructures (Figure 5) [7].

It is intriguing that this principle works on many different length scales. A common problem in modern materials synthesis is hierarchical patterning. In many cases, macroscopic structures have to be combined with micron-scale and nanoscale features to obtain a certain effect. Biological systems master such multiscale synthesis easily because they synthesize materials from the smallest building units under molecular control. This is hard to mimic using conventional microfabrication. Components from different length scales that contain the structural information to form hierarchical structures are an interesting alternative. They can be optimized independently and combined in a modular fashion, an approach much more compatible to engineering principles.

Structure formation due to distributed information is often called "self-assembly" or "self-organization" [8]. As a synthetic tool, self-assembly is not as versatile as conventional microfabrication, and self-assembled structures often exhibit defects and lack long-range order.

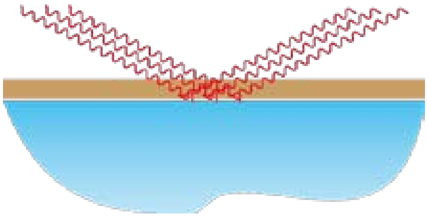


Figure 3: Interference lithography is based on overlapping beams of electromagnetic radiation that pattern a material due to the intensity modulations in the interference pattern.

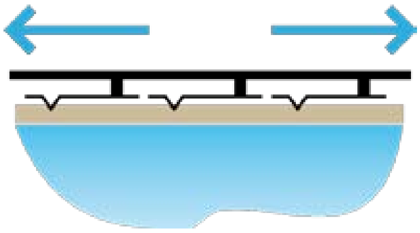


Figure 4: A multiprobe array (here composed of multiple AFM tips) writes identical structures at multiple positions in parallel.

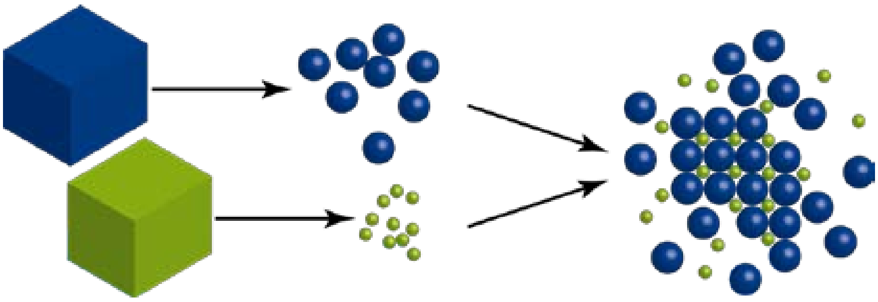


Figure 5: Certain mixtures of colloidal particles spontaneously arrange into ordered superstructures that depend on the particle geometries and interactions.

It is, however, suitable for the synthesis of designed materials. Relevant for such materials are connectivity, symmetry, and termination, and self-assembly processes can efficiently generate those. For example, binary mixtures of nanoparticles form supercrystals with good short-range order and great structural diversity. The resulting materials exhibit interesting electronic properties notwithstanding the fact that the particle agglomeration process cannot produce long-range order [9].

Information enters the particle aggregation process mainly through the geometry and interaction potential of the involved particles. With increasingly narrow size distribution and structural uniformity of the involved particles and increasingly controlled processing, increasingly predictable structures are formed. The particle-based approach is convenient because particle geometry and interaction can be analysed and modified in great detail *a priori*, particles retain their identity and can be observed during the agglomeration process, and many different base materials can be shaped into particles. On the other hand, it is challenging because the quality of the constituent particles has to be very high, many interactions influence the aggregation process and the resulting structure have large interfaces that often govern their behaviour. The approach is not new: in food technology and cosmetics for example, particle aggregation has long been exploited to adjust texture.

It is as yet unclear which processes govern the assembly of nanoparticles into ordered structures, or why certain structures form. In some cases, the reason will be simple energy minimization. The interacting particle system is ergodic and finds a minimum energy configuration in which it then settles. In many cases, however, this principle alone cannot explain the experimental results [7]. In addition, entropic effects are relevant. It is well known that the total entropy of certain, apparently ordered systems is greater than that of many disordered systems. Although the overall configurational entropy in the ordered arrangements is smaller, the entropy contribution of the individual particles can more than balance this reduction. Recent research tries to identify such arrangements using purely geometrical space-filling arguments [10]. If such arguments should hold, many different particle arrangements would be predictable and accessible simply by tuning particle size.

Not all particle ordering processes take place close to equilibrium or end in an equilibrium situation. On the contrary, the frequently used convective particle assembly process is a kinetically driven, non-equilibrium process. Hydrodynamic effects guide particles into their respective positions [11]. Fractal particle agglomeration is another classical mechanism of structure formation; only very short-range order can be generated by it, and the structures depend on particle transport rather than on particle structure. Such processes are very sensitive to process parameters like temperature and viscosity.

Process design is critical even for those structure formation processes in which an equilibrium state is reached. The motion of colloidal particles is slow compared to that of molecules, and its motion is affected by many more processes than that of molecules. Hydrodynamic instabilities, for example, can severely distort the particle order. In addition, structural information in ordering particle systems is less explicit than in conventional patterning processes, so that secondary minima or bifurcation frequently occur and give raise to unwanted structures. Thus, in contrast to classical microfabrication processes, self-assembly suffers less from inhomogeneities over large distances. Instead, typical defects involve drastic local and even global deviations from the desired structure. Suppressing such instabilities is the main challenge in nanoparticle assembly on large scales. It is indeed possible even in relatively simple and cost-effective setups [12], but it requires diligent process optimization and rigorous control of the conditions.

The development of scalable microstructuring technologies, from electron beam patterning to particle self-assembly, has the potential to make engineered materials available for mass applications. In that, it is similar to the development of chemical nanoparticle synthesis: a simple principle yields macroscopic amounts of nanostructures. Like in nanoparticle synthesis, the actual assembly process is rather complex and requires considerable know-how in scale-up. When it is understood, however, the process has the potential for relevant technological innovation.

References

[1] E. Arzt, S. Gorb, R. Spolenak, PNAS, 2003, 100, 10603-10606.

[2] A. S. Dimitrov, K. Nagayama, Langmuir, 1996, 12, 1303-1311.

[3] V. M. Shalaev, W. S. Cai, U. K. Chettiar, H. K. Yuan, A. K. Sarychev, V. P. Drachev, A. V. Kildishev, Opt. Lett., 2005, 30, 3356-3358.

[4] B. Michel, A. Bernard, A. Bietsch, E. Delamarche, M. Geissler, D. Juncker, H. Kind, J. P. Renault, H. Rothuizen, H. Schmid, P. Schmidt-Winkel, R. Stutz, H. Wolf, IBM J. Res. Dev., 2001, 45, 697-719.

[5] J. H. Moon, J. Ford, S. Yang, Polym. Advan. Technol., 2006, 17, 83-93.

[6] S. Sekula, J. Fuchs, S. Weg-Remers, P. Nagel, S. Schuppler, J. Fragala, N. Theilacker, M. Franueb, C. Wingren, P. Ellmark, C. A. K. Borrebaeck, C. A. Mirkin, H. Fuchs, S. Lenhert, Small, 2008, 4, 1785-1793.

[7] E. V. Shevchenko, D. V. Talapin, N. A. Kotov, S. O‘Brien, C. B. Murray, Nature, 2006, 439, 55-59.

[8] G. M. Whitesides, B. Grzybowski, Science, 2002, 295, 2418-2421.

[9] C. B. Murray, C. R. Kagan, M. G. Bawendi, Ann. Rev. Mat. Sci., 2000, 30, 545-610.

[10] Z. Chen, S. O’Brien, ACS Nano, 2008, 2, 1219-1229.

[11] T. Solomon, M. J. Solomon, J. Chem. Phys., 2006, 124, 134905.

[12] L. Malaquin, T. Kraus, H. Schmid, E. Delamarche, H. Wolf, Langmuir, 2007, 23, 11513–11521.

Fast and sensitive screening of transparent composite materials using polarized light image processing

I. M. Weiss and B. Heiland

Abstract

Composite materials of natural origin have remarkable material properties. In order to mimic the various functions of such materials for technical applications, it is necessary to understand the structure and the mechanisms of their formation, a scientific field which is called structural biology. A milestone in the era of structural biology was the application of the polarized light microscope for investigating biological specimens by W. J. Schmidt in 1924. A recent development in polarization technology is the LC-PolScope (Abrio IM™ Imaging System) that has been developed by R. Oldenbourg and his colleagues during the past 20 years. Like conventional polarization microscopy, it probes the local anisotropy of the specimen's optical properties, such as birefringence or dichroism. Since there is a close relationship between such properties and molecular order in bulk materials, polarization microscopy can be regarded as a submicroscopic technique. The LC-PolScope is especially suitable for investigating biological materials without the need for using contrasting agents such as fluorescent markers. We demonstrated the applicability of this technique for the fast and sensitive screening of biological composite materials. Implications for the characterization of biomineralization phenomena in a quantitative manner are discussed.

Introduction

In optically anisotropic materials, the refractive index **n** changes with the polarization of light passing through. With respect to the direction of molecular order, the refractive indices parallel and perpendicular differ by a certain value Δ**n**, which is called birefringence. One century ago, the invention of polarized light microscopy represented a milestone for the investigation of biological materials such as bone, teeth, and sea shells. It became possible to determine the orientation of inorganic crystals with respect to the body plan of biomineralizing organisms. As mentioned above one of the pioneers in the era of structural biology was W. J. Schmidt who applied the polarized light microscope to biological specimens [1]. A few years before, O. Wiener had presented a general theory of the dielectric constant of mixed systems based on expressions for the birefringence of lamellar sheets and suspensions of parallel cylinders [2]. While Schmidt continued to apply the polarized light microscope to all kinds of biological specimens in the macroscopic and microscopic regime of length scales during the following years [3-6], other scientists like Perutz [7] as well as Bragg and Pippard [8] employed Wiener's theory in order to characterize biological molecules and their interaction phenomena such as alignment and crystallization at the nanoscale. Biological macromolecules bear several analytical problems regarding the so-called form birefringence, a phenomenon that applies to objects substantially smaller than the wavelength of light. Thus, the controversies regarding intrinsic and form birefringence continued [9, 10]. The comeback of Wiener's theory happened in 1975, when Inoué and coworkers demonstrated a direct relationship between the birefringence of the mitotic spindle and the alignment of tubulin proteins [11]. It was suggested that Wiener's theory is in good agreement with experimental observations of form-birefringence that were made with DNA and Tobacco Mosaic Virus assemblies. It turned out to be crucial to take solvent effects into account [12]. The past 20 years brought further development of polarized light microscopy by using liquid crystal compensators. Combined with efficient image processing techniques it is now possible to quantify birefringence at different levels of sensitivity [13-15]. The analogies of conventional polarized light microscopy and the LC-PolScope (Abrio IM™ Imaging System, CRi, Woburn, MA, U.S.A.) are summarized in Figure 1. With respect to biological materials it is especially interesting to note that the orientations of extracellular fibrils such as cellulose in wood or plant cell walls correlate with the alignment of certain fibrillar proteins such as microtubules inside of cells [16]. Intracellular cytoskeletal structures are usually visualized by immuno-fluorescence staining techniques with the drawback of fixation that is not compatible with recording dynamic rearrangements *in vivo*. Here we demonstrate the applicability of an imaging platform for the fast and sensitive screening of mineralized biological materials based on an inverted microscope equipped with an Abrio IM™ Imaging System that will allow us to study the synthesis of extracellular matrix polymers and their crystallization behaviour as a function of intracellular cytoskeletal fibril assembly. Based on this fast and sensitive screening method, the relationship between particular calcium oxalate crystals and plant cells turned out very efficient in order to demonstrate the suitability of this instrumentation for screening composite materials of biological origin with respect to quantification of birefringence and the orientation of crystals.

Quantification of birefringence

In principle, the LC-PolScope measures the same properties which can be measured by a traditional polarized light microscope, but with limited speed, sensitivity, and accuracy. The LC-PolScope achieves significant improvements by measuring the birefringence parameters for all resolved specimen points simul-

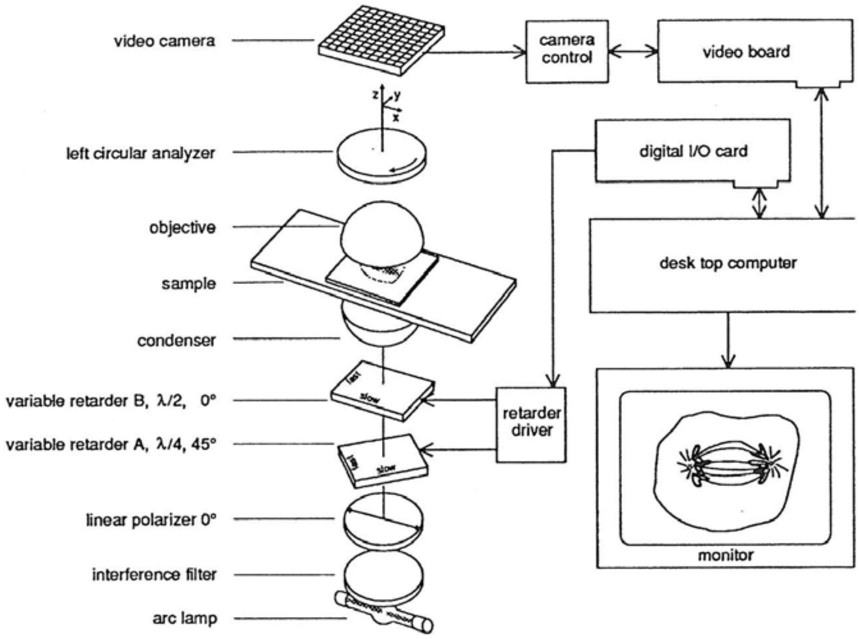


Figure 1: Principles of PolScope imaging technology as described in ref. [17]. The details of relevant imaging parameters are reviewed in ref. [14].

taneously. The heart of the instrument is an extremely sensitive high resolution ccd camera, in addition to an universal compensator which is built from two variable electro-optical retarder plates and a linear polarizer [17]. Depending on whether monochromatic light passes first through the retarder plates or first through the linear polarizer, this device functions as a "variable polarizer" or "polarization analyser", respectively. Any polarization state can be achieved: Linear polarization of any azimuth, or elliptical polarization of any handedness, ellipticity, or azimuth. The voltage for each retarder plate is specifically controlled by a software that synchronizes the image acquisition process and computes values for retardance R = Δn · *t* (relative optical path difference, or phase change, suffered by two orthogonal polarization components of light when interacting with an optically anisotropic material of a given thickness *t*) and principal axis orientation (e.g. slow axis azimuth corresponds to highest refractive index) at each resolved image point. A total of four images, which are in perfect register, are recorded with selected voltages applied to the liquid-crystal devices and processed. Since there is no mechanical change in orientation of the specimen required, inaccuracies due to a potential lack of reproducibility in the manipulation of the microsocope are eliminated. In such a way, high spatial resolution can be achieved. Since LC-PolScope image acquisition is based on circular polarized light, a monochromatic filter (e.g. 546 ±15 nm) is required. Although calibration and correction procedures are employed,

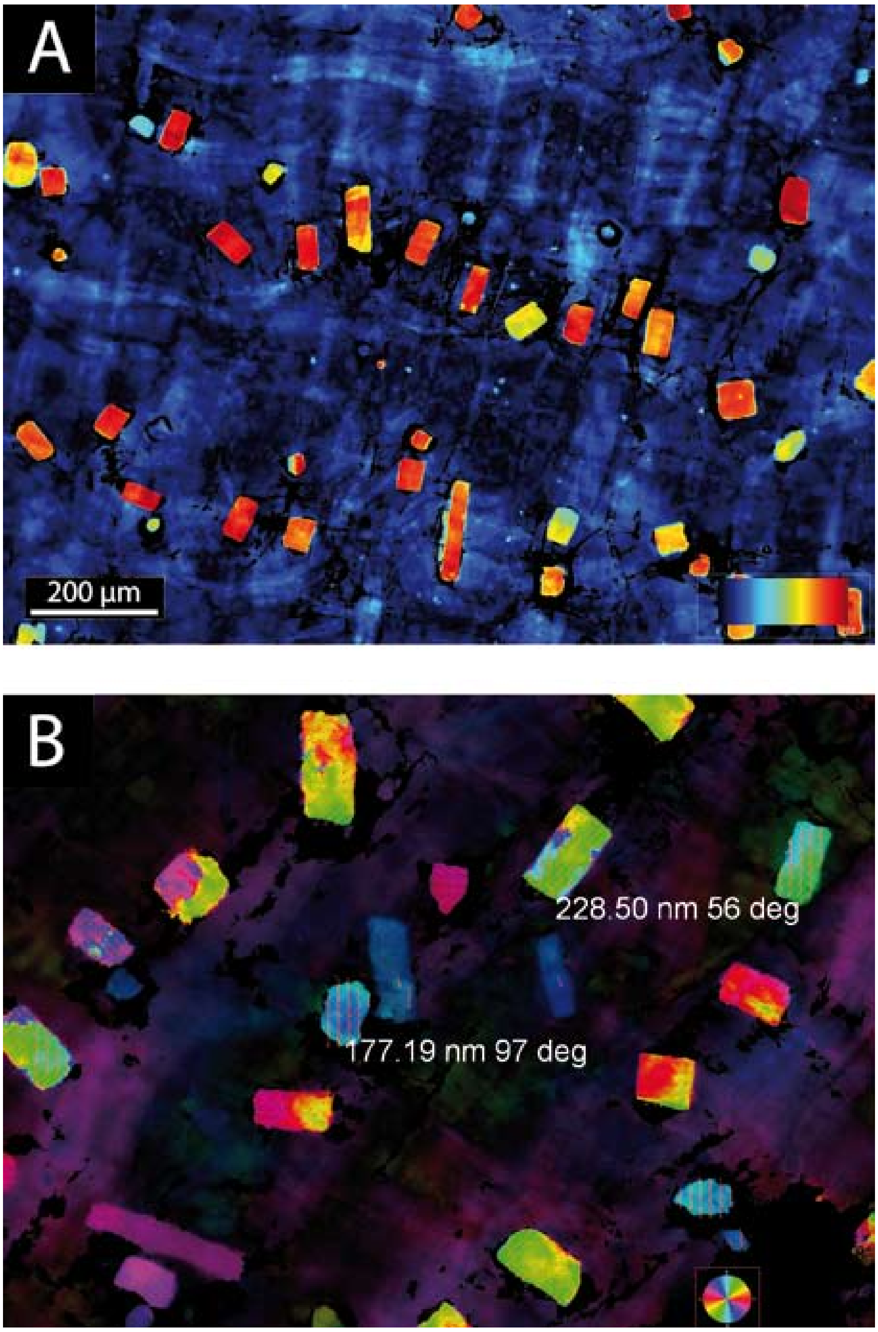


Figure 2: LC-PolScope images taken from *Allium spec.* in the retardance (A) and vector overlay mode (B). The range of retardance values in (A) is calculated from 0 nm - 272 nm (blue → red). The retardance in nm and azimuth of the slow axis in deg in (B) are calculated per pixel and colour encoded in the image. Depending on selected threshold values for the retardance, the azimuth vectors are displayed on regular grid points.

it is still crucial for measuring small retardance values in the range below 0.01 nm that extinction is optimized by the right choice of optics components as described [14]. The speed of image acquisition is to a large extent depending on liquid crystal settling time (e.g. 30 ms), among other parameters.

Investigation of birefringence in biological materials

The suitability of the LC-PolScope for investigating biological materials in an inverted microscope was tested using tissue samples from the onion (*Allium spec.*). It turned out that a large number of irregular cube-shaped objects of about 50 µm in length with high retardance values as compared to the surrounding tissue were observed in the outer epidermis of *Allium spec.*. As shown in Figure 2, the retardance in nm and azimuth in deg per pixel is quantitatively colour encoded in the image. The surrounding tissue gives significantly lower retardance values corresponding to a reduced brightness than obtained from the cube-shaped objects, which correspond well to calcium oxalate crystals that have been described in the literature to occur in *Allium spec.* [18-20]. As shown in Figure 2, it is possible to obtain information regarding the orientation of particular single crystals by vector overlay. As opposed to conventional polarized light microscopy, it was possible to visualize a whole population of crystals within the same image irrespective of their orientation. Furthermore, irregularities were observed within the crystals that would be difficult to detect by using conventional crossed polarizers. It remains, however, to be investigated whether the irregularities are due to crystal defects of the calcium oxalate, or due to any kind of form birefringence caused by surface roughness or liquid inclusions. In 2004, R. Oldenbourg wrote: “The art and science of relating measured retardance and azimuth values to structural information on the molecular level of the specimen is still in its infancy. The potential information in PolScope images and time-lapse movies is enormous” [14]. Our current investigation of *Allium spec.* demonstrates that Wiener’s theory [2] will have to be consulted more extensively in order to see beyond the iceberg’s tip of complex biological materials.

Outlook

We plan to transfer some concepts of mollusc shell formation [21-25] into suitable cellular model systems that allow us to study the formation of composite materials *in vivo* in a comparative manner. The main focus will be the biosynthesis of extracellular matrix polymers and the subsequent steps that are important to create mineralized composite materials. Since this is by no means going to be an easy task, it will be extremely important to apply a fast and sensitive screening technique. It is part of the strategy to obtain high-throughput information on the degree of mineralization and possibly the orientation of crystals with respect to intra- and extracellular polymer structures. Combined with standardized metallographic specimen preparation techniques [26-28], the LC-PolScope will thus be extremely useful for designing biological materials with respect to various applications.

References

[1] W. J. Schmidt, Die Bausteine des Tierkörpers in Polarisiertem Lichte, Cohen: Bonn, 1924.

[2] O. Wiener, Abh. Math. Phys. Kl. Koenigl. Saechs. Ges. Wiss., 1912, 32, 509-604.

[3] W. J. Schmidt, Z. Zellforsch. Mikrosk. Anat., 1936, 25, 181-203.

[4] W. J. Schmidt, Z. Zellforsch. Mikrosk. Anat., 1958, 47, 557-559.

[5] W. J. Schmidt, Chromosoma, 1939, 1, 253-264.

[6] M. M. Swann, J. M. Mitchison, J. Exp. Biol., 1950, 27, 226-237.

[7] M. F. Perutz, Acta Crystallogr., 1953, 6, 859-864.

[8] W. L. Bragg, A. B. Pippard, Acta Crystallogr., 1953, 6, 865-867.

[9] J. Y. Cassim, E. W. Taylor, Biophys. J., 1965, 5, 531-552.

[10] J. Y. Cassim, P. S. Tobias, E. W. Taylor, BBA - Protein Structure, 1968, 168, 463-471.

[11] H. Sato, G. W. Ellis, S. Inoue, J. Cell Biol., 1975, 67, 501-517.

[12] R. Oldenbourg, T. Ruiz, Biophys. J., 1989, 56, 195-205.

[13] K. Katoh, K. Hammar, P. J. S. Smith, R. Oldenbourg, Mol. Biol. Cell, 1999, 10, 197-210.

[14] R. Oldenbourg, In: R. D. Goldman, D. L. Spector, Eds., Live Cell Imaging: A Laboratory Manual, Cold Spring Harbor Laboratory Press: New York, 2004, 205-237.

[15] R. Oldenbourg, E. D. Salmon, P. T. Tran, Biophys. J., 1998, 74, 645-654.

[16] M. C. Ledbetter, K. R. Porter, J. Cell Biol., 1963, 19, 239-250.

[17] R. Oldenbourg, G. Mei, J. Microsc., 1995, 180, 140-147.

[18] I. Ricci, Ann. Bot. (Rome), 1963, 27, 431-450.

[19] C. J. Prychid, P. J. Rudall, Ann. Bot., 1999, 84, 725-739.

[20] V. R. Franceschi, P. A. Nakata, Annu. Rev. Plant Biol., 2005, 56, 41-71.

[21] V. Schönitzer, I. M. Weiss, BMC Struct. Biol., 2007, 7, 71.

[22] I. Weiss, N. Tuross, L. Addadi, S. Weiner, J. Exp. Zool., 2002, 293, 478-491.

[23] I. M. Weiss, F. Marin, In: A. Sigel, H. Sigel, R. K. O. Sigel, Eds., Biomineralization: From Nature to Application, John Wiley & Sons: West Sussex, 2008, 71-126.

[24] I. M. Weiss, V. Schönitzer, J. Struct. Biol., 2006, 153, 264-277.

[25] I. M. Weiss, V. Schönitzer, N. Eichner, M. Sumper, FEBS Lett., 2006, 580, 1846-1852.

[26] H. Schumann, Metallographie, Deutscher Verlag für Grundstoffindustrie: Stuttgart, 1990.

[27] G. Petzow, Metallographisches, keramographisches und plastographisches Ätzen, Borntraeger: Berlin [u. a.], 2006.

[28] B. Heiland, E. Arzt, I. M. Weiss, unpublished.

Group Reports
Gruppenberichte

Biomineralization / Biomineralisation (since 01/2008)

PD Dr. Ingrid Weiss

Den Forschungsschwerpunkt des Programmbereichs Biomineralisation, der im Januar 2008 eingerichtet wurde, bildet die Suche nach biologischen Synthesewegen für neue Materialien. Der Hauptvorteil derartiger Strategien ist sicherlich energetischer Natur: Biologische Materialien werden unter physiologischen Temperaturen synthetisiert. Ebenso lassen sich ambitioniertere Ziele der Materialwissenschaften wie beispielsweise Selbstheilungseffekte implementieren: Defekte werden bereits während der Synthese biologischen Materials erkannt und können *in situ* eliminiert werden, indem die erforderlichen Reparatur-Enzyme und Strukturbausteine nach Bedarf rekrutiert werden. Materialien biologischen Ursprungs sind zudem umweltfreundlich, da sie in bereits bestehende ökologische Kreisläufe für Um- und Abbau in der Natur integriert sind und sich dadurch Risiken der langjährigen Akkumulation in der Umwelt minimieren lassen. Biosynthesewege basieren oftmals auf einfachsten Ausgangsstoffen, die in der Natur praktisch unbegrenzt zur Verfügung stehen, können aber in komplexe und funktionell optimierte hierarchische Strukturen münden. Die Architektur biologischer Materialien vereint oftmals unterschiedliche Designprinzipien wie beispielsweise Stabilität unter Beibehaltung selektiver Permeabilität für Ionen, Moleküle, Photonen usw., wobei die hierfür erforderlichen Strukturen unter minimalem energetischen Aufwand und möglichst geringem Materialeinsatz aufgebaut werden. Die Biologie bedient sich ihrer eigenen Form von „Nano-Technologie” und implementiert sozusagen „automatisch” entsprechende Skalengesetze, um den hierarchischen Aufbau ihrer Materialien über mehrere Längenskalen kontrolliert und funktionsoptimiert zu bewerkstelligen. Anpassungen an eine Vielzahl von ökologischen Nischen über Jahrmillionen bietet uns die Chance, entsprechende biologische Materialsysteme, die möglicherweise bereits viele der für eine erfolgreiche Anwendung erforderlichen Designkriterien erfüllen, zu finden und weiterzuentwickeln.

Wir arbeiten an der Etablierung eines erweiterten Methodenspektrums für die schnelle, empfindliche und effiziente Analyse von Kompositmaterialien biologischen Ursprungs. Die bildverarbeitungsgestützte Quantifizierung der Lichtdoppelbrechung erlaubt die nichtinvasive Untersuchung dynamischer Prozesse in Zellen an der Grenzfläche zur selbst-organisierenden bis hin zu mineralisierenden Extrazellular-Matrix, die den Ausgangspunkt einer Vielzahl biologischer Materialien bildet.

Den zweiten Schwerpunkt bildet die Analyse der Biosynthese von Perlmutt, einem biologischen Hightechmaterial aufgrund seiner enormen Bruchfestigkeit bei extrem geringer Dichte. Hier spielt die Synthese von Chitin eine wichtige Rolle, um indirekt die Mineralisierung von Calciumcarbonat mit einem hohen Organisationsgrad zu bewerkstelligen. Ausgewählte Prinzipien wie Mechanotransduktion werden auf der Basis von Einzelmolekülstudien des molekularen Myosin-Motors der Mollusken-Chitinsynthase untersucht.

Zukünftig sollen diese neuartigen Konzepte der Perlmuttbildung in geeignete zelluläre Modellsysteme übertragen werden, die zum einen dem besseren Verständnis der Synthese mineralisierter biologischer Materialien dienen und zum anderen direkt für die maßgeschneiderte Synthese biologischer Materialien für verschiedene Anforderungsbereiche genutzt werden können. In diesem Kontext erarbeiten wir derzeit die Grundlagen für neue patentierfähige Konzepte am INM.

Towards new biological materials

The general aim of our research is to exploit biological routes towards new materials for a number of reasons, the most obvious being perhaps energetics, towards material synthesis at ambient conditions. Even ambitious goals in materials synthesis such as self-healing can immediately be targeted: Defects that occur during the synthesis route are immediately recognized and eliminated *in situ* by recruiting the appropriate enzymatic machinery. Materials from biological origin are environmentally friendly, since they are integrated into ecological cycles for degradation that already exist in nature, thus minimizing risks of long-term accumulation on earth. Biosynthetic routes usually start from simple compounds that are abundantly available in nature, but eventually result in sophisticated and efficient hierarchical systems such as the photosynthetic chain for light harvesting. The architecture of biological materials often serves multi-purpose demands, such as coordinating stability and selective permeability for ions, molecules, photons etc., while at the same time minimizing energy and resources requirements for building such a functional structure. Biological materials have already implemented scaling laws as in "nanotechnology", in addition to the control of hierarchical assembly over a range of multiple length scales. Their adaptation to a tremendous variety of ecological niches over millions of years allows us to search for a model organism that is already well-adapted to a given application in terms of materials design.

Establishing new methods that allow fast and sensitive screening of biocomposite materials

One century ago, the invention of polarized light microscopy represented a milestone in the investigation of biological materials such as bone, teeth, and sea shells. It became possible to determine the orientation of inorganic crystals with respect to the body plan of biomineralizing organisms. The past 20 years brought further development by using liquid crystal compensators. Combined with efficient image processing techniques, it is now possible to quantify birefringence at different levels of sensitivity. With respect to biological materials it is especially interesting to note that orientations of extracellular fibrils such as cellulose in plant cell walls correlate with the alignment of certain fibrillar proteins such as microtubules inside cells. Intracellular cytoskeletal structures are usually visualized by immuno-fluorescence staining techniques with the drawback that fixation is not compatible with recording dynamic rearrangements in vivo. The use of so-called "living colors", such as expression of fluorescent fusion proteins, is suitable only for established transgenic model organisms. We created an imaging platform for living cells based on an inverted microscope equipped with an Abrio image analysis system (CRi LC-PolScope) that will allow us to study the synthesis of extracellular matrix polymers and their crystallization behaviour as a function of intracellular cytoskeletal fibril assembly. Based on this fast and sensitive screening

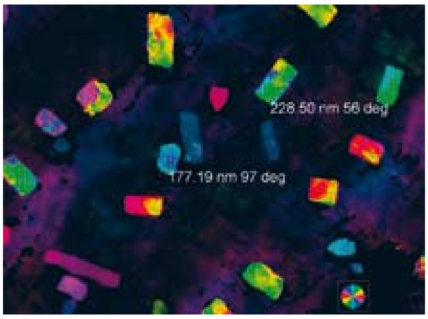


Figure 1: Birefringence analysis of native calcium oxalate crystals in plant tissue using the CRi Abrio Imaging System "LC-PolScope".

method, the relationship between particular calcium oxalate crystals and plant cells has demonstrated the suitability of this instrumentation for screening composite materials of biological origin in an efficient way(see Figure 1).

Nacre formation: Biosynthetic routes towards sophisticated biological materials

The properties of certain biological materials indeed match with industrially relevant man-made materials. The nacreous part of mollusc shells represents a light-weight material comparable in some way to metal alloys, in terms of fracture toughness, for example. We recently found that the biological fabrication process of nacre involves a complex transmembrane enzyme that synthesizes chitin, a biopolymer which is widespread in nature, but difficult to investigate due to its strong tendency to crystallize and to associate with proteins, thereby forming insoluble aggregates in an irreversible manner. In the case of nacre it is not yet understood how the chitin framework is biologically designed and controlled in order to induce the mineralization of about 500 nm thick calcium carbonate tablets in highly ordered stacks. Based on our results we propose a concerted interaction between the intracellular actin cytoskeleton and extracellular chitin fibrils. This would lead to a new level of understanding of the formation of nacre from the viewpoint of mechanotransduction. The myosin domain of the mollusc chitin synthase, a molecular motor that converts chemical energy into mechanical force, has been heterologously expressed and is currently characterized on the functional level (see Figure 2).

Outlook

We plan to transfer some concepts of nacre formation into suitable cellular model systems that allow us to study the formation of composite materials in vivo in a comparative manner. The main focus will be the biosynthesis of extracellular matrix polymers and the subsequent steps that are important to create mineralized composite materials. Once the mechanisms will be better understood in terms of variation of components with time, surface interaction potentials, adhesion phenomena, and phase separation, we can tackle the design of biological materials for various applications. In this context we are currently establishing the knowledge base for new INM patents.

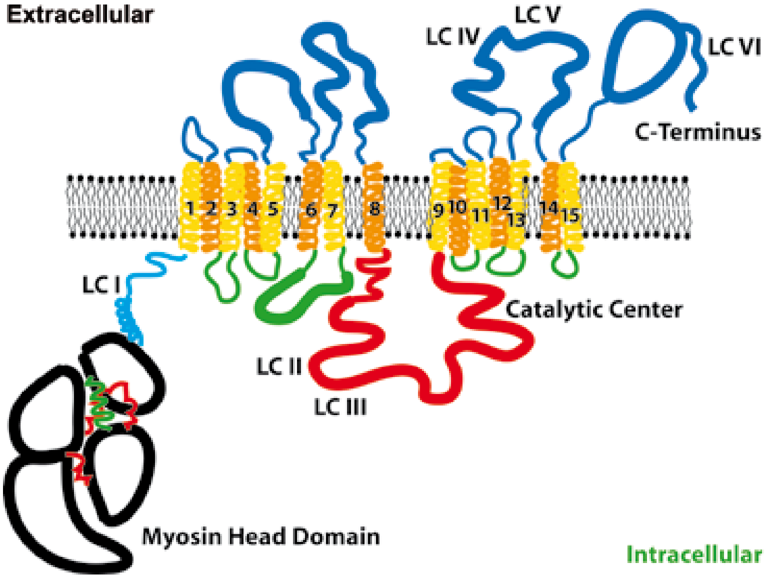


Figure 2: Model of the molecular Myosin motor that coordinates the transmembrane synthesis of a chitin matrix which is the precursor of highly ordered biominerals such as nacre.

CVD/PVD Technologies / CVD/PVD-Technologien

Prof. Dr. Dr. h. c. Michael Veith, Cenk Aktas, Prof. Dr. Sanjay Mathur

Die Forschungs- und Entwicklungsaktivitäten des Programmbereichs CVD/PVD-Technologien konzentrieren sich grundsätzlich auf die Deposition verschiedener kristalliner und amorpher Beschichtungen, die in vielen technologischen Anwendungen, wie zum Beispiel Mikroelektronik, Optik und Schutzbeschichtungen verschiedener Oberflächen, Verwendung finden. Die Forschungsgruppe arbeitet an der Entwicklung neuer molekularer Präkursoren und Depositionstechniken durch bereits etablierte Methoden einschließlich „thermal assisted CVD“, „plasma enhanced CVD“ (PECVD) und „liquid injection CVD“. Spezielle Kenntnisse wurden mit dem „single source precursor“-Konzept erworben, das eine der besonderen Methoden unserer Gruppe ist [1]. Im Jahr 2008 wurden zusätzlich die Techniken der „laser assisted CVD“ (LCVD) und „selective CVD“ (SCVD) entwickelt, um nanostrukturierte Oberflächen zu synthetisieren.

Im Vergleich zu 2007 ist es uns 2008 gelungen, völlig neue Forschungsthemen und Kollaborationen zu etablieren. Besonders im Bereich Implantations- und Biomaterialanwendungen wurde die Zusammenarbeit mit unterschiedlichen Institutionen aufgenommen. Durch diese ergiebigen Kollaborationen galt das Hauptinteresse der Erforschung der Interaktionen verschiedenster Zelltypen mit durch CVD synthetisierten nano- und mikrostrukturierten Oberflächen, um die Biokompatibilität von Implantationsmaterialien durch Oberflächenmodifikation zu optimieren.

Zusätzlich zu den Anwendungen von Biomaterialien beschäftigten wir uns mit der Möglichkeit der Energiespeicherung mit Hilfe nanoporöser Oberflächen. Im Rahmen einer Dissertation wurde (unterstützt durch den DAAD) die Adaption von nanoporösen und extrem-chaotischen eindimensionalen Strukturen zur Wasserstoffspeicherung getestet. Ein weiteres schönes Beispiel unserer Forschungsarbeiten ist die Entwicklung von „PZT thin films“ in Zusammenarbeit mit dem IBMT in St. Ingbert. Diese sollen Verwendung finden für akustische Sensoren. Die Entwicklung von dekorativen Beschichtungen mit Hilfe von CVD, eine umweltfreundliche Alternative zu aniodischer Oxidation, ist ein weiteres Thema unserer Gruppe.

The research and development activities in the program division CVD/PVD Technologies are basically focused on depositing large variety of crystalline and amorphous thin films which are extensively used in various technological applications such as microelectronics, optics, as well as for hard and protective coatings. The research group focused on the development of new molecular precursors and deposition of these precursors through previously well established methods including thermal assisted CVD, plasma enhanced CVD (PECVD) and liquid injection CVD (LICVD). Special knowledge has been assembled in "single source precursor" concept, which is one of the outstanding methods of this group [1]. In 2008, additionally laser assisted CVD (LCVD) and selective CVD (SCVD) methods were developed to synthesize nanostructured surfaces. Compared to 2007, 2008 was successful for initiating totally new research themes and developing strong collaborations. Especially in the field of implant and biomaterial applications, collaborations with different institutions were started. Through these fruitful collaborations, the main interest was given to explore the interactions of various cell types with nano/micro structured surfaces prepared by CVD and improve the biocompatibility of implants by surface modifications. In addition to biomedical applications, nanoporous surfaces, which were prepared, may be used for energy resource. Within a PhD thesis (supported by DAAD) the adaption of nanoporous and extremely chaotic 1D structures for hydrogen storage is tested.

Another example is the development of PZT thin films which will be used for an acoustic-sensor in collaboration with IBMT (St. Ingbert). The development of decorative coatings by a CVD method which is environmentally friendly and an alternative to anodic oxidation is also another interest of our group.

Synthesis of 1D nanoscale heterostructures

After the discovery of carbon nanotubes (CNTs) by Iijma [2] one-dimensional (1D) nanostructures attracted a considerable research interest due to their unique properties and potentials for nanotechnological applications. Today 1D nanostructures such as nanowires and nanotubes are already accepted as new kind of functional and smart materials for electronics, sensors, photonics, electromechanical and biotechnology applications. Especially 1D hetero-, core-shell and hierarchical nanostructres are preferred due

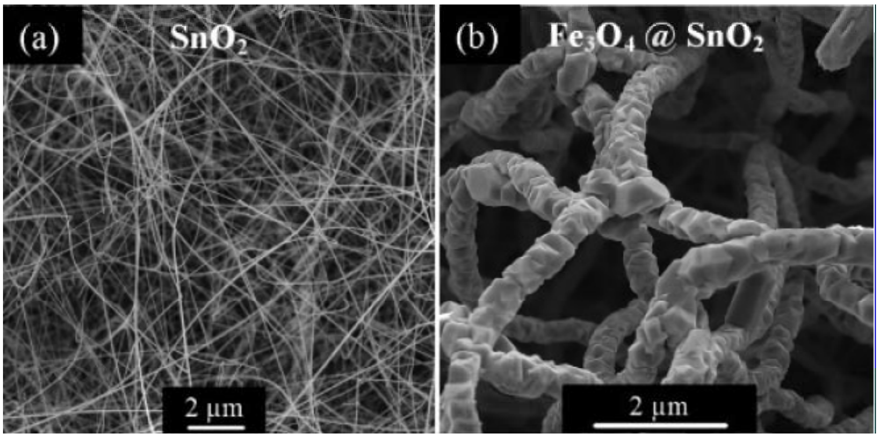


Figure 1: SEM images of (a) SnO_2 nanowires synthesized by deposition of $[Sn(OBu^t)_4]$ (b) SnO_2/Fe_3O_4 core-shell nanowires prepared by deposition of $[Fe(OBu^t)_3]_2$ on SnO_2 nanowires.

to their multifunctionality and tuneable material properties. In this context in 2008, the main interest was given to the development of new synthesis routes to fabricate 1D heterostructures. Basically two different routes were followed to synthesize 1D core-shell nanostructures:

Two-step deposition of 1D core-shell structures

In previous years, we have synthesized SnO_2 nanowires (Figure 1a) by chemical vapour deposition of $[Sn(OBu^t)_4]$ in the temperature range from 650 to 700 °C [3]. Such nanostructures were shown to be used as CO and ethanol sensors successfully. Recently we have used such SnO_2 nanowires as substrates to synthesize multifunctional 1D heterostructures. We have synthesized SnO_2/Fe_3O_4 core-shell structures (Figure 1b) by a two-step growth process, where initially grown tin oxide nanowires (50 nm) were covered by a dense assembly of magnetite nanocrystals forming a polycrystalline layer, which was confirmed by X-ray diffraction analysis. The results presented here demonstrate the strength of the molecule-based chemical vapour growth in producing simple and complex nanoarchitectures with precisely controlled composition and phase configurations.

Direct deposition 1D core-shell structures

Different from the two-step deposition approach, we have shown that core-shell 1D hetero structures can be synthesized via direct deposition of a single source precursor. Al/Al_2O_3 core/shell nanostructures were synthesized by decomposing tert-butoxialane ($^tBuOAlH_2$) at high temperatures (Figure 2a and b). The combination of metal core and dielectric shell make such nanowires multifunctional. We observed that such composite nanowires exhibit a broad plasmon absorption which can be attributed to the crossing geometry of the nanowires. By altering the deposition temperature and flow rate, the diameter and density of the nanowires can be controlled easily. This gives us a chance to modify the surface properties such as wetting. In a joint project with the program division Functional Surfaces (supported by Volkswagen Foundation) we explore the wetting and adhesion properties of the deposited Al/Al_2O_3 core/shell nanowires. We have shown a strong mechanical anchoring between nanowires and polymers. In order to deposit Al/Al_2O_3 core/shell nanowires on larger surfaces, a BMBF project is going on in collaboration with the program division Glass and Optics to develop a CVD system equipped with a continuous sheet feed system.

In addition to synthesis of Al/Al_2O_3 nanowires by thermal CVD, LCVD and SCVD methods were employed to synthesize hierarchical 1D Al_2O_3 nanostructures. Laser assisted CVD yielded synthesis of Al_2O_3 fibrous layers (Figure 3a). Applying selective CVD brought out new kind of Al_2O_3 structures which can be termed as *nanoloops* (Figure 3b). The growth mechanism of such struc-

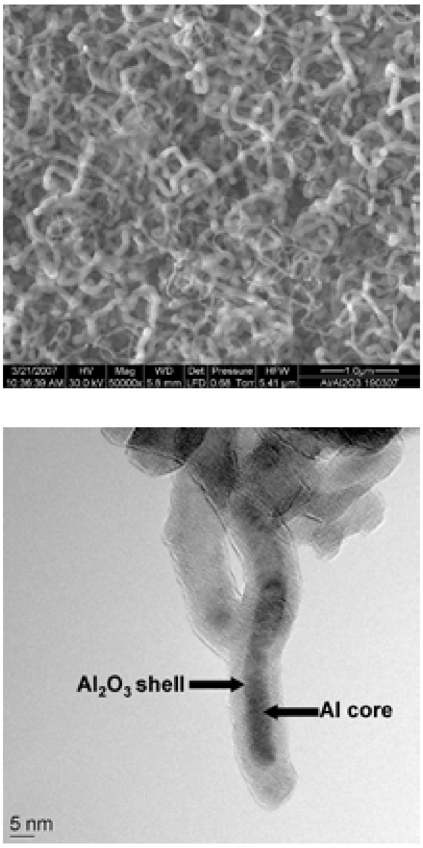


Figure 2: (a) SEM images of chaotic Al/Al_2O_3 nanowires (b) TEM image of a single nanowire showing an Al core and Al_2O_3 shell.

tures seems to be different compared to the well known vapour-liquid-solid (VLS) and other catalyst based 1D growth methods.

Micro- and nanostructure surfaces for bio-applications

The development of surfaces which improve the cell-substrate adhesion is of great interest in biomedical applications. Recently, alumina nanofibers were accepted as promising materials for orthopaedic applications. It is believed that such one dimensional (1D) nanomaterials, especially the nanowires and/or nanotubes with controlled morphology and chemical composition, interact with specific biological entities. In this regard, different cell types (fibroblasts, neurons, and different blood cells) have been tested to explore the interaction of biological entities with deposited 1D aluminium-aluminium oxide (Al/Al_2O_3) nanostructures. It is found that specifically some cell types adhere selectively on deposited Al/Al_2O_3 nanowires (Figure 4a and b). Depending on the distribution and degree of the interpenetration of the nanowires one can control the cell compatibility and adhesion. The cell compatibility shows a parallelism with the change of the contact angle due to various surface topographies at different deposition temperatures.

In addition to the direct deposition of structured surfaces, the post-treatment of deposited Al/Al_2O_3 nanowires by laser induced heat treatment leads to the formation of hierarchically structured alumina surfaces. The combination of micro- and nano-structures is interesting from the point of mimicking the structure of the extra cellular matrix (ECM) which also contains different scaled features. It is clearly seen that cells behave different on micro- and nano-scaled features which co-exist on the same test surface (Figure 5a and b). In case of the microstructures, the interspacing between the individual structures plays also an important role in addition to the size effect.

Nanoporous layers for hydrogen storage

The texture of Al/Al_2O_3 nanowires is very porous and this porosity is of greater interest for the use as hydrogen storage material. Hydrogen storage is suspect to many mechanisms but the most important and of wider interests are the physical adsorption and chemical reaction mechanisms. Al/Al_2O_3 nanowires material has potential for both types of mechanisms. We started investigating physical adsorption of hydrogen by Al/Al_2O_3 nanowires and the results are very encouraging.

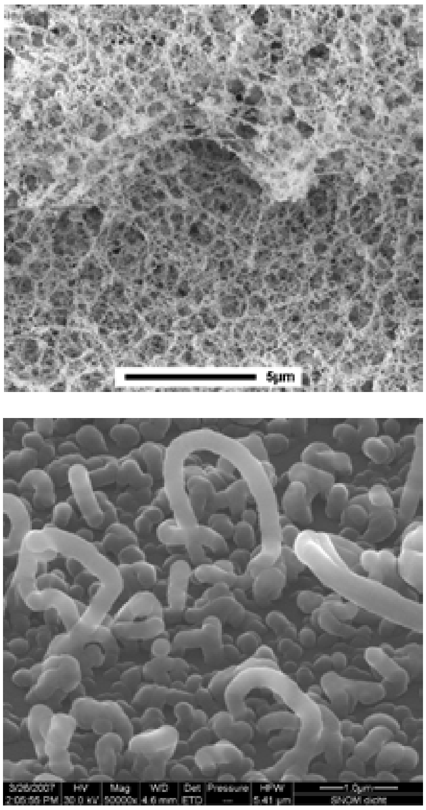


Figure 3: SEM images of (a) 1D highly chaotic Al_2O_3 nanostructres deposited by LCVD (b) Al_2O_3 nanoloops deposited by SCVD.

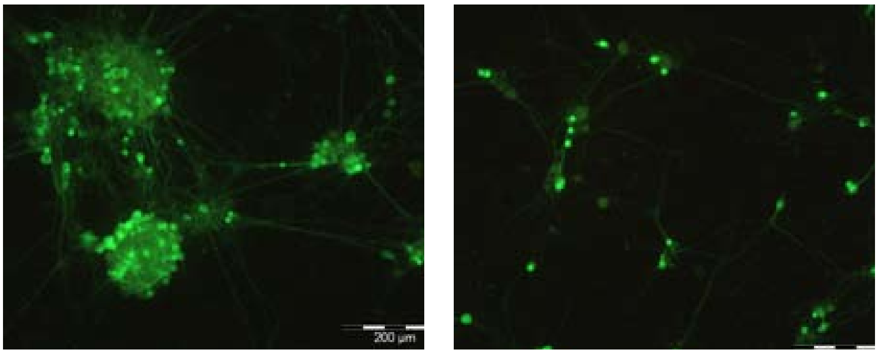


Figure 4: Fluorescence microscopy images of (a) few neuron cells on bare glass control substrates, (b) clusters of neuron cells on Al/Al_2O_3 deposited glass substrates.

PZT coatings

Epitaxial lead zirconate titanate (PZT)-coatings in the sub-µm-range were produced at temperatures below 400 °C using a novel single-source PZT-precursor in a classical thermal CVD-process. The design of two bimetallic alkoxide compounds, a lead titanate and a lead zirconate source with almost identical properties and complementary miscibility resulted in a new single source PZT-precursor, an azeotropic compound, that evaporates at 30 °C at a pressure of 4 x 10^{-1} mbar. The metal-composition of the precursor (Pb:Zr:Ti = 2:1:1) leads to transparent highly (111)-oriented PZT coatings (Figure 6) with a stoichiometry near the morphotropic phase boundary. An additional lead-source is not required. Such high quality PZT thin films are being tested for acoustic sensor development in a collaboration with Fraunhofer Institute for Biomedical Engineering (IBMT).

References

[1] M. Veith, J. Chem. Soc. Dalton, 2002, 2405-2412.

[2] S. Iijima, Nature, 1991, 354, 56-58.

[3] F. H. Ramírez, A. Tarancón, O. Casals, J. Rodríguez, A. Romano-Rodríguez, J. R. Morante, S. Barth, S. Mathur, T. Y. Choi, D. Poulikakos, V. Callegari, P. M. Nellen, Nanotechnology, 2006, 17, 5577-5583.

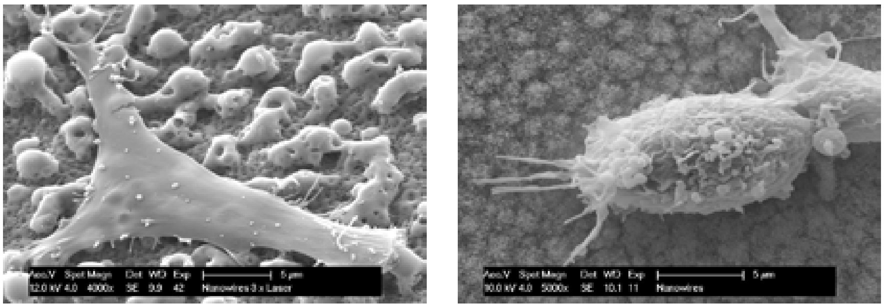


Figure 5: (a) Fibroblasts form only a few filopodia on Al/Al_2O_3 nanowires (b) Fibroblasts adhere well on laser treated Al/Al_2O_3.

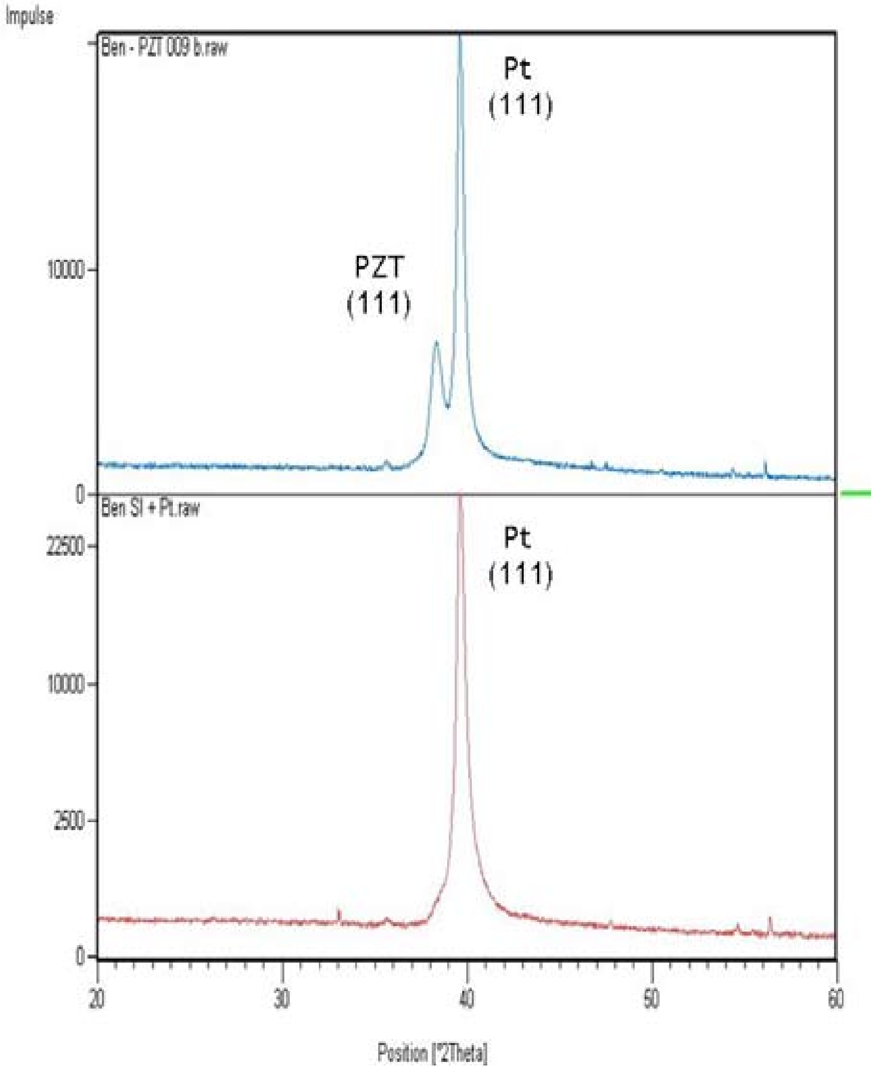


Figure 6: XRD of deposited PZT films showing (111) orientation (blue: PZT film, red: substrate).

Functional Surfaces / Funktionelle Oberflächen

Dr. Aránzazu del Campo

Der Programmbereich Funktionelle Oberflächen beschäftigt sich vorwiegend mit der Erforschung, Herstellung und Charakterisierung von Materialoberflächen und -schichten, deren Strukturierung im Mikro- und Nanobereich besondere mechanische Funktionen erzeugt. Ein Schwerpunkt liegt auf bioinspirierten Oberflächen mit adhäsiven Eigenschaften. Die Untersuchung solcher Oberflächen dient dem näheren Verständnis der Interaktion von Biomolekülen, Zellen und Tieren während der Fortbewegung mit ihrer Umgebung. Modellsysteme werden dazu mit komplexen Verfahren chemisch modifiziert und deren Topographie mittels Mikrostrukturierungstechniken verändert. Darüber hinaus wurden metallische Oberflächen mit spezieller Strukturierung in die Untersuchungen aufgenommen.

Kernthemen des Programmbereichs: Herstellung von mechanisch schaltbaren Strukturen

Durch Verwendung von Shape-Memory-Polymeren kann die Haftkraft adhäsiver Strukturen durch Temperaturänderung beeinflusst werden. Das Einbringen von magnetischen Nanopartikeln erlaubt eine Strukturveränderung mittels eines externen magnetischen Felds.

Herstellung von chemisch schaltbaren Strukturen

Mit Hilfe von UV-reaktiven chemischen Schutzgruppen können Oberflächen so modifiziert werden, dass sich die adhäsiven Eigenschaften durch UV-Bestrahlung lokal ändern. Bei UV-Belichtung werden die Schutzgruppen entfernt und die Oberfläche am Ort der Belichtung aktiviert.

Herstellung hierarchischer Strukturen

Mittels Mikrostrukturierungsmethoden ist es gelungen, Adhäsionsstrukturen mit einer zweistufigen Hierarchie herzustellen.

Hochpräzise Adhäsionsmessungen

Der Neuaufbau eines Adhäsionsmessgeräts ermöglicht Adhäsionsmessungen mit sehr hoher Präzision (Kraftauflösung im sub-µm-Bereich). Mit Hilfe dieses Aufbaus sind erstmalig Messungen von adhäsiven Strukturen gegen unterschiedliche Oberflächenrauhigkeiten und -geometrien möglich.

Zum besseren Verständnis biologischer Haftsysteme werden die Auswirkungen der Geometrie und der Materialeigenschaften der künstlichen Haftstrukturen auf die Haftung untersucht. Gleichzeitig werden weitere Möglichkeiten zur Schaltbarkeit künstlicher Haftsysteme entwickelt. Die so erhaltenen bioinspirierten Haftsysteme können dann mittels oberflächenchemischer Modifizierung weiter optimiert werden.

The program division Functional Surfaces focuses on the design, fabrication and understanding of surfaces and surface films with tuneable properties. These surfaces are developed to mimic adhesion mechanisms by which biomolecules, cells, or even animals interact with solid surfaces during locomotion. Novel micro- and nanofabrication strategies are combined with organic chemistry principles to obtain surfaces with complex compositions, topographies and properties. In this area, two of our recent papers were recognized as among the most-cited in their journals [1, 2]. Complementary topics include the strength and nanoplasticity of fine-scale patterns on metallic surfaces.

Switchable gecko-inspired adhesives using magnetic polymers

We have prepared microstructured surfaces from of an elastomeric material filled with magnetite nanoparticles. By applying an external magnetic field, the magnetic microstructures (pillars of different heights) change their initial shape and bend. The topographical change is reversible and the pillars recover their initial geometry when the magnetic field is removed. This effect can be exploited to obtain surfaces with adhesive properties that can be modulated by means of magnetic fields.

Bioinspired adhesives with hierarchical design [3]

Structured polymer surfaces containing hierarchically ordered pillars were obtained by lithography and soft moulding methods. These structures have helped us elucidate the role of a multilevel structure in gecko-like adhesives. We demonstrated that hierarchy is not a relevant design parameter in adhesion of structured surfaces to planar substrates, but may be relevant in the case of adhesion to rough substrates, where adaptability of the adhesive structure is required.

Geometry optimization in gecko-inspired adhesives [4]

A novel technique for controlled 3-dimensional surface patterning was established using a combination of photolithography, reactive ion etching and soft moulding. With this new technique it is possible to control geometrical parameters of artificial adhesion systems. To characterize the adhesive properties of these systems we have set up a Macroscopic Adhesion measurement Device (MAD) exhibiting high force and displacement resolution. MAD allows the exploration of a variety of novel testing parameters, i.e. surface roughness and substrate geometries (e.g. flat punches, semi-cylinders).

Caged surfaces with different reactive states [5]

We have developed reactive surfaces with latent functional levels that can be individually activated by irradiation at selected wavelengths. Irradiation removes photolabile caging groups from the surface and activates biofunctional ligands.

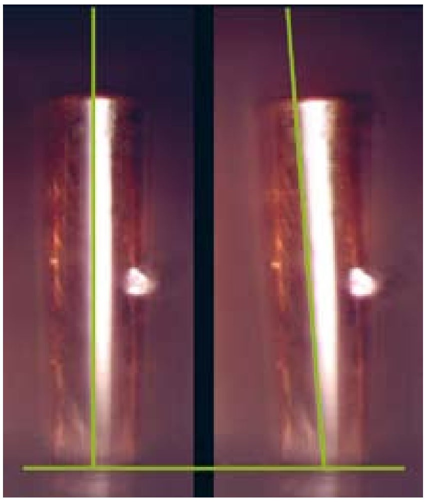


Figure 1: Switching of magnetite/elastomer hybrid microstructures upon application of external magnetic field.

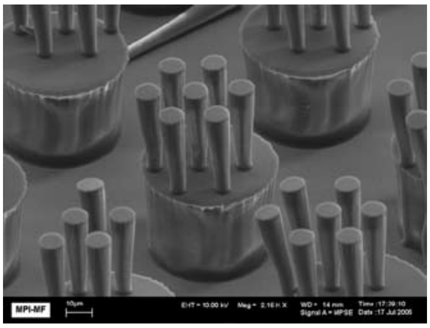


Figure 2: Patterned surface with a two-level hierarchical design.

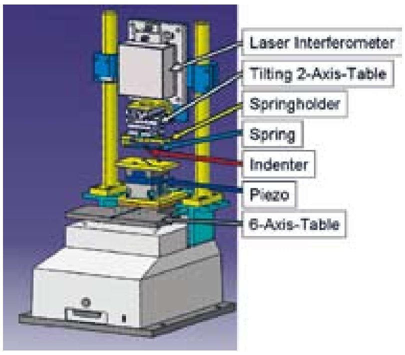


Figure 3: Macroscopic Adhesion measurement Device (MAD).

This strategy has been exploited to immobilize different proteins with spatio-temporal resolution. By using a caged form of RGD peptides, cell patterns have also been obtained.

Outlook

We have identified several design parameters that are important in order to enhance adhesion, among them pillar radius, aspect ratio and tip shape. Nevertheless, the exploration of the parameter space has just begun and thus, geometry and material property optimization are ongoing efforts. Parameters under investigation include the influence of stiffness of the pillars, preload and backing layer thickness. These geometry-property relationships are used to guide the design of novel bioinspired artificial analogues. We are currently exploring gecko-inspired switchable adhesive devices by combining patterning technologies with responsive polymer systems. Dependent on the particular system, switching can be induced using thermal, magnetic, optical or acoustic stimuli. Another focus of future research is the exploration of biomedical applications (collaboration with Saarland University, Homburg). There is a growing need for biocompatible adhesives that can reversibly attach to the underlying tissue. We will investigate the importance of the synthetic pillar geometry, surface chemistry and stiffness in relation to the opposing skin or mucosa surface for optimum adhesion.

The advances in understanding and controlling the properties of bio-inspired adhesive materials, via a collaborative approach among different groups at INM, will ultimately contribute to the development of multifunctional surfaces with tuneable adhesive properties that can be realized over larger areas.

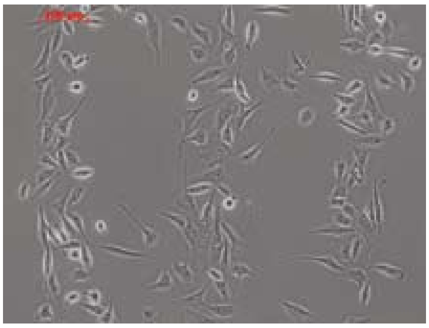


Figure 4: Cell micropattern upon irradiation of caged RGD peptides.

References

[1] A. del Campo, E. Arzt, Macromol. Biosci., 2007, 7, 118-127.

[2] R. Spolenak, S. Gorb, E. Arzt, Acta Biomater., 2005, 1, 5-13.

[3] Ch. Greiner, E. Arzt, A. del Campo, Adv. Mater., 2009, 21, 479-482.

[4] E. Kroner, Ph.D. thesis, in progress.

[5] S. Petersen, J. M. Alonso, A. Specht, P. Duodu, M. Goeldner, A. del Campo, Angew. Chemie, 2008, 120, 3236-3239.

Glass and Optics / Glas und Optik

Dr. Peter W. de Oliveira

Für die zukunftsorientierte Implementierung der grundlagenorientierten Forschungsergebnisse in stärker anwendungsbezogene Bereiche hat der Programmbereich Glas und Optik ein Konzept entwickelt, das die direkte Verknüpfung bestehenden INM-Know-hows, aktueller Forschung und Produktion vorsieht. Aufbauend auf dem Stand der Technik wurden in einem ersten Schritt neue Forschungsfelder und Kooperationsmöglichkeiten identifiziert.

Erste Erfolge daraus zeigen sich in der Materialentwicklung auf Basis von DNS oder ionischen Flüssigkeiten für Informationsspeichermedien. Ferner ist die positive Entwicklung der Anzahl an Journal- und Proceeding-Beiträgen eine direkte Folge der neuen Forschungsstrategie. Darüber hinaus manifestiert sich das obengenannte Konzept in Projekten wie zukunftsorientierten Anwendungsstudien, dem Ausbau INM-interner Kooperationen, der Bildung von Netzwerken mit Universitäten und anderen Forschungseinrichtungen, sowie in der sehr engen Zusammenarbeit mit etablierten, ortsansässigen Firmen und Start-up-Unternehmen. INM-interne Kooperationen bestehen beispielsweise mit dem Programmbereich Nanomere auf dem Gebiet der Nanopartikelfunktionalisierung, mit dem Programmbereich Nanotoxizität zur Entwicklung DNS-basierten holographischen Materials, mit dem Programmbereich Nanoprotekt auf dem Gebiet photopolymerisierbarer Komposite und mit dem PB CVD/PVD-Technologien auf dem Gebiet der Kombination von CVD- und Sol-Gel-Prozess in einer Hybridtechnik.

Die erfolgreiche Zusammenarbeit mit externen, lokalen Partnern zeigte sich unter anderem in der Bildung eines Konsortiums zur Nutzung erneuerbarer Energien unter Beteiligung der lokalen Industrie und der Universität des Saarlandes.

Nicht zuletzt hat das Know-how des Programmbereichs Glas und Optik in den Bereichen optische Materialien und Beschichtungstechnologie wieder zu erfolgreichen Projekten mit deutschen, europäischen und Partnern aus Übersee geführt.

Weitere Schwerpunktthemen umfassen hochtemperaturbeständige Beschichtungssysteme mit Korrosions- und Abriebschutz bis 900° C auf Stahl, kratzfeste Antireflex- und Interferenzschichten. Über die Reduktion der Photokatalyse in Titandioxid für bestimmte optische Anwendungen wird im Artikel 'Work on nonphotocatalytically active titania particles' ausführlich berichtet. Im Bereich der transparenten, leitfähigen Beschichtungen wurden In_2O_3:Sn (ITO) Pellets aus einer alternativen elektrochemischen Syntheseroute elektrisch charakterisiert, sowie Komposite aus Titania und Kohlenstoffnanoröhren entwickelt und untersucht. Experimentelle Techniken zur vertieften Untersuchung der Prozesse bei der Sol-Gel-Synthese mit optischen und spektrometrischen Methoden wurden entwickelt.

In researching materials for optical applications, the program division Glass and Optics combined INM know-how and research and device production. Important considerations have been made to identify new research fields and opportunities using the INM´s existing knowledge in material development. Based on this strategy some of the research fields have been restructured and matched to new goals. The base concept to use nanoparticles, dispersions or nanostructured materials has not been a "sine qua non" condition for the material development. Material based on DNA or ionic liquids for the use in information storage media are examples for this concept.

Basic research management concepts like future orientated applied studies, internal network formation (with other INM´s departments) or external network formation with universities and research centres as well as the very close partnership with local companies and start-ups have been emphasized. Cooperations with other INM´s groups include projects with the PD Nanomers in the area of nanoparticle functionalization, with the PD Nanotoxicity on DNA based holographic materials, with the PD Nanoprotect in the area of photopolymerizable composites and with the PD CVD/PVD Technologies on TCO coatings and hybrid coating technology based on CVD - Sol-Gel process. The increasing output of papers and congress proceedings is a result of the newly applied research strategy.

Partnership with local industry leads to the formation of a consortium for exploitation of renewable energies which consists of Nano-X, Inomat, Prof. S. Hüfner and Prof. R. Hempelmann. The developed know-how in optical materials and coatings technology has attracted projects with German, European and over-seas companies and institutions.

Within the last few years DNA was widely used in optical systems and is also used as a part of holographic security labels. In addition it is known that radical formation within DNA happens easily at e.g. wavelengths between 280 and 320 nm, due to excitation of the heterocyclic bases. With the help of cationic ligands like CTMA it is possible to dilute such modified DNA within organic solvents or matrices. Our objective was to use DNA as an optically active material that gives us the possibility to cause changes in refractive index by irradiation. This can be

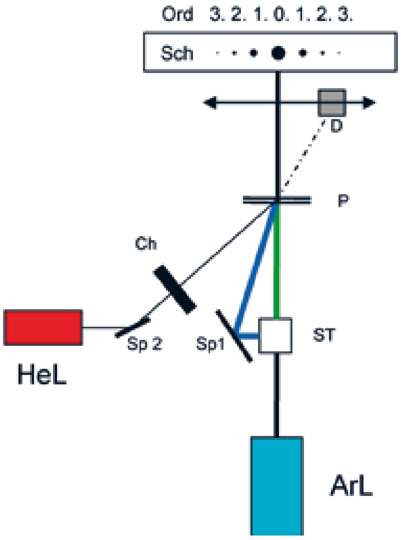


Figure 1: Experimental setup for holography.

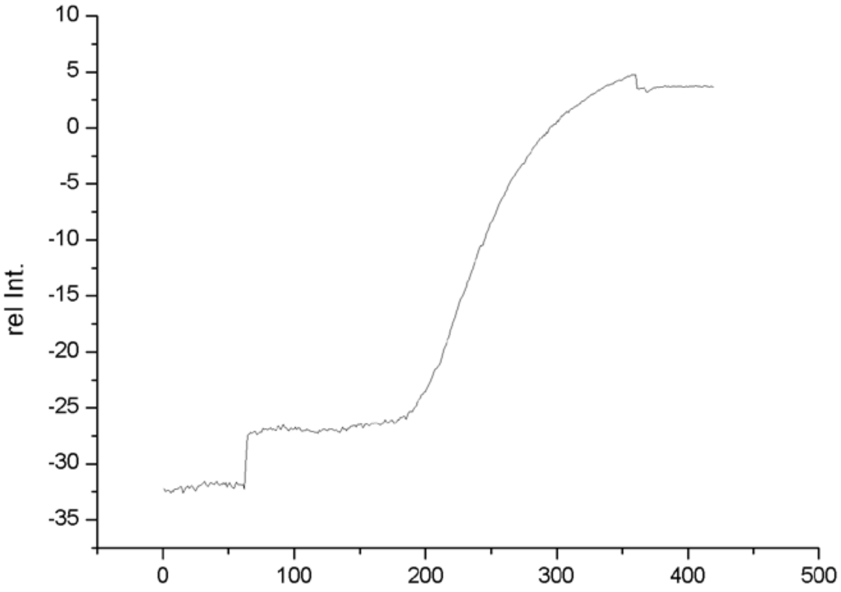


Figure 2: Development of the intensity at first recording diffraction maximum.

achieved either by double strand breaks of the DNA or by using the formed radicals to locally polymerize monomers.

A typical system, which we have used for our experiments, consists of PVAc, acetone, tetraethyleneglycoldimethacrylate and DNA-CTMA-complex in 2-propanol. Films of this mixture were applied to glass slides and dried at various temperatures. After drying, these films have been successfully used in holographic experiments. With an Ar^+-ion-laser it was possible to write a grid hologram. The writing process has been followed by reading the grid hologram with a He-laser and measurement of the intensity at the first diffraction maximum (Figure 1 and 2).

The written grid hologram was further examined by using a light microscope showing the lines of different diffraction indices, the line width of the darker lines is around 2 µm (Figure 3).

From the experiments completed this far it has been shown that it is possible to use modified DNA for the preparation of holograms. However the exact mechanism of this process requires further investigation.

Materials for high temperature stable volume holograms are required for optical devices and decoration. At INM mixtures of titanium or zirconium compounds along with polymerizable monomers, photoinitiators and matrix materials have been developed and used as a holographic recording material or in the preparation of gradient index lenses. Our new objective was to prepare a single precursor which would be able to form a hologram once irradiated within a matrix material. Our reactions start with commercially available titanium or zirconium alcoxides and photoinitiators like Darocur 1173. We have been able to prepare a molecule consisting of a titanium centre and four photoinitiator residues with a classic substitution reaction and also investigate its molecular structure (Figure 4). For holographic experiments with a mixture of these new molecules and PVAc, films have been prepared on glass slides and a grid hologram was written with an Ar^+-ion-laser and read with a He-laser (Figure 5).

The new titanium-photosensitive molecule shows promising results for holographic applications and can also be used as a new radical initiator that already contains a metal centre.

In the area of metals (particularly stainless steel), alloys and glasses have a noble optical appearance, high mechanical stability and are therefore used in many applications. The disadvantages include the sensitivity to corrosion, scratches, oxidation at temperatures above 200-300 °C and the danger of burst, cracks, scratches and splinter (in the case of glass). The adhesion of dirt is often also a problem.

Therefore, the objective of research and development in this area was to synthesize coatings with corrosion and abrasion protection up to 900 °C, a reduced cleaning effort and low cost application techniques for mass production. Composition and densification parameters and their influence on the coating properties were investigated.

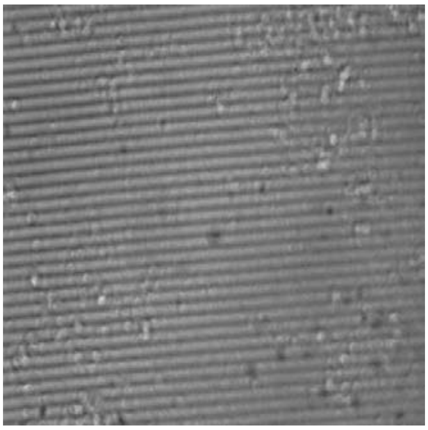


Figure 3: Light microscopic image of the received grid hologram.

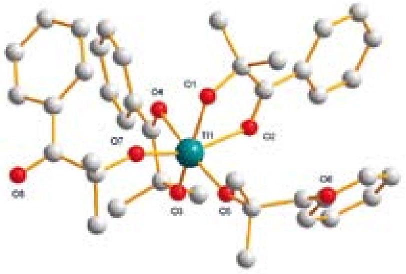


Figure 4: Molecule structure of the product from the reaction of Titanium-tetraisopropanolate with four equivalents of Darocur 1173.

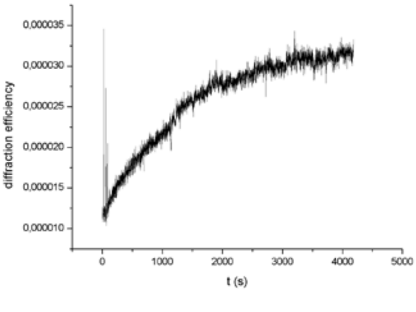


Figure 5: Development of the intensity at first diffraction maximum. The sample has been irradiated three times for two seconds each and after that diffusion processes can be assumed because of the increase in the diffraction efficiency.

For pre-experiments, steel and glass plates were used. The coating sols were mixtures of carbides, graphites, silanes and tribological/hard pigments. After addition of hydroxides of alkali metals and water, the synthesized sol-gel systems were applied by wet chemical methods (spray or dip coating), dried and densified up to 900 °C in air or under inert gases.

For the development of hard optical coatings single crystalline sapphire substrates were spin-coated with two layers of antireflective coatings on one or two sides of the substrate. Various curing temperatures between 600 and 850 °C were used for stack firing of 30 minutes to densify the coatings. The transmittance of the sapphire substrate was enhanced from 85 % to 92 % and 99 % for two layers of antireflective coatings on one side and on two sides, respectively. The scratch resistance of the coating was tested by crockmeter. Tests showed that the samples with curing temperatures of 750 °C and 800 °C started to be scratched after 400 cycles whereas the samples with other curing temperatures started to be scratched after 200 cycles.

Based on the development of functionalization of the optical surfaces for glass and plastics interference coatings for crystals like sapphire were developed. Coatings for crystals need to fulfil stringent requirements like high abrasion resistance and temperature stability. The interference layer system was produced by spin-coating of TiO_2 and SiO_2 layers. TiO_2 (n_D = 1.93) sol was used as the first layer. After a drying at 120 °C for 3 minutes and cooling to room temperature, SiO_2 (n_D = 1.495) sol was coated as a second layer. The whole stack was cured at various temperatures between 600 and 850 °C for 30 minutes to densify the coatings. The transmittance of the coatings was measured by UV-VIS technique. The scratch resistance of the coating was tested by crockmeter. Figure 7 shows the transmittance spectra of the antireflective coatings on the sapphire substrate in the visible range. The transmittance of the sapphire substrate is enhanced from 85 % to 92 % and 99 % for two layers antireflective coatings on one side and on two sides, respectively.

The result of the scratch resistance test by crockmeter is shown in the following table:

Curing temperature [°C]	Number of Cycles until samples started to be scratched
600	200
650	200
700	200
750	400
800	400
850	200

This qualitative scratch resistance test shows that the best curing temperature is between 750 and 800 °C. It can be assumed that below 750 °C the curing process is not completed but on the other hand, temperatures higher than 800 °C

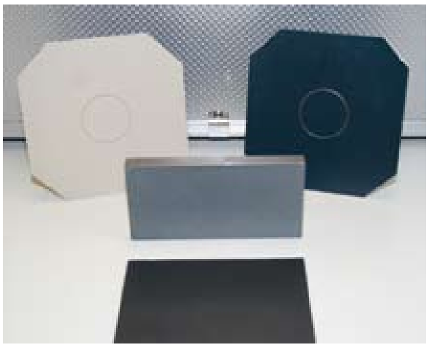


Figure 6: Standard glass-ceramic coatings on steel (top) and borosilicate glass (bottom).

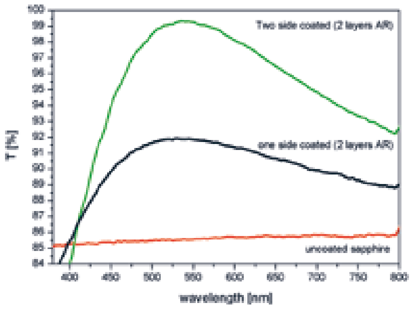


Figure 7: Transmission spectral of antireflective coatings on the sapphire substrate in visible range.

result in a higher porosity in the coatings which reduces scratch resistance.

For many applications in optics the photocatalytical properties of titania are a disadvantage. Due to this reason research aimed at reducing this undesired feature was undertaken. This topic is described in more detail in the article 'Work on non-photocatalytically active titania particles' in the chapter 'scientific articles'.

The electrochemical synthesis of nanoscaled ITO made it possible to work with cheaper raw materials and to avoid expensive and polluting washing processes. Besides the beneficial environmental and cost effects, it aimed at fabricating a product containing less impurities due to the exclusive use of the metals for synthesis. To investigate bulk and surface conductivities on pellets of ITO, pellets were prepared from nanoscaled powder via uniaxial and hydrostatic pressing.

Figure 8 shows the effect of a pre-sintering calcination treatment. Calcination before sintering allows organic components to burn-out more effectively, and thus, higher pellet densities are achieved, resulting in remarkable enhancements of the conductivity for example from 22 Scm^{-1} to 70 Scm^{-1} (900 °C) and from 46 Scm^{-1} to 137 Scm^{-1} (1000 °C). At 1100 °C, the microstructure development is presumably more or less completed and so the effect by the pre-treatment is not observable. It is interesting that the effect of the calcinations only improves the set of samples simply oxidized by sintering. In contrast for the reduced series, lower conductivities are measured: 406 Scm^{-1} instead of 852 Scm^{-1} (for 900 °C), 730 Scm^{-1} instead of 911 Scm^{-1} (for 1000 °C) and 878 Scm^{-1} instead of 1442 Scm^{-1} for 1100 °C. The optical appearance of the samples with prior calcination and sintering and final reduction shows that the reduction is not as distinct as is the case for the samples only sintered and reduced. It is possible that this is due to higher densities resulting from the further developed microstructure, which might have hindered a thorough reduction of the samples.

Conductive coatings based on carbon nanotubes (CNT) have been investigated as an option for transparent conductive ITO coatings. Films of titania/multi-walled carbon nanotube (MWNT) nanocomposites were prepared on borosilicate glass substrate and sintered in air and in forming gas (N_2:H_2 = 95:5) at 300 °C. The electrical and structural properties of the coatings were investigated. It was observed that the sheet resistance of films treated in the reducing atmosphere of N_2:H_2 is two times lower than that of films sintered in air. This could be an indication of a more efficient densification of the films treated under forming gas, as revealed by field-emission scanning electron microscopy (FESEM). Moreover, Raman spectroscopy of the coatings treated under N_2:H_2 revealed a decrease in the defect band of MWNTs (1330 cm^{-1}) compared with those sintered in air. This is probably related to the removal of amorphous carbon from the surface of the coatings treated under N_2:H_2 and may also contribute to the enhancement of the electrical properties of the film. Raman spectroscopy of

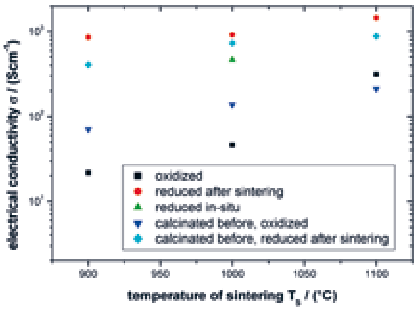


Figure 8: Electrical conductivities for pellets after various treatments: oxidized and reduced, either with and without prior calcination, single point at 1000 °C: reduced in-situ while sintering.

TiO_2/CNT coatings sintered in forming gas and in air shows a decrease in the defect band (D) of MWNTs treated under forming gas. The D/G ratio of films sintered in air is 1.51, against 1.30 in N_2:H_2 sintering (G band ~ 1590 cm^{-1}).

A tool intended to provide some deeper insight into sol-gel coating processes by allowing an *in-situ* characterization of sol-gel films during their formation and (initial) densification was developed. The optical characterization comprises a reflection spectrometer in order to measure the optical thickness of the film, defined as the geometrical thickness multiplied by its refractive index, and a video microscope allowing to monitor the formation of structures such as scattering centers or cracks. An environmental cell to enclose the sample before and after dipping is currently under construction. This will allow for a tight control of the temperature and humidity, such that the effects of small changes can be detected with high sensitivity. It will also allow for a controlled heating up to ~ 150 °C, such that thermal curing processes can also be investigated. A fiber coupled visible light spectrometer (Ocean Optics USB2000) is used to measure the reflection spectra. The CCD detector array measures light from 350 to 1000 nm, but the grating characteristics limit the useful wavelength range to approximately 400 to 900 nm. The spectrometer is combined with a tungsten-halogen lamp via a fiber reflection probe containing six illumination fibers surrounding a single detection fiber. The face of the probe head is imaged to the sample using an achromatic doublet lens, in order to maximize the signal while minimizing the contribution of ambient light and the reflection from the windows of the environmental cell to be added. Details of the optical setup are shown in Figure 9, together with the microscope. Video microscopy was used in preparatory experiments in summer 2008 during a student summer internship from Santa Barbara University.

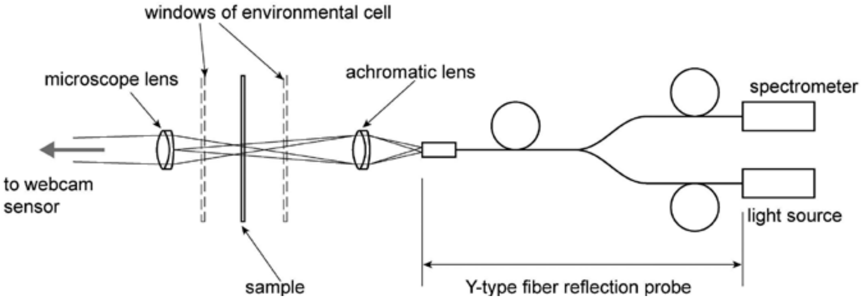


Figure 9: The optical setup integrated into the dip coating apparatus, comprising the reflection spectrometer and the microscope, of which only the lens is shown.

The head of the fiber probe contains 6 illumination fibers surrounding a single detection fiber. This is imaged into a spot of ~ 2 mm diameter on the sample. In order to efficiently couple the reflected light from the illumination fibers into the detection fiber, the focus of the lens on the spectrometer side does not coincide with either of the sample surfaces, but is a few millimetres on the far side of the sample.

Life Science/Biomimetics / Life Science/Biomimetik

Prof. Dr. Dr. h. c. Michael Veith, Dr. Stefan Gerbes

Im Programmbereich Life Science/Biomimetik werden nanoskalige Systeme für medizinische sowie für bio- und lebensmitteltechnische Anwendungen entwickelt. Es wurde vorwiegend grundlagenorientiert in öffentlich geförderten Projekten gearbeitet, jedoch mit einem starken Anwendungsbezug. Im Jahr 2008 standen folgende Arbeiten im Mittelpunkt des Programmbereiches:

Der Schwerpunkt der durch das BMBF unterstützten Tätigkeit lag auf der Entwicklung anorganischer Nanopartikelsuspensionen für den Wirkstofftransport. Diese sollen einen Photoinitiator in die Mitochondrien von Krebszellen transportieren. In der photodynamischen Therapie (PDT) wird der Photoinitiator dann mittels UV-Licht angeregt und zerstört die Krebszelle von innen. Dabei werden die Partikel auch auf Nebenwirkungen insbesondere auf das Immunsystem untersucht. Unter den Partikeln, die eingesetzt werden sollen, sind auch TiO_2-Partikel. Für den Einsatz in Zellkulturversuchen sind die Partikel aber noch zu modifizieren, damit diese auch bei pH 7 stabil in Suspension gehalten werden können. Um den Verbleib der Partikel verfolgen zu können, sind diese Partikel mit Fluoreszenzmarkern versehen worden.

Für das EU-Projekt CellPROM wurden topographisch und chemisch strukturierte Oberflächen entwickelt. Dazu war auch die Stabilität der Anbindung verschiedener Oberflächenmodifikatoren, die als Linker zur Anbindung von Biomolekülen dienen sollten, unter den Bedingungen in der Zellkultur zu untersuchen.

Des Weiteren wurde das BMBF-geförderte Projekt ZOVAN (Zahnoberflächenversiegelung mit einer antiadhäsiven Nanokompositbeschichtung) abgeschlossen. Es wurde nachgewiesen, dass die lichthärtenden und bioverträglichen Nanomer-Materialien aus dem Projekt die Anhaftung von Biofilmen (Plaque) deutlich reduzieren können. Allerdings sind noch weitere Untersuchungen erforderlich, um den Wirkmechanismus aufzuklären und um alle Anforderungen an ein zahnmedizinisches Produkt zu erfüllen.

Based on chemical nanotechnology, the program division Life Science/Biomimetics focuses on the development of inorganic-organic materials for applications in medical science, bio- or food-technology. In the following some highlights of our research and development work done in 2008 are presented.

Inorganic particles as drug-carrier

The interaction between nanoparticles and living cells is a very promising research field from a toxicological as well as a pharmaceutical point of view. In two German-Chinese projects, supported by the German ministry of education and research (BMBF), the interaction of nanoparticles with human cells is investigated by partners in order to improve their usability as drug carriers in the photodynamic therapy (PDT). Therefore, the INM develops inorganic nanoparticles that differ in composition, size and shape.

The specific requirements on the particulate suspensions for this type of applications are rather stringent. First of all the particles have to be non-toxic. The particles also must be stable against agglomeration even in cell-culture media, which is not trivial; since inorganic suspensions are stabilized normally by electrostatic repulsion, they are very sensitive to higher ion-forces and concentrations. A modification of the particles is necessary to shift to another stabilizing mechanism (as the distribution of the drug is monitored in the living cell). Both the drug and the particles must be fluorescent. Last but not least, all suspensions have to be sterile, since cells of the human immune systems are involved which will react even to metabolites of microorganisms.

While working on SiO_2-particles of different sizes and modifications, we started, in a parallel project in 2008, the synthesis of modified TiO_2- and Hydroxyapatite nanoparticles to be used as drug carriers.

The development of a new synthesis route resulted in a monomodal size distribution of the TiO_2-nanoparticles (see figure 1).

Since titania particles have an isoelectrical point at pH-values between 6 and 7, they normally agglomerate under physiological conditions. Thus we introduce molecules

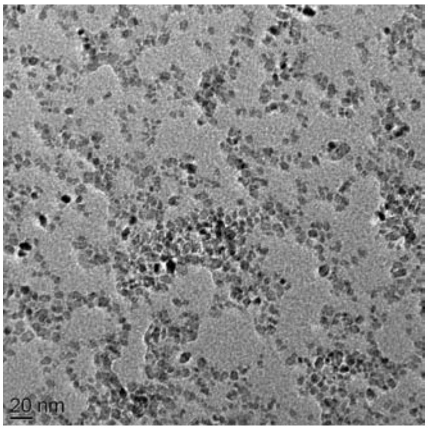


Figure 1: TiO_2-nanoparticles of about 10 nm mean diameter.

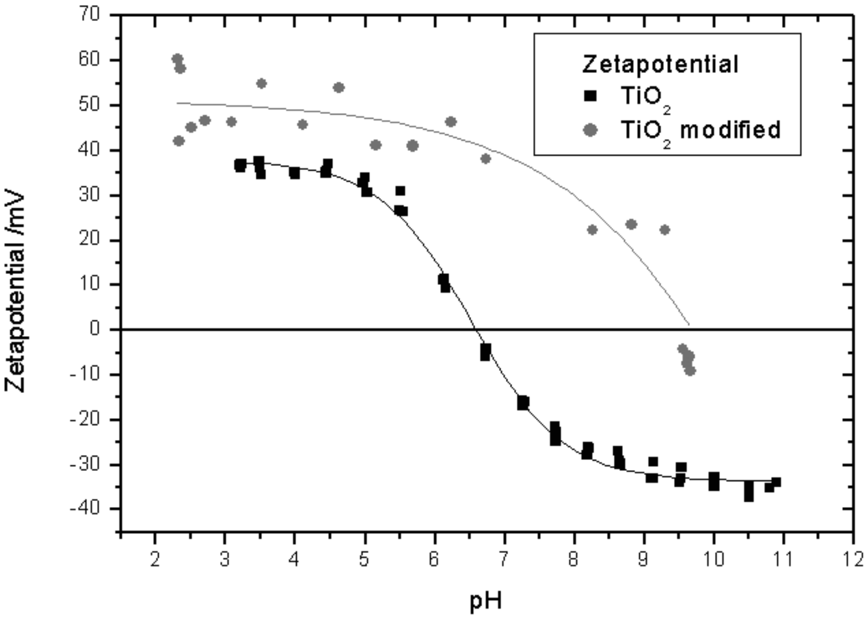


Figure 2: Effect of modification on the zetapotential of TiO_2 nanoparticles; the surface modified particles (circles) show a shift of the zetapotential to higher values, the isoelectric point is shifted from pH 6.5 to pH 9.5.

bearing primary amino-groups in order to obtain additional positive charges on the particle surface. As a result (see figure 2), the isoelectrical point shifts from about 6.5 to pH values beyond 9, which would be sufficient to get stable suspensions in physiological environment.

These particles shall then be loaded with drugs which have to be delivered to specific sites inside the cells. The drugs can be conjugated to the particles covalently, electrostatically or adsorptively

The European project Cellprom was completed in 2008. In the project new surfaces – topographically and chemically structured – were developed. Additionally the stability of the linkages of biomolecules on the nanoparticle-surface was investigated in using different linkers and presence of different cell culture media.

Furthermore, the BMBF-funded project ZOVAN was successfully completed. Aim of the project was the development of a seal for tooth surfaces with antiadhesive properties. It was demonstrated that the light-curing and biocompatible nanomer materials from the project can significantly reduce the attachment of biofilms (dental plaque). However, further investigation is needed to clarify the mechanism of action and to meet all requirements for a dental product.

Modelling/Simulation / Modellierung/Simulation

Prof. Dr. Robert McMeeking

Der Programmbereich, kommissarisch geleitet von Prof. Dr. Robert McMeeking (University of California, Santa Barbara, UCSB), ist derzeit im personellen Aufbau begriffen. Es ist geplant, dass der Programmbereich bis im Jahr 2010 die anderen Programmbereiche maßgeblich unterstützen sowie eigene Forschungsprojekte betreiben wird.

Im Jahr 2008 führte McMeeking einige Forschungsprojekte zu Themen durch, die relevant für gegenwärtige Arbeiten am INM sind. Hierfür konnte er durch seine Kontakte an der UCSB und der University of Aberdeen in Schottland Synergien mit Projekten nutzen, die derzeit an diesen Einrichtungen bearbeitet werden. Einen Schwerpunkt seiner Arbeit stellte die Adhäsion zwischen einer steifen, glatten und einer nachgiebigen, fibrillar sphärischen Oberfläche dar. Hierbei wurden die Abzugskräfte zwischen diesen Oberflächen modelliert. Bei Arbeiten zur Zellmechanik wurde ein Modell zur Kontraktilität, zum Umbau von Zytoskeletten und zur Adhäsion in biologischen Zellen entwickelt, das an Signale gekoppelt ist, die durch Proteine generiert werden. Diese werden ihrerseits durch mechanische Stimulation von Ligamenten erzeugt, an welche die Zellen angelagert sind. Schließlich wurden Modelle entwickelt, die das Erstarrungsverhalten von nanopartikelhaltigen Schmelzen simulieren. Hier wird die Wechselwirkung zwischen der Erstarrungsfront und den Nanopartikeln betrachtet, um Prozesse zu identifizieren, die die Dispersionshärtung mit Hilfe der Partikel erlauben.

Folgende Themen werden aus heutiger Sicht in Zukunft von Interesse sein:

- Kontaktmechanik strukturierter Systeme
- Tribologie von Hybridschichten
- Benetzung und Kapillarität
- Adhäsion
- Optimierung mikrooptischer Systeme
- Mikromechanik natürlicher und künstlicher Kompositsysteme
- Simulation von Wachstumsprozessen
- Modellierung von Zelladhäsion und zellbiologischen Prozessen, insbesondere in Wechselwirkung mit Substraten oder Nanopartikeln

This program division is in a set-up stage. Since 1 April 2008, Prof. Dr. Robert McMeeking from the University of California, Santa Barbara (UCSB) has been its provisional head. Currently, Prof. Dr. Daniel Strauss is a senior member of the division, and he and McMeeking collaborate on both research projects and planning for the future of the program. By 2010, the division should be providing considerable support to the other program divisions, as well as having ongoing research projects selected according to the research interests and activities of division personnel.

Research projects

McMeeking pursued a small number of research projects relevant to topics currently of importance within INM. His contacts at UCSB and the University of Aberdeen, Scotland enabled him to exploit synergies with on-going projects at those two institutions, and, given the limited staff of the simulation and modelling division at INM, to capitalize on available research activities at those places.

Adhesion: Models of adhesion between a stiff, smooth surface and a compliant, fibrillar spherical surface have been developed. These models allow simulation of experiments carried out at INM and elsewhere in which the pull-off forces between such surfaces have been measured. The results of the simulations allow hypothesis testing to be carried out regarding the nature of adhesion between the fibrils on the spherical surface and the flat object. For example, assessments can be made in regard to whether the pull-off condition for an individual fibril is deterministic, or if it obeys probabilistic behaviour in which the force at which separation occurs is controlled by defects in the surfaces that are adhering.

Cell mechanics: A model for contractility, cytoskeleton remodelling, and adhesion in biological cells has been developed, coupled to signalling controlled by proteins generated in response to mechanical stimulations of the ligaments to which the cell is attached. This model is able to simulate various experimental observations such as the scaling of contractile forces with the stiffness of the cell's environment, and the orientation of cytoskeletal stress-fibres during cyclic straining of the cell. The model is being extended to allow for its utilization in particle toxicology simulations, where endocytosis ingests particles, and stimulates remodelling of the cytoskeleton. Such models should be relevant to issues of nanotoxicology that are being considered at INM. In addition, preliminary thought has been developed for developing the cell model in the direction of biomineralization, another topic that is actively pursued at INM.

Solidification of melts containing nanoparticles: Models are being developed for simulating solidification fronts that interact with a family of nanoparticles to identify processes that will enable the dispersion of such particles within the solidified material for purposes of strengthening. Competition among drag on the nanoparticles, their inertia and surface tension among the solid, liquid and particle surfaces can dominate the processes involved, determining whether the nanoparticles are driven into the liquid ahead of the solidification front or are ingested into the solidifying solid. The simulations are being pursued to identify the range of processing parameters (*i.e.* cooling rate, speed of the solidification front, *etc.*) that ensures incorporation of the nanoparticles into the solid and their even dispersion.

Outlook

From today's perspective, the following topics are assessed to be of significance for future developments in modelling and simulation, given directions within INM, and will be considered for future effort as the division builds in strength and resources:

- contact mechanics of structured systems
- tribology of hybrid layered systems
- wetting and capillarity
- adhesion
- optimization of micro-optical systems
- micro mechanics of natural and artificial composite systems
- simulation of growth processes
- modelling of cell adhesion and of cell biological processes, especially in interaction with substrates or nanoparticles

Nanomers / Nanomere

Dr. Carsten Becker-Willinger

Die Forschungs- und Entwicklungsarbeiten des Programmbereiches Nanomere liegen schwerpunktmäßig in der Synthese, Oberflächenmodifikation und Verwendung von Nanopartikeln in polymerartigen bzw. organisch-anorganischen Matrices zur Herstellung von Nanokompositsystemen, den so genannten Nanomeren. Diese Materialklasse besitzt eine hohe werkstofftechnische Variabilität und bietet die Möglichkeit, maßgeschneiderte Eigenschaftsprofile für ganz spezifische Anwendungen einzustellen. Die Aktivitäten des Programmbereiches Nanomere, der aus 13 Mitarbeitern besteht, beschränkten sich dabei im Jahr 2008 nicht nur auf Basisentwicklungen für neue Werkstoffkonzepte, sondern umfassten in großem Maße auch industrielle Forschungsprojekte zur angepassten Materialentwicklung (z. T. öffentlich gefördert über z. B. BMBF). Schwerpunktmäßig wurden dabei tribologische Beschichtungen mit hoher Verschleißbeständigkeit und extrem niedrigem Gleitreibungskoeffizienten, abriebbeständigen Antifingerprintbeschichtungen sowie Niedrigenergieoberflächen mit hoher Dauerhaftigkeit und elektrischen Isolationseigenschaften sowie kompakte optische Materialien bearbeitet. Weiterhin wurde in den meisten Industrieprojekten zusätzlich zur Materialoptimierung auch eine Technologieentwicklung durchgeführt. Hierzu gab es eine enge Zusammenarbeit mit dem Anwendungszentrum des INM, NMO, um die Resultate vom Labormaßstab in die praktische Anwendung zu überführen. Zusätzlich wurden die Entwicklungen des Programmbereiches Nanomere auch weltweit auf entsprechenden Ausstellungen und Industriemessen sowie Roadshows präsentiert. Aufgrund der Resonanz dieser Präsentationen kann auf ein nach wie vor großes Potential der Nanomer-Technologie im Sinne einer breiten industriellen Anwendung gesetzt werden.

The research and development activities in the program division Nanomers are focused on the synthesis, surface modification and dispersion of inorganic nanoscale particles in different media, as well as their transfer into polymeric and organic-inorganic matrices in order to form nanocomposite systems, the so called Nanomers. These types of materials possess a high variability in their chemical structure and open the possibility to fulfil very specific requirements even for contradictory combinations of materials properties. The wet chemical synthesis approach enables to process the materials using technically relevant and cost saving methods, as well as to develop and optimize materials with tailored properties with regard to a broad variety of different, in many cases industrial applications. In 2008 many activities on the generation of basic knowledge and also on industrial research projects have been performed in the program division Nanomers which comprises 13 co-workers. To create basic knowledge, three PhD theses have been carried out over the year aiming at new tribological systems, transparent barrier functions and star-type nanostructures. Most of the industrial projects have been focused on coating applications, mainly in the fields of abrasion resistant functional coatings, tribological layers with high wear resistance, as well as low surface free energy coatings with high durability and special electrical insulation properties. The use of nanoparticles with their intrinsic solid state physical properties enables to add various features with new interesting functionalities to the Nanomer systems.

Another sector of the research activities was the use of nanoparticles for the modification of compact materials such as duroplastic resins in order to change e.g. their electrical, thermal and mechanical properties.

A highly innovative field with big potential for the future is the area of tribological low friction coatings. In addition to the last year, where low friction coating systems containing solid state lubricants were developed combining a low coefficient of friction with excellent adhesion and corrosion protection on low alloyed steel, in 2008 new Nanomer compositions with low friction and high wear resistance were developed for the use on plastic fastening parts for the automotive industry and for steel parts in rotating systems as well as valves. Figure 1 shows the experimental set-up of the pin-on-disc tribometer, the standard testing equipment for the determination of the long term tribological behaviour such as

Figure 1: Pin-on-disc tribometer test on Nanomer coated steel plate.

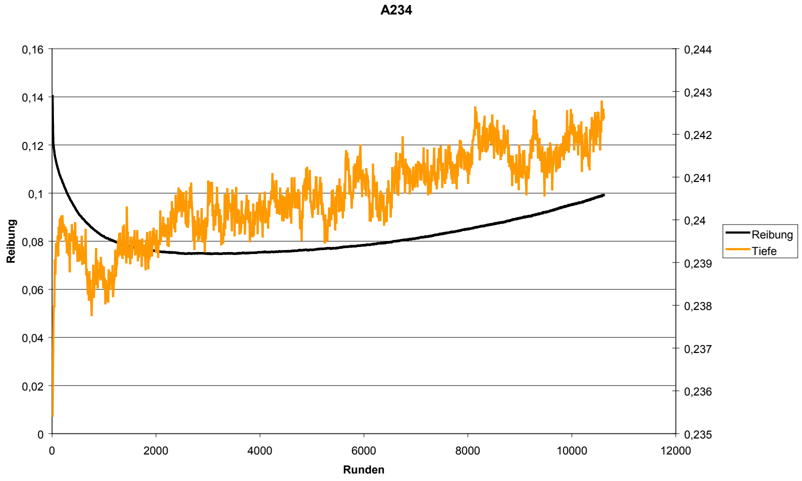


Figure 2: Coefficient of friction and transfer film formation for a tribological Nanomer coating dependencing on the number of rotations in the pin-on-disc tribometer.

coefficient of friction and wear coefficient of coatings.

New materials combinations and processing technologies enabled to approach the region of coefficient of friction which is normally covered by hydrodynamic lubrication only. Figure 2 shows an example of such a nanocomposite coating material containing solid state lubricants but at the same time showing extremely low coefficient of friction without the use of hydrodynamic friction mechanisms usually known from oils.

Figure 2 shows that a coefficient of friction below $\mu = 0.08$ can be achieved for a system containing only solid state lubricants over a long sliding distance. This achievement is an important milestone which shows the great potential of the Nanomer systems.

In particular basic investigations in the field of Nanomer coating systems have shown that even small amounts of special nanoparticles in the lower nanometer size range can dramatically improve the long term electrical insulation properties of commercially available organic coating systems. Nanoparticles with special electronic structure have been successfully incorporated into insulation materials, showing 100 times higher insulation capability compared to conventional coating systems. For this materials principle a patent has been filed. In 2008, this project has been finalized by performing the technology development together with the NMO application centre. The material itself is now running in field tests at the industrial partner company.

In the field of mechanical improvement of coating systems by the use of nanoparticles, a development project has been performed to derive scratch resistant anti-fingerprint coatings for coil coating applications on zinc galvanised steel. Figure 3 shows a coated steel plate in the abrasion test.

In 2009, we hope to develop the appropriate technology to bring the material towards production. In the same sense also an abrasion and impact resistant long term labelling paint for steel parts was developed successfully. This material will be transferred to application in the beginning of 2009.

In 2008, several projects supported by the federal government of Germany have been executed. Considerable investigation effort has also been set on the use of nanoparticulate additives for conventional coatings and paint systems with respect to polymerisation catalysing properties. In a first project supported by the BMBF, the investigations focused on the effect of nanoparticles catalysing, especially UV photo polymerization reactions in acrylic coating systems. Figure 4 shows the nanoparticulate anatase particles, which have been developed for the use as photo polymerisation initiators.

By using particles of this type in a concentration of 2 %, an increase of the polymerization speed up to 75 % was achieved compared to the unfilled acrylic systems. This intrinsic property of the nanoparticles has high industrial demand and can be a useful alternative for conventional

Figure 3: Pin-on-disc abrasion test for linear back-and-forth-movement of the testing head.

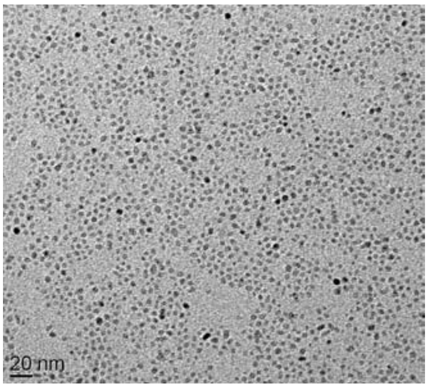


Figure 4: Photopolymerizing anatase nanoparticles from lyothermal synthesis.

approaches using organic molecules as photo initiators.

In a second project supported by the BMBF, nanoscale additives have been investigated as initiators selectively absorbing in the infrared region in order to develop thermally fast curing systems for adhesive bonding.

In the field of anti-adhesive coatings, an industrial project aims to develop materials which improve the emptying behavior of containers for chemicals. The investigations are still running. The results from the basic investigation concerning this topic can be used to optimize the materials with respect to the substrate surface, the surface morphology and the surface free energy in order to find a tailored solution for specific problems in various other relevant industrial applications. For the types of extremely abrasion resistant anti-adhesive nanocomposites, successful field tests could be performed in the area of power generation equipment.

In the area of polymer matrix nanocomposites, nanoparticles have been used in such matrices to tailor optical, thermal and mechanical properties of compact bulk materials. Reactively curable resins such as epoxies and polyurethanes as well as thermoplastic matrices have been filled with nanoscale particles that can be compounded, extruded or injection moulded. By this approach, transparent, temperature-stable resins have been developed that can be used for the fabrication of optical elements. These materials combine easy processing ability with relatively low density compared to glass and tailored ability of optical properties such as refractive index. In this direction, composite systems derived from epoxy-hybrid matrices and nanoparticles with high density have been developed, enabling one to derive a composite with tuneable bulk density which is important to adjust e.g. the acoustic wave transmittance of such type of material. Figure 5 shows the dependence of the acoustic impedance of a nanocomposite layer on the degree of filling with high density nanoparticles.

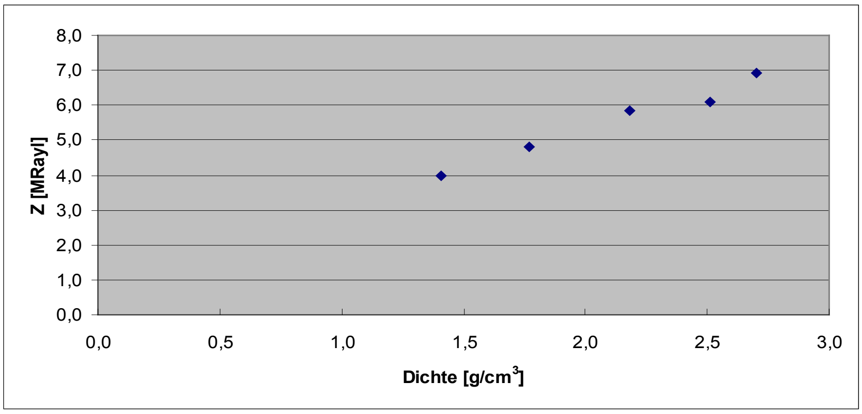


Figure 5: Acoustic impedance Z in dependence on the density of nanocomposites containing increasing amount of high density nanoparticles in epoxy matrix.

This research activity has been financially supported by the ministry of economics and science of the state of Saarland and resulted in a joint patent application which helps to strengthen the position of the local economy.

Investments in new equipment have been made by this division, to enable processing of not only reactive curable systems but also thermoplastic materials for refractive optical and foil applications. Figure 6 shows the new extrusion system for preparation of thermoplastic foils.

A big part of the know-how in this program division can be ascribed to the specific surface modification and compatibilization of nanoparticles with polymer type matrices, important to obtain nanocomposite systems with almost perfectly dispersed nanoparticles. This foundation is an important tool used to develop tailored materials within a short time. Development of new interesting chemical structures and new composite morphologies has been added within the course of three PhD thesis works.

In 2008, many projects with industrial partners have been performed in the group. Furthermore, it is important to note that for most of the cooperation projects a technology development in addition to the pure materials optimisation was of high importance in order to transfer the results from the lab scale to the practical application. This step has been performed together with the NMO, the application centre of the INM. Furthermore the group has also participated in many expositions on fairs and road shows ranging from Germany to Japan and Korea as well as to Argentina and Brazil. The results of these fair and road show activities have made clear that the Nanomer technology still has a big potential for a broad range of industrial applications especially for the energy and power generation, engineering and printing industry. In this sense it is expected that the chemical nanotechnology approach has not tapped its full potential up to now and still should be able to open completely new solutions in the future.

Figure 6: New foil extruder for thermoplastic foil production at the lab scale.

Nanoprotect/Combinatorial material development / Nanoprotekt/Kombinatorische Materialentwicklung

Dr. Matthias Wittmar

Hauptaufgaben des Programmbereichs Nanoprotekt/Kombinatorische Materialentwicklung sind die Synthese, Oberflächenmodifizierung und Anpassung von unterschiedlichen, zumeist oxidischen Partikeln an unterschiedlichste Medien, sowie die Verwendung derartig modifizierter Partikel in anorganisch-organischen Hybridlacken für den Korrosionsschutz von Metallen. Zudem arbeitet der Programmbereich in der Entwicklung von Bindersystemen für viele Materialien und von neuen Schutzsystemen für Leichtmetalllegierungen und Stahl.

Eine weitere Kompetenz des Programmbereichs ist die Entwicklung von neuen Materialien mit kombinatorischen Methoden.

Im Jahr 2008 war der Programmbereich in vier großen, öffentlich geförderten Projekten tätig, MULTIPROTECT, PriMeBits, UNACON und das AiF-Projekt, deren Bearbeitung Hauptthema im Jahr 2008 war. Das AiF-Projekt zur Entwicklung eines transluzenten Schutzlackes für Holz gegen UV-Bestrahlung und Bewitterung wurde in diesem Jahr erfolgreich abgeschlossen. Zudem wurden drei größere Industrieprojekte und viele kleinere Aktivitäten mit Industriepartnern bearbeitet sowie die Suche nach neuen Materialien mit ultrahohen Dielektrizitätskonstanten mit Hilfe eines kombinatorischen Ansatzes fortgesetzt.

2009 werden die Projekte MULTIPROTECT, PriMeBits und UNACON weiter fortgesetzt. MULTIPROTECT wird in diesem Jahr das Ende seiner Laufzeit erreichen. Die Aktivitäten im Bereich kombinatorische Werkstoffentwicklung sollen ebenfalls fortgesetzt werden.

Introduction

The main objectives of the program division Nanoprotect/Combinatorial material development are the synthesis, surface modification and adjustment of different, mainly oxidic, particles in organic solvents and water solutions and the use of such particles in inorganic-organic hybrid lacquers for the corrosion protection of metals and in binder systems. Further objectives are the development of binder systems for a diversity of bindable materials and the development of anticorrosive inorganic-organic hybrid lacquers systems for the protection of light alloys and steel. The development of novel materials by combinatorial techniques also lies among the competences of the division.

During 2008 the division was active in four publicly funded projects. The AiF project dealing with the protection of wood against UV light was successfully finalized. Three major industrial projects were handled and several other smaller activities with industrial clients, such as sample preparation, took place.

Anticorrosive coatings

In 2008 the department continued the coordination of the European project MULTIPROTECT (31 partners from 13 European countries). During the review meeting with the European commission in Bucharest (Romania), the project was successfully defended and assessed. Another outcome of this meeting was the decision of the consortium on the systems to be used for coating demonstration. Two inorganic-organic hybrid lacquers developed at INM were selected: one material for the corrosion protection of aluminium alloy 2024 and a second material for the protection of steel. Both systems were upscaled to 4-5 kg batches and successfully applied by industrial application techniques. Together with an industrial partner in MULTIPROTECT, a coating for magnetic ballasts was adjusted to electrical steel substrates. Better performance than that of the currently used commercial coating of these ballasts was achieved and proofed by a neutral salt spray test. New types of corrosion inhibitors incorporated into inorganic-organic hybrid lacquers were investigated. In particular, the combined use of three different inhibitors secured high barrier properties combined with small delamination at an artificial scratch as observed in accelerated corrosion tests according to ISO 9227. To simplify industrial synthesis while retaining the high barrier and protection abilities of former evolved systems, alternative compositions for anti corrosive lacquers were developed. In another study, a corrosion protection lacquer was mixed with particles of CeO_2 in sub-micron and nanometer range to investigate the influence of the particle size on the barrier properties of the resulting coating. A better performance of the nano doped coating was found. This behaviour seems to be caused by better distribution and better crosslinking in the coating.


Figure 1: INM-anticorrosion coating on a Multiprotect demonstrator: aircraft skin with welded stringers provided by EADS.

Particle development and use

In the PriMeBits project, a printable electric low-voltage nonvolatile memory is developed for printed sensor, media and wireless-ID applications. The main strategy is to utilize printing technology where it has a competitive advantage compared to silicon technology. The project builds on basic research in new materials and components and takes the results into prototyping of new applications. To reduce the research risk, two different technologies for the memory functionality are considered with partially overlapping application areas.

Current printable polymer-based memory technologies typically suffer from i) the required operating voltage being high, ii) insufficient lifetime in room atmosphere, iii) poor temperature stability, iv) chemically reactive materials needing encapsulation and/or v) time-consuming temperature-annealing steps in fabrication. Consequently, for many commercially attractive passive and battery-powered applications, the properties of current printable memories are unsuited. To overcome the drawbacks of prior-art approaches, printable inorganic metal oxide nanoparticle-based ferroelectric FRAM memory and a resistive metallic-based write-once-read-many (WORM) memory will be developed. To print the ferroelectric memory, new printing inks based on, for example, barium titanate ($BaTiO_3$) nanoparticles will be developed. For the WORM memory, the project will aim at utilizing commercial metal nanoparticle inks with possibly some customization. Depending on the application, printed circuitry or an external device is used for the reading and writing of the memory.

The project partners are: VTT, Technical Research Centre of Finland, INM – Leibniz Institut für Neue Materialien gGmbH, MidSweden University, Ecole Polytechnique Fédérale de Lausanne (EPFL), Sensible Solutions Sweden AB, Evonik Degussa GmbH, Motorola GmbH, ARDACO a.s., UPC Consulting Ltd. / UpCode Ltd., Stora Enso Oyj.

Within PriMeBits, INM is responsible for the development of synthesis routes for deagglomerated ferroelectric nanoparticles which are tailored with regard to the envisaged printing process and device. INM provides the required nanoparticles in the form of pastes and dispersions to the partners.

For the FRAM, the obtained results during the first project year are encouraging. At INM, ferroelectric nanoparticles have been successfully synthesized. The nanoparticles have been characterized in depth with respect to size, morphology, structure and composition. Further, ferroelectric properties of commercial and own particles have been characterised at INM on the basis of prototype systems. For the non-printed prototype systems, $Pr > 1.5\ \mu C/cm^2$ at 25 V/µm and a storage time of the order of hours has been demonstrated. For the systems fully-gravure-printed by VTT, $Pr \sim 0.05\ \mu C/cm^2$ at 10 V/µm and storage time of minutes have been demonstrated. Altogether, the project is on track towards developing printed low-voltage WORM and FRAM

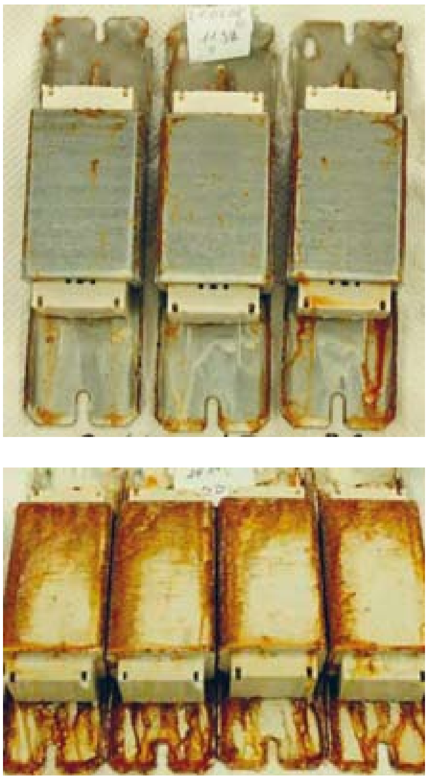


Figure 2: Results of corrosion tests of samples coated with (a) commercial insulation resin for ballasts and (b) with the anticorrosion coating for steel developed by INM after 120 h in neutral salt spray test.

technologies for single-bit event-logging sensors as well as multi-bit data-tag applications.

Project website address:
www.primebits.eu

Combinatorial material development

In 2008, work in the field of combinatorial material development concentrated on fundamental research with a focus on parallel synthesis techniques. The development of a new combinatorial technique based on injection moulding has been finished successfully. Using this method for the ternary system Fe_2O_3-TiO_2-Al_2O_3 gave proof of the reliability of the technique. [1]

In a second example, dielectric properties have been studied in the ternary system CuO-TiO_2-CaO. Samples were made comparably by injection moulding, cutting, mixing and sintering. A phase analysis of the complete phase diagram at 950 °C has been conducted. Five two-phase regions, five three-phase regions and only one ternary compound $CaCu_3Ti_4O_{12}$ (CCTO) were identified. One of these regions shows giant dielectric constants with ε′ over 10,000. A second one has similar properties depending on the preparation conditions and sintering temperatures. The influence of these factors on the dielectric properties and on the microstructure evolution of the resulting solids was determined. A full publication of the results is under way. [2]

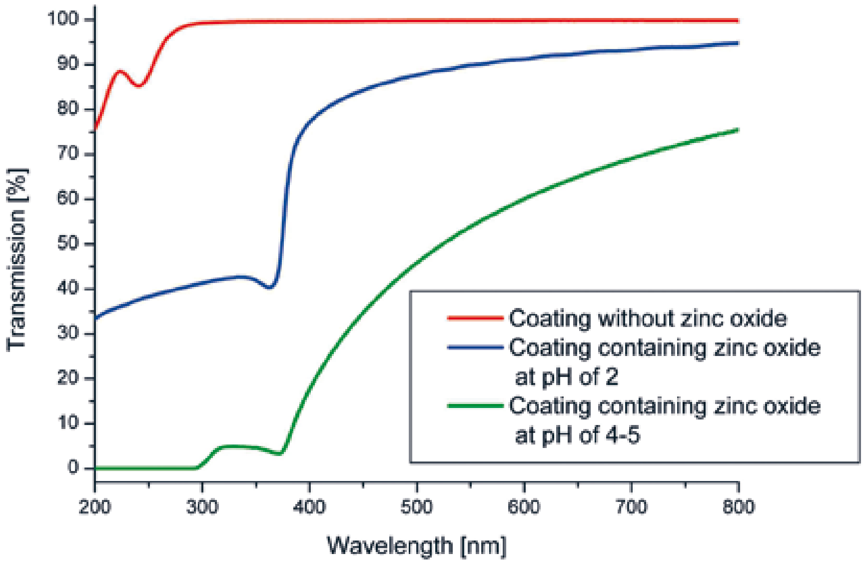


Figure 3: UV-Transmission of coatings on domestic wood.

[1] S. Ren, W. Kochanek, H. Bolz, M. Wittmar, I. Grobelsek, M. Veith, J. Eur. Ceram. Soc., 2008, 28, 3005-3010.

[2] S. Ren, M. Wittmar, H. Bolz, M. Veith, Giant dielectric constant response of the composites in ternary system CuO-TiO_2-CaO, to be published.

Binder and additive development

In 2008 the activities in the AiF-project for the improvement of domestic wood for outdoor applications were continued and successfully completed with the end of the year. In the last project year the UV- and weathering protection could be optimized to a nearly complete resistant outdoor applicable and long lasting system (see figure 3).

In cooperation with our partners BAM and University of Göttingen – responsible for the development of two different impregnation systems – a complete protection for fast growing domestic wood was achieved. The impregnation developed by the partners reduced dimension changes. These characteristics are necessary for the maintenance of the function of the protection coating. Large dimension changes increase the risk of the formation of cracks and the loss of the protection function. Together with the two other partners - Deutsche Gesellschaft für Holzforschung and ift Rosenheim – was decided to manufacture an entire window frame as demonstrator. The several wooden parts of the frame were impregnated by BAM or by University of Göttingen and coated at INM (see figure 4).

The window was assembled at ift Rosenheim. There, the complete window is exposed to outdoor weathering.

The second publicly funded project resides in the European EraSME programm – in Germany founded by the national partner BMWi (UNACON). The consortium consists of three research institutes – BAM, University of Innsbruck and INM – and 9 SME from Germany, Austria, Belgium, Spain, Sweden and Iceland. The industrial partners are producers of architectural concrete masonry units and expect an innovative pulse for the building material industry by the implementation of innovative aspects in their products. The aim of the project is the development of a hydrophobic, dirt-repellant nanoadditive. The additive will be added directly to the liquid concrete mixture and should be incorporated in the cement stone matrix via a pozzolanic bond. In addition to the improved durability of the characteristics, a simplification of the manufacturing process is expected. In the first project year one of the largest challenges of the project was overcome. By a masking of the reactive groups of the additive a homogeneous distribution of the additive in concrete mixtures could be realized. The influence on the strength development was lessened. The cured stones exhibited excellent hydrophobic behaviour.

Outlook

In 2009 the continuation of the three major projects MULTIPROTECT, PriMeBits and UNACON is planned. MULTIPROTECT will be finalized in this period. The activities in the combinatorial development of material with giant dielectric constants will be continued.

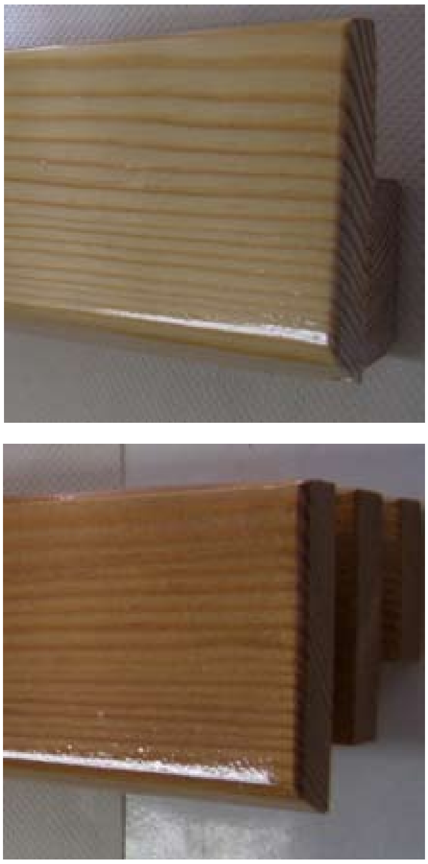


Figure 4: Impregnated and coated wooden window panel.

Nanotoxicity / Nanotoxizität (since 05/2008)

Dr. Annette Kraegeloh

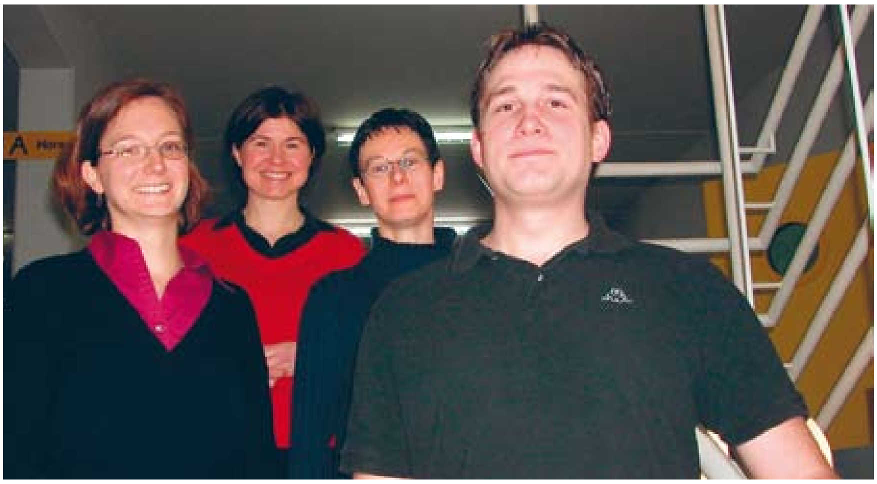
Die Juniorforschungsgruppe Nanotoxizität wurde zum 01. Mai 2008 eingerichtet. Sie untersucht die Wechselwirkungen zwischen lebenden Zellen und nanoskaligen Materialien unter dem Aspekt, die potentielle Toxizität technisch erzeugter Nanopartikel aufzuklären. Da bereits heute technisch hergestellte nanoskalige Materialien in einer Vielzahl von Produkten eingesetzt werden und das Ausmaß und die Vielfältigkeit, in der diese Materialien Einsatz finden, kontinuierlich ansteigt, wird der Mensch in zunehmenden Maße synthetisch hergestellten Nanopartikeln ausgesetzt sein. Deshalb ist die Verträglichkeit von Nanomaterialien nicht nur von wissenschaftlicher, sondern auch von gesellschaftlicher Relevanz.

Unser Ziel ist es, Mechanismen aufzuklären, die eine Toxizität dieser Nanomaterialien gegenüber Zellen und Organismen verursachen. Unsere Untersuchungen führen wir in vitro an humanen und tierischen Zellen durch. Im Mittelpunkt stehen relevante Phänomene wie die Adhäsion einzelner Partikel an der Zelloberfläche, die Partikelaufnahme, der intrazelluläre Partikeltransport sowie der intrazelluläre Verbleib der Partikel. Zur Erfassung struktureller Phänomene werden verschiedene mikroskopische Methoden, vor allem die STED (STimulated Emission Depletion) Mikroskopie, eingesetzt. Ergänzend werden Untersuchungen durchgeführt, die Aussagen über den physiologischen Status der Zellen zulassen. Um ableiten zu können, auf welche Eigenschaften der Nanopartikel (Material, Größe, Oberflächenpotenzial, Modifikation) eine potentiell toxische Wirkung gegenüber Zellen zurückzuführen ist, ist es notwendig, diese Eigenschaften auch unter den experimentellen Bedingungen, die der physiologischen Umgebung der Zellen entsprechen, genau zu kennen. Für unsere Untersuchungen setzen wir unter anderem Modellpartikel ein, die es erlauben, zelluläre Strukturen und Nanopartikel gleichzeitig detektierbar, bzw. sichtbar zu machen. Der vielschichtige Ansatz erfordert eine Kombination biologischer, chemischer und physikalischer Kenntnisse und Methoden.

Eines unserer wichtigsten Arbeitsmittel ist die STED-Mikroskopie, die im Vergleich zur herkömmlichen Konfokalmikroskopie eine deutlich höhere Auflösung ermöglicht. Das Auflösungsvermögen des am INM vorhandenen STED-Systems (Anschaffung gefördert durch das BMBF) der Firma Leica liegt unterhalb 90 nm. Für unsere STED-Untersuchungen setzen wir fluoreszenzmarkierte Nanopartikel ein. Bei der Herstellung der Partikel aus amorphem Siliziumdioxid wurde der Farbstoff kovalent in die Matrix inkorporiert.

Unsere mikroskopischen Untersuchungen werden ergänzt durch Untersuchungen zum physiologischen Status der Zellen. Wichtige Fragestellungen adressieren die Rückführung der experimentellen Ergebnisse auf die Eigenschaften der Nanopartikel und auf dosis-, zeit- und applikationsbedingte Effekte.

Langfristig möchten wir neben der STED-Mikroskopie auch weitere mikroskopische Methoden einsetzen, darunter Elektronenmikroskopie und in Zusammenarbeit mit dem Programmbereich Nanotribologie Rasterkraftmikroskopie. Die Modellierung anhand der experimentellen Daten wird dazu beitragen, Vorhersagen bezüglich der Wahrscheinlichkeit der Aufnahme von Nanopartikeln oder der intrazellulären Transportwege zu treffen. Die Modellierung wird in Zusammenarbeit mit dem im Aufbau befindlichen Forschungsschwerpunkt zur physikalischen Modellierung und Simulation erfolgen.



The junior research group Nanotoxicity was established in May 2008. The group investigates the interactions between living cells and nanoscaled materials in order to elucidate the potential toxicity of engineered nanoparticles. Engineered nanoparticles and nanoscaled materials are already used in a multitude of products. The extent and diversity of applications based on such materials is rising continuously. For this reason, human beings are increasingly exposed to synthetically produced nanoparticles. Hence, the compatibility of nanomaterials is not only of scientific, but also of societal relevance.
Our aim is to elucidate mechanisms causing toxicity of nanomaterials on cells and organisms. We carry out *in vitro* experiments on human and animal cells, concentrating on relevant phenomena like the adhesion of single particles to cellular surfaces, nanoparticle uptake, intracellular transport and fate of nanoparticles. To detect structural phenomena, various microscopical methods are applied, including STED microscopy (STimulated Emission Depletion – explained in greater detail below). Complementary investigations are carried out to probe the physiological state of the cells.

In order to deduce which particle properties – e.g. material composition, size, surface potential or modification - potentially cause toxic effects, these properties have to be defined accurately. The experimental conditions which resemble the physiological environment of the cells have to be accounted for.

For our investigations we use model particles, which allow for the simultaneous detection or visualization of nanoparticles as well as cellular structures. This complex approach requires a combination of biological, chemical and physical skills and methods.

STED microscopy

The acquisition of a STED microscope, a high-resolution confocal microscope, was made possible by the funding of the project “MeNatox – Mechanismen der Nanotoxizität durch Nutzung mikroskopischer Methoden (mechanisms of nanotoxicity by use of microscopical methods)” of the Federal Ministry of Education and Research within the programme “WING – Werkstoffinnovationen in Industrie und Gesellschaft”. At the end of 2008, the STED system was installed at INM and put into operation. Confocal microscopy is an optical method for the three-dimensional, not surface bound imaging of cells and cellular structures, even suitable to observe dynamical processes in living cells. The lateral resolution of the conventional technique is subjected to the diffraction (Abbe) limit and is limited to about 200-250 nm. Until recently, this technique was not suitable to access the scale between 1-100 nm, relevant for representation of biological and artificial nanostructures.

In contrast, the recently developed STED technique overcomes this diffraction limit. The acquired STED-microscope (Leica Microsystems) delivers a resolution below 90 nm, facilitating spatially resolved imaging of nanoparticles and nanoscaled cellular components, such as membrane vesicles or protein filaments. Hence, STED microscopy is similarly suited to visualize the agglomeration of nanoparticles within the environment of biological structures. It will also be used to characterize the interaction of cellular components affected by the presence of nanoparticles.

Preparation of fluorescently labelled model particles

Establishment of the STED-technique necessitates the application of special fluorescently labelled nanoparticles. For this reason, we synthesized nanoparticles composed of amorphous silica. During synthesis, the organic fluorescent dye was incorporated into the particle matrix. The dye was attached covalently and distributed evenly over the particle volume. The size of the nanoparticles, as measured by STED microscopy, compared well with the size determined by transmission electron microscopy. At present, we are working towards establishing a further method suitable for the preparation of various types of labelled core-shell nanoparticles. This method allows for the introduction of label either into the core or the shell. Various sizes of nanoparticles will be used to define the resolution limit of the STED technique in a biological environment.

Outlook

The primary objective of the group is to investigate the potential toxicity of various nanoparticles in human cells. In the short to medium term, we will employ various types of human cells (for example epithelial cells of the lung, skin cells), which are relevant to potential exposition pathways. Important questions are material, surface or size dependent effects of the nanoparticles. We will study the toxic effects as a function of dose, time and mode of application. We will perform assays suited to enable conclusions on the physiological state of the cells; for instance cellular activity, viability or oxida-

Figure 1: STED-system used for the imaging of nanoparticles interacting with biological structures.

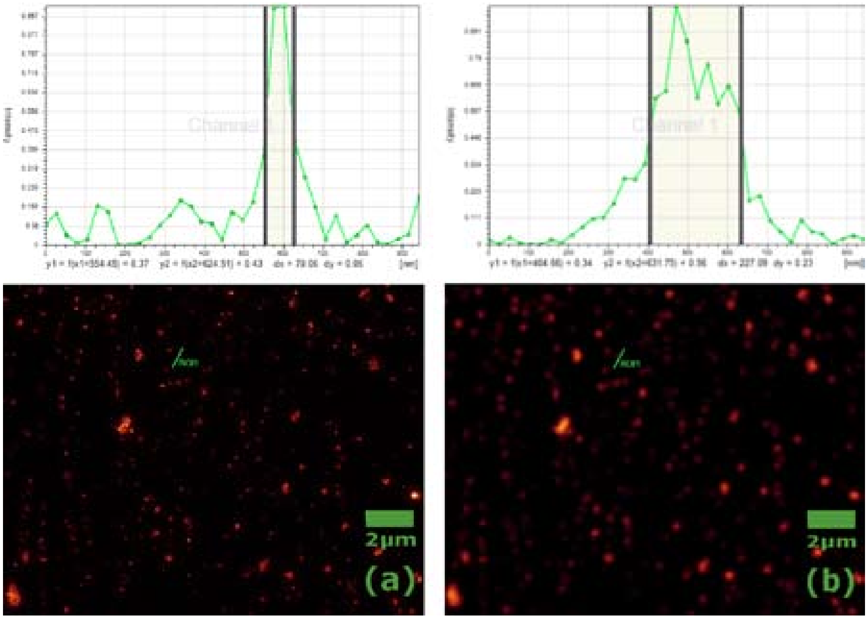


Figure 2: Enhanced resolution of STED-microscopy: a) STED-image of fluorescently labelled silica nanoparticles in comparison to b) a conventional confocal image. Fluorescence profiles (top) along the indicated region of interest (ROI1) were used to determine the size of single nanoparticles (70 nm [STED], compared to 228 nm [confocal]).

tive stress. Additional tests will allow for more detailed information on the effects of nanoparticles (apoptosis, genotoxicity, activation of distinct proteins or signalling factors). Finally, the results will be correlated to the microscopical analysis.

In the long term we plan to apply further microscopical methods in our investigations. Besides optical methods (dark field or reflection), which are particularly suited for investigations on living objects, we also plan to use electron microscopy, for example environmental scanning electron microscopy (ESEM). In addition, we intend to investigate the interactions of single nanoparticles by use of atomic force microscopy (AFM). For example, this approach will enable characterisation of the nanomechanical forces participating for example in particle adhesion or penetration. The latter investigations will be performed in tight cooperation with the program division Nanotribology. The generated data regarding adhesion or mechanical interactions of particles and cells will subsequently be linked with theoretical models in order to predict the probability of nanoparticle uptake or intracellular interaction patterns. Modeling will be performed in collaboration with the program division Modeling and Simulation which is under development. The group already cooperates with partners from Saarland University including Homburg campus regarding intracellular transport of nanoparticles and physical modeling of such phenomena (Pharmaceutical Biology, Theoretical Physics) and effects of nanoparticles on neuronal cells and on the propagation of action potentials (Cellular Neurophysiology).

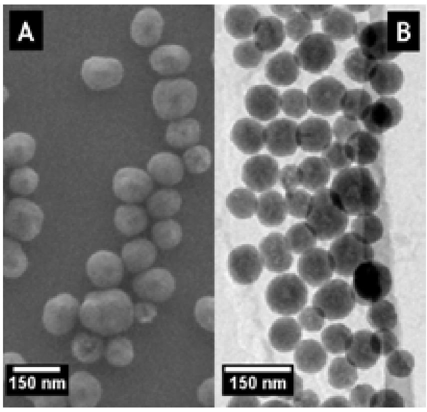


Figure 3: Electron micrographs of fluorescently labelled silica nanoparticles: a) SEM, b) TEM.

Nanotribology / Nanotribologie (since 06/2008)

Prof. Dr. Roland Bennewitz

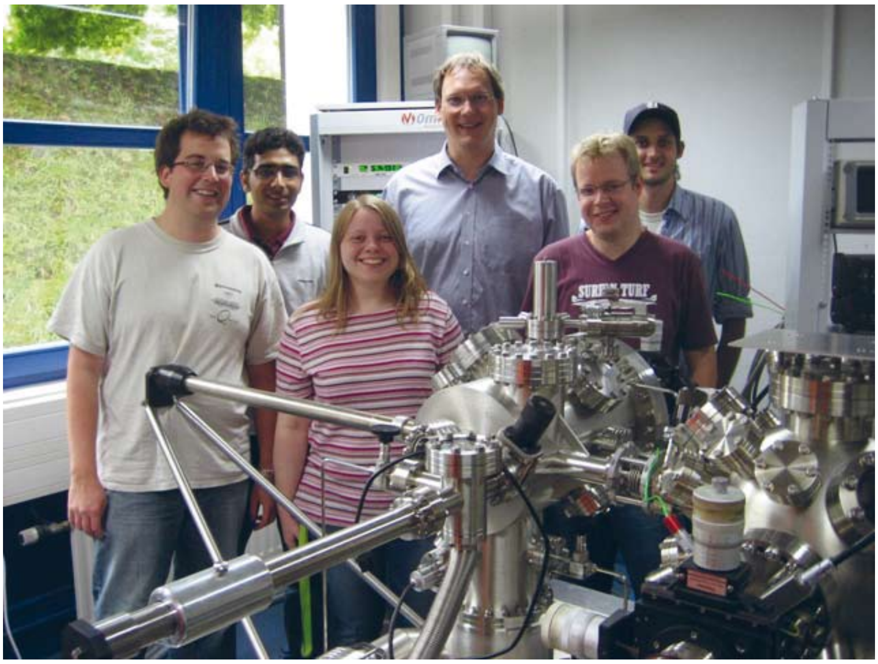
Der Programmbereich Nanotribologie hat sich zum Ziel gesetzt, die mechanischen Phänomene Reibung, Abrieb und Verformbarkeit aus einer mikroskopischen Perspektive heraus zu verstehen. Dazu führen wir modernste nanomechanische Experimente durch, meist mittels Rasterkraftmikroskopie (Atomic Force Microscope, AFM) und entwickeln neue Experimente, die die Lücke zwischen Nanotribologie und makroskopischen Untersuchungen schließen sollen. In den Experimenten bestimmen wir die Abhängigkeit der Reibungskräfte und der Energiedissipation von Parametern wie zum Beispiel Auflagekraft, Geschwindigkeit oder Filmdicke bei festen Schmierstoffen. Die hochauflösende Abbildung mit dem AFM trägt weiterhin Informationen über die Oberflächenstruktur (oftmals mit atomarer Auflösung) und über die ersten Stufen von Abrieb und Verformung bei. Wir berichten über unsere Ergebnisse auf internationalen Konferenzen und in internationalen wissenschaftlichen Zeitschriften.

Aufbauend auf seinem grundlagenwissenschaftlichen Ansatz und seinem methodischen Schwerpunkt arbeitet unser Programmbereich mit allen Bereichen des INM zusammen. Typische Beispiele von gemeinsamen Vorhaben betreffen die tribologischen Eigenschaften mikrostrukturierter Oberflächen (Programmbereich Funktionelle Oberflächen) oder die Kratzfestigkeit in nanopartikelverstärkten Polymerbeschichtungen (Programmbereich Nanomere). In diesen Vorhaben und in zukünftigen Projekten mit externen Partnern möchten wir die Ergebnisse der Nanotribologie für die wissensbasierte Entwicklung neuer Materialien mit erwünschten mechanischen Eigenschaften fruchtbar machen.

Der Programmbereich Nanotribologie wurde im Juni 2008 am INM eingerichtet, als Roland Bennewitz mit einem Teil seiner Arbeitsgruppe von der McGill Universität in Montreal, Kanada, an das INM wechselte. Die wichtigsten Aktivitäten im Jahr 2008 waren die Installation der Labore, die Erzielung von Messergebnissen auf dem letzten Stand der Technik für einfache Materialien und die Diskussion möglicher gemeinsamer wissenschaftlicher Projekte mit den Programmbereichen des INM und Forschungsgruppen an der Universität des Saarlandes.

Der Aufbau der folgenden experimentellen Projekte ist im Wesentlichen abgeschlossen:

- In einem Projekt über atomare Reibung auf dünnen Filmen haben wir eine sehr erfolgreiche Studie über Graphen-Filme auf Siliziumkarbid abgeschlossen. Zwei AFM-Experimente, eines im Vakuum und eines unter elektrochemischer Kontrolle, sind installiert und erlauben Reibungsphänomene auf atomarer Skala zu studieren.
- Die ersten Stufen der mechanischen Verformung werden in einem AFM-Experiment untersucht, das sowohl die Einbringung einzelner Versetzungen während des Eindrückens der Oberfläche beobachtet als auch die Abbildung



der resultierenden Oberflächenstruktur erlaubt.

- Über das Studium einzelner mikroskopischer Kontakte im AFM hinaus weist ein neues Experiment, in dem wir das Reibungsverhalten mikrostrukurierter Oberflächen mit tausenden regelmäßig angeordneten Kontakten mittels Lichtstreuung untersuchen.

Für die nächste Zukunft ergeben sich für den Programmbereich Nanotribologie zwei wesentliche Herausforderungen. Zum einen wollen wir bald wieder durch herausragende experimentelle Ergebnisse zum fundamentalen Verständnis mechanischer Phänomene auf kleinster Skala beitragen. Zum anderen wollen wir in gemeinsamen Projekten mit den Programmbereichen des INM die Ergebnisse der Nanotribologie für die Lösung aktueller und angewandter materialwissenschaftlicher Probleme nutzbar machen.

The program division Nanotribology strives to understand mechanical processes like friction, wear, and plasticity from a microscopic perspective. Towards this goal we perform cutting-edge nanomechanical experiments, mostly based on Atomic Force Microscopy (AFM), and develop novel experimental techniques which bridge the gap between nanomechanical and macroscopic studies. The experiments reveal the dependence of force fluctuations and of energy dissipation on parameters like normal load, sliding velocity, or film thickness for solid lubricants. High-resolution AFM imaging supports the mechanical experiments by revealing the surface structure, often with atomic resolution, and detecting the onset of wear and plasticity. The results of our work are presented at international conferences and in international scientific journals.

With its fundamental approach and methodological focus, our program division collaborates on projects across the INM. Typical areas of collaborative research include the tribological properties of micro-structured surfaces (program division Functional Surfaces) or the wear processes in nanoparticle-enforced polymer coatings (program division Nanomers). Within these collaborations and in future projects with external partners, we want to make the fundamental results of nanotribology useful for the rational design of new materials with dedicated mechanical functions.

The program division Nanotribology was established at the INM in June 2008, when Roland Bennewitz moved here from McGill University in Montreal, Canada, together with a part of his research group. The activities in 2008 have been focused on installing the laboratory, obtaining state-of-the-art experimental results on simple model systems, and discussing scientific interaction with the program divisions at the INM and research groups at the University of the Saarland.

Atomic friction

Our group has a world-wide reputation for advanced atomic friction experiments. With the objective to extend this class of experiments from simple model systems to more relevant materials we perform atomic friction experiments on ultrathin lubricating films. Furthermore, we have started to study friction on surfaces under electrochemical control in order to explore friction and wear under corrosive conditions. In 2007 we have transferred an AFM operating in ultra-high vacuum from McGill University to the INM and added a vacuum chamber for thin film growth to the system. A new Post Doc, Dr. Nitya Nand Gosvami, has learned to operate the instrument during an extended stay at McGill University and has already recorded cutting-edge atomic friction results at the INM, see Figure 1. A PhD student from McGill, Tobin Filleter, has visited the INM for several months, helped to install the AFM, and finalized a study on single and bi-layer graphene on silicon carbide which is now being published (Appl. Phys. Lett. 93, 133117 (2008) and Phys. Rev. Lett, in print 2009). The results show that a single layer of graphene greatly reduces friction and that a bi-layer of graphene outperforms even graphite as a solid lubricant due to reduced adhesion. Furthermore, in collaboration with a photoelectron spectroscopy group at the University of Berkeley we could explain an intriguing difference in friction between single and bi-layer graphene as the result of a difference in electron-phonon coupling. Funding for the continuation of these activities has been granted by the Deutsche Forschungsgemeinschaft within the European Science Foundation project FANAS.

We also have purchased and installed a new AFM operating in liquid environment. We are in the process of thoroughly testing the instrument by recording friction data at various liquid-solid interfaces. A new PhD student, Florian Hausen, with a strong background in electrochemistry has equipped the laboratory with the tools to prepare and manipulate the surface of atomically clean and flat gold surfaces. This is our first test bed for friction in oxidizing and reducing environment enabling us to compare the friction of the samples which we have already characterized in our vacuum experiments. In this project we continue to collaborate with McGill University where a home-built AFM has already demonstrated reversible switching between different oxidation states as revealed by atomic friction patterns.

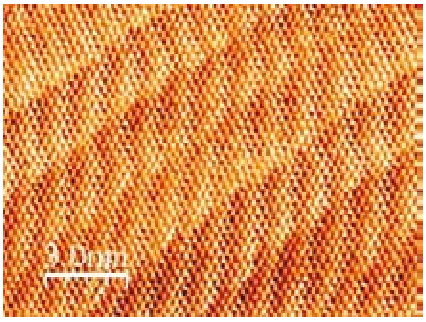


Figure 1: Map of lateral forces recorded on a perfectly flat gold surface in ultra-high vacuum. The atomic structure of the gold surface is revealed through the atomic stick-slip mechanism. Furthermore, the so-called herring-bone reconstruction of the surface modulates the friction force in parallel stripes.

Plasticity and wear

The permanent deformation of crystalline materials under stress occurs through the nucleation, multiplication, and movement of extended structural defects in the crystal called dislocations. Understanding plasticity on the microscopic scale requires experiments which bridge the gap between continuum mechanics and the properties of individual dislocations. We contribute to this field through AFM-based indentation experiments combined with high-resolution imaging of the resulting surface structure. Our method allows us to the incipient stages of plasticity, i.e the nucleation of individual dislocations.

Our research activities on plasticity are the subject of the PhD work of a student at McGill University, Philip Egberts, who has now joined the INM. In the second half of 2008 we have installed new soft- and hardware for controlling the vacuum AFM which allows us to program complex experimental procedures. Furthermore, we have published results which show how a multi-mode use of the AFM can reveal the electric charges attached to individual dislocations (Nanotechnology, in print 2009). In this study we show that even for electrically insulating materials a bias voltage applied to the back of a sample can reveal the charge of single dislocations imaged by the AFM. The charge also helps to identify the position of edge dislocations at the surface which have no topographic signature. The edge character of the dislocation could then be confirmed by imaging the surface with atomic resolution. We have also attempted to extend the project on microscopic plasticity to a light metal, aluminium, which is of high interest in modern automotive manufacturing.

With our project on plasticity we are associate partners in a collaborative research project of Canadian, Indian, and US groups which is supported by the Canadian funding organization NSERC. The goal of the collaboration is a description of the hardness of nanocrystalline materials.

Friction of micro-structured surfaces

Friction between two sliding surfaces is critically determined by the roughness of the surfaces. A multitude of microscopic contacts is continuously formed, deformed, and ruptured in the course of sliding. Our activities using AFM explore the friction of single microscopic contacts. In a complementary approach, we study the friction of surfaces with thousands of microscopic contacts. However, these contacts are well-ordered and well-defined in shape to allow for an easier interpretation of the results. The novel experiment allows to study strain across the field of microscopic contacts by means of light scattering. A new PhD student, Katrin Brörmann, has performed experiments using the so-called Gecko surfaces prepared by the program division Functional Surfaces. These surface materials exhibit regular arrays of elastomer pillars with enhanced adhesion properties. In our experiment they are slid horizontally across a glass surfaces. We find that the whole ensemble of microscopic contacts undergoes an interesting pre-sliding phase and finally enters a so-called stick-slip regime of sliding motion in which the contacts regularly switch between sticking to certain surface positions and slipping to the next one. The periodicity of this stick-slip motion is determined by the periodicity of the engineered microstructure (see Figure 2). This finding may have interesting applications in sliding rubber-glass contacts which exhibit noisy acoustic emissions. In an extension of the experiment we have observed the sliding contact array directly in an optical microscope and started to analyze the recorded movies by means of image correlation methods.

Outlook

After the successful installation of the Nanotribology laboratories at the INM we have two major goals for the near future. On the one hand we hope to further contribute to the fundamental microscopic understanding of friction, plasticity, and wear. Atomic friction on ultrathin films will be explored on new materials and in different environments, in particular for iron halides on iron and for graphene on silicon carbide in liquid environments. A new PhD student, Christian Held, will support the activities from February 2009 on. Regarding the microscopic plasticity of single-crystal materials we plan to study the temporal statistics of dislocation nucleation. The tribology of patterned elastomer samples will be tested as a function of micro-structure, materials properties, and liquid lubricants in the contact.

On the other hand we want to establish well-defined collaborative projects. Examples include a fundamental study of scratch-resistant materials together with the program division Nanomers, for which Dr. Harald Tlatlik will join the INM in May 2009. In collaboration with Prof. Birringer in the Department of Physics of the University of the Saarland we develop nanometer-scale metal particles held in an oxide matrix for mechanical testing at their surface. Mechanical testing of Functional Surfaces and CVD films will be pursued in collaboration with the respective program divisions. For the latter we also want to broaden our instrumental spectrum towards *in-situ* testing in a Scanning Electron Microscope.

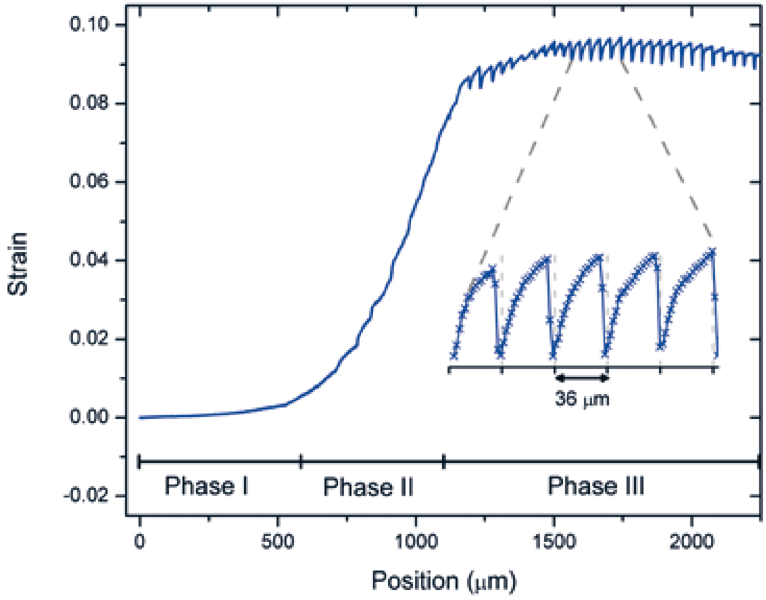


Figure 2: Development of lateral strain as the Gecko surface is pulled over a glass surface. Three phases of compression can be identified: Initial shearing of the elastomer backing without strain (Phase I), pres-sliding compression of the micro-structured surface (Phase II), and finally sliding in a stick-slip fashion (Phase III). The inset reveals that the periodicity of the stick-slip motion is defined by the pitch of the compressed microstructure, i.e. 36 µm.

Application Centre NMO/Chemical Engineering / Anwendungszentrum NMO/Verfahrenstechnik

Dr. Andreas Altherr

Seit der Gründung des Anwendungszentrums ist dieses das zentrale Element des Instituts für den Technologietransfer in die Industrie. Im Jahr 2008 wurden insgesamt elf Projekte, darunter ein eigenständiges Großprojekt zur industriellen Umsetzung von Laborentwicklungen durchgeführt. Im Rahmen von internen Kooperationen übernahm das NMO im Rahmen von zwei weiteren Projekten die Durchführung der Technologiephase bzw. deren Vorbereitung. Die Projekte beinhalteten das Up-Scaling von Rezepturen, die Entwicklung nasschemischer Applikationsverfahren einschließlich geeigneter Applikationswerkzeuge, die Pilotserienfertigung sowie intensive Arbeiten zur chemischen Analytik an Beschichtungswerkstoffen. Innerhalb der Chemischen Verfahrenstechnik wurde eine eigenständige, von Industrieprojekten unabhängige Grundlagenforschung betrieben. Das Thema der 2007 abgeschlossenen Diplomarbeit zur elektrochemischen Abscheidung von oxidischen Materialien wurde im Rahmen einer Promotionsarbeit weiterverfolgt, die auch über das Jahr 2008 hinaus weitergeführt wird.

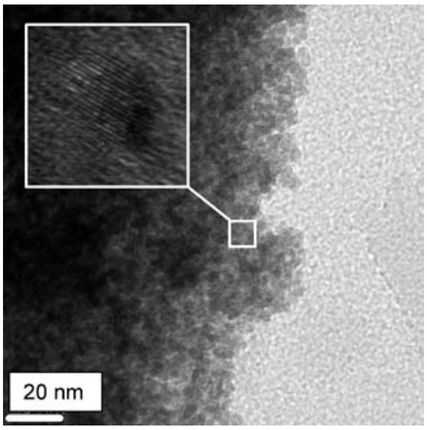


Figure 1: TEM-study of nano-scaled indium-tin hydoxide particles synthesized by the electro chemical route.

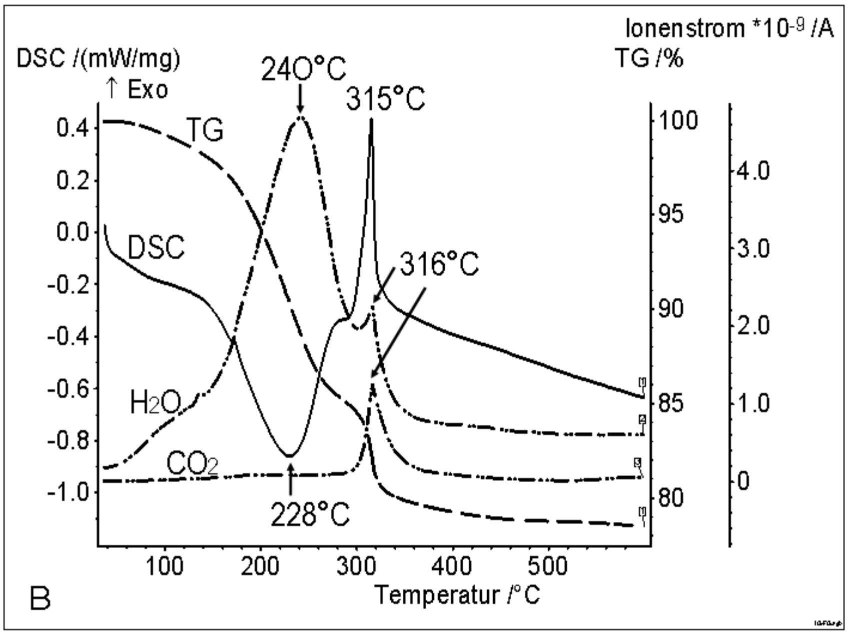


Figure 2: DSC diagram coupled with TG and MS-analysis of indium-tin hydroxid.

Since its foundation, the NMO Application Center has been the central division of the institute concerned with the transformation of lab-scaled processes to industrial applications. To attain this goal, there is an intense interaction between the INM research groups and NMO, as well as with industrial project partners.

The topics of NMO focus on the industrial application and technology transfer of wet coating technologies developed in the basic research groups of INM. NMO also executes independent basic research in the field of new synthesis routes of nano materials. The projects of NMO/ Chemical Engineering were executed by six employees.

In the year 2008, NMO was involved in 13 projects, including:

- Nanomere coatings with Easy to Clean (ETC) properties were applied in an industry project by dip-coating techniques and curing to test the performance of the coating material in real conditions.
- As support for small and medium sized enterprises, NMO rented out technical equipment for materials synthesis lacquers applications and materials analysis.
- Synthesis and application of anti-microbial Nanomere coating material on polymers in a test environment.
- In cooperation with the PD Glass and Optics, tests on wet technologies for the application of new coating materials were performed.
- The technology transfer step of Nanomere industry projects was executed in two projects by the Chemical Engineering subgroup.
- As a service group, the Chemical Engineering subgroup supports all program divisions of INM.

The Education and advanced training of scientists was focused to the basic research:

After a diploma thesis in 2007, the research activity on the electrochemical deposition process for nano scaled metal oxide particles was continued as a doctoral thesis. Findings of synthesis and characterization of the ITO obtained were published in the Journal of Nanoscience and Nanotechnology.

Research activities in 2008 were focused on ZnO and its behaviour under varying process parameters. To control the reaction atmosphere and to enhance the reproducibility of process parameters, a new experimental set-up was designed. The main focus was on process management, parameter variation and their influences on the deposition process itself.

Structure formation at small scales/ Strukturbildung auf kleinen Skalen

Dr. Tobias Kraus

Die Juniorforschungsgruppe Strukturbildung erzeugt Überstrukturen aus Partikeln. Unser Ziel ist es, Materialien mit definierten Nanostrukturen durch die kontrollierte Abscheidung von Nanopartikeln herzustellen. Die Gruppe konzentriert sich auf Anordnungsmechanismen: Wir analysieren das Wachstum geordneter Überstrukturen aus ungeordneten Kolloiden und entwickeln Prozesse für ihre zuverlässige Abscheidung in makroskopischen Mengen. Diese Prozesse sollen zu tradionellen Methoden der Nassbeschichtung kompatibel und auf große Flächen übertragbar sein.

Um die mikroskopischen Vorgänge der Strukturbildung zu verstehen, beobachten wir Partikelabscheidung und Aggregation sowohl direkt als auch mit spektroskopischen Methoden. Wir analysieren die Dynamik der Wachstumsvorgänge und den Zusammenhang mit den entstehenden Strukturen. Mit diesem Verständnis können Parameter und Methoden für die Anordnung bestimmter Überstrukturen ausgewählt werden. Relevant dabei sind Interaktionen zwischen Partikeln, zwischen Partikeln und Lösemitteln sowie zwischen Partikeln und Grenzflächen; all diese Interaktionen können in realen Abscheidungsprozessen gleichzeitig auftreten. Ordnung kann von jeder einzelnen abhängen, und selbst wenn die Interaktionen der Partikel der eigentliche Ursprung von Ordnung sind, so hängen doch Partikelbeweglichkeit, Energielandschaft und deren zeitliche Entwicklung vom Gesamtprozess ab.

Die gewonnenen Erkenntnisse sollen zur Synthese neuer funktionaler Materialen angewandt werden. Geeignete Grundmaterialien werden in Nanopartikel zerteilt, aus denen dann ein strukturiertes Komposit hergestellt wird (Figure 1). Ein solches Material kann beispielsweise elektromagnetisch aktiv sein und kollektive Bandlücken aufweisen, es kann Energie konzentrieren, übertragen oder umwandeln oder als Metamaterial wirken, es kann komplexes mechanisches Verhalten aufweisen oder ungewöhnliche thermische Eigenschaften haben, falls es komplexe Kontakte und Flüssigkeitsgrenzflächen bildet.

Zentrale Themen der Gruppe sind

- die Synthese von Nanopartikeln mit hohen Ansprüchen an Monodispersität, chemische Identität und Charakterisierung;
- die Untersuchung des kollektiven Partikelverhaltens in Hinblick auf Stabilität und Kinetik;
- die Anordnung von Partikeln in spezialisierten, hier entwickelten Aufbauten mit umfangreichen Kontroll- und Analysemöglichkeiten.

Während die derzeit hergestellten Partikelüberstrukturen relativ einfacher Struktur sind (beispielsweise hexagonal dichteste Packungen), sollen in Zukunft komplexere Strukturen durch die Mischung verschiedener Partikel hergestellt werden. Reizvoll ist auch der Übergang von multimodalen zu polydispersen Partikelsystemen, wie sie in der Praxis oft vorkommen.

The junior research group Structure formation prepares particle superstructures. Our goal is to form rationally nanostructured materials through the controlled deposition of nanoparticles. The research is focused on order formation mechanisms: we analyze the growth of regular superstructures from disordered colloids and develop processes for their reliable deposition in macroscopic quantities. The processes are to be compatible with traditional wet coating methods and can be scaled up to large areas.

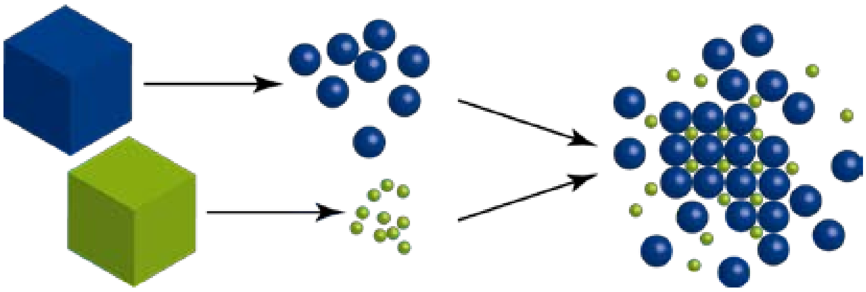


Figure 1: The concept of particle-based materials synthesis: materials are dispersed into nanoparticles which interact and assemble into a superstructure.

To gain microscopic insight into structure formation, particle deposition and aggregation are observed both in real space and using spectroscopic methods. We analyze growth mechanisms and correlate their dynamics with the resulting particle superstructures. General particle processing requirements are derived to help choose parameters and methods for the deposition of specific superstructures. The research covers particle-particle, particle-solvent and particle-interface interactions, all of which occur simultaneously in realistic deposition processes. Order can depend on any of them, and even if particle-particle interactions are the ultimate source of order, particle mobility, the shape of the free energy landscape and its time evolution are dependent on the overall process.

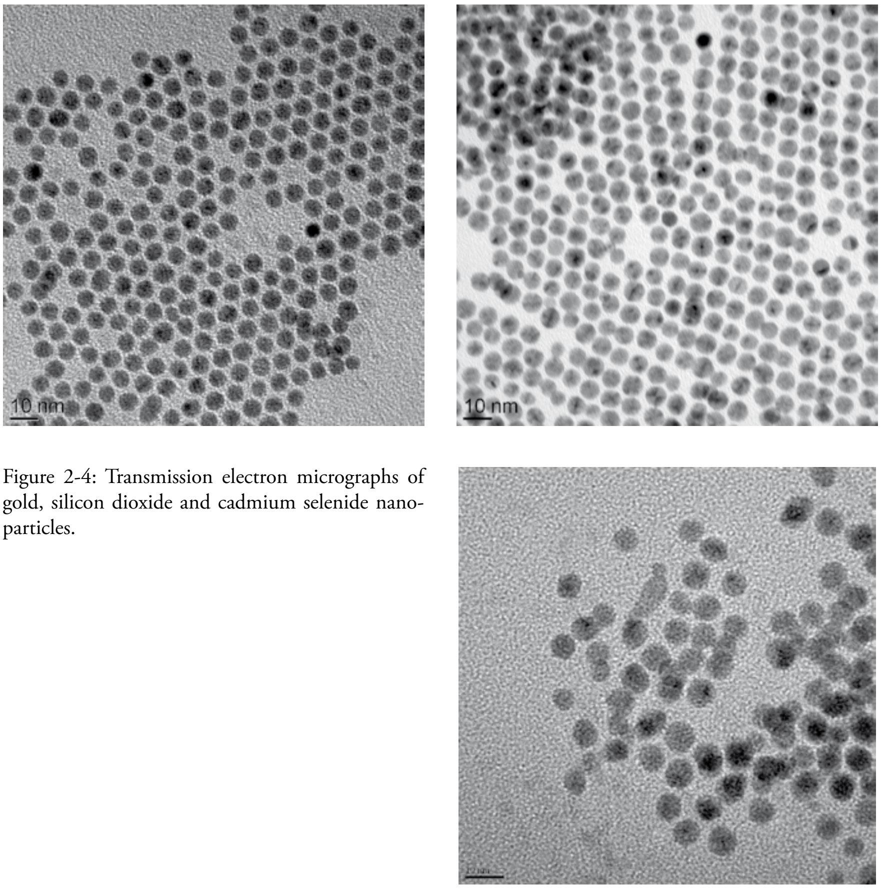


Figure 2-4: Transmission electron micrographs of gold, silicon dioxide and cadmium selenide nanoparticles.

This gained knowledge will be applied in the synthesis of new functional materials. The constituent materials are broken down, cut up into nanoparticles, which are then assembled into a structured composite material (Figure 1). Such a material can be electromagnetically active and exhibit collective bandgaps, it can concentrate, transfer and convert energy and act as a metamaterial, it can exhibit complex mechanical behaviour or have unusual thermal properties if it forms complex contacts and interfaces with liquids.

Nanoparticle synthesis

Our work is based on highly monodispersed, well-defined particles in the size range of 1 to 100 nm with known, reproducible interaction potentials. We have chosen three material systems — gold (Figure 2), silica (Figure 3), and cadmium selenide (Figure 4) — from which we synthesize spherical nanoparticles that are dispersed in various organic solvents and characterize them for size, concentration, residual charges, optical properties, crystal structure and surface state. Other particles, such as silver and polymer particles or non-spherical particles, are used less frequently and synthesized or obtained from commercial sources. The particle surfaces are grafted with self-assembled monolayers to tune interparticle potentials and particle spacing.

Particle behavior

Forming regular particle superstructures requires a stable colloid that is destabilized in a controlled fashion at the appropriate moment. Thus, colloidal stability, which is dependent on concentration, temperature and the presence of other solutes, is critical for the design of a particle deposition process. Besides, the previously mentioned factors affecting colloidal stability, geometrical confinement occurs frequently in processes such as dip-coating or spin-coating, where it often has a profound effect on the particles' stability and aggregation behaviour.

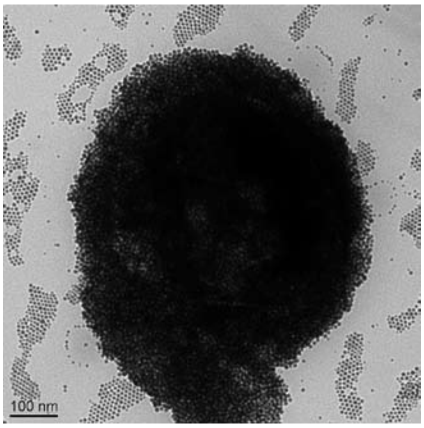


Figure 5: Amorphous agglomerate of 6-nm-Gold nanoparticles.

We investigate the dependence of colloidal dynamics on particle composition, surface coating, temperature, concentration and other variables using dynamic light scattering, optical spectroscopy and small-angle x-ray scattering. Particle deposition is observed where possible. In addition, we create model systems that provide confinement and high particle concentrations in a matrix where it can be easily observed and analyzed.

Beyond colloidal stability, the precise nature of the flocculation and agglomeration process is analyzed. Depending on the process, particle agglomerates can be amorphous (Figure 5) or crystalline (Figure 6), dense or fractal with large voids, mobile or rather static.

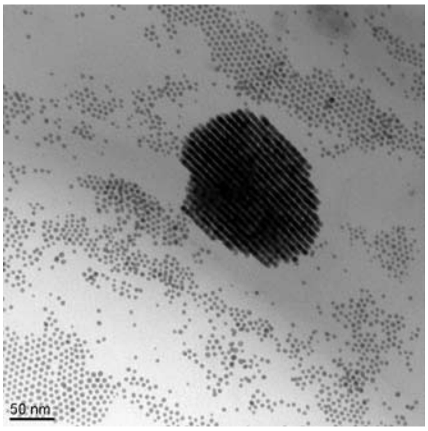


Figure 6: Crystalline agglomerate of 6-nm-Gold nanoparticles.

Figure 7: The capillary assisted particle assembly tool.

Particle assembly methods

Nanoparticles are assembled by this group at gas-liquid or liquid-solid interfaces, in thin wetting layers, in the bulk via temperature reduction, the introduction of incompatible solvents, as well as with other methods. Assembly and deposition take place under tightly controlled conditions and with analysis.

For example, the *capillary assisted assembly-tool* (Figure 7) enables small-scale "dip-coating" experiments at controlled temperature and speed, where the formation of the layer can be observed microscopically and spectroscopically. Emulsion-assisted particle assembly is a complementary method, where small liquid droplets contain the nanoparticles that assemble while the liquid of the droplets is slowly evaporated (Figure 8).

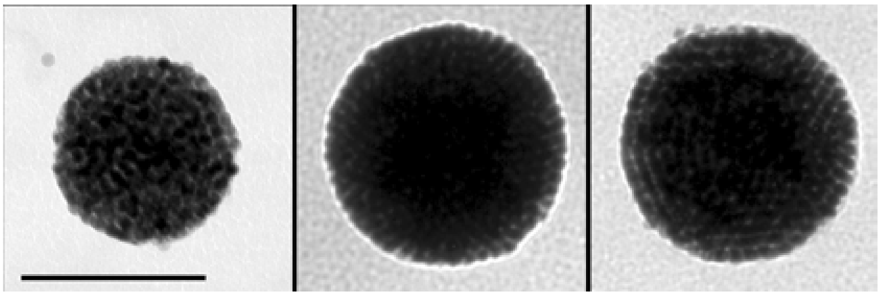


Figure 8: Emulsion-assembled nanoparticles.

Outlook

Nanoparticle-based material synthesis is attractive because it creates complex materials and still can be scaled up efficiently. Our group will collaborate with other INM groups to develop the synthesis of bulk quantities of regularly nanostructured materials from particles.

Initial studies naturally use simple structures, e.g., dense hexagonal layers of nanoparticles (Figure 9), to show the feasibility of extended, functional superstructures. More complex geometries can be attained using mixtures of multiple particles and, ultimately, complex particles with designed, anisotropic interactions. Our long-term goal is to provide a rationale for almost arbitrary structures, including the ability to predict the appropriate particle mixture and deposition method.

Polydispersed particles are another complex, widely used class of materials. They form partially ordered structures that have important implications for existing composite materials, but also for basic questions of packing and jamming. We will apply the methods developed with monodispersed systems in these complex particle dispersions to improve known deposition techniques.

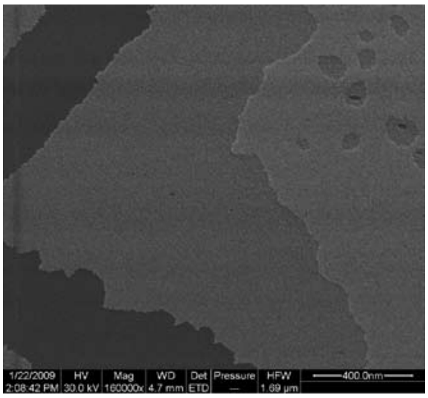


Figure 9: Dense mono- and double-layers of nanoparticles.

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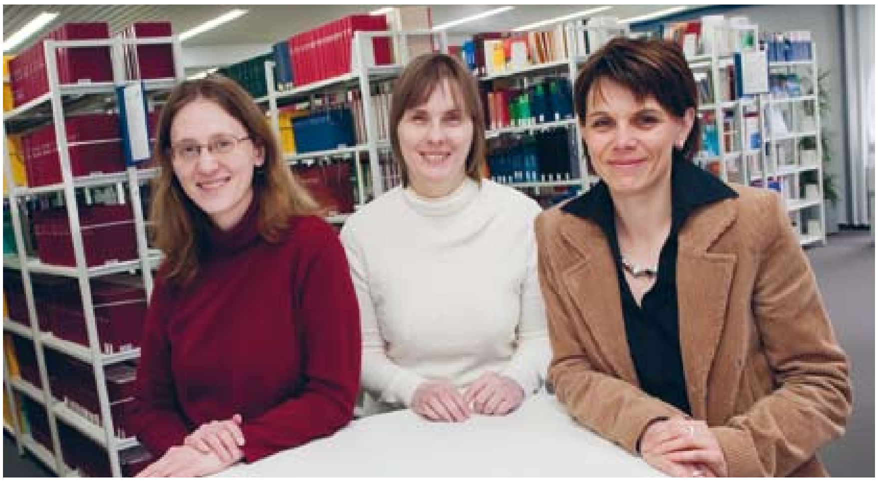
In ihrer Leitlinie zu Open Access formulierte die Leibniz-Gemeinschaft 2007 das Ziel, dass die Forschungsergebnisse ihrer Mitgliedseinrichtungen möglichst digital und frei zugänglich publiziert werden und langfristig an zentraler Stelle nachgewiesen werden sollen. Dies soll schwerpunktmäßig zunächst im Rahmen der elektronischen Zweitveröffentlichungen von Zeitschriftenartikeln unter Berücksichtigung der urheberrechtlichen Bestimmungen erfolgen. Die INM-Bibliothek nutzt zu diesem Zweck SciDok (http://scidok.sulb.uni-saarland.de/), den Wissenschaftsserver der Saarländischen Universitäts- und Landesbibliothek. In einem ersten Schritt wurden ca. 300 wissenschaftliche Publikationen in SciDok veröffentlicht. Dabei handelt es sich im Wesentlichen um Publikationen der wissenschaftlichen Geschäftsführer des INM aus dem Zeitraum 1966-1994, für die der Bibliothek ein Nutzungsrecht übertragen worden ist. Die INM-Bibliothek plant für die Zukunft, unter Berücksichtigung der verlegerischen Bestimmungen, die INM-Publikationen möglichst umfangreich über SciDok elektronisch zur Verfügung zu stellen.

Dokumentation von INM-Publikationen

Wissenschaftliche Publikationen gelten als quantitativer Leistungsindikator bei Audit und Evaluierung innerhalb der Leibniz-Gemeinschaft. In der INM-Bibliothek werden an zentraler Stelle sämtliche Veröffentlichungen des Hauses kontinuierlich und vollständig dokumentiert, um Analysen nach evaluationsrelevanten Kriterien zu ermöglichen. Als Basis dient die Literaturverwaltungssoftware Endnote, für die auch Schulungen für interessierte Wissenschaftlerinnen und Wissenschaftler des Hauses durchgeführt worden sind.

Interne Kommunikation und Information

Innerhalb der Arbeitsgruppe PR / Öffentlichkeitsarbeit des INM ist die Bibliothek an der Konzeption und dem Erstellen von Medien und Kommunikationsmitteln des Instituts beteiligt. Dazu gehören die Mitarbeit bei der Erstellung der Mitarbeiterzeitschrift nanonews, Administration und Weiterentwicklung des INM-Internetauftritts sowie im Berichtszeitraum die Konzeption und Erstellung eines Intranets für das Institut.



Service group Chemical Analysis / Servicegruppe Chemische Analytik

Dr. Claudia Fink-Straube

Für die Ermittlung der Zusammensetzung von im INM synthetisierten Materialien und die Charakterisierung der Reaktionsabläufe bei der Materialherstellung werden die Methoden der Flüssig- (HPLC, LC/MS, SEC) und Gaschromatographie (GC, GC/MS), atomspektrometrische Messverfahren (AAS, ICP OES) sowie die Flüssig- und Festkörper-NMR-Spektroskopie eingesetzt.

Im Jahr 2008 wurden vom Servicebereich Chemische Analytik ca. 80 % der analytischen Arbeiten für interne Untersuchungen und Kooperationen mit der Universität des Saarlandes, ca. 10 % für Routinemessungen der Wareneingangskontrolle und etwa 10 % für externe Auftraggeber, hauptsächlich Spin-offs des INM, durchgeführt.

Neben den analytischen Untersuchungen zu den laufenden Projekten des INM lag ein weiterer Schwerpunkt auf der Anpassung, Erweiterung und Optimierung der bestehenden Methoden auf die Anforderungen der neuen Programmbereiche. So wurde beispielsweise für die Analyse von Aminosäuren in biologischen Materialien im Bereich Chromatographie der Autosampler des bestehenden LC/MS-Systems für die Onlinederivatisierung von primären und sekundären Aminosäuren mit o-Phthalaldehyd (OPA) bzw. 9-Fluorenyl-Methyl-Chloroformiat (FMOC) eingerichtet und optimiert. Anhand von Testproben (Hydrolysate von Pfauenfederkiel und -schaumkern) konnte gezeigt werden, dass somit eine schnelle Quantifizierung der Aminosäuren in Proteinhydrolysaten ohne aufwändige manuelle Probenvorbehandlung möglich ist (siehe Abbildung 1).

Zur Bestimmung der Größenverteilung von DNA-Materialien wurden neue Säulen für die Gelfiltrations-Chromatographie beschafft sowie Eichkurven erstellt, und es konnten erste Realproben (Heringssperma) analysiert werden.

Mit kontinuierlichen GC/MS-Untersuchungen konnte die Umsetzung der Synthese des funktionellen Silans 1-(2-(2-(2-aminoethoxy)ethoxy)ethyl)-3-(3-(triethoxysilyl)propyl)urea aus 3-Isocyanato propyltriethoxysilan und 2,2 ethylendioxy-bis-ethylamine, die unter unterschiedlichen Bedingungen hergestellt wurden, quantitativ verfolgt werden.

Für die beiden verfügbaren GC/MS wurde ein neues automatisches Probengebersystem mit Headspace- und SPME-

Funktion installiert. Neben Platz- und Kostenersparnis erlaubt dieser Autosampler die Ansteuerung beider GCs nebeneinander, so dass die Proben gleichzeitig auf zwei verschiedenen Säulenmaterialien analysiert werden können.

Um das Probenspektrum für die ICP OES zu erweitern, wurden zwei neue Zerstäuber, „PolyCon“ und „VeeSpray“, zum einen für sehr geringe Probenvolumina mit einem Durchfluss von nur 0,1 ml/min und zum anderen für HF-haltige Suspensionen mit Partikelgrößen bis 300 µm beschafft.

Im Rahmen eines Industrieprojektes erfolgte der Vergleich von Analyseergebnissen des INM mit den Messergebnissen eines akkreditierten Labors anhand von zwei pharmazeutischen Proben. Die Analyse umfasste die Messung mit zwei verschiedenen Verfahren (INM: ICP OES, Fremdlabor: GF AAS) inklusive entsprechender Probenvorbehandlung. Die Abweichungen der Ergebnisse zwischen INM und akkreditiertem Labor betrugen < 0,4 %.

Bereits Ende 2006 wurde eine Kooperation mit Frau Prof. Dr. Petra Bauer von der Arbeitsgruppe für molekulare Pflanzenbiologie und Botanik des Fachbereichs Biowissenschaften an der Universität des Saarlandes gestartet, die im Jahr 2008 intensiviert wurde. Dabei analysiert das INM quantitativ die Eisen-, Kupfer-, Zink- und Mangangehalte in gefriergetrockneten Pflanzenmaterialien (bisher Arabidopsis und Tomate) mit direkter Feststoff-AAS.

Eine weitere Zusammenarbeit mit der Universität des Saarlandes erfolgte auf dem Gebiet der NMR-Spektroskopie. Für den Lehrstuhl Adhäsion und Interphasen in Polymeren von Herrn Prof. Dr. Wulff Possart des Fachbereichs Materialwissenschaften und Werkstofftechnik wurden Protonenmessungen bei tiefen Temperaturen bis 193 K an Dicyandiamid, das als Härter für Epoxidharze eingesetzt wird, durchgeführt, um Hinweise auf die Struktur des Moleküls zu erhalten. Außerdem wurden über Kohlenstoffmessungen mögliche Reaktionsprodukte dieser Umsetzung identifiziert.

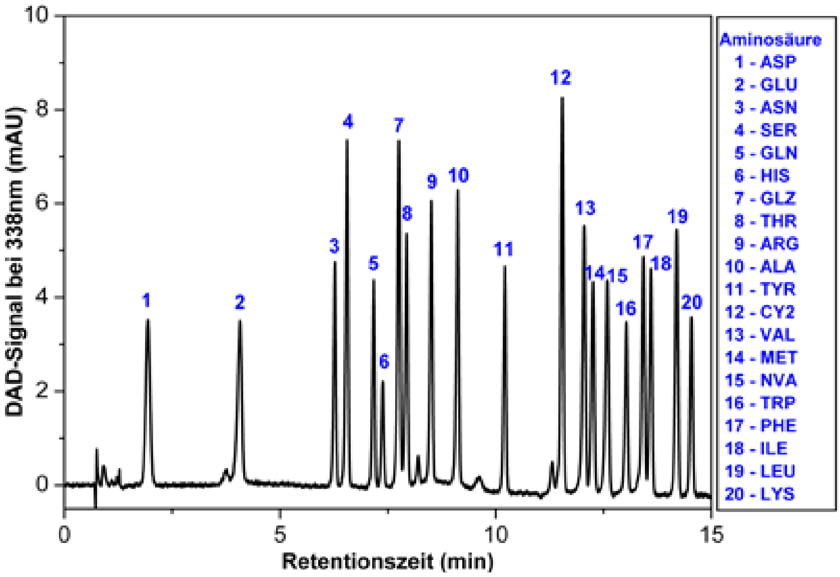


Figure 1: High-resolution analysis of 20 primary amino acids (online derivatisation with o-phthalaldehyde) using ZORBAX Eclipse AAA column (4.6 mm x 150 mm, 5 µm).

Service group Physical Analysis / Servicegruppe Physikalische Analytik

Dr. Herbert Schmid

Für eine umfassende Charakterisierung nanostrukturierter Materialien bezüglich deren Struktur, Phase, Morphologie und chemischer Zusammensetzung stehen in der Physikalischen Analytik folgende Methoden zur Verfügung:

Röntgenanalytik:

- Konventionelle Pulver-Röntgendiffraktometrie (XRD)
- Hochtemperatur-XRD bis 1800° C unter Vakuum oder Inertgas (HTK-XRD)
- Vier-Kreis-Diffraktometrie für Reflektometrie und Textur-/Spannungsanalyse
- Röntgen-Fluoreszenzspektroskopie (XRF)

Mikroskopie:

- Analytische Transmissions-Elektronenmikroskopie (TEM) mit Röntgenspektrometer (EDS)
- Konventionelle Raster-Elektronenmikroskopie (SEM) mit EDS
- Raster-Elektronenmikroskopie unter variablen Vakuumbedingungen (ESEM)
- Raster-Kraftmikroskopie (AFM)
- Konfokale Laser-Raster-Mikroskopie (CLSM)

Die Hauptaktivitäten der Physikalischen Analytik betreffen Serviceleistungen für die Programmbereiche des INM sowie Kooperationen im Rahmen von Projekten mit internen und externen Partnern. Die Betreuung der Geräte, insbesondere deren regelmäßige Justierung und Kalibrierung, wurde im Rahmen allgemeiner Serviceleistungen durchgeführt. Mitarbeiter aus anderen Abteilungen wurden im Rahmen längerfristiger Projekte in Gerätebedienungen eingewiesen und beratend unterstützt und betreut.

An umfangreicheren laufenden Aktivitäten im Jahr 2008 sind zu nennen:

(1) INM-interne Kooperationen/Serviceleistungen:

- SEM- und HTK-XRD-Messungen an ITO zu Fragen der Transformation amorph → kristallin in Abhängigkeit von Temperatur und Atmosphäre (NMO/Verfahrenstechnik)
- HTK-XRD-Messungen zur Klärung der Umwandlung $Zn(OH)_2 \rightarrow ZnO$ (NMO/Verfahrenstechnik)
- HTK-XRD-Messungen an $BaTiO_3$ zur Phasentransformation tetragonal → kubisch (Nanoprotekt/Kombinatorik)

- SEM- und XRD-Messungen zur Phasenanalyse im ternären System Fe_2O_3-TiO_2-Al_2O_3 (Nanoprotekt/Kombinatorik)
- kombinierte TEM-, SEM- und XRD-Untersuchungen zur Klärung der Core/Shell-Struktur von Al/Al_2O_3-, SnO_2- und In_2O_3-Nanowires (CVD/PVD)
- analytische TEM-Untersuchungen zur Morphologie von Fe_3O_4-Nanopartikeln mit Pt-Kern und deren Anordnung in zweidimensionalen Gitterstrukturen (Glas und Optik)

(2) Methodische Entwicklungen:

- Software-Lösungen zur Zuordnung zwischen Beugungsreflexen in Elektronenbeugung und Kristallorientierung; automatische Erfassung von Kristallstruktur, Dimension und Morphologie von Nanopartikeln

- Benetzungsphänomene auf strukturierten Oberflächen wurden mittels hochauflösender ESEM in Abhängigkeit von Wasserdampf-Partialdruck und Probentemperatur untersucht. Es konnte gezeigt werden, dass bei geeigneter Oberflächenstrukturierung auch hydrophile Materialien super-hydrophobe Eigenschaften annehmen können.

- Weiterentwicklung des ESEM-Modus zu WET-STEM-Modus, d. h. Beobachtung mittels Raster-Transmissions-Abbildung (STEM) von Nanopartikeln in flüssigen Medien

(3) Externe Kooperationen

- dynamische XRD-Messungen zum Kornwachstum in nanokristallinem Palladium (Technische Physik/UdS)

- ESEM-Abbildungen zum Verhalten von Ag-Nanopartikel an Zellwänden von E. Coli-Bakterien (Uniklinik Homburg/UdS)

- analytische TEM-Untersuchungen zur Struktur von Ni/a-C Sensorschichten (HTW Saarbrücken)

Service group Engineering/Workshop / Servicegruppe Engineering/Werkstatt

Dietmar Serwas

Die Servicegruppe Engineering/Workshop setzt sich zusammen aus den Arbeitsbereichen Konstruktion, Elektronik, Elektrotechnik sowie mechanische und Elektrowerkstatt. Der Servicebereich versteht sich als methodische Arbeitsgruppe im INM zur Durchführung von Dienstleistungen sowohl für die wissenschaftlichen Abteilungen als auch für alle anderen Bereiche des Hauses wie z. B. Analytik, Verwaltung oder Haustechnik. Weiterhin führt die Servicegruppe im Rahmen einer Kooperation die Werkstattaufgaben für den Lehrstuhl Technische Physik der Universität des Saarlandes durch.

Das Hauptarbeitsgebiet liegt in der Entwicklung und im Bau von wissenschaftlichen Anlagen und Komponenten für die Forschungsabteilungen im Rahmen von Projekten und im Bereich der Grundlagenforschung. Die Bandbreite reicht hierbei von kleinen Laborgeräten bis hin zu großen Pilotanlagen. Der Servicebereich schließt hierzu an die Forschung der wissenschaftlichen Abteilungen an und setzt dieses in Produkte um. Aus den Vorgaben der Forschung werden Konzepte entwickelt, mit denen nach Präzisierung der Anforderungen eine Konstruktion erstellt wird. Planung, Konstruktion und Bau erfordern eine enge Verzahnung mit den wissenschaftlichen Abteilungen des Hauses. Die praktische Umsetzung erfolgt in den Werkstätten sowohl durch weitestgehend eigene Fertigung von individuellen Steuerungen und Softwareentwicklungen als auch durch die mechanische Herstellung der einzelnen Komponenten einschließlich Zusammenbau zur kompletten Anlage.

Ein weiteres Arbeitsgebiet ist die Messwerterfassung zur Charakterisierung elektrischer Materialeigenschaften.

Zur Bewältigung dieser Aufgaben setzen wir in unserem Team auf modernste Technologie. So stehen für Konstruktionen die 3D-CAD-Software Catia V5 und für die Erstellung von Schaltplänen die CAD-Software CADDY++electrical zur Verfügung. Der Bereich Elektronik/Elektrotechnik/Messwerterfassung arbeitet mit hochpräzisen Mess- und Steuergeräten. Der Bereich Mechanik verfügt über einen CNC-Maschinenpark der bis zur 5-Achsen-gesteuerten Maschine reicht. Zur optimalen Nutzung der Fertigungsmöglichkeiten wird ein Offline-CAM-System von Cenit verwendet, das die CAD-Daten der Konstruktion in Fertigungsdaten generiert. Neben diesen Dienstleistungen legen wir großen Wert auf eine umfassende Unterrichtung unserer Auszubildenden.

Im Berichtszeitraum wurde die Ausstattung um einen leistungsfähigen Heliumlecktester sowie eine 6-Achs-Wasserstrahlschneidanlage erweitert. Damit ist es möglich, fast alle Materialien zu schneiden – auch beschichtete Materialien ohne Wärmeeinfluss und ohne Gefügeveränderungen. Das Konzept sah eine volle 3D-Funktion sowie die Kompatibilität mit dem vorhandenen CAD/CAM-System vor. Das Besondere dieser Anlage ist, neben dem Arbeiten im vollen 3D-Bereich, die Offlineprogrammierbarkeit und Datenerzeugung/-generierung mittels CAD/CAM-System auch im Abrasivschneidbereich. Um diese komplexen Konturen im 3D-Bereich schneiden zu können, wird der Schneidkopf von einem 6-Achs-Knickarmroboter geführt. Zur Erzielung hoher Absolutgenauigkeiten ist die Anlage mit einem Kalibriersystem ausgerüstet, das die Abweichungen des Roboters im Bereich der Absolutgenauigkeiten ausgleicht. Um die geforderten Sicherheitsanforderungen zu erfüllen, wurde die Anlage neben einer softwaremäßigen Beschränkung der Schneidbereiche mit einer begehbaren Glassicherheitseinhausung mit Sicherheitsabschaltung bei unbefugtem Zutritt im Betrieb ausgerüstet. Die Doppelverglasung mit Sicherheitsglas führt bei Auftreffen des Wasserstrahls durch spezielle Sensoren zum sofortigen Abschalten der Anlage. Dadurch ist einerseits die volle Beobachtung möglich und andererseits bei Fehlfunktionen ein sicheres Abschalten der Anlage gewährleistet. Eine weitere Neuheit ist die von der Firma Kunert für diese Anlage entwickelte Schutzhülle, um die beweglichen Teile des Roboters vor dem Eindringen von abrasiven Stoffen zu schützen.

Damit ist es möglich, ohne Wärmeeintrag und unter Vermeidung von Gefügeveränderungen, fast alle – auch beschichtete – Materialien zu schneiden.

Service Group Materials Testing/Powder synthesis / Servicegruppe Materialprüfung/Pulversynthese

Karl-Peter Schmitt, Robert Drumm

Service group Computer Services /Servicegruppe EDV

Wolfgang Türk

Service Group Materials Testing/ Powder synthesis / Servicegruppe Materialprüfung/Pulversynthese

Die prinzipielle Aufgabe der Servicegruppe Materialprüfung liegt in der Charakterisierung der am INM gefertigten Materialien im Hinblick auf ihre mechanischen Eigenschaften. Die dafür eingesetzten Messeinrichtungen spiegeln die große Varietät der am INM untersuchten Materialien und ihrer Formen wider. Sie erlauben nicht nur die Charakterisierung monolithischer Materialien wie beispielsweise von Keramiken, Gläsern, Polymeren oder Metallen, sondern auch die Messung der mechanischen Eigenschaften sowohl von Kompositen als auch von Schichtsystemen. Neben der Durchführung von Standardtestprozeduren unterstützt die Servicegruppe auch die Forschung am INM in Fragen der Materialmechanik und des Prüfens. Als weitere Aufgabe tritt neben die Materialprüfung die Synthese nanoskaliger Pulver. Hier steht vor allem das Up-Scaling von Syntheseprozessen, d. h. die Übertragung von Laborprozessen in den Pilotanlagenmaßstab im Vordergrund.

Service group Computer Services / Servicegruppe EDV

Der IT-Service des INM kümmert sich um die elektronischen Datenverabeitungssysteme des Hauses und stellt die Funktion der Server, PCs und Netzwerke sicher. Neben einer kontinuierlichen Weiterentwicklung der Geräte und Komponenten werden existierende Techniken auf den neusten Stand gebracht beziehungsweise an aktuelle Anforderungen angepasst. Dies gilt sowohl für komplette funktionelle Einheiten wie z. B. Datenserver individueller Arbeitsbereiche, als auch für periphere Geräte wie z. B. Drucker und zentral verfügbare Hochgeschwindigkeitsgeräte.

Facts and Figures/ Fakten und Zahlen

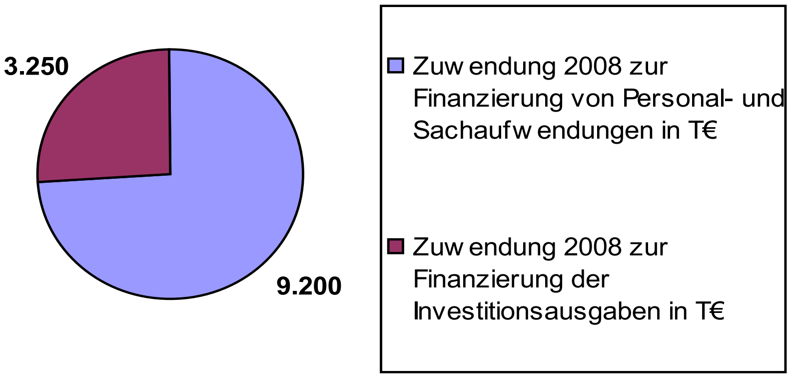
Statusbericht

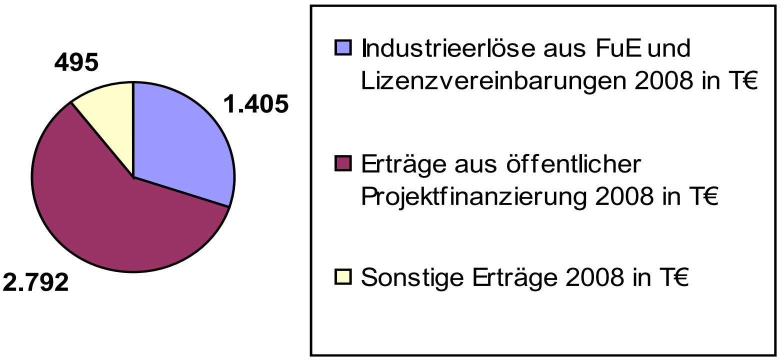
Finanz- und Ertragslage / Vermögenslage der Gesellschaft

Als Forschungseinrichtung der Leibniz-Gemeinschaft hat das INM auch im Haushaltsjahr 2008 eine gemeinsame Förderung durch den Bund und die Länder erhalten. Diese belief sich auf 12.450 T€; hiervon 9.200 T€ zur Finanzierung von Personal- und Sachaufwendungen sowie 3.250 T€ für erforderliche Neu- und Ersatzinvestitionen.

Die nominale Steigerung der Zuwendung gegenüber dem Vorjahr um 1.250 T€ ist durch eine allgemeine Zunahme zum Ausgleich von Tariflohn- und Preissteigerungen sowie eine Sonderfinanzierung zum Aufbau neuer Forschungsschwerpunkte bedingt.

Im Geschäftsjahr 2008 erzielte das Leibniz-Institut für Neue Materialien eigene Erlöse aus Forschung und Entwicklung sowie sonstige betriebliche Erträge in Höhe von 4.692 T€ (Vorjahr: 4.427 T€). Die Industrieerlöse aus Forschung und Entwicklung sowie aus Lizenzvereinbarungen beliefen sich hierbei auf 1.405 T€ (Vorjahr: 1.560 T€). Im Rahmen öffentlicher Projektfinanzierungen erzielte das INM im Jahr 2008 Erträge in Höhe von 2.792 T€ (Vorjahr: 1.976 T€). Sonstige Erträge resultierten im Geschäftsjahr 2008 vornehmlich aus der Weiterbelastung von Gebäude-, Patent- sowie sonstigen Kosten.





Im EU-Projekt „Multiprotect“ ist das Leibniz-Institut für Neue Materialien Projektkoordinator und hat in dieser Funktion im Geschäftsjahr 2008 weitere Fördermittel der Europäischen Union in Höhe von 2.193 T€ vereinnahmt und an die Projektpartner weitergeleitet.

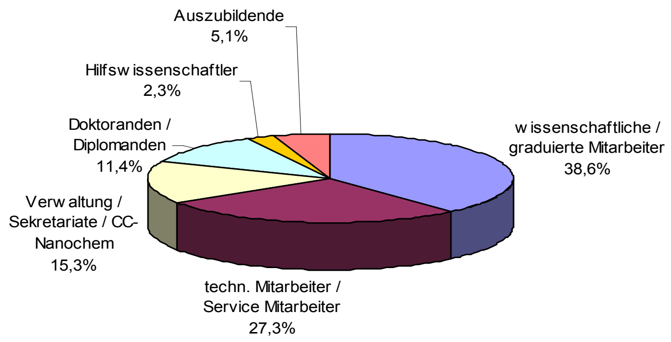
Der Gesamtumsatz 2008 der Gesellschaft betrug 17.166 T€ (Vorjahr: 14.881 T€). In erster Linie resultiert diese Erhöhung aus den Erträgen aus der gemeinsamen Finanzierung durch Bund und Länder. Der Anteil der Drittmittel sowie der sonstigen Erträge im Geschäftsjahr 2008 belief sich somit auf 27,3 % des Gesamtbudgets (Vorjahr 29,7 %).

Die Bilanzsumme der Gesellschaft zum 31. Dezember 2008 betrug 16.223 T€; gegenüber dem Vorjahr eine deutliche Erhöhung um 1.468 T€. Während sich das Umlaufvermögen gegenüber dem Bilanzstichtag des Vorjahres geringfügig um 240 T€ verringerte, ist beim Anlagevermögen der Gesellschaft eine beträchtliche Zunahme um 1.702 T€ zu verzeichnen. Die aktiven Rechnungsabgrenzungsposten haben sich gegenüber dem Vorjahr um 6 T€ erhöht. Die Investitionstätigkeit der Gesellschaft (3.684 T€) überstieg im Geschäftsjahr 2008 erneut deutlich die Abschreibungsquote in Höhe von 1.982 T€. Die Verbindlichkeiten der Gesellschaft beliefen sich zum Bilanzstichtag auf 752 T€ (Vorjahr: 765 T€).

Personalentwicklung

Die Anzahl der Beschäftigten in 2008 blieb gegenüber dem Jahr 2007 nahezu konstant. So waren Ende 2008 176 Mitarbeiter (160 Vollzeitäquivalente) gegenüber 178 Mitarbeiter (157 Vollzeitäquivalente) Ende 2007 beschäftigt. Hiervon waren 54 Mitarbeiterinnen und Mitarbeiter (dies entspricht 51 Vollzeitäquivalenten) im Drittmittelbereich beschäftigt. Die Anzahl der Auszubildenden konnte erneut auf nunmehr neun gesteigert werden; der Anteil der Mitarbeiterinnen und Mitarbeiter im Verwaltungsbereich / Sekretariate / CC-Nanochem erhöhte sich auf 15,3 %. Der Anteil der Doktoranden / Diplomanden verringerte sich mit 11,4 % gegenüber 11,8 % in 2007 geringfügig, der der Hilfswissenschaftler sank auf 2,3 % gegenüber 6,7 % im Vorjahr. Der Anteil der wissenschaftlichen und graduierten Mitarbeiter konnte gegenüber dem Vorjahr um 3,2 Prozentpunkte von 35,4 % auf 38,6 % angehoben werden, während der Anteil der Servicemitarbeiter und der Mitarbeiter im technischen Bereich mit 27,3 % gegenüber 27,5 % in 2007 nahezu konstant blieb.

Personal nach Beschäftigtengruppen in 2008

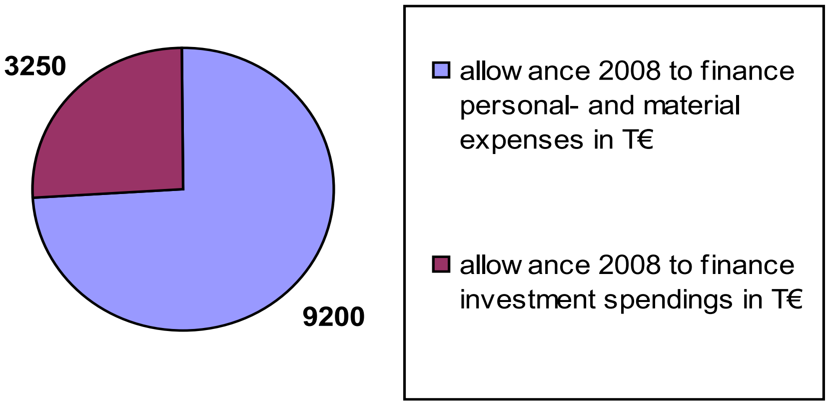


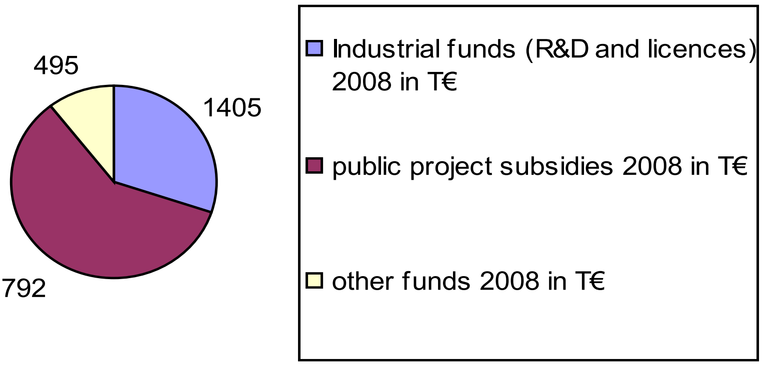
Financial and income situation of the company

As a research institute of the Leibniz Association, INM obtained common grants from the federal government and the states in the financial year 2008. These amounted to 12,450 T€, of which 9,200 T€ were used to finance personnel and material expenses and 3,250 T€ for new investments and reconstruction measures.

The nominal increase of the obtained common grant amounting to 1,250 T€ results from a general rise for equalizing the increase of the standard wages and price rise and also from a special grant for the development of new main research fields.

In the financial year 2008, INM – Leibniz Institute for New Materials generated own proceeds of 4,692 T€ (preceding year 4,427 T€) from research and development as well as from other operating income. Industry revenues from research and development as well as from patents/licences amounted to 1,405 T€ (preceding year 1,560 T€). Within public project subsidies, INM made a profit in the amount of 2,792 T€ in 2008 (preceding year 1,976 T€). Other income principally results from cost transfer for buildings, patents and other costs.





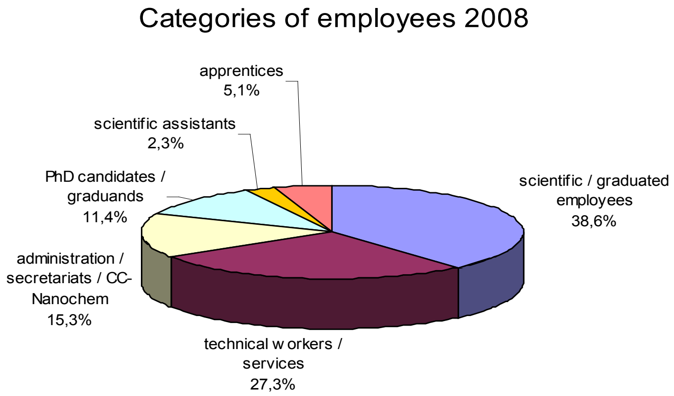
INM is the project coordinator of an EU-project named “Multiprotect” and collected in this function another subsidy amounting to 2.193 T€ from the European Union. These subsidies were transferred to the corresponding project partners.

The total turnover of INM in 2008 added up to 17,166 T€ (preceding year 14,881 T€). This increase primarily results from obtained common grants from the federal government and the state. The share in third-party revenues as well as in other revenues in the business year 2008 amounted to 27.3 % of the total turnover (preceding year 29.7 %).

The balance sheet total of the corporation was 16,223 T€ on 31 December 2008, compared to the preceding year a remarkable increase by 1,468 T€. While current assets decreased slightly by 240 T€ compared to the reporting date of the previous year, the non-current assets of the corporation increased remarkable by 1,702 T€. The investment activity of the corporation (3,684 T€) anew exceeded noticeably the allowance rate amounting to 1,982 T€ in the financial year 2008. The liabilities of the corporation amounted to 752 T€ at reporting date (preceding year 765 T€).

Personnel development

In comparison to 2007, the number of employees remained nearly constant. At the end of 2008, 176 employees (160 full-time equivalents) worked at INM, compared to the preceding year with 178 employees (157 full-time equivalents). 54 employees (51 full-time equivalents) were financed by third-party funds. The number of apprentices increased from eight to nine, while the proportion of the administration staff / secretariats / CC-Nanochem increased from 14.0 % to 15.3 %. The proportion of PhD and other graduate students remained constant (11.4 % in 2008, 11.8 % in 2007) while the proportion of scientific and graduated employees increased about 3.2 percentage points from 35.4 % to 38.6 %. The proportion of manual workers and workers in technical services remained constant (27.5 % in 2008, 27.3 % in 2007), and the proportion of scientific assistants decreased from 6.7 % in 2007 to 2.3 % in 2008.



Members of the Board of Directors / Mitglieder des Kuratoriums
Members of the Scientific Board / Mitglieder des wissenschaftlichen Beirats

Activities in committees / Aktivitäten in Gremien

Members of the Board of Directors (Status: End of 2008) Mitglieder des Kuratoriums (Stand: Ende 2008)

Dr. Christian Ege

Staatssekretär
Ministerium für Wirtschaft und Wissenschaft des Saarlandes
– Vorsitzender –

Dr. Gerhard Felten

Geschäftsleiter Zentralbereich Forschung und Vorausentwicklung
Robert Bosch GmbH, Stuttgart

Prof. Dr. Horst Hahn

Geschäftsführender Direktor
Institut für Nanotechnologie (INT), Forschungszentrum Karlsruhe
Eggenstein-Leopoldshafen

Dr. Max Häring

Vorstandsvorsitzender a.D.
Landesbank Saar Girozentrale, Saarbrücken

Liane Horst

Bundesministerium für Bildung und Forschung, Berlin
– Stellvertr. Vorsitzende –

Prof. Dr. Volker Linneweber

Präsident der Universität des Saarlandes
– Stellvertr. Vorsitzender –

Prof. Dr. Dr. E.h. Kurt Mehlhorn

Wissenschaftlicher Direktor
Max-Planck-Institut für Informatik, Saarbrücken

Prof. Dr. Frank Mücklich

Lehrstuhl für Funktionswerkstoffe
Universität des Saarlandes

Dr. Peter William de Oliveira

Wissenschaftlicher Mitarbeiter
INM gGmbH, Saarbrücken

Dr. Roland Rolles

Leiter der Abteilung C – Mittelstand und Innovation
Ministerium für Wirtschaft und Wissenschaft des Saarlandes

Ralf Zastrau

Vorstandsvorsitzender
Nanogate AG, Göttelborn

Members of the Scientific Board (Status: End of 2008) Mitglieder des wissenschaftlichen Beirats (Stand: Ende 2008)

Prof. Dr. Horst Hahn

Geschäftsführender Direktor
Institut für Nanotechnologie (INT), Forschungszentrum Karlsruhe GmbH
Eggenstein-Leopoldshafen
– Vorsitzender –

Ralf Becker

Leitung Geschäftsfeld Sanitärkeramik und Küche
Villeroy & Boch AG, Mettlach

Prof. Dr. Rainer Birringer

Lehrstuhl für Technische Physik
Universität des Saarlandes

Prof. Dr. Ludwig Gauckler

Institut für Nichtmetallische Werkstoffe
ETH-Hönggerberg, Zürich, Schweiz

Dr. Klaus Harste

Mitglied des Vorstandes
Saarstahl AG, Völklingen

Prof. Dr. Martin Möller

Lehrstuhl für Textilchemie und Makromolekulare Chemie
RWTH Aachen

Prof. Dr. Andreas Mortensen

École Polytechnique Fédérale de Lausanne (EPFL), Schweiz

Prof. Dr. Erich Sackmann

Lehrstuhl für Biophysik
Technische Universität München, Garching

Dr. Stefan Sepeur

Geschäftsführer
NANO-X GmbH, Saarbrücken

Prof. Dr. Robert F. Singer

Lehrstuhl Werkstoffkunde und Technologie der Metalle
Universität Erlangen-Nürnberg

Activities in committees / Aktivitäten in Gremien

Prof. Dr. Eduard Arzt

Korrespondierendes Mitglied der Österreichischen Akademie der Wissenschaften

Mitglied der Deutschen Akademie der Naturforscher Leopoldina, Halle

Mitglied, Professorial Committee, IST Austria, Wien

Mitglied, Beirat von Garching Innovation GmbH, der Patentverwertungsagentur der Max-Planck-Gesellschaft

Mitglied, Corporate Research and Technology Board, Böhler Uddeholm AG, Wien

Mitglied, Internationaler Beirat der Christian-Doppler-Gesellschaft, Wien

Mitglied, Kuratorium des Erich-Schmid-Instituts für Materialwissenschaft der Österreichischen Akademie der Wissenschaften, Leoben

Mitglied, Scientific Advisory Board des Netherlands Institute for Metals Research (NIMR)

Mitglied, Scientific Advisory Board, CCMX - Comptenece Centre for Materials Science and Technology, Lausanne, Switzerland

Mitglied, Wissenschaftlicher Beirat der Alfried Krupp von Bohlen und Halbach Stiftung, Essen

Editor von „Progress in Materials Science" Oxford, UK (Reviewzeitschrift)

Gutachtertätigkeit bei

- Harvard University, USA
- Massachusetts Institute of Technology, USA
- Stanford University, USA
- Columbia University, USA
- Alexander-von-Humboldt-Stiftung

Mitglied im Editorial Board der Zeitschriften

- MRS Bulletin of the Materials Research Society, USA
- Materials Science and Technology, London, UK
- Advanced Engineering Materials, Weinheim
- Zeitschrift für Metallkunde, forts.: International Journal of Materials Research, München
- Materials Science and Engineering / C, Materials for Biological Applications.

Referee bei Zeitschriften (u.a.):

- Acta Materialia
- Applied Physics Letters
- Journal of Applied Physics
- Journal of the Royal Society / Interfaces
- Langmuir
- Nanotechnology
- Nature
- Nature Materials
- Scripta Materialia

Dr. Alexandra Barau

Referee der Zeitschrift “Journal of Non-Crystalline Solids”

Dr. Carsten Becker-Willinger

Gutachtertätigkeit bei Forschungsanträgen „Forschungsverbund Baden-Württemberg“

Prof. Dr. Roland Bennewitz

Adjunct Professor, Physics Department der McGill University, Montreal, Kanada

Honorarprofessor der Universität des Saarlandes, Saarbrücken

Mitglied im Editorial Board der Zeitschrift “Review of Scientific Instruments”

Referee der Zeitschriften

- Applied Physics Letters
- Journal of Physics / D, Applied Physics
- Physical Review / B
- Review of Scientific Instruments
- Thin solid films
- Tribology letters

Jochen Flackus

Vorstandsvorsitzender des NanoBioNet e.V.

Gründungsmitglied von Saarland empowering nano

Editor der Zeitschrift „empowering nano“

Dr. Annette Kraegeloh

Referee der Zeitschrift „Microbiology“

Dr. Tobias Kraus

Referee der Zeitschriften

- Applied Physics Letters
- Tribology Letters

Prof. Dr. Sanjay Mathur

Organisator, 2nd Symposium on Nanostructured Materials and Nanotechnology im Rahmen der 31st International Cocoa Beach Conference & Exposition on Advanced Ceramics & Composites, Daytona Beach, USA January 27-February 1st, 2008 (Organisator)

Mitglied, External Advisory Board to Strategic Research Cluster “Forme” Science Foundation Ireland

Mitglied, Inorganic Chemistry Division II der IUPAC 2004-2009

Mitglied, ECD Award Komitee der American Ceramic Society

Mitglied, IUPAC Projekt “Nanotechnology for Energy Production”

Mitglied im Editorial Board der Zeitschriften

- Journal of Applied Ceramic Technology (ACT)
- Ceramics International
- International Journal of Nanotechnology

Associated Editor von

- Journal of Nanomaterials
- International Journal of Applied Ceramic Technology (ACT)

Referee bei zahlreichen führenden chemischen und material-wissenschaftlichen Fachzeitschriften

Dr. Thomas Müller

Gutachtertätigkeit bei E.ON International Research Initiative

Referee der Zeitschrift „Thin Solid Films“

Dr. Peter W. de Oliveira

Mitglied, Round Table Lateinamerika des BMBF

Gutachtertätigkeit bei IWT-Vlaanderen, Instituut voor de aanmoediging van innovatie door Wetenschap & Technologie in Vlaanderen, Brüssel, Belgien

Referee der Zeitschriften

- Materials Letters
- Proceedings of the 32nd Internat. Conf. on Advanced Ceramics and Composites ICACC

Dr. Mario Quilitz

Gutachtertätigkeit bei Machbarkeitsstudien im Rahmen des AGeNT-De Netzwerkes der nationalen Kompetenzzentren für Nanotechnologien und Nanoanalytik

Gutachtertätigkeit bei ITAS-Expertengruppe „Nanowissenschaften & Nanotechnologie“ des Institutes für Technikfolgenabschätzung und Systemanalyse des FZK Karlsruhe

Gutachtertätigkeit bei E.ON International Research Initiative

Referee der Zeitschrift “Solid State Ionics”

Dr. Peter Rogin

Referee der Zeitschrift „Plasma Processes & Polymers“

Prof. Dr. Dr. Daniel J. Strauss

Visiting Professor, Technical University Malaysia Melaka

Mitglied, Empower Deutschland / Cluster Medizintechnik

Mitglied, Cognitive Neuroscience Society CNS

Mitglied, Institute of Electrical and Electronic Engineers IEEE

Mitglied, Engineering in Medicine and Biology Society EMBS

Mitglied, Society for Industrial and Applied Mathematics SIAM

Gutachtertätigkeit bei Medical Research Council, UK

Gutachtertätigkeit bei University Malaysia Perlis

Referee der Zeitschriften

- Journal of Neuroscience Methods
- Artificial Intelligence in Medicine
- IEEE Trans. on Neural Networks
- IEEE Trans. on Biomedical Engineering
- IEEE Trans. on Neural Systems & Rehabilitation Engineering
- Medical Engineering and Physics
- Annals of Biomedical Engineering
- IEEE International Conference Neural Engineering
- IEEE International Conference of the Engineering in Medicine and Biology Society
- Computers in Medicine & Biology

Sowie verschiedener nationaler Konferenzen

Prof. Dr. Dr. h. c. Michael Veith

Korrespondierendes Mitglied, Akademie der Wissenschaften und der Literatur, Mainz

Mitglied, Deutsche Akademie der Naturforscher Leopoldina, Halle

Fellow of the Royal Society of Chemistry, London

Berater, Universität Lyon

Bevollmächtigter der DFH für den Studentenaustausch mit Strasbourg (ECPM)

Externes Beratungsmitglied, LCC Toulouse

Sprecher, Internationales Graduiertenkolleg GRK 532

Stellvertretender Leiter, Frankreichzentrum, Universität des Saarlandes, Saarbrücken

Unirat, Universität des Saarlandes, Saarbrücken

Vorstand, AGeNT-De Netzwerk der nationalen Kompetenzzentren für Nanotechnologien und Nanoanalytik

1. Vorsitzender, cc-NanoChem

Vorstandsmitglied, International Ring Systems (IRIS), International Meetings: Germanium, Tin, Lead (GTL)

Wissenschaftlicher Beirat, Papiertechnische Stiftung PTS München

Wissenschaftlicher Beirat, Qiagen, Hilen

Mitglied, Ecole Doctorale Metz-Nancy

Mitglied, Expertengruppe Metropolprojekt Saarbrücken - Moselle Est

Mitglied, Verwaltungsrates der EEIGM Nancy

Mitglied, Programm-Komitee Nanotechnology International Forum, Moskau Dezember 2008

Mitglied, Arbeitsausschuss “Angewandte Anorganische Chemie” DECHEMA e.V.

Mitglied, Steering Komitee MiNaT

Gutachtertätigkeit, Deutsche Forschungsgemeinschaft (DFG)

Gutachtertätigkeit, FWF Wissenschaftsfonds, Wien

Gutachtertätigkeit, Deutsche Bundesstiftung Umwelt DBU

Gutachtertätigkeit, Deutsche Akademie der Naturforscher Leopoldina (Stipendien)

Gutachtertätigkeit, Projektträger Jülich / Forschungszentrum Jülich

Gutachtertätigkeit, Academy of Sciences, Tschechien

Gutachtertätigkeit, Universität Stockholm, Schweden

Gutachtertätigkeit, Qiagen GmbH (Innovation Award)

Gutachtertätigkeit, Helmholtz Gemeinschaft ((Hochschul-)Nachwuchsgruppen)

Gutachtertätigkeit, Alexander-von-Humboldt-Stiftung (Stipendium)

Gutachtertätigkeit, Universität Stuttgart (W3-Professur)

Gutachtertätigkeit, Universität Hannover (Berufungsverfahren W2)

Mitglied im Editorial Board der Zeitschriften

- New Journal of Chemistry (NJC)
- Comptes Rendus

- Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry (European Ed.)

Referee der Zeitschriften

- Inorganic Chemistry
- Canadian Journal of Analytical Sciences and Spectroscopy
- Chemistry – an Asian Journal
- Chemical Communications
- Advanced Materials
- Chemistry of Materials
- European Journal of Inorganic Chemistry
- Journal of Adhesion, Science and Technology
- Journal of Organometallic Chemistry
- Materials Science and Engineering / C
- Organometallics
- Dalton Transactions
- Zeitschrift für Anorganische und Allgemeine Chemie ZAAC
- Solid State Sciences
- Zeitschrift für Kristallographie

Dr. Ingrid Weiss

Gutachtertätigkeit, German-Israeli Foundation for Scientific Research and Development

Referee der Zeitschriften

- ChemBioChem
- Chemical Reviews
- CompBiochemPhysiol
- Journal of Structural Biology
- Marine Biology

Dr. Matthias Wittmar

Referee der Zeitschrift “Corrosion Science”

Awards / Auszeichnungen

Dr. Aranzazu del Campo und Prof. Dr. Eduard Arzt

Wiley-Interscience : Highly downloaded paper
A. del Campo and E. Arzt, Design parameters and current fabrication approaches for developing bioinspired dry adhesives, Macromol. Biosci. 2007, 7, 118-127

Prof. Dr. Eduard Arzt

ISI Highly Cited Article
R. Spolenak, St. Gorb, E. Arzt, Adhesion design maps for bio-inspired attachment systems, Acta Biomater., 2005, 1, 5-13

Graciela Castellanos

Fin de Carrera in Industrial Technical Engineering specialized in Chemistry
Universidad de Oviedo, Spain

Dr. Nitya Nand Gosvami

Best Poster Award and Price: Atomic-Scale Friction Study of Gold Surfaces Electrochemical Environment
International Nanotribology Forum, Nano-Sikkim III: Mechanics and Friction at the Nanoscale, Norbu Ghang Resort, Pelling, Sikkim, India, Nov. 10-14, 2008

Dr. Nitya Nand Gosvami

Alexander von Humboldt-Research Fellowship

Pan Jun

Best Poster Award: Controlled Synthesis and Growth Mechanism Study of Tin Oxide Nanowire Arrays
MSE Materials Science and Engineering, Nürnberg, Germany, Sept. 01-04, 2008

Prof. Dr. Sanjay Mathur

IOP Publishing: Top Paper Collection
F. Hernández-Ramírez, A. Tarancón, O. Casals, J. Rodríguez, A. Romano-Rodríguez, J.R. Morante, S. Barth, S. Mathur, T. Y. Choi, D. Poulikakos, V. Callegari and P. M. Nellen, Fabrication and electrical characterization of circuits based on individual tin oxide nanowires, Nanotechnology, 2006, 17, 5577-5583

Prof. Dr. M. Veith

Honorary Doctorate Degree
Universitè Paul Sabatier; Toulouse, France

Completed PhD theses / Abgeschlossene Dissertationen

S. Barth

Eindimensionale oxidische Nanostrukturen mittels chemischer Gasphasenabscheidung und Synthese heterometallischer Übergangsmetallalkoxide (Co, Mn und Fe)
Saarbrücken, Univ., Diss (2008)

E. Hemmer

Darstellung lanthanoidhaltiger Oxid- und Hydroxidphasen - Neue Konzepte zur Kontrolle von Morphologie und funktionellen Eigenschaften
Saarbrücken, Univ., Diss (2008)

J. Hopf

In-situ Herstellung von Al$_2$O$_3$/SiC-Nanokompositen
Saarbrücken, Univ., Diss (2008)

M. Klook

Kompositwerkstoffe auf Basis von Cellulosen, Cellulosefasern, Zucker und Ausschnitten aus Silikaten
Saarbrücken, Univ., Diss (2008)

C. Köhler

Verschleißverhalten von gefüllten Polymeren
Saarbrücken, Univ., Diss (2008)

H. Smail

Reactivity of alumosiloxanes for the synthesis of new metal derivatives
Saarbrücken, Univ., Diss (2008)

Completed diploma theses / Abgeschlossene Diplomarbeiten PhD students / Doktoranden Guest Scientists / Gastwissenschaftler

E. A. Sow

Importance of CVD-process parameters for the synthesis of novel Al/Al_2O_3 and Ga/Ga_2O_3 composite nanostructures
Saarbrücken, Univ., Diss (2008)

M. Uyanik

Synthesis and characterization of TiO_2 nanostars
Saarbrücken, Univ., Diss (2008)

Completed diploma theses / Abgeschlossene Diplomarbeiten

G. Castellanos

Estudio técnico y económico de la fabricación de adhesivos reversibles basados en polímeros magnéticos -Technical and economical study of the fabrication of switchable adhesives based on magnetic polymers-
Gijón <Spain>, Univ., Dipl (2008)

S. K. Gurram

Plasma enhanced chemical vapour deposition of silicon and tin oxides
Saarbrücken, Univ., Dipl (2008)

PhD students / Doktoranden

Aktas, Oral Cenk
Prof. Dr. M. Veith

Altmayer, Jessica
Prof. Dr. S. Mathur

Bender, Michael
Prof. Dr. M. Veith

Born, Philip
Prof. Dr. E. Arzt

Brörmann, Katrin
Prof. Dr. R. Bennewitz

Bubel, Carsten
Prof. Dr. M. Veith

Cavelius, Christian
Prof. Dr. S. Mathur

Egberts, Philip
Prof. Dr. R. Bennewitz

Gossmann, Kai
Prof. Dr. H. Schmidt

Hausen, Florian
Prof. Dr. R. Bennewitz

Kasper, Christoph
Prof. Dr. M. Veith

Kroner, Elmar
Prof. Dr. E. Arzt

Lehnert, Tobias
Prof. Dr. M. Veith

Moh, Karsten
Prof. Dr. M. Veith

Moll, Jana
Prof. Dr. M. Veith

Ndiaye, Amadou
Prof. Dr. M. Veith

Rabung, Benjamin
Prof. Dr. M. Veith

Ren, Shuhua
Prof. Dr. M. Veith

Ullah, Hameed
Prof. Dr. M. Veith

Weber, Eva
PD Dr. habil. I. Weiß

Xiao, Lisong
Prof. Dr. M. Veith

Guest Scientists / Gastwissenschaftler

Aktas, Oral Cenk
Türkei

Asbai, Abdellah
Marokko

Barau, Dr. Alexandra
Rumänien

Bingham, Ruth Violet
Großbritannien

Bylda, Caroline
Frankreich

Caparrotti, Dr. Hinka
Bulgarien

Castellanos, Graciela
Spanien

Castro, Dr.-Ing. Mayra Rúbia Silva
Brasilien

De Jesús, Carolina
Venezuela

De Souza, Dr. Emerson
Brasilien

Del Campo, Dr. Aránzazu
Spanien

Egberts, Philip
Kanada

Egorov, Dr. Yuri
Russland

Fiala, Jan
Tschechien

Filleter, Tobin
Kanada

Girault, Dr. Baptiste
Frankreich

Gosvami, Dr. Nitya Nand
Indien

Guidoni, Dr. Griselda
Argentinien

Gurram, Sanjeev Kumar
Indien

Jehanathan, Neerushana
Indien

Kamperman, Dr. Marleen
Niederlande

Laurent, Dr. Antoine
Frankreich

Lin, Dr. Hechun
China

Markova, Aleksandra
Bulgarien

McMeeking, Prof. Dr. Robert
USA

Murray, Dr. Eoin
Irland

Ndiaye, Amadou
Senegal

Negash, Amanuel
USA

Pan, Jun
China

Ren, Shuhua
China

Sam, Dr. Ebru Devrim
Türkei

Shanmugasundaram, Dr. Sakthivel
Indien

Shen, Dr. Hao
China

Smail, Dr. Hakima
Frankreich

Sow, Dr. Eve Awa
Frankreich

Trenado, Carlos
Mexiko

Ullah, Hameed
Pakistan

Uyanik, Dr. Mehmet
Türkei

Wei, Jiandong
China

Xiao, Lisong
China

Yazdani-Assl, Dr. Omid
Iran

Yu, Dr. Eung Chul
Südkorea

Publications / Publikationen

Book chapter / Buchkapitel

M. Ehses and M. Veith

Bachelor/Master-Reform in den Natur- und Ingenieurwissenschaften: Fragen grenzüberschreitender Zusammenarbeit
In: Jahrbuch des Frankreichzentrums. Am Wendepunkt - Deutschland und Frankreich um 1945 - zur Dynamik eines ‚transnationalen‘ kulturellen Feldes, P. Oster Ed.,Transcript: Bielefeld, 2008, pp 309-335

M. Veith

Vorwort
In: Nanomaterialien in Architektur, Innenarchitektur und Design, S. Leydecker Ed.,Birkhäuser: Basel, 2008, pp 10-11

I. M. Weiss and F. Marin

The role of enzymes in biomineralization processes
In: Biomineralization. From Nature to Application, A. Sigel, H. Sigel and R. K. O. Sigel Eds.,Wiley: Chichester <UK>, 2008, Vol. 4, pp 71-126

Journal articles / Publikationen in Journalen

J. Adam, M. Aslan, R. Drumm and M. Veith

Mechanisms of bonding effected by nanoparticles in zirconia coatings applied by spraying of suspensions
Annual Report, Jahresbericht 2007 - INM Leibniz-Institut für Neue Materialien 2008, 60-68 [-]

J. Adam, R. Drumm, G. Klein and M. Veith

Milling of zirconia nanoparticles in a stirred media mill

J Am Ceram Soc 2008, 91, (9), 2836-2843 [01.792 (2007)]

M. A. Aegerter, R. M. Almeida, A. Soutar, K. Tadanaga, H. Yang and T. Watanabe

Coatings made by sol-gel and chemical nanotechnology
J Sol-Gel Sci Techn 2008, 47, (2), 203-236 [01.300 (2007)]

A. Al-Kahlout, S. Heusing and M. A. Aegerter

Brown coloring electrochromic devices
J Sol-Gel Sci Techn 2008, 47, (2), 215-216 [01.300 (2007)]

M. Ames, J. Markmann, R. Karos, A. Michels, A. Tschöpe and R. Birringer

Unraveling the nature of room temperature grain growth in nanocrystalline materials
Acta Mater 2008, 56, (16), 4255-4266 [03.624 (2007)]

C. O. Avellaneda, D. F. Vieira, A. Al-Kahlout, S. Heusing, E. R. Leite, A. Pawlicka and M. A. Aegerter

All solid-state electrochromic devices with gelatin-based electrolyte
Sol Energ Mat Sol C 2008, 92, (2), 228-233 [02.002 (2007)]

S. Barth, S. Mathur, F. Hernandez-Ramirez and A. Romano-Rodriguez

One-dimensional oxide nanostructures: Growth, applications and devices
Annual Report, Jahresbericht 2007 - INM Leibniz-Institut für Neue Materialien 2008, 52-60 [-]

M. Bender, R. Drumm, J. Adam, A. Jakob, R. Lemor and M. Veith

Preparation of acoustic lenses by mechanochemical synthesis and electrophoretic deposition of lead zirconium titanate (PZT) films
Annual Report, Jahresbericht 2007 - INM Leibniz-Institut für Neue Materialien 2008, 68-75 [-]

R. Bennewitz and J. T. Dickinson

Fundamental studies of nanometer-scale wear mechanisms
MRS Bull 2008, 33, (12), 1174-1180 [05.168 (2007)]

M. R. S. Castro, A. F. Lasagni, H. K. Schmidt and F. Mücklich

Direct laser interference patterning of multi-walled carbon nanotube-based transparent conductive coatings
Appl Surf Sci 2008, 254, (18), 5874-5878 [01.406 (2007)]

M. R. S. Castro, P. W. Oliveira and H. K. Schmidt

Enhanced mechanical and electrical properties of antimony-doped tin oxide coatings
Semicond Sci Tech 2008, 23, (3), 035013 [01.899 (2007)]

M. R. S. Castro, E. D. Sam, M. Veith and P. W. Oliveira

Structure, wettability and photocatalytic activity of CO_2 laser sintered TiO_2/multi-walled carbon nanotube coatings
Nanotechnology 2008, 19, (10), 105704 [03.310 (2007)]

M. R. S. Castro and H. K. Schmidt

Preparation and characterization of low- and high-adherent transparent multi-walled carbon nanotube thin films
Mater Chem Phys 2008, 111, (2-3), 317-321 [01.871 (2007)]

E. J. De Souza, M. Brinkmann, C. Mohrdieck and E. Arzt

Enhancement of capillary forces by multiple liquid bridges
Langmuir 2008, 24, (16), 8813-8820 [04.009 (2007)]

E. J. De Souza, M. Brinkmann, C. Mohrdieck, A. J. Crosby and E. Arzt

Capillary forces between chemically different substrates
Langmuir 2008, 24, (18), 10161-10168 [04.009 (2007)]

E. J. De Souza, L. Gao, T. J. McCarthy, E. Arzt and A. J. Crosby

Effect of contact angle hysteresis on the measurement of capillary forces
Langmuir 2008, 24, (4), 1391-1396 [04.009 (2007)]

A. del Campo and E. Arzt

Fabrication approaches for generating complex micro- and nanopatterns on polymeric surfaces
Chem Rev 2008, 108, (3), 911-945 [22.757 (2007)]

W. Delb, D. J. Strauss, Y. F. Low, H. Seidler, A. Rheinschmitt, T. Wobrock and R. D'Amelio

Alterations in event related potentials (ERP) associated with tinnitus distress and attention
Applied Psychophysiology and Biofeedback 2008, 33, (4), 211-221 [01.226 (2007)]

J. K. Deuschle, G. Buerki, H. M. Deuschle, S. Enders, J. Michler and E. Arzt

In situ indentation testing of elastomers
Acta Mater 2008, 56, (16), 4390-4401 [03.624 (2007)]

V. Dhayal, R. Bohra, M. Nagar, A. Kaushik, S. Mathur and S. Barth

Low-temperature sol-gel transformation of methyl silicon precursors to silica-based hybrid materials
Appl Organomet Chem 2008, 22, (11), 629-636 [01.224 (2007)]

T. Filleter, K. V. Emtsev, T. Seyller and R. Bennewitz

Local work function measurements of epitaxial graphene
Appl Phys Lett 2008, 93, (13), 133117 [03.596 (2007)]

C. P. Frick, B. G. Clark, S. Orso and E. Arzt
Size effect on strength and strain hardening of [111] nickel sub-micron compression pillars
Mat Sci Eng A-Struct 2008, 489, 319-329 [01.457 (2007)]

C. P. Frick, B. G. Clark, S. Orso, P. Sonnweber-Ribic and E. Arzt
Orientation-independent pseudoelasticity in small-scale NiTi compression pillars
Scripta Mater 2008, 59, (1), 7-10 [02.481 (2007)]

P. A. Gruber, J. Böhm, F. Onuseit, A. Wanner, R. Spolenak and E. Arzt
Size effects on yield strength and strain hardening for ultra-thin Cu films with and without passivation: A study by synchrotron and bulge test techniques
Acta Mater 2008, 56, (10), 2318-2335 [03.624 (2007)]

P. A. Gruber, S. Olliges, E. Arzt and R. Spolenak
Temperature dependence of mechanical properties in ultrathin Au films with and without passivation
J Mater Res 2008, 23, (9), 2406-2419 [01.916 (2007)]

P. A. Gruber, C. Solenthaler, E. Arzt and R. Spolenak
Strong single-crystalline Au films tested by a new synchrotron technique
Acta Mater 2008, 56, (8), 1876-1889 [03.624 (2007)]

F. Guyon, A. Hameau, A. Khatyr, M. Knorr, H. Amrouche, D. Fortin, P. D. Harvey, C. Strohmann, A. L. Ndiaye, V. Huch, M. Veith and N. Avarvari
Syntheses, structures, and photophysical properties of mono- and dinuclear sulfur-rich gold(I) complexes
Inorg Chem 2008, 47, (17), 7483-7492 [04.123 (2007)]

F. Hernandez-Ramirez, J. D. Prades, A. Tarancon, S. Barth, O. Casals, R. Jimenez-Diaz, E. Pellicer, J. Rodriguez, J. R. Morante, M. A. Juli, S. Mathur and A. Romano-Rodriguez
Insight into the role of oxygen diffusion in the sensing mechanisms of SnO$_2$ nanowires
Adv Funct Mater 2008, 18, (19), 2990-2994 [07.496 (2007)]

S. Heusing and M. A. Aegerter
Blue and grey coloring electrochromic devices
J Sol-Gel Sci Techn 2008, 47, (2), 214-215 [01.300 (2007)]

G. Huber, S. Orso, R. Spolenak, U. G. K. Wegst, S. Enders, S. N. Gorb and E. Arzt
Mechanical properties of a single gecko seta
Int J Mater Res 2008, 99, (10), 1113-1118 [00.478 (2007)]

V. Huch, R. Kumar, S. Mathur and R. Ratnani
Trans-dichlorodioxobis(triphenylphosphate) molybdenum(VI), MoO$_2$Cl$_2$[OP(OPh)$_3$]$_2$
J Chem Crystallogr 2008, 38, (8), 605-608 [00.597 (2007)]

L. Jiang, M. T. Perez-Prado, P. A. Gruber, E. Arzt, O. A. Ruano and M. E. Kassner
Texture, microstructure and mechanical properties of equiaxed ultrafine-grained Zr fabricated by accumulative roll bonding
Acta Mater 2008, 56, (6), 1228-1242 [03.624 (2007)]

V. Kozhukharov, S. Kozhukharov, G. Tsaneva, J. Gerwann, M. Schem, T. Schmidt and M. Veith
Investigation on the corrosion protection ability of nanocomposite hybrid coatings
Bulgarian chemical communications 2008, 40, (3), 310-317 [-]

S. Kozukharov, G. Tsaneva, V. Kozhukharov, J. Gerwann, M. Schem, T. Schmidt and M. Veith
Corrosion protection properties of composite hybrid coatings with involved nanoparticles of zirconia and ceria
J Univ Chem Technol Met 2008, 43, (1), 73-80 [-]

C. Kuemin, T. Kraus, H. Wolf and N. D. Spencer
Matrix effects on the surface plasmon resonance of dry supported gold nanocrystals
Opt Lett 2008, 33, (8), 806-808 [03.711 (2007)]

A. Kurz and M. A. Aegerter
Novel transparent conducting sol-gel oxide coatings
Thin Solid Films 2008, 516, (14), 4513-4518 [01.693 (2007)]

M. Legros, G. Dehm, E. Arzt and T. J. Balk
Observation of giant diffusivity along dislocation cores
Science 2008, 319, (5870), 1646-1649 [26.372 (2007)]

E. J. Lima, A. D. Arelaro, H. R. Rechenberg, E. L. Duarte, R. Itri, C. Cavelius, H. Shen, S. Mathur and G. F. Goya
Magnetic characterization of ferrihydrite nanoparticles synthesized by hydrolysis of metal-organic precursor
Physica B 2008, 403, (23-24), 4156-4159 [00.751 (2007)]

E. J. Lima, T. B. Martins, H. R. Rechenberg, G. F. Goya, C. Cavelius, R. Rapalaviciute, H. Shen and S. Mathur
Numerical simulation of magnetic interactions in polycrystalline YFeO$_3$
J Magn Magn Mater 2008, 320, (5), 622-629 [01.704 (2007)]

H. Lin, P. W. Oliveira and M. Veith

Ionic liquid as additive to increase sensitivity, resolution, and diffraction efficiency of photopolymerizable hologram material
Appl Phys Lett 2008, 93, (14), 141101 [03.596 (2007)]

S. Lu and H. K. Schmidt

Photoluminescence and XPS analyses of Mn^{2+} doped ZnS nanocrystals embedded in sol-gel derived hybrid coatings
Mater Res Bull 2008, 43, (3), 583-589 [01.484 (2007)]

W. Lu and H. K. Schmidt

Lyothermal synthesis of nanocrystalline $BaSnO_3$ powders
Ceram Int 2008, 34, (3), 645-649 [01.360 (2007)]

W. Lu and H. K. Schmidt

Synthesis of tin oxide hydrate ($SnO_2 \cdot xH_2O$) gel and its effects on the hydrothermal preparation of $BaSnO_3$ powders
Adv Powder Technol 2008, 19, (1), 1-12 [00.494 (2007)]

S. Mathur and S. Barth

One-dimensional semiconductor nanostructures: Growth, characterization and device applications
Z Phys Chem 2008, 222, (2-3), 307-317 [01.112 (2007)]

S. Mathur, S. Barth, U. Werner, F. Hernandez-Ramirez and A. Romano-Rodriguez

Chemical vapor growth of one-dimensional magnetite nanostructures
Adv Mater 2008, 20, (8), 1550-1554 [08.191 (2007)]

S. Mathur, T. Rügamer, N. Donia and H. Shen

Functional metal oxide coatings by molecule-based thermal and plasma chemical vapor deposition techniques
J Nanosci Nanotechno 2008, 8, (5), 2597-2603 [01.987 (2007)]

R. M. McMeeking, E. Arzt and A. G. Evans

Defect dependent adhesion of fibrillar surfaces
J Adhes 2008, 84, (7), 675-681 [01.000 (2007)]

K. Moh, S. Schumacher and M. Veith

Wet chemical syntheses of Ag-nanoparticles
Annual Report, Jahresbericht 2007 - INM Leibniz-Institut für Neue Materialien 2008, 75-79 [-]

M. T. Northen, C. Greiner, E. Arzt and K. L. Turner

A gecko-inspired reversible adhesive
Adv Mater 2008, 20, (20), 3905-3909 [08.191 (2007)]

C. Piana, S. Toegel, I. Güll, S. Gerbes, H. Viernstein, M. Wirth and F. Gabor

Growth surface-induced gene and protein expression patterns in Caco-2 cells
Acta Biomater 2008, 4, (6), 1819-1826 [03.113 (2007)]

C. Piana, M. Wirth, S. Gerbes, H. Viernstein, F. Gabor and S. Toegel

Validation of reference genes for qPCR studies on Caco-2 cell differentiation
Eur J Pham Biopharm 2008, 69, (3), 1187-1192 [02.611 (2007)]

J. D. Prades, R. Jimenez-Diaz, F. Hernandez-Ramirez, S. Barth, A. Cirera, A. Romano-Rodriguez, S. Mathur and J. R. Morante

Ultralow power consumption gas sensors based on self-heated individual nanowires
Appl Phys Lett 2008, 93, (12), 123110 [03.596 (2007)]

J. D. Prades, R. Jimenez-Diaz, F. Hernandez-Ramirez, L. Fernandez-Romero, T. Andreu, A. Cirera, A. Romano-Rodriguez, A. Cornet, J. R. Morante, S. Barth and S. Mathur

Toward a systematic understanding of photodetectors based on individual metal oxide nanowires
J Phys Chem C 2008, 112, (37), 14639-14644 [-]

S. Promnimit, C. Cavelius, S. Mathur and J. Dutta

Growth of gold/zinc sulphide multilayer films using layer-by-layer assembly of colloidal nanoparticles
Physica E 2008, 41, (2), 285-291 [00.834 (2007)]

J. Pütz and M. A. Aegerter

Direct gravure printing of indium tin oxide nanoparticle patterns on polymer foils
Thin Solid Films 2008, 516, (14), 4495-4501 [01.693 (2007)]

J. Pütz and M. A. Aegerter

Sol-gel coatings on thin glasses
J Sol-Gel Sci Techn 2008, 47, (2), 226-227 [01.300 (2007)]

J. Pütz and M. A. Aegerter

Improvement of surface roughness and electrical properties of sputtered ITO electrodes with a thin sol-gel film
J Sol-Gel Sci Techn 2008, 47, (2), 227-228 [01.300 (2007)]

J. Pütz and M. A. Aegerter

Wet chemical deposition of transparent conducting coatings in glass tubes
J Sol-Gel Sci Techn 2008, 47, (2), 228-230 [01.300 (2007)]

J. Pütz, S. Heusing and M. A. Aegerter

Gravure printing of nanoparticulate coatings for display application
J Sol-Gel Sci Techn 2008, 47, (2), 230-231 [01.300 (2007)]

M. Quilitz, M. Schubert and M. Veith
Neue Entwicklungen der chemischen Nanotechnologie. Innovative Schichtsysteme
JOT - Journal für Oberflächentechnik 2008, 48, (7), 8-10 [-]

S. Ren, W. Kochanek, H. Bolz, M. Wittmar, I. Grobelsek and M. Veith
Combinatorial preparation of solid-state materials by injection moulding
J Eur Ceram Soc 2008, 28, (16), 3005-3010 [01.562 (2007)]

M. Schubert
Die Veredelung des Edlen. Materialoptimierung mit Hilfe von Nanotechnologie
medAmbiente 2008, 2, 32-33 [-]

N. Sharma, V. Sharma, M. Nagar, R. Bohra, A. Kaushik, S. Mathur and S. Barth
Glycol modified cis-diisopropoxy-bis (N-phenylsalicylideneiminato)zirconium(IV): syntheses, characterization and low temperature transformation to nanocrystalline zirconia
J Coord Chem 2008, 61, (14), 2234-2245 [00.867 (2007)]

A. V. Spuskanyuk, R. McMeeking, V. S. Deshpande and E. Arzt
The effect of shape on the adhesion of fibrillar surfaces
Acta Biomater 2008, 4, (6), 1669-1676 [03.113 (2007)]

P. Stegmaier, J. M. Alonso and A. del Campo
Photoresponsive surfaces with two independent wavelength-selective functional levels
Langmuir 2008, 24, (20), 11872-11879 [04.009 (2007)]

D. J. Strauss, W. Delb, R. D'Amelio, Y. F. Low and P. Falkai
Objective quantification of the tinnitus decompensation by synchronization measures of auditory evoked single sweeps
IEEE Trans Neural Syst Rehabil Eng 2008, 16, (1), 74-81 [02.489 (2007)]

G. Tsaneva, V. Kozhukharov, S. Kozukharov, M. Ivanova, J. Gerwann, M. Schem and T. Schmidt
Functional nanocomposite coatings for corrosion protection of aluminum alloy and steel
J Univ Chem Technol Met 2008, 43, (2), 231-238 [-]

M. Veith
Precursorchemistry with metalalkoxides and their use for nano-scaled materials
Z Phys Chem 2008, 222, (2-3), 287-305 [01.112 (2007)]

M. Veith
New synthetic routes to nano-composites with ceramic particles, using lanthanide compounds
J Sol-Gel Sci Techn 2008, 46, (3), 291-298 [01.300 (2007)]

M. Veith, A. Altherr and M. Klook
Geräte und Anlagen für neue Lacke mit nanoskaligem Zirkondioxid
Chem-Ing-Tech 2008, 80, (11), 1705-1710 [00.401 (2007)]

M. Veith, C. Belot, L. Guyard, V. Huch, M. Knorr and M. Zimmer
Synthesis and crystal structure investigations of trivalent rare earth (Y^{3+}, Nd^{3+}, Er^{3+}) thienyl-substituted methoxides
Eur J Inorg Chem 2008, (15), 2397-2406 [02.597 (2007)]

M. Veith, A. L. Ndiaye, V. Huch, H. Cui and C. Wickleder
Luminescent study on Nd^{3+} complexes containing carboxylate-dithiolene and akoxide-dithiolene ligands
Z Anorg Allg Chem 2008, 634, (14), 2551-2556 [01.260 (2007)]

M. Veith, C. Petersen, O. C. Aktas, W. Metzger, M. Oberringer, T. Pohlemann, M. Müller and S. Gerbes
Chemical vapour deposition of aluminium based micro- and nanostructured surfaces for biological applications
Mater Lett 2008, 62, (23), 3842-3845 [01.625 (2007)]

M. Veith, H. Smail and V. Huch
Reactivity of $[(Ph_2Si)_2O_3]_4[Al(OH)]_4$ and $[(Ph_2Si)_2O_3]_4[Al(OLi)]_4$ towards NaOEt and LiOH. Synthesis of new twelve-membered alumopolysiloxane rings
Z Anorg Allg Chem 2008, 634, (15), 2867-2872 [01.260 (2007)]

M. Veith, E. A. Sow, U. Werner, C. Petersen and O. C. Aktas
The transformation of core/shell aluminium/alumina nanoparticles into nanowires
Eur J Inorg Chem 2008, 2008, (33), 5181-5184 [02.597 (2007)]

M. Veith, K. Valtchev and V. Huch
Tetraalkoxyaluminates of nickel(II), copper(II), and copper(I)
Inorg Chem 2008, 47, (3), 1204-1217 [04.123 (2007)]

M. Zaiser, J. Schwerdtfeger, A. S. Schneider, C. P. Frick, B. G. Clark, P. A. Gruber and E. Arzt
Strain bursts in plastically deforming molybdenum micro- and nanopillars
Philos Mag 2008, 88, (30-32), 3861-3874 [01.486 (2007)]

Proceedings / Konferenzbeiträge

E. Arzt and A. Del Campo
Bio-inspired adhesion devices
In: Solid-state sensors, actuators, and microsystems workshop 2008, June 01-05, 2008, Hilton-Head Island <South Carolina>, K. L. Turner and L. C. Spangler, Eds., Transducer Research Foundation, (2008), pp 26-27

D. Benyoucef and D. J. Strauss

Optimized Computational Instrumentation in Biomedicin: A Review
In: Proceedings of the Third International Conference on Intelligent Information Hiding and Multimedia Signal Processing of the IEEE (IIHMSP), November 26-28, 2007, Kaohsiung City <Taiwan>, B.-Y. Liao, Ed., (2008), pp 217-220

M. Busse, Y. F. Low, F. I. Corona-Strauss, W. Delb and D. J. Strauss

Neurofeedback by neural correlates of auditory selective attention as possible application for tinnitus therapies
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 5136-5139

F. I. Corona-Strauss, W. Delb, M. Bloching and D. J. Strauss

On the single sweep processing of auditory brainstem responses: Click vs. chirp stimulations and active vs. passive electrodes
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 4166-4169

W. Delb, Y. F. Low, F. I. Corona-Strauss and D. J. Strauss

Fast detection of ABR-responses using neural synchronization stability and single sweep analysis
In: 8th EFAS Congress, 10th Congress of the German Society of Audiology, June 06-09, 2007, Heidelberg, (2008), pp 120 (CD-ROM)

W. Delb, Y. F. Low, F. I. Corona-Strauss and D. J. Strauss

Auditory attention and tinnitus: the objective determination of tinnitus decompensation
In: 8th EFAS Congress, 10th Congress of the German Society of Audiology, June 06-09, 2007, Heidelberg, (2008), pp 109 (CD-ROM)

A. R. Harris, K. Schwerdtfeger and D. J. Strauss

Optimized shift-invariant wavelet packet feature extraction for electroencephalographic evoked responses
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 2685-2688

A. R. Harris and D. J. Strauss

Adaptive frame decomposition in slow wave cortical responses to transcranial magnetic stimulation
In: Proceedings of the Third International Conference on Intelligent Information Hiding and Multimedia Signal Processing of the IEEE (IIHMSP), November 26-28, 2007, Kaohsiung City <Taiwan>, B.-Y. Liao, Ed., (2008), pp 201-204

S. Heusing, P. W. Oliveira, E. Kraker, A. Haase, C. Palfinger and M. Veith

Development of printed ITO coatings on PET and PEN foil for flexible organic photodiodes
In: Organic Optoelectronics and Photonics III, April 07, 2008, Strasbourg <France>, P. L. Heremans, M. Muccini and E. A. Meulenkamp, Eds., SPIE, (2008), p 69992I

M. H. Jilavi, P. W. Oliveira, E. D. Sam, U. Werner and M. Veith

An overview for production and characterization of reflective and antireflective multilayer coatings by sol gel method on glass, sapphire, quartz and plastic foils
In: 4. Thüringer Grenz- und Oberflächentage & 1. Thüringer Kolloquium "Dünne Schichten in der Optik", September 16-17, 2008, Jena, (2008), pp 45-50

H. Lin, P. W. Oliveira and M. Veith

Ionic liquids in photopolymerizable hologram materials
In: 4. Thüringer Grenz- und Oberflächentage & 1. Thüringer Kolloquium "Dünne Schichten in der Optik", September 16-17, 2008, Jena, (2008), pp 288-289

Y. F. Low, H. Argstatter, H. V. Bolay and D. J. Strauss

Evaluation of a compact tinnitus therapy by electrophysiological tinnitus decompensation measures
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 5132-5135

M. Mariam, W. Delb, F. I. Corona-Strauss, M. Bloching and D. J. Strauss

Extraction of habituation correlates in single sweeps sequences of auditory evoked late potentials using time-scale coherence: Objective detection of uncomfortable loudness level
In: Proceedings of the Sixth IASTED International Conference on Biomedical Engineering, February 13-15, 2008, Innsbruck, A. Hierlemann, Ed., (2008), pp 83-87

M. Mariam, W. Delb, A. R. Harris, M. Bloching and D. J. Strauss

Detection of the uncomfortable loudness level by electroencephalographic data: A novelty detection approach using habituation correlates
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 4976-4979

S. Mathur, E. Hemmer, S. Barth, J. Altmayer, N. Donia, I. Kumakiri, N. Lecerf and R. Bredesen

Microporous ZrO_2 membrane preparation by liquid-injection MOCVD

In: Nanostructured materials and nanotechnology, Proceedings of 31st International Conference on Advanced Ceramics and Composites, January 21-26, 2007, Daytona Beach <Florida, USA>, S. Mathur and J. Salem, Eds., (2008), pp 165-173

S. Mathur and M. Singh
Ceramic Engineering and Science Proceedings: Preface
In: Nanostructured materials and nanotechnology, Proceedings of 31st International Conference on Advanced Ceramics and Composites, January 21-26, 2007, Daytona Beach <Florida, USA>, S. Mathur and J. Salem, Eds., (2008), pp IX-X

I. Mustaffa, C. Trenado, K. Schwerdtfeger and D. J. Strauss
Evoked response denoising using nonlinear diffusion filtering of single-trial matrix representations
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 3999-4002

A. Ramachandran, W. Poppendieck, K. P. Koch, N. Donia and S. Mathur
Investigations on the stability of platinum nanostructures on implantable microelectrodes - A first approach
In: Nanostructured materials and nanotechnology, Proceedings of 31st International Conference on Advanced Ceramics and Composites, January 21-26, 2007, Daytona Beach <Florida, USA>, S. Mathur and J. Salem, Eds., (2008), pp 65-72

P. Rogin, J. Hartmann, K. Schmitt, P. W. Oliveira and M. Veith
Thermische Verdichtung von niedrig schmelzenden Glasfilmen auf Polyimidfolie
In: Proceedings der 82. Glastechnische Tagung, May 19-21, 2008, Hameln, (2008), pp CD-ROM

A. Romano-Rodriguez, F. Hernandez-Ramirez, R. Jimenez-Diaz, J. D. Prades, O. Casals, S. Barth and S. Mathur
Gas sensing devices based on 1D Metal-oxide nanostructures: Fabrication, testing and device integration
In: Dielectrics for nanosystems 3: materials science, processing, reliability, and manufacturing, May 18-22, 2008 Phoenix <Arizona, USA>, D. Misra, Ed., (2008), pp 57-64

A. Romano-Rodriguez, F. Hernandez-Ramirez, J. D. Prades, A. Tarancon, O. Casals, R. Jimenez-Diaz, M. A. Juli, J. R. Morante, S. Barth, S. Mathur, A. Helwig, J. Spannhake and G. Mueller
Bottom-up fabrication of individual SnO$_2$ nanowires-based gas sensors on suspended micromembranes
In: Microelectromechanical Systems - Materials and Devices. 2007 MRS Fall Meeting, November 26-28, 2007, San Francisco <Calif., USA>, Materials Research Society, (2008), pp 291-296

M. Schem, T. Schmidt, H. Caparrotti, M. Aslan, M. Wittmar and M. Veith
The use of Al$_2$O$_3$-micro containers for storage of corrosion inhibitors
In: Managing corrosion for sustainability : EUROCORR 2008, the European Corrosion Congress, September 07-11, 2008, Edinburgh <UK>, (2008), p 50

D. J. Strauss, F. I. Corona-Strauss and M. Froehlich
Objective estimation of the listening effort: Towards a neuropsychological and neurophysical model
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 1777-1780

C. Trenado, L. Haab and D. J. Strauss
An integrative multiscale modeling approach to the study of tinnitus decompensation neural correlates
In: Proceedings of the 30th Annual International Conference of the IEEE Engineering in Medicine and Biology Society : "Personalized Healthcare through Technology", August 20-24, 2008, Vancouver <Canada>, (2008), pp 2473-2476

C. Trenado, D. J. Strauss and M. Wittmar
Hybrid multiscale modeling of corrosion nanoinhibitors transport
In: 2. Europäische COMSOL Conference, November 04-06, 2008, Hannover, (2008), pp CD-ROM

R. Würz, A. Eicke, O. Yazdani-Assl, F. Händel and E. Häberle
Diffusions- und Isolationsbarrieren für flexible Cu(In,Ga)Se$_2$-Dünnschichtsolarzellen auf unlegiertem Stahlsubstrat
In: 4. Thüringer Grenz- und Oberflächentage & 1. Thüringer Kolloquium „Dünne Schichten in der Optik", September 16-17, 2008, Jena, (2008), pp 40-44

Publications in magazines / Magazinartikel

M. Amlung and P. W. Oliveira
Sauber und sicher - Neue Schutzschichten für Glas und Stahl
Glaswelt 2008, (10), 50

J. Flackus and M. Geerkens
Technologietransfer auf Augenhöhe. Forschungsinstitute bauen Brücken zwischen Grundlagenforschung und industrieller Anwendung
Empowering Nano 2008, (4), 23-25

S. Mathur, S. Barth, F. Hernandez-Ramirez, A. Romano-Rodriguez and J. D. Prades
One-dimensional oxide nanostructures: Growth, applications and devices
MST news 2008, (3), 6-9

M. Quilitz, S. Heusing and P. W. Oliveira

Strom in unsichtbaren Leitern
Empowering Nano 2008, (4), 20-22

M. Quilitz, M. Schubert and M. Veith

Chemische Nanotechnologie. Anwendungen in industriellen Beschichtungen
nanoworld 2008, (1), 38-40

M. Schubert

Alle Jahre am INM - Sommerschule Chemische Nanotechnologie
nanotechnologie aktuell 2008, (1), 76-79

M. Schubert

Mit Mikro und Nano zu neuen Technologien
MiNaT Journal 2008, 2, 3

Posters / Poster

M. Amlung, P. W. Oliveira and M. Veith

New glass-ceramic coatings with corrosion protection and anti-adhesive properties
7th International Conference on Coatings on Glass and Plastics (ICCG7), June 15-19, 2008, Eindhoven/Veldhoven <Netherlands>

K. Brörmann and R. Bennewitz

Friction on microstructured surfaces
12. Deutsche Physikerinnentagung, November 06-11, 2008, Münster

M. R. S. Castro, P. W. Oliveira and M. Veith

Sintering of TiO$_2$/CNT coatings in a reducing N$_2$:H$_2$ atmosphere
Graphene Week 2008, August 25-29, 2008, Trieste <Italy>

P. Egberts, T. Filleter and R. Bennewitz

FM-Kelvin probe study of nanoindentations on KBr(100)
11th International Conference on Non-contact Atomic Force Microscopy: NCAFM-2008, September 16, 2008, Madrid <Spain>

S. Heusing, P. W. Oliveira, A. Haase, E. Kraker and M. Veith

Development of printed ITO coatings on PET and PEN foil for flexible organic photodiodes
Organic optoelectronics and photonics III, SPIE, 6999, April 07-10, 2008, Strasbourg <France>

E. Kroner

Bioinspired adhesion systems
Junior EuroMat 2008, July 14-18, 2008, Lausanne <Schwitzerland>

A. Labuda, N. N. Gosvami, B. Pietrobon, Y. Guo, P. Grutter, B. Lennox and R. Bennewitz

Atomic-scale friction study of gold surfaces in electrochemical environment
International Nanotribology Forum, NANO-SIKKIM-III: Mechanics and Friction at the Nanoscale, November 10-14, 2008, Pelling <Sikkim, India>

S. Mathur

Functional nanostructures through chemistry: Processing and device applications
TFOC, Transatlantic Frontiers of Chemistry Symposium, Cranage Hal, July 31-August 03, 2008, Cheshire <UK>

A. L. Ndiaye, M. Knorr and M. Veith

NIR and visible luminescents lanthanides (Ln^{3+})-dithiolene complexes
XXI. Tage der Seltenen Erden (Terrae Rarae) 2008, December 04-06, 2008, Bochum

J. Pan, J. Altmayer, J. Wei, N. Donia, H. Shen and S. Mathur

Controlled synthesis and growth mechanism study of tin oxide nanowire arrays
MSE Materials Science and Engineering, September 01-04, 2008, Nürnberg

J. D. Prades, R. Jimenez-Diaz, F. Hernandez-Ramirez, L. Fernandez-Romero, T. Andreu, A. Cirera, A. Cornet, J. R. Morante, S. Barth, S. Mathur and A. Romano-Rodriguez

Design rules and modeling of photodetectors based on individual nanowires
MRS Fall Meeting, December 01-05, 2008, Boston <Mass., USA>

J. D. Prades, M. Manzanares, R. Jimenez-Diaz, F. Hernandez-Ramirez, T. Andreu, A. Cirera, J. R. Morante, S. Barth, S. Mathur and A. Romano-Rodriguez

Chemical electrical transduction phenomena in single SnO$_2$ nanowires at room temperature under UV illumination
MRS Fall Meeting, December 01-05, 2008, Boston <Mass., USA>

B. Rabung and M. Veith

Electrochemical synthesis of differently shaped nanocrystalline ZnO in an aqueous system
107. Bunsentagung, May 01-03, 2008, Saarbrücken, p P 111

Talks / Vorträge

J. Adam

Verschiedene chemische Verfahren zur Herstellung von Nanopartikeln. a) Methoden zur Oberflächenmodifikation und Dispergierung, b) Top-down: Nassmahlverfahren
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 17-18, 2008; Saarbrücken
Eingeladener Vortrag

J. Adam, M. Aslan, R. Drumm and M. Veith

Mechanisms of bonding effected by nanoparticles in zirconia coatings applied by spraying of suspensions
Summer School “Chemische Nanotechnologie”; October 07, 2008; Saarbrücken
Eingeladener Vortrag

E. Arzt

Mechanical size effects in sub-micron dimensions
Physics Colloquium, Saarland University; January 24, 2008; Saarbrücken
Eingeladener Vortrag

E. Arzt

Haftungseigenschaften von Mikrostrukturen
54. Metallkunde-Kolloquium; April 14, 2008; Lech/Arlberg <Austria>
Eingeladener Vortrag

E. Arzt

Klein, stark, smart - durch Skaleneffekte zu neuen Materialien
Antrittsvorlesung, Universität des Saarlandes; April 18, 2008; Saarbrücken
Eingeladener Vortrag

E. Arzt

Biomimetic functional surfaces: lessons from files and geckoes
Vaalsbroek Synthetic Fiber Talks; May 08, 2008; Vaalsbroek <Netherlands>
Eingeladener Vortrag

E. Arzt

Bioinspired adhesion devices
The 2008 Solid-State Sensor, Actuator and Microsystems Workshop; June 01-03, 2008; Hilton-Head Island <South Carolina, USA>
Eingeladener Vortrag

E. Arzt

Bioinspired adhesion surfaces
1st International Symposium on Functional Surfaced, Volkswagen-Stiftung; June 19, 2008; Bremen
Eingeladener Vortrag

E. Arzt

Development of gecko surfaces
Gecko NiRT-Meeting, UCSB; July 09-15, 2008; Santa Barbara <Calif., USA>
Eingeladener Vortrag

E. Arzt

Kooperation Wissenschaft - Industrie am Beispiel des INM
German Scholar Organisation (GSO); October 16-19, 2008; San Francisco <Calif., USA>
Eingeladener Vortrag

E. Arzt

Biomimetische Oberflächen
Gesellschaft Deutscher Chemiker; October 23-24, 2008; Dresden
Eingeladener Vortrag

E. Arzt

Biomimetic adhesion systems
Hoffmann-La Roche; November 05-06, 2008; Beatenberg <Switzerland>
Eingeladener Vortrag

E. Arzt

Naturwissenschaftler/Ingenieure in Lehre und Forschung
Workshop an der KWT; November 24, 2008; Saarbrücken
Eingeladener Vortrag

E. Arzt

Alles Nano?! Forschung am Unsichtbaren
Leibniz-Frühstück; December 09, 2008; Berlin
Eingeladener Vortrag

E. Arzt

Surface templates for enhanced adhesion
SSSI 2008 - Self-assembly and Self-organization at Surfaces and Interfaces; December 09-12, 2008; Cambridge <USA>
Eingeladener Vortrag

S. Barth, S. Mathur, F. Hernandez-Ramirez, S. Estrade and A. Romano-Rodriguez

Molecule-based chemical vapor deposition of one-dimensional oxide nanostructures: Growth, characterization and devices
MRS Fall Meeting; December 01-05, 2008; Boston <Mass., USA>

C. Becker-Willinger

Nanostructured materials for industrial applications
Nanoroadshow 2008; October 20-26, 2008; Buenos Aires <Argentina>

C. Becker-Willinger, G. Heppe, F. Hollmann, P. Kalmes, C. Kasper, M. Wittmar, A. Altherr and M. Veith

Nanomers: Functional coatings and nanocomposites for industry
Nanotech 2008; February 13-15, 2008; Tokyo <Japan>

C. Becker-Willinger, G. Heppe, P. Kalmes, S. Albayrak, F. Hollmann, S. Schmitz-Stöwe, P. W. Oliveira and M. Veith

Neue Oberflächeneigenschaften auf Kunststoffen über Nanopartikel
VDI-Seminar 355803, Oberflächen von spritzgegossenen Kunststoffteilen; April 02, 2008; Mannheim

C. Becker-Willinger, F. Hollmann, G. Heppe, S. Albayrak, P. Kalmes, P. W. Oliveira and M. Veith

Nanostructured materials for industrial applications
Nanokorea 2008, KINTEX; August 27-29, 2008; Seoul <South Korea>

C. Becker-Willinger and S. Schmitz-Stöwe

Nanopartikel in Polymeren
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

R. Bennewitz

Atome bei der Arbeit: Experimente zu den mikroskopischen Phänomenen der Reibung
Allgemeines Physikalisches Kolloquium, Fachbereich Physik, Universität Münster; June 06, 2008; Münster
Eingeladener Vortrag

R. Bennewitz

Atome bei der Arbeit: Experimente zu den mikroskopischen Phänomenen der Reibung
Physikalisches Kolloquium, Fachrichtung Physik, Universität des Saarlandes; July 03, 2008; Saarbrücken
Eingeladener Vortrag

R. Bennewitz

Atomic friction of ultrathin KBr films and graphene
European Conference on Surface Science (ECOSS25); July 07, 2008; Liverpool <UK>
Eingeladener Vortrag

R. Bennewitz

Atomic friction of ultrathin films
IBM Zurich Research Center; August 25, 2008; Zürich
Eingeladener Vortrag

R. Bennewitz

Kelvin probe force microscopy measurements of epitaxial graphene
11th International Conference on Non-contact Atomic Force Microscopy: NCAFM-2008; September 17, 2008; Madrid <Spain>
Eingeladener Vortrag

R. Bennewitz

Force microscopy of epitaxial graphene: Work function and friction contrasts
DFG Rundgespräch Graphene, Kloster Banz; September 25, 2008; Bad Staffelstein

R. Bennewitz

Friction experiments from epitaxial graphene to rough elastomers
Seminar der Kondensierten Materie, Universität Basel; September 29, 2008; Basel
Eingeladener Vortrag

R. Bennewitz

Moiré patterns and contacts driven out of equilibrium: What high-resolution AFM can do for the Graduiertenkolleg
Seminar des Graduiertenkolleg 1276/1 "Strukturbildung und Transport in komplexen Systemen", Universität des Saarlandes; October 28, 2008; Saarbrücken
Eingeladener Vortrag

R. Bennewitz

Dissipation in single-asperity experiments
International Nanotribology Forum, NANO-SIKKIM-III: Mechanics and Friction at the Nanoscale; November 10-14, 2008; Pelling <Sikkim, India>
Eingeladener Vortrag

R. Bennewitz

Friction experiments from epitaxial graphene to rough elastomers
Max-Planck-Institut; December 15, 2008; Stuttgart
Eingeladener Vortrag

M. Busse, Y. F. Low, F. I. Corona-Strauss, W. Delb and D. J. Strauss

Future for tinnitus therapies: Neurofeedback by neural correlates of auditory selective attention
X. International Conference on Cognitive Neuroscience (ICON X); September 01-05, 2008; Bodrum <Turkey>

M. R. S. Castro, E. D. Sam, M. Veith and P. W. Oliveira

Optical, structural and photocatalytic characterization of laser sintered TiO_2/multi-walled carbon nanotubes composites
32nd International Conference on Advanced Ceramics and Composites; January 27-February 1, 2008; Daytona Beach <Florida, USA>

A. del Campo

Aktive Oberflächen mit steuerbarer Haftung
NanoSurface 2008: Biochemisch und chemisch aktive Oberflächen; August 28, 2008; Gießen
Eingeladener Vortrag

A. del Campo

Active surfaces with tunable adhesion
Marie Curie Conference "Synthesis and applications of self-assembling materials at nano-scale"; April 14-18, 2008; Funchal <Madeira, Portugal>
Eingeladener Vortrag

A. del Campo and E. Arzt

Patterned adhesive surfaces: recent advances
CIMTEC Conference: Smart Materials, Structures and Systems; June 08-13, 2008; Acireale <Italy>
Eingeladener Vortrag

N. Donia

Chemische Gasphasenabscheidung
GDCh Workshop „Chemische Nanotechnologie"; June 17-18, 2008; Saarbrücken
Eingeladener Vortrag

R. Drumm

Kolloidales Processing von ZrO_2-Nanopartikeln in Rührwerkskugelmühlen
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

S. Gerbes

Neue Materialien für LifeScience-Anwendungen
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

S. Gerbes and T. S. Müller

Nanotechnology at INM
Workshop "Co-operations under the framework of FP7", Confindustria Padove; March 27-28, 2008; Padova <Italy>

A. Haase, E. Kraker, J. Krenn, C. Palfinger, S. Heusing, P. W. Oliveira and M. Veith

Organic photodiodes on flexible substrates
International Symposium on Flexible Electronics (ISFE); April 06-09, 2008; Tarragona <Spain>

A. Haase, E. Kraker, C. Palfinger, A. Fian, J. Krenn, S. Heusing, P. W. Oliveira and M. Veith

Organic photodiodes on flexible substrates
2nd International Symposium on Transparent Conductive Oxides (TCO 2008); October 22-26, 2008; Hersonissos <Crete, Greece>

E. Hemmer, S. Mathur, V. Colquhoun and C. Cavelius

Synthesis of new lanthanide precursors for the preparation of lanthanide containing nanomaterials by solvothermal processes
32nd International Conference on Advanced Ceramics and Composites; January 27-February 1, 2008; Daytona Beach <Florida, USA>

S. Heusing

Electrochromic coatings and windows
DPG-Frühjahrstagung; February 25-29, 2008; Berlin
Eingeladener Vortrag

S. Heusing, P. W. Oliveira, E. Kraker, A. Haase, C. Palfinger and M. Veith

Wet chemical deposited ITO coatings on flexible substrates for organic photodiodes
2nd International Symposium on Transparent Conductive Oxides (TCO 2008); October 22-26, 2008; Hersonissos <Crete, Greece>

R. Jimenez-Diaz, F. Hernandez-Ramirez, M. A. Juli, J. D. Prades, S. Barth, O. Casals, T. Andreu, A. Tarancon, J. R. Morante, E. Pellicer, A. Helwig, J. Spannhake, G. Mueller, S. Mathur and A. Romano-Rodriguez

Design and fabrication of a portable prototype of gas sensor and UV photodetector based on individual semiconductor nanowires
MRS Spring Meeting; March 24-28, 2008; San Francisco <Calif., USA>

M. Koch

Der Lotus-Effekt aus Sicht des ESEMs
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

A. Kraegeloh

Silbernanopartikel in Wecheselwirkung mit Zellen
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

T. Kraus

Transport in templated particle assembly
Directed Assembly of Functional Materials and Devices. National Institute of Standards and Technology; March 19-21, 2008; Gaithersburg <Maryland, USA>
Eingeladener Vortrag

T. Kraus

How to make gradients by directed mass transport - and how to use them to direct mass transport
Gordon Research Conference on Surface Gradients; July 14-18, 2008; Waterville Valley <NH, USA>
Eingeladener Vortrag

T. Kraus

Ordered materials from disordered colloids
IBB, Georgia Institute of Technology; July 21, 2008; Atlanta <GA, USA>
Eingeladener Vortrag

T. Kraus

Particle assembly for materials synthesis
Graduiertenkolleg 1276/1 „Strukturbildung und Transport in komplexen Systemen". Universität des Saarlandes; April 15, 2008; Saarbrücken
Eingeladener Vortrag

E. Kroner

Was wir vom Gecko lernen können
Tag der Offenen Tür der Universität des Saarlandes; June 21, 2008; Saarbrücken

S. Mathur, J. Altmayer, T. Rügamer, N. Donia, C. Cavelius and J. Kondratiuk

Nanostructured surfaces through chemical nanotechnology: Opportunities and applications
2nd IEEE International Nanoelectronics Conference (INEC 2008) in conjunction with Shanghai Nanophotonics & Electronics Forum; March 24-27, 2008; Shanghai <China>
Eingeladener Vortrag

S. Mathur, S. Barth, F. Hernandez-Ramirez, J. D. Prades and A. Romano-Rodriguez

Growth of one-dimensional metal oxide nanostructures and nanowire-based devices
MRS Fall Meeting; December 01-05, 2008; Boston <Mass., USA>
Eingeladener Vortrag

S. Mathur, S. Barth, T. Rügamer, H. Shen, R. Ganesan and P. Born

Metal oxide nanowires: Growth, applications and devices
2nd IEEE International Nanoelectronics Conference (INEC 2008) in conjunction with Shanghai Nanophotonics & Electronics Forum; March 24-27, 2008; Shanghai <China>
Eingeladener Vortrag

S. Mathur, N. Donia and J. Altmayer

Protective and bio-compatible nanostructured surfaces by CVD techniques: Controlled modulation of surface and phase structures
European Coatings Conference Medical Coatings & Adhesives; April 10-11, 2008; Berlin
Eingeladener Vortrag

S. Mathur, R. V. Ganesan, N. Donia, S. Barth, T. Rügamer and P. Born

Enhanced ethanol sensing by SnO_2 nanowires functionalized with (Sn/Au) metal additives through post-deposition plasma treatment
32nd International Conference on Advanced Ceramics and Composites; January 27-February 1, 2008; Daytona Beach <Florida, USA>
Eingeladener Vortrag

K. Moh

Core-Shell-Nanopartikel
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

T. S. Müller

Utilizing the knowledge about co-operation building a bow between R&D partners
Interdisziplinärer Workshop "Interdisziplinärer Austausch, gemeinsame Ideen für Neuentwicklungen", Institut für Angewandte Physik; September 05, 2008; Bern <Switzerland>
Eingeladener Vortrag

T. S. Müller

Utilizing the knowledge about co-operation building a bow between R&D partners
Hot Spot WGL - MiNaT, Internationale Fachmesse für Feinwerktechnik, Ultrapräzision, Mikro- und Nanotechnologie; October 08, 2008; Stuttgart
Eingeladener Vortrag

A. L. Ndiaye and M. Veith

Luminescents properties in dithiolene complexes
Clustertreffen im Rahmen des SPP1166-Projekts; July 10, 2008; Siegen

P. W. Oliveira, M. H. Jilavi, E. D. Sam, U. Werner and M. Veith

Development of nano particles for optics applications
32nd International Conference on Advanced Ceramics and Composites; January 27-February 1, 2008; Daytona Beach <Florida, USA>

P. W. Oliveira, T. S. Müller and M. Quilitz

Nanoparticles for optics
Unileste - Centro Universitario do Leste de Minas Gerais; June 04, 2008; Coronel Fabriciano, Minas Gerais
Eingeladener Vortrag

J. D. Prades, R. Jimenez-Diaz, F. Hernandez-Ramirez, S. Barth, O. Casals, A. Tarancon, T. Andreu, M. A. Juli, A. Cirera, A. Cornet, A. Peret-Rodriguez, S. Mathur, A. Romano-Rodriguez and J. R. Morante

Extraction of the electrical parameters of individual metal oxide nanowires using two-point probing to FIB-fabricated platinum electrodes
MRS Spring Meeting; March 24-28, 2008; San Francisco <Calif., USA>

J. Pütz

Nasschemische Beschichtung und Strukturierung
Nanokomposite Beschichtungen. OTTI-Seminar; February 27-28, 2008; Regensburg
Eingeladener Vortrag

J. Pütz

Nanokomposite Farbschichten
Nanokomposite Beschichtungen. OTTI-Seminar; February 27-28, 2008; Regensburg
Eingeladener Vortrag

J. Pütz

Einführung - Gedruckte Elektronik
Gedruckte Elektronik. OTTI-Seminar; March 03-04, 2008; Regensburg
Eingeladener Vortrag

M. Quilitz

Chemische Nanotechnologie und das INM
Partnering Day am Werkstoffimpulszentrum (IZW) Leoben; September 11, 2008; Leoben <Austria>
Eingeladener Vortrag

M. Quilitz

Chemical nanotechnology for glass and optics
tp21 Workshop: Technology for industry, Science Park 2; September 18, 2008; Saarbrücken

M. Quilitz and P. W. Oliveira

Chemische Nanotechnologie für den Klima- und Umweltschutz
Deutsch-Argentinische Woche der wissenschaftlichen und technologischen Kooperation Innovationsforum; November 14, 2008; Buenos Aires <Argentina>
Eingeladener Vortrag

M. Quilitz, P. W. Oliveira and C. Becker-Willinger

Neue Oberflächen über Chemische Nanotechnologie
Radical Surface Innovation Workshop; March 12, 2008; Bad Wurzach
Eingeladener Vortrag

M. Quilitz and H. Thedens

Charakterisierung nanoskaliger Pulver mit Röntgenstrahlen
GDCh-Kurs „Chemische Nanotechnologie“; June 17-18, 2008; Saarbrücken
Eingeladener Vortrag

P. Rogin, J. Hartmann, K. Schmitt, P. W. Oliveira and M. Veith

Thermische Verdichtung von niedrig schmelzenden Glasfilmen auf Polyimidfolie
82. Glastechnische Tagung; May 19-21, 2008; Hameln

A. Romano-Rodriguez, F. Hernandez-Ramirez, R. Jimenez-Diaz, J. D. Prades, O. Casals, E. Pellicer, A. Tarancon, S. Barth and S. Mathur

Individual metal oxide nanowire; fundamental properties and device applications
Materials Science and Engineering Conference; September 01-04, 2008; Nürnberg

A. Romano-Rodriguez, J. D. Prades, R. Jimenez-Diaz, A. Cirera, O. Casals, J. R. Morante, X. Illa, T. Andreu, S. Barth, F. Hernandez-Ramirez and S. Mathur

Individual zinc oxide nanowires as UV photodetectors
Materials Science and Engineering Conference; September 01-04, 2008; Nürnberg

M. Schem, T. Schmidt, H. Caparrotti, M. Aslan, M. Veith and M. Wittmar

The use of Al_2O_3-micro containers for storage of corrosion inhibitors
Managing corrosion for sustainability : EUROCORR 2008, the European Corrosion Congress; September 07-11, 2008; Edinburgh <UK>

K.-P. Schmitt

Nasschemische Pulversynthese
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

M. Schubert

Was ist Nanotechnologie?
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken

C. Trenado, Y. F. Low, L. Haab, W. Delb and D. J. Strauss
Multiscale modeling of attention and habituation effects in the tinnitus decompensation
X. International Conference on Cognitive Neuroscience (ICON X); September 01-05, 2008; Bodrum <Turkey>

M. Veith
The principle of recognition between surfaces and (biological) macromolecules
Conference Universitaire de Suisse Occidentale; April 07-11, 2008; Lausanne <Schwitzerland>
Eingeladener Vortrag

M. Veith
From molecules to materials (Part I)
Conference Universitaire de Suisse Occidentale; April 07-11, 2008; Fribourg <Switzerland>
Eingeladener Vortrag

M. Veith
Biorelevant surface engineering
Conference Universitaire de Suisse Occidentale; April 07-11, 2008; Neuchâtel <Switzerland>
Eingeladener Vortrag

M. Veith
Tuning of physical properties of materials for biological applications
Conference Universitaire de Suisse Occidentale; April 07-11, 2008; Bern <Switzerland>
Eingeladener Vortrag

M. Veith
From molecules to materials (Part II)
Conference Universitaire de Suisse Occidentale; April 07-11, 2008; Fribourg <Switzerland>
Eingeladener Vortrag

M. Veith
From molecular precursors to nano materials
Universität Szczecin; May 28-29, 2008; Szczecin <Poland>
Eingeladener Vortrag

M. Veith
Rationelle Beschichtung von Oberflächen durch Sol-Gel-Technik
Sol-Gel Fachtagung. Grundlagen und industrielle Anwendungen; June 17, 2008; Nürnberg
Eingeladener Vortrag

M. Veith
Vom Molekül zum Material
Technische Universität Graz; June 03, 2008; Graz
Eingeladener Vortrag

M. Veith
Biomimetics of surfaces (Biomimetisme de surfaces)
Verleihung Ehrendoktorwürde, Université Paul Sabatier; October 13-16, 2008; Toulouse <France>
Eingeladener Vortrag

M. Veith
Oberflächen nach dem Vorbild der Natur für Zellseparation
Workshop „Bionik - die Evolution als Ingenieur", Akademie der Wissenschaften und der Literatur Mainz; October 24, 2008; Mainz
Eingeladener Vortrag

M. Veith
Forschung und Zelladhäsion und deren Beeinflussung durch nanostrukturierte Oberflächen
BfR-Forum: Nanotechnologie im Fokus des gesundheitlichen Verbraucherschutzes; November 10-11, 2008; Berlin
Eingeladener Vortrag

M. Veith
Nano surfaces and applications in technology and life sciences
Nanotechnology International Forum December 03-05, 2008; Moscow <Russia>
Eingeladener Vortrag

M. Veith and M. Quilitz
Chemische Nanotechnologie - Grundlagen der Herstellung und Oberflächenmodifikation von Nanopartikeln über chemische Verfahren
GDCh-Kurs „Chemische Nanotechnologie"; June 17-18, 2008; Saarbrücken
Eingeladener Vortrag

I. M. Weiss
Chitin and chitin synthase in mollusks
2008 Gordon Conference on Biomineralization; August 10-15, 2008; New London <NH, USA>
Eingeladener Vortrag

I. M. Weiss
Enzymatic control of biomineralizing interfaces: Structuring of mollusc shells via myosin-dependent chitin synthesis
Biological Materials Science (BMS) Symposium at the 2008 TMS Annual Meeting; March 09-13, 2008; New Orleans <Louisiana, USA>
Eingeladener Vortrag

U. Werner
Elektronenmikroskopie - Abbildung der inneren und der Oberflächen-Struktur
Summer School Chemische Nanotechnologie; October 06-10, 2008; Saarbrücken
Eingeladener Vortrag

U. Werner
Charakterisierung von Nanopartikeln mittels Transmissions-Elektronenmikroskopie
GDCh-Kurs „Chemische Nanotechnologie"; June 17-18, 2008; Saarbrücken
Eingeladener Vortrag

M. Wittmar
Multiprotect - Advanced, environmental friendly, multifunctional corrosion protection by Nanotechnology as substitution of Cr(VI)
IntAircoat Conference; May 15-16, 2008; München
Eingeladener Vortrag

Lectures / Lehrveranstaltungen

Lectures / Lehrveranstaltungen

E. Arzt
Mittwochskolloquium
Universität des Saarlandes
(WS 08/09)

A. del Campo
Biophysikalische Chemie für Studierende der Biotechnologie, Bachelor
Universität Heidelberg
(WS 07/08)

S. Mathur
Anorganisch-chemisches Fortgeschrittenenpraktikum für Studierende des Lehramts an Gymnasien
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Anorganisch-chemisches Praktikum für Studierende der Chemie (Schwerpunktfach)
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Anorganisch-chemisches Praktikum für Studierende der Chemie (Wahlfach)
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Anorganisch-chemisches Praktikum II für Studierende der Chemie
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Seminar für Diplomanden und Doktoranden
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Seminar für wissenschaftliche Mitarbeiter
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Seminar zum Anorganisch-chemischen Praktikum II für Studierende der Chemie
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Übungen im Vortragen und Demonstrieren für Studierende des Lehramts an Gymnasien
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Übungen im Vortragen und Demonstrieren für Studierende des Lehramts an Grund-, Haupt- und Realschulen
Julius-Maximilian-Universität Würzburg
(WS 07/08)

S. Mathur
Anorganisch-chemisches Praktikum für Studierende der Chemie (Schwerpunktfach)
Julius-Maximilian-Universität Würzburg
(SS 08)

S. Mathur
Anorganisch-chemisches Praktikum für Studierende der Chemie (Wahlfach)
Julius-Maximilian-Universität Würzburg
(SS 08)

S. Mathur
Anorganisch-chemisches Praktikum II für Studierende der Chemie
Julius-Maximilian-Universität Würzburg
(SS 08)

S. Mathur
Seminar für Diplomanden und Doktoranden
Julius-Maximilian-Universität Würzburg
(SS 08)

S. Mathur
Seminar für wissenschaftliche Mitarbeiter
Julius-Maximilian-Universität Würzburg
(SS 08)

S. Mathur
Seminar zum Anorganisch-chemischen Praktikum II für Studierende der Chemie
Julius-Maximilian-Universität Würzburg
(SS 08)

D. Strauss
Grundlagen der Medizinischen Messtechnik (Bachelor)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 07/08)

D. Strauss
Einführung in das Neural Engineering (Bachelor)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 07/08)

D. Strauss
Neural signal analysis and modeling (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 07/08)

D. Strauss
Audiologie (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 07/08)

D. Strauss
Einführung in die Biosignalverarbeitung (Bachelor)
Hochschule für Technik und Wirtschaft des Saarlandes
(SS 08)

D. Strauss
Biomedizinische Signal- und Bildverarbeitung (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(SS 08)

D. Strauss
Neural und Cognitive Systems (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(SS 08)

D. Strauss
Grundlagen der Medizinischen Messtechnik (Bachelor)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 08/09)

D. Strauss
Einführung in das Neural Engineering (Bachelor)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 08/09)

D. Strauss
Neural Signal Analysis and Modeling (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 08/09)

D. Strauss
Audiologie (Master)
Hochschule für Technik und Wirtschaft des Saarlandes
(WS 08/09)

M. Veith
Seminar für Mitarbeiter
Universität des Saarlandes
(WS 07/08)

M. Veith
Allgemeine Chemie
Universität des Saarlandes
(WS 07/08)

M. Veith
Grundlagen der Hauptgruppenchemie
Universität des Saarlandes
(WS 07/08)

M. Veith
Allgemeine Chemie für Pharmazeuten
Universität des Saarlandes
(WS 07/08)

M. Veith
Anorganisches und analytisches Kolloquium
Universität des Saarlandes
(WS 07/08)

M. Veith
Einführung in das Fortgeschrittenenpraktikum
Universität des Saarlandes
(WS 07/08)

M. Veith
Fortgeschrittenenpraktikum für Lehramtsstudierende
Universität des Saarlandes
(WS 07/08)

M. Veith
Chemisches Praktikum mit Seminar für Studierende der Biologie, der Physik, der Werkstoffwissenschaften und der Metalltechnik, Kurs 1 und 2
Universität des Saarlandes
(WS 07/08)

M. Veith
Einführung zum Praktikum für Studierende der Biologie, der Physik, der Werkstoffwissenschaften und der Metalltechnik
Universität des Saarlandes
(WS 07/08)

M. Veith
Seminar zum fachdidaktischen Schulpraktikum
Universität des Saarlandes
(WS 07/08)

M. Veith
Chemische Fachdidaktik inkl. Seminar zum Schulpraktikum Kurs B
Universität des Saarlandes
(WS 07/08)

M. Veith
Chemische Fachdidaktik Kurs A
Universität des Saarlandes
(WS 07/08)

M. Veith
Anorganisches Praktikum für Fortgeschrittene (ACF)
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Einführung in das Fortgeschrittenenpraktikum für Lehramtsstudierende (FGLa1)
Universität des Saarlandes
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Einführung und Seminar zum chemischen Praktikum für Naturwissenschaftler
Universität des Saarlandes
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Materialien aus molekularen Vorstufen (AC9)
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Metallorganische Chemie (AC6)
Universität des Saarlandes
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Molekülchemie der Hauptgruppenelemente (AC7)
Universität des Saarlandes
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M. Veith

Molekülchemie der Hauptgruppenelemente II (AC12)
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Spezielle Kapitel der Anorganischen Chemie (AC11)
Universität des Saarlandes
(SS 08)

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Vorlesungen im Rahmen des DEA
Ecole Polytechnique, Paris Palaiseau
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Anorganisches Kolloquium
Universität des Saarlandes
(WS 08/09)

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Chemische Fachdidaktik, Kurs A (FD1)
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(WS 08/09)

M. Veith

Chemische Fachdidaktik inklusive Seminar zum Schulpraktikum, Kurs B (Fd1+FD2)
Universität des Saarlandes
(WS 08/09)

M. Veith

Seminar für eigene Mitarbeiter
Universität des Saarlandes
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Seminar zum fachdidaktischen Schulpraktikum (FD2)
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Übungen zu Grundlagen der Hauptgruppenchemie (AC01Ü)
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(WS 08/09)

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Übungen zur Allgemeinen Chemie (AC00Ü)
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M. Veith

Allgemeine Chemie (AC00)
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(WS 08/09)

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Biochemisches Großpraktikum I / Teil B (Bioinformatik und Enzymologie) im Bachelor Studiengang
Universität Regensburg
(WS 08/09)

Other courses / Weitere Lehrveranstaltungen

M. Schubert

Sol-Gel for New Materials
Workshop in co-operation with Hong Kong Metal Finishing Society
25. - 28.02.2008, Saarbrücken

M. Schubert

Summer School Chemische Nanotechnologie
06. – 10.10.2008, Saarbrücken

M. Veith, M. Quilitz, et al.

GDCh-Kurs "Chemische Nanotechnologie"
26. – 27.06.2008, Saarbrücken

Patents / Patente

Im Jahr 2008 wurden sieben neue Patentanmeldungen hinterlegt, die noch nicht offengelegt sind. Es wurden 12 Patente erteilt, davon zwei innerhalb von Europa und zehn auf internationaler Ebene. Somit wurden insgesamt ca. 145 Patentanmeldungen durch das Leibniz-Institut für Neue Materialien vorgenommen. Rund die Hälfte dieser Schutzrechte ist erteilt.

In 2008, INM has filed seven new patent applications which are not yet published and 12 patents have been granted. Two of these patents are granted in Europe and ten in foreign countries. INM applied around 145 patent families in total and around 50 percent of them are granted until today.

Erteilte europäische Patente:

EP 1532219 B1

Titel: „Liquid repellent, alkali resistant coating composition and coating suitable for pattern forming“
Erfinder: Carsten Becker-Willinger, Helmut Schmidt, Pamela Kalmes

EP 1438361 B1

Titel: „Beschichtete Titandioxid-Teilchen“
Erfinder: Martin Schichtel

Erteilte internationale Patente:

Japanisches Patent Nr. JP 4092513

Stammanmeldungstitel: „Kompositklebstoff für optische und optpelektronische Anwendungen“
Erfinder: Volker Gerhard, Gerhard Jonschker, Martin Mennig, Helmut Schmidt

Japanisches Patent Nr. JP 4129564

Stammanmeldungstitel: „Verfahren zur Herstellung schwach agglomerierter nanoskaliger Teilchen“
Erfinder: Detlef Burgard, Rüdiger Naß, Helmut Schmidt

US- Patent Nr. US 7,390,532

Stammanmeldungstitel: „Verfahren zur Herstellung optischer Elemente mit Gradientenstruktur“
Erfinder: Ulrike Dellwo, Martin Mennig, Peter William de Oliveira, Helmut Schmidt, Heike Schneider

Japanisches Patent Nr. JP 4147496

Stammanmeldungstitel: „Verfahren zum Versehen einer metallischen Oberfläche mit einer glasartigen Schicht“
Erfinder: Gerhard Jonschker, Martin Mennig, Helmut Schmidt

Japanisches Patent Nr. JP 4168412

Stammanmeldungstitel: „Nanostrukturierte Formkörper und Schichten sowie Verfahren zu deren Herstellung“
Erfinder: Ertugrul Arpac, Herbert Krug, Peter Müller, Peter William de Oliveira, Helmut Schmidt, Stefan Sepeur, Bettina Werner

Japanisches Patent Nr. JP 4172034

Stammanmeldungstitel: „Verfahren zur Herstellung agglomeratfreier nanoskaliger Eisenoxidteilchen mit hydrolysebeständigem Überzug“
Erfinder: Christoph Lesniak, Rüdiger Naß, Thomas Schiestel, Helmut Schmidt

Japanisches Patent Nr. JP 4196129

Stammanmeldungstitel: „Verfahren zur Herstellung von Zusammensetzungen auf der Basis von Epoxidgruppen-haltigen Silanen“
Erfinder: Ertugrul Arpac, Volker Gerhard, Reiner Kasemann, Helmut Schmidt, Georg Wagner, Elisabeth Wintrich-Geiter

Japanisches Patent Nr. JP 4207178

Stammanmeldungstitel: „Verfahren zum Schutz eines metallischen Substrats vor Korrosion“
Erfinder: Gerhard Jonschker, Stefan Langenfeld, Helmut Schmidt

US-Patent Nr. US 7,449,245

Stammanmeldungstitel: „Substrate mit photokatalytischer TiO_2-Schicht“

Erfinder: Murat Akarsu, Ertugrul Arpa, Helmut Schmidt

Saudi Arabisches Patent Nr. SA 2063
Stammanmeldungstitel: „Konsolidierungsmittel und dessen Verwendung zur Konsolidierung von Formkörpern und geologischen Formationen aus porösen oder partikulären Materialien“
Erfinder: Klaus Endres, Helmut Schmidt, Bernd Reinhard

Cooperations / Kooperationen (Selection / Auswahl)

Academy of Sciences of the Czech Republic / Prague, Tschechien

Agricultural University of Athens / Athen, Griechenland

Austrian Research Centers GmbH – ARC / Wien, Österreich

BAM – Bundesanstalt für Materialprüfung / Berlin

Bilkent Üniversitesi / Bilkent, Türkei

Bogazici Universitesi / Bogazici, Türkei

CASTI Centro per L'Assistenza Scientifica e Tecnologica alle Imprese / L'Aquila, Italien

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CNRS Institut des Materiaux Jean Rouxel / Nantes, Frankreich

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Helsinki University of Technology / Helsinki, Finnland

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IEE Bratislava / Bratislava, Slowakei

IHPP, Institute of High Pressure Physics / Warschau, Polen

Illinois Institute of Technology / Chicago, USA

Inasmet Technalia / San Sebastian, Spanien

Indian Institute of Science / Bangalore, Indien

Institut für Mikrotechnik Mainz GmbH / Mainz

Institute of Physical Chemistry / Bukarest, Rumänien

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McGill University / Montreal, Kanada

MidSweden University / Östersund, Schweden

Moscow Aviation Institute / Moskau, Russland

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Nanoscience Centre / Cambridge, UK

National Center for Scientific Research Demokritos / Athen, Griechenland

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University of Chemical Technology and Metallurgy / Sofia, Bulgarien

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University of Manchester / Manchester, UK

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University of Oklahoma / Oklahoma City, OK, USA

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University of Udine / Udine, Italien

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Yarmouk University / Yarmouk, Jordanien

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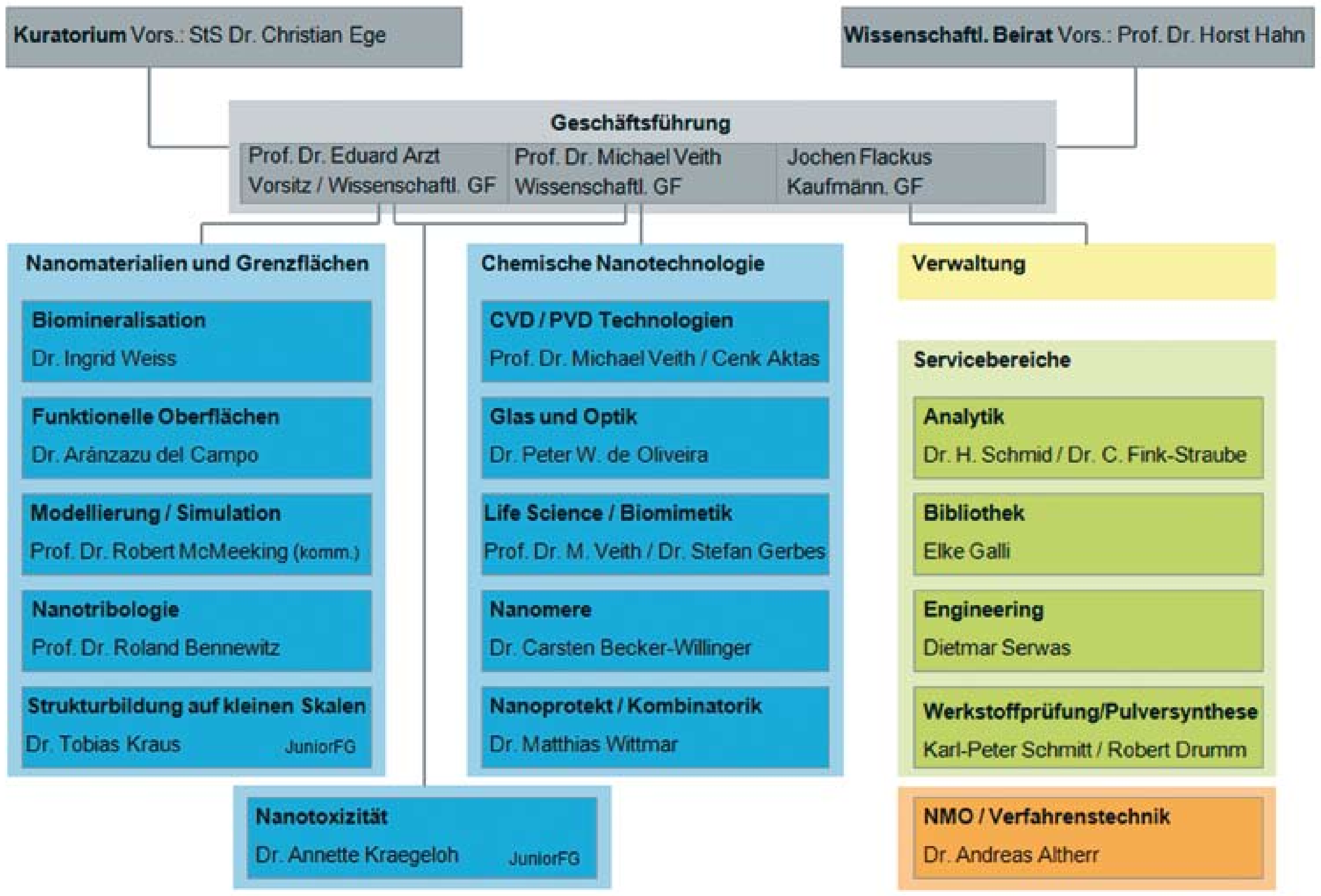

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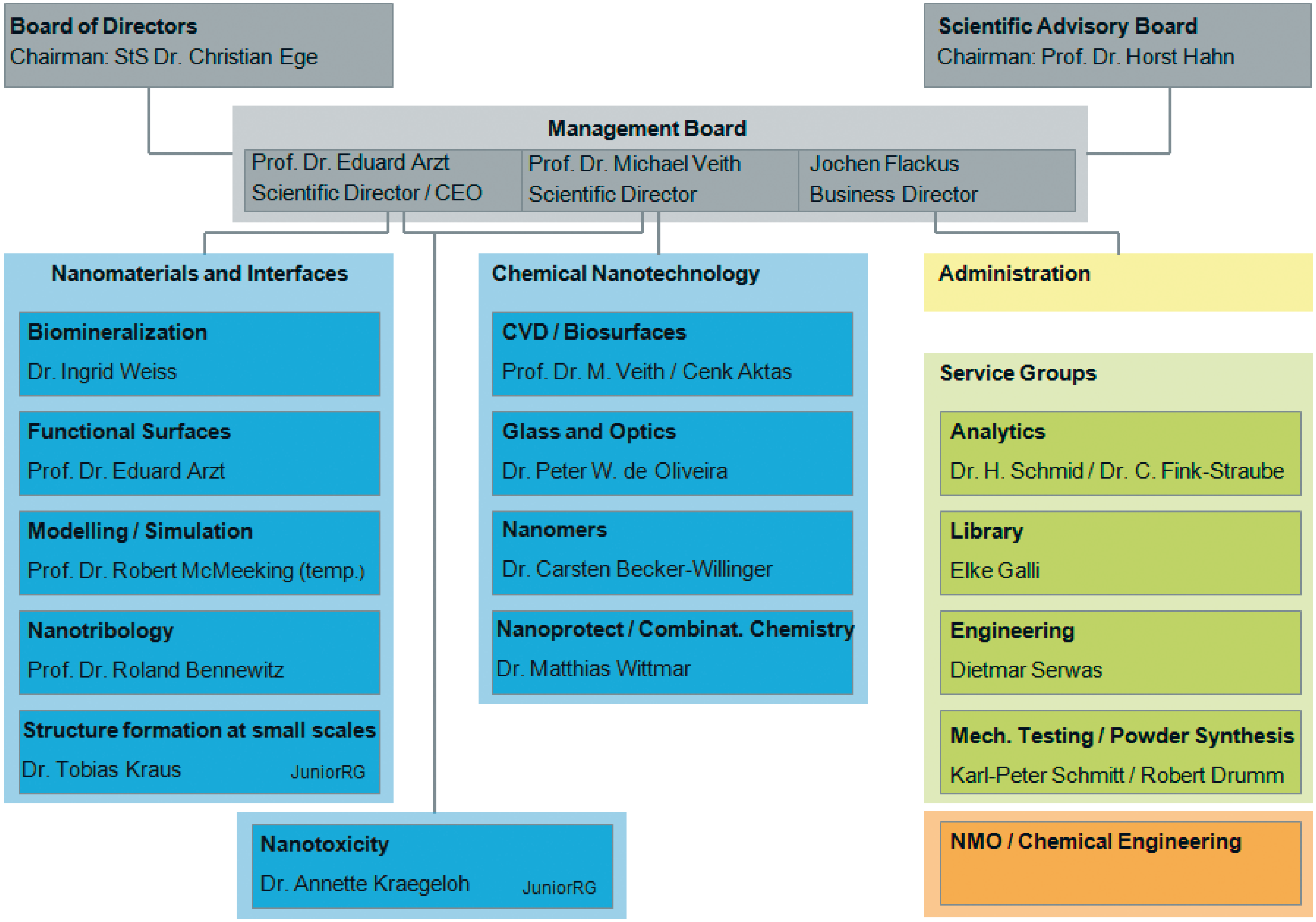

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NMO / Chemical Engineering

as of December 31, 2008

INM wird Preisträger beim „Land der Ideen“: Feier mit Nacht der Wissenschaften

So etwas gibt es nicht alle Tage. Dass Forscher bereitwillig ihre Labore öffnen, sich bei der Arbeit über die Schulter schauen lassen. Die „Nacht der Wissenschaften“ im INM machte es aber möglich.

„Entdecke eine Welt voller Chancen!“, hieß am 18. April 2008 das Motto, und rund 120 Oberstufenschüler ließen sich das nicht zweimal sagen. Geführt von fachkundigen Scouts durchliefen sie einen Parcours, der an mehr als einem Dutzend Stationen zu Themen der Materialwissenschaft und der Nanotechnologie hinführte.

„Wie kommt die Perle aus dem Webstuhl?“, „Färben mit Licht“ und „Warum mein Deo so gut duftet“ lauteten die Titel von Vorführungen und Experimenten, die auch Lust auf die Naturwissenschaften und auf das Forschen machen sollten.

Hintergrund der „Nacht der Wissenschaften“: Mit der Vielfalt seiner Arbeitsgebiete wurde das INM „Ausgewählter Ort“ beim bundesweiten Wettbewerb „Land der Ideen“. In dessen Slogan steckt auch ein Motto des INM: „Hier wird Zukunft gemacht!“ Prof. Dr. Eduard Arzt, Vorsitzender der INM-Geschäftsführung, freute sich über die Auszeichnung:

„Die Auszeichnung ‚Land der Ideen‘ passt hervorragend zum INM. Ideen sind unser Hauptprodukt, sowohl in unserer kreativen Forschung als auch in unseren Problemlösungen für den Alltag.“

Buntes INM 2008

Nanomer ist ein vom INM eingetragener Markenname.

Gestaltung und Layout: J. Pütz · Redaktion: Dr. M. Quilitz, Dr. Ch. Sauer

Korrektur: Dr. H. Bolz · P. Egberts · M. Groh · Ch. Hartmann · Dr. M. Kamperman · Dr. T. Kraus · K. Moh · Dr. T. S. Müller · Dr. E. Murray · Dr. P. Rogin

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Druck: Digitaldruck Pirrot GmbH, Saarbrücken-Dudweiler