

Annual Report

Jahresbericht

2007

Leibniz-Institut für Neue Materialien

Ein Institut der Leibniz-Gemeinschaft

Saarbrücken



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Vorwort

Das Jahr 2007 wurde von einigen herausragenden Ereignissen geprägt. Eduard Arzt hat im Oktober seine Tätigkeit als Wissenschaftlicher Geschäftsführer und Vorsitzender der Geschäftsführung des INM aufgenommen und baut das neue Forschungsfeld „Nanomaterials and Interfaces“ auf. Er wird das Institut zusammen mit Michael Veith als weiterem Wissenschaftlichen Geschäftsführer und Jochen Flackus als Kaufmännischem Geschäftsführer leiten.

Im Januar besuchte Bundespräsident Horst Köhler das INM und zeigte sich beeindruckt von der Kompetenz und Praxisnähe des Instituts. Ein weiterer Höhepunkt war das zwanzigjährige Bestehen des INM, das im Oktober mit einem großen Festakt gefeiert wurde, an dem u.a. Ministerpräsident Peter Müller und der Präsident der Leibniz-Gemeinschaft, Prof. Dr. Ernst Rietschel, teilnahmen.

Mit dem vorliegenden Jahresbericht 2007 blickt das Leibniz-Institut für Neue Materialien auf ein Jahr zurück, in dem die bereits in den Vorjahren eingeleiteten Veränderungen konsequent fortgeführt wurden. Die chemische Nanotechnologie bietet nach wie vor ein enormes Potenzial für die Entwicklung und die industrielle Herstellung neuer Materialien, und auch die chemische Strukturierung von Oberflächen spielt hierbei eine zunehmend größere Rolle. So sind besonders diese beiden Schwerpunkte weiterhin im Grundlagenbereich sowie in wesent-

lichen Projekten und Industriekooperationen des INM vertreten. Wie in den Vorjahren lag daher in 2007 der Fokus der Arbeiten im Institut in der Synthese, Verarbeitung und Anwendung von nanoskaligen Partikeln, teilweise in Kombination mit unterschiedlichen Stoffsystemen. Die gezielte Modifizierung der Partikeloberflächen mittels unterschiedlichster funktioneller Gruppen dient der Herstellung maßgeschneiderter Materialeigenschaften und ist daher in fast allen Programmbereichen ein präzentes Thema. Einen weiteren wichtigen Arbeitsschwerpunkt des Hauses stellt die Applikation, Verarbeitung und Charakterisierung von Schichten und Schichtsystemen dar.

Auch 2007 wurden zahlreiche, sowohl öffentlich- als auch industriegeförderte, Projekte akquiriert, bearbeitet und erfolgreich abgeschlossen. In der Arbeit an diesen Projekten konnte eine Verbesserung der interdisziplinären Zusammenarbeit über alle Programmbereiche hinweg beobachtet werden. Im Bereich der Grundlagenentwicklung und -forschung zeigten sich ebenfalls positive Tendenzen, was sich beispielsweise in der gegenüber dem Vorjahr erhöhten Zahl von Publikationen in Journalen widerspiegelt. Das Vorhaben, ein Gleichgewicht zwischen Grundlagen- und Anwendungsentwicklung am INM zu erreichen, trägt also bereits sichtbare Früchte.

Im Jahr 2007 ist eine besondere Beteiligung des INM an Netzwerkaktivitäten zu verzeichnen. So blickt das am INM beheimatete Kompetenzzentrum cc-NanoChem auf sein erstes Jahr in der neuen

Form als gemeinnütziger Verein cc-NanoChem e. V. zurück.

Die 2006 am INM eingerichtete Koordinierungsstelle Nanotechnologien der Leibniz-Gemeinschaft WGL konnte die zentrale Rolle des Institutes in der nationalen und internationalen Nanotechnologieszene stärken und die Sichtbarkeit der WGL durch eine gemeinsame Kongressbeteiligung (MiNaT, Stuttgart) verbessern.

Ebenfalls 2007 wurde die Arbeitsgemeinschaft der Nanotechnologie-Kompetenzzentren in Deutschland (AGeNT-D) gegründet, deren Mitglieder die bisher vom BMBF geförderten Zentren oder ihre Nachfolgeorganisationen sind. Michael Veith ist Mitglied im Vorstand dieser Institution.

Im Netzwerk NanoBioNet, das neben der Nanotechnologie auch die Biotechnologie und Nanobiotechnologie einschließt, wurde Jochen Flackus im Dezember 2007 zum Vorsitzenden des Vereins gewählt.

Wichtigste Aufgabe des INM bleibt es, die Empfehlungen der Evaluationskommission aus dem Jahr 2005, aufbauend auf den seither bereits erfolgten Veränderungen, konsequent weiter umzusetzen. Um dies zu erreichen, wurde eine Reihe von Zielen beschlossen, deren Umsetzung die nächste Zeit prägen wird. Hierzu zählen neben der weiteren Erhöhung der wissenschaftlichen Qualität und Sichtbarkeit des Instituts vor allem die Strukturverbesserung und -erneuerung, die Optimierung und Verste-

Preface

tigung der Drittmittelstruktur, die Analyse und Strategie des Industrietransfers sowie schließlich die Öffnung des Instituts als Wissensdrehscheibe.

Inhaltlich kommt auf das INM ebenfalls eine Weiterentwicklung zu. Zu den bestehenden Programmbereichen werden weitere Gruppen entstehen, deren Themen interessante neue Materialgrundlagen erschließen und gleichzeitig die bereits bearbeiteten Gebiete gut ergänzen sollen. Dazu wird das bisherige Forschungsfeld des Instituts durch zukunftssträchtige Aspekte aus der Materialwissenschaft, Physik und Biologie erweitert. Eine weitere Neuerung wird die Einführung von Juniorforschungsgruppen sein, in denen Forscherinnen und Forscher bereits kurz nach der Promotion eigenverantwortlich ihren Ideen nachgehen können. Erste wesentliche Schritte konnten bereits unternommen werden: Die erste Juniorforschungsgruppe („Strukturbildung auf kleinen Skalen“; Leitung: Dr. Tobias Kraus) hat im Oktober 2007 ihre Arbeit aufgenommen, und der erste neue Programmbereich („Funktionelle Oberflächen“; Leitung: Dr. Aránzazu del Campo) wurde im Dezember eingerichtet.

Ein großer Teil des Weges bei der Neuorientierung des INM liegt noch vor uns, und wir bedanken uns schon jetzt bei allen, die ihn mit uns gehen werden. Ein solcher Umbruch und Neuaufbau, wie wir ihn planen, benötigt flexible und findige Mitstreiter, aber auch Vertrauen und Zeit – zwei Ressourcen, die für den Aufbau kreativer Forschung unabdingbar sind.

Auf den folgenden Seiten erwartet Sie ein umfassender Überblick über die Tätigkeiten des INM im Jahr 2007. Allen Mitarbeiterinnen und Mitarbeitern, den Mitgliedern des Wissenschaftlichen Beirats und des Kuratoriums, sowie den Zuwendungsgebern und den Partnern aus Wirtschaft und Forschung danken wir herzlich für ihre Unterstützung des Instituts.



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



Prof. Dr. Michael Veith
(Wissenschaftlicher Geschäftsführer)

Preface

The year 2007 was characterized by several outstanding events. Eduard Arzt took up his function as Scientific Director and Chairman of the management of INM in October 2007 and started to build the new research area „Nanomaterials and Interfaces“. He will head the institute together with Michael Veith as further Scientific Director and Jochen Flackus as Business Director.

In January, the President of the Federal Republic of Germany Horst Köhler visited the INM and appeared to be impressed by the institutes competence and practical approach. Another highlight of the year was the twentieth anniversary of the INM. It was celebrated with a ceremonial act in October, which was attended by the Prime Minister of the federal state of Saarland, Peter Müller, and the president of the Leibniz Association, Prof. Dr. Ernst Rietschel, as well as many other prominent guests.

With the annual report for 2007 the Leibniz Institute for New Materials looks back to a year in which the changes initiated in the previous years have been consequently continued. Chemical nanotechnology still bears an enormous potential for the development and the industrial production of new materials. Also the chemical structuring of surfaces plays an increasing role in this context. Therefore, especially these two focal areas are present both in the field of basic research and in cooperations with industry. As in previous years, highlights of our work concentrated on synthesis, processing and application of nanoscaled particles, partly in combination with various materials systems. The designed modification of particle surfaces via various functional groups allows the preparation of materials with tailored properties and is therefore an important topic for almost all program divisions. Another important focus is application, processing and characterization of coatings and coating systems.



In 2007 again many projects – public as well as industrially funded – were acquired, executed and successfully completed. The work in these projects revealed a remarkable improvement of interdisciplinary cooperation across all program divisions. Also in the field of basic research and development, positive tendencies are apparent, as reflected in an increased number of journal publications. The intention to establish an equilibrium between basic and application oriented research and development bears visible fruits.

In 2007 a strong participation of the INM in networking activities can be reported. Especially the INM-based center of competence “cc-NanoChem” looks back on its first year in the newly organized form as a non-profit association (cc-NanoChem e. V.).

The coordination bureau nanotechnologies of the Leibniz Association established at the INM in 2006, served to strengthen the central role of the institute in the national and international nanotechnology scene and improve the visibility of the WGL by a common participation at a conference (MiNaT, Stuttgart, Germany).

The working consortium of the competence centers of nanotechnology in Germany (AGeNT-D) was also founded in 2007. Members comprise the formerly state funded centers or their successor organisations. Michael Veith is a member of the board of directors of this institution.

Jochen Flackus was elected chairman of NanoBioNet in December 2007. NanoBioNet is a networking association that includes, besides nanotechnology, the areas bio- and nanobiotechnology.

For INM, the important task remains to consequently implement the recommendations of the evaluation committee in 2005, based on the alterations which were performed since then. To achieve this, a couple of aims were determined, whose implementation will influence the next time. Among these are, besides the further enhancement of scientific quality and the visibility of the institute, the structural improvement and renovation, the optimization and perpetuation of the third party funding structure, the analysis and strategy of the transfer to industry and last but not least the opening of the institute as a knowledge platform.

Regarding content, INM will also face a further development. Supplementary to the existing program divisions more groups will be founded whose research fields will create interesting new materials basics and at the same time complement the already active areas. Additionally, the former research portfolio will be expanded by seminal aspects from materials science, physics and biology. Another innovation will be the implementation of junior research groups, in which researchers can follow their ideas in sole responsibility short after their PhD-thesis. By now already first measures were taken up: The first junior research group („Structure Formation at Small Scales“; Head: Dr. Tobias Kraus) took up its work in Octo-

ber 2007 and the first new program division („Functional Surfaces“; Head: Dr. Aránzazu del Campo) was founded in December 2007.

A great deal of the way to the new orientation of the INM still lies ahead, and we already thank all those who will go it together with us. Such a reorganisation and change, as we planned, requires flexible and creative co-workers, but also confidence and time – two resources, inevitable for the constitution of creative research.

On the following pages you will find a comprehensive overview over the activities of the INM in the year 2007. Special thanks once again to our employees, the members of the Board of Directors and of the Scientific Board, our sponsors and last but not least the partners from industry and research.

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

Prof. Dr. Michael Veith
(Scientific Director)

Statusbericht

Statusbericht

Finanz- und Ertragslage / Vermögenslage der Gesellschaft

Als Forschungseinrichtung der Leibniz-Gemeinschaft hat das INM auch im Haushaltsjahr 2007 eine gemeinsame Förderung durch den Bund und das Saarland erhalten. Diese belief sich auf 11.200 T€; hiervon 9.000 T€ zur Finanzierung von Personal- und Sachaufwendungen sowie 2.200 T€ für erforderliche Neu- und Ersatzinvestitionen.

Der nominale Rückgang der Zuwendung gegenüber dem Vorjahr um 73 T€ ist durch die- einmalig im Haushaltsjahr 2006- für notwendige Sanierungsmaßnahmen an den Flachdächern des Institutsgebäudes bewilligten Haushaltsmittel bedingt.

Im Geschäftsjahr 2007 erzielte das Leibniz-Institut für Neue Materialien eigene Erlöse aus Forschung und Entwicklung sowie sonstige betriebliche Erträge in Höhe von 4.427 T€ (Vorjahr: 3.627 T€). Die Industrierlöse aus Forschung und Entwicklung sowie aus Lizenzvereinbarungen beliefen sich hierbei auf 1.560 T€ (Vorjahr: 1.717 T€). Im Rahmen öffentlicher Projektfinanzierungen erzielte das INM im Geschäftsjahr 2007 Erträge in Höhe von 1.976 T€ (Vorjahr: 1.346 T€). Die sonstigen Erträge des Instituts im Geschäftsjahr 2007 belaufen sich auf 891 T€ (Vorjahr: 564 T€).

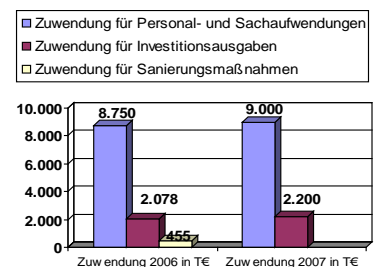
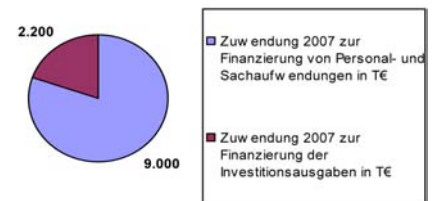
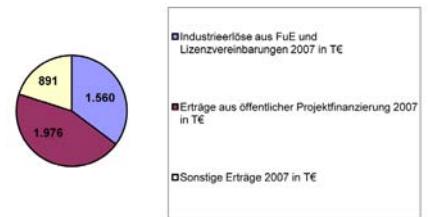
Der Gesamtumsatz 2007 der Gesellschaft betrug 14.881 T€ (Vorjahr: 14.352 T€).

Der Anteil der Drittmittelerlöse sowie der sonstigen Erträge im Geschäftsjahr 2007 belief sich somit auf 29,7% des Gesamtumsatzes (Vorjahr 25,3%). Wie bereits in den beiden Geschäftsjahren zuvor feststellbar, tragen zunehmend öffentliche Projektförderungen zur Gesamtfinanzierung des INM bei.

Die Bilanzsumme der Gesellschaft zum 31. Dezember 2007 betrug 14.755 T€, gegenüber dem Vorjahr eine deutliche Erhöhung um 923 T€. Während sich das Umlaufvermögen gegenüber dem Bilanzstichtag des Vorjahres geringfügig um 164 T€ erhöhte, ist beim Anlagevermögen der Gesellschaft eine beträchtliche Zunahme um 730 T€ zu verzeichnen. Die Investitionstätigkeit der Gesellschaft (2.861 T€) überstieg im Geschäftsjahr 2007 erneut deutlich die Abschreibungsquote in Höhe von 1.968 T€. Die Verbindlichkeiten der Gesellschaft beliefen sich zum Bilanzstichtag auf 765 T€ (Vorjahr: 410 T€).

Personalentwicklung

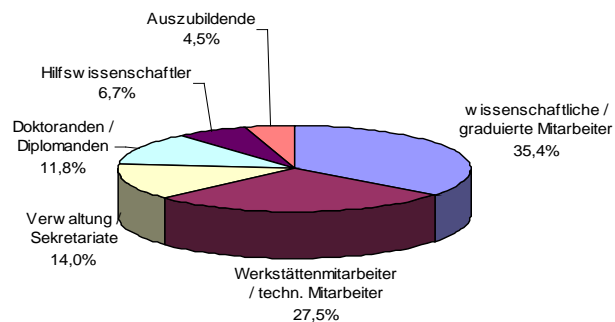
Die Beschäftigtenanzahl konnte 2007 gegenüber dem Jahr 2006 deutlich angehoben werden. So waren Ende 2007 178 Mitarbeiter (157 Vollzeitäquivalente) gegenüber 156 Mitarbeiter (130 Vollzeitäquivalente) Ende 2006 beschäftigt. Hiervon waren 52 Mitarbeiterinnen und Mitarbeiter (dies entspricht 46 Vollzeitäquivalenten) im Drittmittelbereich beschäftigt. Auch die Anzahl der Auszubildenden konnte auf acht gesteigert werden, während der Anteil der Mitarbeiter





im Verwaltungsbereich und der Sekretariate mit 14 % gegenüber 2006 (14,1 Prozent) nahezu konstant blieb. Der Anteil der Doktoranden/Diplomanden sank geringfügig um 1,0 Prozentpunkt von 12,8 % auf 11,8 %. Der Anteil der wissenschaftlichen und graduierten Mitarbeiter nahm gegenüber dem Vorjahr um 1,8 Prozentpunkte von 37,2 % auf 35,4 % ab; der Anteil der Mitarbeiter in den Werkstätten und im technischen Bereich konnte um 3,2 Prozentpunkte von 24,4 % auf 27,5 % erhöht werden.

Personal nach Beschäftigtengruppen in 2007



Status Report

Financial and income situation of the company

As a research institute of the Leibniz Community INM obtained common grants from the federal government and the state of Saarland also in the financial year 2007. These grants amounted to 11,200 T€, of which 9,000 T€ were used to finance personnel and material expenses and 2,200 T€ for necessary new investments and reconstruction measures.

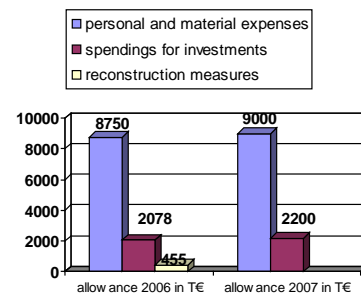
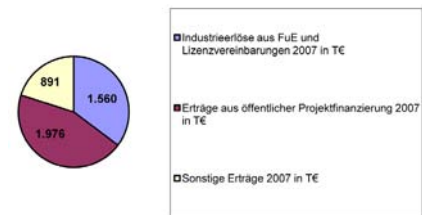
Because of one-time approved budget funds in 2006 for necessary reconstruction measures on the flat roofs of the institute building, there is a nominal decrease of allowance of 73 T€ in the financial year 2007.

In the financial year 2007, INM Leibniz-Institute of New Materials generated own proceeds of 4,427 T€ (preceding

year 3,627 T€) from research and development as well as from patent operating income. Industry revenues from research and development as well as from patents/licences amounted to 1,560 T€ (preceding year 1,717 T€). Within public project subsidies, INM made a in the amount of 1,976 T€ in 2007 (preceding year 1,346 T€). Other institution's profits in the financial year 2007 amounted to 891 T€ (preceding year 564 T€).

The total turnover of INM in 2007 was 14,881 T€ (preceding year 14,352 T€). So, the part of in third-party funds as well as in other revenues in the business year 2007 amounted to 29.7% of the total turnover (preceding year 25.3%). As already shown in the last two business year's reports, public projects grants contribute increasingly to the total turnover of INM.

The balance sheet total of the corporation came to 14,755 T€ on 31 December 2007, compared to the preceding year a remarkable increase of 923 T€.



While current assets increased slightly by 164 T€ compared to the reporting date of the previous year, the noncurrent assets of the corporation increased considerably by 730 T€. The investment activity of the corporation (2,861 T€) exceeded again noticeably the allowance rate in the amount of 1,968 T€ in the financial year 2007. The liabilities of the corporation amounted to 765 T€ at reporting date (preceding year 740 T€).

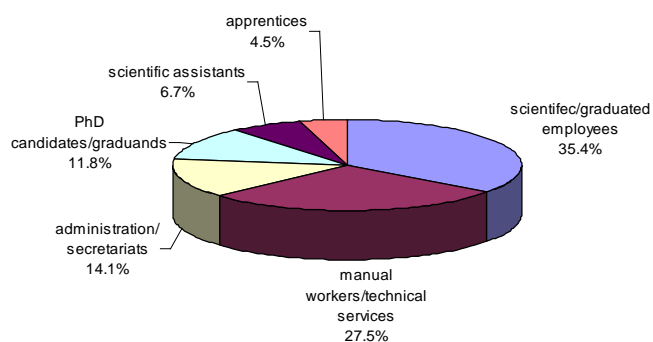
Personnel development

In comparison to 2006, INM increase could the number of employees considerably.

At the end of 2007, 178 employees (157 full-time equivalents) worked at INM, compared to the preceding year with 156 employees (130 full-time equivalents). 52 employees (46 full-time equivalents) were financed by third-party funds.

The number of apprentices increased from six to eight, while the proportion of the administrative and secretary staff remained nearly at the same level (2007: 14.0%, 2006: 14.1%). The rate of the PhD candidates and graduates declined slightly 1.0 percentage point from 12.8% to 11.8% and the part of scientific and graduated employees declined about 1.8 percentage points from 37.2% to 35.4%. The rate of the manual workers and the workers in the technical services increased about 3.1 percentage points from 24.4% to 27.5%.

Categories of employees 2007



Mitglieder des Kuratoriums (Stand: Ende 2007) / Members of the Board of Directors (Status: End of 2007)

Staatssekretär Dr. Christian Ege

Ministerium für Wirtschaft und Wissenschaft des Saarlandes

- Vorsitzender -

Klaus Gerstner

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

- Stellvertr. Vorsitzender -

Prof. Dr. Hans-Joachim Güntherodt

Institut für Physik
Universität Basel

Dr. Wolf-Dieter Haecker

Direktor a. D.
Robert Bosch GmbH, Stuttgart



Wissenschaftlicher Beirat



Dr. Max Häring

Vorsitzender des Vorstandes
Landesbank Saar Girozentrale, Saarbrücken

Liane Horst

Bundesministerium für Bildung und Forschung, Berlin

Prof. Dr. Volker Linneweber

Präsident der Universität des Saarlandes

Prof. Dr. Sanjay Mathur

Institut für Anorganische Chemie
Universität Würzburg und
Leiter PB PVD/CVD-Technologien
INM gGmbH, Saarbrücken

Prof. Dr. Hans-Heinrich Moretto

Direktor a.D.
Bayer Industry Services GmbH & Co. OHG,
Leverkusen

Prof. Dr. Frank Mücklich

Lehrstuhl für Funktionswerkstoffe
Universität des Saarlandes

Prof. Dr. Frank Pobell

Hochfeld-Magnetlabor
Forschungszentrum Rossendorf e.V., Dresden

Prof. Dr. Wulff Possart

Lehrstuhl für Thermodynamik der Werkstoffe
Universität des Saarlandes

Mitglieder des wissenschaftlichen Beirates (Stand: Ende 2007) /
Members of the Scientific Board
(Status: End of 2007)

Ralf Becker

Villeroy & Boch AG
Leitung Geschäftsfeld
Sanitärkeramik und Küche
Mettlach

Prof. Dr. Ludwig Gauckler

Institut für Nichtmetallische Werkstoffe
ETH Zürich

Prof. Dr. Horst Hahn

Geschäftsführender Direktor
Institut für Nanotechnologie (INT) Forschungs-
zentrum Karlsruhe GmbH
Eggenstein-Leopoldshafen

Dr. Klaus Harste

Mitglied des Vorstandes
Saarstahl AG, Völklingen

Prof. Dr. Andreas Mortensen

Ecole polytechnique fédérale de Lausanne

Prof. Dr. Martin Möller

Lehrstuhl für Textilchemie und Makromolekulare Chemie
RWTH Aachen

Prof. Dr. Erich Sackmann

Lehrstuhl für Biophysik
Technische Universität München

Prof. Dr. Robert F. Singer

Lehrstuhl Werkstoffkunde und Technologie der Metalle
Universität Erlangen-Nürnberg



Programmbereich Glas und Optik

Dr. Peter W. de Oliveira



Program Division Glass and Optics / Programmbereich Glas und Optik

Der Hauptarbeitsschwerpunkt des Programmbereiches Glas und Optik bestand auch im Jahr 2007 in der Entwicklung neuer Materialien und ihrer Anpassung auf Anwendungen in der Optik. Forschungs- und Entwicklungsarbeiten des Programmbereiches Glas und Optik wurden dementsprechend vor allem auf den Gebieten neuer photosensitiver Materialien für die Phasenstrukturierung, Techniken für die Informationsspeicherung, transparenter und leitfähiger Beschichtungen, Barrierschichten, Core-shell-Partikeln, Antireflexschichten, Lichtmanagementfolien, strukturierten Schichten für kontrolliertes Zellwachstum und nicht zuletzt glasartiger Schichten für den Korrosions- und Verschleißschutz durchgeführt.

Ein wesentliches Ziel für 2007 bestand darin, die Entwicklung in Richtung eines Gleichgewichtes zwischen Grundlagenarbeit und angewandter Industrieforschung auszubauen. Diesem Ziel wurde mit der weiteren Erhöhung der Grundlagenaktivitäten gearbeitet, was sich vor allem in einer deutlichen Steigerung der Publikationstätigkeit in 2007 niederschlug. Durch diese Aktivitäten wurden aber auch eine Reihe neuer Ansätze für weitere Grundlagen- und zukünftige Projektarbeiten eröffnet.

Weiterhin sehr erfolgreich entwickelte sich die Projektarbeit, wobei zunächst

die öffentlichen Projekte zu nennen sind, von denen eine ganze Reihe in 2007 bearbeitet wurden (FlexNet, ROLLED, FLEX-OPTEC, NOVACOAT, CellPROM). Neben den nationalen (BMBF) spielen hierbei besonders EU-Kooperationen eine wichtige Rolle. Weitere Projekte wurden für ihre Einreichung in 2008 vorbereitet (z. B. EMABO).

Diese Arbeiten wurden durch eine Reihe erfolgreicher Industrieprojekte mit diversen internationalen Partnern ergänzt. Ein besonderes Highlight des Technologietransfers im Jahr 2007 war der Produktionsbeginn in Japan der am INM entwickelten Lichtmanagementfolien. Diese Produktion konnte erfolgreich an die von den Grundlagen bis zur Pilotfertigung reichende Produkt- und Prozessentwicklung des INM anknüpfen. Zusätzlich wurden die Entwicklungen des Programmbereiches Glas und Optik auf nationalen und internationalen Ausstellungen und Industriemessen mit sehr positiver Resonanz präsentiert.

Auch 2007 wurde wieder in die Infrastrukturentwicklung des Programmbereiches investiert. Neben Geräte- und prozesstechnischen Entwicklungen – ein besonderes Highlight war 2007 die Arbeiten an der neuen Folienbeschichtungsanlage, die realitätsnahe Fertigungstests unter Industriebedingungen erlaubt – wurde die Optimierung des Gruppenmanagements durch Einführung weiterer Managementtools vorangetrieben.



The main work of the Glass and Optics Group is the development of new materials and the adaptation of these materials for applications in optics. The research has been focused on the development of new photosensitive materials for phase μ -patterning as well as on techniques for the storage of information. Simulations of the optical properties of thin films and GRIN-coatings (Graded Refractive INdex) have shown that the wet chemistry has a big impact on new material development for optical storage systems. In order to maintain scientific productivity special attention has been given to the basic research in order to improve the sustainability of the Glass and Optics Group. More than the year 2006, 2007 was a successful year for industry projects as well as for public founded research programs as a result of an optimization of the groups management based on the activity of all group members.

Transparent conductive films like ITO (tin doped indium oxide) with a sheet resistance of 2 to 3 $k\Omega/sq$ were produced by gravure printing process on various foil substrates as PET (polyethylene terephthalate) and PEN (polyethylene 2,6-naphthalate) foils. The printing paste consisted of ITO nanoparticles which were dispersed in a solvent by using a surfactant. The dispersion was mixed with a binder and a photo initiator before printing. The printed films were hardened under UV-irradiation at low temperatures ($< 130^\circ C$). The sheet resistance could be decreased by heat treatment at $120^\circ C$ under reducing atmosphere (for-

ming gas atmosphere ($N_2/ 5vol \% H_2$) to 1.5 $k\Omega/sq$. The transmission of the ITO coated PET foil is more than 80 % in the visible range.

The ITO films were directly used as the bottom electrode (figure 1) in an organic photodiode (OPD) at JR-NMP (Joanneum Research - Institut für Nanostrukturierte Materialien und Photonik) in Austria. Results on these OPDs demonstrated the suitability of the printed ITO layers as anode for organic photodiodes. This work was financed by the transnational ERASPOT project „FLEX-OPTEC - Flexible Organic Photodiodes with Transparent Electrodes fabricated by wet Chemical deposition methods” (03/2007 – 03/2008), whereby the german part was financed by the BMBF (FKZ: 13N9230). The aim of the work at the INM is to reduce the sheet resistance of the ITO coatings to 1 $k\Omega/sq$, whereby the transmittance should be $> 80 \%$. (figure 2, 3)

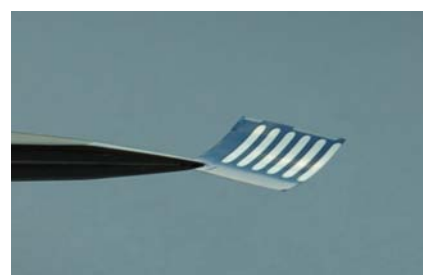


Figure 1: Organic photodiode (fabricated in the JR-NMP institute) with a printed ITO coating on PET foil (fabricated in the INM) as bottom electrode.

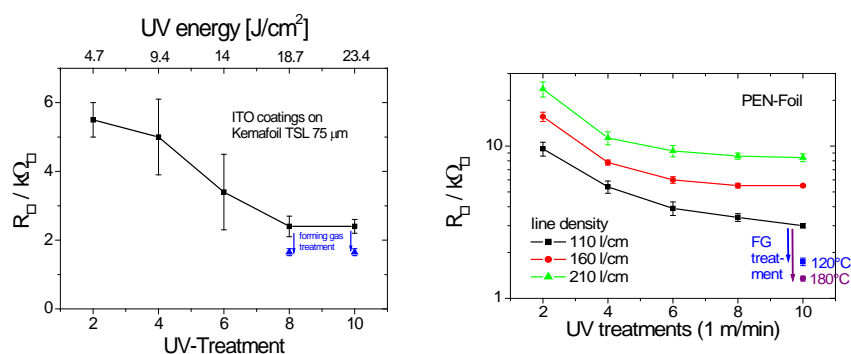


Figure 2: Sheet resistance R_{sq} of printed ITO coatings (line density 110 l/cm) on PET foil (left, Kernafol TSL75) and on PEN foil (right) after UV treatment (conveyor speed 1 m/min, measured 5 min after 2, 4, 6, 8 and 10 UV passes) and after forming gas treatment at $120^\circ C$ (PET foil and PEN foil) and at $180^\circ C$ (PEN foil).

Besides the conductivity of ITO layers their ductility is important for some applications. If it is too low the layer cracks during the following processing steps and is thus not conductive anymore. The optimization of the ductility with respect to conductivity and the optical properties by variation of the ITO-composition and the process parameters during film processing were investigated. A method for the measurement of the ductility was defined and a variety of different ITO-layers were tested with this method. Microscopy of the ITO-layers after the mechanical loading according to the above mentioned test gave an additional indication of their ductility.

In a cooperation with NMO pellets of indium tin oxide (ITO) nanoparticles from an electrochemical synthesis process were characterized with regard to their electrical properties. The results of this characterization gave valuable insights into the influence of various process parameters on the microstructure and the electrical properties of ITO. Especially the use of additional calcination and reduction processes enhanced the electrical conductivity by more than one order of magnitude.

For the optimization of the electrical properties of MgB_2 ceramics the use of nanoscaled materials for the synthesis of MgB_2 and the respective precursors for ex-situ- and in-situ-synthesis routes were investigated aiming at improved microstructures and homogeneities and finally at the improvement of the corresponding electrical properties. Besides the screening of precursors, alternative syn-

thesis routes were examined including electrochemical synthesis in aqueous and non-aqueous systems and with additional modifiers as well as reduction respective precipitation techniques.

In the european collaborative research project ROLLED (2004-2008) the development of flexible transparent barrier layers for encapsulating organic light emitting diodes was carried on towards roll-to-roll (R2R) processing. The novel approach, for which a patent application was filed in 2006 [DE 10 2006 046961], is based on preparing a glass layer initially prepared on a temperature resistant substrate by wet coating and high temperature densification before transferring this layer to a transparent base film like PET. The adaptation of this concept to R2R processing introduced new techniques and equipment for coating and high temperature treatment on the existing pilot coating line, which turns out to be useful also in other work.

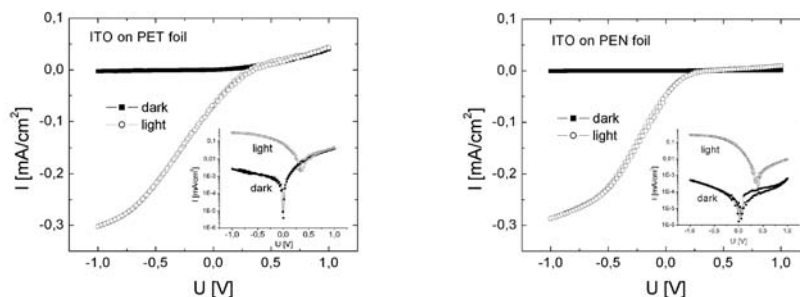


Figure 3: Current-voltage (I-V) characteristics of organic photodiodes with the following structure: substrate/ ITO/ PEDOT/ PSS/ CuPc/ PTCBi/ Alq3/ Ag with PEN foil (left) and PET foil (right) as substrate. The insets show the I-V characteristic in logarithmic scale. (The OPD were fabricated and characterized at JR-NMP with the printed ITO coatings of the INM).



In the collaborative research project Flex-Net funded by the BMBF (2006-2009) initial investigations were done on preparing glass like diffusion barrier layers on mild steel and polyimide substrates to be used for the preparation of thin film photovoltaic cells afterwards. The purpose of these barrier films is to prevent the diffusion of harmful components from the substrate into the active layers during processing, and also to provide electrical insulation in the case of metallic substrates. First results already indicate good electrical insulation properties and suitable diffusion barrier properties, although some work still has to be done to improve adhesion and film homogeneity.

The year met the completion of the EU Craft Project NOVACOAT (COOP-CT-2005-005799), in which hybrid coatings for ceramic tiles were developed based on the idea to replace traditional high temperature glaze in tile manufacturing and to access new decorations and designs. Therefore coating systems based on several different hybrid coating compositions of INM were improved in order to meet the specific requirements of tile manufacturers especially towards chemical durability and application behaviour as well as their potential to incorporate color and functional effects. In the materials part, the development concentrated on water based hybrid coating systems to meet the H&S regulations and on the application side on the use of conventional ceramic coating methods – in particular curtain coating and spraying.

The synthesis and characterization of metal and metal@metal oxide core shell particles with a potential use in e.g. catalytic especially photocatalytic applications has been another focus, concentrating in 2007 on the development of new wet-chemical synthesis routes for monodisperse silver particles (figure 4) and on particles based on binary intermetallic compounds such as FePt. The difficulty lies in the development of an easy strippable intermediate surface modification. This surface modification must be stable enough to stabilize the freshly prepared core particles during processing, but must be removable in order to allow the growth of the desired metal oxide shell in the following step. A further topic is to understand how a variation in the process parameters influences size and shape as well as crystallinity of metal and metal-oxide particles. Therefore metal-organic complexes were synthesized and decomposed by heat treatment in a solvent where the organic fraction in these complexes serves as a surface modifier for the forming particles.

Alternative conductive coatings based on multi-walled carbon nanotubes (MWNTs) have been investigated in the frame of a cooperation with Prof. H. Schmidt. In this thesis, the performance of MWNTs as networks and as additional conductive elements in various matrices was demonstrated. Additionally, studies of the effects of CO₂ laser sintering on the properties of TiO₂/MWNT nanocomposites, such as photocatalytic activity, wettability and structure, besides optical and

electrical responses has been performed. (figure 5) CO₂ laser irradiation was presented as an efficient method for the sintering of TiO₂ anatase phase while preserving the MWNT's structure in the nanocomposites. The enhancement of laser intensity during the sintering contributes to the densification of the coatings, leading to hydrophilic surfaces with enhanced electrical conductivity. The addition of CNTs to the TiO₂ matrix has improved the photocatalytic activity of TiO₂ coatings, what has been demonstrated by the degradation of stearic acid layer deposited on the films. In addition, higher CO₂ laser intensities during the sintering resulted in an enhanced photocatalytic activity of the nanocomposites.

Coating solutions based on surface modified high (TiO₂) and low (SiO₂) refractive index nanoparticles were used to produce a five layer reflective system on glass substrate.

UV-VIS-spectroscopy measurements showed that the reflection of the glass substrate can be increased to ca. 80% in the visible region.

A five reflective layers system was investigated by high resolution transmission electron microscope (HRTEM) using a cross-sectional specimen preparation method (figure 6). HRTEM results showed that the layers were very homogeneous without pores and cracks in the layers. The thicknesses of the TiO₂ layers were found to be 45, 49 and 55 nm respectively. The thickness of the two SiO₂ layers were very similar with around 65 nm. The diffraction patterns of the TiO₂

and SiO₂ layers indicated that there were crystalline and amorphous phases respectively. (figure 7)

In 2007 an INM product has taken up production in Japan. The light management films were produced by a sol-gel process which was developed at INM previously. The goal of the project was a product development starting from laboratory research, and comprising activities up through pilot plant stage to a state where mass production was possible. The production process based on a nanocomposite material containing high refractive index nano particles with a surface modification allowing for UV induced cross-linking. The nano particles are mobile in a low refractive index gel-

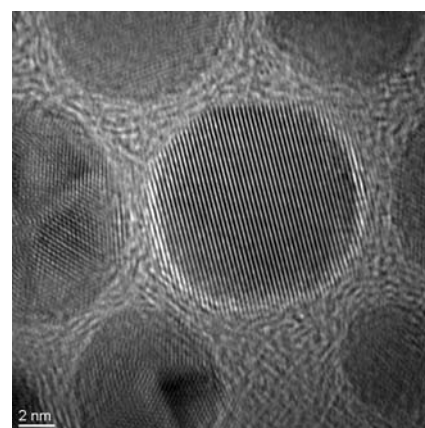


Figure 4: Single-crystalline silver particle, obtained by thermal decomposition of a metal-organic complex in a high-boiling solvent.

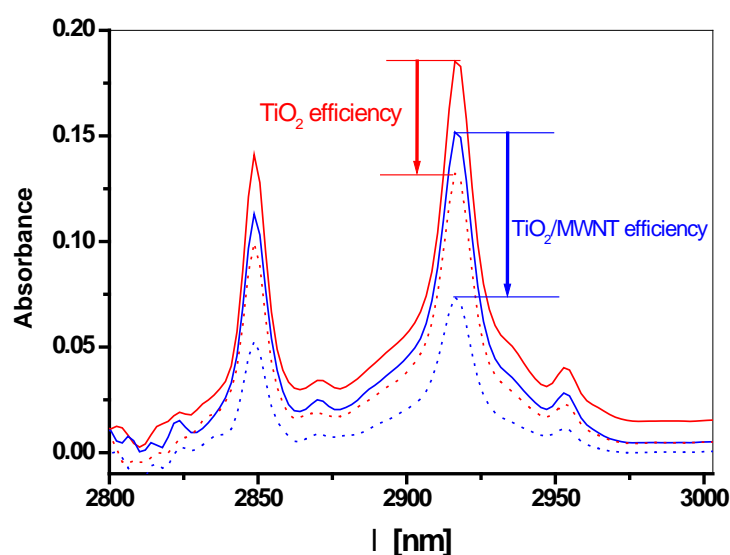


Figure 5: Loss of absorbance in the C-H stretching region observed after UV irradiation for 1 h for a stearic acid layer deposited on TiO₂ and TiO₂/MWNT coatings sintered with CO₂ laser irradiation of 17 W/m².



like matrix and the structuring is realized by a lithographic process step.

Various profiles (asymmetric, symmetric) of the diffusers (figure 8, 9) have been put into practice during the project and also the processes suitable for mass production were developed. A further part of the cooperation was the scaling-up of the sol-gel material production. INM has developed a complete checklist for all production parameters and steps to assure a good quality and reproducibility of the sol. Overall 900 kg (700 kg were shipped to Japan) sol-gel material were produced at INM during this industrial project.

One of the main topics of the EU project CellPROM - Embossed micro structures for controlled cell growth was the controlled growth of cell material. Further micrometer structures were developed and reproduced for this challenge. A laser was used for the production and PDMS embossing stamps were used for the replication of this micro structures which were sent to other institutes all over Europe for further experiments with cell growth on them (figure 10).

Since several years, pure glass-like coatings are well known for corrosion and wear protection up to 500°C [DE 10 2004 001 097 A1]. Based on this, new glass-ceramic coatings were developed, which withstand temperatures up to 900°C without loss of the protection properties. In addition, tribological and easy-to-clean functions are incorporated by ceramic components like SiC. These results were obtained by combination of a glass matrix with a ceramic matrix, which

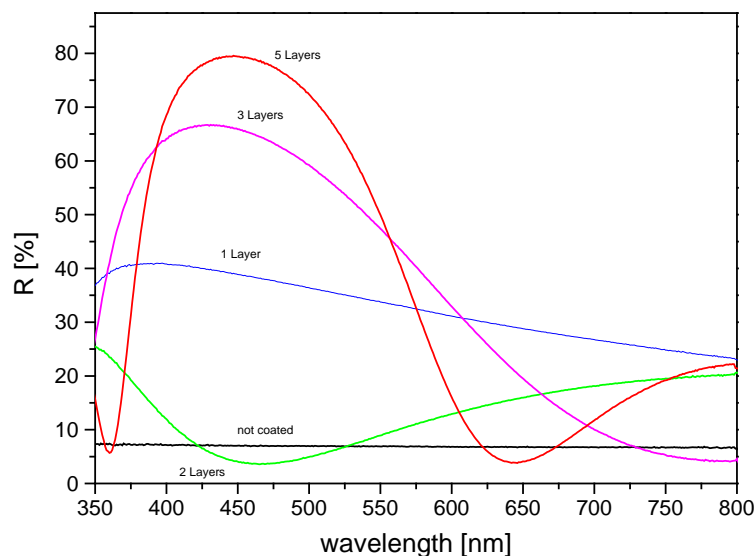


Figure 6: The spectra of the glass substrate before coating, the single layer (H), two layers system (HL), three layers system (HLH) and five layers system (HLHLH) show the reflectance (R) versus the wavelength between 350 to 800 nm.

showed an increased stability compared to a pure glass-like matrix [DE 197 14 949 A1] after sintering at temperatures between 500°C and 900°C (figure 11).

It can be seen that the glass-like coating cannot withstand 800°C without damages, caused by shrinkage and burn out of residual organic components. In contrary, the glass-ceramic maintains its homogeneous and amorphous appearance up to 900°C.

Investigations of corrosion protection and wear resistance have shown, that the known protection by alkali glass-like systems is combined with a higher hardness, depending from the amount of ceramic. While these samples possess increased hardness (up to an average of 10.000

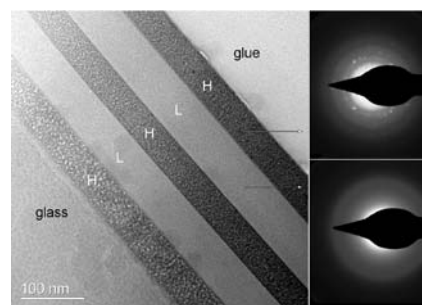


Figure 7: High magnification of cross sectional view of multilayers by HRTEM. Dark layers are TiO₂ layers and bright layers are SiO₂ layers.

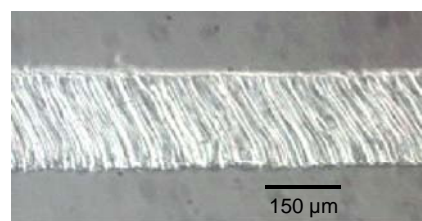


Figure 8: Cross Section View of a Diffuser.

MPa with 20 wt.% SiC) they are further on flexible and can be formed to a certain extent.

Another advantage of the new coating is the easy-to-clean function combined with tribological behaviour. Especially if the coating is sintered under inert atmosphere (nitrogen, argon) at 500°C to 800°C, the contact angles against water have values of 100° to 110° and the surface energy was calculated to be 20,1 mJ/m², which is best result for these temperatures.

For the ball-disc tribometer measurements, a glass-ceramic coating on steel, densified at 800°C at atmosphere, was investigated with CSM instruments. The result can be seen in figure 12.

The glass-ceramic coating withstands the stainless steel ball without damages. It was even superior to the ball, which got abraded itself.

Generally with the new coating an excellent combination of very hard glass-like coatings (with corrosion and scratch/wear protection) and high temperature stable ceramics (with anti-adhesive properties and flexibility) was obtained.

On the base of the newly developed glass-ceramic coatings, variations and optimizations were performed in the scope of an industrial project to trim the glass-ceramic for an insert of extruders. With pigment incorporation (e.g. corundum, titania, mullite) the wear and abrasion resistance should be further increased with parallel improvement of sliding friction by incorporation of tribological powders (e.g. graphite, MoS₂, SiC).

At first, the best systems of the screening were endurance tested. The resistance of the glass-ceramic coatings against PVC powder or melt should be several weeks at 200°C. The surface of the coatings was examined morphological after the exposition and compared with the initial state before exposition. The gained results were base of the optimization phase. The advanced systems of the optimization will be tested and characterized analogous (end of project in July 2008).

Sooner or later, heating elements tend toward corrosion and calcinations depending on used water. Thereby, the heat output will be strongly limited or even cut off due to safety aspects. Therefore, at the INM investigations were performed with two different materials (glass-like and glass-ceramic) to check their anti-limestone properties at high temperatures.

It was shown that an intact coating withstands the industrial used rapid tests without damages. But if thicker wet films are applied, some micro cracks are formed during densification due to shrinkage. These let to defects during rapid tests. Intact areas showed no formation of limes-

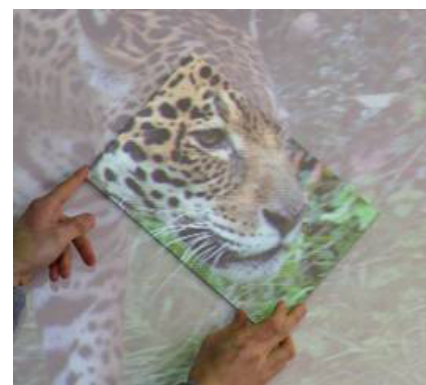


Figure 9: Diffuser in front of a projector screen.

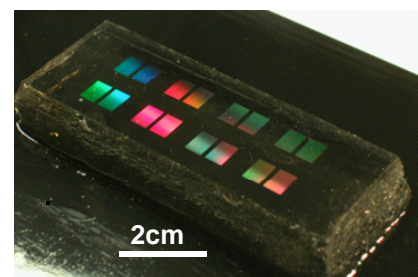


Figure 10: PDMS embossing stamp with micro structures.

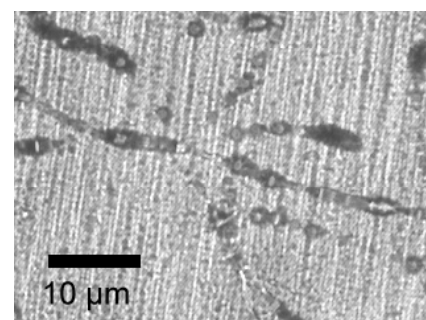
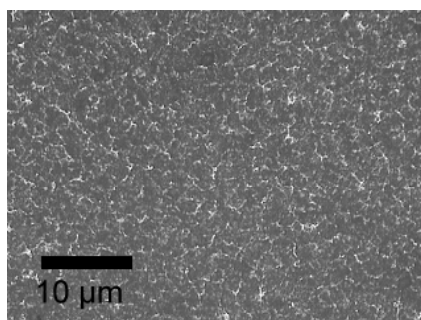


Figure 11: Glass-like (left, thickness 3 μm) and glass-ceramic coating (right, thickness 3 μm) on borosilicate glass after heat treatment at 800°C.



tone (figure 13), which was documented by SEM/EDX.

For future coatings it is very important to know the upper thickness of glass-ceramic wet films, which results in totally defect free coatings (even without micro cracks). Because up to now the spray coating is done manual, the right settings are experience of the spray master. In later on planned applications with spray robots, the adaptation and reproducibility will be improved.

Program Division Nanomers® / Programmbereich Nanomere®

Die Forschungs- und Entwicklungsarbeiten der Abteilung Nanomere® liegen schwerpunktmäßig auf der Synthese, Oberflächenmodifikation und Verwendung von Nanopartikeln in polymerartigen bzw. organisch-anorganischen Matrices zur Herstellung von Nanokomposit-systemen, den so genannten Nanomeren®. Diese Materialklasse besitzt eine hohe werkstofftechnische Variabilität und bietet die Möglichkeit, maßgeschneiderte Eigenschaftsprofile für ganz spezifische Anwendungen einzustellen. Die Aktivitäten der Abteilung Nanomere®, bestehend aus 18 Mitarbeitern, beschränkten sich dabei im Jahre 2007 nicht nur auf Basisentwicklungen für neue Werkstoffkonzepte, sondern umfassten in großem Maße auch industrielle Forschungsprojekte (z.Z. öffentlich gefördert über BMBF bzw. AiF/DECHEMA) zur an-

gepassten Materialentwicklung. Schwerpunktmäßig wurden dabei transparente Kratzfest-Beschichtungen, tribologische Beschichtungen mit hoher Verschleißbeständigkeit sowie Niedrigenergie-Beschichtungen mit hoher Dauerhaftigkeit und elektrischen Isolationseigenschaften sowie kompakte optische Materialien bearbeitet. Weiterhin wurde in den meisten Industrieprojekten zusätzlich zur Materialoptimierung auch eine Technologie-Entwicklung durchgeführt. Hierzu gab es eine enge Zusammenarbeit mit der Applikationsabteilung NMO, um die Resultate vom Labormaßstab in die praktische Anwendung zu überführen. Zusätzlich wurden die Entwicklungen der Abteilung Nanomere® weltweit auf entsprechenden Ausstellungen und Industriemessen 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.



Figure 12: Ball-disc tribometer of glass-ceramic coating (thickness 5 µm) on steel plate (10 cm x 10 cm) after 20 m with 2 N load on stainless steel ball (100Cr6).

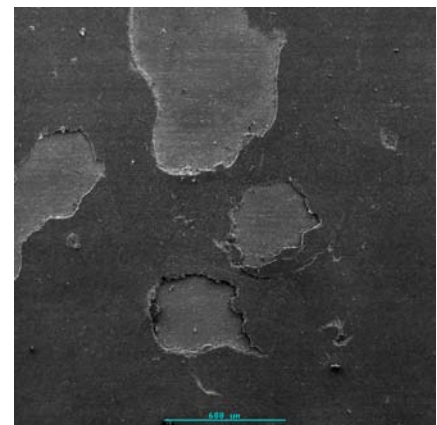
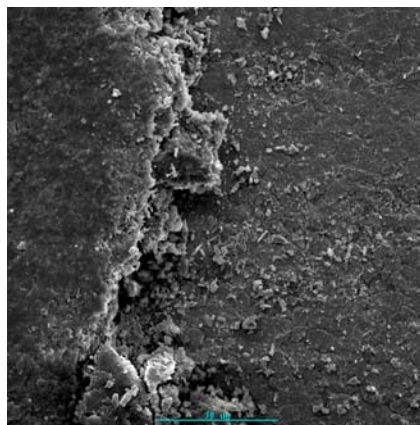


Figure 13: SEM picture of flakings (left 600 µm, right 30 µm) with beginning of limestone at the areas of breaking edges.

The research and development activities in the department 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®. The Nanomers® possess a high variability in their chemical structure and open the possibility to achieve very specific requirements even in difficult combinations. 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 applications. In the year 2007 many activities on the generation of basic knowledge and also on industrial research projects have been performed in the division Nanomers® consisting of 18 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 dealing with coating applications mainly in the fields of abrasion resistant coatings, tribological coatings 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 vari-

ous 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 polyurethane or epoxy resins in order to change e.g. their electrical 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 year before, where low friction coating systems containing solid state lubricants have been developed, which combine a low coefficient of friction with excellent adhesion and corrosion protection on low alloyed steel, also new Nanomer® compositions with low friction and high wear resistance could be developed for the use on plastic parts for the automotive industry and for steel parts in rotating systems. Figure 1 shows a pendulum with a transmission shaft which has been partially coated with a wear resistant Nanomer® low friction coating.

New material combinations and processing technologies allowed to approach the region of the coefficient of friction which is normally covered by hydrodynamic lubrication only. This is an important milestone which shows the great potential of the Nanomer® systems.

In particular in the field of Nanomer® coating systems basic investigations have shown that the use of special nanoparticles in the lower nanometer size range even in a small amount can improve dramatically the long term electrical insulation properties of commercially availab-

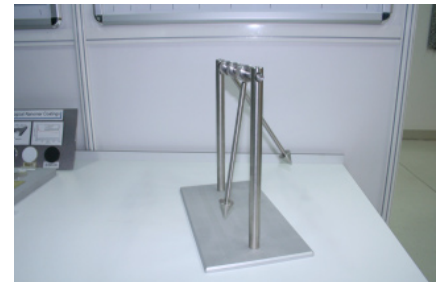


Figure 1: Pendulum with a Nanomer® coated (back side) and an uncoated (front side) transmission shaft.

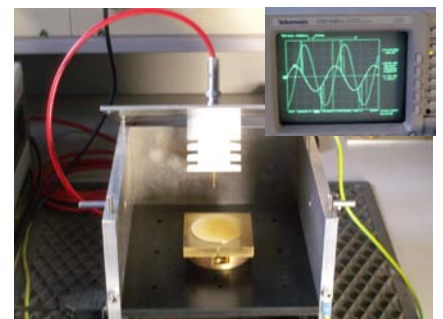


Figure 2: Experimental setup for the determination of electrical insulation properties of Nanomer® coating systems.



le organic coating systems. Nanoparticles with special electronic structure could be successfully incorporated into insulation materials showing 100 times higher insulation capability compared to conventional coating systems. Figure 2 shows the experimental setup for the determination of breakthrough voltage and resistance against partial discharge in insulation coatings.

This principle could be protected with respect to intellectual property and has been successfully used in R&D projects to develop new materials solutions for the electronic and display industry.

It also has been found that nanoparticles can be of interest to improve the mechanical behaviour in UV curable nanocomposite systems. As a result new coating systems with specifically surface tailored nanoparticles could be developed which are applied as reactive curable coating systems showing extremely low intrinsic stress after curing. In addition comparably high loading levels of these nanoparticles could be created in composite coatings which showed a very high elastic modulus and can be patterned in a very fine structure (cp. figure 3). These types of materials are of interest in many areas of the electronic and optoelectronic industry. This approach also has been protected by an appropriate patent application and now serves as a basis for new industrial projects in the future.

During the year 2007 also various projects supported by the federal government of Germany have been executed.

In the area of anti-adhesive coatings a project supported by the AiF/ Dechema (AiF Nr. 14228 N/2) concerning the development of adhesion minimizing coatings for wet processes in the food production industry has been completed as a basic research and development work. New compositions for coating materials useful for the application on already existing equipment in the food production industry with interesting new surface properties could be developed in a tailored way. Figure 4 shows the wetting behaviour of water droplets on such a surface.

The structure - property relationships related to adhesion mechanisms of specific adhering substances and food products could be investigated in more detail together with the R&D cooperation partner in the project. The results could 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 the relevant industrial applications favoured in this project. A practical test has been performed at the end of the project with promising intermediate results, which will help to further optimize the materials in future work.

A quite high investigation effort has also been set on the use of nanoparticulate additives for conventional coatings and lacquer systems with respect to polymerisation catalyzing properties. In a first project supported by the BMBF (FKz.: 13N9119) the investigations have been focused on the effect of nanoparticles ca-

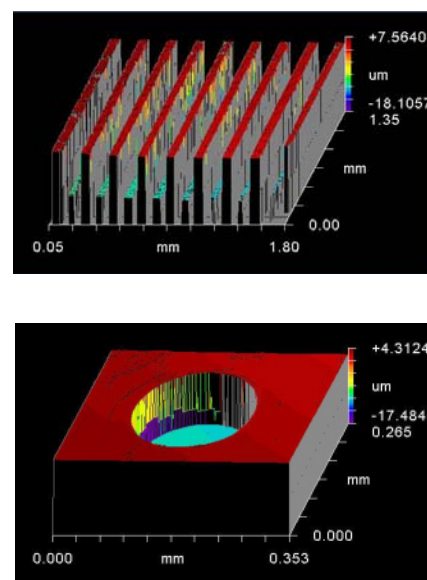


Figure 3: Interferometer micrographs of various patterns generated by photolithography.

talyzing especially UV photo polymerisation reactions in organic coating systems. This intrinsic property of the nanoparticles has high industrial demand and can be a useful alternative for conventional approaches using organic molecules as photo initiators. In a second project supported by the BMBF (FKz.: 03X0032) 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 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 which can be compounded, extruded or injection moulded. By this approach transparent temperature stable resins have been developed which can be used for the fabrication of optical elements. These materials combine easy processability with relatively low density compared to glass and tailor ability of optical properties such as refractive index. In this direction also composite systems derived from epoxy-hybrid matrices and nanoparticles with high density have been developed, which enable to derive a composite with tuneable bulk density which is important to adjust e.g. the acoustic wave transmittance of such type of material. This research activity has been financially supported by the ministry of economics

and science of the state of Saarland. Investments have been made for the future with respect to the equipment, to enable to process not only reactive curable systems but also thermoplastic materials for refractive optical and foil applications.

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

During the year 2007 many projects with industrial partners have been performed in the division, most of them cannot be mentioned because of specific non disclosure agreements. Furthermore it is important to note that for most of the cooperation projects a technology development in addition to the pure materials optimization 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 application centre of the INM, NMO. Furthermore the division has also participated in many expositions on fairs ranging from Germany to Japan and Korea. From the results of these fair activities it could be derived that the Nanomer® technology still has a big potential for a broad ran-

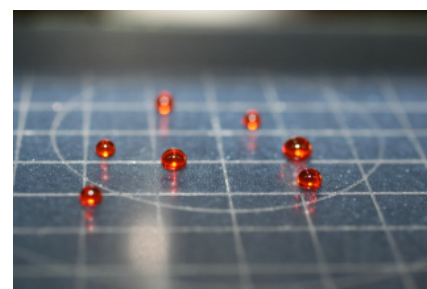


Figure 4: Wetting behaviour of water droplets on an anti-adhesive Nanomer® surface with high durability.



ge of industrial applications especially for e.g. the consumer electronics, 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.

Program Division CVD/PVD Technologies / Programmbereich CVD/PVD-Technologien

Der Fokus des Programmbereiches CVD/PVD-Technologien lag auf der Abscheidung funktioneller Schichten und Nanostrukturen unter Einsatz des plasmaunterstützten und thermischen CVD-Verfahrens. Die gewünschten Materialeigenschaften lassen sich mittels der chemischen Gasphasenabscheidung gezielt variieren, da das Verfahren eine hohe Reinheit sowie eine gleichmäßige Oberflächenbeschaffenheit der abgeschiedenen Schichten aufweist und zudem noch eine konturgetreue Bauteilbeschichtung gewährleistet.

Thin film deposition by CVD techniques plays an important role in the development of both decorative and functional coatings, important for their technological implications. Usually, multi-component materials are prepared from a mixture of precursors; however the efficiency of such processes is hampered by the mis-

match of chemical parameters among the precursor species such as thermal stability, vapour pressure etc. Given the inherent differences in the physico-chemical properties phase separation and elemental segregation are commonly observed in CVD deposited materials (Figure 1).

Functional Nanostructured Coatings by Molecule-based PECVD Techniques

We are exploring low-temperature methods for depositing functional coatings by combining our precursor-based approach with plasma-assisted CVD techniques (Figure 1).

The de-mixing of elements in multi-component systems is thermodynamically driven and sensitive to the chemical behaviour of the precursors. Transformation of precursor compounds possessing bonding features inherent to the solid-state lowers the need of diffusion and counterbalances the thermodynamic impediments. Recently, we have developed several new metal-organic systems and used

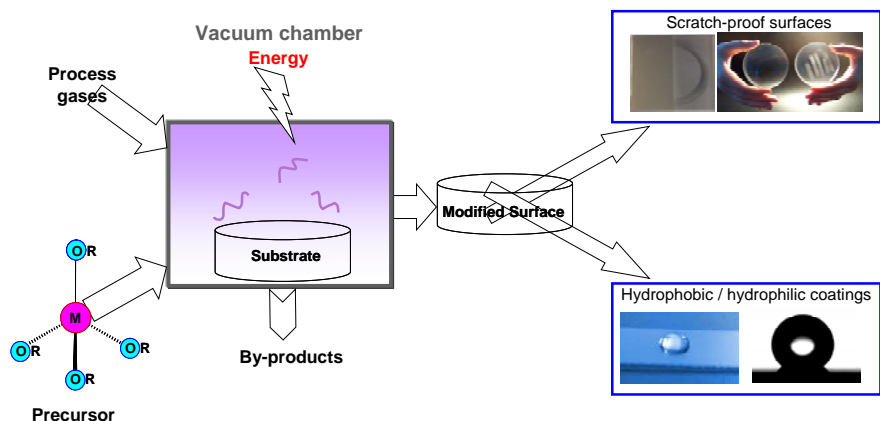


Figure 1: Schematic representation of a PE-CVD process and examples of the functional coatings.

them in plasma-assisted CVD method to deposit decorative and functional coatings on metallic and non-metallic substrates. For instance, we have investigated the decomposition of single molecular sources like $\text{Hf}(\text{O}^i\text{Bu})_4$, $\text{Ti}(\text{O}^i\text{Pr})_4$, $\text{Zn}[\text{N}(\text{SiMe}_3)_2]_2$ and hexamethyldisiloxane (HMDSO) in plasma-enhanced CVD techniques to deposit hafnium oxide (HfO_2), titanium oxide (TiO_2), zinc oxide (ZnO) and silicon oxide (SiO_x) coatings on different substrates as single-layer or multi-layer systems, and have investigated their application as anti-reflective and scratch-resistant surfaces. As basic process we used the deposition of SiO_x nanostructured coatings which act on the one hand as functional layer and on the other hand simultaneously as a matrix for depositing a secondary metal-organic component. The development of these functional thin film systems comprises first the alternating utilization of various precursor systems for adequate multilayer systems and second the parallel use of the precursors to obtain mixed or graded materials with new compositions and properties.

The plasma-activation process enhances the deposition rates and is suitable for deposition at low temperature substrate like polycarbonate, the film attributes are sensitive to the deposition conditions and a small variation can be detrimental to the film properties.

In addition, we have developed in collaboration with Korean Institute of Technology (KITECH) a new class of tribological coatings on Al substrates (Al 7075, Al 6065). For this purpose, a ti-

tanium amide precursor was decomposed in a plasma activated process to obtain uniform dense coatings containing Ti, N and C as main constituents. These films are strongly adherent to substrate and show metallic glaze (Figure 2).

The analytical work related to their functional properties and scaling up of the coating procedure is underway.

Post-deposition plasma treatment of SnO_2 nanowires

Gas sensors based on nanostructured metal oxides are emerging as a powerful platform for the detection and quantification of gases, even at lower concentration due to their enhanced ability compared to bulk materials, the confined electronic proper-

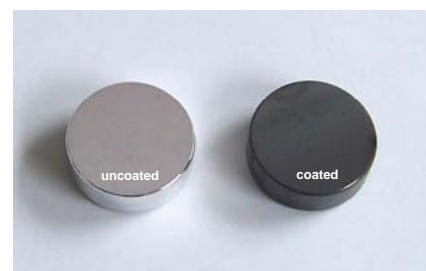


Figure 2: Uncoated (left) and coated (right) Al substrates.

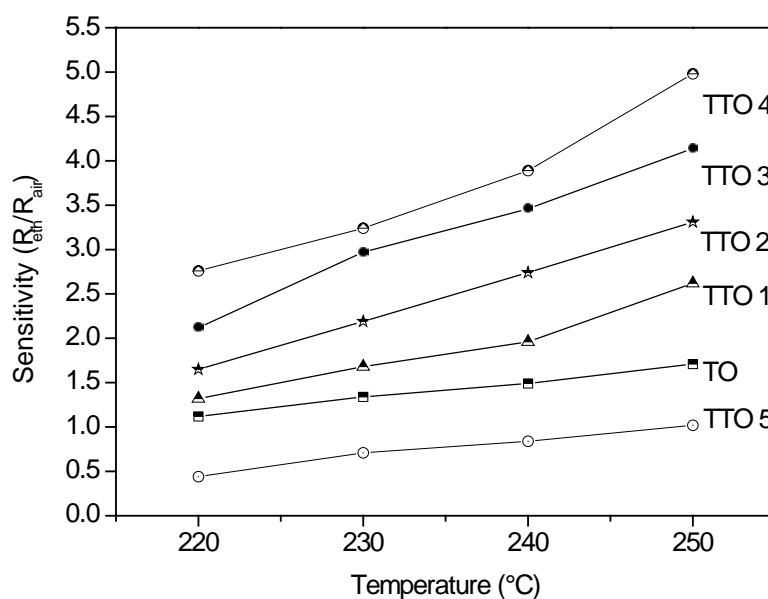


Figure 3: Temperature-dependent sensor response of as-deposited (TO) and plasma-treated tin oxide (TTO) nanowires.

ties as well as the huge surface-to-volume ratio, augment the sensor response. In this context tin oxide nanostructures are excellent primary transducers for producing signals, as the diameters of those nanowires are comparable to the charge penetration layer (Debye length, λ), which offer novel and unique properties to improve the efficiency of the gas sensing mechanism.

SnO₂ nanowires configured as CO and ethanol sensors, were synthesized by chemical vapor deposition of [Sn(O^tBu)₄] in the temperature range from 650 to 700°C. Post-deposition modifications of tin oxide nanowires to study the physical and chemical properties of the tin nanowires were carried out by treating them in radio frequency plasma of Ar and O₂. We could show that the plasma treatments modify the chemical composition and lead to incorporation of metallic impurities (Au/Sn) in as-deposited nanowires. Tin-in-Tin Oxide nanowires (TTO) were obtained by preferential etching of lattice oxygen atoms from the surface of SnO₂ nanostructures through treatment in Ar/O₂ plasma environment. Gold-in-Tin Oxide (GTO) nanowires were obtained by sputtering Au (0.5-1.3 nm) followed by low plasma treatment for 10 minutes to permeate Au into SnO₂ matrix. Metal-functionalized nanowires showed better sensitivity for CO and ethanol at lower operating temperature and exhibited improved response towards changing gas atmospheres (Figure 3).

The enhanced sensing functionality of the nanowires was attributed to the ca-

talytic dissociation of molecular adsorbate on metal functionalized SnO₂ surfaces and subsequent diffusion of atomic species to the semiconductor surface. TEM observations (Figure 4) confirmed the morphological changes of the surface due to the sputter-redeposition mechanism as a result of the reducing plasma treatment.

For Au-doped samples, dramatic improvement in sensing performance was attributed to the spill over effect caused on the oxygen molecule into atoms by Au sensitised surfaces.

Program Division Life Sciences / Biomimetics / Programmbereich Life Science / Biomimetik

Im Programmbereich „Life-Science / Biomimetik“ werden nanoskalige Systeme für medizinische sowie für bio- und lebensmitteltechnische Anwendungen entwickelt. Zur Zeit wird stark grundlagenorientiert in öffentlich geförderten Projekten gearbeitet, jedoch mit einem großen Anwendungsbezug.

Es wurden Arbeiten an anorganischen Nanopartikeln durchgeführt, die auf ihre Eignung als Träger von Wirkstoffen in der photodynamischen Therapie (PDT) eingesetzt werden sollen. Dazu sollen verschiedene anorganische Nanopartikel entwickelt und deren Wechselwirkung mit humanen Zellen aus unterschiedlichen Geweben untersucht werden. Um den Verbleib der Partikel verfolgen zu

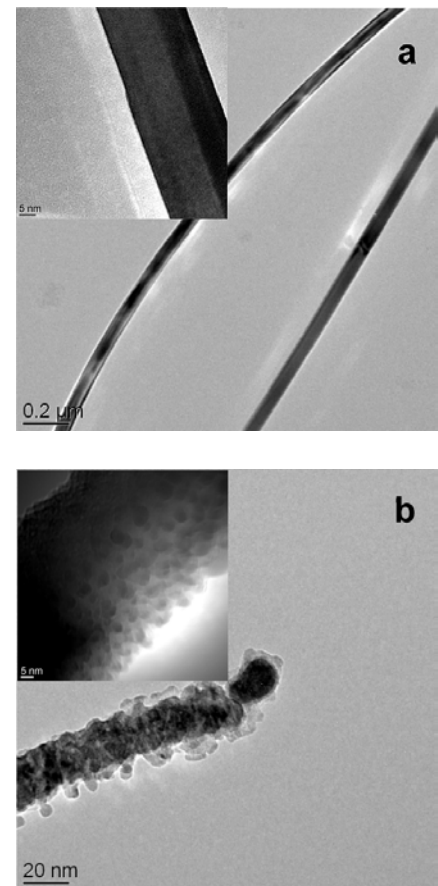


Figure 4: TEM images of as-deposited (a) and plasma treated (b) nanowires.

können, sind diese Partikel mit Fluoreszenzmarkern versehen worden.

Für das EU-Projekt CellPROM wurden topographisch und chemisch strukturierte Oberflächen entwickelt. Dazu ist auch die Stabilität der Anbindung von Oberflächenmodifikatoren, die als Linker zur Anbindung von Biomolekülen dienen sollen, auf diesen Materialien unter den Bedingungen in der Zellkultur untersucht worden.

In dem BMBF-Projekt ZOVAN wurde eine Beschichtung entwickelt, die Zähnen eine unter den Bedingungen der Mundhöhle antiadhäsiv wirkende Oberfläche verleiht.

Based on chemical nanotechnology, the program division "Life Science/ Biomimetics" focuses upon 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 2007 is shortly presented.

Inorganic Particles as Drug-Carriers

The interaction between nanoparticles and living cells is a very promising field, from a toxicological as well as from a pharmaceutical point of view. In two German-Chinese projects, supported by the German ministry of education and research (BMBF) (FKN: 0312026A and 0312025H), the interaction of nanoparticles with human cells is investigated in order to improve their usability as

drug carrier in the photodynamic therapy (PDT). For this purpose the INM develops inorganic nanoparticles of various compositions, sizes and shapes.

The specific requirements to the particulate suspensions for this type of applications are rather tough. First of all the particles have to be non-toxic. Then the particles have to be stable against agglomeration even in cell-culture media, which is not evident; since inorganic suspensions are normally stabilized by electrostatic repulsion, they are very sensitive to stronger ionic forces and concentrations. A modification of the particles is necessary to shift to another stabilizing mechanism. Because the distribution of the drugs is monitored in the living cell, both the drug as well as the particles have to be fluorescent. And last but not least, all suspensions have to be sterile, since cells of the human immune system are involved which will react even to metabolites of microorganisms.

The first type of particles synthesized were fluorescent silica nanoparticles, which were obtained by a modified Stöber-process (figure 1).

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

Participation in CellPROM

The task of the INM within this EU-funded integrated project was to develop physically and chemically nanostructu-



red surfaces for cell imprinting. The surface was expected to have a nanostructured topography as well as it should bear functional groups for the covalent binding of biomolecules.

Two approaches were made: 1. Nano-composite coatings structured by embossing and equipped with functional groups necessary for conjugating biomolecules. 2. Nanostructured Al/Al_2O_3 composite surfaces derived by gas phase methods. In order to improve the biocompatibility this surface-material can be functionalized as well.

For grafting of biomolecules monolayers of a linker can be put on the surfaces.

Because TiO_2 -surfaces were also employed, the stability of the grafting of linker to this material was tested. Therefore, patterned TiO_2 surfaces were modified with an aminosilane (3-aminopropyl-triethoxysilane, APTES). Silanized samples of TiO_2 layers on glass slides were stored for various times (3, 7 and 15 days) under phosphate buffered saline-(PBS)-solution at $37^\circ C$. Then, the surface-composition was determined by X-ray-photoelectron-spectroscopy (XPS). Figure 2 shows a XPS spectrum of such a modified surface before immersing in PBS-solution.

In all samples titanium could be found only in traces on the surface, because the surface was well covered by the aminosilane-molecules. The concentrations of all measured elements (Ti, O, Si, C, N) seemed to be independent of the time the sample was immersed in PBS, within the tolerance of the measurement. This re-

sult proves that the Si-O-Ti-bond exhibits fairly good stability during storage in a buffer solution during the observation time.

Development of a Dental Coating

Another activity was related to the development of a new dental material. A nano-composite coating had to be developed preventing the building up of thick plaque-layers and the colonization of the dental surface by biofilm-forming microorganisms. The idea was to establish a permanent anti-adhesive surface on teeth. This antiadhesive layer was based on compounds having perfluorinated side chains, which enrich themselves at the surface of the coating during curing.

The thicknesses of these fluorine-rich layers were investigated by XPS combined with a sputtering process. At the surface

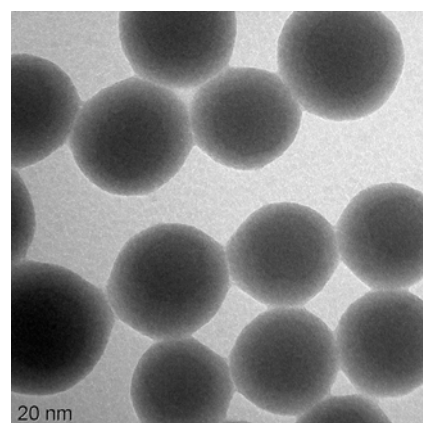


Figure 1: Fluorescent SiO_2 -nanoparticles.

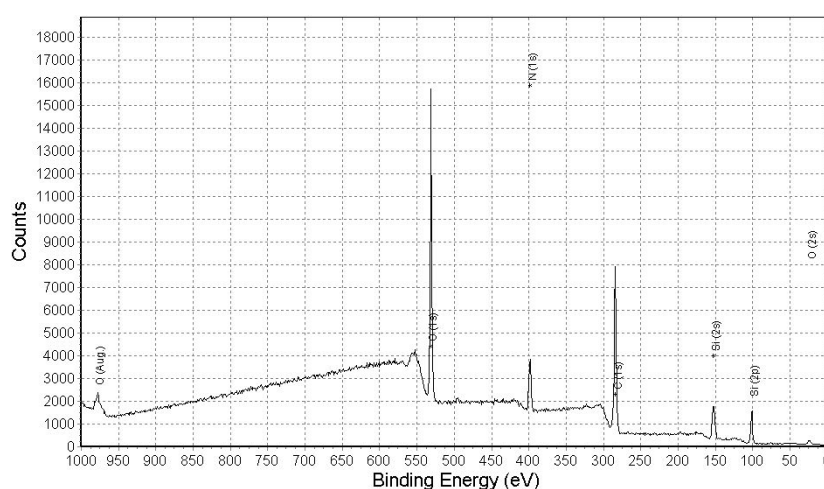


Figure 2: XPS spectrum of a TiO_2 surface modified by APTES before immersing in PBS-solution.



Programmbereich Nanoprotekt und Kombinatorische Werkstoffentwicklung

Dr. Matthias Wittmar



very high fluorine contents were found (figure 3). The thickness of these fluorine rich zone was relatively thin, below 200 nanometers. Their thickness can be influenced only slightly by incorporating the fluorine compounds into larger polymeric units.

Toxicological tests undertaken by project-partners with our coating material showed a good compatibility.

This project was also supported by German ministry of education and research (BMBF) (FKN: 03X1501A).

Program Division Nanoprotect / Combinatorial Materials Development / Programmbereich Nanoprotekt / Kombinatorische Werkstoffentwicklung

Im Laufe des Jahres 2007 wurden die Gruppen Keramik, Binder und Isolationsstoffe, Oxidationskatalyse, Korrosionsschutz und Kombinatorische Werkstoffentwicklung unter der Leitung von Dr. Matthias Wittmar zu einem Programmbereich „Nanoprotekt/Kombinatorik“ zusammengefasst. Der Programmbereich hat die alten in den Untergruppen bearbeiteten Projekte übernommen und im Laufe des Jahres sowohl Industrieprojekte als auch öffentlich geförderte Projekte akquiriert. Neben Industrieprojekten, die hier nicht näher genannt werden dürfen, und dem EU-Projekt MULTIPROTECT, das unter der Leitung des

INM fortgesetzt wurde, wurden im letzten Jahr die Arbeiten im AiF-Projekt zur Verbesserung der Haltbarkeit von Holz gegenüber UV-Licht fortgesetzt. Außerdem wurden einige neue EU-Projekte erfolgreich eingeworben.

During the last year the former research groups ceramics, corrosion protection, oxidation catalysis, combinatorial materials development as well as binder and insulation materials were merged into one new program division “Nanoprotect / Combinatorial material development”. Head of this new division is Dr. Matthias Wittmar, since the end of 2006 responsible for the corrosion protection group, the oxidation catalysis and combinatorial material development.

In 2007 the main project within the division was the EU-project MULTIPROTECT. Other important projects were made with industrial partners and AiF. A new 3-years-project concerned with soil proofing of horizontal precast concrete block areas has started in 2007.

In the combinatorial material development experiments for the EU-project MULTIPROTECT were continued. New equipment was integrated into the automated liquid handling system. A micro- and nano-litre pump was purchased from the Micro-Mechatronics Technologies AG. With its help the dosage of liquids in micron and submicron range is now possible. This will enable the screening of mixtures of liquid substances

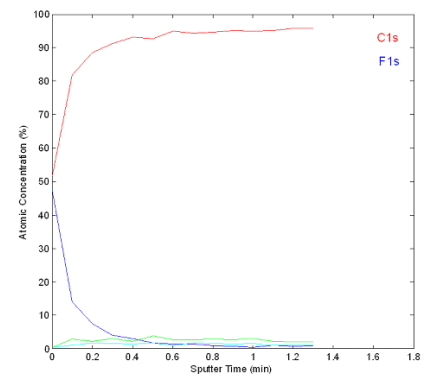


Figure 3: Profile of the fluorine content in the layer.



with rather different amounts of the single components. Efforts were undertaken to find new corrosion inhibitors by the combination of various inorganic salt solutions. The mixtures were tested in drop test for their potential in the inhibition of aluminium corrosion. At the end of the year a special balance for automated liquid handling systems were purchased. Its integration is currently under way. After completion a direct transfer of lab scale coating receipts to the robot and back will be clearly simplified.

The work of the corrosion protection group was mainly focussed on the EU-project MULTIPROTECT (Contract N° NMP3-CT-2005-011783, speaker Prof. M. Veith), which focuses on the replacement of hexavalent chromium in the field of corrosion protection of metals. Within the scope of the project the coating systems that proved to be optimal on the substrate materials steel and aluminium were further optimized in order to fit the industrial needs.

During 2007 further research efforts were undertaken in order to find and incorporate corrosion inhibitors like porous containers in nanometric range filled with inorganic or organic compounds into corrosion protection coatings. Especially the use of benzotriazole filled containers showed synergistic effects in the corrosion protection of aluminium alloys, while the use of cerium salts as inorganic compounds yielded no positive effects.

In a series of experiments the amount of inhibiting material released from the coating materials was monitored over time.

These experiments were carried out to determine the release kinetics of the coating material. It turned out that materials with a high inhibitor release rate tend to show a limited corrosion protection capability while coatings with a negligible inhibitor release tend to yield better protection.

Other inhibitors like cerium nitrate and 3-amino-1,2,4-triazole were tested on different steel substrates DC01 and DC04 with measurable success.

The effect of doping the coating system with cerium nitrate in regard to the long-term barrier performance of the Zr primed DC04 samples in comparison to the undoped coating system were tested by means of EIS. The impedance modulus Z at 0.01 Hz of the coated steel samples versus the immersion time in a 3.5 wt. % NaCl solution is presented. Contrary to the bad performance of the unfilled system on Zr primed DC04, the cerium nitrate doped system showed excellent barrier properties and the test is still running without visible coating defects up to now.

Combinations of CeO_2 -nanoparticles with conductive nanoparticles were examined for their corrosion protection abilities. The idea was that a sol-gel coating filled with both conductive and inhibiting nanoparticles should result in an increased activity of the inhibiting substances. In case of an injury of the coating not only the inhibiting particles in direct neighbourhood to the scratch should deploy their self-healing or protection potential, also the inhibiting particle farther

from the scratch should help to protect the metal surface. Due to the included conductivity a permanent reaction between the metal surface, the inhibitor near the scratch and the inhibitor in the coating away from the scratch should be possible because of a broadening of the potential drop over the conductive particles. With the addition of an amount of conductive nanoparticles comparable to that of CeO_2 it was tried to verify this hypothesis.

In order to determine the influence of CeO_2 , conductive nanoparticles and a possible synergistic effect, four different sets of samples were produced: pure coating material, coating material doped with CeO_2 , coating material doped with conductive particles, and coating material doped with CeO_2 and conductive particles, respectively. All coatings were prepared from one batch of coating material. In order to exclude the influence of the milling process, the blank coating was also milled for the same time as the other materials. Afterwards the coatings were applied via the dip coating process.

The pure coating material showed the first pitting after 1100 h SST. At the artificial scratch creepage could be observed from the beginning. The coating material doped with conductive nanoparticles showed a comparable performance, the creepage was even more pronounced than in the former case. The coating material containing CeO_2 showed first pittings after 166 h SST. The coating material including a combination of CeO_2 and conductive particles showed even more pit-

ting than the CeO_2 doped coating material after the same time. In the two CeO_2 containing coatings creepage was negligible after 330 hours.

From these results can be concluded that CeO_2 suppresses creepage to some extent. When CeO_2 particles are missing in the coating the creepage is very pronounced from the beginning. On the other hand no pitting is observed in the pure coating material and the conductive particle doped coating. CeO_2 particles seem to weaken the barrier properties of the coating material. The combination of CeO_2 and conductive particles in the coating matrix thus did not lead to an improvement in the corrosion protection abilities but helped to determine the influence of particles.

In the reporting period, an industrial project has been continued. It covers the cleaning of gases from organic contaminations in the ppt-range by catalytic oxidation. Parameter studies with a test bench under realistic conditions have been conducted successfully after a time consuming adaptation of the analytical procedure to the test conditions (cp. last annual report). New types of porous substrates have been coated with catalytic coatings in order to develop a prototype together with the project partner.

In cooperation with other members of the Nanoprotect department, two more projects in the fields of ceramic and binder development have been assisted. Additionally, supervision and operation of thermal analysis equipment like the Netzsch DSC-TG-FTIR-MS coupling as a service



for other groups of INM has been expanded noticeably.

Fundamental research has been done in the frame of a PhD study by application of a new parallel synthesis technique (cp. last annual report) to study dielectric properties in the ternary system CuO-TiO₂-CaO. The samples with compositions near CaCu₃Ti₄O₁₂, sintered at 1050°C showed highest dielectric constants, which is consistent with the previous results. The dielectric analysis for lower melting samples in the CuO-rich region sintered at 950°C showed more than 20 compounds having dielectric constants larger than 1000. Phase relationship analysis was partially done. Further detailed investigation to study the composition-structure-property relationship in this system is underway.

The research topics of the group binder and insulation materials (head: Dr. B. Reinhard) were continued in 2007 and will be aligned with respect to potential synergy effects with the other research areas of the department. In general the proved material basis with binder, fire protection material and special glass-like, functional coatings should be retained and used for further special applications requiring multifunctional or innovative aspects. Investigations in reactivity control, aging stability, VOC-content, potlife and recurrent use show high potential for optimized applications in foundry, automotive, oil, wood and building material industry. Analytical investigations with dynamic mechanical analysis, HP-rheometry, HP-DSC, DSC/TG/FTIR/MS

and NMR were useful methods in the characterization of such materials. The new acquisition of a microwave oven at the end of 2007 will open access to new materials via enhanced microwave assisted syntheses. With respect to this topic further acquisitions were made for the installation of the new research field "natural products chemistry".

A nationally funded project for the improvement of domestic wood for outdoor applications was started in 2006 and has been continued in 2007. Aim of project is the protection of fast growing domestic wood against the effect of weathering and UV irradiation. By impregnation with nanocomposites, the dimensional stability of the wooden substrates under changing climate conditions could be optimized. An effective protection of the wood surface with sol-gel-based materials against the destructive effects of weathering and UV irradiation is directly connected to an efficient dimensional stability of the wooden body. UV protection could be realized by UV absorbing ZnO nanoparticles containing sol-gel-based coating compositions. The transparent coated wooden substrates were characterised by artificial weathering and outdoor exposure according to the protecting effects (AiF14897 N/4).

A second public funded project was started in November 2007 and resides in the INONET funding program of BMWi (UNACON, Aktenkennzeichen: NETE012, Verbundnummer: INE3036). This project will be concerned with soil proofing of horizontal precast concrete

block areas and will release an innovative pulse for the building material industry. The development of a multifunctional nanoadditive with compatibility to concrete mixtures and intrinsic soil resistant characteristics of concrete stones will be the aim of this project.

In quarter four of 2007 the ceramics group (head: Dr. J. Adam) was merged in the program division “Nanoprotect and Combinatorial material development” due to the reorganisation of the INM. The long lasting work of the group on ceramic nanoparticles and on nanoparticle deagglomeration continued in 2007.

A series of experiments comprised the treatment of zirconia nanoparticle dispersions in a stirred media mill. It can be concluded that this method combining mechanical deagglomeration and chemical surface modification is advantageous for the manufacture of stable colloids. Further, for the “larger” nanoparticle types comminution effects occur. Results and conclusions are briefly described in an article that has been submitted to a journal in Q1/08.

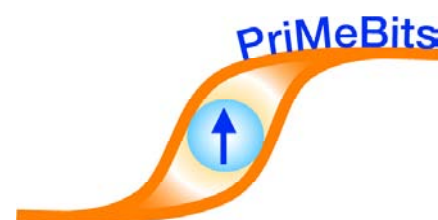
In 2006 we started to work on special ceramic coatings. They are based on wet coating methods using particle suspensions as starting material. After the application of the suspension (for example by spraying) and its drying on a substrate it is possible to obtain porous ceramic coatings after thermal treatment at 500°C, for example. Considering that this temperature is far below usual sintering temperatures the coatings undergo a remarkable mechanical consolidation due to

bonding caused by nanoparticles. Two related articles will be published in the proceedings of the 10th International Conference of the European Ceramic Society which took place in June 2007 in Berlin.

Under the coordination of the Finnish research centre VTT, the INM was part of a consortium (10 partners) applying for a project in the frame of EU / FP7 / ICT. INM’s contributions to the proposal have been coordinated and prepared by the ceramics group, strongly supported by INM colleagues from other departments. Since January 1st, 2008 the EU funds this project under the acronym PriMeBits (Grant agreement no.: 215132, Call FP7-ICT-2007-1) and the title ‘Printable memory solutions for sensor, ID, and media applications’ (<http://primebits.epfl.ch>). INM’s main role in the project is the synthesis, dispersion and characterization of ferroelectric nanoparticles for printable memories.

In the reporting period a special ZrO_2 dispersion has been prepared, characterized and provided to support a PhD study at the Institute of Pharmaceutical Biology of the Saarland University. ZrO_2 nanoparticles (themselves having a low zeta-potential at pH ~7) have been successfully surface modified with a silane to equip them with an electrosteric stabilisation mechanism in pH neutral aqueous media for in vitro cell tests.

Since the beginning of 2008 Bühler PARTEC GmbH is offering ZnO nanoparticle dispersions based on several organic dispersion media, e.g. for UV protection applications. The ceramics group parti-





participated in the development of this commercial product by executing a related feasibility study for Bühler PARTEC in 2006 and 2007.

The work on the potential corrosion inhibitors has been continued in 2007. Nanoparticles have been synthesized, dispersed and characterized or doped with corrosion inhibitors. The colleagues of the MULTIPROTECT project incorporated the particles in corrosion protection coatings and tested these coatings on metal substrates.

In September 2007 our former Korean colleague Taejin Hwang (KITECH) defended his PhD thesis on “Silica / Polyaniline Composite Nanoparticle Produced in an Inverse Microemulsion Solution for the Electrochromic Applications” successfully.

Program Division Application Centre NMO / Chemical Engineering / Programmbereich Anwendungszentrum NMO / Verfahrenstechnik

Seit der Gründung des Anwendungszentrums ist dieses das zentrale Element des INM für den Technologietransfer. 2007 wurden insgesamt 6 Kleinprojekte und 2 eigenständige Großprojekte zur industriellen Umsetzung von Laborentwicklungen durchgeführt. Im Rahmen von internen Kooperationen übernahm das NMO in 6 Projekten die Durchführung der Technologiephase. Die Projekte be-

inhalteten das Scaling-Up von Rezepturen, die Entwicklung nasschemischer Applikationsverfahren einschließlich geeigneter Applikationswerkzeuge sowie die Pilotserienfertigung. Unterstützt wurde das NMO durch die 2007 eingegliederte Chemische Verfahrenstechnik, die auch eine eigenständige Grundlagenforschung betreibt. Es wurde hier eine Diplomarbeit zur elektrochemischen Abscheidung von oxidischen Materialien abgeschlossen, sowie eine Dissertationsschrift zur Herstellung neuer temperaturstabiler Cellulose-Siloxan-Komposite eingereicht.

Since its foundation the Application Centre represents the central element at INM concerning the transfer of lab scaled developments to industrial application. To reach this an intensive interaction between NMO and the other program divisions is needed. With regard to the close linking to the process engineering group – which dealt with one of the biggest work packages during the industrialization of lab results – this group was integrated in the program division to form the Program Division Application Centre NMO / Chemical Engineering.

As in the last years the topics in the Application Centre were focused on the industrial application and technology transfer of results obtained during the investigations in the R & D departments of INM, while the Chemical Engineering supported NMO and executed independent ba-



Figure 1: Clean room facility

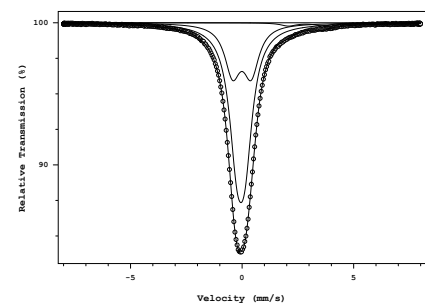


Figure 2: ^{119}Sn Mössbauer spectrum of $\text{In}(\text{OH})_3:\text{Sn}$. Doublet fitting giving an amount of 98 % Sn^{4+} and 2% Sn^{2+} with a corresponding isomer shift of 2.85 mm/s for the Sn^{2+} component.

sis research in the field of new synthesis routes of nano materials syntheses.

The projects of NMO / Chemical Engineering were executed by seven employees (chemists, physicist, process engineer, physical-technical-assistant, laboratory assistant, varnisher).

In 2007 NMO performed 6 small scale projects, 2 independent large scale projects and 6 projects in cooperation with other INM program divisions.

In the small scaled projects - typically provision of samples - the services varied in a wide range:

- NMO synthesized in a project easy-to-clean lacquers (ETC) in small amounts (up to batches of 5 l) for pilot productions in the industries.
- Nanomere® coatings with ETC properties were applied in an industry project by dip-coating techniques and curing.
- As support for small and medium sized enterprises NMO rented technical equipment for materials synthesis and lacquers applications.
- Large scaled projects dealt with the synthesis of lacquers and their application.
- A pilot line was transferred to production scale. After technology transfer the company was ready to produce the coating by its own, renting the needed equipment and services at NMO/INM for a transition time before starting the own production plant.

- Synthesis and application of ETC Nanomere® coatings on metal sheets in pilot scale.
- Internal cooperation.
- In cooperation with the program division Glass and Optics, in three projects lacquers were synthesized in batch synthesis (max. batch size 100 l).
- The technology transfer step of Nanomere® industry projects was executed in three projects by NMO.

Education and advanced training of scientists was focused on basic research:

- Benjamin Rabung finished his diploma thesis which dealt with the examination of an electrochemical deposition process to synthesize nano-scaled (mixed) metal oxide particles and the characterization of the obtained materials. It could be shown that starting with metal or alloy electrodes with the desired metal composition in a water based electrolyte a pre-product could be obtained which formed nano scaled oxides after annealing. The products were characterized by chemical and physicochemical methods like EDX, SEM, TEM, XRD, XPS and Mössbauer spectroscopy. To get more detailed information about the chemical processes at the surface of the electrode the studies of the synthesis of nano-scaled ITO and nano-scaled ZnO were continued within the doctoral thesis of Benjamin Rabung started in June 2007.



Figure 3: Three step glass cleaning automat (Buxtrup)



- Mark Klook finished his practical work for his doctoral thesis and submitted his thesis about new materials based on renewable-materials for new production technologies to the deanery. The new compound materials were synthesized with the main focus on the relation between mechanic strength and temperature resistance. Tension rods for mechanic examinations were formed in a moulding press followed by a drying step. The analysis of the experiments demonstrated that it is possible to create new compounds based on renewable materials. Looking to the data of the dynamic mechanic investigations it could be shown, that the new material has an interesting thermal stability with a glass transition temperature of approximately >100 °C. In 2008 the thesis will be finished with the colloquium.

Service Group Chemical Analytics / Servicebereich Chemische Analytik

Interne analytische Untersuchungen zu laufenden Projekten des INM bildeten im Jahr 2007 den Schwerpunkt des Servicebereichs Chemische Analytik, ca. 20 % wurden für Routinemessungen der Wareneingangskontrolle und ca. 10 % für externe Auftraggeber, hauptsächlich Spin-offs des INM, durchgeführt.

Für die Charakterisierung von Reaktionsabläufen bei der Materialherstellung und die Ermittlung der Zusammensetzung und des Aufbaus von im INM synthetisierten Materialien werden die Flüssig- und Festkörper-NMR-Spektroskopie, Flüssig- und Gaschromatographie (HPLC, GC / MS) sowie atomspektrometrische Methoden (ICP OES, AAS) genutzt.

Mit gaschromatographischen Verfahren wurden im Berichtszeitraum beispielsweise nicht hydrolysierte flüchtige Anteile, wie z. B. MPTS, FTS, MTEOS, PhTES und Lösemittel in synthetisierten Lacken und Nanopulvern quantitativ bestimmt, die unterschiedlichen Stufen der Waschwässer der Nano-ZrO₂-Synthese des Institutes auf Abfallprodukte untersucht und verschiedene Chargen Erdöle

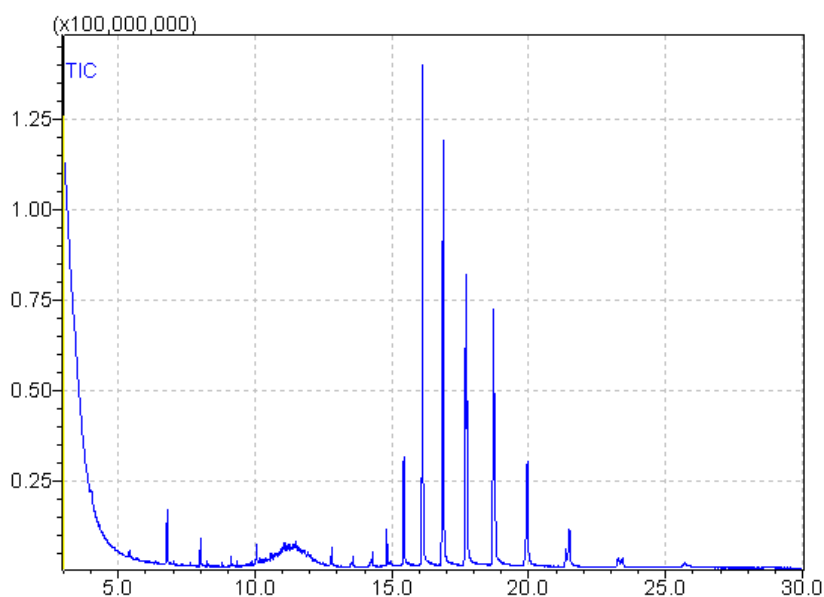


Figure 1: Gas chromatogram of coconut oil (dilution 1:1000 in hexane, 0.5 µl).

und herkömmliche Pflanzenöle (siehe Figure 1) qualitativ analysiert.

Für die qualitative und quantitative Bestimmung nichtflüchtiger Komponenten in flüssigen Proben mit HPLC wurde ein automatischer Probengeber gekauft, der die zeitaufwendige manuelle Einzelinjektion der Proben ersetzt. Mit HPLC wurden beispielsweise Rückstände an Photoinitiatoren in Druckerfarbe auf bedrucktem Papier und Polypropylenfolie quantitativ ermittelt (siehe Figure 2) und die Konzentrationen an Benzotriazol in Wasser, in denen Korrosionsschutzschichten lagerten, analysiert.

Für die ICP OES wurde eine neue Zerstäuberkammer nach Scott, die gekühlt für die Analyse organischer Probenmatrices eingesetzt werden kann, und ein Ringspaltzerstäuber mit 0,7 mm Platin-Kapillare für den Durchsatz von Suspensionen gekauft.

Im November wurde eine Kooperation mit Frau Professor Bauer von der Arbeitsgruppe Molekulare Pflanzenbiologie und Botanik des Fachbereichs Biowissenschaften an der Universität des Saarlandes gestartet. Frau Professor Bauer untersucht die verschiedenen Transportphänomene von Metallen in Pflanzen, z. B. Arabidopsis und Tomate, und das INM analysiert quantitativ die Eisen-, Zink-, Kupfer- und Mangan-Gehalte mit direkter Feststoff-AAS. Diese Graphitrohr-Messmethodik (siehe Figure 3, Feststoff-AAS) erweist sich als besonders günstig, da nur kleinste Probenmengen zwischen 50 und 500 µg pro Feststoffeinwaage benötigt werden und die unverfälschte Ori-

ginalprobe ohne vorherige zeitintensive Aufschlussverfahren und Verdünnungen analysiert werden kann. Dadurch können Blindwert- und Kontaminationsprobleme erheblich minimiert werden.

Bereits Ende des Jahres 2006 wurde ein neues NMR-Gerät Avance II der Fa. Bruker (siehe Figure 4) angeschafft. Es handelt sich hierbei um ein hochauflösendes digitales NMR-Spektrometer mit einem Magnetfeld von 7,05 T, was einer Protonenresonanzfrequenz von 300 MHz entspricht. Die Abschirmung des Magnetfeldes erfolgt mit der UltraShield™-Technologie der Fa. Bruker, durch die die Magnetfeldstärke auf einen Wert von 0,5 T im Abstand von lediglich 70 cm vom Magneten reduziert wird. Dies führt einerseits zu einem kleineren Risiko des Bearbeiters durch elektromagnetische Felder und erlaubt es andererseits, das

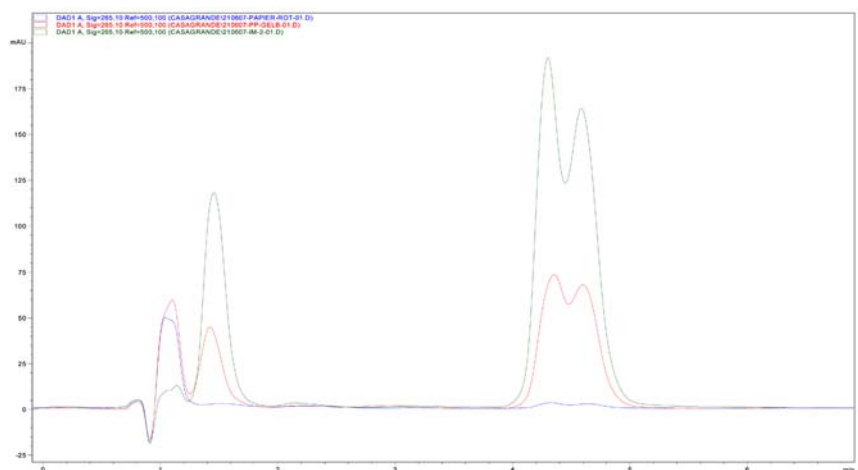


Figure 2: HPLC chromatogram at wavelength 265 nm, green: photoinitiator 0,016 % in ethanol, red: yellow printing paint on polypropylene film, 1x2 cm², leaching 10 min in 1 ml ethanol, blue: red printing paint on paper, 1x2 cm², leaching 10 min in 1 ml ethanol.



bereits vorhandene NMR-Gerät im gleichen Raum problemlos weiter zu betreiben.

Das Spektrometer ist mit zwei austauschbaren Probenköpfen ausgestattet:

- Ein BBFO-Breitband-Probenkopf zur Messung von ^{19}F sowie aller Kerne im Frequenzbereich von 31P bis 15N. Die Frequenz des Probenkopfes kann automatisch abgestimmt werden.
- Ein Si-selektiver Dual-Probenkopf zur Bestimmung von ^{29}Si über den vollständigen Resonanzbereich.

Das neue NMR-Spektrometer und die Auswertungssoftware TopSpin® sind zur Durchführung aller gängigen ein- und zweidimensionalen Untersuchungsmethoden geeignet.

Die NMR-Spektroskopie wird am Institut neben der Durchführung von Routinemessungen, hierzu zählen Wareneingangskontrollen von Silanen und anderen Chemikalien, insbesondere zur Reaktionskontrolle bei der Solsynthese durch Bestimmung von Hydrolyse- und Kondensationsgraden, sowie zur Untersuchung von Polymerisationsreaktionen und zur Identifizierung der dabei entstehenden Reaktionsprodukte eingesetzt.

The most important area of activities of the service group chemical analysis is the suitable solution of analytical tasks for current projects of INM. This focuses on the selection of the suitable method and

the development and adaptation of the method parameters.

As every year a vitally important task (about 20 %) was the routine inspection of incoming goods such as solvents and precursors for the coating sol synthesis of the new materials, which are delivered in barrels. The reagent grade of these chemicals is proofed and the quantitative analysis of trace elements is performed continuously by methods of GC / MS and atomic spectrometry.

10 % of all applications are external investigations, especially for spin-offs of INM.

For the characterization of reactions during the material development and the determination of compositions of the prepared new materials, the service group is well equipped with various instrumental methods:

- Liquid and Solid State Nuclear Magnetic Resonance Spectroscopy (NMR).
- Gas Chromatography Mass Spectrometry (GC / MS) in combination with headspace, thermal desorption and pyrolysis.
- High Performance Liquid Chromatography (HPLC) equipped with different detectors (refractive index detector, diode array detector and mass spectrometry detector) and Size Exclusion Chromatography (GPC, GFC).
- Atomic Absorption Spectrometry (AAS) with both flame and graphi-

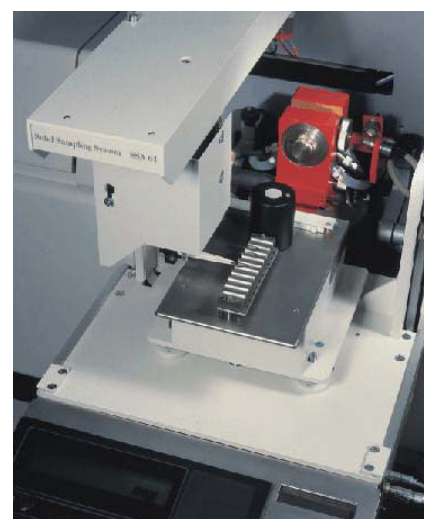


Figure 3: Graphite furnace AAS vario 6 with solid sampling system SSA 61 from Analytik Jena AG.



Figure 4: Bruker NMR-spectrometer Avance II with a resonance frequency for protons of 300 MHz.

te furnace with auto samplers for liquids as well as solids and Optical Emission Spectrometry with Inductively Coupled Plasma (ICP OES);

- CHN analyser.

Among others, the group analysed non hydrolysed precursors like MPTS, FTS, MTEOS, PhTES and solvents in coating sols of INM by gas chromatographic methods of GC-MS quantitatively, examined washing water of the Nano-ZrO₂ synthesis of INM and investigated different charges of conventional vegetable oils and crude oil qualitatively (see figure 1).

The manually operated injector of the HPLC was changed by an automatic sampler for 100 vials for the analysis of non-volatile components in liquids. Residues of photo initiators applied in printers ink were investigated quantitatively on printed paper and printed polypropylene films by this method for example (see figure 2).

A new nebulizer chamber “Scott”, which can be cooled, was installed for ICP OES and has been used for organic solutions. The new pneumatic nebulizer “Ring-spalt”, equipped with a 0.7 mm diameter platinum needle with zirconium nozzle, has been applied to suspensions.

In November 2007 a cooperation with Frau Professor Bauer from the Department of Biosciences-Botany / Plant Molecular Biology of the Saarland University and INM has been started. The team of Professor Bauer determined different transport mechanism of metals (e.g. Fe, Zn, Cu, and Mn) in plants, like toma-

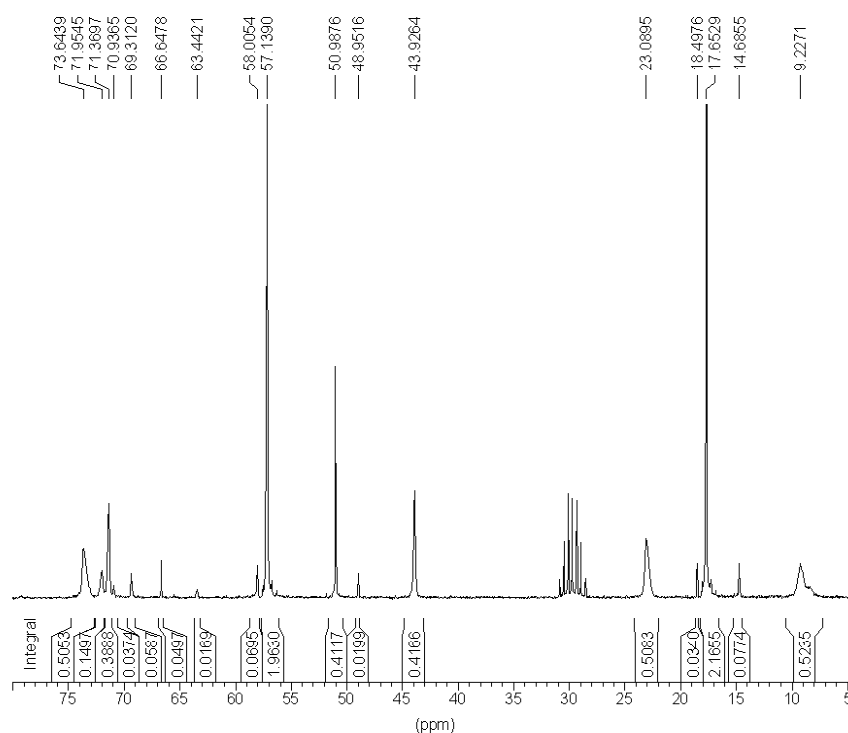


Figure 5: ¹³C-NMR spectrum of GPTES after starting of the organic polymerisation reaction.

to and arabidopsis and INM analysed the metal-content quantitatively by graphite furnace atomic absorption spectrometry with direct solid sampling (see figure 3). This direct solid technique offers significant advantages:

- Analysis of the original sample without the addition of reagents.
- Time consuming digestion technique is avoided.
- Small sample quantities (50-500 µg) are required.
- Reduced analytical error (e.g. contamination, blank value and dilution errors).



- High sensitivity and very good detection limits, therefore suitable for trace analysis.
- Very well suited for homogeneity studies.

At the end of the year 2006, a new NMR-spectrometer, shown in figure 4, was bought and installed. It is a high-resolution digital spectrometer with a magnetic field strength of 7.05 T, which corresponds to a resonance frequency for protons of 300 MHz. The NMR is equipped with an actively self-shielded UltraShield™ magnet system to improve field stability and achieve maximum spectrometer performance. It also leads to smaller risks caused by electromagnetic stray fields and to less stringent site requirements so that it is possible to use the old spectrometer in the same room.

The spectrometer is equipped with two probes:

- A BBFO-broadband-probe for 5 mm sample diameters to observe ^{19}F or any nucleus in the frequency range from 31P to ^{15}N . The outer coil is tuned for proton decoupling or observation. The probe can automatically be tuned and matched.
- A dual probe for 10 mm sample diameters for the observation of ^{29}Si and proton decoupling. This probe is a special manufacture without any glass elements, so no overlapping with the signals of the sample will occur (when using sample tubes made of teflon instead of glass).

The new NMR spectrometer and the software TopSpin® are capable of proces-

sing and analysing all common one and two dimensional spectra.

The NMR spectroscopy is used for routine inspections of incoming silanes and other chemicals, and for the determination of the degree of hydrolysis and the degree of condensation of silanes in reactions. Beyond this, the NMR spectroscopy is used for analytical investigations in current projects. For example, the investigation of organic polymerisation and the identification of resulting products are matters of particular interest. Figure 5 shows a ^{13}C -NMR spectrum of GPTES after the organic polymerisation has started. The signals in the area from 60 ppm to 75 ppm represent the various products.

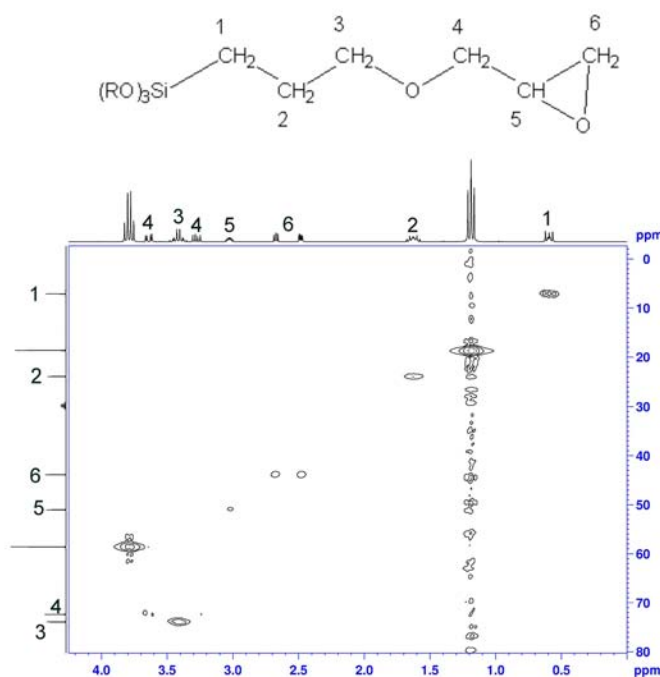


Figure 6: 2D-HMQC of GPTES; ^{13}C -spectrum on the left hand side, ^1H -spectrum on the upper side. The numbers refer to the corresponding carbon atoms and protons.



Servicebereich Analytik: Mikroskopie und Röntgenanalyse

Dr. Ulf Werner



Especially, the two dimensional NMR spectroscopy opens up new ways in the research of organic polymerisation. Figure 6 presents a simple two dimensional spectra of monomeric GPTES. The ^{13}C -spectrum is shown on the right hand side and the ^1H -spectrum is shown on the upper side. The cross peaks demonstrate the relation between the carbon atoms and the bonded protons. This could be a possible method to identify and quantify the different compounds resulting from the ring opening reaction.

Service Group Analytics: Microscopy and X-ray Analysis / Servicebereich Analytik: Mikroskopie und Röntgenanalyse

Die Eigenschaften von Materialien werden nicht nur von deren Aufbau im Makroskopischen, sondern auch von deren chemischer, kristalliner und morphologischer Struktur in nanoskaligen Dimensionen bestimmt. Eine gezielte Synthese neuer Materialien erfordert daher Methoden, die sowohl globale Aussagen über Materialparameter als auch über individuelle Strukturen über einen ausgedehnten Skalenbereich liefern, der bis hinab auf die atomare Ebene reicht. Dies kann nur ein Verbund von Methoden leisten, die ihre Informationen auf der Basis unterschiedlicher Wechselwirkungsprozesse und methodischer Techniken gewinnen. Im INM werden konfokale Laser-Scanning-, Rastersonden- und Elektro-

nen-Mikroskope sowie Röntgen-Diffraktometer eingesetzt, die mittels Photonen, Elektronen oder diverser interagierender Felder im Fern- oder Nahfeld durch simultane oder sequentielle Signalerfassung umfassende Informationen über die Struktur der Materialien gewinnen.

Zur mikroskopischen Oberflächen-Analytik stehen die hochauflösende Raster-Elektronen- (SEM–Scanning Electron Microscopy), die Raster-Sonden- (SPM–Scanning Probe Microscopy) und die konfokale Laser-Raster-Mikroskopie (CLSM–Confocal Laser Scanning Microscopy) zur Verfügung. Diese Methoden ergänzen sich in vielen Fällen hinsichtlich ihrer Aussagen zu Topographie, Morphologie und Artefakten von Oberflächen. Eine dreidimensionale metrologische Beschreibung von Oberflächenstrukturen ist sowohl mit dem SPM als auch dem CLSM möglich, wobei das SPM seine Stärken auf der Nanometer-Skala, das CLSM auf der Mikrometer-Skala zeigt. Die Oberflächen stark gasender und isolierender Proben können mit einem Feldemissions-Raster-Elektronenmikroskop untersucht werden, das über zwei Vakuum-Moden verfügt, die eine Arbeit bei höheren Drücken erlauben (Low Vacuum Mode: bis 1.5 torr, ESEM-Mode: bis 20 torr). Zum Einsatz dieses Mikroskops zur Abbildung biologischer Oberflächen siehe unten.

Die mikroskopische Untersuchung der inneren Materialstruktur bleibt vor allem dem hochauflösenden Transmission-Elektronenmikroskop (HR-TEM–High-Resolution Transmission Electron

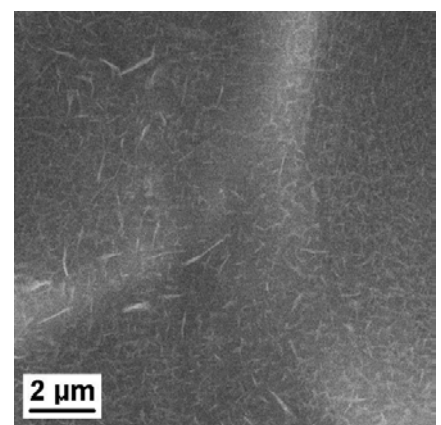
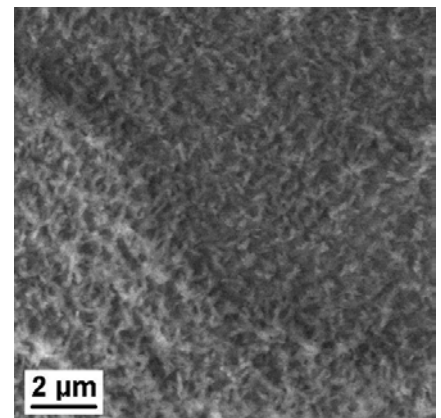
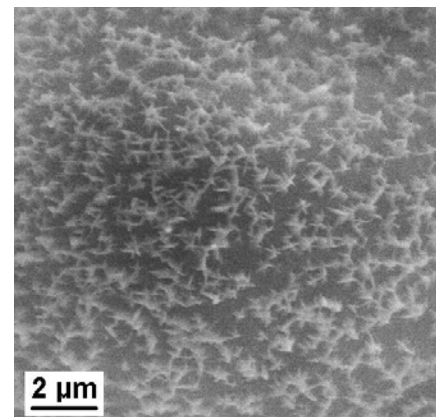


Figure 1: Scanning electron microscopy images of poinsettia (a), nasturtium (b) and red clover (c) showing wax crystals on the surface.



Microscope) vorbehalten: Die exzellente räumliche und zeitliche Kohärenz des Elektronenstrahls dieses Mikroskops erlaubt Raumschwingungen weit unterhalb der Punktauflösung des Mikroskops (0.24 nm) zu erfassen. Die Registrierung derart kleiner Raumschwingungen ermöglicht die Abbildung von Netzebenen mit relativ hoher Indizierung und liefert kontrastreiche Bilder atomarer Strukturen. Dieses Potenzial wird genutzt, um die atomare Kristallstruktur eines Anatas-Nanodrahtes zu charakterisieren (siehe zweiter Abschnitt weiter unten).

Low Vacuum Scanning Electron Microscopy of Plant Surfaces

Some of the most interesting objects for high resolution microscopy are plant surfaces. They are quite rough and chemical sensitive because of a soft surface layer of wax crystals with low melting point, so it is often difficult to measure the topography with AFM or High Vacuum Electron Microscopy. By using Low Vacuum Mode in Scanning Electron Microscopy it's possible to measure the cuticula surface artefact-free down to sub-micrometer resolution, as can be seen in Fig. 1 for poinsettia (*Euphorbia pulcherrima*), nasturtium (*Tropaeolum majus*) and red clover (*Trifolium pratense*). Although surface morphologies are quite different, these plants show similar water repellent behavior. The reason of this lies in the structure period of the wax crystals from some hundred nanometers, which enhances the hydrophobicity of the wax. For a technical

application surfaces with hydrophobic coatings can be made superhydrophobic by structuring the surface in the sub-micron range.

Characterization of Anatase Nanorods by High-Resolution Transmission Electron Microscopy

The general difficulty of transmission electron microscopic imaging of atomic crystal structures of nanoparticles arises from the circumstance that the particles are deposited on an amorphous carrier film and mostly embedded in solvent remnants. Therefore, the structure contrast of the nanoparticles is highly affected by the phase contrast of surrounding amorphous matter. In order to get an optimum structure information from such images a combination of Fourier filtering and morphologic operations can be used: In case of the present investigation of an individual anatase nanorod (synthesis: Dr. D. Bentz) it must be ensured in a first step that the relevant reflexes of the anatase structure are visible in the power spectrum. For this purpose, Fourier filtering using a strong anatase reflex is performed and by binarization and smoothing of the resulting lattice plane image a mask of the nanorod is created. By windowed Fourier transformation of the masked nanorod a power spectrum arises in which numerous additional reflexes emerge from the diffuse background. These reflexes allow a detailed evaluation, i.e. a comprehensive indexing of diffractogram reflexes on the one side, and a specific selection of reflexes for in-verse transformation on the other. Due to

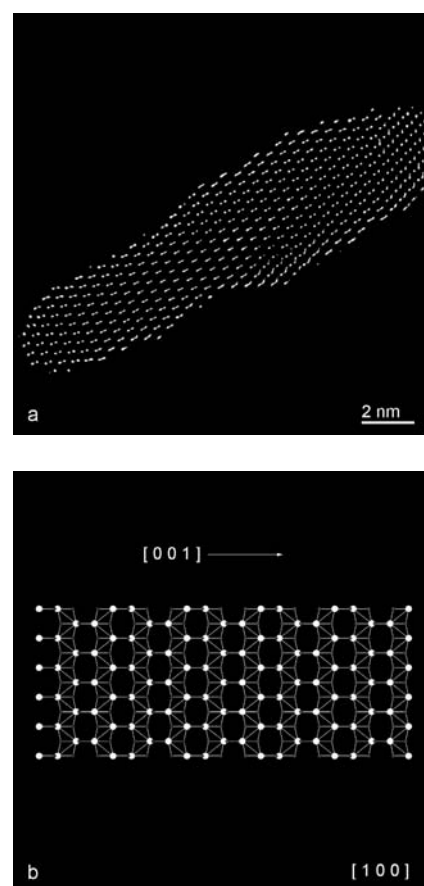


Fig. 2: a) Imaging of the atomistic crystal structure of an individual anatase nanorod (synthesis: Dr. D. Bentz) by means of high-resolution transmission electron microscopy. Image processing: Fourier filtering, binarization and smoothing to generate a particle mask, reflex-selective Fourier filtering of the masked particle, diverse morphologic cleaning operations. b) Structure model of anatase in [100] orientation with the drawn direction of the longitudinal nanorod axis.



the masking, these reflexes contain hardly frequencies of 'amorphous' image structures. This fact reduces image processing artefacts considerably. In case of Fig. 2a all reflexes of type $\{011\}$ were utilized for the inverse Fourier transformation. The binarized and by diverse morphologic operations cleaned image 2a indicates obviously atom column pairs. Owing to superposition of the nanorod structure by a further nanorod (because of filtering not visible) the perceptibility of the pairs is lost in the middle of the particle. This shows the disturbing influence of structures which are arranged 'between' the electron beam and the sample. Comparing the depicted crystal structure with the structure model posed in Fig. 2b reveals that merely the relatively heavy Ti atoms are registered by the microscope. Furthermore, the comparison discloses the orientation of the anatase particle and its longitudinal axis to be $[100]$ and $[001]$, respectively. Since this result was confirmed by several other images, it can be deduced that the particles preferably contact the carrier film surface with (200) lattice planes.

Service Group Engineering / Workshop / Servicebereich Engineering / Werkstatt

Das Aufgabengebiet der Arbeitsgruppe erstreckt sich von der Durchführung von Dienstleistungen wie Reparatur und Wartungen bis zur Entwicklung und dem Bau von wissenschaftlichen Anlagen und Komponenten für die Forschungsabteilungen im Rahmen von Projekten und im Bereich der Grundlagenforschung. Die praktische Umsetzung erfolgt durch eigene Konstruktion und Fertigung. Dies beinhaltet sowohl die Erstellung von individuellen Steuerungen und Softwareentwicklungen als auch die Konstruktion mittels 3D-CAD einschließlich der mechanischen Herstellung mit Hilfe von CAM-Systemen sowie dem Zusammenbau der Anlagen. Der Berichtszeitraum war geprägt durch zwei Hauptaufgaben:

Bildung des Arbeitsbereiches Engineering/Workshop

Die beiden Bereiche „Elektrotechnik/Elektronik“ und „Mechanische Entwicklung“ mit den angeschlossenen Werkstätten wurden zur Arbeitsgruppe „Engineering/Workshop“ vereint. Neben Synergieeffekten wurde damit auch der Vorteil für die wissenschaftlichen Abteilungen des Hauses geschaffen, Anlagen aus einer Hand zu beziehen.

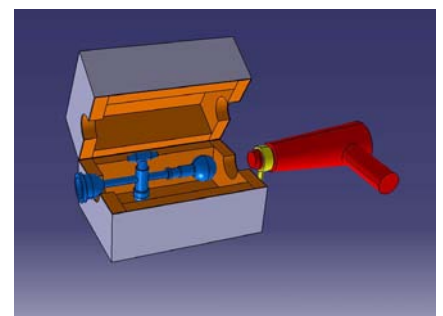


Figure 1: Precursor Heating.



Modernisierung des Bereiches

Die Konstruktion und Fertigung wurde mit einem 3D-CAD/CAM-System ausgerüstet, das die Erstellung von Volumenmodellen mit Simulation ermöglicht und die damit erstellten Daten in fertigungsrelevante Daten generiert. Damit sind mit der vorhandenen 5-Achs-CNC-Fräsmaschine jetzt auch Simultanarbeiten mehrerer Achsen gleichzeitig möglich. Die Wahl fiel aufgrund der weiten Verbreitung auf die marktführende Software CATIA V 5 von Dassault/CENIT. Im elektrotechnischen Bereich wurde die Ausrüstung um eine moderne hochauflösende Wärmebildkamera erweitert.

The assignment of the working group contains the realization of services which range from repairing and maintenance to the development and the construction of scientific plants and components for the research departments within projects and in the field of fundamental research. Here the scope varies from small laboratory equipment to pilot installations. For this the service group goes along with the inventions of the scientific departments and implements them. The practical implementations take place by own developments and fabrication which contains the construction phase of individual control systems and software development as well as the construction using 3D CAD including mechanical fabrication by **means of CAM systems and assembly of machines**. The report period was marked by two main tasks:

Establishment of the working group Engineering/Workshop

The two groups “Electrotechnology/Electronics” and “Mechanical Development” with their respective workshops were merged to the working group “Engineering/Workshop”. This implied besides the structural and personal also a spatial fusion in the workshop building of INM. Combined with synergetic effects we can offer now constructions from one single source to the INM scientists.

Modernization of the group

The construction group was equipped with a 3D CAD system which allows the design of solids with simulation. We chose the market-leading software CATIA V5 by Dassault/CENIT.

Dassault/CENIT offers a CAM module for CNC milling machines which can be used for the creation of construction-required data from the data as-received. Because of this the different axes of the 5-axial CNC milling machine can work simultaneously. To expand the possibilities of handling (cutting almost all material from very soft to very hard) a rotor-based 6-axial water-jet cutting plant with abrasive cutting tool was ordered during the report period. The plant will be delivered at the beginning of 2008.

The equipment of the electrotechnical group was extended by a modern and high resolution infrared camera. This enabled us to display two-dimensional thermograms as well as to take pictures of thermal gradient in the service depart-



Figure 2: Simulation Dip-Coating.



Servicebereich Modelling

Prof. Dr. Dr. Daniel J. Strauss



ment (e. g. ascertainment of damage) and by scientific experiments.

An example for a plant engineering and construction, in which the whole group of the department took part, is the construction of a dip coating plant as described as followed:

A clean room compatible dip plant should be constructed which can handle substrates up to 1000 mm x 1000 mm with a dip angle that can adjust from 0° to 20° and with a drawing rate variable from 0 to 15 mm/s. As guiding mechanism we chose an Isel linear unit. The power is provided by a multiphase motor. Its number of steps was increased by a belt transmission to reduce the vibration. Furthermore we used vibration dampers, a rigid frame, high bulk, and sand filling in the hollow space to reduce the vibration. The substrate clamping is actuated by a pneumatic foot switch. To adjust the dip angle the dip cuvette was constructed rotatable and movable.

The 3D construction enables the display and simulation of the dipping process against the tilting angle. The construction results from CAM programming.

A Siemens S7 CPU 313C was used as control system. The self-made program analyses via sensors all possible positions and locations of the axes and thus controls the actors.

The operation was implemented by a text-oriented display.

Service Group Modelling / Servicebereich Modelling

In 2007 bestand eine Hauptaktivität der Modelling-Gruppe in der Unterstützung des EU-Projektes MULTIPROTECT hinsichtlich der Charakterisierung und Optimierung der Korrosionsschutzschichten. In dem EU-Projekt „MULTIPROTECT - Advanced environmentally friendly multifunctional corrosion protection by nanotechnology“ soll computer-gestützte Modellierung die Optimierung eines selbstheilenden aktiven Korrosionsschutzmaterials unterstützen und ein besseres Verständnis für den Selbstheilungseffekt an sich liefern. Der Schwerpunkt in diesem Jahr lag auf der Integration von multimodalen experimentellen Ergebnissen in ein zuvor von der Gruppe „Modelling“ entwickeltes heterogenes Multiskalen-Modell zur Selbstheilung im Korrosionsschutz. Im Fokus stand hier insbesondere die Anpassung der Modellkinetik an die experimentell erhobenen Daten. Das erstellte, numerisch stabile Modell lieferte bereits eine sehr gute Korrelation zu experimentellen Daten für weit aufgespannte Parameterräume.

Neben den Arbeiten für MULTIPROTECT wurden einige neue numerische Schemen neu entwickelt wie z. B. der 3D-Blutfluss in elastischen Gefäßen unter Nutzung von angiographischen DICOM (Digital Imaging and Communications in Medicine) Standardbildern für In-Vivo-Simulationen von Blut-Material-Wechselwirkungen von perfusionsinduzierten Scher-Spannungs-Profilen. Zu-

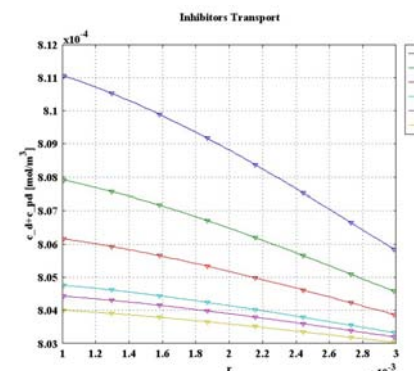
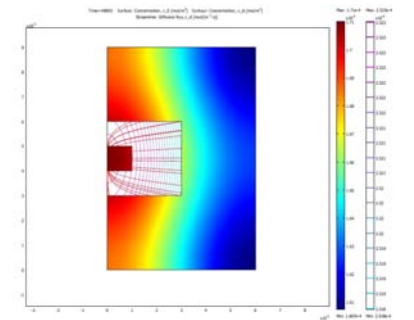


Figure 1: (top) simulation of the self-healing process, (bottom) inhibitors release kinetics on space and time.



sammen mit anderen Gruppen am INM starteten die Partner im Forschungsschwerpunkt “Computational Biomaterials Sciences” der Saarländischen Universitätsklinik ein Projekt zur Oberflächenoptimierung von Stents, bei dem auch zwei Industriepartner einbezogen wurden. Dieses Projekt wird durch das vorher beschriebene Projekt zur numerischen Simulation des In-Vivo-Blutflusses unterstützt. Darüberhinaus wurden fortgeschrittene Phasenraummethoden zur Bildverarbeitung für die computergestützte Charakterisierung und Quantifizierung von Korrosionseffekten auf Bildern von Proben aus dem Salzsprühkammer test entwickelt.

The main objective of the modelling group during 2007 has been primarily the support of the EU-project MULTIPROTECT regarding the computational characterization and optimization of corrosion protection coatings. Apart from MULTIPROTECT, several new numerical schemes have been developed such as the 3D blood flow in elastic vessels using angiographic DICOM (Digital Imaging and Communications in Medicine) standard images for in vivo simulations of blood-material interactions in perfusion induced shear stress profiles. Together with other groups at the INM, the partners in the research focal point “Computational Biomaterials Sciences” from the Saarland University Hospital, in particular the Clinic for Diagnostic and Interventional Neuroradiology and the Com-

putational Diagnostics and Biocybernetics Unit a project regarding the Stent surface optimization has been launched with two industrial partners. This project will directly be supported by the numerical simulation of the in vivo blood flow described before. Moreover, advanced phase space methods of image processing have been developed for the computational characterization and objective quantification of corrosion in images obtained from specimens in salt spray tests.

The mathematical modelling group at INM has been actively participating within the framework of the EU-project: MULTIPROTECT - Advanced environmentally friendly multifunctional corrosion protection by nanotechnology. In 2007, our main activities have focused on the incorporation of experimental findings and novel measurements to select the most relevant parameters and kinetic mechanisms that characterize the functionality of MULTIPROTECT coatings.

The basic heterogeneous multiscale model of the self-healing process by corrosion inhibitors remains incorporating the basic kinetic and thermodynamic mechanisms of corrosion, including the mass transport of inhibitors with gradients of potential and concentration as main driving forces, together with coating properties such as porosity, viscosity as well as physical dimensions of the system. Figure 1 shows a simulation on a 2D domain (6mm x 9mm) of the control release of inhibitors from a cathode (3mm x 3mm, corrosion reservoir) to an anode (1mm x

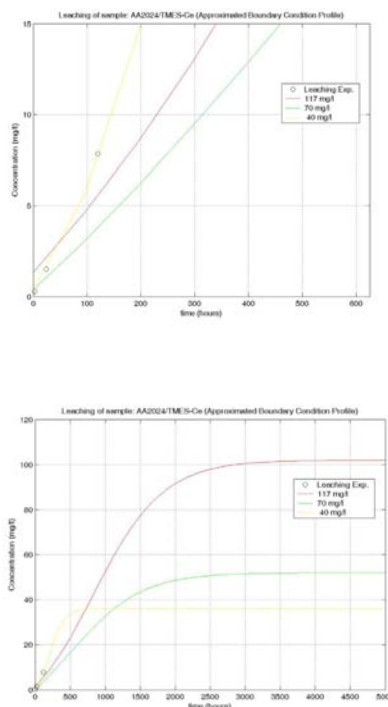


Figure 2: Boundary profiles obtained from coating leaching experiments in NaCl and water solutions.



1mm, which is assumed to be the place where corrosion takes place).

Coating leaching experiments have also been important in setting the boundary conditions for the mathematical model. Figure 2 shows an instance of the boundary profile obtained by means of MULTIPROTECT coating leaching experiments in NaCl and aqueous media.

The incorporation of techniques such as GDOES (glow discharge optical emission spectroscopy) and SVET (scanning vibrating electrode technique) in order to set up artificial corrosion conditions, namely, pH values, anode and cathode currents, and concentrations has offered a great possibility to study the effects of such parameters as in the case of confined microdefects. In Figure 3 we show simulations based on experimental data obtained from SVET for the case of the concentration of inhibitors in the vicinity of a microdefect.

In the next step, the mathematical modelling activities will be focused on the numerical fitting of experimental data and the possible predictions arising from the mathematical model. In this direction, we are implementing experimental strategies in cooperation with UNIMAN (UK), ICV (Spain), and University of Aveiro (Portugal) to establish a synergetic approach that can provide a compromise and realistic industrial solution.

Service Group Library / Servicebereich Bibliothek

Als einer der Servicebereiche des INM ist die Bibliothek verantwortlich für die wissenschaftliche Informations- und Literaturversorgung der Mitarbeiterinnen und Mitarbeiter des Hauses. Ein Schwerpunkt ist der Ausbau elektronisch zugänglicher Informationsressourcen. Ausbau und Aktualisierung der Institutsinternetpräsenz ist ebenfalls Aufgabe der Bibliothek. Die Bibliothek ist außerdem Dokumentationsstelle für Publikationen, Präsentationen und Vorträge des Instituts.

In 2007, after extensive renovation with new chattels and high-capacity hardware, the INM library presented itself in a new and modern design.

The library was manned with a second full-time position. And in the second half of the year a trainee from Korea Maritime University, Busan, South Korea, was supervised.

Customers of the INM library are the scientists of INM as well as members and students of the Saarland University. Furthermore the library provides to an increasing degree information services **within the legal licensing system** to external users, especially to spin-offs from INM.

Electronic resources play a significant role in the information supply for INM scientists. Therefore the range of electronic re-

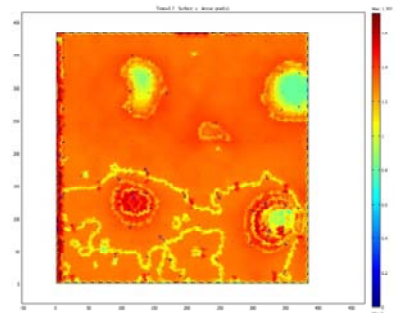
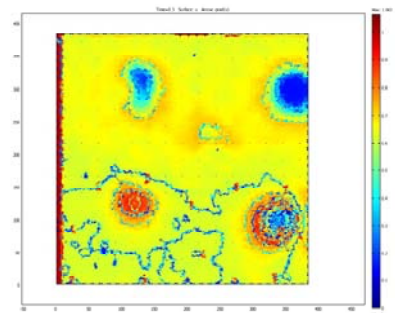
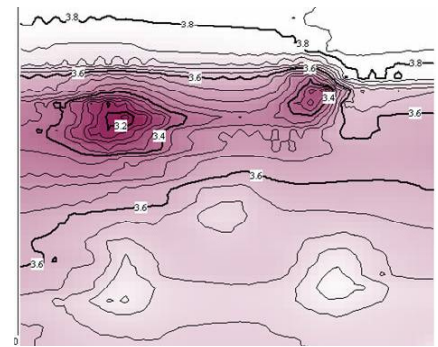


Figure 3: Simulation of the self-healing process by making use of SVET experimental data.



sources and the associated infrastructure was improved. Besides the existing licence contracts like access to ISI-Web of Science (WGL) and to the range of the American Chemical Society the library concluded new bilateral agreements of electronic access for example to SCOPUS and Scencedirect (Elsevier) or the participation in the national licences of the DFG (German Research Foundation).

INM library focuses on supply with literature that isn't available in the library itself, concerning the performance ratio and delivery rate. The used document delivery services are Tiborder and Subito. In the reporting period 97% of the 1276 ordered documents could be fulfilled, 79 % of them within 3 working-days.

Furthermore in 2007 the responsibilities of the library were extended by task in the field of INM public relations, including cooperation in the compilation of 'nanonews' the quarterly institution-owned magazine, the development of a web platform for INM alumni and in particular the support and the further development of INM homepage. Especially noteworthy is the translation of the INM homepage to Japanese and Korean which was realised at the end of 2007.

Scientific publications of an institute are considered as a quantitative performance indicator in scientific evaluations. A major task of the library is the complete documentation of the scientific output of the co-workers. Thus, all institute publications are collected continuously both in an internal INM publication database which allows special analysis according to evalua-

tion relevant criteria and in an internet database with free access to the public.

The INM library keeps contact to other libraries at Saarland University. For instance there is cooperation with the Saarland University and State Library (SULB) in the acquisition of journals and magazines. The book and journal inventory balance of INM library is stored in the library catalogue of SULB and in the sup-raregional union catalogue of Suedwestdeutscher Bibliotheksverbund (SWB) and Zeitschriftendatenbank (ZDB).

The library of Schloss-Dagstuhl, Wadern, and the INM library initiated a working group for regular communication together with libraries of the extra-university research institutions at Saarland University campus. The first working group meeting about copyright has been already held.

The INM library is a member of the "Arbeitskreis Bibliotheken und Informationsrichtungen der Leibniz-Gemeinschaft" (working group of libraries and information institutions of the Leibniz Association). Its spokesman organizes annual meetings which we appreciate very much. And we express our special thanks to Mrs. C. Hasemann, the speaker of the work group and vice-chairman of the German National Library of Science and Technology (TIB). She is the negotiation party in the consortium agreements of the use of electronic resources like databases and journals. Therefore small libraries in the Leibniz Association can provide a competitive and high value offer of information to their co-workers.



Materialprüfung / Karl-Peter Schmitt

EDV / Wolfgang Türk

INM als starker Netzwerk-Partner



Other Service Groups / Weitere Servicegruppen

Materials Testing / Materialprüfung

The principal task of the service group Materials testing comprises the characterization of the materials fabricated at INM with regard to their mechanical properties. The equipment for measurement therefore orientates on the wide variety of the materials investigated at INM and their respective shapes. Allows not only to characterize monolithic materials as e.g. ceramics, glasses, polymers or metals but also the measurement of the mechanical properties of composites as well as coating systems. Besides the performance of standard testing procedures it is an important task to support the research at INM in matter concerning materials mechanics and testing.

Computer Services / EDV

The IT-service of the INM cares for the electronic data processing systems of the house and guarantees the smooth function of the servers, PC and the network. Apart from a continuous improvement of the plants, just as strengthened it is made certain that the existing mechanisms are always updated and adapted to new re-

quirements. This applies likewise to complete functional units (e.g. data servers of individual work areas), as well as to peripheral devices (e.g. plotters, department printers and central available high speed devices).

INM als starker Netzwerk-Partner

Wissenschaftlicher Erfolg wird von der Intensität und der Qualität des Austauschs mit anderen Wissenschaftlern bestimmt. Der Übergang von der Invention zur Innovation gelingt, wenn auch der Dialog mit Unternehmen der Wirtschaft gepflegt wird. Spielt beides zusammen, wird Technologietransfer zum ständigen Begleiter der Institutsarbeit. Netzwerke können dazu einen wichtigen Beitrag leisten.

Für das Jahr 2007 sind vier Netzwerke herauszuheben, deren Arbeit das Institut maßgeblich mit bestimmt hat.

Saarland Empowering Nano

Die Initiative „Saarland Empowering Nano“, die 2006 unter maßgeblicher Mitwirkung des INM ins Leben gerufen wurde, ist eine Plattform für saarländische Nanotechnologie, ihre Anwendungen und Produkte. Sie will über gemeinsames Marketing, Messeauftritte und Veranstaltungen dazu beitragen die führende Rolle des Saarlandes in der Nanotechnologie zu kommunizieren und auszubauen.



Vier mal im Jahr erscheint seit November 2007 das Journal „Saarland empowering Nano“. (Herausgeber u.a. Jochen Flackus). Das Journal berichtet neben Schwerpunktthemen und wissenschaftlichen Artikeln über neue Produkte, sowie über Firmen und Märkte.

Zu den internationalen Aktivitäten gehört die Organisation gemeinsamer Messeauftritte etwa auf der Nanotech-Tokyo, der NanoKorea in Seoul oder der NanoSolutions, Frankfurt am Main, im November des Jahres. Unter der Dachmarke von Saarland Empowering Nano finden sich alle Kompetenzen der saarländischen Nanotechnologie. Von der Forschung am Institut bis zum Massenartikel für den Konsumenten – hier kann Technologietransfer auf allen Ebenen stattfinden.

cc-NanoChem e. V.

Das von 1999 bis 2006 vom BMBF als Kompetenzzentrum geförderte cc-NanoChem wurde in einen Verein überführt und hat seine Arbeit 2007 am INM weitergeführt. Den Vorsitz im Vorstand des Vereins hat Prof. Veith. Er wird im Vorstand von Prof. Fuhr vom Fraunhofer-Institut für Biomedizinische Technik in St. Ingbert und Prof. Matthias Hannig von der Klinik für Zahnerhaltung, Parodontologie und Präventive Zahnheilkunde am Universitätsklinikum des Saarlandes unterstützt. Die Zahl der Mitglieder konnte im Laufe des ersten Jahres als gemeinnütziger Verein von 13 auf 23 erhöht werden. Die Verankerung des Vereins ins Leibniz-Institut für Neue Materialien ist für viele der Mitglieder von be-

sonderem Interesse. Gedankenaustausch und Kooperation werden gefördert.

Der Verein hat 2007 seine Seminarreihe „Innovationen durch Nanotechnologie“ mit zwei Veranstaltungen ebenso fortgesetzt wie die Summer School Chemische Nanotechnologie, die in diesem Jahr bereits zum fünften Mal stattfand. Die Summer School ist zu einer festen Größe am Institut geworden, die auch über die regionalen Grenzen bekannt ist. Viele der Referenten nutzen die Reise nach Saarbrücken auch, um mit Wissenschaftlern des INM in einen Dialog zu treten.

Einer der Schwerpunkte der Vereinsarbeit lag in der Weiterentwicklung des Schüler-Experimentierkastens zur Chemischen Nanotechnologie. Der Kasten mit fünf Experimenten, die aktuelle Forschungsthemen am INM abbilden, wurde 2007 zur Serienreife gebracht. Über die Fa. Aug. Hedinger GmbH & Co. KG kann der Kasten seit Herbst von Schulen erworben werden.

NanoBioNet e. V.

Intensive Zusammenarbeit des INM gibt es auch mit dem NanoBioNet e.V, einem weiteren vom BMBF bis 2006 geförderten Kompetenzzentrum auf dem Saarbrücker Campus. NanoBioNet ist eine gemeinsame Initiative des Saarlandes und des Landes Rheinland-Pfalz, hat sich aber mittlerweile mit über 100 Mitgliedern weit über die Region hinaus entwickelt. Thematisch ist das Netzwerk breiter aufgestellt und umfasst neben der Nanotechnologie auch die Biotechnologie und die Nanobiotechnologie.



One-Dimensional Oxide Nanostructures: Growth, Applications and Devices

S. Barth, S. Mathur, F. Hernandez-Ramirez, A. Romano-Rodrigueza



Dem immer wieder geäußerten Wunsch nach einem stärkeren Engagement des INM im NanoBioNet wurde seit Ende 2005 Rechnung getragen. Schließlich wählte die Mitgliederversammlung im Dezember 2007 Jochen Flackus zum Vorsitzenden des Vereins.

Beide Kompetenzzentren, cc-NanoChem und NanoBioNet, haben sich auf ihren jeweiligen Mitgliederversammlungen 2007 für eine engere Zusammenarbeit der Vereine ausgesprochen, wobei auch die Möglichkeiten einer Fusion evaluiert werden sollen.

Leibniz-nano!

Innerhalb der Leibniz-Gemeinschaft forschen eine Reihe von unterschiedlichen Instituten an nanotechnologischen Themenstellungen auf hohem Niveau. Im vorangegangenen Jahr wurde daher der Wunsch nach einer engeren Zusammenarbeit innerhalb der Gemeinschaft artikuliert und am INM die Koordinierungsstelle Nanotechnologien der Leibniz-Gemeinschaft eingerichtet.

Um eine effiziente Vernetzung zu gewährleisten, wurde von Anfang an auf persönliche Kontakte gesetzt. Die beteiligten Institute haben jeweils einen kompetenten Ansprechpartner für die Nanoaktivitäten im Hause benannt, die Anfang 2007 von Herrn Dr. Schubert vor Ort besucht wurden. Auf der Liste der Institute standen neben dem INM zunächst das IKZ in Berlin, das INP in Greifswald, das IHP in Frankfurt (Oder), das ISAS in Dortmund und Berlin, das FZD in Dresden, das IOM in Leipzig und das FIZ in

Karlsruhe. Die beiden Dresdner Institute IPF und IFW kamen Mitte des Jahres dazu. Auch hier wurde bei einem Besuch der persönliche Kontakt aufgebaut. Damit hat die Koordinierungsstelle einen guten Überblick über die Aktivitäten und Kompetenzen der Partner.

Eines der zentralen gemeinsamen Ziele ist es, die Sichtbarkeit der WGL zu erhöhen. Ein Schritt in diese Richtung war die konzentrierte Beteiligung am Stuttgarter MiNaT-Kongress. Nicht nur in der Zahl der Beiträge konnte sich die WGL gegenüber den anderen Wissenschaftsgesellschaften behaupten. Auch im Marketing der Veranstaltung fand das Logo der Leibniz-Gemeinschaft seinen Platz neben Fraunhofer-Gesellschaft, Max-Planck-Gesellschaft und Helmholtzgemeinschaft.

Auch gemeinsame Messeauftritte sind für die Zukunft geplant. So haben sich INP und INM bereits über den gemeinschaftlichen Auftritt auf der Hannover Messe 2008 verständigt.

[One-Dimensional Oxide Nanostructures: Growth, Applications and Devices](#)

One dimensional (1D) inorganic materials are gaining high attention due to their structural stability and unique structural features^[1]. Among them, oxides are widely studied due to their well established application potential and mechanical as well as chemical stability. We have deve-



veloped a generic approach for size-selective and site-specific growth of oxide nanowires by combination of a catalyst-assisted growth mechanism and a molecular precursor approach, which is a viable alternative to other gas phase and solution procedures and produces well-defined (morphology and composition) materials.

Tin Oxide Nanowires

We reported the size-selective synthesis of tin oxide NWs in earlier studies, which allowed the investigation of diameter-dependent photo-conductance with nanowire diameters in the range of 50-1000 nm^[2]. In addition, the stable response over several on-off cycles demonstrated their potential as photo detectors^[2]. Besides multiwire devices, individual tin oxide wires, contacted by FIB nanolithography, were investigated via a combination of two- and four-probe geometries towards their electrical properties^[3]. An alternative strategy to FET measurements was described to evaluate conductivity σ_2 , charge carrier density η_d and mobility μ in SnO₂ NWs circuits.^[4] However, self heating effects and metrological precautions should be taken into account in order to prevent destruction or degradation of these devices as shown in figure 1.

Resistive-type metal oxide sensor, such as tin oxide, uses surface chemical reactions and the subsequent transduction of its electronic properties (e.g., change in the density of conduction band electrons or valence band holes) to detect various gaseous species^[5]. However selecti-

vity of SnO₂ surfaces towards atmospheric moisture and water vapor (H₂O) greatly influences their application as reliable gas sensing devices^[6]. Since electrical response of SnO₂ nanostructures critically depends on pre-adsorbed species at the oxide surface, understanding the role and influence of water vapour in sensing mechanism is essential for the optimization of phenomenological experiments and more importantly in evaluating the potential of tin oxide-based devices^[7]. The interfering effect of water vapor has been investigated in some cases, for example in the detection of carbon monoxide (CO), however the different interaction mechanisms of H₂O molecules and SnO₂ surface are only vaguely described.^[7] Heiland and Kohl proposed a widely accepted model of the metal oxide surface interaction with water molecules^[8]. A realistic interaction model between water and SnO₂ should, however, consider the effects introduced by chemisorbed oxygen at the surface of the metal oxide. Henrich and Cox proposed a displacement of pre-adsorbed oxygen by water adsorption^[9], which was supported by reliable hints for an influence of water vapor on oxygen chemisorption found by several groups. Similarly, Caldararu et al. assumed the blocking of oxygen absorption sites by water molecules^[10]. All these mechanisms describe the major role surface states play in the interaction of metal oxide and H₂O.

We have performed humidity sensing experiments close to the temperature (~ 300 °C) required for the detection of carbon mono-

xide, which is one of the most prominent applications of SnO₂-based sensors^[11]. More than 10 % variation in the sensor resistance values was observed when water vapor pulses with concentrations as low as 1400 ppm (equivalent to 5 % humidity at 25°C) were introduced in N₂ atmosphere at 295°C. Up to 80 % variation in the resistance values were obtained after injection of water vapor pulses equivalent to 100 % of humidity at 25°C in Standard Atmosphere. The electrical response (change of resistance) can be approximated by the following power law outlined in eq. 1

$$R = R_0 (1 - [H_2O]^\beta) \quad (1)$$

where R₀ is the NW resistance in dry nitrogen or pure synthetic air, [H₂O] the water vapor concentration (in ppm units), and β a constant parameter. A double-logarithmic plot of the sensor response, shown in figure 2, revealed a linear dependence of device responses on water vapor concentration.

The electrical change, which is directly related to the concentration of the water vapor pulses, is almost the same in SA and N₂ experiments. It must be pointed out that the relative resistance difference under N₂ and SA atmospheres is almost constant with similar β values (β(N₂) = 0.21, β(SA) = 0.19), which suggests that the water molecules adsorb on the coordination sites and do not necessarily compete with pre-adsorbed species. However, a chemical reaction of H₂O molecules upon increasing concentration with the adsorbed oxygen species (O²⁻, O⁻ and O²⁻) can not be ruled out.^[12] Fi-

nally, the sensor stability was evaluated as function of operating time, where none of the measured NWs was damaged after several operating cycles. In addition, no significant changes in their sensing capabilities were observed after one month in open air atmosphere, which suggested that a long lifetime and trustworthy responses could be expected for these components.

In order to fabricate portable devices based on individual nanostructures SnO₂ nanowires were contacted to microelectrodes on free-standing micromembranes by FIB nanolithography techniques. The electrical characterization was performed using an electronic circuit designed to prevent the destruction of such devices due to uncontrolled current fluctuations. Modulation of the device temperature was shown to be reproducible and fast due to the integrated heater on the micromembranes. The obtained devices showed stable and reproducible responses for different CO concentrations demonstrating their potential as gas sensors. The article dealing with this topic was selected by the journal Nanotechnology as one of the hot papers and cover page (figure 3)^[13].

Alignment of SnO₂ NWs on surfaces was achieved by controlling the lattice mismatch between NW material and substrate ((aSnO₂ - aTiO₂) / aTiO₂ = 2.6 % and (cSnO₂ - cTiO₂) / cTiO₂ = 7.7 %). The aligned growth of SnO₂ nanowires was achieved by catalyzing the nucleation of one-dimensional nanostructures through gold nanoparticles, and exploit-

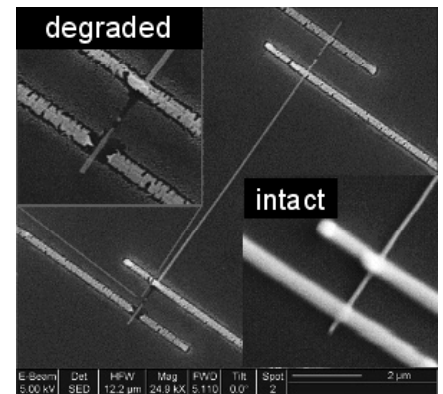


Figure 1: FIB nanolithography fabricated circuit based on an individual SnO₂ NW showing intact and degraded electrical contacts.



ting the structural relationship between TiO_2 and SnO_2 systems. Since the lattice parameters of tin and titanium oxides showed considerable mismatch (up to 7.7 %), the interface needs to relax to accommodate the strain [14]. In-plane SEM view of tin oxide NWs confirmed an oriented growth with a mesh-like network of straight NWs of regular dimensions (figure 4). The diameters of oriented as-grown NWs ranged from 15-25 nm and the length was typically found to be in the range 0.5 - 5 μm . Cross-sectional view in scanning electron microscope (SEM) revealed a characteristic angular orientation of NWs with respect to the substrate, presumably imposed by the four-fold axis of symmetry present along the c -axis in TiO_2 (001) substrate, which offered the growing tin oxide nanowires four equivalent growth directions due to mirror images of the unit cell. In order to elucidate the influence of substrate orientation on observed growth, TiO_2 (100) was also used to grow SnO_2 NWs (figure 4b), which resulted in two main growth directions and appearance of secondary alignment effects, caused by the substitution of equivalent sides with low lattice mismatch (a -axis 2.6 %) through c -axis orientation (7.7 %). Figure 4c reveals randomly oriented NW nucleation on (111)-oriented TiO_2 , where the lattice mismatch is too large to favour NW alignment.

High-resolution TEM images of several tin oxide nanowires revealed that the majority of them exhibited preferred growth along $\langle 031 \rangle$ direction, which contrasts

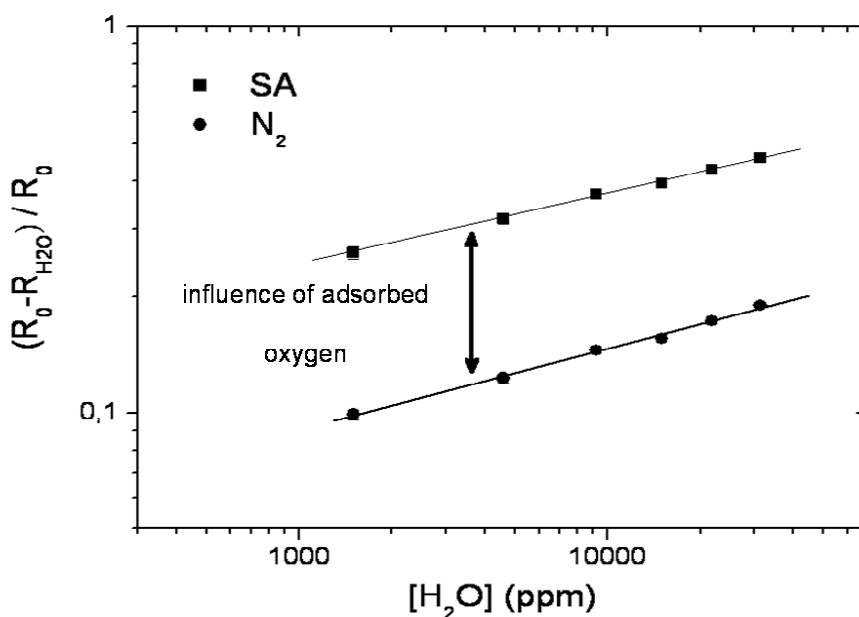


Figure 2: Double logarithmic plot of the response of an individual SnO_2 NW ($r = 50$ nm) to different humidity concentrations in SA and N_2 environments. An offset introduced by ionsorbed oxygen contribution in SA atmosphere is observed.

the expected growth axis ($\langle 001 \rangle$) based on the crystallographic relationship between the substrate and the NW material. This could be caused by an energetically favoured growth axis, with contributions of the bulk energy of tin oxide, the bulk energy of the liquid droplet, the interfacial tension of the liquid solid interface and the surface tensions of the droplet and the nanowire, respectively [15].

Despite proven experimental strategies for site-selective growth of NW ensembles, numerical control over lateral density of NWs remains a crucial challenge. The Au-catalyzed growth of nanowire arrays was downsized to grow individual NW by depositing gold colloids on TiO_2 (001) substrates using spin-on tech-

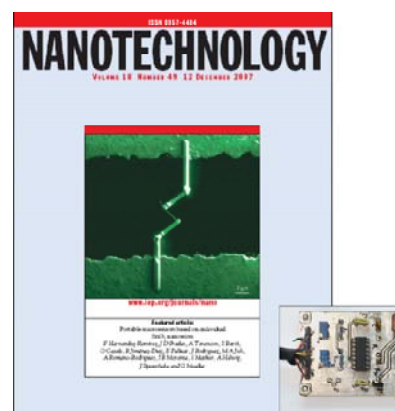


Figure 3: Portable SnO_2 NW device (inset) developed in collaboration with University of Barcelona was featured on the cover page of the journal *Nanotechnology* (2007, Vol. 18, 495501) [13].

niques. Controlled decomposition of the precursor at 700°C on individually dispersed Au nanoparticles provided singly grown NWs with growth axis identical to that found in NWs grown in bundles. In addition to oriented nanowires, the rest of the substrate was covered with a nanocrystalline film (figure 5) illustrating the competing catalyzed (1D) and uncatalyzed (2D) growth. Selectivity of the nanowire growth is limited by the fact that only a fraction (approximately 30-70 %) of catalyst particles act as growth initiators. A similar observation has been recently made in vertically oriented Ge nanowires grown from gold colloids on silicon substrates [16]. The observation that the density of gold colloids is not comparable to the density of nanowires may relate to several factors such as the surface charge (due to citrate ions present on Au particles) and agglomeration, which may influence the activity and melting point of the gold nanoparticles, respectively.

Iron oxide (Fe₃O₄)

Nanoscaled iron oxides are promising for magnetic (e.g., spintronics) and manifold biomedical applications such as drug delivery [17, 18], gene therapy [17, 19], hyperthermic cancer treatment [17, 20], and contrast agents in magnetic resonance imaging (MRI) [17, 21]. When compared to large body of data available on synthesis, characterisation and applications of magnetic nanoparticles [20], reports on anisotropic nanostructures such as nanowires and nanotubes are limited to templated [22], hydrothermal [23], and catalyst-

assisted pulsed laser deposition processes [24] as well as reduction of pre-grown α -Fe₂O₃ [25].

CVD of [Fe(O^tBu)₃]² enabled the formation of 1-D magnetite nanostructures at temperatures between 750-850 °C. The thermal fragmentation of Fe³⁺ species under low pressure partially reduced Fe³⁺ to Fe²⁺ leads to the formation of nanostructured magnetite films at elevated temperatures (figure 6) [26]. The phase purity of the as-obtained nanostructures was investigated by XRD analysis, which exhibited a pure Fe₃O₄ composition devoid of other Fe:O phases. Deposition on MgO (100) had an aligning effect on the growing structures, which is suited for growing highly oriented magnetite coatings due to its low lattice mismatch (0.3 %) with Fe₃O₄ [27] and produced both wiry and saw-like morphologies [28].

Four equivalent growth orientations evident in SEM images (figure 7a), indicated the influence of crystallographic relationship between the oriented substrate and magnetite nanostructures. Figure 7b shows transmission electron microscope (TEM) images of straight (figure 7a) and zigzag magnetite nanostructures (inset). Both, HR-TEM and corresponding FFT images revealed the single crystalline nature of the 1D magnetite nanostructures. Interplanar spacings and corresponding FFT revealed the preferred growth direction to be <110> (figure 7c). Lattice spacings of 0.2980 nm match well with the (220) lattice planes of magnetite (0.2967 nm, PDF 19-0629). The catalyst particle was clearly identified as a globu-

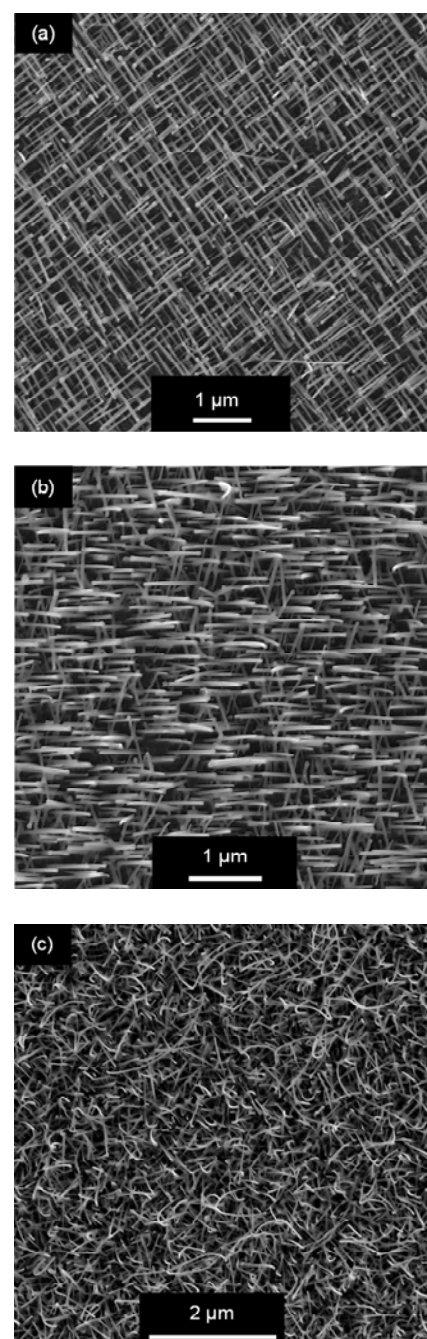


Figure 4: SnO₂ NWs on single crystalline TiO₂ substrates of (a) (001), (b) (100) und (c) (111) orientation.



lar tip, whereby the gold metal was found to align with the cubic oxide structure providing a coincidence grain boundary, where the (111) planes of Au align along the (220) lattice planes of Fe_3O_4 and the resulting strain at the interface is relaxed through one dimensional defects. During the course of the CVD process, growth of new 1 D structures via catalyst-assisted mechanism and further growth on initially formed nanowires, supported through layer-by-layer growth was observed, which resulted in stepwise tapered structures (figure 7b).

Formation of facets due to kinetic barriers is common in the gas phase growth of crystalline materials. If the deposition rates do not vary substantially the diffusion kinetics of adatoms is determined by the diffusion distances and the associated kinetic barriers on flat surfaces and near surface steps. Apparently, the migration along $\langle 110 \rangle$ step edge is thermodynamically favoured and thus fast diffusion path for the atoms, which limits the formation of flat surface and leads to the pronounced formation of faceted nanostructures as observed in figure 7b. As a result both axial and radial dimensions of the nanowires grow during the CVD process, due to competing catalyst-assisted growth occurring at the nanowire tip and diffusion-assisted growth active on the nanowire body.

Individual magnetite NWs were contacted to Pt electrodes using FIB nanolithography procedures, as described in literature [12]. I-V characteristics were investigated in two point configuration. The observed ohmic response of this device

could be attributed to metallic conduction of the Fe_3O_4 NW at 300 K. The resistivity value was in the range of $\sim 9 \cdot 10^{-6} \Omega \cdot \text{m}$, which is in the range of reported data for single crystals.[29] Two out of three measured devices showed resistivities in the range of $9\text{-}10 \cdot 10^{-6} \Omega \cdot \text{m}$, however high contact resistance contributions must be taken into account, which was already described for FIB contacts to SnO_2 NWs.[4]

1D Oxide Heterostructures

In order to grow longitudinal heterostructures, SnO_2 nanowires formed by the CVD of $\text{Sn}(\text{O}^t\text{Bu})_4$ [2] were used as substrates to obtain tin oxide/magnetite nanostructures (figure 8). We have synthesized $\text{SnO}_2/\text{Fe}_3\text{O}_4$ core-shell structures by a two-step growth process, where initially grown tin oxide NWs ($\varnothing 50 \text{ nm}$) were covered by a dense assembly of magnetite nanocrystals forming a polycrystalline layer (figure 8a), which was confirmed by X-ray diffraction analysis. Sub-

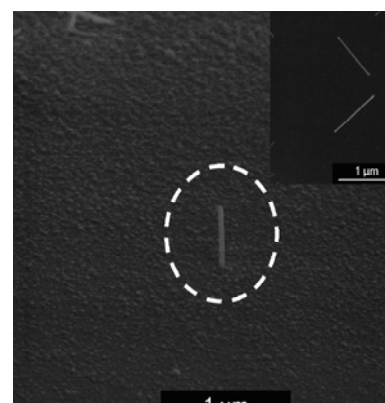


Figure 5: SEM image of an individual SnO_2 NW grown on TiO_2 (001) surrounded by a nanocrystalline film (picture taken under 30° angle to the substrate surface) and plane view of oriented grown wires (inset).

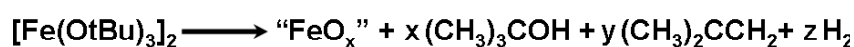
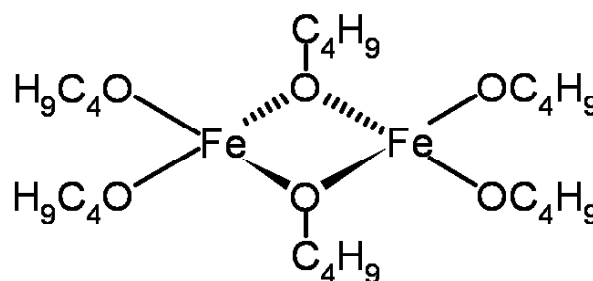


Figure 6: Molecular structure of $[\text{Fe}(\text{O}^t\text{Bu})_3]_2$ and scheme of the thermally activated decomposition in a CVD process.

ject to the precursor flux and adjustment of CVD parameters, the magnetite shell sometimes connected several pre-formed crossed NWs of the SnO_2 substructure to form interconnected magnetite superstructures with several junctions at the micro-scale. Differences in electrical and magnetic properties between single crystalline wires and polycrystalline core-shell structures are currently underway.

Branched one-dimensional heterostructures were fabricated by growing vanadium oxide nanostructures on pre-grown SnO_2 backbones by decomposition of $[\text{VO}(\text{O}^i\text{Pr})_3]$. Hierarchical structures involving second generation growth of nanowires, have been realized using a sequential two-step strategy. Merely few data exists in the literature dealing with epitaxial growth of a high axial ratio oxide nanostructure on a one-dimensional oxide host structure possessing different chemical compositions and crystal structures [30]. This is a generic approach and should in principle provide access to various combinations of materials especially in systems where an intrinsic doping is not possible due to solubility limitations. Hierarchical growth exhibiting strong interaction of individual vanadium oxide sub-structure with the tin oxide backbone was observed in 1D and 2D configurations (parallel or/and normal to tin oxide NW axis). The geometrical features of the V_2O_5 nanostructures were modulated by varying the precursor flux (temperature), whereby elongated structures were formed at higher gas phase saturation. HR-TEM images of SnO_2 - V_2O_5

interface revealed a strong alignment of the two different crystal lattices, resulting in a heteroepitaxial growth of vanadium oxide on tin oxide. The sharp SnO_2 - V_2O_5 interface indicate the epitaxial bonding driven by the low mismatch factors of the two single crystalline materials, confirmed by corresponding FFT images [14]. Given the different work functions of the two oxides and the possibility of epitaxially anchoring V_2O_5 nanostructures on SnO_2 NWs, this concept represents a viable strategy for locally modifying the electronic properties of the host materials [31].

In summary, we have developed a molecule-based catalyst assisted growth mechanism for the synthesis of single crystalline functional 1D oxides. We were able to synthesize and structurally characterize tin and iron oxides of high aspect ratio. Further a universal methodology was investigated to contact individual nanoscaled objects for the production of microcircuits, which are suitable for fast and reliable gas detection and extraction of intrinsic material properties by electrical characterisation. In addition, various shapes of oxide heterostructures based on tin oxide NWs were grown and structurally characterized for the investigation of growth phenomena and interface effects on functional properties.

Acknowledgements

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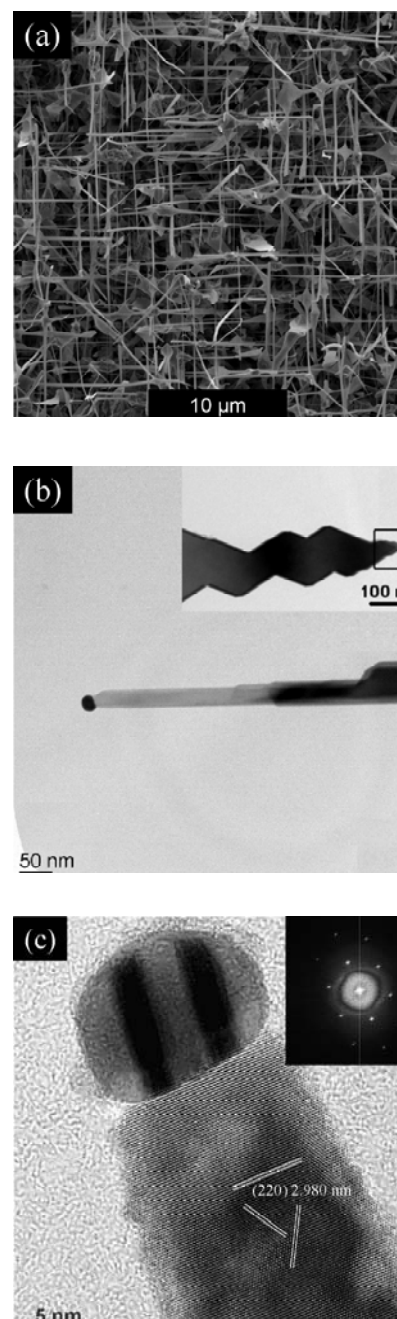


Figure 7: (a) SEM image of magnetite nanowires array. (b) TEM images of synthesized 1D Fe_3O_4 and (c) HR-TEM and FFT images of a magnetite NW close to the catalyst particle.



ation (DFG) is gratefully acknowledged for supporting our research (priority program SFB-277).

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References

[1] Z. L. Wang in „Nanowires and Nanobelts“, Vol. 1 and Vol. 2 (Ed. Z. L. Wang), Springer, Berlin 2006.

[2] S. Mathur, S. Barth, H. Shen, J.-C. Pyun, U. Werner, *Small* 2005, 1, 713.

[3] F. Hernandez-Ramirez, A. Tاراcon, O. Casals, J. Rodriguez, A. Romano-Rodriguez, J. R. Morante, S. Barth, S. Mathur, T. Y. Choi, D. Poulidakos, V. Callegari, P. M. Nellen, *Nanotechnology* 2006, 17, 5577.

[4] F. Hernandez-Ramirez, A. Tاراcon, O. Casals, E. Pellicier, J. Rodriguez, A. Romano-Rodriguez, J. R. Morante, S. Barth, S. Mathur, *Phys. Rev. B* 2007, 76, 085429.

[5] (a) M. E. Franke, T. J. Koplín, U. Simon, *Small* 2006, 2, 36.

[6] M. Batzill and U. Diebold, *Progress in Surface Science* 2005, 79, 47.

[7] N. Bárzan, U. Weimar, *J. Phys.: Condens. Matter* 2003, 15, R 813.

[8] G. Heiland, D. Kohl in T. Seiyama (ed.), *Chemical Sensor Technology*, Vol. 1, 1–35, Kodansha, Tokyo.

[9] V. A. Henrich, P. A. Cox, *The Surface Science of Metal Oxides*. University Press, Cambridge 1994, 312.

[10] M. I. Baraton, L. Merhari, *Nanostr. Mater.* 1998, 10, 699.

[11] F. Hernandez-Ramirez, A. Tاراcon, O. Casals, J. Arbitol, A. Romano-Rodriguez, J. R. Morante, *Sensors Actuators B* 2007, 121, 3.

[12] F. Hernandez-Ramirez, S. Barth, A. Tاراcon, O. Casals, E. Pellicier, J. Rodriguez, A. Romano-Rodriguez, J. R. Morante, S. Mathur, *Nanotechnology* 2007, 18, 424016.

[13] F. Hernandez-Ramirez, J. D. Prades, A. Tاراcon, S. Barth, O. Casals, R. Jimenez-Diaz, E. Pellicier, J. Rodriguez, M. A. Juli, A. Romano-Rodriguez, J. R. Morante, S. Mathur, A. Helwig, J. Spannhake, G. Mueller, *Nanotechnology* 2007, 18, 495501.

[14] S. Mathur, S. Barth, *Small* 2007, 3, 2070.

[15] V. Schmidt, S. Senz, U. Gösele *Nano Lett.* 2005, 5, 931.

[16] J. H. Woodruff, J. B. Ratchford, I. A. Goldthorpe, B. C. McIntire, C. E. D. Chidsey, *Nano Lett.* 2007, 7, 1637.

[17] A. Q. Pankhurst, J. Connolly, S. K. Jones, J. Dobson, *J. Phys. D: Appl. Phys.* 2003, 36, R167.

[18] C. C. Berry, A. S. G. Curtis, *J. Phys. D: Appl. Phys.* 2003, 36, R198.

[19] M. Shinkai, M.- Yanase, M. Suzuki, H. Honda, T. Wakabayashi, J. Yoshida, T. Kobayshi, *J. Magn. Magn. Mater.* 1999, 194, 176.

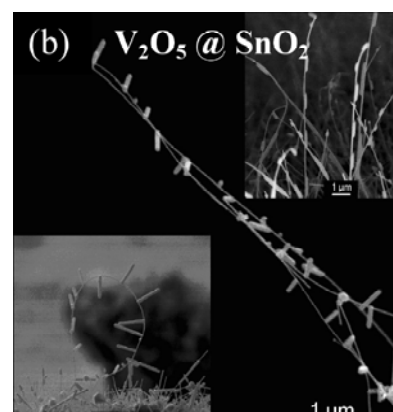
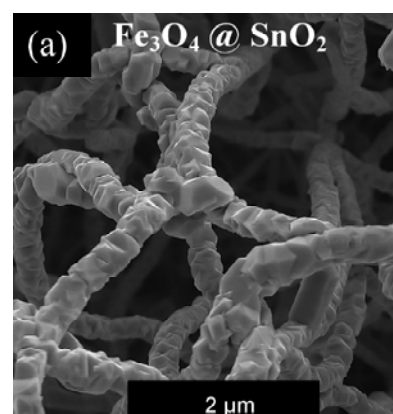


Figure 8: SEM images of (a) $\text{Fe}_3\text{O}_4/\text{SnO}_2$ core-shell and (b) $\text{V}_2\text{O}_5/\text{SnO}_2$ hierarchical nanostructures.



Mechanisms of Bonding Effected by Nanoparticles in Zirconia Coatings Applied by Spraying of Suspensions

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



- [20] T. Sugimoto, in *Monodisperse Particles*, Elsevier, Amsterdam, Netherlands 2001.
- [21] N. Nitin, L. E. W. LaConte, O. Zurkiya, X. Hu, G. Bao, *J. Biol. Inorg. Chem.* 2004, 9, 706.
- [22] Z. Liu, D. Zhang, S. Han, C. Li, B. Lei, W. Lu, J. Fang, C. Zhou, *J. Am. Chem. Soc.* 2005, 127, 6.
- [23] J. Wang, Q. Chen, C. Zeng, B. Hou, *Adv. Mater.* 2004, 16, 137.
- [24] J. R. Morber, Y. Ding, M. S. Haluska, Y. Li, J. P. Liu, Z. L. Wang, R. L. Snyder, *Phys. Rev. B* 2006, 110, 21672.
- [25] J. Bachmann, J. Jing, M. Knez, S. Barth, H. Shen, S. Mathur, U. Gosele, K. Nielsch, *J. Am. Chem. Soc.* 2007, 129, 9554.
- [26] (a) S. Mathur, M. Veith, V. Sivakov, H. Shen, V. Huch, U. Hartmann, H. B. Gao, *Chem. Vap. Depos.* 2002, 8, 277; (b) S. Mathur, V. Sivakov, H. Shen, S. Barth, C. Cavalius, A. Nilsson, P. Kuhn, *Thin Solid Films* 2006, 502, 88; (c) S. Mathur, H. Shen, V. Sivakov, U. Werner, *Chem. Mater.* 2004, 16, 2449.
- [27] D. M. Lind, S. D. Berry, G. Chern, H. Mathias, L. R. Testardi, *Phys. Rev. B* 1992, 45, 1838.
- [28] (a) S. Mathur, S. Barth, U. Werner, F. Hernandez-Ramirez, A. Romano-Rodriguez, *Adv. Mater.* 2008, in print; (b) S. Mathur, S. Barth, *Z. Phys. Chem.* 2008, 222, 307.
- [29] M. Bohra, N. Venkataramani, S. Prasad, N. Kumar, D. S. Misra, S. C. Sahoo, R. Krishnan, *J. Magn. Magn. Mater.* 2007, 310, 2242.
- [30] (a) A. Vorniero, M. Ferroni, E. Comini, G. Fagkia, G. Sberveglieri, *Nano Lett.* 2007, 7, 3553; (b) L. Xu, Y. Su, S. Li, Y. Q. Chen, Q. T. Zhou, S. Yin, Y. Feng, *Phys. Chem. B* 2007, 111, 760.
- [31] J. E. Evans, K. W. Springer, J. Z. Zhang, *J. Chem. Phys.* 1994, 101, 6222.

Mechanisms of Bonding Effected by Nanoparticles in Zirconia Coatings Applied by Spraying of Suspensions

Abstract

Zirconia coatings consisting of a mixture of coarse and fine grained zirconia powders prepared by spraying of suspensions and subsequent thermal treatment at limited temperatures (up to 500°C) are poor in adherence and in intrinsic mechanical strength. We have shown elsewhere that mechanical properties of these coatings can be improved clearly by adding a small amount of nanoscaled zirconia.

Here, the structural and the chemical development of this coating material and of the nanoparticles is examined to gain information about the underlying bonding mechanisms. The applied temperature is relatively low in comparison to the usual onset temperature of accelerated sintering. Nevertheless, the results show that diffusion controlled material transport mechanisms play their role in bonding. The condensation of surface OH groups may participate in bonding, too.



These first results confirm the potential of nanoparticles to act as inorganic binder. Additional research effort to clarify the underlying mechanisms in detail is of interest. For the practical side, it can be concluded that the resulting effect of mechanical consolidation of ceramic structures at relatively low temperatures enables new ceramic applications, for example a new type of ceramic coatings on metallic substrates.

Keywords: ZrO_2 , ceramic coatings, nanoparticles, bonding, colloidal processing.

Introduction

Different surface functionalities can be realised on metals by thick ceramic coatings. PVD/CVD and sol-gel routes lead to a limited coating thickness. Usually coatings with a thickness in the order of 100 μm are applied by using high temperature processes. Corresponding main routes are enamelling and various thermal spraying processes [1]. These coatings are also suitable for long term operation at high temperatures. When coating methods on the basis of particle suspensions with subsequent heat treatment are used, drying and sintering shrinkage are detrimental effects on formation of dense and well adhering coatings on rigid substrates.

One approach to reduce shrinkage is to include fractions of larger particles. Lan and Xiao [2] minimized drying stress and cracking of ZrO_2 coatings during drying by adding sub- μm or μm particles to a suspension of nanoparticles.

Their colleagues, Wang et al. [3] avoided overall shrinkage with the so-called 'bricks and mortar' concept using 10-60 μm ZrO_2 particles as structural backbone. The coatings with a thickness of 200-600 μm were fired at 1200°C with the 'mortar' consisting of 14 nm ZrO_2 particles to obtain mechanical strength.

However, many substrates do not allow such high heat treatment temperatures. Applications that require neither sophisticated coating strengths nor dense coatings would be simply and economically accessible by colloidal coating methods (dipping, spin coating, spraying) if bonding could be realised at low application temperatures. At 500°C we achieved ZrO_2 coatings with a remarkable strength if ~ 8 wt.-% of the ceramic fraction consisted of nanoparticles [4]. This interesting result directly leads to the question about the underlying bonding mechanisms.

Surface diffusion is the first expected sintering mechanism in the low temperature sintering stage. A rough estimation of the onset temperature of surface diffusion is given by the "Hüttig temperature" $T_H = 0.3 \cdot T_{\text{melt}}$ (T in K) which is 620°C in the case of ZrO_2 . Akash [5] determined the temperature dependence of the surface diffusion coefficient in the temperature range of 900-1050°C for yttria stabilized zirconia particles with a mean particle size of 15 nm, determined by XRD/Scherrer equation. Extrapolating the surface diffusion coefficient given by Akash down to a temperature of 500°C results in a massively smaller surface dif-

fusion coefficient (13 orders of magnitude lower). At first sight, it can be concluded from this extrapolation and from the Hüttig temperature, that surface diffusion should not be relevant at 500°C. But, the results of Skandan [6] show that the impact of particle size on sintering in the lower nm-range has to be considered. Compacts made of 6 nm zirconia particles exhibit a linear dilatometric shrinkage of ~ 2 % [6] with increasing temperatures up to 500°C due to coarsening and particle rearrangement. Particle coarsening and shrinkage give the hint that diffusion is relevant at 500°C for particles < 10 nm. Next to surface diffusion even volume and grain boundary diffusion would take place because these two mechanisms are connected with shrinkage. Other work [7] confirms by specific surface area measurements, XRD and TEM that ZrO₂ particles in the same size range are coarsening with increasing calcination temperatures from 400 to 450 and 600°C. For example from XRD data crystallite sizes of 6, 8, 11 nm for the respective temperatures were calculated.

Coalescence has to be mentioned as a further particle coarsening mechanism. In-situ TEM observations at 890°C revealed this mechanism for 20-200 nm zirconia particles next to formation of sintering necks [8].

Physisorbed and chemisorbed surface water becomes quantitatively relevant for nanoparticulate powders because of their high specific surface area. For their study the authors of [9] annealed ZrO₂ powder with a specific surface area of 80 m²/g

at 600°C to remove organics. Then, they found a reversible water uptake of 2.5 wt.-% from air as determined by weight loss up to 400°C by TG analysis. In agreement with this result and on the basis of quasielastic neutron scattering experiments, they conclude the existence of two hydration layers on top of the layer of surface OH groups. The number of layers results from the assumed areal density of about 4.6 molecules per nm² which was a cited literature value, given for silica surfaces. The question arises if a higher water amount could have been found if final TG analysis temperature had been higher. A report [10] leads to this conclusion because their FTIR spectral analysis shows that OH groups are still present on zirconia surfaces after heat treatment at 400°C under vacuum.

Anyway, several OH groups per nm² are present on zirconia surfaces representing an amount being worth to consider when nanoparticles are used generally. Dehydration alters chemical bonds and is also causing material transport. So, it may contribute to the formation of interparticle bonds, as will be discussed.

The present work investigates the chemical and microstructural development of coatings based on a nanoparticulate “mortar” in a temperature range up to 500°C in order to identify possible bonding mechanisms in our coatings [4].

Experimental

In our work we compare the behaviour of a) the pure ZrO₂ nanoparticle fraction



("n-ZrO₂") and b) the coating material (termed as the "mix") that did not undergo thermal consolidation at 500°C prior to the examinations.

The n-ZrO₂ (doped with 4 mole-% Y₂O₃) was produced by a precipitation and hydrothermal treatment. Afterwards, as continuation of the nanoparticle preparation process [11], the particles were chemomechanically surface modified to obtain stable colloids ($d_{50}(\text{vol}) = 14 \text{ nm}$) in ethanol. XRD reveals tetragonal crystallites with an average size of 6.1 nm. A specific surface area of 164 m²/g can be calculated ($\text{Aspec} = 6000 / [d_{\text{XRD}} \cdot \rho]$) from this value which is in good agreement with BET measurements: 166 m²/g for the powder being free of organics.

The ethanolic suspension for the spray application of the coatings contained 80 wt.-% ZrO₂ and 1.3 wt.-% 3,6,9-trioxadecanic acid as surface modifier / dispersing agent. The ceramic fraction consisted of 65.8 wt.-% calcined spray dried granules (Tosoh TZ-3Y, exhibiting 4.6 m²/g after calcination at 1200°C), 26.3 wt.-% of de-agglomerated ZrO₂ (Tosoh TZ-3Y, 13.5 m²/g) and 7.9 wt.-% of n-ZrO₂. This suspension was sprayed on steel substrates, dried, and the coating was removed to gain the "mix". For the results in [4] the coatings have been consolidated at 500°C and it is shown there that their abrasion resistance is clearly higher, in comparison to coatings without nanoparticles. Further details on suspension and coating preparation can be found in [4].

Here, the mix and n-ZrO₂ were examined by ...

- Dilatometry - Linseis "Dilatometer L25". Samples have been compacted without binders or pressing additives by axial and isostatic pressing, 10 K/min heating rate in the dilatometer
- N₂ ad-/ desorption - Quantachrome "Autosorb-AS6", for BET specific surface area determination and to receive the BJH pore size distribution
- TG/DSC - Netzsch "STA 449 C", 10 K/min heating rate, in air atmosphere
- C/H analyser (carrier hot gas extraction method) - Leco "RC-412", 50 K/min heating rate
- XRD - Philips "X'Pert MRI"

Results and Discussion

Sintering shrinkage measurements as well as measurements to determine the development of the specific surface area and of the pore structure have been executed to allow conclusions about sintering mechanisms. The accompanying characterization of mass loss, heat flow and C/H release completes the examinations on the side of chemical reactions taking place upon heating the material up to 500°C.

1 Dilatometry

Shrinkage curves of the mix and n-ZrO₂ are shown in figure 1. Due to the use of

a coarse ZrO_2 fraction (calcined granules in the size range 10-100 μm) the mix material does not undergo relevant shrinkage, even up to 1000°C, which is an important requirement to receive crack free and adhering coatings on a rigid substrate.

In contrast, the shrinkage of n- ZrO_2 starts above 200°C and passes to accelerated sintering at about 900°C. This broad temperature range of first linear shrinkage and the observed shrinkage of 3.2 % at 500°C leads to the conclusion that material transport mechanisms (particle rearrangement, diffusion) take place. Thus, neck growth and shrinkage are relevant for n- ZrO_2 at 500°C, although those mechanisms appear at much higher temperatures for sub- μm particles.

The coarsening of n- ZrO_2 particles is confirmed by XRD measurements (6.1 \rightarrow 7.8 nm after heat treatment at 500°C). From dilatometry it can be concluded that nanoparticles themselves can undergo shrinkage at comparatively low temperatures. Therefore, nanoparticles in the coatings being in contact with larger particles can act as binding bridges by the formation of sintering necks without shrinkage. The binding effect of the nanoparticles can be explained on the basis of material transport mechanisms that have been proven indirectly by dilatometry, XRD and specific surface area measurements, as will be shown in the next section.

2 Specific surface area and pore size

The specific surface area of the n- ZrO_2 decreases from 166 m^2/g at room temperature to 115 m^2/g at 500 °C (table 1). The reduction of the specific surface area is in good agreement with the theoretical values calculated from XRD data due to particle coarsening and formation of particle contact areas that are not longer accessible for N_2 adsorption.

n- ZrO_2	calculated from XRD data	measured
RT	6.1 nm \rightarrow 164 m^2/g	166 m^2/g (a)
500°C	7.8 nm \rightarrow 128 m^2/g	115 m^2/g

Table 1: Specific surface area of n- ZrO_2 , (a): mean value of measurements on 2 samples after stripping off organics

Table 2 summarises specific surface areas for the mix. Values slightly above 21 m^2/g have been measured. The calculated value was obtained by adding the weighted

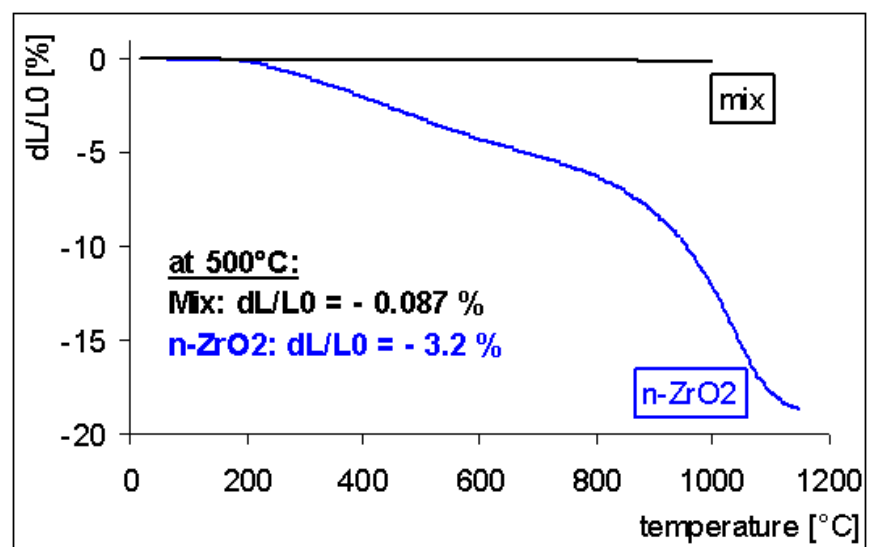


Fig. 1: Linear shrinkage of mix and n- ZrO_2 , dilatometric measurement (see text for specification of mix).



areas of the ZrO_2 fractions. The fact that measured values are higher than the calculated values can be explained by a slight sedimentation of coarse zirconia granules in the suspension prior spray application and their resulting depletion in the coating. This effect probably causes variations of the fractional ZrO_2 coating composition which may also result in variations of surface areas. Another factor influencing surface area measurements can be given by the presence of organics (1.6 wt.-% = 6.2 vol.-% surface modifier). To check if the organic fraction masks a part of the surface area, the mix was cleaned/washed with Na(OH) to strip off the organics. The so treated mix exhibits a larger surface area of 23.8 m^2/g . Compared to the value after heat treatment at 500°C, it results in an apparent decrease of surface area. This is a hint to conclude that surface reducing sintering mechanisms can also be present in the mix. But, regarding the discussed possible compositional variations, this conclusion has to be made with care.

Mix	measured	calc. from comp.
RT	21.1(b) / 23.8(c) m^2/g	19.7 m^2/g
500°C	21.6 m^2/g	15.7 (d) m^2/g

Table 2: Specific surface area of the mix. (b): with organics, (c): free of organics, (d) used value for n- ZrO_2 : 115 m^2/g (see text for specification of mix).

Increase in pore volume and average pore size (11.3 \rightarrow 13.7 nm) can be observed in figure 2, when the mix in room temperature state / after heating to 500°C is compared (figure 2). On the basis of pre-

sent results, it cannot be concluded if this is due to organics burn out and/or due to sintering neck formation and “pore smoothing” [5].

A complete disappearance of the micropores (< 2 nm) being present in the room temperature state of the mix give a strong hint for sintering neck formation and/or coalescence of particles at 500°C.

3 DSC / TG measurements

Figure 3 shows the DSC and TG plots of the mix upon heating. There is one exothermic DSC peak at 333°C which can be ascribed to organics burn out and correlates to the mass loss in this temperature range. A further step of mass loss below 200°C can be explained with the loss of adsorbed water.

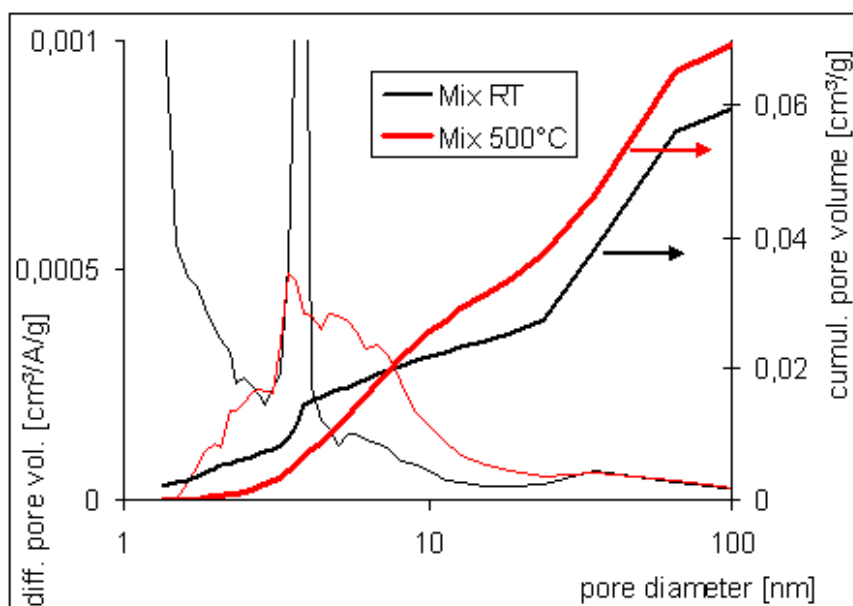


Fig.2: Differential and cumulative pore volume distributions in the mix (see text for specification of mix).

4 C/H analyser

The DSC and TG data can be underlined by measurements with the C/H analyser, as shown in figure 4. Here, the appearance of carbon coming from the sample shows a maximum at 287°C. This lower peak temperature can be explained by the use of pure oxygen as analysis gas in comparison to air in the DSC/TG.

Water originates from different sources like water adsorbed on the surface and in the pores (appears at lower temperatures than C), from oxidized H from organics (signal decreases parallel to that of C) and from surface OH groups (at higher temperatures). Considering a C content of 50 wt.-% and a H content of 10.5 wt.-% in the organics (this is close to the values of the surface modifier and of potential residual ethanol in the pores), it is possible to allocate 2.7 wt.-% of the measured water to the organics. From two Karl-Fischer titrations, a mean content of adsorbed water in the mix of 0.44 wt.-% was determined. Subtracting this physisorbed water and the amount of water due to organics from the total measured water amount (3.54 wt.-%) leads to the result that 0.4 wt.-% water may have surface hydroxyl groups as origin.

This equals to 11 OH groups per nm² of the ZrO₂ surface area, which is significantly higher than given in literature (4.6 OH groups / nm²) [9]. These authors assumed the existence of three layers with this density and stated that the adsorbed H₂O layers being directly on the layer of OH groups are very immobile, too. So, these inner immobile adsorbed water lay-

ers may not be detected by the Karl Fischer method. This is a possible reason why we found a higher number of surface OH groups.

Anyway, from this calculation and the general state of knowledge the existence of several OH groups per nm² on surfaces of oxides can be taken as fact. As they become quantitatively relevant for nanoparticles with e.g. surface areas > 100 m²/g, this publication suggests to consider their role in binding effects by nanoparticles. At low temperatures where diffusion mechanisms are not yet relevant, binding could take place as condensation of OH groups to form Me-O-Me bonds (Me = metal).

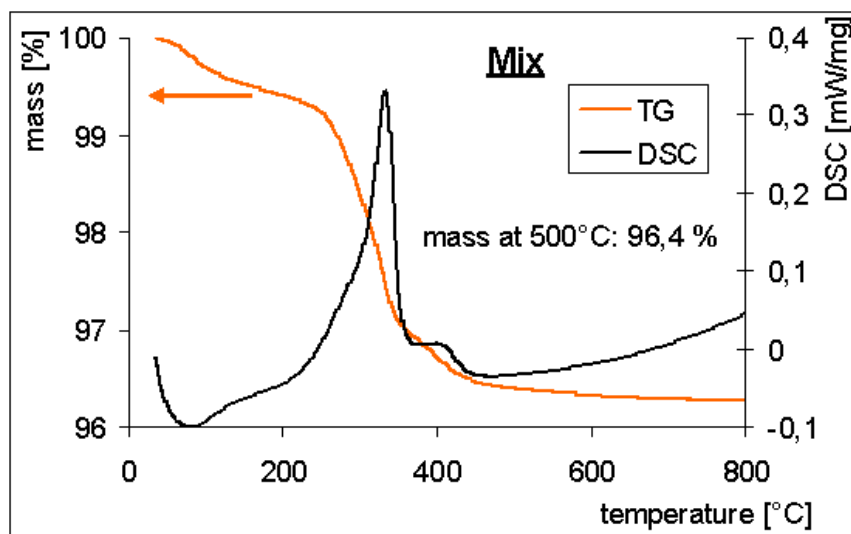


Fig. 3: Thermo-gravimetric and differential scanning calorimeter signal of the mix (see text for specification of mix).



Conclusions

The remarkable bonding effect [4] of ~ 8 wt.-% zirconia nanoparticles in ZrO_2 coatings that have been heat treated at 500°C is examined and discussed for the first time. For this temperature, in agreement with the literature it was concluded that sintering mechanisms are active for the nanoparticles used, even those mechanisms that cause grain coarsening and shrinkage locally. Further, the condensation of surface hydroxyl groups may contribute to inter particle bonding. This effect may be superimposed by sintering.

More research is needed to identify and quantify the relevant bonding mechanisms in detail. Further information would be gained if neck growth and/or coalescence of ~ 6 nm ZrO_2 particles at 500°C could be confirmed by imaging, for example with TEM observations.

The observed bonding effect was combined with the use of multiple particle fractions with different sizes to achieve densely packed structures inhibiting overall shrinkage. This approach allows the preparation of porous, well adhering ceramic coatings on rigid substrates with strength values sufficient for applications with limited requirements on the strength. It opens the way to new applications because a large variety of substrates can be equipped with such coatings via simple wet coating processes using suspensions and subsequent heating at moderate temperatures as 500°C.

For the development of new applications for those coatings it seems to be promi-

sing to apply further research effort. This will utilise the described principles for the realization of other ceramic systems. It also seems to be realistic to achieve further optimization of coating strength, density and thickness.

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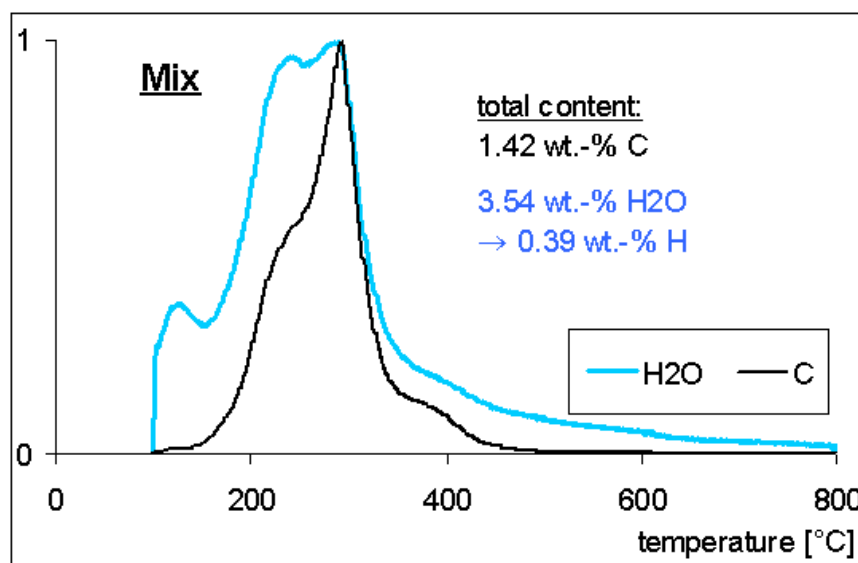


Fig. 4: Temperature dependent C and H₂O release from the mix, normalized signal (see text for specification of mix).



Preparation of Acoustic Lenses by Mechanochemical Synthesis and Electrophoretic Deposition of Lead Zirconium Titanate (PZT) Films

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References

- [1] H. Salmang, H. Scholze, R. Telle (ed.): *Keramik*. Springer, Berlin, Heidelberg (2007)
- [2] W. Lan, P. Xiao: Drying stress of yttria-stabilized-zirconia slurry on a metal substrate. *J. European Ceramic Soc.* 27 (2007) 3117-3125
- [3] X. Wang, W.H. Lan, P. Xiao: Fabrication of yttria stabilized zirconia coatings by a novel slurry method. *Thin Solid Films* 494 (2006) 263-267
- [4] M. Aslan, J. Adam, B. Reinhard, M. Veith: Preparation of thick zirconia coatings by spraying of colloidal suspensions. *Proceedings of the 10th International Conference and Exhibition of the European Ceramic Society*, 17.-21. June 2007 in Berlin
- [5] M.J.M. Akash: Zr surface diffusion in tetragonal yttria stabilised zirconia. *J. Materials Science* 35 (2000) 437-442
- [6] G. Skandan: Processing of nanostructured zirconia ceramics. *NanoStructured Materials* 5 (1995) 111-126
- [7] W. Li, L. Gao: Compacting and sintering behaviour of nano ZrO₂ powders. *Scripta materialia* 44 (2001) 2269-2272
- [8] J. Rankin, B.W. Sheldon: In situ TEM sintering of nano-sized ZrO₂ particles. *Materials Science and Engineering A204* (1995) 48-53
- [9] E. Mamontov: Dynamics of surface water in ZrO₂ studied by quasielastic neutron scattering. *J. Chemical Physics* 121 (2004) 9087-9097
- [10] G. Cerrato, S. Bordiga, S. Barbera, C. Morterra: Surface characterization of monoclinic ZrO₂, I. Morphology, FTIR spectral features, and computer modelling. *Applied Surface Science* 115 (1997) 53-65
- [11] H. Schmidt, K.-P. Schmitt, K. Schmitt, F. Tabellion: Method for production of nanoparticles with custom surface chemistry and corresponding colloids. Patent application WO 2006037591 A3 (priority: 2004)

Preparation of Acoustic Lenses by Mechanochemical Synthesis and Electrophoretic Deposition of Lead Zirconium Titanate (PZT) Films

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Abstract

PZT powder has been synthesized via reactive dry milling using PbZrO₃ and PbTiO₃ as starting materials. Stable suspensions of the PZT particles in ethanol ($d_{50}(\text{Vol}) = 115 \text{ nm}$) were obtained by a chemomechanical disper-



sion step. The electrophoretic deposition has been optimized varying the cell voltage and the PZT solid content in the suspension. PZT films have been deposited on platinum coated sapphire. After drying, the films are densely packed and free of cracks. By using lithium acetate and lead acetate as a sinter aid it was possible to reduce the sintering temperature to 1050°C. A gold electrode has been sputtered onto the piezoelectric films which then have been poled by the corona method. The circular PZT dots ($\varnothing = 2$ mm) with a thickness of 1 μm show the expected oscillation resonance at about 2 GHz and can be used in acoustic lenses, for example in acoustic microscopes.

Introduction

In liquids acoustic waves in the gigahertz range are known to have wavelengths comparable to that of visible light [1]. In acoustic microscopy this property is used to determine the elastic structure of samples in a mechanical scanning system.

The basic component in an acoustic microscope is the acoustic lens. This is a simple device made of a single-crystal silicon-substrate supporting the piezoelectric unit which is composed of the piezoelectric ceramic (mostly ZnO) and two electrodes [1]. The working-frequency of the lens depends on the thickness of the ceramic layer.

The piezoelectric properties of PZT (stoichiometry in this work: $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$) exceed the ones of ZnO. So the cou-

pling factor of PZT is higher by a factor of 3 than that of ZnO [2]. The piezoelectric constant d_{33} is higher about a factor of 10 [3].

So it should be promising to replace the ZnO ceramic by a PZT ceramic to make the advantages of PZT available for acoustic microscopy. In this work an acoustic lens, in which a piezoelectric PZT-ceramic is built by electrophoretic deposition (EPD), has been produced. EPD is chosen because of the easy process control in comparison to sputtering or chemical vapour deposition of PZT. Via EPD, stoichiometry and phase purity of the end-product is already fixed before the deposition process.

In the EPD-process it is possible to create highly uniform ceramic films [11]. Electrophoretic shaping is suitable for handling powders in the nanometer-range, since deposition velocity is independent from particle size. An adequately stabilised suspension of the particles is a precondition for a successful deposition. The stability of colloidal suspensions is described by the DLVO-theory [12, 13], labelled by Derjaguin, Landau, Verwey and Overbeek. According to that theory surfaces of colloidal particles are regarded as capacitor plates. In a suspension, electrochemical double layers form at the capacitor's surfaces. The range of the resulting repulsive forces is higher than the range of the attractive Van-der-Waals forces, which leads to a stabilization of the suspension.

Due to an applied electric field, charged particles in the suspension move to the

oppositely charged electrode, where they agglomerate to a dense green film. This green film has to be sintered to high densities and to be poled to make it suitable for sensors and actuators.

Experimental

The PZT powder used in this work is synthesised by a mechanochemical process. In a high energy ball mill provided with a jar (125 ml) and balls made of tungsten-carbide, commercially available PbZrO_3 (Aldrich) and PbTiO_3 (Aldrich) powders with a particle size distribution in the sub- μm -range are mechanochemically treated to get a single phase PZT-powder with a stoichiometry of $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$, which is near the morphotropic phase boundary. To determine optimal parameters for the mechanochemical process, rotation velocity, ball-to-powder weight ratio and rotation time are varied. The phase transition to PZT is observed by an X-ray diffractometer (Siemens D500). In previous works different conditions are described to get PZT by mechanochemical treatment [4, 5].

After the mechanochemical synthesis of the powders, the particles are strongly aggregated. So, a further chemomechanical dispersion step is applied. This step is accomplished in a laboratory ball mill (Fritsch, Pulverisette 6) with a ZrO_2 -jar and ZrO_2 -balls with 0.3 mm diameter in ethanol charged with an organic surface modifier, an oxocarboxylic acid. In using a carboxylic acid, among electrostatic stabilisation as described in the DLVO-

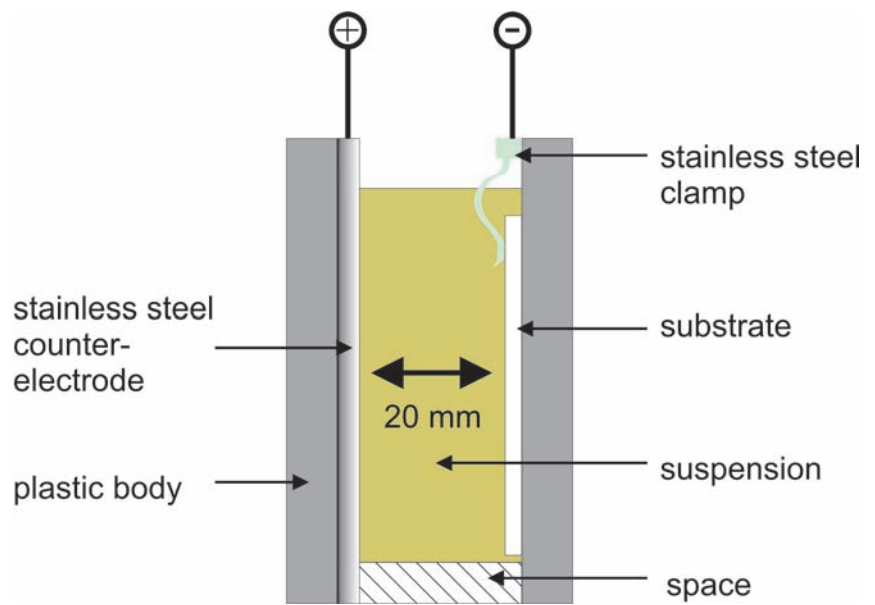


Fig. 1: EPD-cell.

theory, a steric stabilisation by the carbon chains is given. This combination is known as electrosteric stabilisation. In this way, the particles are deaggregated in a stabilized PZT/ethanol-suspension for use in the electrophoretic deposition process. Ethanol is used as dispersion medium because of experiences made by Ma [8, 9, 10]. The particle size distribution is determined by an ultrafine particle analyzer (UPA 400, Grimm). Further on the particles are characterized by SEM and EDX (JEOL, JSM 6400 F).

16 short-circuited bottom-electrodes made of platinum are sputtered on to the sapphire-substrates. To avoid flaking of the electrodes during the sintering a sputtered 10 nm adhesive layer between substrate and platinum electrodes is used. The dimensions of the substrates are 5 cm x 5 cm x 2 mm. The resulting



Pt-dots have a diameter of 5 μm and a thickness of 200 nm – 1 μm . For use in acoustic microscopy, the thickness of the electrodes should be as low as possible to avoid loss of sonic energy.

To optimise the electrophoretic process, it is necessary to vary the cell voltage, the solid content in the suspension and the deposition time. The ζ -potential of the PZT-suspensions are measured at various pH values adjusted by NaOH and the surface modifier, an oxocarboxylic acid. After that, the pH has to be regulated to the maximum value of the ζ -potential to get a stabilized suspension. The design of the EPD-cell is shown in Figure 1. The substrate with the short-circuited electrodes is fixed and electrically contacted by a stainless-steel clamp. The metrics of the counter electrode made of stainless-steel are 80 mm x 50 mm x 2 mm. The distance of the electrodes is fixed at 20 mm. The cell is connected to a DC voltage source, counter electrode at positive pole, substrate at negative pole.

After deposition, green films are treated with a sintering aid. Van Tassel and Randall propose a sintering aid made of methyl ethyl ketone containing lead ethylhexanoate and lithium acetate with a small addition of methanol to dissolve the acetate [6]. In this work a similar aid is used made of lithium acetate-dihydrate, lead acetate-dihydrate, methyl ethyl ketone and methanol.

Sintering is accomplished in a cement sealed Al_2O_3 -crucible with a lead-source to avoid the loss of lead in the PZT-film during the sintering process. Lead-source

is a commercially available PZT-Powder (EPC, Ltd., PZT 856). The influence of the sintering aid is determined by a dilatometer and by SEM-images (JEOL, JSM 6400 F).

After the sintering process the top-electrode made of gold is sputtered on the PZT-films and the films are poled using the corona-method [7]. Now the functionalised coatings are ready to be characterised in a network analyzer (Hewlett Packard, HP 8719D). The analyzer generates a microwave-signal, which is supplied into a coaxial cable enclosed by the sample. The configuration is shown in Figure 2.

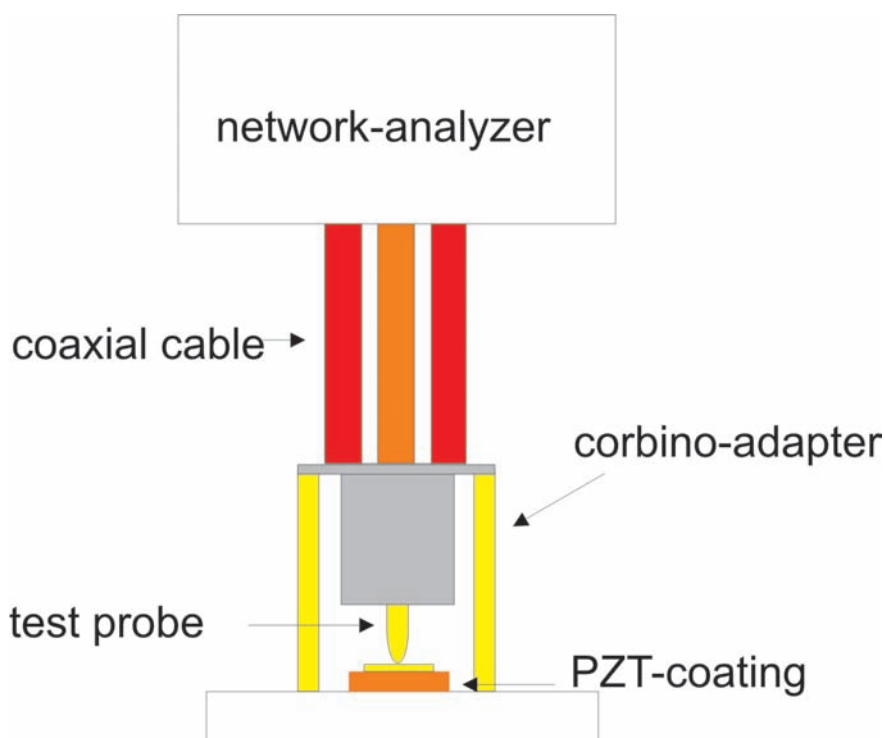


Fig. 2: Configuration for electrical characterisation of functional coatings.

The result of this measurement is the reflection factor S_{11} . It describes the percentage of the reflected energy at different frequencies. So a high reflection factor means a low absorption of energy by the sensor. Analysis is focused at the frequency range around the resonant frequency of the system, that depends on PZT-film- thickness.

Results and Discussion

In Figure 3 the X-ray patterns for mechanochemical synthesised PZT-Powder are shown. The ball-to-powder weight ratio was 20:1 and the rotation velocity was at 300 rpm. To reveal the formation of the PZT-phase, samples are taken out of the milling-jar after 15 min, 30 min, 60 min and 120 min and analyzed by X-ray-diffraction.

The phase transformation to $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ can be observed by investigating the (120)-Peak of PbZrO_3 and the (100)-Peak of PbTiO_3 . Within 120 minutes the lattices of the raw materials PbZrO_3 (orthorhombic) and PbTiO_3 (tetragonal) transform to the new lattice of $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$ (trigonal/tetragonal).

Contamination by tungsten-carbide is neither detectable by X-ray-diffraction nor by EDX. The product is a yellow PZT-powder with aggregates of 300 nm to 1 μm (Figure 4).

The measured particle size distribution confirms the SEM-results (Figure 5).

In the following dispersion step, the PZT-powder is deaggregated in ethanol

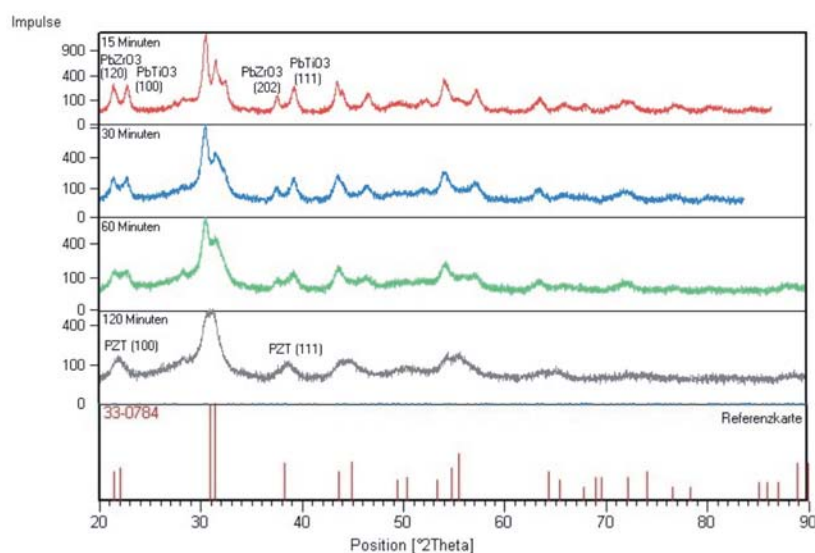


Fig. 3: X-ray-patterns of mechanochemical synthesised $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$, ball-to-powder weight ratio 20:1, 300 rpm and reference pattern for $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$.

charged with an organic surface modifier within 7 hours to a relatively narrow particle size distribution with a d_{50} value of 115 nm (Figure 6).

The ζ -Potential of the modified powder shows a maximum at pH 8.5 (Figure 7), measured in ethanol. Values of pH are adjusted by NaOH and the surface modifier, an oxocarboxylic acid.

According to that, EPD is carried out at pH 8.5 adjusted by the surface modifier. The solid content in the suspension is 2 g PZT per 100 ml ethanol. Higher contents lead to cracks in the coatings, lower contents lead to porous coatings. In a similar way, the morphology of the coatings depends on deposition time. There is only a narrow working-window to get dense coatings, free of cracks. Best results are achieved with suspensions of 2 g

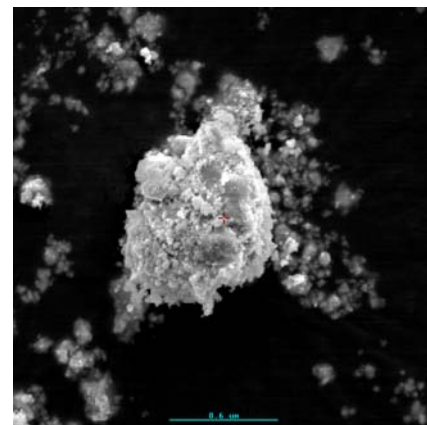


Fig. 4: SEM-image of mechanochemical synthesised PZT-aggregates.



PZT per 100 ml ethanol and deposition times between 10 and 15 seconds under an electric field of 30 V/cm. The thickness of the coatings is about 1.4 – 2 μm , depending on deposition time.

Figure 8 shows a fracture cross-section of a PZT-green-film deposited on a sputtered Pt-electrode on polycrystalline Al_2O_3 . Deposition time was 10 sec.

After deposition the green-films are rewetted with the sintering aid, a mixture of lithium acetate-dihydrate, lead acetate-dihydrate, methyl ethyl ketone and methanol. While drying, most of the solvents evaporate leaving behind the metal organics, which are then burned out during the sintering process. The films are sintered at 1050 $^\circ\text{C}$ for 30 min.

The average grain size, as determined by inspection of a fracture cross-section of the film, is about 500 nm. The thickness of the films is about 1 μm , which corresponds to a resonance frequency at 2 GHz.

Figure 9 shows a fracture surface cross-section of a sintered PZT-film on a sputtered Pt-electrode on sapphire. Top view of the coatings shows a dense crack free surface (Figure 10).

X-ray diffraction pattern for the films indicates that the films are phase pure PZT. Reactions with the substrate are not observed (Figure 11).

After sputtering the gold-top-electrode onto the PZT-films, the films are polarised by the corona method for 60 min at

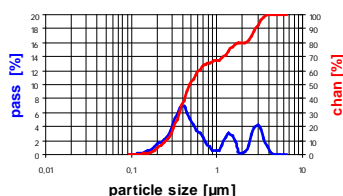


Fig. 5: Particle size distribution by volume of mechanochemical synthesised PZT-powder.

a voltage of 4 kV/cm and a temperature of 150 $^\circ\text{C}$, shown in Figure 12.

Now, the completed sensors are characterised by a network analyzer. Figure 13 shows the result of the analysis. As expected, there is a resonance at 2 GHz. At that frequency nearly 80 % of the incoming energy is absorbed by the sensor-system, what relates to the oscillation of the PZT-film in thickness mode.

Figure 14 shows the top view of one of the 16 completed sensors. In the middle the gold electrode is visible. The PZT ceramic is located between the little gold electrode and a bigger platinum electrode on the sapphire. The sapphire becomes apparent by the shadow of the platinum electrode in the background. It can be observed that the PZT ceramic is transparent for visible light. This can be ascribed to a grain size of 500 nm inside the ceramic, comparable with the wavelength of visible light.

Conclusions

Transparent crack-free PZT thin films for use in acoustic microscopy are pro-

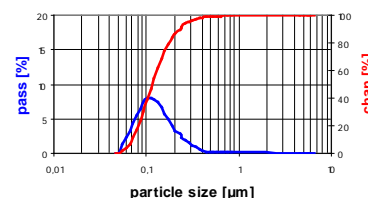


Fig. 6: Particle size distribution by volume of mechanochemical synthesised PZT-powder, dispersed in ethanol for 7 hours in a ZrO_2 -jar.

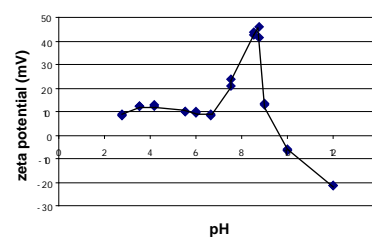


Fig. 7: ζ -potential of the synthesised PZT-powder in ethanol at various pH values.

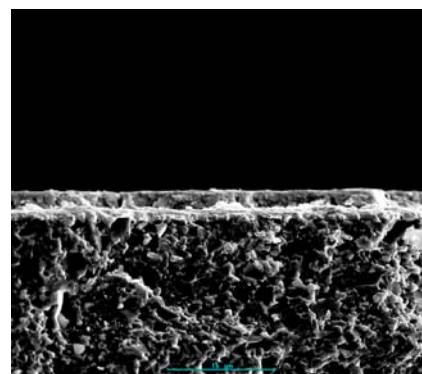


Fig. 8: Fracture surface cross-section showing a PZT-green-film deposited on a sputtered Pt-electrode on polycrystalline Al_2O_3 .

duced by mechanochemical synthesis, electrophoretic deposition and sintering of PZT-powder. As expected, the polarised films show a significant resonance at about 2 GHz.

Problems like delamination of the sputtered ground electrode were solved by using an adhesive layer.

The sinter aid, the surface modifier and the synthesised nano-powders lead to homogeneous crack-free PZT-coatings.

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M. Bender, R. Drumm, J. Adam, A. Jakob, R. Lemor, M. Veith "Preparation of Acoustic Lenses by Mechanochemical Synthesis and Electrophoretic Deposition of Lead Zirconium Titanate (PZT) Films" Proc. 10th ECerS Conf. (2007), edited by J.G. Heinrich and C.G. Anziris, Göller Verlag, Baden-Baden, pp. 848-853.

References

1. Quate C.F., Atalar A., Wickramasinghe H.K.: Acoustic microscopy with mechanical scanning – A review, proceedings of the IEEE, Aug. 1979, Vol. 67, Issue: 8, 1092-1114
2. M.E. Motamedi: Acoustic Sensor Technology, Microwave Symposium Digest, 1994, IEEE MTT-S International
3. W. Tjhen, T. Tamagawa, C.-P. Ye, C.-C- Hsueh, P. Schiller, D.L. Pol-

la: Properties of piezoelectric Thin Films for Micromechanical Devices and Systems, Micro Electro Mechanical Systems, 1991, MEMS '91, Proceedings. 'An Investigation of Micro Structures, Sensors, Actuators, Machines and Robots'. IEEE

4. Z. Brankovic, G. Brankovic, C. Jovalekic, Y. Maniette, m. Cilense, J.A. Varela: Mechanochemical Synthesis of PZT Powders, Materials Science and Engineering A345 (2003) 243-248
5. D. Stojanovic: Mechanochemical synthesis of ceramic powders with perovskite structure, Journal of Materials Processing Technology 143-144 (2003) 78-81
6. J. Van Tassel and C.A. Randall: Electrophoretic Deposition and Sintering of Thin/Thick PZT Films, Journal of the European Ceramic Society 19, (1999), 955-958
7. David Waller, Tariq Iqbal, Ahmad Safari: Poling of Lead Zirconate Titanate Ceramics and Flexible Piezoelectric Composites by the Corona Discharge Technique, J.Am. Ceram. Soc., 72, (1989), 2, 322-24.
8. J. Ma, R. Zhang, C.H. Liang, L.Weng: Colloidal characterization and electrophoretic deposition of PZT, Materials Letters 57 (2003), 4648-4654
9. J. Ma, Wen Cheng: Electrophoretic Deposition of Lead Zirconate Titanate Ceramics. Journal of the

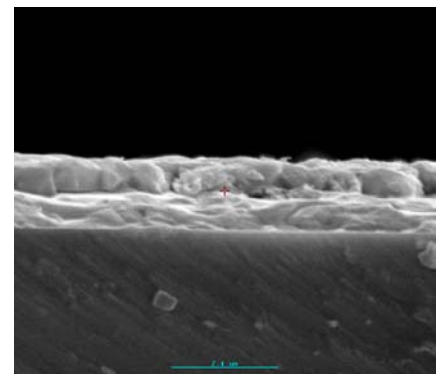


Fig. 9: Fracture surface crosssection showing a sintered PZT-film on a sputtered Pt-electrode on sapphire.

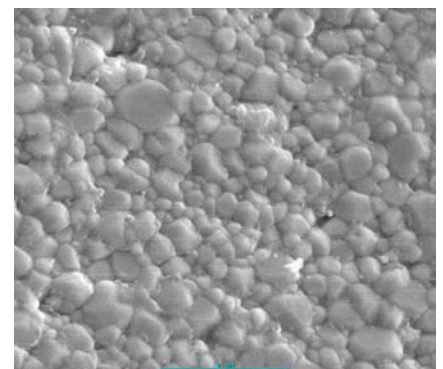


Fig. 10: Top view of a sintered PZT-coating by SEM.

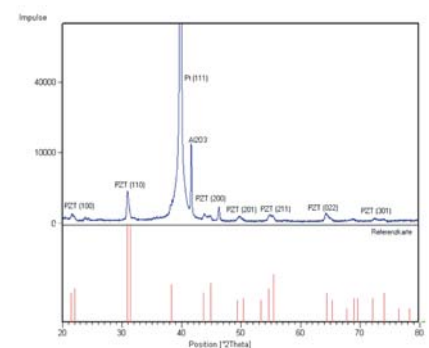


Fig. 11: X-ray diffraction pattern of a sintered PZT-film on a sputtered platinum electrode on sapphire and reference pattern for $Pb(Zr_{0.52}Ti_{0.48})O_3$



Wet Chemical Syntheses of Ag-Nanoparticles

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- American Ceramics Society 85, 7, (2002), 1735-1737
10. J. Ma, Wen Cheng: Deposition and packing study of sub-micron PZT ceramics using electrophoretic deposition, *Materials Letters* 56, (2002), 721-727
 11. H. Wittwer and H.G. Krüger, Möglichkeiten und Grenzen der Elektrophorese. *Cfi/Ber. DKG* 72, (1995), 556-560
 12. B. Derjaguin, On the Repulsive Forces between Charged Colloid Particles and on the Theory of Slow Coagulation and Stability of Lyophobic Sols. *Trans. Faraday Soc.* 36, (1941), 203-215
 13. E. J. W. Verwey and J. T. G. Overbeek, *Theory of the Stability of Lyophobic Colloids The Interaction of Sol Particles having an Electric double Layer*, (1948), Amsterdam-New York: Elsevier Publishing Company

Wet Chemical Syntheses of Ag-nanoparticles

Introduction

The synthesis of nanoparticles with well defined size, morphology and crystallinity becomes more and more important to advance in the field of nanotechnology. Especially high-temperature wet-chemical syntheses are very promising to realise the production of particles with narrow

size-distribution and a sufficient yield [1-4]. Beside this goal, for industrial purposes it must be taken into account, that the chemicals costs as well as the expenses for processing are within acceptable limits. In addition, the nanoparticles have to be sufficiently stabilised to ensure their subsequent treatment. In this article, a synthesis-route for silver-nanoparticles is described which fulfils the above mentioned criteria to a large extent. Furthermore, particle size and size-distribution can be varied to some degree by changing the solvent, the heating-rate, the maximum-temperature and the duration of synthesis.

Experimental

Materials

Diphenylether 99 % (DPE), dibenzylether 99 % (DBE), dioctylether 99 % (DOE), 1-hexadecene 92 % (1-HD) and oleic acid 90 % (OA) were purchased from Aldrich, 1-octadecene 90 % (1-OD) from Alfa Aesar, 1-eicosene 85 % (1-Eic) from ABCR, tri-n-octylamine 98 % (TNO) from Acros, silver nitrate 99 % (AgNO_3) and oleylamine 70 % (OAA) from Fluka. All chemicals were used without further purification.

Syntheses

Synthesis route 1

In a typical synthesis, 10 ml solvent (for example DBE) and 8 mmol (2.26 g) OA were mixed in a 100 ml three-necked

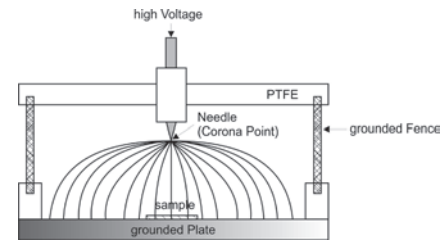


Fig. 12: Polarisation by the corona method

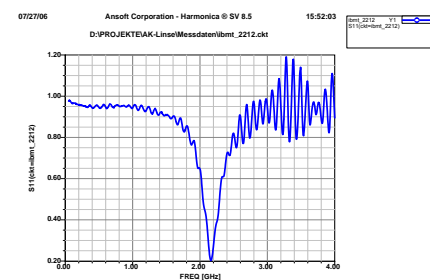


Fig. 13: Dependence of the reflection factor S 11 of various frequencies

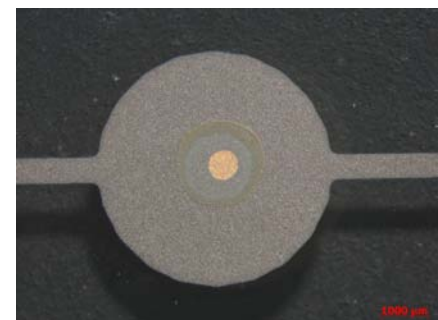


Fig. 14: Completed sensor, image made by light-optical microscope

round-bottom flask and heated under stirring to $T = 200\text{ }^{\circ}\text{C}$ under a nitrogen atmosphere with a predefined heating rate $\text{h.r.} = 3\text{ K/min}$. At $T = 140\text{ }^{\circ}\text{C}$, 1 mmol (0.169 g) AgNO_3 was added as a powder, followed by 2 mmol (0.535 g) OAA. Within a few seconds, the formerly colourless solution became first yellow, then brown, indicating the formation of silver particles. After refluxing for 60 minutes, the heating source was removed and the solution was allowed to cool down to room temperature.

Synthesis route 2

In an alternative route, Ag-oleate was prepared similar to a process for Fe-oleate [5] and used as the silver precursor instead of AgNO_3 . 1 mmol Ag-oleate was mixed with 10 ml TNO, 2 mmol OAA and 7 mmol OA to achieve the same concentrations of precursor, surface modifier and solvent as in the synthesis route mentioned before. The mixture was heated to $T = 200\text{ }^{\circ}\text{C}$ with $\text{h.r.} = 3\text{ K/min}$ and allowed to reflux at this temperature for $t = 60\text{ min}$ before cooling down to room temperature.

Independent of the synthesis route, the brown product was precipitated by mixing firstly 0.5 g of the particle dispersion with 1 g cyclohexane and then adding 6 g ethanol. This mixture was centrifuged at an acceleration of $a = 10,000\text{ g}$ for $t = 20\text{ min}$ at $T = 20\text{ }^{\circ}\text{C}$ and the yellowish supernatant was discarded. This procedure was repeated once. Finally, the particles were dispersed in 1 g cyclohexane.

Characterisation

To prepare a sample for transmission electron microscopy, one drop of the solution was put onto a carbon coated copper grid. TEM was carried out using a Philips CM 200 FEG high-resolution microscope. To determine the particle-size and size distribution, comparable micrographs of the respective samples were binarised and analysed using the software Digital Micrograph v. 3.8 and ImageJ v. 1.39u.

For revealing the crystallinity of the as prepared silver nanoparticles, X-ray diffraction analyses were carried out. Therefore, the particles were purified four times by a procedure similar to the above mentioned to minimize the amorphous background due to the organic residues.

Results and Discussion

Synthesis

Each synthesis system consists of the following components: silver-precursor, solvent, and organic surfactant. For synthesis route 1, the silver-precursor (AgNO_3) is known to be a salt. Raman and FTIR measurements indicate that this is also the case for Ag-oleate. By heating to a sufficiently high temperature the silver precursor dissociates. Although the reaction mechanism is not yet understood in detail, it is assumed that the Ag^+ -ions are reduced to Ag^0 by electron transfer from the ligands L.

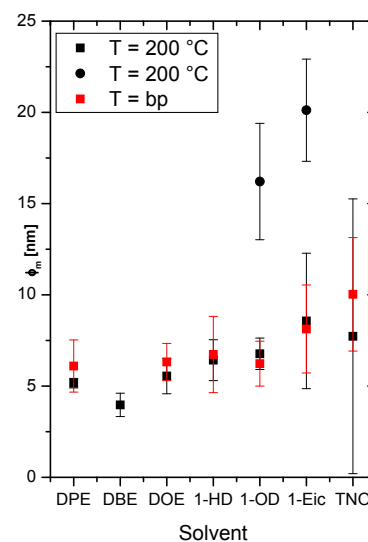
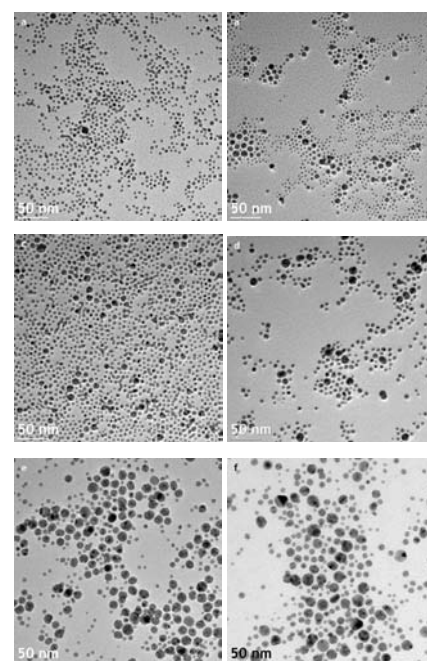
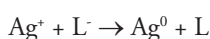


Fig. 1: TEM-micrographs (a-n) of silver particles synthesised with DPE, DBE, DOE, 1-HD, 1-OD, 1-Eic and TNO at $T = 200\text{ }^{\circ}\text{C}$ (a-g) and $T = \text{bp}$ [$^{\circ}\text{C}$] (h-n) and the respective size and size distribution (Fig. 1o). Note that for DBE at $T = \text{bp}$ [$^{\circ}\text{C}$] (Fig. 1i), no particle size and distribution could be determined because the particles aggregated to clusters of $0.5 - 1\text{ }\mu\text{m}$ in diameter.



Capping of the nuclei with surfactant molecules then promotes the controlled growth [4,6]. As the detailed understanding of the reaction kinetics is crucial for establishing new, advanced synthesis methods, an experimental setup for in situ investigations with mass-spectrometry, FT-infrared spectrometry and UV-Vis spectrometry shall be realised soon.

Influence of the solvent

To investigate the influence of the solvent in the synthesis of silver nanoparticles, we repeated the synthesis based on route 1, using different ether (DPE, DBE, DOE), alkenes (1-HD, 1-OD, 1-Eic) and a tertiary amine (TNO). Mixtures were heated with a constant rate of h.r. = 3 K/min and allowed to reflux for $t = 60$ min not only at $T = 200$ °C but also at the respective boiling point of the solvents as given in Table 1.

The TEM-micrographs show the particles synthesized at $T = 200$ °C in DPE, DBE, DOE, 1-HD and 1-OD as the respective solvent. (fig. 1a-e)

Analysis of the mean particle size ϕ_m and size distribution σ indicates that ϕ_m slightly increases with increasing molar weight of the solvent, whereas higher temperature does not seem to effect particle size. Concerning the size distribution σ , for solvents with low molar weight σ increases with temperature while it decreases for solvents of high molar weight. To understand this, different aspects have to be taken into account [1,2,4]. First of all in our case the structure and with

that the sterical hindrance of the solvents used, drastically changes with increasing molar weight leading to a different coordination to the silver nuclei. Secondly, by keeping the solvent volume in the synthesis constant at 10 ml, the molar rate decreases with increasing molar weight. Therefore the number of solvent molecules surrounding each nucleus decreases and during growth, particles become more polydisperse. Thirdly, in the case of TNO, the tertiary amine can compete with the stabilising surface modifiers in coordinating to the silver ions and thus inhibit a controlled nucleation and particle growth. To proof this assumption, we made another particle synthesis with TNO (route 2), using Ag-oleate as the metal precursor. In this case the stabilisation of the nuclei should be much better because a stabilising agent is already introduced in the metal precursor. As assumed, this synthesis route leads to crystalline particles with decreased size and narrowed size distribution (fig. 2).

Influence of the heating rate

Another parameter in particle synthesis is the heating rate. To synthesise nearly monodisperse particles by thermal decomposition it is favourable to have a large gap between the initial temperature for nucleation T_n and particle growth T_g [5]. In an ideal situation one would keep the temperature constant at T_n until nucleation is finished and then jump to T_g to initiate particle growth. The particle size can then be tuned by varying the ratio of solvent, surface modifier and precursor.

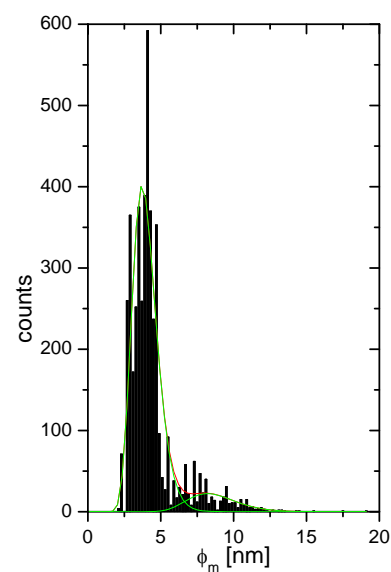
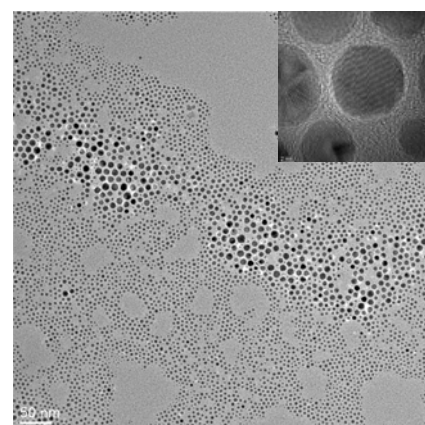


Fig. 2: TEM-micrograph (a) of particles synthesised using Ag-oleate as the metal-precursor. The inset shows an individual particle at high resolution, revealing the crystallinity of the particles. Fig. 2b depicts the particle size and size distribution.

By heating with a constant heating rate, the situation is a little different. Heating too fast, initiates the particle growth before the nucleation has finished and the particles become bigger and more poly-disperse. To verify this, we synthesised particles using DBE as the solvent with $T = 200\text{ }^{\circ}\text{C}$ and $t = 60\text{ min}$ and varied the heating rate with h.r. = 3, 5, 10, 20 and 40 K/min, respectively. As predicted, the particle size increases with h.r. and the size distribution broadens (Fig. 3 f). It must be noted, that the broad size distribution for h.r. = 10 K/min must be ascribed more to problems with particle size analysis due to the formation of a particle bilayer onto the TEM-grid, than on a real effect.

Influence of the dwelling time

The dwelling time at a temperature suitable for particle growth is another parameter that can influence particle size and size distribution [7]. In a first step the particles grow until all of the precursor is decomposed completely. In a second step, small particles dissolve while bigger ones grow. This process is called Ostwald ripening. Therefore we synthesised silver nanoparticles at a constant heating rate of h.r. = 3 K/min with DBE as the solvent by varying the dwelling time at $T = 200\text{ }^{\circ}\text{C}$ from $t = 0, 30, 60$ and 720 min , respectively. As expected, the particle size distribution narrows from $t = 0$ to $t = 60\text{ min}$ but interestingly becomes bimodal for $t = 30\text{ min}$ while the particle size decreases (Fig. 4 a-c). The assumption is, that in this case the final particle size is pri-

marily determined by the ratio of surface modifier to metal precursor. At $t = 0\text{ min}$ the initial particle growth is not completed and the size distribution is therefore broad. At $t = 30\text{ min}$ due to Ostwald ripening, the bigger particles further grow while the smaller ones shrink to a certain size, determined by the amount of surface modifier. In a third step, also the bigger particles shrink to the optimum size determined by the surface modifier. For $t = 720\text{ min}$ it must be noted that due to the continuous flushing of the flask with nitrogen, nearly all of the solvent was carried off although using a reflux cooler and that the surface modifiers (OAA and OA) were already polymerised as can be seen in Fig. 4 d, so that these results are not representative to verify the theory.

XRD measurements were made to determine the crystallinity of the as prepared silver nanoparticles. Although the diffractograms are not suitable to determine the particle size based on the Debye-Scherrer equation, they reveal that the particles are partly (multiply)twinned and crystalline.

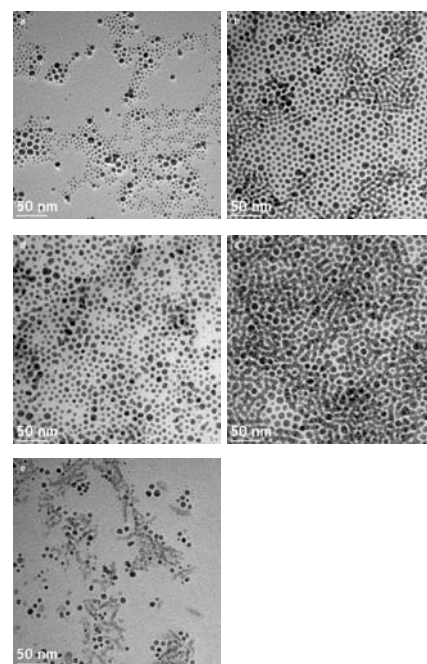


Fig. 3: TEM-micrographs (a-e) of particles synthesised at h.r. = 3, 5, 10, 20 and 40 K/min and the respective size and size distribution (Fig. 3 f)

Chemical	CAS	M [g/mol]	bp [$^{\circ}\text{C}$]	d [g/ml] at $25\text{ }^{\circ}\text{C}$
Diphenylether (DPE)	101-84-8	170.21	259	1.073
Dibenzylether (DBE)	103-50-4	198.26	298	1.043
Di-n-octylether (DOE)	629-82-3	242.45	287	0.806
1-Hexadecene (1-HD)	629-73-2	224.43	274	0.783
1-Octadecene (1-OD)	112-88-9	252.48	317	0.789
1-Eicosene (1-Eic)	3452-07-1	280.54	330	0.7916
Tri-N-octylamine (TNO)	1116-76-3	353.68	365 - 367	0.809

Table 1: Physical and chemical data of the solvents used in the particle synthesis.



Conclusions

We used two different wet chemical routes based on thermal decomposition and reduction to achieve crystalline silver nanoparticles and systematically changed the solvent, temperature, heating rate, and dwelling time to investigate their influence on particle size and size distribution. Most of the results fit well with the theory of nucleation burst, particle growth and Ostwald-ripening.

[1] J. Park, K. An, Y. Hwang, J-G. Park, H-J. Noh, J-Y. Kim, J-H. Park, N-M. Hwang, T. Hyeon, *Nature Mat.*, 3, 2004, pp. 891 - 895

[2] C. de Mello Donegá, P. Liljeroth, D. Vanmaekelbergh, *small*, 1(12), 2005, pp. 1152 – 1162

[3] E. E. Finney, R. G. Finke, *J. Coll. Inter. Sci.*, 317, 2008, pp. 351 – 374

[4] Y. Yin, A. P. Alivisatos, *Nature*, 437, 2005, pp. 664 – 670

[5] L. M. Bronstein, X. Huang, J. Retrum, A. Schmucker, M. Pink, B. D. Stein, B. Dragnea *Chem. Mater.*, 19(15), 2007, pp. 3624 - 3632

[6] A. V. Gaikwad, G. Rothenberg, *Phys. Chem. Chem. Phys.*, 8, 2006, pp. 3669 – 3675

[7] C. R. Bullen, P. Mulvaney, *Nano Lett.*, 4(12), 2004, pp. 2303 – 2307

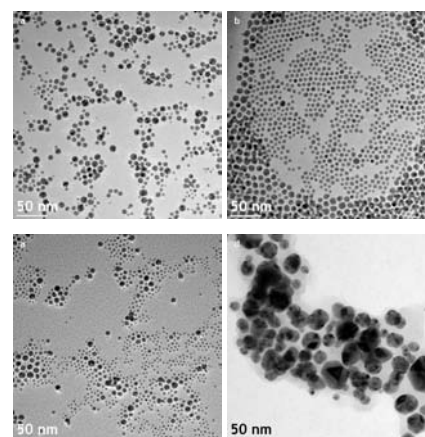


Fig. 4: TEM-micrographs (a-d) of particles synthesised at $t = 0, 30, 60$ and 720 min and the respective size and size distribution (Fig. 4 e)



Aktivitäten in Gremien



Activities in committees / Aktivitäten in Gremien

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Korrespondierendes Mitglied der Österreichischen Akademie der Wissenschaften

Editor von Progress in Materials Science

Mitglied, Scientific Advisory Board, CCMX – Competence Centre for Materials Science and Technology, Lausanne, Switzerland

Mitglied, Professorial Selection Committee des Institute of Science and Technology – Austria, Bundesministerium für Bildung, Wissenschaft und Kunst, Wien

Beirat von Max Planck Innovation GmbH, der Patentverwertungsgesellschaft der Max-Planck-Gesellschaft

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

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

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

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

Mitglied des Visiting Committee des Département des Matériaux an der Eidgenössischen Technischen Hochschule Lausanne (EPFL)

Mitglied des Editorial Boards/Advisory Boards: “MRS Bulletin of the Materials Research Society”, USA; “Materials Science and Technology”, London/UK; “Advanced Engineering Materials”, Weinheim, “International

Journal of Materials Research (Zeitschrift für Metallkunde)”, München; „Materials Science and Engineering C: Materials for Biological Applications“

Mitglied des Review Panel für das Los Alamos National Laboratory im Bereich Basis Energy Sciences, Los Alamos, New Mexico, U.S.A.

Gutachter für internationale Fachzeitschriften, u.a. “Acta Materialia”, “Scripta Materialia”, “Nanotechnology”, “Journal of Applied Physics”, “Applied Physics Letters”

Prof. Dr. Michael Veith

Sprecher des ersten Europäischen Graduiertenkollegs in Chemie (einschließlich: Physik, Materialwissenschaften, Medizinische Chemie), GRK 532, Universitäten von Metz, Nancy, Strasbourg, Luxembourg, Saarbrücken

Vorsitzender des Kompetenzzentrums Nanotechnologie cc-Nanochem e.V.

Vizepräsident des Frankreichzentrums der Universität des Saarlandes, Saarbrücken

Mitglied der Deutschen Akademie der Naturforscher Leopoldina, Halle

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Mitglied des Verwaltungsrates der EEIGM (INPL), Nancy/Frankreich

Mitglied des Komitees “Anorganisch-Technische Chemie” der DECHEMA, Frankfurt

Mitglied des Advisory Board der Zeitschrift Anorganische und Allgemeine Chemie (ZAAC)

Mitglied des Editorial Board des New Journal of Chemistry (NJC)

Mitglied des Editorial Board von Comptes Rendus

European Editor von Synthesis and Reactivity in Inorganic and Metal-Organic Chemistry

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

Korrespondierendes Mitglied der Akademie der Wissenschaften und der Literatur, Mainz

Mitglied der Expertengruppe Metropolprojekt Saarbrücken – Moselle Est

Advisory Board der Fa. Qiagen, Hilden

Beirat der Papiertechnischen Stiftung, München

Externes Beratungsmitglied der LCC Toulouse

Mitglied der Ecole Doctorale Metz- Nancy

Berater für die Universität Lyon

Bevollmächtigter der DFH für den Studentenaustausch mit Strasbourg (ECPM)

Prof. Dr. Sanjay Mathur

Mitglied der Inorganic Chemistry Division II of the IUPAC 2004-2009

Mitglied des Komitees “Materials Chemistry” of the IUPAC 2004-2007

Project Leader des IUPAC Projektes “Nanotechnology for Energy Production”

Mitglied des ECD Awards Komitees der American Ceramic Society

Mitglied des Editorial Board International Journal of Applied Ceramic Technology (ACT)

Mitglied des Editorial Board Ceramics International

Mitglied des Editorial Board International Journal of Nanotechnology

Associated Editor des Journal of Nanomaterials

Associated Editor des International Journal of Applied Ceramic Technology (ACT)

Organizer of Symposium 7, 31st International Cocoa Beach Conference & Exposition on Advanced Ceramics & Composites Daytona Beach, USA, January 21 – 26, 2007

Scientific committee member of the symposium Q “Protective Coatings and Thin Films 07”

Auszeichnungen

Abgeschlossene Dissertationen

Abgeschlossene Diplomarbeiten

Doktoranden

E-MRS Spring Meeting, Strasbourg, May 28-June 1, 2007

Co-Organizer of the Symposium Nanostructured Ceramic Materials: Science and Technology, MS&T'07 Materials Science & Technology COBO Center, Detroit, USA, September 16-20, 2007

Referee zahlreicher führender chemischer und materialwissenschaftlicher Fachzeitschriften.

Awards / Auszeichnungen

Sanjay Mathur and Eva Hemmer

Microporous ZrO₂ Film Preparation by Chemical Vapor Deposition, MS&T'07 Materials Science & Technology COBO Center, Detroit, USA

September 16-20, 2007

Der Posterbeitrag wurde ausgezeichnet mit dem Best Poster Award.

Completed PhD theses / Abgeschlossene Dissertationen

Castro, Mayra R. S.

Alternative conductive coatings based on multi-walled carbon nanotubes
Saarbrücken, Univ., Diss. (2007)

Dreier, Uwe

Entwicklung von Absorberwerkstoffen für die Lösungsmittelabsorption aus der Gasphase
Saarbrücken, Univ., Diss. (2007)

Hwang, Taejin

Silica/Polyaniline Composite Nanoparticle Produced in an Inverse Microemulsion Solution for the Electrochromic Applications
Saarbrücken, Univ., Diss. (2007)

Kuhn, Patrick

Abscheidung titan-sauerstoff-stickstoff-haltiger Schichten im Plasma CVD Prozess durch Implementierung einer metall-organischen Vorstufe
Saarbrücken, Univ., Diss. (2007)

Petersen, Christian

Mikro- und nanostrukturierte Schichten mittels Gasphasenabscheidung von [H₂AlOtBu]₂ für den Einsatz in der Nanobiotechnologie
Saarbrücken, Univ., Diss. (2007)

Rueff, Andreas

Herstellung und Ansteuerung elektrochromer Anzeigeelemente
Saarbrücken, Univ., Diss. (2007)

Completed diploma theses / Abgeschlossene Diplomarbeiten

Kondratiuk, Jens

Transparente kratzfeste Schichten mittels Plasma aktivierter chemischer Gasphasenabscheidung
Saarbrücken, Univ., Dipl. (2007)

Rabung, Benjamin

Untersuchung und Herstellung nanoskaliger Oxide durch ein neues elektrochemisches Verfahren München, Univ., Dipl. (2007)

Siegert, Benny

Neue Erkenntnisse zu Laserstrukturierung im System Wasserstoff-Aluminium-Sauerstoff
Saarbrücken, Univ., Dipl. (2007)

PhD students / Doktoranden

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Prof. Dr. S. Mathur

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Prof. Dr. M. Veith

Bubel, Carsten
Prof. Dr. M. Veith

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Hemmer, Eva
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Klook, Mark
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Prof. Dr. M. Veith

Rabung, Benjamin
Prof. Dr. M. Veith

Ren, Shuhua
Prof. Dr. M. Veith

Rügamer, Thomas
Prof. Dr. S. Mathur



Gastwissenschaftler Publikationen



Trenado, Carlos
Prof. Dr. Dr. D. J. Strauß

Ullah, Hameed
Prof. Dr. M. Veith

Uyanik, Mehmet
Prof. Dr. H. Schmidt

Xiao, Lisong
Prof. Dr. M. Veith

Yip, Siew Siew
Prof. Dr. H. Schmidt

Guest Scientists / Gastwissen- schaftler

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Dr. Al-Kahlout, Amal
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Dr. Castro, Mayra Rúbia Silva
Brasilien

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Mexiko

Dr. Egorov, Yuri
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Tschechische Republik

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Indien

Jehanathan, Neerushana
Indien

Kaushik, Ajay
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Dr. Lin, Hechun
China

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Senegal

Pan, Jun
China

Promnimit, Sujira
Thailand

Ren, Shuhua
China

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Spanien

Dr. Sam, Ebru Devrim
Türkei

Dr. Shanmugasundaram, Sakthivel
Indien

Dr. Shen, Hao
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Trenado, Carlos
Mexiko

Tsaneva, Gabriela
Bulgarien

Ullah, Hameed
Pakistan

Uyanik, Mehmet
Türkei

Wei, Jiandong
China

Xiao, Lisong
China

Dr. Yazdani-Assl, Omid
Iran

Dr. Yu, Eung Chul
Südkorea

Publications / Publikationen

Book chapter / Buchkapitel

D. J. Strauss, W. Delb and P. K. Plinkert
Identification of central auditory processing disorders by binaurally evoked brainstem responses
Handbook of Neural Engineering, Wiley-IEEE Press: 2007, pp 59-80

C. Trenado and D. J. Strauss
Magnetic nanoparticles for in vivo applications: A numerical modelling study

Mathematical modeling of biological systems. Cellular biophysics, regulatory networks, development, biomedicine, and data analysis, A. Deutsch, L. Brusch, H. Byrne, G. de Vries and H. Herzel Eds., Birkhäuser Boston: 2007, pp 275-280

M. Veith, M. Schubert and M. Kölbl
Nanotechnologie in der Diskussion und ihre regulatorischen Instrumente
Sicherheit der Nanotechnologie, N. e. V. Ed. Saarbrücken, 2007, Vol. 2, pp 70-93

Journal articles / Publikationen in Journalen

A. Al-Kahlout and M. A. Aegerter
Coloration mechanisms of sol-gel NiO-TiO₂ layers studied by EQCM
Sol Energ Mat Sol C 2007, 91, (4), 213-223 [02.321]

C. O. Avellaneda, D. F. Vieira, A. Al-Kahlout, E. R. Leite, A. Pawlicka and M. A. Aegerter
Solid-state electrochromic devices with Nb₂O₅: Mo thin film and gelatin-based electrolyte
Electrochim Acta 2007, 53, 1648-1654 [02.955]



- J. Bachmann, J. Jing, M. Knez, S. Barth, H. Shen, S. Mathur, U. Gösele and K. Nielsch
Ordered iron oxide nanotube arrays of controlled geometry and tunable magnetism by atomic layer deposition
J Am Chem Soc 2007, 129, (31), 9554-9555 [07.696]
- F. Barroso-Bujansa, J. L. G. Fierro and M. Veith
Grafting of poly(methyl vinyl ketone) onto aluminum surface
J Colloid Interf Sci 2007, 314, (1), 160-166 [02.233]
- M. R. S. Castro and H. K. Schmidt
Transparent conducting antimony-doped tin oxide films containing functionalized multi-walled carbon nanotubes
Phys Status Solidi A 2007, 204, (10), 3380-3386 [01.221]
- M. R. S. Castro, M. Veith and P. W. Oliveira
Electro-optical characterization of carbon nanotube-based coatings under CO₂ laser irradiation
Phys Status Solidi B 2007, 244, (11), 3998-4001 [00.967]
- S. Garcia Gomez de las Heras, N. Pütz, P. Mestres, M. Koch and U. Werner
Environmental scanning electron microscopy of the choroid plexus of the chick embryo
Microsc Microanal 2007, 13, (Supplement S03), 242-243 [02.108]
- R. Gerdes, P. Kalmes and C. Becker-Willinger
Investigation on thermal and chemical stability of polymer based easy-to-clean nanocomposite systems
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 77-86 [-]
- M. Hannig, L. Kriener, W. Hoth-Hannig, C. Becker-Willinger and H. K. Schmidt
Influence of nanocomposite surface coating on biofilm formation in Situ
J Nanosci Nanotechnol 2007, 7, (12), 4642-4648 [02.194]
- F. Hernandez-Ramirez, S. Barth, A. Tarancon, O. Casals, E. Pellicer, J. Rodriguez, A. Romano-Rodriguez, J. R. Morante and S. Mathur
Water vapor detection with individual tin oxide nanowires
Nanotechnology 2007, 18, 424016 [03.037]
- F. Hernandez-Ramirez, J. D. Prades, A. Tarancon, S. Barth, O. Casals, R. Jimenez-Diaz, E. Pellicer, J. Rodriguez, M. A. Juli, A. Romano-Rodriguez, J. R. Morante, S. Mathur, A. Helwig, J. Spannake and G. Mueller
Portable microensors based on individual SnO₂ nanowires
Nanotechnology 2007, 18, 495501 [03.037]
- F. Hernandez-Ramirez, A. Tarancon, O. Casals, E. Pellicer, J. Rodriguez, A. Romano-Rodriguez, J. R. Morante, S. Barth and S. Mathur
Electrical properties of individual tin oxide nanowires contacted to platinum electrodes
Phys Rev B 2007, 76, (8), 85429-1 - 85429-5 [03.107]
- S. Heusing
A contribution to climate protection - Electrochromic windows fabricated with the sol-gel technology
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 90-98 [-]
- T. Hwang, M. Quilitz and H. K. Schmidt
Comparison of the electrochromic properties of films made of silica/polyaniline composite nanoparticles and films made of pure polyaniline
J New Mat Elect Syst 2007, 10, (4), 237-242 [01.095]
- M. H. Jilavi, E. D. Sam, U. Werner, P. W. Oliveira and M. Veith
Microstructure investigation of reflective coatings interference multilayers produced by sol-gel method
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 72-76 [-]
- T. Krajewski
Quantitative spectrochemical analysis of Na₃AlF₆, ZrSiO₄ and InSb with the analytical electron microscope (TEM & SEM)
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 86-90 [-]
- W. Lu, M. Quilitz and H. K. Schmidt
Nanoscaled BaTiO₃ powders with a large surface area synthesized by precipitation from aqueous solutions: Preparation, characterization and sintering
J Eur Ceram Soc 2007, 27, (10), 3149-3159 [01.576]
- W. Lu and H. K. Schmidt
Synthesis of nanosized BaSnO₃ powders from metal isopropoxides
J Sol-Gel Sci Techn 2007, 42, (1), 55-64 [01.009]
- W. Lu and H. K. Schmidt
Preparation and characterization of BaSnO₃ powders by hydrothermal synthesis from tin oxide hydrate gel
J Mater Sci 2007, 42, (24), 10007-10013 [00.999]
- L. Ma, R. McMeeking and E. Arzt
Adhesive contact between flat punches with finite edge radius and an elastic half-space
Int J Mater Res 2007, 98, (11), 1156-1162 [-]
- S. Mathur and S. Barth
Molecule-based chemical vapor growth of aligned SnO₂ nanowires and branched SnO₂/V₂O₅ heterostructures
Small 2007, 3, (12), 2070-2075 [06.024]
- S. Mathur, R. Ganesan, I. Grobelsek, H. Shen, T. Rügamer and S. Barth
Plasma-assisted modulation of morphology and composition in tin oxide nanostructures for sensing applications
Adv Eng Mater 2007, 9, (8), 658-663 [01.402]
- S. Mathur, R. Ganesan, T. Rügamer, H. Shen and S. Barth

- Plasma-assisted modulation of morphology and composition in tin oxide nanostructures for sensing applications
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 99-104 [-]
- S. Mathur, T. Rügamer, H. Braunschweig and G. D'Andola
Investigations on chemical vapour deposition of magnesium-boron-containing metal-organic precursors
Z Anorg Allg Chem 2007, 633, (13-14), 2459-2462 [01.241]
- S. Mathur, T. Rügamer and I. Grobelsek
Phase-selective CVD of vanadium oxide nanostructures
Chem Vapor Depos 2007, 13, (1), 42-47 [01.679]
- S. Mathur, H. Shen and J. Altmayer
Nanostructured functional ceramic coatings by molecule-based chemical vapor deposition
Rev Adv Mater Sci 2007, 15, (1), 16-23 [01.019]
- S. Mathur, H. Shen, N. Donia, T. Rügamer, V. Sivakov and U. Werner
One-step chemical vapor growth of $\text{Ge/SiC}_x\text{N}_y$ nanocables
J Am Chem Soc 2007, 129, (31), 9746-9752 [07.696]
- A. L. Ndiaye, F. Guyon, M. Knorr, V. Huch and M. Veith
Modification of the hydrogen bonds network in a hydroxyl functionalized dithiolene ligand by HgX_2 complexation
Z Anorg Allg Chem 2007, 633, 1959-1963 [01.241]
- C. Petersen, A. Lasagni, C. Holzapfel, C. Daniel, F. Mücklich and M. Veith
SEM/TEM characterization of periodical novel amorphous/nano-crystalline micro-composites obtained by laser interference structuring: The system $\text{HAIO-Al-Al}_2\text{O}_3$
Appl Surf Sci 2007, 253, (19), 8022-8027 [01.436]
- C. Piana, I. Güll, S. Gerbes, R. Gerdes, C. Mills, J. Samitier, M. Wirth and F. Gabor
Influence of surface modification on vitality and differentiation of Caco-2 cells
Differentiation 2007, 75, (4), 308-317 [03.745]
- M. Praetorius, C. Brunner, B. Lehnert, C. Klingmann, H. K. Schmidt, H. Staecker and B. Schick
Transsynaptic delivery of nanoparticles to the central auditory nervous system
Acta Oto-Laryngol 2007, 127, (5), 486-490 [00.738]
- J. Pütz
Flexibel und druckbar. Transparente leitfähige Oxide als Additive
Metalloberfläche : MO 2007, 61, (4), 38-40 [-]
- M. Schem, T. Schmidt, H. Caparrotti, M. Wittmar and M. Veith
Corrosion inhibiting cerium compounds for chromium-free corrosion protective coatings on AA 2024
Annual Report, Jahresbericht 2006 - INM Leibniz-Institut für Neue Materialien 2007, 65-72 [-]
- T. Schmidt, M. Mennig and H. K. Schmidt
New method for the preparation and stabilization of nanoparticulate t-ZrO₂ by a combined sol-gel and solvothermal process
J Am Ceram Soc 2007, 90, (5), 1401-1405 [01.396]
- T. Schmidt, P. W. Oliveira, M. Mennig and H. K. Schmidt
Preparation of optical axial GRIN components through migration of charged amorphous ZrO₂ nanoparticles inside an organic-inorganic hybrid matrix by electrophoresis
J Non-Cryst Solids 2007, 353, (30-31), 2826-2831 [01.362]
- V. Schönitzer and I. M. Weiss
The structure of mollusc larval shells formed in the presence of the chitin synthase inhibitor Nikkomycin Z
BMC Struct Biol 2007, (Art. No. 71), [01.975]
- G. Schultes, M. Schmidt, M. Truar, D. Goettel, O. Freitag-Weber and U. Werner
Co-deposition of silver nanoclusters and sputtered alumina for sensor devices
Thin Solid Films 2007, 515, (20-21), 7790-7797 [01.666]
- F. L. Souza, M. A. Aegerter and E. R. Leite
Performance of a single-phase hybrid and nanocomposite polyelectrolyte in classical electrochromic devices
Electrochim Acta 2007, 53, 1635-1642 [02.955]
- F. L. Souza, M. A. Aegerter and E. R. Leite
Solid hybrid polyelectrolyte with high performance in electrochromic devices: Electrochemical stability and optical study
Sol Energy Mat Sol C 2007, 91, (19), 1825-1830 [02.321]
- D. Sun, S. Heusing and M. A. Aegerter
Li⁺ ion exchange in CeO₂-TiO₂ sol-gel layers studied by electrochemical quartz crystal microbalance
Sol Energy Mat Sol C 2007, 91, (12), 1037-1050 [02.321]
- M. Veith
Nils Wiberg (1934-2007)
Nachr Chem 2007, 55, (7-8), 781 [00.161]
- M. Veith
Glanzlichter chemischer Experimentierkunst
Angew Chem 2007, 119, (1-2), 1025 [-]
- M. Veith, M. Gasthauer, M. Zimmer and V. Huch
Zur Bildung einer Zinn-Zinn-Bindung in Bis(hexamethyldisilazyl)-Zinn(II) unter C-H Bindungsbruch
Z Anorg Allg Chem 2007, 633, (13-14), 2274-2277 [01.241]
- M. Veith, H. Hreleva-Caparrotti and V. Huch
Two new metal derivatives of the aluminosiloxane $[\text{Ph}_2\text{SiO}]_8[\text{AlO}(\text{OH})]_4$; $[\text{Ph}_2\text{SiO}]_8[\text{AlO}_2(\text{Na})]_4 \cdot 5(\text{THF})$ and $[\text{Ph}_2\text{SiO}]_8[\text{AlO}(\text{OH})]_2[\text{AlO}_2]_2[\text{Zn}(\text{OH})]_2 \cdot 2(\text{OEt}_2)$



- J Organomet Chem 2007, 692, (13), 2784-2788 [02.332]
- M. Veith and M. Kölbl
Potenzial für die Oberflächentechnik. Nanotechnologie in der Praxis
JOT - Journal für Oberflächentechnik 2007, 47, (7), 16-19 [-]
- M. Veith, A. Rammo, V. Huch and J. Biegler
The reaction behaviour of the polycyclic oligoalumosiloxane $[\text{Ph}_2\text{SiO}]_8[\text{AlO}(\text{OH})_4]$ towards hexamethyldisilazane
Z Anorg Allg Chem 2007, 633, (2), 246-250 [01.241]
- M. Veith, M. Schubert and M. Kölbl
Regulatorische Instrumente für die Nanotechnologie: Weltweite Suche in vollem Gange
CLB Chemie in Labor und Biotechnik 2007, 58, (7), 261-267 [-]
- N. Wiberg, S. K. Vasisht, G. Fischer, P. Mayer, V. Huch and M. Veith
Reactivity of the unusually structured silicon cluster compound $\text{Si-8}(\text{SiBu}_3)_6$
Z Anorg Allg Chem 2007, 633, (13-14), 2425-2430 [01.241]
- M. Wittmar
„Multiprotect“: Gemeinsam gegen den Rost. Umweltfreundlicher Korrosionsschutz als europäisches Forschungsprojekt
Besser Lackieren 2007, (7), [-]
- Proceedings / Konferenzbeiträge**
- J. Adam, M. Aslan, R. Drumm and M. Veith
Mechanisms of bonding effected by nanoparticles in Zirconia coatings applied by spraying of suspensions
In: 10th International Conference and Exhibition of the European Ceramic Society (ECerS), June 17-21, 2007, Berlin, J. G. Heinrich and C. Aneziris, Eds., Götter Verlag, (2007), pp 307-311
- M. Aslan, J. Adam, B. Reinhard and M. Veith
Preparation of thick zirconia coatings by spraying of colloidal suspensions
In: 10th International Conference and Exhibition of the European Ceramic Society (ECerS), June 17-21, 2007, Berlin, J. G. Heinrich and C. Aneziris, Eds., Götter Verlag, (2007), pp 322-327
- 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
In: 10th International Conference and Exhibition of the European Ceramic Society (ECerS), June 17-21, 2007, Berlin, J. G. Heinrich and C. Aneziris, Eds., Götter Verlag, (2007), pp 848-853
- F. I. Corona-Strauss, W. Delb, M. Bloching, S. Hussain and D. J. Strauss
The johor screening scheme: is an area-wide newborn hearing screening possible in Malaysia?
In: Intelligent Information Hiding and Multimedia Signal Processing. Proceedings of the Third International Conference of the IEEE IHHMSP, November 26-28, 2007, Kaohsiung City <Taiwan>, (2007),
- F. I. Corona-Strauss, W. Delb, M. Bloching and D. J. Strauss
Ultra-fast quantification of hearing loss by neural synchronization stabilities of auditory evoked brainstem activity
In: Proceedings of the 29th Annual International Conference of the IEEE EMBS, August 23-26, 2007, Lyon <France>, (2007), pp 2476-2479
- F. I. Corona-Strauss, W. Delb, D. J. Hecker and D. J. Strauss
Ultra-fast detection of hearing thresholds by single sweeps of auditory brainstem responses: A new novelty detection paradigm
In: Proceedings of the Third International IEEE EMBS Conference on Neural Engineering, May 02-05, 2007, Kohala Coast <Hawaii, USA>, (2007), pp 638-641
- 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, (2007), p 120
- 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, (2007), p 109
- A. R. Harris, K. Schwerdtfeger, M.-A. Luszpinski, G. Sandvoss and D. J. Strauss
Adaptive time-scale feature extraction in electroencephalographic responses to transcranial magnetic stimulation
In: Proceedings of the 29th Annual International Conference of the IEEE EMBS, August 23-26, 2007, Lyon <France>, (2007), pp 2835-2838
- A. R. Harris and D. J. Strauss
Adaptive frame decomposition in slow wave cortical responses to transcranial magnetic stimulation
In: Proceedings of Third International Conference of the IEEE IHHMSP, Kaohsiung City <Taiwan>, (2007), pp 201-204
- Y. F. Low, F. I. Corona-Strauss, P. Adam and D. J. Strauss
Extraction of auditory attention correlates in single sweeps of cortical potentials by maximum entropy paradigms and its application
In: Proceedings of the Third International IEEE EMBS Conference on Neural Engineering, May 2-5, 2007, Kohala Coast <Hawaii, USA>, (2007), pp 469-472

- Y. F. Low, C. Trenado, W. Delb, F. I. Corona-Strauss and D. J. Strauss
The role of attention in the tinnitus decompensation: Reinforcement of a large-scale neural decompensation measure
In: Proceedings of the 29th Annual International Conference of the IEEE EMBS, August 23-26, 2007, Lyon <France>, (2007), pp 2485-2488
- S. Mathur and N. Donia
Nanostructured materials and coatings through chemistry: From molecules to product applications
In: Nanotechnology in industrial applications : EuroNanoForum 2007, June 19-21, 2007, Düsseldorf, (2007), pp 105-109
- S. Mathur, E. Hemmer, S. Barth, J. Altmayer and N. Donia
Microporous ZrO₂ membrane preparation by liquid-injection MOCVD
In: Proceedings of 31st Cocoa Beach Conference, (2007), pp 165-173
- B. Reinhard, A. Altherr, C. Trenado, D. S. Strauss and M. Veith
Chemische Nanotechnologie im Brandschutz
In: Nasschemische nanokomposite Beschichtungen. OTTI-Fachforum, February 7-8, 2007, Regensburg, OTTI, (2007),
- A. Romano-Rodriguez, F. Hernández-Ramírez, A. Tarancon, O. Casals, J. D. Prades, J. R. Morante, S. Barth and P. T. Mather
Fabrication strategies and electrical characterization of nanodevices contacted using focused ion beam techniques
In: Nanostructured Ceramic Materials: Science and Technology. MS&T07, September 16-20, 2007, Detroit <Mich., USA>, Acers, (2007),
- C. Trenado, L. Haab and D. J. Strauss
Modeling neural correlates of auditory attention in evoked potentials using corticothalamic feedback dynamics
In: Proceedings of the 29th Annual International Conference of the IEEE EMBS, August 23-26, 2007, Lyon <France>, (2007), pp 4281-4284
- C. Trenado and D. J. Strauss
A markovian-type approach for stream selection in auditory selective attention
In: Proceedings of the Dagstuhl seminar 07461 "Numerical Methods for Structured Markov Chains", November 11-14, 2007, Wadern, (2007),
- C. Trenado and D. J. Strauss
Multiscale multiphysics modeling
In: Proceeding of Invention 2007: Innovation in Electric Power Plants, October 25-26, 2007, Ustron <Poland>, (2007),
- C. Trenado, D. J. Strauss, B. Reinhard, A. Altherr, T. Smuc and A. Maksimovic
Modeling of fire protective materials
In: Proceedings of the Fifth Vienna Symposium on Mathematical Modelling (Mathmod), February 8-10, 2006, Wien, (2006), p 355
- M. Veith
nanoDe 2006: Branchendialog Bauwesen
In: nanoDE 2006. Strategien für Produkte von morgen. 3. BMBF-Nanotechnologietage, November 06-07, 2006, Berlin, Bundesministerium für Bildung und Forschung, (2006), pp 147-149
- M. Veith, M. Quilitz and M. Schubert
Chemical nanotechnology for industrial coatings
In: European Coatings Daily, May 8-10, 2007, Nürnberg, Vincentz, (2007), p 6
- Publications in magazines / Magazin-artikel**
- A. Altherr
INM entwickelt innovative Beschichtungen für die Automobilindustrie
OEM & Lieferant automotive.Saarland. Messeausgabe zur IAA 2007 2007
- H. Shen
NanoFutur: Design und Modifikation von Metalloxid-Nanodrähten - MONOGAS Nanospotlight 2007, 4
- Posters / Poster**
- M. R. S. Castro, M. Veith and P. W. Oliveira
Electrical properties of titania/multi-walled carbon nanotube nanocomposites irradiated with CO₂ laser
XXI International Winterschool on Electronic Properties of Novel Materials - IWEPM, March 10-17, 2007, Kirchberg <Austria>
- S. Garcia Gomez de las Heras, N. Pütz, M. Koch, U. Werner and P. Mestres
Environmental scanning electron microscopy of the choroid plexus of the chick embryo
Microscopy Conference 2007. 33rd Conference of the DGE, Deutsche Gesellschaft für Elektronenmikroskopie e. V., September 2-7, 2007, Saarbrücken
- S. Gerbes, O. C. Aktas, M. Müller, M. Koch, S. Kiefer, B. Schäfer and P. W. Oliveira
Development of biocompatible nanostructured surfaces
Nano2Life-Kongress, March 19-21, 2007, Saarbrücken
- J. Gerwann, S. Brück, K. Endres, S. Gerbes, M. Hannig, M. Hoffmann, N. Moszner, D. Poppe, U. Salz and H. Schmidt
ZOVAN-ahnoberflächenversiegelung mit einer antiadhäsiven Nanokompositbeschichtung
3. WING-Konferenz, October 22-24, 2007, Berlin
- E. Hemmer and S. Mathur
Homo- und heteroleptische Lanthanoidverbindungen für die Herstellung Ln-haltiger Nanostrukturen
DFG Kolloquium, November 28-29, 2007, Bonn-Röttgen



E. Hemmer, T. Noisser and S. Mathur
Homo- and heterometallic alkoxides as precursors to functional materials
iNANOSCHOOL. PhD Summer School, June 10-15, 2007, Ebeltoft <Denmark>

A. Kraegeloh, M. Koch, S. Gerbes and M. Veith
Investigations on the microbicidal effectiveness of silver based nanomaterials
Nano2Life-Kongress, March 19-21, 2007, Saarbrücken

T. Krajewski, M. Naumann, K. Moh, T. S. Müller, M. Akarsu and H. Schmidt
Morphologic-structural characterization of nano-scaled TiO₂ and TiO₂ doped photocatalytic organic-inorganic gradient layers on glass by analytical electron microscopy
14. Tagung Festkörperanalytik, July 16-18, 2007, Wien

S. Mathur, J. Altmayer, S. Barth and M. Müller
Nanostructured metal oxide surfaces for bio-medical applications: Modulation of surface properties through chemical precursor design
BioStar 2006 2nd International Congress on Regenerative Biology and ICBN 2006 2nd International Congress on Bio-Nano-Interface. In: Tissue Engineering, October 9-11, 2006, Stuttgart pp 906-906

S. Mathur, C. Cavalius, C.-M. Lehr, N. Daum, H. Thielecke and H. Büth
Cellular binding, translocation and differentiation as key responses to surface modified metal oxide nanoparticles
DFG Kolloquium, September 5, 2007

S. Mathur and E. Hemmer
Microporous ZrO₂ film preparation by chemical vapor deposition
MS&T'07 Materials Science & Technology, September 16-20, 2007, Detroit <Mich., USA>

M. Veith, S. Gerbes, C. Petersen, W. Metzger, S. E. Motsch, M. Oberinger, R. Gerdes, P. W. Oliveira, S. Kiefer and B. Schäfer

Structured surfaces for cell differentiation
BioStar 2006 2nd International Congress on Regenerative Biology and ICBN 2006 2nd International Congress on Bio-Nano-Interface. In: Tissue Engineering, October 9-11, 2006, Stuttgart pp 906-907

Talks / Vorträge

J. Adam
Surface modification of nanoparticles
MULTIPROTECT Training Course „Nanoparticles preparation for coatings technology“; February 15-17, 2007; Szczecin <Poland>

J. Adam
Verschiedene chemische Verfahren zur Herstellung von Nanopartikeln: Methoden zur Oberflächenmodifikation und Dispergierung
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken

J. Adam
Verschiedene chemische Verfahren zur Herstellung von Nanopartikeln: Top-down: Nassmahlverfahren
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken

J. Adam, M. Aslan, R. Drumm and M. Veith
Mechanisms of bonding effected by nanoparticles in Zirconia coatings applied by spraying of suspensions
10th International Conference and Exhibition of the European Ceramic Society (ECerS); June 17-21, 2007; Berlin

J. Adam, W. Lu, R. Drumm and M. Schem
Synthesis of nanoparticles for Multiprotect [CeP_{0.4}, Ce₂(M₀O₄)₃]

MULTIPROTECT Training Course „Nanoparticles preparation for coatings technology“; February 15-17, 2007; Szczecin <Poland>

E. Arzt
Mechanics of adhesion in biological and artificial systems
43. Meeting of the German Colloid Society; October 8-10. 2007; Mainz
Eingeladener Vortrag

E. Arzt
Haftung durch Mikrostrukturierung: Auf dem Weg zu smarten Oberflächen
Volkswagen AG, AutoUni Wolfsburg; November 21, 2007; Wolfsburg
Eingeladener Vortrag

M. Aslan, J. Adam, B. Reinhard and M. Veith
Preparation of thick zirconia coatings by spraying of colloidal suspensions
10th International Conference and Exhibition of the European Ceramic Society (ECerS); June 17-21, 2007; Berlin

C. Becker-Willinger
Funktionelle Beschichtungen - Neue Materialien mit maßgeschneiderten Eigenschaften
Innovationen durch Nanotechnologie: Mehrwert für Zulieferer der Automobilindustrie; June 5, 2007; Köln

C. Becker-Willinger and S. Schmitz-Stöwe
Verarbeitung von Nanopartikeln: Dispersion - Folien und Schichten - Einarbeitung in Kunststoffen
GDCh-Kurs „Chemische Nanotechnologie“; June 26, 2007; Saarbrücken

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
10th International Conference and Exhibition of the European Ceramic Society (ECerS); June 17-21, 2007; Berlin

M. R. S. Castro
Multi-walled carbon nanotube-based conductive coatings

- Leibniz-Institut für Neue Materialien; April 4, 2007; Saarbrücken
- M. R. S. Castro
Exploring potential applications of TiO₂/multi-walled carbon nanotube nanocomposites
NanoteC'07 - International Conference on carbon nanoscience and nanotechnology; September 1, 2007; Brighton <UK>
- F. I. Corona-Strauss, W. Delb and D. J. Strauss
Fast detection of ABR-responses using neural synchronization stability and single sweep analysis
8th EFAS Congress, 10th Congress of the German Society of Audiology; June 06-09, 2007; Heidelberg
- W. Delb, Y. F. Low, F. I. Corona-Strauss and D. J. Strauss
Auditory attention and tinnitus: the objective determination of tinnitus decompensation
8th EFAS Congress, 10th Congress of the German Society of Audiology; June 06-09, 2007; Heidelberg
- N. Donia
Chemische Gasphasenreaktionen
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken
- N. Donia and S. Mathur
Nanostrukturierte Oberflächen durch Gasphasenabscheidung
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- R. Drumm
Charakterisierungsmethoden für Nanopartikel, Teil 1
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken
- S. Gerbes and M. Veith
Developing of nanoscaled materials for life science applications
- Internationale Fachmesse für Feinwerktechnik, Ultrapräzision, Micro- und Nano-Technologien (MiNaT-Kongress); June 12-14, 2007; Stuttgart
- S. Gerbes and M. Veith
Developing of nanoscaled materials for life science applications
German-Korean Symposium: Interdisciplinary Research Symposium on BioMEMS, BioMedical, Nano-, System Technology; November 08, 2007; Saarbrücken
- M. Gros and P. W. Oliveira
Mikrostrukturierung durch Prägen von nanopartikelmodifizierten Systemen
Nanomaterialien: Kleinste Teilchen - Großer Nutzen. Aktuelle Entwicklungen für die tägliche Praxis; July 19, 2007; Bayreuth
- S. Heusing
Elektrochromie. Grundlagen und Anwendungen
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- A. Kraegeloh and M. Koch
Nanosilber und Mikroorganismen: Wirkung und Wechselwirkung
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- T. Kraus
Assembly and printing of micro and nano objects
Universität Basel, Fakultät Physik; November 19, 2007; Basel
Eingeladener Vortrag
- S. Mathur
Chemically designed nanoparticles and nanowires: Controlled growth, applications and devices
211th ECS Meeting. A2 Tutorials in Nanotechnology: Focus on High Temperature Materials; May 6-10, 2007; Chicago <Ill., USA>
- S. Mathur
Nanostructures and thin film systems
- Workshop INM/ISC; January 9, 2007; Bronnbach
- S. Mathur
Chemische Nanotechnologie: Vom Molekül zur Anwendung
Universität zu Köln. Institut für Anorganische Chemie; January 16, 2007; Köln
Eingeladener Vortrag
- S. Mathur
One dimensional semiconductor nanostructures
31st International Cocoa Beach Conference & Exposition on Advanced Ceramics & Composites; January 21-26, 2007; Daytona Beach <Florida, USA>
- S. Mathur
Synthesis of lanthanide containing nanomaterials by chemical vapor deposition and hydrothermal processes using new lanthanide precursors
31st International Cocoa Beach Conference & Exposition on Advanced Ceramics & Composites; January 21-26, 2007; Daytona Beach <Florida, USA>
Eingeladener Vortrag
- S. Mathur
Chemische Nanotechnologie: Vom Molekül zur Anwendung
GDCh-Kolloquium; February 6, 2007; Kassel
Eingeladener Vortrag
- S. Mathur
Chemically processed nanomaterials
University of Genoa; February 19, 2007; Genoa <Italy>
Eingeladener Vortrag
- S. Mathur
Chemical nanotechnology: Opportunities and applications
EuroIndiaNet; March 3, 2007; New Delhi <India>
Eingeladener Vortrag
- S. Mathur
Molecular-based synthesis of nanomaterials



- Indian Institute of Technology; March 6, 2007; New Delhi <India>
Eingeladener Vortrag
- S. Mathur
Chemically designed nanomaterials: Controlled growth, applications and devices
Nano and Giga Challenges in Microelectronics; March 13, 2007; Phoenix <Arizona, USA>
Tutorial Lecture
- S. Mathur
Chemically designed nanoparticles and nanowires: Controlled growth, application and devices
MRS Spring 2007; April 13, 2007; San Francisco <Calif., USA>
adWorkshop of the Belgian IAP project P6/16. Advances complex inorganic materials by a novel bottom-up (nano)chemistry approach: processing and shaping; November 29, 2007; Gent <Belgium>
Eingeladener Vortrag/Keynote Lecturer
- M. Quilitz
Von den Grundlagen bis zum industriellen Up-Scaling - das INM in Saarbrücken
Forum Science2Business, Nanosolutions 2007; November 22, 2007; Frankfurt am Main
- M. Quilitz
Nanomaterials Through Chemistry: Controlled Growth, Application and Devices
2007 International Conference & Exhibition on Nanotechnology and Advanced Materials; December 13, 2007; Hong Kong
Eingeladener Vortrag
- M. Quilitz, P. W. Oliveira and C. Becker-Willinger
Neue Oberflächeneigenschaften über Nanopartikel
1. Materialica Kongress „Innovative und nachhaltige Oberflächeneigenschaften“; October 18, 2007; München
Keynote Lecturer
- B. Reinhard, A. Altherr, C. Trenado, D. S. Strauss and M. Veith
Chemische Nanotechnologie im Brandschutz
Nasschemische nanokomposite Beschichtungen. OTTI-Fachforum; February 7-8, 2007; Regensburg
- M. Schem and J. Adam
Nanoparticle production in general
MULTIPROTECT Training Course „Nanoparticles preparation for coatings technology“; February 15-17, 2007; Szczecin <Poland>
- K.-P. Schmitt
Pulversynthese und kontrollierte Fällung
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- K.-P. Schmitt
Verschiedene chemische Verfahren zur Herstellung von Nanopartikeln: Bottom-up: Fällungsprozesse, Emulsionsprozesse etc.
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken
- S. Schmitz-Stöwe
Nanomere
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- S. Schmitz-Stöwe
Funktionelle Beschichtungen - Neue Materialien mit maßgeschneiderten Eigenschaften
Innovationen durch Nanotechnologie: Neue Materialien für Architektur, Möbel und Design. Materials ConneXion; September 20, 2007; Köln
- M. Schubert
Nanotechnologie und Schule
Lehrer-Workshop „Industrie trifft Schule“. Lehrerakademie für Fortbildung und Personalentwicklung an Schulen in Baden-Württemberg; March 15, 2007; Esslingen
- M. Schubert
Was ist Nanotechnologie? Grundlagen und Anwendungsbereiche
Innovationen durch Nanotechnologie: Mehrwert für Zulieferer der Automobilindustrie; June 5, 2007; Köln
- M. Schubert
Mit Nanotechnologie zu neuen Oberflächen
AK Neue Materialien in der Messewirtschaft. Faircycle-Netzwerk;
August 15, 2007; Kassel
- M. Schubert
NanoScience in der Schule Moderne Physik in Sek I und Sek II
EduNetwork 07;
September 15, 2007; Berlin
- M. Schubert
Was ist Nanotechnologie? Grundlagen und Anwendungsbereiche
Innovationen durch Nanotechnologie: Neue Materialien für Architektur, Möbel und Design. Materials ConneXion; September 20, 2007; Köln
- H. Shen
Design and modification of single and ordered metal oxide nanowires for optical and gas sensing applications
2. network meeting of BMBF-NanoFutur;
June 14-15, 2007; Maria Laach
- M. Veith
Chemische Nanotechnologie - Was ist das?
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- M. Veith
Vom Molekül zum Material: Neue Erkenntnisse beim Stoffsystem Aluminium/Sauerstoff/Wasserstoff
GDCh-Kolloquium des Ortsverbandes Hannover; January 25, 2007; Hannover
- M. Veith
Nanotechnology with respect to protecting surfaces
Nanotechnology - Products and Processes for Environmental Benefit. Royal Society London; May 16-17, 2007; London <UK>



Vorlesungen



Lectures / Vorlesungen

- M. Veith
Structuring of surfaces
Internationale Fachmesse für Feinwerktechnik, Ultrapräzision, Micro- und Nanotechnologien (MiNaT-Kongress); June 12-14, 2007; Stuttgart
Keynote Lecturer
- M. Veith
Selbstorganisation bei Molekülen und Feststoffen
Plenum der Akademie der Wissenschaften und der Literatur; June 22, 2007; Mainz
- M. Veith
New synthetic routes to nano-composites with ceramic particles, using lanthanide compounds
XIV International Sol-Gel Conference; September 2-7, 2007; Montpellier <France>
Eingeladener Vortrag
- M. Veith
Nano-Schichten
Dünne Schichten mit großer Wirkung in der Nanotechnologie. Fachtagung NanoSurface 2007; September 19, 2007; Gießen
- M. Veith
Nano-materials by chemical single source precursor techniques
Kolloquium der Technischen Fakultät der Christian-Albrechts-Universität zu Kiel; November 19, 2007; Kiel
- M. Veith
Vom Molekül zum Material
GDCh-Vortrag Darmstadt; December 4, 2007; Darmstadt
- M. Veith, M. Ehses, A. Laurent and M. Gasthauer
Ge, Sn and Pb in rings and cages, and in solid materials: From molecules to materials
International Conference on the Coordination and Organometallic Chemistry of Germanium, Tin and Lead. ICCOC-GTL-12; July 9-13, 2007; Galway <Ireland>
- M. Veith and M. Quilitz
Grundlagen der Herstellung und Oberflächenmodifikation von Nanopartikeln über chemische Verfahren
GDCh-Kurs „Chemische Nanotechnologie“; June 26, 2007; Saarbrücken
- M. Veith, M. Wittmar, M. Quilitz and M. Schubert
Nanotechnology with respect to protecting surfaces
Nanotechnology - Products and Processes for Environmental Benefit; July 11, 2007; London <UK>
Eingeladener Vortrag
- U. Werner
Elektronenmikroskopie - Abbildung der inneren und der Oberflächen-Struktur
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken
- U. Werner
Untersuchung von Nanopartikeln mittels Transmissions-Elektronenmikroskopie
GDCh Fortbildungskurs „Chemische Nanotechnologien“; June 26-27, 2007; Saarbrücken
- M. Wittmar
Dünne Multifunktionsbeschichtungen für Glas, Metalle und Kunststoffe
MST Aerospace Kooperationsforum; November 27, 2007; Köln
- S. Mathur
Anorganische und Analytische Chemie
Julius-Maximilians-Universität Würzburg, (WS 06/07)
- D. J. Strauss
Programmierung
(WS 06/07)
- D. J. Strauss
Audiologie
(WS 06/07)
- D. J. Strauss
Neuronale Signalanalyse und Modellierung
(WS 06/07)
- D. J. Strauss
Grundlagen der Medizinischen Messtechnik
(WS 06/07)
- D. J. Strauss
Einführung in die Biosignalverarbeitung
(SS 07)
- D. J. Strauss
Einführung in das Neural Engineering I
(SS 07)
- D. J. Strauss
Neural and Cognitive Systems
(SS 07)
- D. J. Strauss
Biomedizinische Signal- und Bildverarbeitung
(SS 07)
- M. Veith
Allgemeine Chemie und Grundlagen der Hauptgruppenchemie
(WS 06/07)
- M. Veith
Metallorganische Chemie
(SS 07)
- M. Veith
Molekülchemie der Hauptgruppenelemente
(SS 07)
- M. Veith
Materialien aus molekularen Vorstufen
(SS 07)



Praktika

Weitere Lehrveranstaltungen

Patente



M. Veith et al.
Spezielle Kapitel der Anorganischen Chemie
(WS 06/07)

M. Veith
Allgemeine Chemie für Pharmazeuten
(WS 06/07)

Practical courses / Praktika

S. Mathur
Übungen im Vortragen und Demonstrieren
für Studenten des Lehramts an Gymnasien
Julius-Maximilians-Universität Würzburg

S. Mathur
Übungen im Vortragen und Demonstrieren
für Studenten des Lehramts an Grund-,
Haupt-, und Realschulen
Julius-Maximilians-Universität Würzburg

S. Mathur
Anorganisch-Chemisches Fortgeschrittenen-
praktikum für Studenten des Lehramts an
Gymnasien
Julius-Maximilians-Universität Würzburg

M. Veith et al.
Fortgeschrittenenpraktikum "Nichtmetalle I"
(WS 06/07)

M. Veith et al
Anorganisch-Chemisches
Praktikum für Fortgeschrittene
(SS 07)

M. Veith et al
Vertiefungspraktikum Anorganische Chemie
(SS 07)

M. Veith et al
Fortgeschrittenenpraktikum für Lehramtsstu-
dierende
(WS 06/07, SS 07)

M. Veith et al
Chemisches Praktikum für Studierende der
Physik, Werkstoffwissenschaften, der Biologie
und der Metalltechnik
(WS 06/07, SS 07)

M. Veith et al
Chemisches Grundpraktikum Einführungs-
praktikum
Allgemeine und Anorganische Chemie
(SS 07)

M. Veith et al
Schwerpunktpraktikum Anorganische Chemie
(WS 06/07)

Other courses / Weitere Lehrver- anstaltungen

M. Schubert
Innovationen durch Nanotechnologie:
Mehrwert für Zulieferer der Automobilindu-
strie
05.06.2007, Köln

M. Schubert
Innovationen durch Nanotechnologie:
Neue Materialien für Architektur, Möbel
und Design
20.09.2007, Köln

M. Schubert
Summer School Chemische Nanotechnologie
08. – 12.10.2007, Saarbrücken

M. Veith et al.
GDCh-Kurs "Chemische Nanotechnologie"
26. – 27.06.2007, Saarbrücken

M. Veith et al.
Fachdidaktik (WS 06/07)

M. Veith et al.
Fachdidaktisches Seminar für Lehramtsstu-
dierende
(SS 07)

M. Veith et al
Seminar zum Schwerpunktpraktikum
Anorganische Chemie (WS 06/07)

Patents / Patente

Im Jahr 2007 wurden sechs neue Patentan-
meldungen hinterlegt, die noch nicht offen-
gelegt sind. Es wurden 18 Patente erteilt,
davon fünf innerhalb von Europa und drei-
zehn auf internationaler Ebene. Somit wur-
den insgesamt ca. 145 Patentanmeldungen
durch das Leibniz-Institut für Neue Materi-
alien vorgenommen. Rund die Hälfte dieser
Schutzrechte ist erteilt.

In 2007, INM has filed six new patent ap-
plications which are not yet published and
18 patents have been granted. Five of these
patents are granted in Europe and thirteen
in foreign countries. INM applied around
145 patent families in total and around 50
percent of them are granted until today.

Erteilte europäische Patente:

DE 10164904 B4
Titel: „Verfahren zur Herstellung eines
Kern-Hülle-Teilchens, wobei der Kern ein
nanoskaliges Teilchen ist und die Verwen-
dung des Teilchens“
Erfinder: R. Nonninger

DE 195 44 763 B4
Titel: „Verwendung einer fluorhaltige anor-
ganische Polykondensate enthaltenden Be-
schichtungszusammensetzung zum Schutz
vor Graffiti“
Erfinder: S. Brück, V. Gerhard, G. Jonsch-
ker, R. Kasemann, M. Mennig, H. Schmidt

EP 1299191 B1
Titel: „Metallische Nickelhydrierkatalysa-
toren, deren Herstellung und deren Verwen-
dung“
Erfinder: : D. Vanoppen, M. Veith, V. Kroum

EP 1687244 B1
Titel: „Isolationsmaterial“
Erfinder: K. Endres, S. Goedicke, B. Rein-
hard, H. Schmidt



Kooperationen



EP 1633822 B1

Titel: „Antiadhäsive Hochtemperaturschichten“

Erfinder: M. Aslan, R. Drumm, K. Endres, H. Nair, B. Reinhard, H. Schmidt

Erteilte internationale Patente:

Chinesisches Patent Nr. ZL 98804996.1

Stammanmeldungstitel: „Nanokomposit für thermische Isolierzwecke“

Erfinder: G. Jonschker, M. Mennig, H. Schmidt

US-Patent Nr. 7279173 B2

Stammanmeldungstitel: „Mikrobizid beschichteter Gegenstand, Verfahren zu dessen Herstellung und dessen Verwendung“

Erfinder: D. Buxmann, H. Schiestel, H. Schirra, H. Schmidt

Koreanisches Patent Nr. 10-0763696

Stammanmeldungstitel: „Mikrobizid beschichteter Gegenstand, Verfahren zu dessen Herstellung und dessen Verwendung“

Erfinder: D. Buxmann, H. Schiestel, H. Schirra, H. Schmidt

Japanisches Patent Nr. 4003810

Stammanmeldungstitel: „Verbundwerkstoffe“

Erfinder: G. Jonschker, M. Mennig und H. Schmidt

Japanisches Patent Nr. 4000423

Stammanmeldungstitel: „Nanokomposit für thermische Isolierzwecke“

Erfinder: G. Jonschker, M. Mennig, H. Schmidt

Koreanisches Patent Nr. 10-0737657

Stammanmeldungstitel: „Verfahren zur Herstellung von Suspensionen und Pulver auf Basis von Indium-Zinn-Oxid und deren Verwendung“

Erfinder: R. Nonninger, C. Göbbert, R. Drumm, S. Sepeur, H. Schmidt

Koreanisches Patent Nr. 10-0737554

Stammanmeldungstitel: „Verfahren zur Herstellung eines mikrostrukturierten Oberflächen-reliefs durch Prägen thixo-

troper Schichten und mikrostrukturiertes Oberflächenrelief“

Erfinder: A. Gier, N. Kunze, M. Mennig, P. W. Oliveira, B. Schäfer, H. Schmidt, S. Sepeur

Chinesisches Patent Nr. ZL 03808123.7

Stammanmeldungstitel: „Substrate mit Biofilm hemmender Beschichtung“

Erfinder: C. Becker-Willinger, H. Schmidt

Chinesisches Patent Nr. ZL 01818076.0

Stammanmeldungstitel: „Mikrobizid beschichteter Gegenstand, Verfahren zu dessen Herstellung und dessen Verwendung“

Erfinder: D. Buxmann, H. Schiestel, H. Schirra, H. Schmidt

Polnisches Patent Nr. 193895 B1

Stammanmeldungstitel: „Verbundwerkstoffe“

Erfinder: R. Angenendt, G. Jonschker, M. Mennig und H. Schmidt

Japanisches Patent Nr. 3931246 B2

Stammanmeldungstitel: „Verfahren zur Herstellung von strukturierten anorganischen Schichten“

Erfinder: H. Krug, M. Mennig, H. Schmidt

Japanisches Patent Nr. 3909389 B2

Stammanmeldungstitel: „Verfahren zur Herstellung von funktionellen glasartigen Schichten“

Erfinder: A. Bauer, T. Burkhart, C. Fink-Straube, G. Jonschker, M. Mennig, H. Schmidt, M. Schmitt

US-Patent Nr. 7,169,834 B2

Stammanmeldungstitel: „IR-absorbierende Zusammensetzungen“

Erfinder: M. Döbler, W. Hoheisel, R. Nonninger, M. Schichtel, H. Schmidt, M. Jost

Cooperations / Kooperationen

Neben der Kooperation mit der Universität des Saarlandes arbeitet das INM unter anderem mit folgenden nationalen und internationalen Institutionen zusammen (Auswahl):

CeNTech, Universität Münster

CNRS Laboratoire de Chimie de Coordination (LCC), Toulouse, Frankreich

Deutsche Gesellschaft für Holzforschung e.V.

Fraunhofer-Institut für Biomedizinische Technik, St. Ingbert

Fraunhofer-Institut für Silicatforschung, Würzburg

Fraunhofer-Institut für Solare Energiesysteme, Freiburg

Fraunhofer-Institut für Zerstörungsfreie Prüfverfahren, Saarbrücken

Humboldt-Universität, Berlin

IBM Zurich Research Laboratory, Rüschlikon, Schweiz

Instituto de Ceramica y Vidrio, Madrid, Spanien

KIST EUROPE Forschungsgesellschaft mbH, Saarbrücken

Max-Planck-Institut für Metallforschung, Stuttgart (Außenstelle des INM)

McGill University, Montreal, Kanada

Nanoscience Centre, Cambridge, Großbritannien

Technische Universität Delft, Niederlande

University of California, Santa Barbara, U.S.A.



University of Chicago, U.S.A.

Université de Franche-Comté, Besançon,
Frankreich

Universität Heidelberg

Université Louis Pasteur, Strasbourg, Fran-
kreich

University of Manchester, School of Materi-
als, Großbritannien

University of Massachusetts, U.S.A.

Universität Stuttgart

Université de Toulouse, Frankreich

Weizmann Institute of Science, Israel

Weiterhin bestehen diverse Kooperationen
im Rahmen des DFG-Sonderforschungsbe-
reich 277 und in zahlreichen Projekten.

Besuch des Bundespräsidenten Horst Köhler



Gast aus Berlin ließ sich von INM-Atmosphäre begeistern

Bei seinem Saarlandbesuch im Januar war Bundespräsident Horst Köhler auch Gast des INM.

Begleitet wurde er von Ministerpräsident Peter Müller, den Ministern Dr. Hans peter Georgi und Karl Rauber sowie dem Kuratoriumsvorsitzenden des INM, Staatssekretär Dr. Christian Ege.

Der Rundgang durchs Institut zeigte die enorme Bandbreite der Forschungs- und Entwicklungsarbeiten. Das Thema „Material- und Energieeffizienz“ interessierte Köhler besonders. Nach seiner Auffassung müsse in diese Forschungsbereiche noch mehr investiert werden.

Sein Augenmerk galt auch der wirtschaftlichen Umsetzung von Forschung und den Chancen deutscher Nanotechnologie auf den internationalen Märkten. Intensiv wurde auch das Thema „Nachwuchs und Ingenieurlücke“ angesprochen.

Der Bundespräsident genoss sichtlich die Atmosphäre am INM. Was er denn nun mitnehme, wurde er am Ende seines Besuches gefragt. Die Antwort kam schnell: Dass das Saarland ein ganz wichtiger Standort für Nanotechnologie in Deutschland sei und dass er selbst das INM und sein Know-how in Berlin noch bekannter machen werde.



20 Jahre Institut für Neue Materialien



Der runde Geburtstag ist ein Signal für den Aufbruch

Es war wie eine Generalversammlung der saarländischen Nano-Familie: Zum Festakt zum 20-jährigen Jubiläum des INM kamen fast alle, die in Wissenschaft oder Wirtschaft dieses Feld bearbeiten.

Der saarländische Ministerpräsident Peter Müller sagte in seiner Ansprache, das Institut sei nicht nur ein Leuchtturm der regionalen Wissenschaftslandschaft, sondern auch ein zentraler Ort des Technologietransfers. Es vor zwei Jahrzehnten aus der Taufe zu heben, sei „ein visionärer Akt“ gewesen.

Prof. Dr. Ernst Th. Rietschel, der Präsident der Leibniz-Gemeinschaft, lobte das INM als „Vorzeige-Einrichtung“ seiner Organisation. Durch den Ansatz der „vertikalen Interdisziplinarität“ und seine Partnerschaft mit der Universität habe es Vorbildfunktion für andere Forschungseinrichtungen.

Dass das Institut den runden Geburtstag nicht zum Anlass von Selbstzufriedenheit nehme, betonte der Vorsitzende der INM-Geschäftsführung, Prof. Dr. Eduard Arzt. Es gehe vielmehr darum, durch neue Orientierungen einen neuen Aufbruch zu schaffen. Einen lebendigen Festvortrag hielt Prof. Dr. Herbert Gleiter, Gründungsdirektor des INM. Wer ihm zuhörte, ahnte etwas von der Leidenschaft, die im Spiel sein muss, wenn jemand in der Wissenschaft Neuland sucht. Seine Empfehlung: keine Denkverbote, keine Trägheit durch Gewohnheit. Am INM, war er sicher, gehöre das zur Unternehmenskultur!





Erratum:

Ergänzung zu Talks / Vorträge



Diese Vorträge wurden aufgrund eines technischen Fehlers nicht in die Druckversion aufgenommen.

S. Mathur
Chemically designed functional nanostructures
211th ECS Meeting; May 6-10, 2007; Chicago <Ill., USA>

S. Mathur
Chemical nanotechnology: From molecules to product applications
NANOMAT; June 5-7, 2007; Bergen <Norway>
Eingeladener Vortrag

S. Mathur
Chemically designed nanoparticles and nanowires: Controlled growth, applications and devices
iNANOSCHOOL. PhD Summer School; June 10-15, 2007; Ebeltoft <Denmark>

S. Mathur
Chemical nanotechnology: From molecules to applications
10th International Conference and Exhibition of the European Ceramic Society (ECerS); June 17-21, 2007; Berlin
Eingeladener Vortrag

S. Mathur
Chemical nanotechnology: From molecules to applications
Nanotechnology in industrial applications : EuroNanoForum 2007; June 19-21, 2007; Düsseldorf
Keynote Lecture

S. Mathur
Nanostructured functional surfaces through chemistry: Processing and applications
ISTMC 2007. International Symposium on Technology for Materials & Components; June 22, 2007; Siheung <South Korea>
Eingeladener Vortrag

S. Mathur
Chemically designed nanomaterials: Controlled growth, applications and devices
ICMAT 2007. International Conference on Materials for Advanced Technologies; July 1-6, 2007; Singapore
Eingeladener Vortrag

S. Mathur
Chemical nanotechnology: From molecules to product applications
IHPC. The Institute of High Performance Computing; July 3, 2007; Singapore
Eingeladener Vortrag

S. Mathur
Nanomaterials through chemistry
2nd International Workshop on Smart Materials and Structures; August 29-31, 2007; Kiel
Eingeladener Vortrag

S. Mathur
One dimensional oxide nanostructures: Growth, applications and devices
Euromat 2007; September 10-13, 2007; Nürnberg
Eingeladener Vortrag

S. Mathur
Funktionelle- und Barriere-Schichten mittels chemischer Gasphasenabscheidung
Cluster-Forum. Funktionalisierte Oberflächen. Schutzfunktion - Barrierewirkung - Bioaktivität; November 22, 2007; Augsburg

S. Mathur
Tin oxide nanowires by chemical vapor deposition
MS&T'07 Materials Science & Technology; September 16-20, 2007; Detroit <Mich., USA>
Eingeladener Vortrag

S. Mathur
Chemically designed nanomaterials: Controlled growth, applications and devices
ACTSEA-2007. International Symposium on Advanced Ceramics and Technology for Sustainable Energy Applications; November 04-07, 2007; Kenting <Pingtung, Taiwan>
Eingeladener Vortrag

S. Mathur
Chemically designed nanoparticles and nanowires
PacRim7 - 7th Pacific Rim Conference on Ceramic and Glass Technology; November 11-14, 2007; Shanghai <China>
Eingeladener Vortrag

S. Mathur
Chemically designed nanoparticles and nanowires: Controlled growth, applications and devices
MRS Fall Meeting; November 26-30, 2007; Boston <Mass., USA>
Eingeladener Vortrag

S. Mathur, S. Barth, T. Rügamer and H. Shen
Metal oxide nanowires: Growth, applications and devices
211th ECS Meeting, J5 - Sensors Based on Nanotechnology 3; May 6-10, 2007; Chicago <Ill., USA>

K. Moh
Core-Shell-Nanopartikel
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken

K. Moh
Photokatalyse: Von der Idee zum Projekt - arbeiten und promovieren an einem anwendungsorientierten Forschungsinstitut
Kolloquium des SFB 445 / Graduiertenkolleg 1240/1 "Nanotronics". Universität Duisburg Essen; February 5, 2007; Duisburg
Eingeladener Vortrag

T. S. Müller
Selbstreinigende Oberflächen - Immer sauber durch Photokatalyse
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken

J. Pütz
Sol-Gel-Prozess & Nassbeschichtung. Von den Grundlagen zur Anwendung
Summer School Chemische Nanotechnologie; October 8-12, 2007; Saarbrücken

J. Pütz
Nasschemische Beschichtung und Strukturierung
Nasschemische nanokomposite Beschichtungen. OTTI-Fachforum; February 7-8, 2007; Regensburg



Diese Vorträge wurden aufgrund eines technischen Fehlers nicht in die Druckversion aufgenommen.

J. Pütz
Funktionelle Schichten auf der Basis von Sol-Gel-Materialien & Nanopartikeln
Wissenschaftliches Kolloquium, Staatliche Materialprüfanstalt Darmstadt / Institut für Werkstoffkunde, TU Darmstadt; January 25, 2007; Darmstadt

J. Pütz
Flexible printing of transparent conducting oxides for display applications
pro flex 2007. Vacuum roll-to-roll processing of flexible materials; September 10-11, 2007; Dresden
Eingeladener Vortrag

J. Pütz
Sol-Gel-Farbschichten
Nasschemische nanokomposite Beschichtungen. OTTI-Fachforum; February 7-8, 2007; Regensburg

J. Pütz
Fundamentals of sol-gel deposition
Workshop of the Belgian IAP project P6/16. Advances complex inorganic materials by a novel bottom-up (nano)chemistry approach: processing and shaping; November 29, 2007; Gent <Belgium>
Eingeladener Vortrag/Keynote Lecturer

J. Pütz
Transparente leitfähige Oxide als nanoskalige Lackadditive
Seminar "Nanoteilchen in Farben und Lacken"; February 26-27, 2007; Esslingen

J. Pütz
Drucken funktioneller Materialien auf Glas
Funktionalisierung von Glasoberflächen durch Beschichtungen. OTTI-Seminar; May 21-22, 2007; Regensburg

J. Pütz
Nass-chemische Aufbringung von TCO - Eine flexible Alternative
Transparente leitfähige Schichten. OTTI-Seminar; September 17-18, 2007; Würzburg

J. Pütz
Prinzipien der Sol-Gel-Beschichtungen im industriellen Einsatz
Funktionale Oberflächenbeschichtungen im industriellen Einsatz; November 07-08, 2007; Regensburg

J. Pütz
Direct gravure printing of electronic materials - towards roll-to-roll manufacturing of OLED devices
International Symposium for Electronics on Polymer & Web-Coating Technology; March 20-21, 2007; Tokyo <Japan>
Eingeladener Vortrag