Leibniz-Institut
für
Oberflächenmodifizierung e. V.

BIANNUAL REPORT 2006/2007

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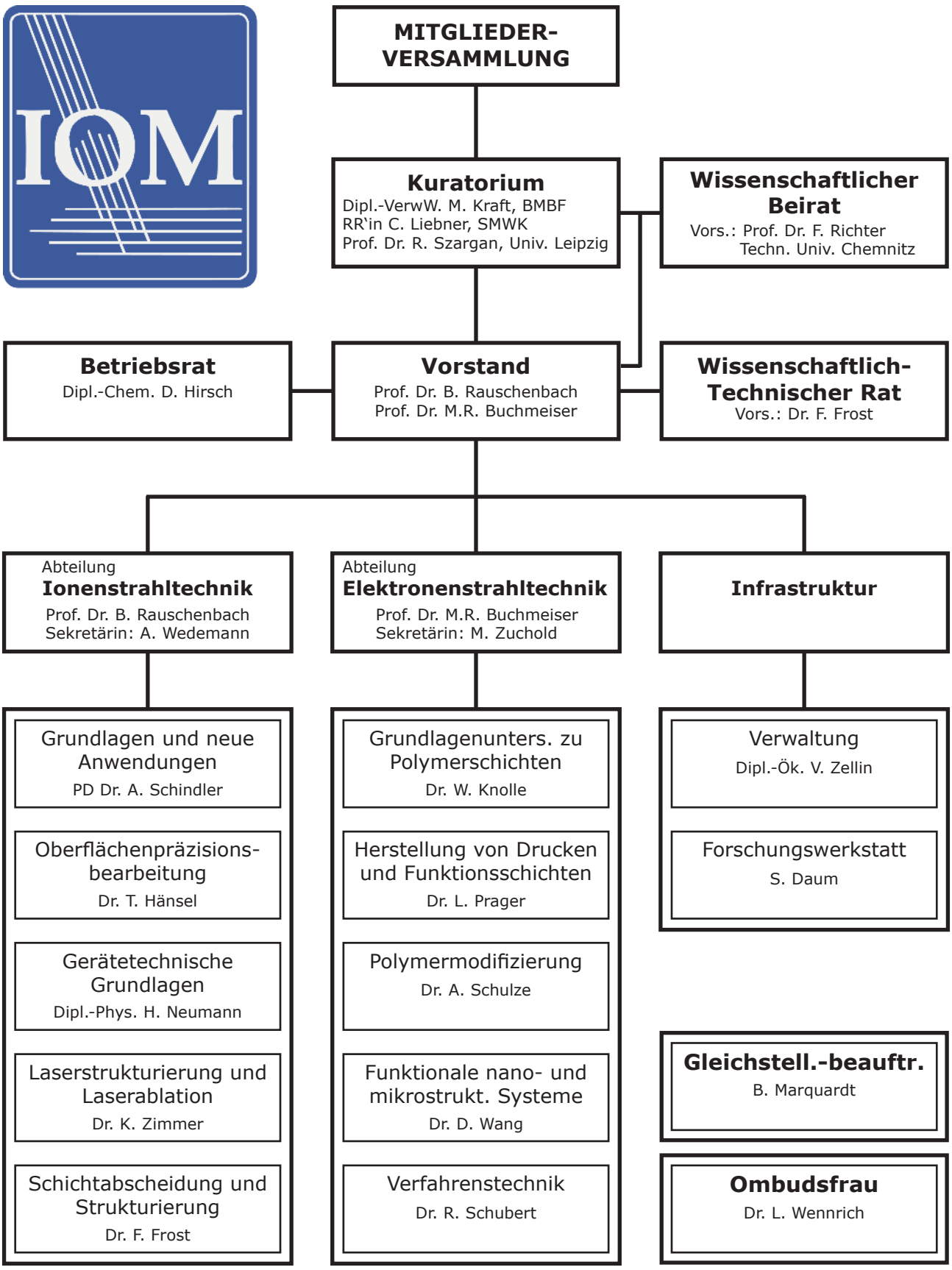


Contents

Preface

The Leibniz-Institut für Oberflächenmodifizierung e. V (IOM), a member of the Leibniz Association, presents this Biannual Report as summary of the scientific work performed in 2006/2007 and as documentation of publication, patents, and other relevant scientific activities.

The IOM deals with application-oriented fundamental research in chosen areas of the modification of surfaces and thin films by pursuing complete lines from explorative fundamental research to the point of near-industrial applications.

Research and development areas of the Institute are

- Ion and plasma assisted ultra-precision shaping and smoothing
- Micro- and nanodimensional structuring and structure transfer
- Thin film deposition and modification
- Fundamental principles of polymeric coatings
- Manufacture of printings and functional coatings
- Functional nano- and microstructured systems

In its research, the IOM puts strong emphasis on collaborations with industry, small and medium enterprises, universities, and other research laboratories. The IOM also participates in joint projects directly funded by industry or Federal Agencies such as the BMBF or by the Free State of Saxony. Among extensive research other activities, the participation in DFG research units, and main focus programs should be mentioned. The last two years were again copious years for the commencement of new activities in this direction. The successful cooperation with chemical, optical, and semiconductor industry was continued. Results of both fundamental and applied research could be jointly transferred into industry.

Highlights of cooperation with the University Leipzig in the last two years were also the successful applications of the centre of excellence "Translational Centre for Regenerative Medicine" and the graduate school "Building with Molecules and Nano-objects" in the framework of the German "Competition for Excellence".

Another big success story for the IOM were the successful applications of the DFG Research Group № 845 "Self-organized nanostructures by low-energy ion beam erosion" and the BMBF Young Scientist Research Group "Ultra-precision processing with atomic particle beams"

In this report the IOM presents its scientific activities and major achievements in the years 2006 and 2007. In this context, the Biannual Report 2006/2007 presented here gives a comprehensive summary of our results. In the first part, overviews on selected projects are given, arranged according to the structure of the IOM research program. These overviews are supplemented by feature articles on selected topical highlights. Finally, the appendices give a full list of publications, talks, teaching activities, and other achievements of the IOM staff.

The Institute would like to thank all friends and organisations who supported its progress in the last two years. Special thank is due to our Board of Trustees and Scientific Advisory Board. Our partners from industry and other research institutes

play an essential role for the IOM. The Board of the Institute would like to thank all members and guests of the institute for their active and excellent contributions to a successful development.

Leipzig, March 2008

Prof. Dr. Bernd Rauschenbach
Director

In 2006, Thomas Höche was awarded the Max-von-Laue Price of the *Deutsche Gesellschaft für Kristallographie* in appraisal of "his manifold contributions to the structure solution of aperiodic crystals" by neutron, X-ray, and electron diffraction.

Rico Böhme received the Young Scientist Award of the European Material Research Society in the year 2006 for his studies on the fundamentals of laser-induced backside wet etching of transparent materials.

The 9th International Workshop on "Plasma-Based Ion Implantation and Deposition", a joint venture of the Leibniz-Institut für Oberflächenmodifizierung Leipzig and the Forschungszentrum Dresden-Rossendorf, was held at the Kubus in Leipzig from September 2nd to 6th, 2007.

Scientific and Technology Results

Reports and Selected Results

Monolithic supports prepared via electron beam curing

R. Bandari, B. Schlemmer, C. Elsner, W. Knolle, M.R. Buchmeiser

Introduction

Monolithic media consist of a single piece of highly porous material and have been intensively developed since the 1990s, especially as supports for separation science and heterogeneous catalysis. Two types of monoliths can be distinguished according to the used material, i.e. silica-based and organic-polymer based. Polymer based monoliths can be prepared either by controlled, TEMPO-initiated radical or living metathesis polymerization, polycondensation, poly-addition or free radical polymerization. In the latter case, radical polymerization can be triggered thermally, via UV or γ-irradiation [1]. Especially radiation based polymerization techniques are suitable for the preparation of monoliths in micro-dimensional systems, e.g. capillaries or chips, because of the easiness of their filling by liquid monomer precursors and the position-resolved control of polymerization and functionalization by focused beams and masking techniques [2]. However, UV and γ-irradiation suffer from serious drawbacks. UV curing can only be realized in case an expensive UV transparent carrier material is used. This is contraindicative to the majority of miniaturized devices, which consist of large scale producible (e.g. produced by hot embossing, injection, or UV molding), inexpensive, UV intransparent materials. Additionally, the low penetration depth of photons confines the use in large scale dimensions. An alternative is γ-irradiation. However, it is significantly restricted by security and environmental requirements. Therefore, we used a polymerization technique based on electron beam (EB) curing for the preparation of polymeric monolithic materials [3-5]. Herein, we present results concerning their synthesis, post-curing functionalization and use in separation science.

Synthesis

A general schema for the preparation of EB derived monoliths and their post-curing functionalization is shown in Figure 1. Typically, EB curing was carried out using ethyl methacrylate (EMA) and trimethylol-propane triacrylate (TMPTA) as the crosslinking monomers, 2-propanol and 1-dodecanol as the macroporogens, and toluene as the microporogen. Additionally, copolymerizable monomers with orthogonal functional groups can be incorporated into the polymerization mixture for copolymerization or post-curing functionalization.

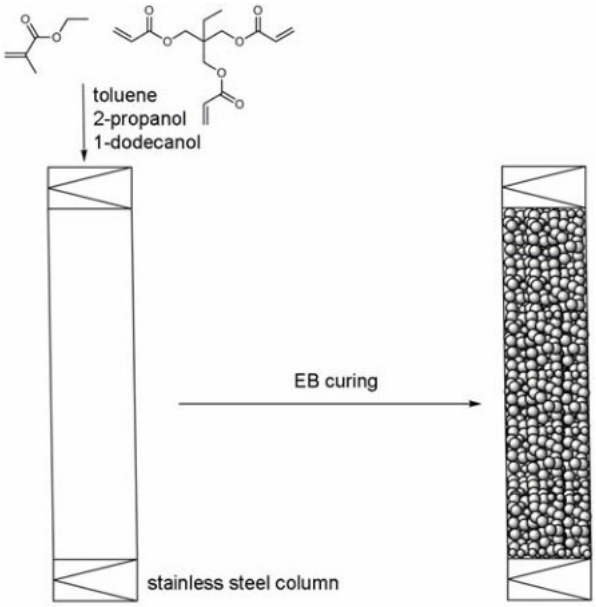


Figure 1: Synthesis of monolithic materials prepared by EB curing.

Monomer concentrations between 15-25 wt.-% and macroporogen concentrations between 20-30 wt.-% were found to be suitable for phase separation during the irradiation process. Electron-beam irradiations were performed at the 10 MeV linear accelerator ELEKTRONIKA (Toriy Company, Moscow). The accelerator was operated at a 50 Hz repetition rate and 4 μs pulse length using a scanning horn (width up to 40 cm, scanning frequency 1 Hz) and a movable table to irradiate the samples. The correlation between the applied dose and the monomer conversion is shown in Figure 2. Figure 3 represents a scanning electron micrograph of an EB curing derived monolith which displays macroporous structures and microglobules with average diameters in the sub-micrometer range. Inverse size exclusion chromatography revealed a mainly macroporous

structure with a low percentage of pores < 100 A. Depending on their composition, specific surface areas varied in the range of 15-23 m^2/g, pore volumes between 60 and 216 mL/g and total porosities in the range of 44 to 83%.

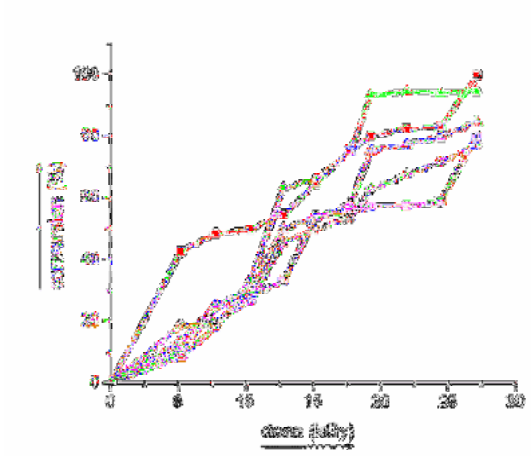


Figure 2: Conversion vs. dose for the polymerization/crosslinking carried out in a mixture of 2-propanol and 1-dodecanol solutions: 10 vol.-% (open squares), 30 vol.-% (open circles), 40 vol.-% (open triangles), 45 vol.-% (full triangles), and 50 vol.-% (full squares) of EMA and TMPTA (1:1). Experiments were carried out at 2 °C.

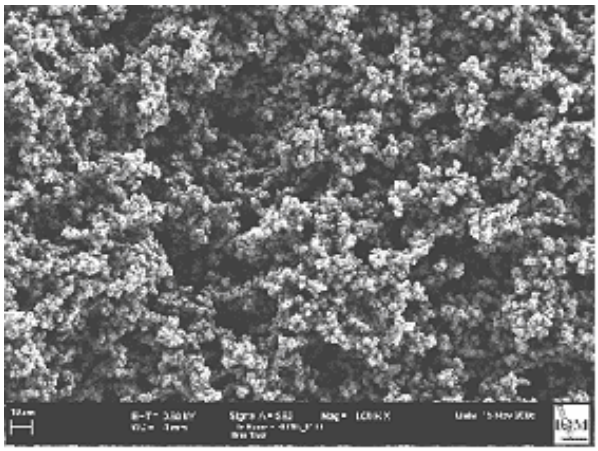


Figure 3: SEM image of a EB curing derived monolith.

Functionalization

Further processing of EB curing derived monoliths were carried out based on the copolymerization of monomers with orthogonal functional groups. Straightforward functionalization of EB curing derived monolithic supports via the copolymerization of N,N-dipyrid-2-ylmethy-acryl amide resulted in a continuous distribution of the functional monomer within the monolithic support. Loading with Pd (II) resulted in a monolithic continuous flow reactor for heterogeneous catalysis with a final Pd- loading of 7.8 mg/g. A simple Heck coupling of iodobenzene with styrene was used to monitor the reactivity and determine the leaching of palladium. A constant, almost quantitative conversion (> 95%) was observed over a range of 5 hours. The highly macroporous nature of the support facilitated mass transfer and gave rise to a low back pressure (< 1.5 Mpa at a flow rate of 0.7 cm/min). Low amounts of palladium leached into the reaction mixture (4.2%). A trans:cis ratio in stilbene formed from styrene and iodobenzene of 93:7 was found, which is characteristic for the dipyridylamide ligand.

However, the copolymerization of functional monomers may be less attractive in several cases. Since they become incorporated into the polymeric matrix, a major part of the functional monomer is not accessible and the surface density of functional groups for further immobilization or adjusting of surface interfacial properties is low. Moreover, special functional groups may inhibit free radical polymerization. Therefore, a post-functionalization technique based on ring-opening metathesis polymerization (ROMP) giving access to a broad range of surface enriched functional units was developed. ROMP is a transition metal triggered polyinsertion which requires the use of cyclic olefins with a substantial ring strain, e.g., norborn-2-enes and cyclooctenes.

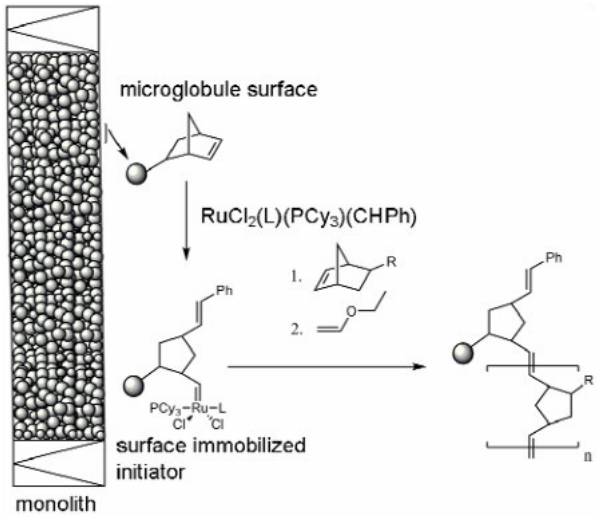
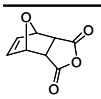
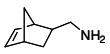
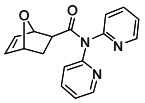
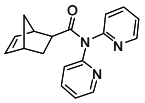
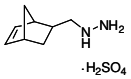


Figure 4: ROMP-based post-functionalization of EB curing derived monolith.

Major advantages of ROMP are the truly living polymerizations that may be accomplished as well as the possibility of using functional monomers. Among the large number of well-defined initiators that are presently available, those based on ruthenium (i.e., the Grubbs initiators) display a higher functional group tolerance compared to those based on molybdenum (Schrock catalysts). Thus, norborn-5-ene-2-ylmethyl acrylate was added to the EB-curable polymerization mixture for subsequent post-functionalization by ROMP (Figure 4).

Although an irradiation dose of 22 kGy was applied over a time period of 15 minutes giving raise to typically 70-90% conversion of the acrylic moieties, the norborn-2-ene units were retained and could in fact be used for post-synthesis functionalization by initiation with either $RuCl_2(PCy_3)_2(CHPh)$ (**1**) or $RuCl_2(PCy_3)$(1,3-dimesityl-4,5-dihydroimida-zol-2-inylidene) (CHPh) (**2**) followed by ROMP of various functional monomers, e.g. pyridine, carboxylic acid, hydrazine, and amine-containing monomers (Table 1).

Table 1: Results of the grafting of different monomers by ROMP.

Monomer	Initiator	Ru [µmol g^{-1}]	Grafting density [µmol g^{-1}]
	1	249	290
	2	56	180
	1	400	240
	2	74	191
	1	193	17
	2	505	73
	1	263	42
	2	37	13
	1	212	37
	2	50	29

The amounts of grafted catalysts and monomers were determined by elemental analysis and ICP-OES. The extent of initiator immobilization was found to be substantially higher for **1** than for **2**. Grafting yields of monomers were found to be dependent on both the nature of the monomer and the amount of initiator immobilized onto the monolith and could be varied within a range of 13-290 µmol.

Application

As already shown, EB curing derived monoliths can be used as supports for the immobilization of catalysts. However, a major application field is the fast separation of low and high molecular weight analytes. Monolithic columns exhibit an enhanced mass transport and lower backpressures allowing for higher flow rates without loss in efficiency. Thus, extremely low separation times for LC-applications are achieved and therefore monolithic columns become more and more feasible in high throughput (HT) applications combined with coupling techniques like LC-MS. To meet the requirements of HT processes, further miniaturization is needed.

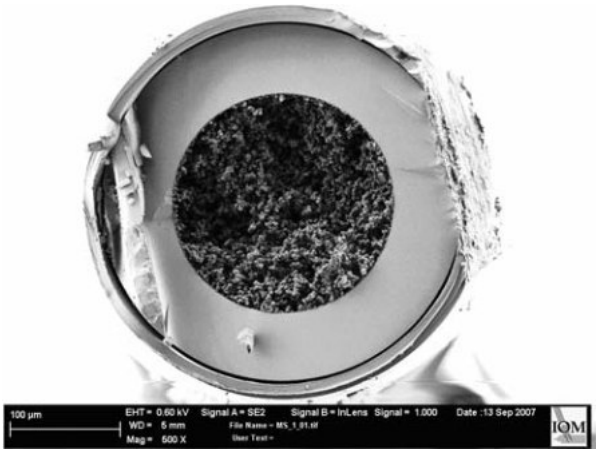


Figure 5: SEM image of a cross section of a capillary with an EB derived monolith inside.

Starting with column dimensions in the range of 3 mm i.d. x 100 mm, capillary columns with dimensions in the range of 0.05-0.2 mm i.d. x 150 mm were developed (Figure 5). Additionally, monolithic media were successfully implemented on lab-on-the-chip devices.

The developed systems were tested for the separation of proteins and amino acids. Exemplarely, chromatograms for the separation of proteins and dansylated (DNS) aminoacids are shown in Figure 6 and Figure 7, respectively.

The possibility to scale up monolithic devices in order to gain access to real preparative scale sepa-

ration units or heterogeneous catalytic systems of technical relevance is of utmost importance.

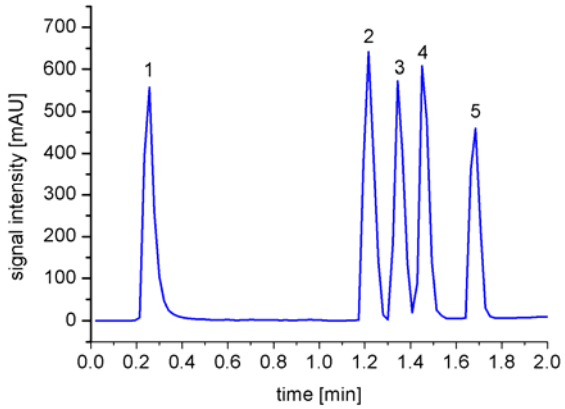


Figure 6: Separation profile of a protein standard on an EB-derived monolith. Column dimensions: 3 x 100 mm, peak order: (1) lysozyme, (2) ribonuclease A, (3) insulin, (4) cytochrome C, (5) albumin; mobile phase A: 95% water, 5% acetonitrile, 0.1% TFA; mobile phase B: 20% water, 80% acetonitrile, 0.1% TFA; linear gradient, 10-90% B in 2.0 min.; flow rate, 3 mL/min; detection, UV (200 nm).

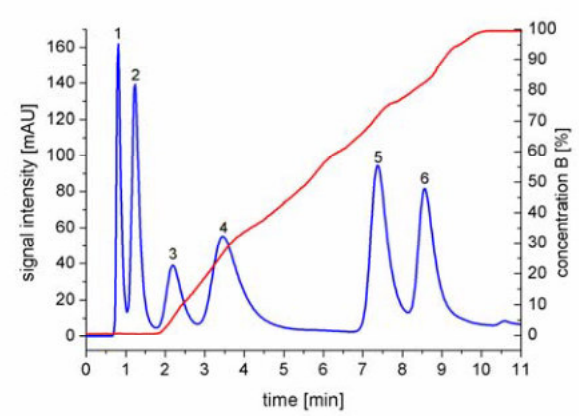


Figure 7: Separation profile of DNS-amino acids on a EB-derived monolith. Column dimensions: 3 x 100 mm; mobile phase A: 58% water + 42% methanol + 0.6% acetic acid and 0.008% triethylamine; mobile phase B: 5% water + 95% methanol + 0.6% acetic acid and 0.008% triethylamine; linear gradient, 0-100% B in 1-10 min and then constant up to 11 min flow rate, 1 mL/min; detection, UV (310 nm); elution order: (1) a-DNS-L-arginine, (2) DNS-L-aspargine, (3) DNS-L-glutamic acid, (4) DNS-L-alanine, (5) DNS-L-isoleucine, and (6) DNS-L-tryptophane. Injection volume 5 µL, concentration of DNS-amino acids was 1.2 mg/mL for 1, 2, 3, and 5; 0.8 mg/mL for 6; 1 mg/mL for 4.

In this context, it has to be lined out that free radical polymerization-derived, large diameter monoliths, i.e., those with ids > 10 mm have not been accessible by a one-step procedure so far. Thus, extensive heat formation occurs in thermally triggered systems which influences the structure of the monolith in a negative way. Therefore, existing large diameter systems have been prepared by modules from monolithic annuluses that are assembled after synthesis. However, EB irradiation can be performed stepwise applying a pulsed mode, thus providing sufficient time to dissipate the polymerization enthalpy and obtaining homogeneous monolithic structures within the confines of a 20 x 300 mm column [6].

Conclusions

An electron beam curing-based process for the manufacture of small- and large-diameter, monolithic materials has been developed. The novel monoliths are applicable to the separation of both, low and high molecular weight analytes as well as supports for heterogeneous catalysis. So far, the functionalization of the novel devices was accomplished via the copolymerization of an orthogonal functional monomer and ROMP mediated surface grafting by the use of Grubbs first and second generation initiators.

Acknowledgements

The work was supported by a grant from the Deutsche Forschungsgemeinschaft (DFG, BU2174/1-1), by the Federal Government of Germany, and by the Freistaat Sachsen.

Literature

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[2] Logan, T.C.; Clark, D.S.; Stachowiak, T.B.; Svec, F.; Frechet, J. M. J. Anal. Chem. 2007, 79, 6592-6598.
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In-line monitoring of conversion and thickness of polymer coatings by NIR spectroscopy

T. Scherzer, G. Mirschel, K. Heymann, M.R. Buchmeiser
in collaboration with
H. Lucht, A. Feierabend, LLA Instruments GmbH, Berlin

Introduction

Process control plays an increasingly important role in chemistry and chemical engineering. Sophisticated analytical techniques such as near-infrared (NIR) spectroscopy can supply detailed information about the chemical and physical parameters of the system. The continuous control of such parameters can assist to ensure a constantly high quality of the product.

In UV curing technology, there are two parameters which have to be controlled: the conversion after irradiation and the thickness of the coating which is applied to the substrate. Most functional properties of a coating strongly depend on the conversion of the reactive groups. The conversion, however, is influenced by a large number of factors which can be controlled only in part. The coating thickness should not fall below a minimum level given by the specification since a sufficient thickness is vitally important for most of the properties of the final coating. In addition to that, most applications require a high homogeneity of the thickness. NIR reflection spectroscopy was shown to be a powerful analytical method for the in-line monitoring of both parameters [1].

Monitoring of the conversion in acrylate coatings

Usually, NIR spectroscopy is used as an indirect analytical method, i.e. quantitative results refer to data of a reference method. This requires the preparation of large amounts of well-defined calibration samples as well as complex calibration procedures. Sophisticated chemometric methods such as multivariate partial least squares (PLS) regression assist in the quantitative evaluation of the spectral data. Since this procedure is mostly too laborious for the determination of the conversion in UV cured coatings, an alternative quantification method was developed which is based on the direct determination of the conversion from the overtone band of the acrylic double bonds at 1620 nm according to the Beer-Lambert law [2]. Figure 1 compares the conversion which was obtained in this way from the NIR reflection spectra of UV-cured clear coats with reference data from FTIR transmission spectroscopy.

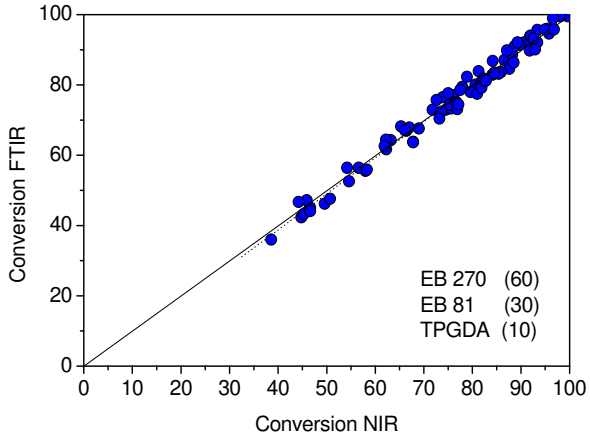

Figure 1: Comparison of the conversion in UV-cured acrylate layers determined from NIR reflection spectra with reference data from FTIR transmission spectroscopy.

Figure 1 shows that quantitative results with high precision are obtained with this method which is much easier to handle than the chemometric approach. So, it can be used for the rapid evaluation of NIR spectra that are recorded during in-line monitoring of the conversion in real coating processes. Figure 2 shows the development of the conversion in an acrylic clear coat applied to polypropylene foil on a roll-coating machine. Both the intensity of the UV lamp and the line speed were varied in order to vary the irradiation dose. The resulting changes of the conversion can be easily seen in the recorded trace. With this method, monomer conversion can be followed at line speeds of 100 m/min or more.

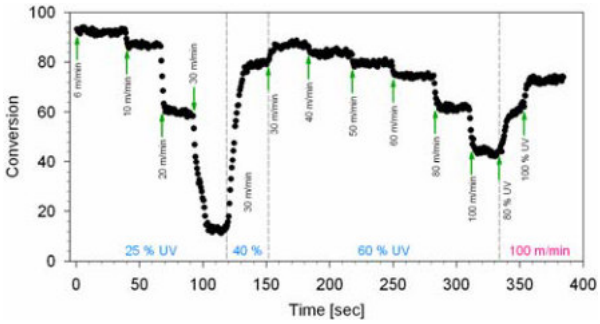

Figure 2: In-line monitoring of the conversion in a 15 g m^{-2} acrylate coating on 20 µm OPP foil after UV curing with variable irradiance and at various line speeds.

Monitoring of the conversion in cationic epoxide/vinyl ether blends

Cycloaliphatic epoxy resins such as 3,4-epoxycyclohexylmethyl-3',4'-epoxycyclohexane carboxylate (EEC) are cross-linked by UV-induced cationic photopolymerization. Due to the high viscosity of these resins, they are often diluted with vinyl ethers (VE) which also polymerize by a cationic mechanism. However, the conversion in EEC cannot be followed by NIR spectroscopy as performed for acrylates since the cycloaliphatic epoxy groups do not have a specific absorption band in the near infrared. In contrast, vinyl bonds can be easily detected in the spectrum using the characteristic overtone band around 1612 nm. Figure 3 shows the gradual decrease of this band in a mixture of EEC and tetraethyleneglycol divinyl ether (DVE-4) after UV irradiation.

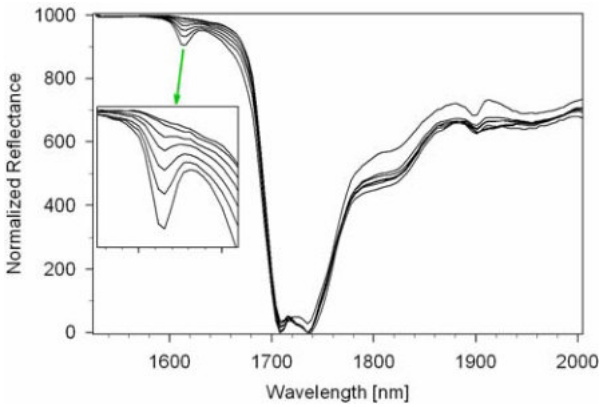

Figure 3: NIR reflection spectra of an EEC/DVE-4 formulation (80/20 wt./wt.) before UV irradiation and after 1, 5, 10, 30, 60, and 120 min postcuring, respectively (in direction of the arrow).

It was shown, that the epoxy groups in mixtures with VE react with similar or even higher rates than the vinyl double bonds [3]. So, it is possible to deduce the overall conversion of the system from the conversion of the vinyl ether. Consequently, NIR spectroscopy can be used to monitor the conversion in coatings from cycloaliphatic epoxide/vinyl ether blends.

In general, cationic formulations show a marked postcuring behavior. In contrast to acrylates, which polymerize in a radical process, only minor conversion is achieved during UV irradiation. However, it successively increases during the subsequent dark reaction. NIR spectroscopy can be used to follow the postcuring process. Figure 4 shows the dark reaction in coatings from an EEC/DVE-4 blend after exposure to various UV irradiation doses.

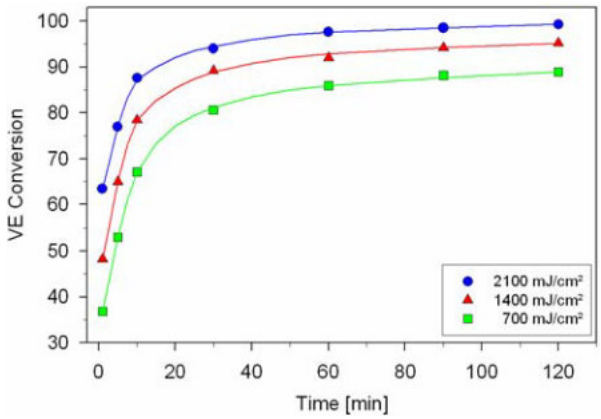

Figure 4: Conversion of the vinyl ether in an EEC/DVE-4 formulation (85/15 wt./wt.) after irradiation with various UV doses.

The strong postcuring behavior of cationic formulations has also consequences for the in-line monitoring of the conversion of coatings after UV curing. After leaving of the irradiation zone, the conversion is still rather low. A typical example is shown in Figure 5. Initially, the conversion is scarcely higher than 60%, but rapidly increases afterwards.

It should be noted that conversion data with very low scattering were obtained although a sampling rate of almost 15 spectra per second was used. The quality of the data as well as the time resolution are sufficient for the requirements of process control.

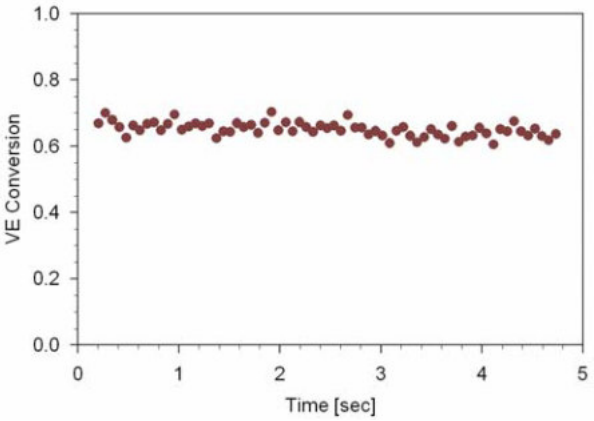

Figure 5: In-line monitoring of the vinyl ether conversion in an EEC/DVE-4 coating at a sampling rate of 15 NIR spectra per second.

In order to study the capability of the method to detect variations of the conversion caused by fluctuation or drift of the UV dose, coatings were irradiated at various line speeds and with various UV intensities. Moreover, the concentration of the photoinitiator was varied. Figure 6 shows the initial conversion in dependence on the line speed.

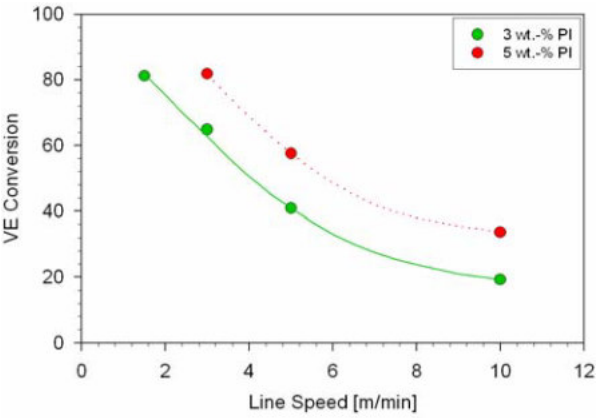

Figure 6: Actual conversion of the vinyl ether in EEC/DVE-4coatings just after leaving the irradiation zone in dependence on the line speed and the concentration of the photoinitiator (PI).

It can be clearly seen that an increasing speed of the conveyor leads to a drastic decay of the initial conversion. In part, this can be compensated by a higher concentration of the photoinitiator.

Monitoring of the thickness of acrylate coatings

Surprisingly, no analytical method was available so far which allowed for the in-line monitoring of the thickness of UV-cured or other polymer coatings. In this project, it was shown that the thickness of such coatings can be determined by NIR reflection spectroscopy [4]. The quantitative evaluation is based on powerful chemometric methods, since calibration samples with well-defined thickness can be easily prepared. Figure 7 shows a PLS-based calibration for the determination of the thickness of acrylic clear coats. In order to check the capability of this calibration function to predict the applied thickness, a series of independent

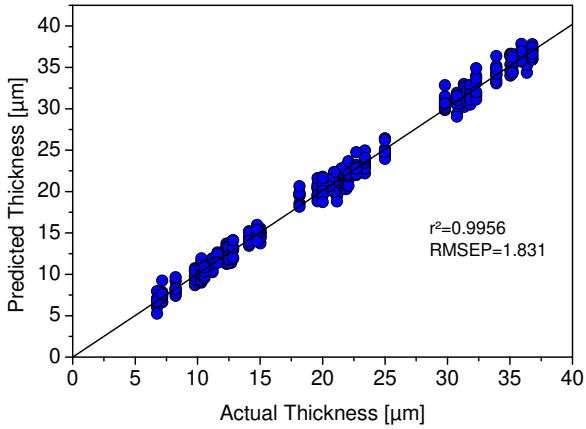

Figure 7: PLS calibration of the NIR spectra of acrylic clear coats to the applied thickness of the layers.

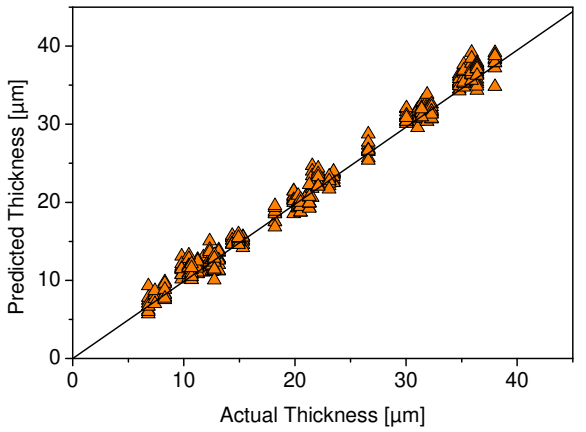

Figure 8: Prediction of the thickness of independent validation samples using the PLS calibration function shown in Figure 7.

validation samples, which were not used for calibration, was characterized. Results are shown in Figure 8. It is obvious that there is an excellent correlation between the actual thickness and the predicted data.

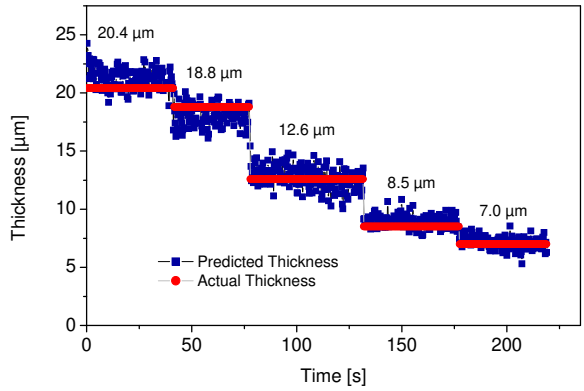

Figure 9: In-line monitoring of the thickness of an acrylic clear coat applied to OPP foil on a roll-coating machine.

For in-line monitoring of the coating thickness, the probe head was again mounted to a roll-coating machine. The acrylate formulation was applied to polypropylene foil by nip coating. The nip was varied stepwise. The thickness of the coatings was determined from the NIR spectra recorded simultaneously by use of the PLS calibration function. In addition, the actual thickness was determined off-line with a digital length gauge. Results are summarized in Figure 9.

Determination of the thickness of silazane-based SiO_x coatings

In a further research project (see contribution by L. Prager et al.), SiO_x barrier layers were applied to foils from polyethylene terephthalate (PET) by deposition and VUV-induced conversion of various silazanes. After application on a coating machine, the uniformity of the applied layer had to be checked. It was tested wheter this challenging problem could be solved by NIR spectroscopy in spite of the extremely low thickness of the layers (~100 to 800 nm). Since NIR spectroscopy predominantly detects bonds involving hydrogen, it is particularly suited for organic coatings. Nevertheless, it was possible to establish a calibration function which relates changes in the recorded NIR signals of the SiO_x coatings (which are probably due to changes of the reflectivity of the samples) to the coating weight (Figure 10).

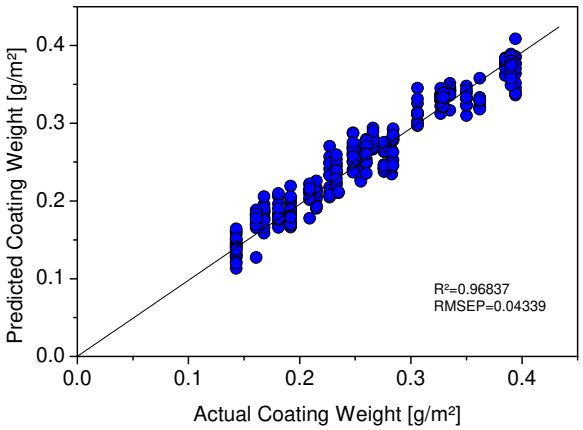

Figure 10: PLS calibration of the NIR signal of silazane-based SiO_x coatings on PET foil to the applied coating weight .

In spite of the strong scattering of the data due to the low thickness of the layers it was possible to monitor the uniformity of the thickness of SiO_x layers which were applied to PET foil by a pilot-scale coating machine. Figure 11 shows a lateral profile of the thickness.

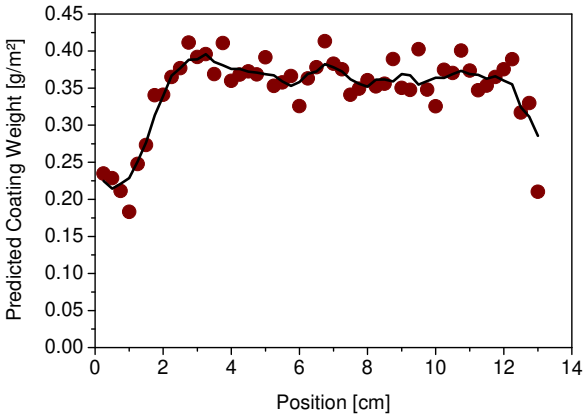

Figure 11: Monitoring of the uniformity of a silazane-based SiO_x coating on PET foil lateral to the web.

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Cationic Ru^{II}-N-heterocyclic carbene complexes for UV-induced ring-opening metathesis polymerization

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Introduction

Recently, there appears an increasing number of reports on latent Ru-based initiators. Such pre-catalysts are of particular interest in technical applications of ROMP, since they allow for the pre-formulation of a certain monomer-pre-catalyst mixture, its storage over a longer period of time at even elevated temperatures (usually < 45 °C) and, most important, the shaping and profiling of such mixtures prior to polymerization ("curing"). All latent Grubbs-type initiators which have been reported recently are triggered thermally. By contrast, particularly the areas of surface modification and functionalization require the existence of UV-triggerable pre-catalysts. Few systems have been reported so far, however, most of them possess significant disadvantages. Thus, either they show low activity and polymer yields (< 30%) in the photochemically triggered process, or the wavelength necessary to trigger ROMP is ≥ 360 nm. In the latter case, the initiators' thermal stability is generally poor, thus aggravating their application in photo-ROMP. However, none of the systems reported was entirely thermally stable above or even at room temperature and, therefore, does not fulfil the requirements of a truly latent photocatalyst.

Synthesis and structure

The following cationic NHC-Ru^{II}-complexes $[(Ru(IMesH_2)(CF_3CO_2)_2(t\text{BuCN})_4)^+ (CF_3COO^-)]$ and $[(Ru(IMes)(CF_3CO_2)_2(t\text{BuCN})_4)^+(CF_3COO^-)]$ (**PI-1** and **PI-2**, respectively) were prepared from $Ru(CF_3CO_2)_2(L)(p\text{-cymene})$[1,2] (L = IMes = 1,3-dimesitylimidazol-2-ylidene; L = $IMesH_2$ = 1,3-dimesityl-4,5-diyhdroimidazolin-2-ylidene) via reaction with excess of *t*BuCN. Both compounds can be handled in air. ^{1}H- and ^{13}C-NMR data and elemental analysis reveal the presence of one N-heterocyclic carbene (NHC), two inequivalent trifluoroacetates and four *t*BuCN ligands, suggesting cationic Ru(II) complexes. The structure of both **PI-1** and **PI-2** was confirmed by x-ray analysis; the one of **PI-1** is shown in Fig. 1.

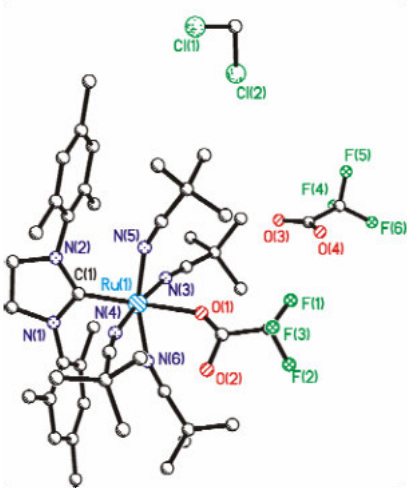


*Figure 1: Structure of **PI-1**.*

Polymerization

In a typical polymerization procedure 4 mg of **PI-1** (4×10^{-3} mmol) and 1 mmol of monomer were dissolved in 5 ml of $CDCl_3$ and transferred into a quartz Schlenk tube. The mixture was exposed to UV irradiation for 60 minute, and then it was poured into 50 ml of methanol. The polymer was isolated by filtration, washed thoroughly with methanol and pentane and dried in vacuo overnight at 40 °C.

Upon mixing of either **PI-1** or **PI-2** with a series of norborn-2-ene- or 7-oxanorbornene-derived monomers **3-7** or with cyclooctene (**8**) (Fig. 2), no reaction was observed at room temperature at all within 24 hours. Even highly reactive (distilled) dicyclopentadiene (**4**) did not react with either **PI-1** or **PI-2** at room or elevated temperature in the absence of light. However, exposing mixtures of either **PI-1** or **PI-2** in chloroform with these monomers to 308 nm light at room temperature resulted in the formation of the corresponding polymers (Fig. 3). Yields were in the range of 21-99%. The molecular weights of the polymers

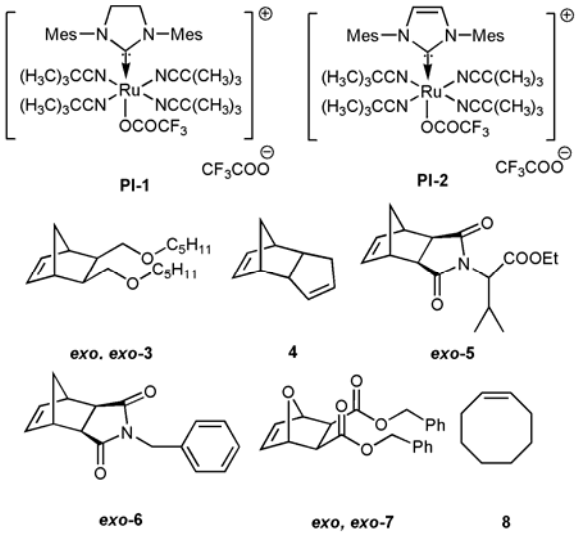


*Figure 2: Structures of **PI-1, PI-2** and monomers **3-8**.*

were in the range of 4×10^4-2.1×10^6 g mol^{-1}; polydispersity indices were in the range $1.2 < PDI < 4.53$. Increasing the energy of the light by switching from 308 nm to a 254 nm Hg-lamp gave raise to virtually quantitative yields for monomers **3**, **4** and **8** (Table 1).

Even the functional monomers **5-7** could be polymerized in high yields, particularly by the action of **PI-1**. In general, initiation efficiencies were low with both PIs. The comparably low molecular weights of poly-**8** with both **PI-1** and **PI-2** are believed to result from significant chain transfer. ^{1}H- und ^{13}C-NMR investigations clearly revealed the ROMP-derived structure of all polymers. As expected, the polymerization of **4** resulted in the formation of crosslinked bulk material. The significantly enhanced reactivity of **PI-1**, bearing the $IMesH_2$ ligand, e.g. in the photopolymerization of **5**, is in accordance with reports on the superior reactivity of $IMesH_2$-based Grubbs type catalysts compared to IMes-based systems [3]. Though we were not able to identify the propagating Ru-carbene species, both the NMR data on the polymer structure and theoretical investigations [2] strongly suggest the formation of Ru^{IV}-based Grubbs-type initiators from both **PI-1** and **PI-2**.

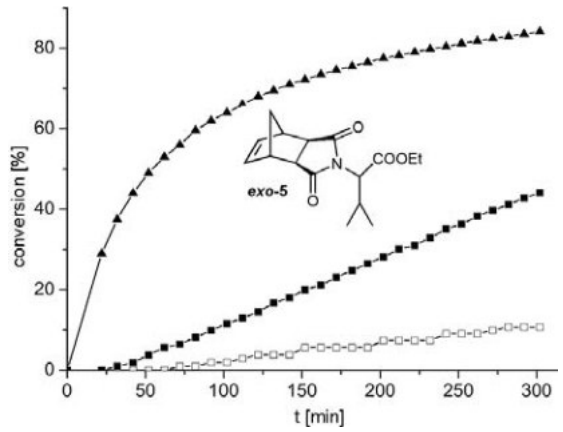


*Figure 3: Polymerization kinetics of **exo-5** by the action of **PI-1** and **PI-2**, respectively, and with different initial irradiation times ▲ 6 min, **PI-1**, □ 3 min, **PI-1**, ■ 6 min, **PI-2**.*

		yield (%)[a]		M_n/PDI[a]
PI	monomer	308 nm	254 nm	254 nm
1	***exo, exo*-3**	40 [b]	95 [b]	$4.8 \cdot 10^5$/1.8
1	**4**	82	99	-
1	***exo*-5**	69	85	$2.1 \cdot 10^5$/1.9
1	***exo*-6**	90	92	$8.8 \cdot 10^5$/1.92
1	***exo, exo*-7**	<5 [b]	90	$2.6 \cdot 10^5$/3.7
1	**8**	33 [b]	99 [b]	40,000/1.2
2	***exo, exo*-3**	41 [b]	92 [b]	-
2	**4**	>99	99	-
2	***exo*-5**	61	67	$4.4 \cdot 10^5$/2.45
2	***exo*-6**	91	90	$8.8 \cdot 10^5$/2.0
2	***exo, exo*-7**	<5 [b]	86	$4.5 \cdot 10^5$/4.53
2	**8**	21 [b]	>99 [b]	49,000/1.8

*Table 1: Polymerization results for monomers **4-8** with **PI-1** and **PI-2**, monomer:initiator ratio = 200:1 [a] in 5 ml of $CDCl_3$, 30 °C/1 h, isolated yield; [b] in 5 ml of $CDCl_3$, 30 °C/1 h, yield determined by ^{1}HNMR.*

Surface coating

It was checked whether the findings found for the solution polymerizations described above could be applied to surface modification. For that purpose, glass plates were coated with a mixture of **4** and **PI-1**, covered with a mask, and subjected to irradiation for one minute. Removal of the mask and unreacted monomer provided fully transparent poly-DCPD coatings (Fig. 4). For the poly-DCPD coating typical contact angles of 95.5° were found, while the parent glass surface showed an angle of 50.7°.

*Figure 4: Poly-**4** coatings on glass prepared by the action of **PI-1**.*

Quantum chemical calculations

On the basis of quantum chemical calculations (B3LYP/LACVP* level and TD-DFT, Jaguar 7.0 program [4]) the following mechanism is proposed. Dissociation of one *t*BuCN in **PI-1** is expected to proceed easily (dissociation energy E_{diss} = 12 kcal mol^{-1}, ΔG = -4 kcal mol^{-1}), while dissociation of the CF_3COO ligand is impossible (E_{diss} = 160 kcal mol^{-1}). Calculations of the electron distribution of the precatalyst PI-1 reveal that the LUMO orbital has strong anti-bonding character between the Ru and the N atoms of the *t*BuCN ligands (Fig. 5 (A) and Fig. 6 (B)), facilitating the release of one ligand in the excited state. This finding is in contrast to another new Ru-complex $Ru(IMesH_2)(CF_3CO_2)_2(CO)_3$ where excitation into non-bonding orbitals occurs (Fig. 5 (B)). In the latter case the release of a CO-ligand in the excited state is not promoted, and the complex does not show significant photocatalytic

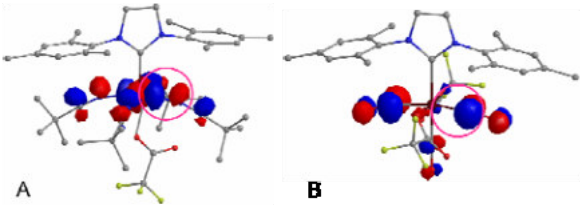


*Figure 5: Calculated LUMO orbitals of **PI-1** (A) and a $Ru(IMesH_2)(CF_3CO_2)_2(CO)_3$ complex (B).*

activity.

The intermediate C in case of **PI-1** (Fig. 6) is stabilized by coordination of the second oxygen of the CF_3COO ligand forming a μ-carboxylato species (Fig. 6) $[Ru(IMesH_2)(CF_3COO)(\mathbf{t}BuCN)_3^+(CF_3COO)^-]$. The next step is likely to be dissociation of a second *t*BuCN-ligand. This reaction is strongly endothermic (approx. 28 kcal mol^{-1}, ΔG = +15 kcal mol^{-1}), however, analysis of the electronic structure of $[Ru(IMesH_2)(CF_3COO)(tBuCN)_3]^+$ reveals that the electron distribution in the LUMO–as in the case of the parent compound–has a strong antibonding character between the Ru and the N atom of one *t*BuCN ligand leading to the weakening of the corresponding bond in the excited (triplet) state (Fig. 6, (D)).

Thus, in agreement with experiments, the possible excess energy for the dissociation of the second

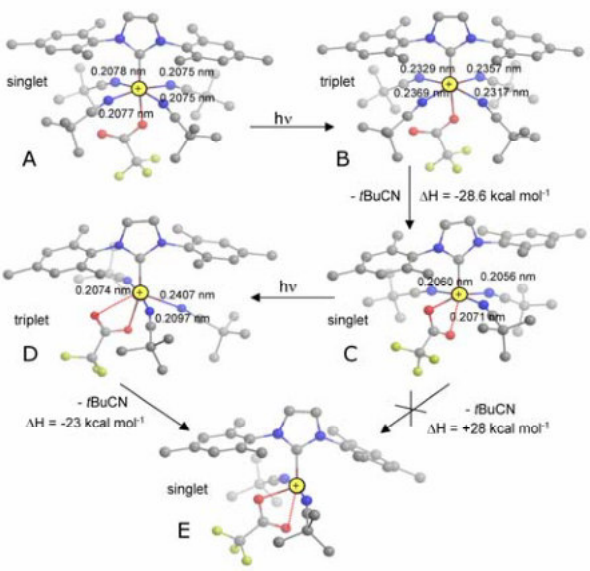


*Figure 6: Structures (H atoms omitted), reaction enthalpies, and reaction scheme of the UV activation of **PI-2**. As can be seen, the Ru–N bonds are essentially longer in both triplet states. Calculated with B3LYP/LACVP*, Jaguar 7.0 program.*

*t*BuCN ligand can only be achieved via excitation of the Ru-complex with high-energy UV-B light. This dissociation is followed by the formation of a stable π-complex with the monomer followed by the formation of the Ru-alkylidene through H-shift within the C=C double bond.[2]

Photolysis experiments

These calculations were supported by laser flash and steady state photolysis experiments which were carried out to investigate the primary reaction steps of the photoactivation of **PI-1**.

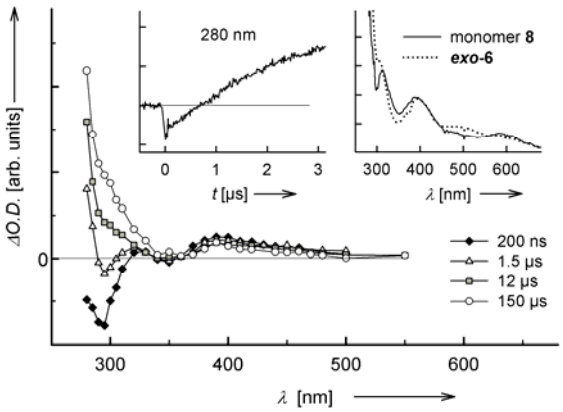


*Figure 7: Laser flash photolysis of a N_2-saturated 2×10^{-4} mol·l^{-1} **PI-1** dichloroethane solution (times as indicated). Insets: formation of the meta-stable transient (left); spectra observed in solutions containing **8** and **exo-6**, 5 min after irradiation (right).*

Immediately after the laser pulse, the depletion of the parent compound was observed (Fig. 7). There was no indication for a long-lived triplet state absorption suggesting a fast cleavage process from either the excited singlet state, a higher excited triplet state or a dissociative *LF (ligand field) state. A short-lived triplet state as suggested by QC calculations below the time-resolution of the laser flash setup (t < 1 ns) cannot be excluded. The delayed formation of a new band in the range of ~30 nm with a very weak shoulder at 350 nm (Figure 7, spectra ≥ 1.5 µs and inset at 280 nm), which did not decay during the time window of the laser flash photolysis (sub-ms), can be related with reasonable care to the formation process of meta-stable (µ-carboxylato) species [Ru(IMesH$_2$)(CF$_3$COO)(*t*BuCN)$_3$$^+$ (CF$_3$COO)$^-$].

Laser photolysis of aerated solutions did not show significant differences in the kinetics or the transient spectra, proving insensitivity towards O_2. In case monomer was added, a completely new band around 400 nm is formed; this formation continued even after the end of the irradiation, and also occurred in case the monomer was added after irradiating a solution of **PI-1**. Since basically the same band at 400 nm (Figure 7, right inset) was observed in the presence of different monomers, e.g. **8** and ***exo*-6**, and is most likely indicative for the catalyst-monomer complex.

NMR measurements on **PI-1** following successive irradiations of the pre-catalyst solution revealed that already at low exposure doses (0.13 J cm^{-2}, 308 nm) the signals of coordinatively bound *t*BuCN decreased and the corresponding signals

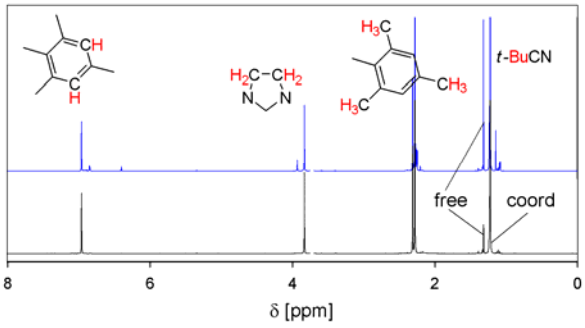
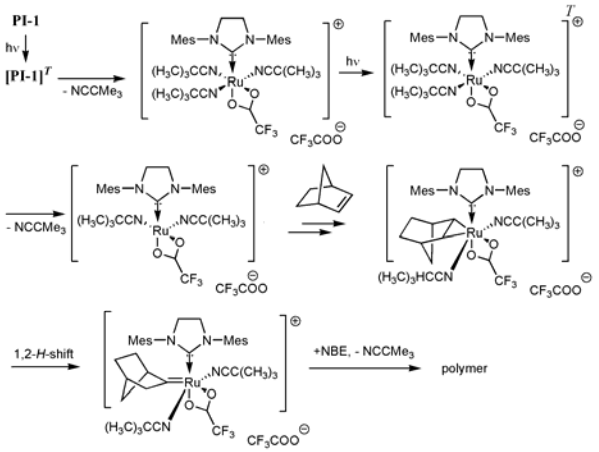


*Figure 8: ^{1}H NMR spectra of a N_2 saturated 2×10^{-2} mol l^{-1} solution of **PI-1** in ClCD$_2$CD$_2$Cl before (bottom) and after (top) irradiation (~1 J cm^{-2}, 311 nm). Corresponding H atoms are marked. Note the formation of free tBuCN.*

of free *t*BuCN increased linearly with irradiation time (Fig. 8). At the same time, the intensities of the other parent NMR signals decreased and new signals appeared which could be clearly assigned to the [Ru(IMesH$_2$)(CF$_3$COO)(*t*BuCN)$_3$]$^+$ species. Quantification of these signals confirmed the QC calculations and suggested in fact removal of only one *t*BuCN upon irradiation and formation of the µ-carboxylato species [Ru(IMesH$_2$)(CF$_3$COO)-(*t*BuCN)$_3$]$^+$. Subsequent photolysis of the latter in presence of monomer leads finally to the active Ru^{IV}-alkylidene complex [Ru(CF$_3$COO)$_2$(IMesH$_2$)L(CHR)] (L=*t*BuCN, monomer).



Scheme 1: Reaction scheme for the formation of the ROMP active species.

Summary

In summary, we have developed truly UV triggerable cationic Ru-based ROMP precatalysts with unprecedented activity and elucidated some key steps of initiation (Scheme 1) [5]. Current investigations focus on systems with improved initiation efficiencies.

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Self-organization on surfaces by low-energy ion beam erosion

F. Frost, B. Ziberi, B. Rauschenbach

Introduction

Today, conventional top-down approaches like lithographic techniques utilizing (UV) photons are very complex and cost-intensive. In contrast, bottom-up techniques based on self-organization phenomena offer an alternative route for the cost-efficient realization of a variety of nanostructures. One of these alternative approaches is the sputtering of surfaces with low-energy ions.

Beside the actual removal of material induced by atomic recoils and the sputtering of atoms from the surface this surface erosion process often results in a pronounced topography evolution, generally accomplished by a kinetic roughening of the surface. Typically, during ion sputtering, the surface of the solid is far from equilibrium and a variety of atomistic surface processes and mechanisms become effective. It is the complex interplay of these processes that either tends to roughen or to smoothen the surface which, finally, can result in a rich variety of surface topographies. Under special circumstances ion beam erosion can, despite the statistical nature of the process, create well-ordered nanostructures on surfaces.

Two prominent examples are the spontaneous formation of well-ordered ripple and dot patterns. Both special cases are of high interest for many potential applications in nanotechnology. For instance, using broad beam ion sources with appropriate beam dimensions, an alternative cost-efficient route exists to produce large-area nanostructured surfaces in a one-step process. In this context, it should be noted that ion beam induced self-organized nanostructures can be observed for a variety of materials (semiconductors, metals, dielectrics), therefore it is a universal process not limited to material classes as self-organization in semiconductor heteroepitaxy. Highly ordered surface structures are promising for quantum dot arrays with specific optoelectronic properties, patterned magnetic media for high-density storage, or passive optical elements based on sub-wavelength structured surfaces. In addition to this mainly technological driven motivation, fundamental studies of the pattern formation mechanisms can gain insight into the behaviour of non-equilibrium processes at surfaces and, furthermore, of complex processes in nature. Therefore, the surface evolution by low-energy ion sputtering comes into focus of extensive theoretical and experimental work, especially in the last three decades.

In this report the current status in the field of tailoring the topography of Si and Ge surfaces at the nanometer and micron scale by low-energy ion beams will be summarized, with the main focus on the unique possibility offered by low-energy ion beams for self-organized pattern formation. Finally a short outlook for current and future work will be given.

Topography evolution of Si and Ge surfaces under ion beam erosion

In general, there are many parameters which can play a crucial role for the formation of nanostructures on the surface, especially if broad beam ion sources are used: beginning with the geometrical parameters of the ion-optical system of the ion source, continuing with the extraction voltages applied on the grid systems, and ending with the parameters that influence the primary ion-target interactions. This compilation of process parameters indicates that there are many degrees of freedom for influencing surface topographies arising under ion bombardment. Without going in details Fig. 1 gives a first impression of the diversity of structures and pattern that can result from the ion beam erosion of Si and Ge surfaces. To get a rough idea, the individual patterns are formed under various erosion conditions where different ion energies (between 500 eV and 2000 eV), different ion species (Ar^+, Kr^+, Xe^+), and ion incidence angles (between 0° and 75°) were used. Partly the ion sputtering was done with simultaneous rotation. On the one hand from Fig. 1 the unique possibilities for tailoring of the surface topography become immediately clear. On the other hand it shows also that there is a big chal-

lenge to solve the puzzle of surface evolution under ion beam erosion.

In a first step efforts have been made to find the key parameters responsible for the various topographies [1]. At the current state the following key parameters were identified: angle of ion incidence, erosion time, ion beam energy, divergence of the ion beam/angular distribution within the ion beam, ion species used as projectile (Ne^+, Ar^+, Kr^+, Xe^+), temperature, and sample manipulation during processing.

In the course of these investigations, it has been found that domains of hexagonally ordered dots were formed for oblique ion incidence (e.g., for 75°) with simultaneous sample rotation but with a higher degree of ordering compared to normal ion incidence [2]. Ordered ripple pattern were observed on Si and Ge surfaces at near-normal ion incidence angles (e.g, at 15°) with a ripple orientation perpendicular to the projection of the incident ion beam onto the surface (i.e., ripple wave vector parallel to ion beam projection) [3]. Typically, in the range from approx. 35-60° the surfaces are stable against topographical changes by ion beam erosion and no structures have been observed for both materials offering that these ion beam parameters can be used for direct ion-beam smoothing of surfaces.

Among the ion incidence angle the influence of ion species on pattern formation was verified. It seems that a minimum mass is necessary to induce the pattern formation. Thus, no pattern formation on Si takes place when using Ne^+ ions. For Ge no pattern can be found when the surface is eroded by Ne^+ and Ar^+ ions. Concerning potential reasons for these findings it has been speculated that highly energetic sputtered target atoms as well as backscattered projectile ions become more important for decreasing ion mass. These sputtered particles might contribute to additional sputtering of peaks compared to valleys and, in combination with further involved smoothing mechanisms, prohibit the evolution of ripples leading to stable and smooth surfaces. The importance of the angle of ion incidence, erosion time, ion beam energy, and divergence of the ion beam will be addressed in more detail in the next paragraph.

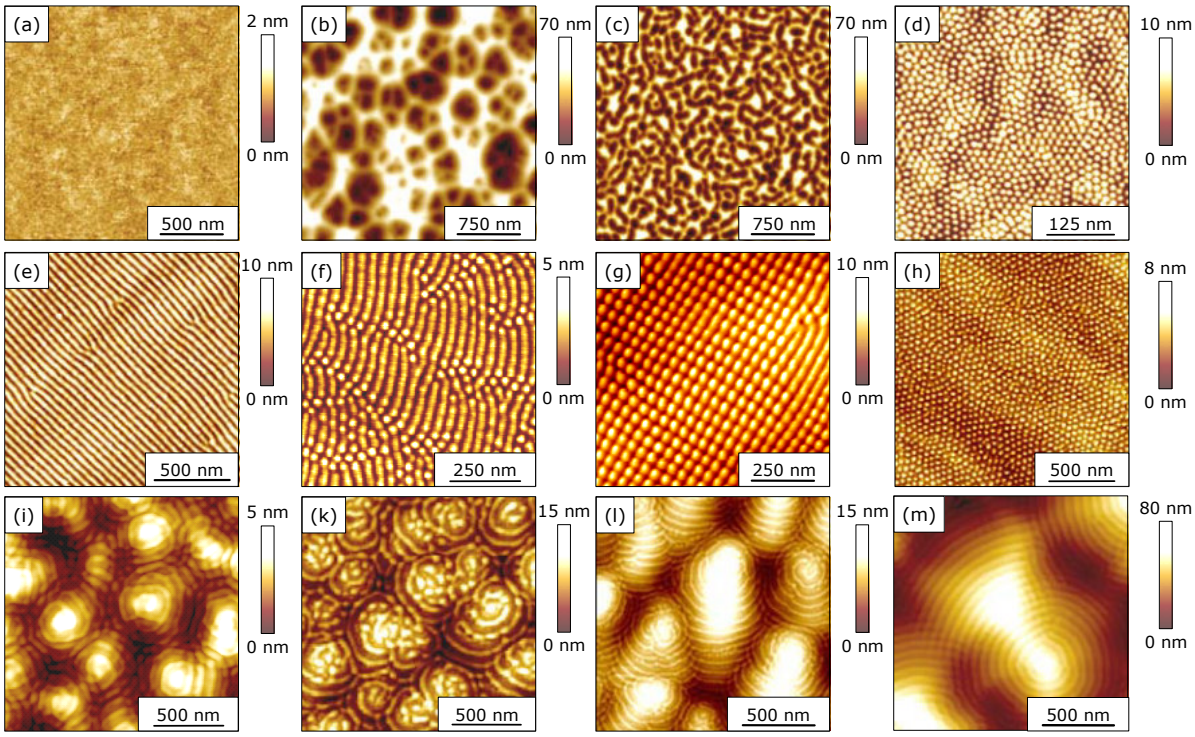

Figure 1: Example of diversity of pattern on Si (a, b, d-g, i, m) and Ge (c, h, k, l) surfaces by low-energy ion beam erosion. The following surface topographies measured by AFM are shown: (a) ultra smooth surface, (b ,c) meshworks of randomly arranged troughs, (d) domains of hexagonally ordered dots, (e) highly ordered ripple pattern, (f) coexistence of dots and ripples, (g) long range square ordered dots on Si, (h) long range hexagonally ordered dots, (i-m) curved ripples. The individual patterns are formed under various erosion conditions.

Control of self-organized pattern formation

As already described, for the given experimental conditions ripple pattern can be formed for ion incidence angles between approx. 5-30° whereas dot pattern occur at 75° with simultaneous sample rotation. For both cases the influence of ion energy and ion fluence on the size of the structures and their ordering were systematically investigated (E_{ion} = 500-2000 eV, ion fluences up to 4×10^{19} cm^{-2}, typical ion flux 1.87×10^{15} cm^{-2} s^{-1}).

Summarizing these studies the most important results are: (i) The ion energy basically determines the size of the evolving ripples and dots. The period or wavelength of the structure increases with ion energy and also their amplitudes. Typically, periods between 30 and 70 nm are measured. The behaviour is in accordance with theory, assuming that non-thermal surface relaxation processes like ion induced viscous flow or ballistic drift mechanisms are dominant. (ii) For both types of patterns (ripples and dots, respectively) the influence of ion energy on the degree of ordering of the nanostructure is negligible. As a measure of ordering the system correlation length was used. (iii) For both types of pattern the wavelength/size of the structures is constant with respect to the ion fluence. In contrast, the amplitude of the pattern exponentially grows until saturation for ion fluences of approx. 1×10^{18} cm^{-2} is observed. The amplitude saturation is in accordance with current models and points to non-linear mechanisms that start to act for this fluence. (iv) Specific for the time evolution of the dot pattern is a saturation of ordering with increasing ion fluences equivalent to the saturation of the size of the individual domains. In contrast, the defect density in the ripple pattern decreases continuously. For the highest fluence applied in this work (4×10^{19} cm^{-2}, corresponding to a total erosion time of 6 h) the pattern with a ripple wavelength of 50 nm has an average defect density of approx. 1 defect/μm^2. A characteristic example for the temporal evolution of a ripple pattern on Si (Kr^+, E_{ion} = 1200 eV, α_{ion} = 15°, j_{ion} = 300 μAcm^{-2}) is shown in Fig. 2.

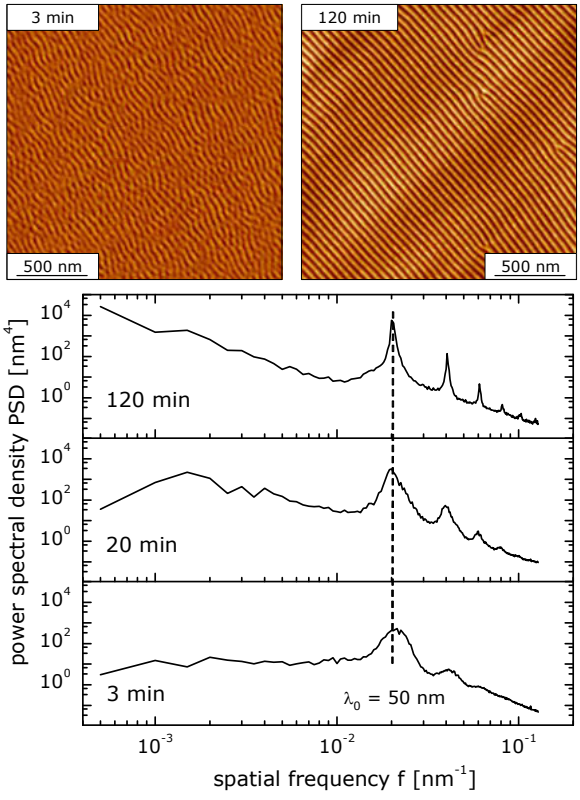

Figure 2: Temporal evolution of ripple pattern on Si. The two AFM images show the ripple pattern after erosion times of 3 min and 120 min, respectively. The number of imperfections is reduced from more than 200 to 6. From the PSD graph it is deduced that the ripple period is constant with time and the ordering improves with erosion time, which can be seen by the reduced width of the first order peak and the higher number of harmonics which becomes visible.

There are two parameters of the sputtering process that can contribute significantly to the achievement of a long range ordering. These are the ion incidence angle and the secondary ion beam parameters [4, 5]. So it was found that small step variations of the ion incidence angle can induce a transition from ripples to dots with increasing ion incidence angle. Additionally, the evolving dots have a large scale ordering, i.e., a spatial correlation over the whole irradiated sample area. In this context the specific role of beam divergence and the angular distribution of ions within the ion beam on the surface evolution have been explored. These parameters, neglected up to now in the studies for nanostructuring with ion beams, play a crucial role in surface evolution processes.
The angular distribution can be controlled by changing the voltages applied to the geometrically defined ion optical elements of the broad beam ion source. For the given experimental set-up, the voltage U_{scr} applied to the screen grid determines

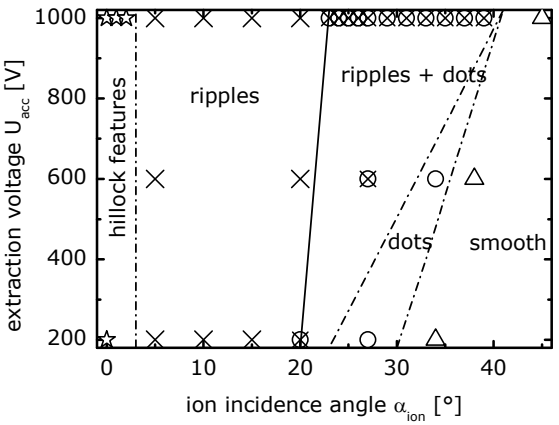

Figure 3: Topography diagram for 2000 eV Xe^+ ion beam erosion of Si surfaces for different acceleration voltages U_{acc} and ion incidence angles α_{ion}. The symbols denote different patterns obtained for different pairs of (α_{ion}, U_{acc}): ☆, hillock structures; ×, parallel mode ripples ⊠, co-existing ripples and dots ○, dots; △, smooth surfaces.

the energy of ions. While the voltage applied to the accelerator grid U_{acc} mainly controls, in a well defined range depending on the grid geometry as well as plasma parameters, the angular distribution under which the ions leave one aperture of the grid. The consequence of this ion beam parameter on the experimentally evolving pattern can be summarized in a so-called topography diagram [5]. The diagram in Fig. 3 shows the variety of different patterns formed for different pairs of variates (α_{ion}, U_{acc}) and the transitions between the patterns. The results clearly demonstrate the importance of the angular distribution of ions, that means, the local angle under which the ions hit the sample surface. In this way, the variation of U_{acc} can be roughly considered as a fine adjustment of the average ion incidence angle which is effective. Consequently, these secondary ion beam parameters that are inherent to all broad beam ion sources can be used as an additional parameter during the sputtering process for controlling the resulting surface topography.

Current and future activities

Current and future activities in this research area are integrated in the DFG research unit 845 „Self-organized nanostructures by low-energy ion beam erosion“ started in 2007 [6]. Regarding IOM activities this research program focuses on the experimental exploration of fundamental processes, the combination of conventional lithographic techniques with patterning by self-organization, and to the application for nanostructured optical surfaces. Thus, the complex relationships of the ion beam induced pattern formation on Si and Ge model surfaces will be considered including a classification of all erosion patterns as well as the detailed investigation of the transition between the patterns. In a further sub-project guided self-organization processes will be addressed by the combination of ion beam induced self-organization with conventional lithographic techniques. Form this work an improved positional control, an enhanced ordering of the nanostructures, and the formations of new types of patterns are expected. From future exploration on this topic we also expect to gain new insight on the process of pattern formation itself and, furthermore, potential applications in micro- and nanooptics, e.g., in bio-inspired functional nanooptics. Therefore, in a third project the potential application of the process for the realization of anti-reflection surfaces in the VUV spectral range based on sub-wavelength structures will be evaluated.

Acknowledgment

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Architecture of nano- and microdimensional building blocks (FOR 522)

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Introduction

Within the DFG research unit 522, "Architecture of nano- and microdimensional building blocks", the IOM is working on two projects with complementary approaches. While laser-beam patterning of ultrathin films in the project "Fabrication of nanostructures by diffraction mask projection laser ablation (DiMPLA)" represents a top-down approach, glancing angle deposition (GLAD) technique employed in the project "Constructive fabrication of three-dimensional, periodic structures using ion beam assisted deposition" is a bottom-up process. DiMPLA can provide templates for various nanostructure growth techniques including GLAD and ion-beam assisted molecular-beam epitaxy.

Diffraction mask projection laser ablation

Highly ordered nano particles attached to a substrate allow for manyfold applications. Using the particles as seeds for 1D (nanowire) or 2D (nanoporous film) growth is as promising as taking advantage of field enhancing or absorbing effects of metal particles with dimensions in the few-nanometer range. DiMPLA is a fast, versatile, and precise method to create such nanostructures [1-4].

The process can be split up into two main preparation steps [3, 4]. The first is to deposit an ultrathin (mostly metallic) film onto a suitable substrate that is in the second step structured by a laser ablation process.

The deposition of thin films for DiMPLA is mainly done by magnetron sputtering. Magnetic materials like Ni are deposited by electron beam evaporation, instead. The thicknesses of the metal films, ranging from about 6 to 15 nm [4], must be in the order of the optical skin depth of the material for the laser wavelength the sample is afterwards illuminated with. By X-ray reflectometry (XRR), the thin-film thickness can be determined precisely. Compactness and roughness of the films are investigated by scanning electron microscopy (SEM) and atomic force microscopy (AFM) as these parameters have a major impact on the mechanism of nanoparticle formation [4].

The second main step of preparation is to structure the thin film (metal henceforth exemplary). For this purpose, it is illuminated by a laterally varying intensity pattern of a KrF excimer laser beam (wavelength $\lambda = 248$ nm, pulse length $\tau \approx 30$ ns). Figure 1 shows the calculated intensity pattern that is used for most of the experiments described here.

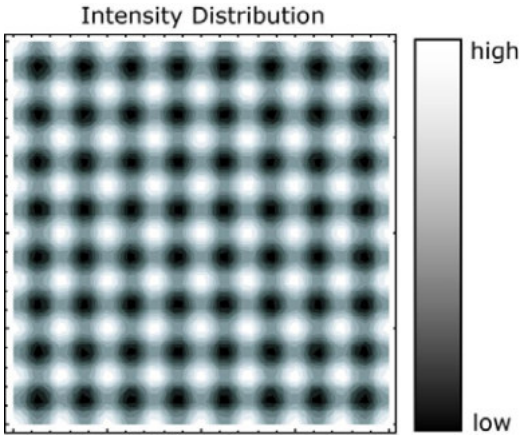

Figure 1: Calculated lateral intensity distribution of the laser beam.

The lateral intensity distribution is obtained by sending the excimer laser beam through a phase mask, where it is diffracted. The first orders of diffraction that are enhanced because of the special mask design [4] are used further while the higher orders and the 0^{th} order are blocked by geometrical arrangements. A reflective objective is used to demagnify the pattern imposed by the mask and project it onto the sample. Figure 2 shows a schematic sketch of the whole set-up.

Materials constituting the sample have to be chosen appropriately [4]. The substrate must have a higher ablation threshold than the film. In this case, the average fluence of the laser beam can be

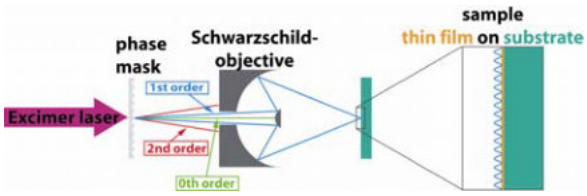

Figure 2: Schematic sketch of the DiMPLA set-up. The laser beam is diffracted by the phase mask and only the first orders of diffraction are used further. The reflective objective demagnifies the pattern that is then projected onto the thin film.

adjusted such that intensity maxima exceed the ablation threshold of the film but not of the substrate and the minima are below both thresholds. There is more than one combination of film and substrate material that fulfils this condition. One combination, that turned out to give decent results for well-ordered nanostructures in terms of successful formation and homogeneity, is a thin gold film on a sapphire substrate.

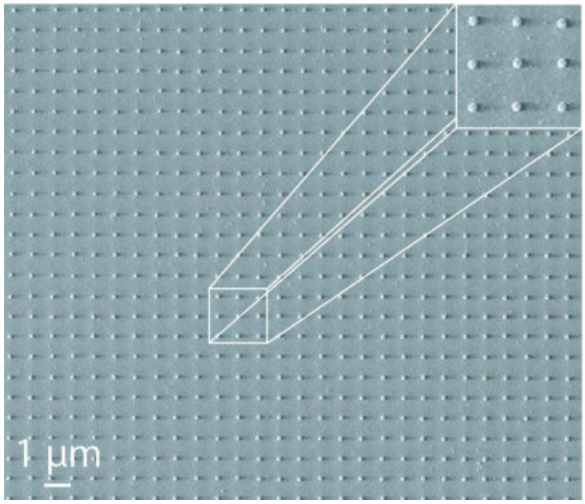

Figure 3: Top view of gold dots on sapphire made with DiMPLA. The micrograph shows only a small section of the 100 x 100 µm² area that is patterned at once with a single laser pulse.

In Figure 3, a plane-view SEM micrograph of Au nanoparticles on a sapphire substrate that were made with the DiMPLA technique is depicted. Materials combinations that also show formation of well ordered nanostructures include titanium on sapphire, nickel on sapphire, aluminium on fused silica [4], or copper on fused silica [4].

The formation process of the structures is not fully understood yet. It is assumed, however, that film material is removed from the substrates at positions of the maxima in intensity (ablated volume of material over the whole patterned area is calculated to be about 60 %). Driven by surface tension, the molten gold then forms structures of minimized surface energy. Cross-sections of the samples, however, reveal that not only the thin film is affected by the laser illumination. In the near-surface region of the single crystalline substrate, a thin amorphous layer enclosing the gold nanospheres is formed. The transmission electron micrograph (TEM) in Figure 4 shows a cross-section of gold spheres embedded in the amorphous alumina layer.

Two different hypotheses on the formation process are made considering this situation. The first

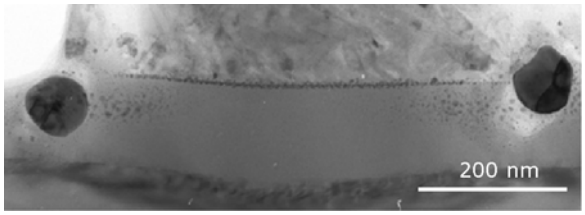

Figure 4: Cross-section of gold dots, embedded in amorphous sapphire, that were originally thought to sit on top of the still crystalline sapphire substrate.

is that surface Plasmon-related field-enhancing effects occur during the patterning process. The gold film might–while melting–form structures of small sizes that subsequently enhance the illuminating field such that the sapphire underneath can be molten. According to another approach, thermal effects play a key role. The temperature rise in the gold film during the ablation process is very high. Hence, the gold film becomes hot enough to melt the top layers of the sapphire substrate. Due to surface tension effects the two molten liquids then form the structures that can be seen in Figure 4 and rapidly solidify.

There are manyfold applications for the well-ordered nanostructures presented here. A first application is to use them as templates for other nanostructure growth techniques. Well-ordered structures created by GLAD on a DiMPLA pre-patterned substrate are described in the following section where it is shown that GLAD structures adopt the arrangement of gold dots on sapphire.

Nano-porous ZnO thin films grown by pulsed laser deposition (PLD) on DiMPLA treated substrates are shown in Figure 5. Even though the

substrate did not show well-ordered nanodots, but rather statistically arranged gold dots with an average diameter of about 2 to 3 nm, the highly porous thin film only grew on the pre-patterned parts of the gold film.

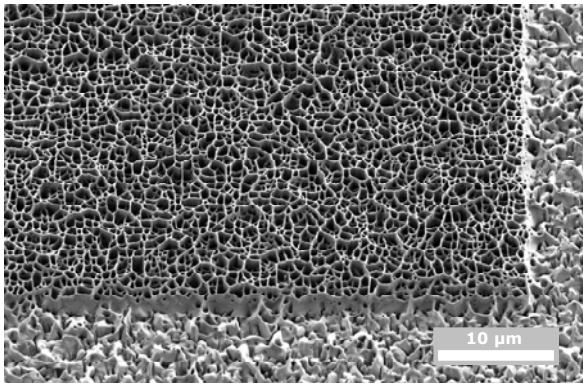

Figure 5: ZnO nanowalls grown on a DiMPLA patterned template.

Glancing angle deposition

In usual vacuum deposition processes like sputter deposition, evaporation, or pulsed laser deposition, the flux of particles that contribute to the growing film reaches the substrate perpendicular to the substrate surface, resulting in a dense, compact film. However, in the glancing angle deposition (GLAD) process, the particle flux strikes the substrate under a highly oblique angle β to the substrate normal. Under such deposition conditions, the first nuclei that form in the beginning of the growth process act as obstacles for the subsequent particle flux which is captured by those seeds and, consequently, does not reach the shadowed region behind the seed any more. By this, a highly underdense sculptured thin film (STF) that consists of needle-like structures, slanted in the direction of the incoming particle flux, is generated (Figure 6). In combination with a suitable substrate rotation mechanism, these needles can be sculptured to form nearly arbitrary shapes like nanocolumns, spirals, screws and chevrons [6,7].

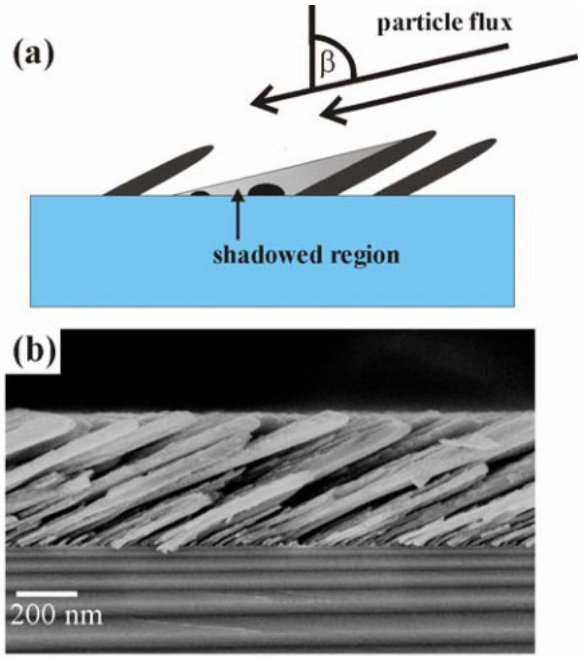

Figure 6: Principle of GLAD without substrate rotation (a) and SEM micrograph of Si needles grown on a Si substrate with GLAD (b).

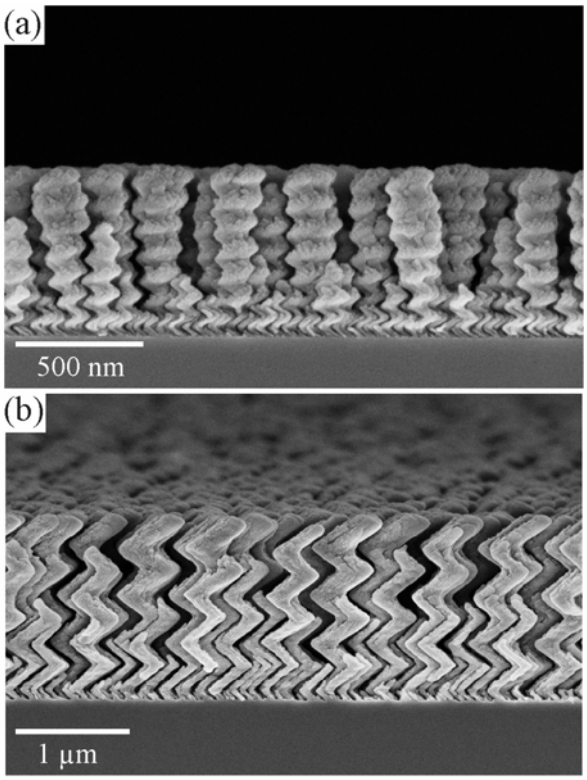

Figure 7: Si screws are grown with continuous substrate rotation (a) and chevron-like structures with symmetric stepwise substrate rotation of 180° (b).

At the IOM, the growth of sculptured Si nanostructures is done by means of ion beam sputtering, with $\beta \approx 85°$ and a computer-controlled substrate rotation. Examples of different Si GLAD nanostructures can be seen in Figure 7. SiO_x and Si/SiO_x multistructures were achieved by growth in oxygen atmosphere during the growth process

or parts thereof. An example of a multilayered STF is shown in Figure 8.

On conventional planar substrates, a competitive growth regime due to self-shadowing of adjacent nanostructures (evolutionary selection) can lead to the extinction of some of the structures.

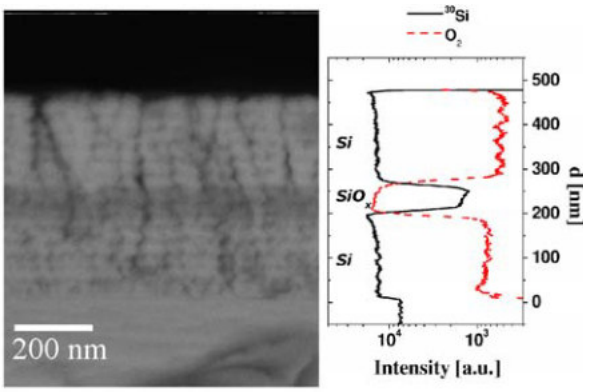

Figure 8: Backscattered electrons micrograph of a screw-like multilayered STF and the corresponding TOF-SIMS composition depth profile.

Using patterned substrates, however, this evolutionary selection can be delayed when the distances between artificial seeds for GLAD growth and seed height are both properly chosen. By this, GLAD provides a method to grow periodically arranged nanostructures with distinct structure diameters and inter-structure distances in a bottom-up process [6]. Periodically arranged seeds for GLAD of differently shaped nanostructures can be prepared with various patterning techniques such as electron beam lithography (EBL) and nanosphere lithography (NSL). DiMPLA proved to be an appropriate patterning technique to provide artificial seeds for the growth of periodically arranged nanostructures, too (Figure 9).

The ability to tailor both diameter and inter-structure-distances of arbitrary shaped nanostructures can lead to interesting applications in many fields. Size-exclusion chromatography and filters for nanoparticles with distinct porosities could be possible with GLAD grown films. A high application potential can be found in the field of optics, where GLAD applications span a wide range including photonic band gap materials and polarizing filters. Additionally, due to the very high surface-to-volume ratio of GLAD sculptured thin films, sensoric applications are possible.

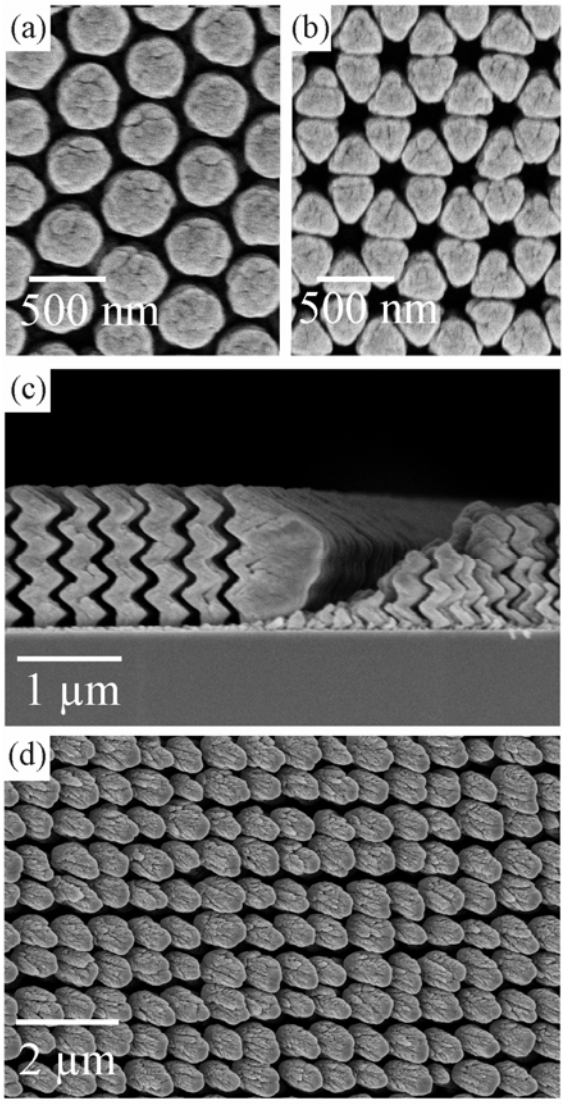

Figure 9: Plane-view micrographs of cylindrically shaped Si nanocolumns on a hcp-arranged template (a) and of Si nanocolumns with triangular cross section on a honeycomb-like arranged template (b). Transition zone between periodically arranged Si chevrons on an EBL patterned substrate and the unpatterned part of the substrate (c). Plane-view of chevrons on a DiMPLA patterned substrate (d).

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Plasma immersion ion implantation of Ti alloys for medical applications

D. Manova, J. Lutz, S. Mändl

Introduction

During the last decades, a paradigm shift has been occurring in medicine. Infectious diseases are on the retreat with some close to extinction, which results in an increased lifetime. Coupled with an aging population and a demand for an increased quality of life, this leads to different quality of demands on medical care. Nowadays, the main causes of death in developed countries are cardiovascular diseases, cancer, heart attacks, non-communicable diseases, and road accidents.

Presently, a third generation of smart biomaterials designed to stimulate specific cellular responses is under development and testing. The challenge for the next generation of materials is a further combination of properties, including stimulation of autointegration, biomechanic in vivo characteristics, growth responsive, and adaptive as well as individualization for specific traits. However, biocompatibility itself is still a concept easy to understand and difficult to define, necessitating further fundamental research on surface interactions.

Several modes of interaction are identified, albeit it is not possible to give a succinct description of their respective mechanisms: (i) surface roughness on the µm- and nm-scale, determining the adhesion of cells and receptor molecules, (ii) electronic density of state at the surface responsible for electron transfer and the distribution of the electrical potential, both effects which may interrupt normal cell behavior, (iii) outdiffusion of metallic cations leading to toxic effects and apoptosis in the surrounding tissue, especially critical for Ni-containing metals, (iv) generation of wear particles and their transport, e.g. in macrophages towards a final agglomeration in the lung and spleen.

Going one step further towards materials science, the microstructure and chemical composition of the surface exposed to a biochemical environment is the influencing factor. By a thermochemical modification using energetic ions additional influences as ion energy, temperature evolution with time, surface sputtering, and ion flux rate will come to importance. Using oxygen and nitrogen plasma immersion ion implantation (PIII) at elevated temperatures, it is possible to improve the biocompatibility of titanium alloys [1]. The underlying physical processes include diffusion, phase formation, and–as a result–modified mechanical properties of the resulting surface layers. Furthermore, an enhanced osseointegration is observed as an increased abiotic mineralization after PIII treatment is observed.

Experiment

For fundamental investigations, flat polished coupons were used in the experiments with Ti grade 2 (Ti > 99.2 wt.%, O ≤ 0.25 wt.%), Ti grade 5 (Ti_6Al_4V with 6 wt.% Al and 4 wt.% V), SM 495 (54.5 wt.% Ni, 45.5 wt.% Ti) and SE 508 (55.8 wt.% Ni, 44.2 wt.% Ti) as base materials. Additionally, total hip replacements (THR) consisting of pure Ti were treated with PIII.

The ion implantations were carried out in a HV chamber with a base pressure better than 10^{-4} Pa at a gas flow of 50 sccm (either nitrogen or oxygen), resulting in a working pressure of 0.2 Pa. Plasma generation by an RF source operating at 40.68 MHz resulted in a plasma density around $6\text{-}10 \times 10^9$ cm^{-3} and an electron temperatures of 1.0-1.5 eV, depending on the gas. The high voltage pulses with a rise time of less than 0.5 µs and a length of 15 µs were varied between 5 and 30 kV across the presented experiments, resulting in incident ion fluences between 1.75 and 3.75×10^{11} atoms/cm^2 per pulse.

The phase composition was studied by X-ray diffraction (XRD) in Bragg-Brentano geometry using Cu K_α radiation. The elemental concentration distributions were measured with elastic recoil detection analysis (ERDA) using $^{197}Au^{15+}$ ions at energies around 200 MeV [2]. Additionally, secondary ion mass spectrometry (SIMS) was performed using a time-of-flight set-up with Ga^+ as primary beam for analysis and O_2^+ or Cs^+

as secondary beam for depth profiling and enhancing the secondary ion yield.

The wear tests on coupons were performed using a rotating ball-on-disc configuration with an alumina ball, diameter 0.476 cm, in continuous sliding contact. A track diameter of 0.65 cm and a rotation speed between 50 and 200 rpm translates into a speed of 1.7-6.8 cm/s. The applied loads were between 0.66 and 9.07 N, corresponding to contact pressures of 0.42-1.0 GPa [3]. The wear investigation of the THRs was performed in a proprietary hip simulator against a bone cemented with and without zirconia additions with the wear debris collected and analyzed [4].

Simulated body fluid (SBF), a useful tool to predict the in-vivo bone bioactivity, was prepared according to the standard protocol suggested. Titanium samples with a titania surface formed by PIII were immersed in SBF, kept at a constant temperature of 37 °C for 48 h in these preparatory experiments. The same procedure was used for pure Ti samples [5].

Results & Discussion

Fig. 1 gives an overview on selected oxygen or nitrogen depth profiles in different materials. The data for Ti_6Al_4V were taken with SIMS, the others with ERDA. For all low temperature implantations (265-400 °C) sharp box-like profiles of the oxygen were found coinciding with the implantation range calculate by TRIM. This indicates high fluences beyond sputter saturation without any additional diffusion of the implanted atoms.

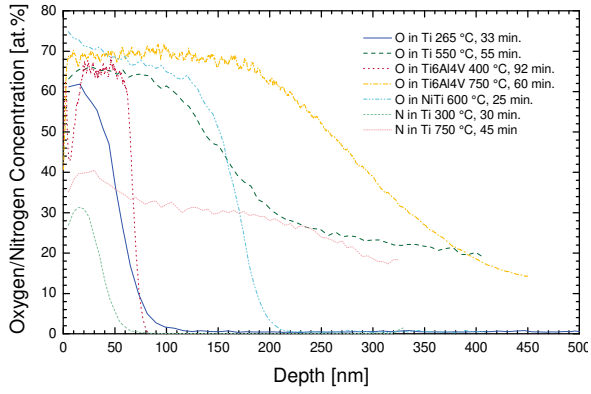

Figure 1: Oxygen and nitrogen depth profiles after implantation into Ti, Ti_6Al_4V or NiTi at different temperatures.

With increasing temperature, three different regimes, depending on the ion species and the base material, have to be distinguished.

For NiTi, box-like profiles are found, independent of temperature, which is indicative of a mass transport of Ni from the surface towards the bulk, leading to a TiO_2 surface layer without any Ni at the surface and an enriched Ni_3Ti layer below the surface [6,7].

In contrast, diffusion of nitrogen in titanium at temperatures beyond 300 °C leads to the evolution of profiles which closely resemble complementary error functions obtained in diffusion theory [8]. No transport of Ti is observed in this case.

Oxygen in Ti and Ti_6Al_4V leads to a mixture of cation and anion transport with oxygen diffusion towards the bulk and titanium diffusing towards the surface. Thus, a closed titania compound layer is formed at the surface, followed by a diffusion layer with interstitial oxygen within the titanium matrix [9].

The corresponding diffusion constants for the oxygen implantations are shown in Fig. 2. The obtained absolute values depend strongly on the composition and structure of the base material while the activation energies are typical for vacancy and interstitial diffusion within metallic and intermetallic solids. It has to be pointed out that for all systems no influence of the ion energy on the growth rate was found, hence radiation enhanced diffusion could be ruled out.

Despite the consistent diffusion mechanism across the whole temperature range, a strong influence of

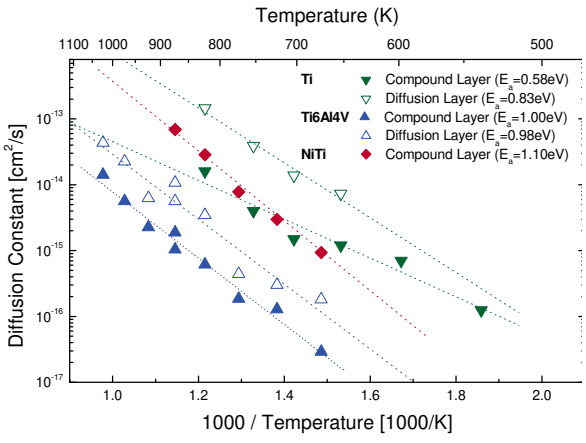

Figure 2: Resulting diffusion constants for oxygen implantation. Please note that no diffusion layer is observed for NiTi.

the temperature on the phase formation, in addition to the microstructure, was observed in some systems. Oxygen implantation into NiTi and pure Ti always leads to the formation of rutile, as shown with Raman spectroscopy independent of the temperature and the heating regime. Oxygen in Ti_6Al_4V leads to mixture of anatase and rutile, together with the formation of alumina at temperatures beyond 600 °C. When the samples are preheated to 400 °C before starting the ion implantation, anatase, the metastable low temperature phase, is favored against rutile while rutile is more pronounced without preheating.

A similar effect is observed during nitrogen implantation into pure Ti, as shown in Fig. 3. Starting the implantation at room temperature leads to the parallel formation of δ-TiN and ε-Ti_2N, as observed by X-ray diffraction. In contrast, additional external heating with a start of the ion implantation at 350 °C results in the dominance of Ti_2N over TiN. The nitrogen surface concentration is in all cases 30-35 at.%. Different activation energies and kinetics were found for the processes (1) Ti → ε-Ti_2N, (2) Ti → δ-TiN and (3) ε-Ti_2N → δ-TiN, with the activation energy increasing from (1) to (2) and (3) [8].

Investigating the microstructure and surface topography of these samples after PIII, no large differences are found between the systems. For Ti + O, the grain size increases from 10-30 nm at 265 °C to 40-100 nm at 550 °C, while elliptical grains with a similar size evolution are observed for Ti_6Al_4V + O. For NiTi, an increase of the grain size from around 40 nm at 400 °C towards around 100 nm at 600 °C is measured.

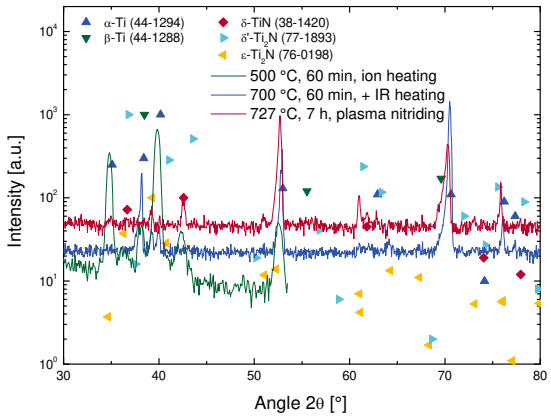

Figure 3: XRD spectra for nitrogen implanted titanium.

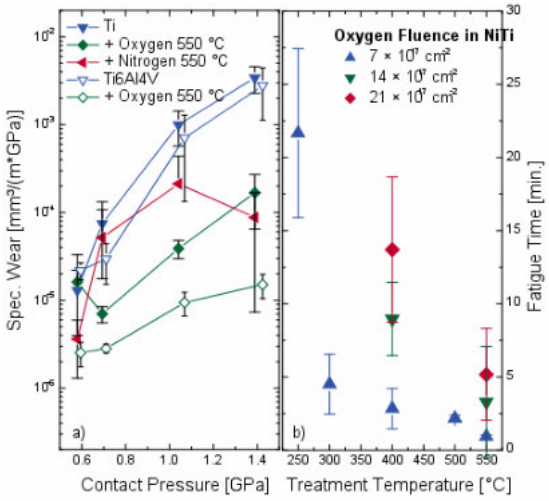

Figure 4: a) specific wear and b) fatigue time for oxygen implantation at different contact pressures, implantation temperatures and ion fluxes.

Correspondingly, influences of the treatment time, the temperature, and the heating regime should lead to different mechanical properties as mediated by the layer thickness, phase composition, and microstructure. Detailed investigations are available for oxygen implantation into Ti, Ti_6Al_4V and NiTi. Using a rotating ball-on-disc test, no significant difference in the wear rate of untreated Ti and Ti_6Al_4V was found (see Fig. 4a), whereas oxygen implantation leads to a wear reduction of two orders of magnitude for the former material, increasing to 2.5 orders of magnitude for the latter one. In contrast, nitrogen ion implantation leads even to lower reductions of the specific wear at low load, while values similar to those of the oxides were found at the highest contact pressure of 1.4 GPa.

For Ti_6Al_4V, the wear rate is independent on the temperature and ion flux for temperatures up to 400 °C, despite a different layer thickness. Beyond that, significantly reduced wear rates were observed with higher temperatures and ion fluxes. As oxygen implantation into pure titanium leads to a reduction of the wear by a factor of three when the ion flux is increased by the same factor, a strong influence of the layer thickness should be responsible as the diffusion in Ti_6Al_4V is slower than in Ti. However, as no temperature effect was found at all for Ti, the microstructure must have an additional influence, especially as thinner lay-

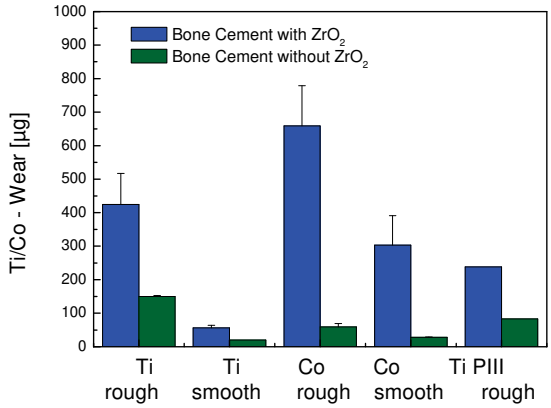

Figure 5: Generation of wear debris as a function of surface condition and bone cement composition.

ers on Ti_6Al_4V have a lower wear rate than thicker layers on pure Ti.

As the fatigue time of oxygen implanted NiTi, as depicted in Fig. 4b, actually increases with decreasing implantation temperature and increasing ion fluence, a complex interplay of adhesion problems due to the sharp interface between the rutile surface and the NiTi intermetallic base material, coupled with thermal stress annealing can be proposed in this system.

Using a more realistic wear test in a hip simulators (cf. Fig. 5), a reduction of the wear volume by one third was found in modular hip implants, whereas a value of 75% was found for implants tested against bone cement. Here, a strong influence of the layer thickness is observed as the wear rates depend on the total time. When the layer is removed, the initial wear rate of the base material is observed.

The next logical step for in vitro testing should involve mechanical characterization in SBF. Results of preliminary experiments show that titania surfaces formed after treatment of pure Ti with oxygen PIII exposed to SBF at elevated temperatures of 37 °C allow to a fast and extensive formation of hydroxyapatite nanoparticles on the surface in an abiotic environment [1]. Consequently, the improved biocompatibility of these PIII treated surfaces can be ascribed to this biomimetic surface functionality. The elemental composition and phase characterization are supported by SIMS, XRD and EDX data. Control experiments with pure titanium including a native oxide layer on the surface yielded negative results concerning the hydroxyapatite formation.

PIII treated titanium and NiTi implants were tested in rats and dogs with the final aim to introduce them into maxillofacial surgery, e.g. as osteosynthesis plates or autocompression braces, in collaboration with the Klinik rechts der Isar in Munich and the Kantonsspital in Basel.

In these environments, which are characterized by a lower number of loading and unloading cycles than THRs, the wear rate is much more reduced. After oxygen PIII treatment, considerably higher pull-out forces were found for Titanium and NiTi, together with a much improved osseointegration. At the same time, the surrounding tissue reaction to NiTi implants, as measured with fluorescence markers, is reduced by about 50%.

Summary

Oxygen and nitrogen PIII of Ti, Ti_6Al_4V and NiTi lead to the formation of hard and wear resistant surface layers with the phase formation and diffusion depending on the process conditions. Subsequently, the mechanical properties, as determined by wear tests, correlate with the final microstructure of the samples. Successful applications have been shown in animal models with osseointegration plates more suitable than replacements for articulating joints.

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Surface modification of nano-sized zeolites

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The transport of molecules through the channels of zeolite catalysts often limits the process efficiency. To reduce transport resistance and diffusion path length, nano-sized zeolites can be applied, thus facilitating the accessibility of molecules to the internal zeolite surface. However, unselective sites present on the external surface area of zeolite crystallites should be inactivated by post-synthesis modifications to avoid secondary transformation of target products. The effects of surface modification by chemical liquid deposition of organosilicon compounds and the pre-coking technique on selectivity and reaction mechanism of isomerisation of n-butene over HZSM-5 and HFER have been studied [1].

Skeletal isomerization of n-butene over pre-coked HFER resulted in similar yields of isobutene and undesired pentenes (Fig. 1), i.e., coke residues formed by pre-coking do not increase isobutene selectivity as proposed by the pseudo-monomolecular reaction pathway. For silanized HFER and HZSM-5 (not shown), a significantly lower isobutene selectivity was obtained. These findings are consistent with results from sorption measurements which revealed that silanization treatment by hydrolyzed TEOS leads to a reduction of pore size whereas the pre-coking treatment hardly affected the pore size distribution. The simultaneous formation of ethene, propene, cis-/trans-2-butene, iso-butene, and pentenes showed the predominance of bimolecular reactions during n-butene conversion over HFER containing many protonic sites ($SiO_2/Al_2O_3 = 20$). For detailed mechanistic studies, trace amounts of expected intermediates, such as dodecene (Fig. 2), were added to the n-butene feed as ^{14}C-labeled substances. Thus, Radio-HPLC results revealed the incorporation of dodecene carbon atoms into all other olefins during n-butene transformation over HFER, even into long-chain species (see arrow). It is apparent that oligomerization and cracking reactions yield an intense ^{14}C scrambling between all olefinic molecules. Therefore, C_3 and C_5 olefins should not exclusively be considered as cracked products of C_8 intermediates but rather as components of an olefinic pool [2]. On strongly acidic HFER, the distribution of olefinic products is, therefore, controlled by both the zeolite pore structure and the thermodynamic conditions. In summary, surface modifications have an impact on isobutene selectivity of nano-zeolites via variations of zeolite properties rather than via changes in the reaction mechanism.

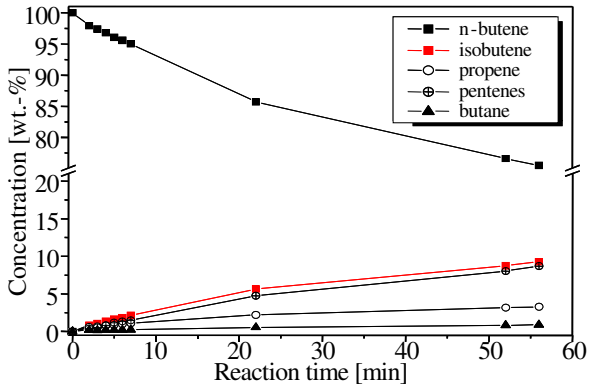

Figure 1: Effect of reaction time on yield during skeletal isomerization of n-butene over pre-coked HFER at 300 °C.

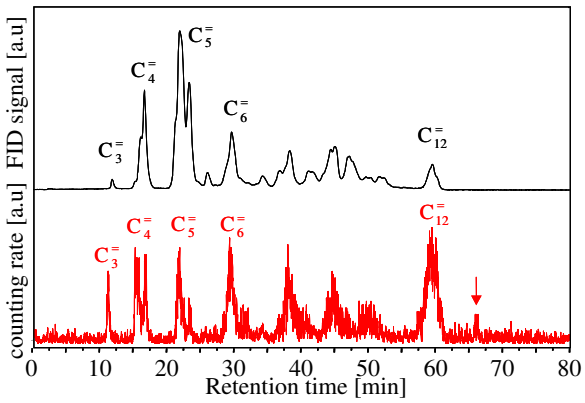

Figure 2: Radio-HPLC chromatogram of ^{14}C-dodecene/n-butene feed after isomerization on HFER at 350 °C and 9 g/(g_{cat}·h).

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Nanocorundum based nanocomposites

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Organic-inorganic hybrid materials have received significant interest in the past few years. Improved material properties including scratch and heat resistance, stiffness as well as gas barrier effects have been reported for nanocomposites formed from inorganic nanoparticles accommodated in a polymeric matrix. In this regard, amorphous aluminium carboxylate nanoparticles were checked for their potential as precursors for nanocorundum powders prepared by a high-temperature conversion process [1].

ALMAL particles were prepared from aluminium 2-propoxide and maleic acid at room temperature whereas their thermal conversion at 1100 °C in the presence of air resulted in the formation of nanocorundum. SEM micrographs revealed that both particulate species were apparently of the same geometrical size and displayed the same aspect ratio as well as spherical particle morphologies. A detailed analysis of the size distribution has been accomplished on the basis of

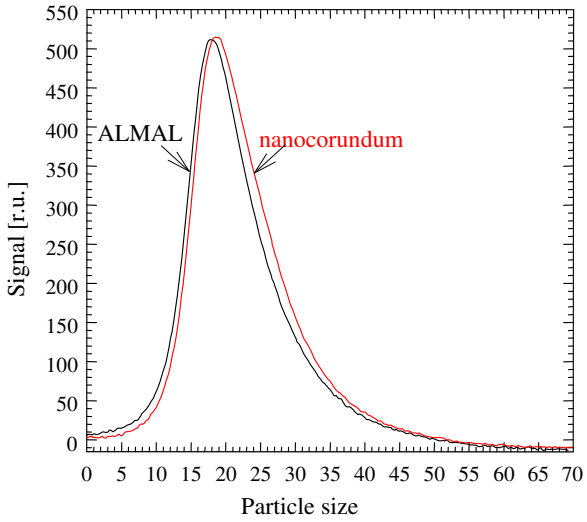

Figure 1: Size distributions of ALMAL and nanocorundum powder as determined by ultracentrifugation.

Stokes' sedimentation law using a disc centrifuge. The size-separated particle fractions showed uniform distributions for both ALMAL and nanocorundum with size maxima at approximately 18 and 19 nm, respectively (Figure 1).

The abrasion resistance of nanocorundum powders was determined by Taber abraser tests shown in Figure 2 which reveal the superiority of the ALMAL/microcorundum (Plakor 15) modification over pure ALMAL/nanocorundum. However, further enforcement by post-tempering and by a

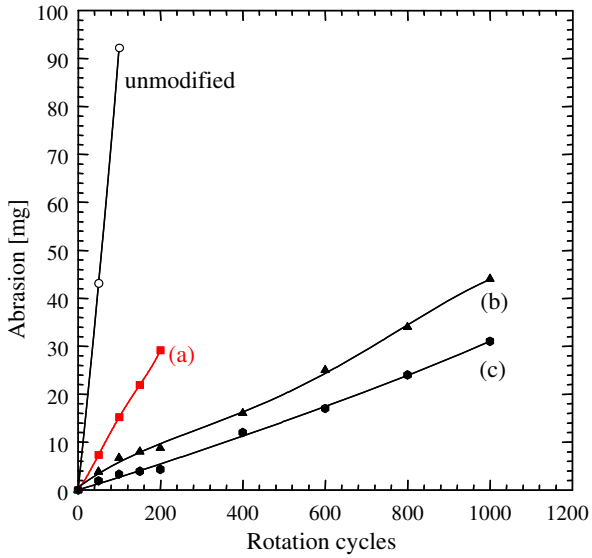

Figure 2: Abrasion for composite coatings after post-tempering at 100 °C for 5 min. (a) (30/15 wt.-% ALMAL/nanocorundum; (b) 30/15 wt.-% ALMAL/Plakor 15; and (c) 30/7.5/7.5 wt.-% ALMAL/nanocorundum/ Plakor 15.

combined ALMAL/nano/micro-corundum modification (Figure 2, line c) demonstrated the potential of the nanopowder. In the case of the most efficient hybrid composite as many as 1 000 rotation cycles of the testing disc removed only approximately one third of the coating by abrasion. This represents an improvement by the factor of 2 compared to pure ALMAL systems.

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Photo-induced radicals in silazanes, silanes, and siloxanes: An EPR and QC study

W. Knolle, L. Prager, S. Naumov, K. Czihal, L. Wennrich

The photo-induced conversion of Polysilazanes into SiO_x layers is an interesting route for producing gas barriers, especially against oxygen and water vapor, on polymer foils at room temperature and normal pressure [1]. By reason that only little is known about the primary reaction steps of the photo-initiated conversion, and to get a deeper understanding of the photochemical reactions following the irradiation of polysilazanes with high energy photons (λ = 172, 185, and 222 nm), the photo-induced radical formation in model compounds was investigated.

Radicals were generated either directly in the EPR cavity by the exposure to 254 or 185 nm light (at 100 K), or externally by irradiation with 222 nm light (finger dewar, 77 K). Temperature-dependent transformations and/or the decay of the radicals was followed up to a temperature of ~ 200 K.

In the simple case of hexamethyldisilane (HMDS) trimethylsilyl radicals were detected (multiplet of 9 equivalent protons with *a(9H)* ~ 0.6 mT (Fig. 1, top)), formed by cleavage of the Si–Si bond.

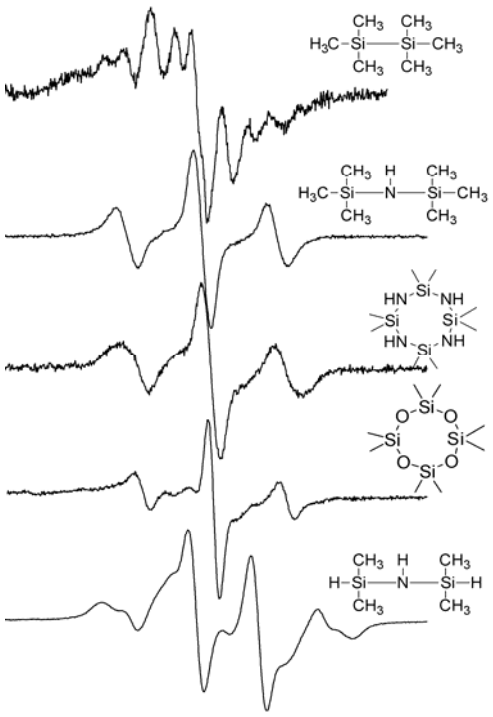

Figure 1: EPR spectra observed after UV irradiation of silanes, silazanes and siloxanes.

Surprisingly, for the investigated siloxanes and silazanes like hexamethyldisilazane (HMDSN), tetramethyldisiloxane (TMDSO), octamethylcyclotetrasilazane and –siloxane (OMCTSN and OMCTSO), no silyl radicals could be detected. Furthermore, the spectra observed for different compounds are very similar (triplet due to two equivalent protons). The only possible assignment is a secondary methylene radical of the structure $RSiCH_2^{\bullet}$, formed by H abstraction through primary radicals. This assignment is supported by the observation of a quartet spectrum in case of tetramethyldisilazane (TMDSN) with the additional splitting of one β-proton (Fig. 1, bottom).

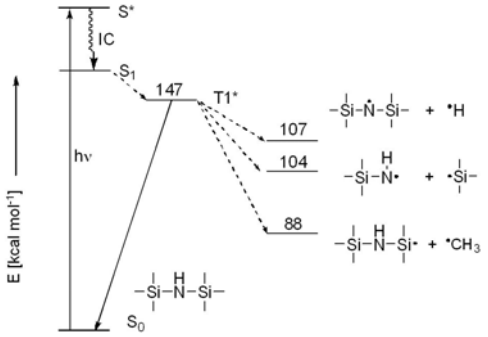

Figure 2: Energy scheme for the photolysis of HMDSN.

Further radicals like $(CH_3)_2Si^{\bullet}H$ and $R_2Si{=}N^{\bullet}$ could be identified in the case of TMDSN; $H^{\bullet}$ atoms and $^{\bullet}CH_3$ radicals were observed after irradiation of frozen HMDSN/acetonitril solutions. Quantum chemical calculations assist in the assignment of the EPR spectra and in the interpretation of a possible reaction mechanism. It was shown that different routes of cleavage of the (excited) triplet state are energetically possible (Fig. 2). However, the primary radicals are very reactive and transform quickly into more stable $R_2SiCH_2^{\bullet}$. Investigations on the reaction pathway under aerobic conditions are in progress.

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Thin oxygen and water vapor barrier layers

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Thin barrier layers are required in many high-tech fields. Metal layers provide the highest barrier values; however, in applications where high transparency is required, silicon and aluminum oxides still dominate.

In the framework of the BMBF research project „*Herstellung von SiO_x-Barriereschichten aus Polysilazanen bei Normaldruck und niedriger Temperatur mittels VUV-Strahlung*" (12/06-11/09), the photochemical conversion of 50-500 nm layers of perhydropolysilazane -$(SiH_2$-$NH)_n$- (PHPS) in the presence of oxygen into an SiO_x network has been studied. Different UV sources in the wavelength range of 160-240 nm, i.e., 172 nm Xe_2* and 222 nm KrCl* excimer and 185 nm low pressure (LP-) Hg lamps, were used for these purposes. The role of both ozone and $O(^1D)$ as well as of catalytic amounts of tertiary amines in the degradation process of PHPS and the formation of SiO_x were studied.

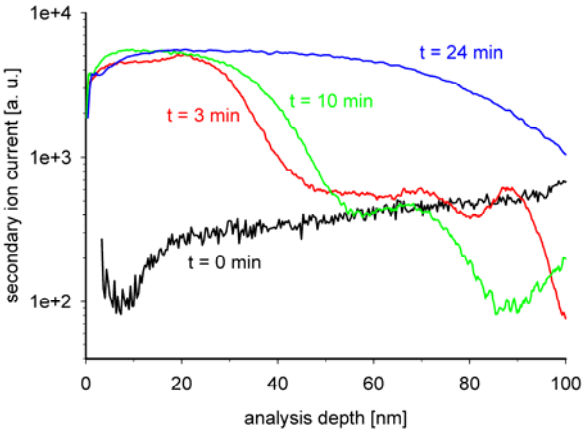

Figure 1: $^{29}SiO_3^-$ TOF-SIMS depth profiles of VUV treated PHPS layers; t – irradiation time.

In this context, the kinetics of the entire reaction were elucidated and allowed for establishing both a continuous and a discontinuous process for the production of fully transparent, flexible barrier coatings. Barrier improvement factors (BIF) of 400 were achieved with one single layer on 23 µm PET, translating into oxygen transmission rates (OTRs) of 0.20 $cm^3 \cdot m^{-2} \cdot d^{-1} \cdot bar^{-1}$. Double layers prepared by this technique allowed for the realization of OTRs ≤ 0.1 $cm^3 \cdot m^{-2} \cdot d^{-1} \cdot bar^{-1}$, corresponding to BIFs ≥ 800. [1, 2]

A different approach to high performance barriers was pursuit in the research project "*Flexible transparente Verkapselung von fotoelektrischen Systemen*" (SAB: 9720/1483): the concept entailed the realization of top-of-the-class magnetron sputtered AlO_x-barrier layers on specially suited PET with a thermo-mechanically adapted polymeric barrier aid layer. Disruptive (e.g. highly crosslinked, stiff, and thus stressed UV cured coatings), indifferent (most coatings), and synergistic (extremely low internal stress laminates or dispersion-derived films) were investigated as the three different classes of polymeric layers on inorganic barrier layers, depending on the relative coating stress level. While disruptive coatings reduce the overall barrier by deteriorating the inorganic barrier, indifferent coatings just add another layer and thus improve the barrier by up to a factor of two. By using a very low internal stress coating from cross-linked, film-forming urethane acrylate dispersions, the compressive stress found in magnetron sputtered AlO_x can be compensated for, resulting in delayed crack formation upon elongation and an at least 20-fold increase in barrier, which was actually below the limits of the measuring apparatus [3].

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Fundamental investigations of photochemical micro-folding

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The surface structuring of radiation-curable coatings by short-wave UV light is well known, and the VUV technology with 172 nm excimer lamps is already state of the art [1]. Nevertheless, further investigations on the kinetics of micro-folding and the characterisation of the random structure in correlation with the application parameters such as gloss, surface feel, scratch, and chemical resistance are required.

Using a small tailor-made chamber (Fig. 1) the kinetics of surface wrinkling of acrylate-based clear coats and UV-curable powder coatings were monitored in the real-time modus.

Figure 1: Real-time measuring device for the obser vation of the kinetics of micro-folding:

1 172 nm excimer lamp (XERADEX type),
2 additional lamp,
3 CCD camera,
4 mobile VUV sensor "FlatLog" with GaP-diode.

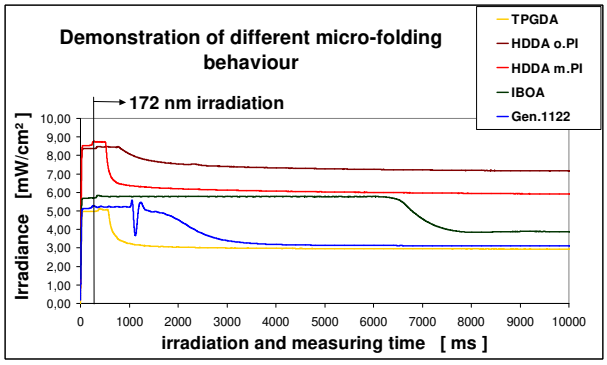

Figure 2: Different micro-folding behaviour.

The metering time period of the mobile VUV sensor was 1 millisecond, and so the different kinetics of various acrylate-based varnishes could be observed by measuring the decrease in transparency of the clear coat during the micro-wrinkling process (Fig. 2). All wrinkling processes were additionally monitored by a CCD camera, especially required for non-transparent UV powder coatings. The roughness parameters of the surface structure were determined by use of the optical profiler FRT MicroProf®. The topology could be described by an aspect ratio and by the power structural density (PSD). The PSDs (Fig. 3) of different surface structures were calculated using the AAP software developed at the IOM/IST Department [2].

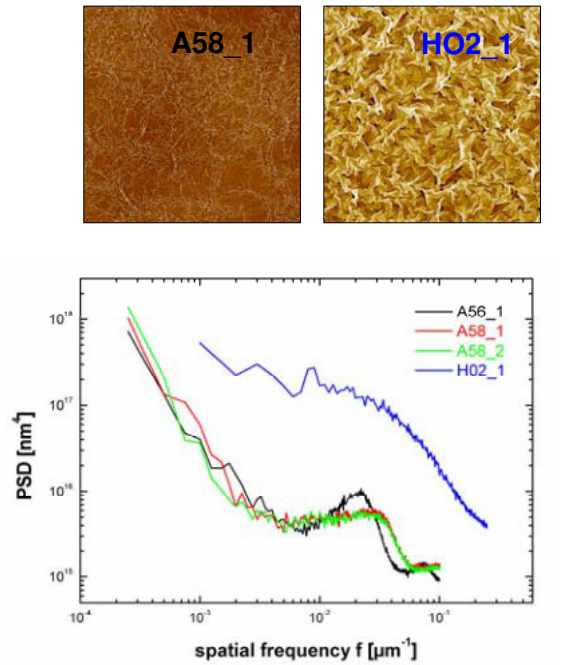

Figure 3: PSD of TPGDA (A58_1) and a screen-printing ink (H02_1).

This PSD permits the comparison of topologies with regards to the repeatability of the micro-folding process.

Acknowledgment

Financial support was provided by the *Sächsische Aufbaubank Dresden*, Germany.

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Argon VUV excimer lamp

A. Sobottka, R. Blaue, L. Drößler, M. Lenk, L. Prager, M.R. Buchmeiser

Rare gases and rare gas halogenide mixtures excited by a barrier discharge can emit UV excimer radiation. In the case of argon, the wavelength of the second continuum emission is centred in the vacuum UV region at 128 nm. For higher wavelengths, silica glass tubes are usually used as the discharge tubes. Since the absorption edge of the purest fused silica is at about 160 nm, only materials like MgF_2 or LiF could be used. However, for a more extended geometry these materials are too expensive. To avoid this problem, an open configuration without a window was chosen, where a continuous flow of the excimer gas Ar inhibits the diffusion of atmospheric oxygen and nitrogen into the discharge gap [1]. For investigation of this type of lamps and of the potential applications such as the etching of polymer surfaces, degradation of organic surface residues, and surface activation [2] several prototypes were built. In addition, different configurations of high voltage electrodes and their geometrical assemblies were examined.

The shape of the electric field in the discharge gap and the distribution of the argon gas flow have a constitutive influence on the formation of the barrier discharge. Streamers formed by micro discharges affect directly the intensity of the VUV emission.

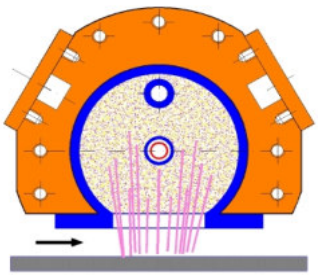

Figure 1: Configuration of an Ar excimer lamp.

An argon excimer lamp was designed which has an 270° enclosed discharge gap with an aperture as the photon exit (Fig. 1). Placing a second anode beneath the belt of the treatment conveyor excited and ionized species from the discharge gap can be extracted onto the sample surface exposing the sample to both the VUV radiation and the discharge plasma.

As electrical power supply, HF generators are used working at frequencies around 750 kHz and at high voltage up to 8 kV producing a field strength of about 800 V cm^{-1}.

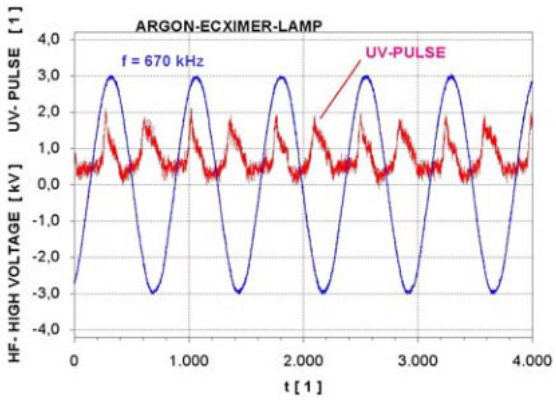

Figure 2: Oscillogram of the high voltage and the UV emission.

At an Ar consumption rate of 5 $m^3\ h^{-1}$ the residual oxygen content in the discharge gap is about 10 ppm. The power of the VUV radiation amounts to 20 mW cm^{-2}. By use of a second anode it reaches 600 mW cm^{-2}. The VUV lamp was assembled on a conveyor for a maximum sample width of 300 mm (Fig. 3).

Figure 3: Irradiation unit.

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Cold reactive plasma jet etching of thin SiO_2 films

T. Arnold, G. Böhm, A. Schindler

Microwave-excited reactive plasma jets generated at atmospheric pressure are versatile tools for surface etching, cleaning and chemical modification. Having an active area of less than 1 mm² such plasma jets are predestined for highly deterministic local processing.

"Cold" reactive plasma jets are of technological importance especially for high etching uniformity and the treatment of heat sensitive surfaces. In the case of plasma jet etching using gases like CF_4, where fluorine radicals are produced, thermally driven chemical mechanisms prevail during material removal. Since the plasma jet acts also as the main heat source which influences the surface temperature distribution the etching rate depends on the local surface temperature. This might result in spatio-temporal etching rate variations.

Treatments of surfaces sensitive to spatial or temporal temperature gradients are a challenging task. Hence the heat flux produced by the plasma jet has to be minimized. One possibility, the reduction of the input power per gas flow unit, leads to a decrease of the plasma temperature but at the same time instabilities of the plasma discharge arise limiting its technological application.

Our solution is to excite the plasma by short rectangular shaped microwave pulses. A special microwave pulse generator has been developed for this purpose by the Ing.-Büro ELEKON. Pulse durations of 3-9 µs with repetition rates of 2-12 kHz and a power range between 150 and 250 W can be adjusted obtaining a stable plasma jet of 5 mm length. The resulting mean power is in the range of 2-9 W. The plasma jet is fed by 400-1500 sccm Helium and 0.5-5 sccm CF_4.

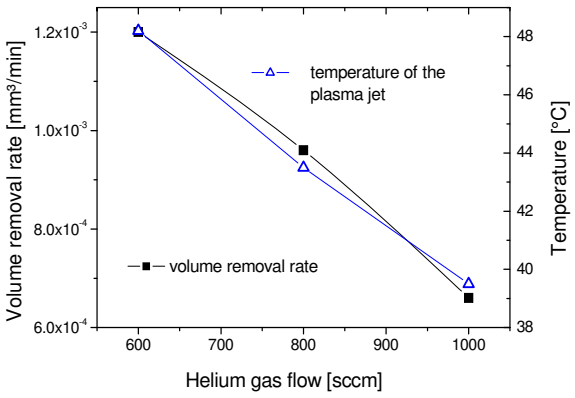

Figure 1: SiO_2 removal rates and plasma jet temperatures at 3 mm off the nozzle tip as a function of the Helium gas flow rate. Microwave mean input power was kept constant at 5 W.

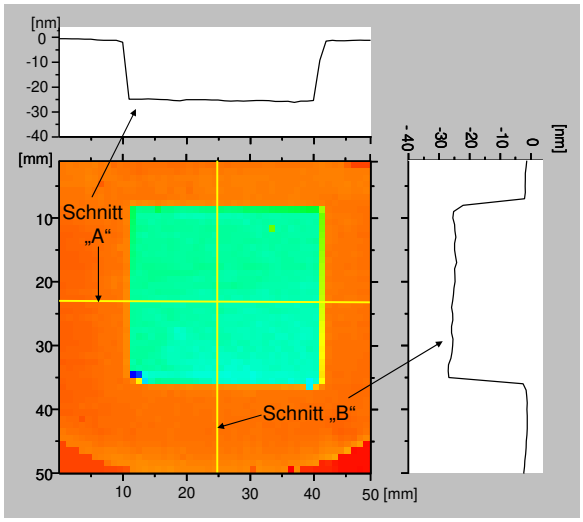

Figure 2: Optically measured thickness of an 100 nm SiO_2 film on a Si wafer with a 25 nm deep plasma jet etched area of 30 x 30 mm².

Gas temperatures of the plasma jet have been measured by a thermocouple in the range of 38-120 °C. Figure 1 depicts the plasma jet temperature and the volume removal rates as a function of the gas flow.

For the performance testing a thermally grown SiO_2 layer (100 nm) on top of a Si wafer was uniformly scanned by the reactive plasma jet obtaining a mean depth of 25 nm. Figure 2 shows the etched area measured by optical depth profiling. The surface shape error in x and y direction is 0.4 nm rms. The plasma jet etching process exhibits an excellent stability and uniformity. Thus, the small-sized plasma jet tool is capable for surface machining with high spatial resolution and nanometer depth accuracy. Potential applications for this tool are flattening of nanotopologies on Si or SOI wafers, cleaning of temperature sensitive surfaces of optical elements, or machining of small-sized mirrors and lenses made from Si, SiC, SiO_2, or Si_3N_4.

Epitaxial gadolinium nitride thin films deposited by low-energy nitrogen ion-beam assisted molecular-beam epitaxy

J.W. Gerlach, J. Mennig, B. Rauschenbach
in collaboration with K. Potzger
Forschungszentrum Dresden-Rossendorf, Institut für Ionenstrahlphysik und Materialforschung

For the past few years, rare earth nitrides have been the focus of an increasing amount of research groups with theoretical and experimental background. In particular, gadolinium nitride (GdN) has raised much interest due to its magnetic and electronic properties [1, 2]. Being ferromagnetic with a Curie temperature of 60 to 70 K, the reported electronic properties range from semiconducting to semimetallic to insulating. Even halfmetallic properties are predicted.

While in literature polycrystalline and randomly oriented GdN films are reported of, at the IOM epitaxial GdN films of relatively high crystalline quality could be achieved on magnesium oxide (MgO) substrates by applying the ion-beam assisted molecular-beam epitaxy (IBA-MBE) technique [3]. For the growth experiments, an appropriate substrate material with a low lattice mismatch to GdN was required, but another prerequisite turned out to be that the substrate material is sufficiently chemically inert. Otherwise, the deposited Gd atoms, evaporated by a high-temperature effusion cell, would chemically react with the substrate due to their high reactivity. Exemplary, epitaxial GdN films could be deposited on yttria-stabilized zirconia (YSZ) substrates, too, but time-of-flight secondary ion mass spectrometry (TOF-SIMS) revealed that the highly oxygen-affine Gd attracted the oxygen in the substrate leading to a severe increase in the oxygen contamination level of the GdN films. Another issue was that GdN is unstable in air and the films oxidized rapidly. This could be avoided by depositing a 50 nm thick polycrystalline gallium nitride (GaN) protective layer on top of the GdN film.

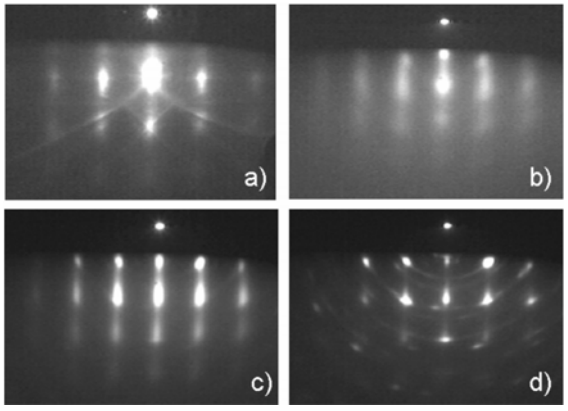

Figure 1: 30 keV RHEED patterns obtained with the electron beam along MgO[100]: MgO substrate prior to deposition (a), after 5 min (b), 15 min (c), and 55 min (d) of GdN deposition (final film thickness: 60 nm).

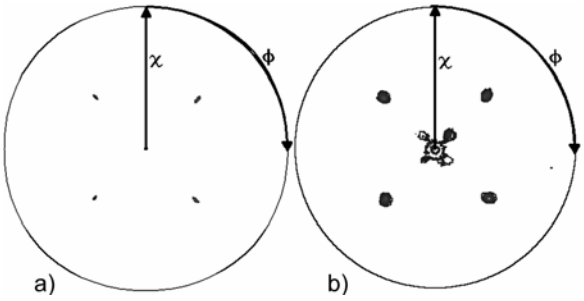

Figure 2: XRD pole figures MgO{111} of the substrate (a) and GdN{100} of the GaN-capped GdN film (b) deposited on top of this substrate, proving the epitaxial growth.

For identifying and monitoring the epitaxial GdN growth, reflection high energy electron diffraction (RHEED) proved as a valuable method (Figure 1). Ex situ, epitaxial relationships and crystalline quality of the GdN films were examined by X-ray diffraction (XRD). Figure 2 shows texture goniometry results of an epitaxial GdN film on a MgO(100) substrate. First magnetic measurements showed that the GdN films exhibit ferromagnetism below a Curie temperature of 70 K.

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[3] J.W. Gerlach, J. Mennig, B. Rauschenbach, Appl. Phys. Lett. 90 (2007) 061919.

Small tool size ion beam and reactive plasma jet surface figuring

T. Hänsel, G. Böhm, A. Nickel, P. Seidel, T. Arnold, A. Schindler

Two major requirements from large high-end optics performances are guiding the development in highly deterministic surface figuring technology (i) to allow sub-0.5 nm depth accuracy with high spatial resolution of up to sub-1.0 mm and (ii) high processing stability and convergence. A third goal for the development of the technologies is to qualify them for efficient processing of small-sized optics of medium optical performance.

We developed a small tool ion beam figuring (IBF) technology with sub-mm FWHM Gaussian ion beam for the figuring of surfaces with curvatures of up to 50° slope angle using a three axes (x, y, z) motion system. The new developed processing software based on a dwell time algorithm include the variation of the ion beam tool removal function with the slope angle of the surface to be processed. This dependence has been measured experimentally in advance of the processing. The first testing of the new technique is shown in Fig. 1 demonstrating a successful surface shape error correction of a small fused silica lens with an improvement from 4.5 nm rms to 0.56 nm rms shape error.

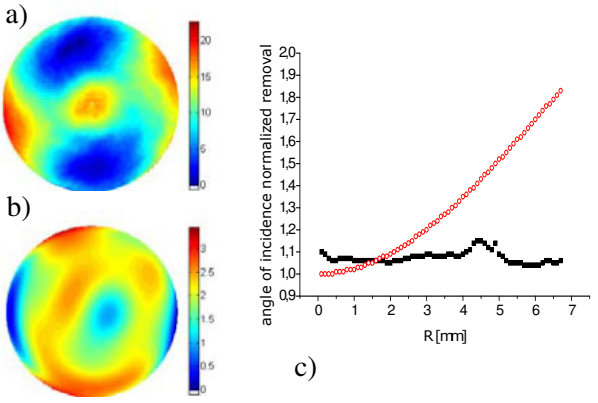

Figure 1: Shape error correction of a convex quartz lens of 13,5 mm diameter and R = -10,44 mm radius of curvature by a 1.1 mm FWHM Gaussian ion beam; a) surface error topology before IBF: 22.6 nm PV and 4.5 nm rms, b) ZERNIKE surface data fit after IBF with resulting 3.0 nm PV and 0.5 nm rms, c) experimentally measured correction function of the angle of incidence dependence of the removal rate used in the IBF modelling (open red circles) and the ratio between the desired and the measured removal using the correction function.

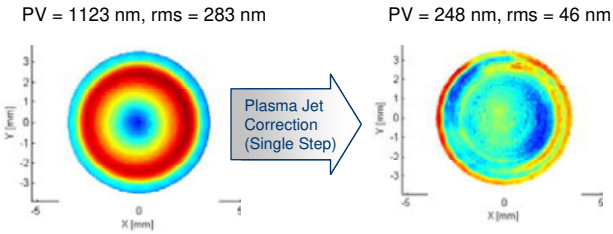

Figure 2: Shape error correction of a small-sized aspheric quartz lens using PACE with a sub-mm FWHM plasma jet tool machining and a standard dwell time algorithm.

The development of atmospheric microwave driven small-sized reactive plasma jet tools having about 1 mm² active area for highly deterministic local surface processing based on plasma assisted chemical etching (PACE) by the chemical reaction with fluorine radicals focused on the figuring of Si and Si-based materials like fused silica, quartz glass, SiC. From the status of demonstration of the figuring at all we aim to make it mature for being an effective technique for the figure finishing of small-sized lenses. On the one hand major progress has been achieved by the development of a dedicated microwave pulse generator for low average plasma jet power regime resulting in very stable jet etching characteristics. On the other hand further improvements in the plasma etch chemistry optimized by the admixture of oxygen to the processing gas mixture led to a more stable and more efficient removal process. Thus, the small-sized plasma jet tool is now mature for surface figuring with millimetre range spatial resolution achieving nanometre range machining depth accuracy. Fig. 2 shows the surface shape error correction of a small-sized convex aspheric quartz lens of 7 mm in diameter and about 50 mm best fit sphere radius of curvature resulting in a six-fold improvement of the rms surface error in only 20 minutes processing time.

The improved small-sized ion beam and plasma jet figuring techniques open new applications, e.g. for effective small lens figuring and nanotopology correction of silicon wafers.

Inductive coupled radio frequency plasma bridge neutralizer

F. Scholze, M. Tartz, H. Neumann

For many applications of surface processing the neutralization of the ion beam current on the target is necessary. Particularly the charging up of isolating surfaces has to be compensated by supplying a sufficient amount of electrons onto the surface. During operation of an ion thruster for spacecraft propulsion charged particles are ejected which produces the thrust; however, the remaining opposite charge leads to a charging up of the spacecraft. In order to keep the spacecraft nearly at ground potential, the excess charge has to be removed from the spacecraft. Usually a neutralizer is employed to deliver an electron beam which fulfils the above-mentioned tasks. Several types of neutralizers have been developed in the past, such as hot cathode neutralizer, plasma bridge neutralizer with hot cathode, or hollow cathode. Hot cathodes are known to have a limited lifetime. Hollow cathodes are very sensitive against oxygen or other reactive gases. To overcome these application constraints a cathodeless inductively coupled radio frequency plasma bridge neutralizer RF-PBN has been developed. Such a concept eliminates all components inside the plasma chamber such as cathodes; there are also no gas-sensitive components. A robust design

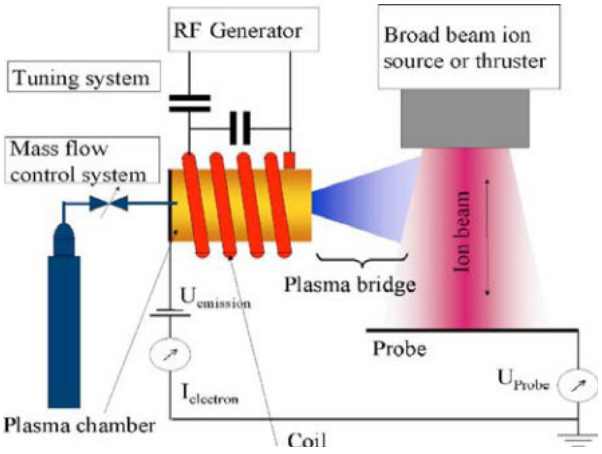

Figure 1: Schematic set-up of the radio frequency.

is expected to result in a long-duration operation. When the extraction aperture of the neutralizer comes close to the ion beam, a plasma bridge would be formed. Across this plasma bridge the neutralizer electrons run toward the positive space charge of the ion beam to compensate this.

The set-up of the inductive coupled plasma bridge neutralizer is shown in Fig. 1. It is composed of a 1.3 cm in diameter and 2.5 cm long ceramic tube located in the center of a cylindrical inductor. The ceramic tube is closed at one side by a thin ceramic plate with a small hole for electron extraction and by a metallic plate as plasma anchor at the reverse side. In the plasma anchor the gas inlet is integrated. The discharge is driven by a 13.56 MHz RF generator which can deliver a maximum power of 300 W. A fixed matching network is used to match the impedance between RF generator and cylindrical inductor consisting of a package of two capacitors. One capacitor is connected in parallel with the cylindrical coil. This capacitor and the coil build an oscillating circuit for the plasma excitation. The second capacitor is connected in series with the oscillating circuit minimizing the reflected RF power to the generator.

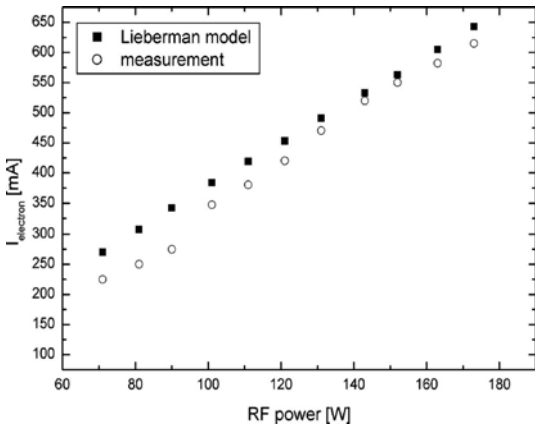

Figure 2: Comparison of measured electron current and calculated values (argon operation, 1 sccm).

Fig. 2 illustrates the measured electron currents in an Argon discharge in dependence on the RF power and the qualitative good agreement between this measured electron emission current and the values calculated with the developed plasma equilibrium model [1] based on the Lieberman model [2].

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Fabrication of sub-100 nm patterns in fused silica by electron beam writing (EBL) and reactive ion beam etching (RIBE)

J. Zajadacz, R. Fechner, K. Zimmer, A. Schindler
in collaboration with
M. Helgert Carl Zeiss AG – BS Jena

Submicron and nanoscale structures are of growing importance in current and future applications. In contrast to bottom-up approaches top-down techniques require high-resolution writing techniques. The combination to pattern transfer techniques is needed for applications requiring advanced materials or films. However, with reduced pattern size the demands on the pattern writing as well as to the transfer technique increase, e.g., due to the maintained pattern depth resulting an increased aspect ratio of the structures.

At the IOM an ultrahigh resolution EBL workstation (e LiNE, Raith) for electron beam lithography is available. The minimum line width with high-resolution positive tone PMMA (polymethylmethacrylate) or negative tone novolac resins (naphthochinondiazides) is less than 20 nm as shown in Fig. 1 (left) for a 70 nm PMMA resist film.

Fig. 1 (centre) shows trenches etched into a 100 nm SiO_2 layer on c-Si. For EBL pattern writing the 120 nm thick PMMA resist AR-P 671.02 from Allresists was exposed at 20 keV with doses ranging from 128 to 152 µC/cm^2 depending on the line width. The optimal exposure parameter and proximity effect corrections were deducted from the analysis of line widths/shape vs. the writing dose. For an exposure dose of 152 µC/cm^2 the resist was slightly overexposed and the designed 100 nm lines broaden to 106 nm width. To transfer the resist pattern into a 100 nm thick SiO_2 layer fluorine based RIBE process was used. For SiO_2 an etch rate of about 35 nm/min and a selectivity of ~ 7 has been achieved using a standard RIBE-process with CHF_3. For applications calling for a lower etch selectivity it can be adjusted by the etch gas mixture ratio of CHF_3 and O_2 in the range from 0.5 to 6. However, size, shape, and sidewall slope of structures are also altered by the gas mixture. For instance, 100 nm resist patterns increase in width to 120 nm at RIBE with CHF_3/O_2 gas mixture of 80:20.

Fig. 1 (right) shows a linear grating with 550 ± 10 nm depth after RIBE pattern transfer into fused silica. The average line width of 80 ± 10 nm is less the desired (resist) line width of 100 nm due to resist shrinking effects. Nevertheless a side wall slope and an aspect ratio of 87° and 5.5 has been achieved, respectively. The resist patterning was designed for a wavelength of 333 nm and ~ 3000 lines/mm and realized by means of holographic lithography at the Carl Zeiss Jena GmbH.

For further size reduction of functional structures EBL and RIBE processes must be optimized with respect to the material, the thickness of the resist mask, and the etch chemistry for pattern transfer depending on the application requirements.

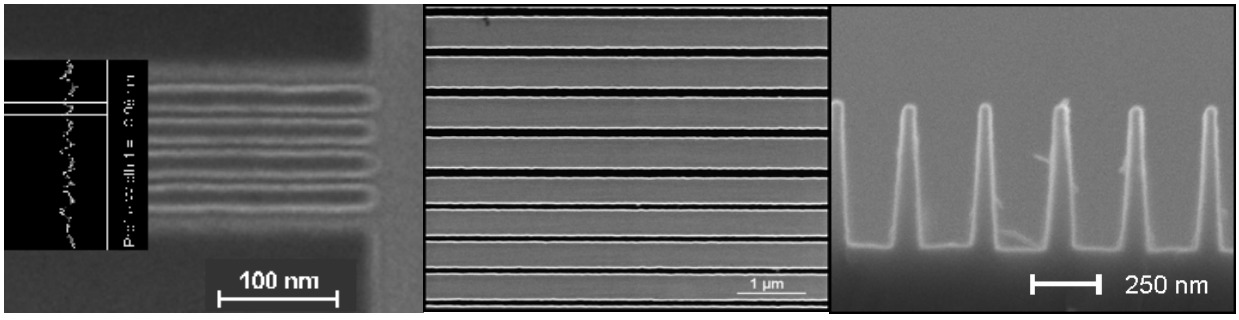

Figure 1: Examples for EBL and RIBE pattern transfer. Left: EBL in a 70 nm PMMA resist, a line width of 19 nm is demonstrated; centre: sub-µm lines (120 to 300 nm) after pattern transfer by RIBE into a 100 nm SiO_2 layer; right: high aspect ratio sub-µm structures transferred by RIBE into fused silica.

Laser etching of 3D phase shaping microoptical elements in fused silica

K. Zimmer, R. Bellmann, R. Böhme

High quality etching of transparent materials for applications in micro- and nano-structuring as well as in precision engineering is still a challenge for current laser processing techniques. The recently developed laser-induced backside wet etching (LIBWE) method [1] allows the direct and precise etching of transparent materials, e.g., fused silica, at low laser fluences, with etch rates in the nanometer scale, and with an optical-grade roughness at optimized processing conditions.

The combination of specific laser processing techniques, e.g., scanning contour mask technique and direct writing with a small laser spot [1], with LIBWE allows the direct writing of analogous and binary microstructures for applications in miniaturized optics [2, 3]. This technology utilizes the full degree of freedom of the laser beam tool and can be applied for Rapid Prototyping of diffractive and refractive microoptics onto planar or non-planar surfaces. Furthermore, hybrid optics (combination of refractive and diffractive optical functions into one element), aspheres, and freeform surfaces can be processed into transparent materials, too.

To demonstrate the capabilities of this processing approach highly sophisticated microoptical elements like spiral phase plate and micro axicon have been etched directly by pulsed laser processing into fused silica. Both elements have a 3D surface shape with depths in the micron range, lateral dimensions in the millimetre scale, and a phase shaping optical function. The spiral phase plate and the axicon can be used to generate advanced optical effects such as optical vortex and Bessel beams, respectively.

First the scanning path of a rhombic mask with a side length of 90 µm across the sample surface was optimized. The velocity distribution of the scanning laser beam was calculated from the local etch depth considering the etch rate, the focus size etc. The laser etching was carried out with a pyrene/toluene solution and at a fluence of 0.86 J/cm².

The etched surface topography of a spiral phase plate measured by interference microscope is depicted in Fig. 1. The phase element that corresponds with the target topography very well has a diameter of 2 mm, a continuously increasing depth with the azimuthal angle with maximum at 1.38 µm. The surface deviation and the optically measured surface roughness are less than 5% and ~ 10 nm rms, respectively. The radius and depth of the laser-etched micro axicon are 0.5 mm and 15 µm giving a wedge angel of about 0.79°. The roughness/waviness increases with the etch depth but is better λ/20. As shown in Fig. 2, the laser written axicon produces a Bessel beam with a 16 µm diameter over a distance of 30 mm.

Literature

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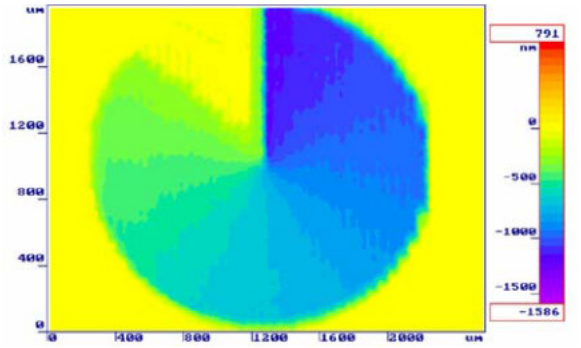

Figure 1: 3D topography of etched spiral phase plate in fused silica recorded with white light interference microscopy.

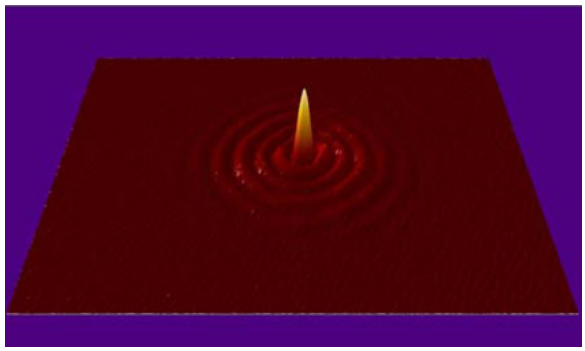

Figure 2: Optical function of the laser-etched micro axicon recorded by CCD camera. A HeNe-laser beam was used for inspection.

Personal Activities and Scientific Events

Doctoral and Diploma Theses

Activities in Scientific Organisations

Honours and Awards

Lectures and Seminars

Scientific Meetings and Institute Colloquia

Personal Activities

Doctoral and Diploma Theses

Doctoral Theses

Emre Erdem
Size effects in ferroelectric $PbTiO_3$ nonopowers observed by electron paramagnetic resonance spectroscopy
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Nasser Razek
Ultrahochvakuum Wafer-Direktbonden nach Reinigung durch Beschuss mit niederenergetischen Wasserstoffionen
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Bashkim Ziberi
Ion beam induced pattern formation on Si and Ge surfaces
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Rico Böhme
Laser-induced backside wet etching of glasses and crystals
Martin-Luther-Universität Halle-Wittenberg, Zentrum für Ingenieurwissenschaften, 2007

Diploma Theses

Inga-Marie Eichentopf
Härte- und Verschleißmessungen an rostfreien Edelstahl
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Andreas Hoffmann
Ionenstrahlgestützte Molekularstrahlepitaxie von Galliumnitrid-Dünnschichten auf Lithiumaluminat-Substraten
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Christoph Klement
Abscheidung dünner Multischichten mittels PLD und deren Charakterisierung
Hochschule Mittweida (FH), Fachbereich für Maschinenbau und Feinwerktechnik, 2006

Andrea Löber
Monolithisch strukturierte Materialien für die Regenerative Medizin
Martin-Luther-Universität Halle-Wittenberg, Fakultät für Chemie und Physik, 2006

Johanna Lutz
Plasmabildung und –diffusion an verteilten Mikrowellen-ECR-Quellen
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Theresa Lutz
Ionenstrahl-induzierte Selbstorganisation auf vorstrukturierten Oberflächen
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Bettina Schlemmer
Neue ROMP-basierte monolithische Trennmedien für die Cap-HPLC
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2006

Andreas Wolfstetter
In-situ REED-Messungen während der ionenstrahlgestützten Deposition dünner Nitridschichten
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2006

Antje Lehmann
Plasma-Immersions-Ionenimplantation in CoCr-Legierungen für biomedizinische Anwendungen
Fachhochschule Osnabrück, Fakultät für Ingenieurwissenschaften und Informatik, 2007

Christine Leschinski
Synthese nitridischer Dünnfilme mittels gepulster Femtosekunden-Laserdeposition
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2007

Julius Mennig
Ionenstrahlgestützte Molekularstrahlepitaxie von Gadoliniumnitrid-Schichten
Universität Leipzig, Fakultät Physik und Geowissenschaften, 2007

Johannes Meister
Verfahrensentwicklung zur Anwendungserweiterung der Verweilzeitmethode
Hochschule für Technik, Wirtschaft und Kultur Leipzig (FH), Fachbereich Informatik und Mathematik, 2007

Sabine Schirmer
Oberflächenbeschichtungen auf Zirkon- und Titanbasis für den Einsatz im Dentalbereich
Fachhochschule Osnabrück, Fakultät für Ingenieurwissenschaften und Informatik, 2007

Tino Stephan
Mikrostruktur und Verbindungstechnologie von Solarzellen
Westsächsische Hochschule Zwickau (FH), Fachbereich Physikalische Technik und Informatik, 2007

Hendrik Zachmann
Untersuchungen von strukturellen und elektrischen Schichteigenschaften bei der ionenstrahlgestützten Abscheidung von $Cu(In,Ga)Se_2$
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2007

Activities in Scientific Organisations

F. Bauer

- Member of the Steering Committee of the 'International Symposium on Ionizing Radiation and Polymers – IRaP 2008'

M.R. Buchmeiser

- Member of the International Advisory Boards of 'Macromolecular Rapid Communications' and 'Macromolecular Chemistry and Physics'
- Chairman of the Scientific Advisory Board of the Institut für Nichtklassische Chemie, University of Leipzig

W. Knolle

- Member of the Scientific Committee of the 'International Symposium on Ionizing Radiation and Polymers – IRaP 2008'
- Member of the Organizing Committee of the 'European Young Investigator Conference – EYIC 2007'
- Associate Editor of 'Radiation Physics and Chemistry'

S. Mändl

- Member of the International Committee of the International Workshop 'Plasma Based Ion Implantation & Deposition – PBII&D'

B. Rauschenbach

- Speaker of the Thin Film Division of the German Physical Society (DPG) (up to 2006)
- Member of the Advisory Board of the International Union of Vacuum Science, Technology and Application (IUVSTA)
- Member of the Curatorship for 'Innovation and Science'
- Member of the Advisory Board of the German Physical Society (DFG)
- Member of the Advisory Board of the German Vacuum Society (DVG) (up to 2006)
- Member of the Council 'Condensed Mater' of the German Physical Society
- Member of the Coordination Board 'Plasma Surface Technologies'

- Member of the Steering Committee of the Leipzig School of Natural Science 'Building with Molecules and Nano-objects'
- Member of the Scientific Board 'Translational Centre for Regenerative Medicine - TRM'

Honours and Awards

R. Böhme:

- Young Scientist Award of the European Material Research Society, 2006

M.R. Buchmeiser

- Ruf an die Technische Universität Dresden, Professur für 'Makromolekulare Chemie'
- Program Chairman of the '18th International Symposium on Olefin Metathesis and Related Chemistry – ISOM XVIII', Leipzig, 2009

T. Höche

- Max-von-Laue-Preis 2006 der Deutschen Gesellschaft für Kristallographie

B. Rauschenbach

- Chairmen of '16th International Conference Ion Beam Modification of Materials – IBMM 2008' Dresden, 2008
- Member of the Advisory Board '11th International Conference on Plasma Surface Engineering – PSE 2008', Garmisch-Patenkirchen, 2008
- Member of the International Advisory Committee '14th International Conference of Thin Films – ICTF14', Ghent, 2008
- Ruf auf eine Gastprofessur an die Wuhan University, Center for Nanosience and Nanotechnology, China

Lectures and Seminars

Lectures

F. Bauer

- *Radioaktivität, Kernenergie und Strahlenschutz*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 06/07

- *Radioaktivität, Kernenergie und Strahlenschutz*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 07/08

M.R. Buchmeiser

- *Molekulare heterogene Katalyse*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 06

- *Makromolekulare Chemie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 06/07

- S*pezial- und Funktionspolymere*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 06/07

- *Makromolekulare Chemie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 07/08

- S*pezial- und Funktionspolymere*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 07/08

J.W. Gerlach

- *Beschichtung und Beschichtungstechnologien*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 06/07

- *Beschichtung und Beschichtungstechnologien*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 07/08

T. Höche

- *Elektronenmikroskopie*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 06

- *Mikro- und Nanostrukturcharakterisierung mit elektronenmikroskopischen Techniken*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 06/07

- *Mikro- und Nanostrukturcharakterisierung mit elektronenmikroskopischen Techniken*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 07/08

S. Mändl

- *Plasmaphysik II: Plasmatechnologie*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 06

- *Physikalische Oberflächenmodifizierung von Werkstoffen der Medizintechnik*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 06/07

- *Schichtabscheidung mit Vakuumlichtbogen und Laserablation*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 07

- *Oberflächenanalytik in Astronomie, Archäologie und Kunstgeschichte*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 07/08

B. Rauschenbach

- *Ionen-Festkörper-Wechselwirkung*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 06

- *Physics of Micro- and Nanostructures*
  University Wuhan, Center of Nanophysics and Nanotechnology, Wuhan, China
  winter 06/07

- *Physik dünner Schichten*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 07

- *Oberflächen- und Dünnschichtanalytik*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 07/08

- *Physics of Thin Films*
  University Wuhan, Center of Nanophysics and Nanotechnology, Wuhan, China
  winter 07/08

A. Schindler

- *Moderne Oberflächentechnologien - Physikalische Grundlagen, Analytik und Anwendungen*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 06

Seminars

M.R. Buchmeiser

- *Materialwissenschaftliches Seminar*
  Universität Leipzig, Fakultät für Chemie und Mineralogie und Leibniz-Institut für Oberflächenmodifizierung Leipzig
  winter 06/07, summer 07, winter 07/08

- *Wissenschaftliches Seminar für Diplomanden und Doktoranden*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 06, winter 06/07, summer 07, winter 07/08

T. Höche and B. Rauschenbach

- *Materialwissenschaftliches Seminar*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften und Leibniz-Institut für Oberflächenmodifizierung Leipzig
  summer 06, winter 06/07, summer 07

Scientific Events

Scientific Meetings and Institute Colloquia

Scientific Meetings

Workshop der DFG-Forschergruppe 522 '*Architektur von nano- und mikro-dimensionalen Strukturelementen*'
Leipzig, 14.03.2006

XIII. Workshop '*Oberflächentechnologien mit Plasma- und Ionenstrahlprozessen*'
Mühlleithen, 14.-16.03.2006

CC-UPOB Workshop '*Ultrapräzise Oberflächenbearbeitung mit Ionen-, Plasma- und Laserstrahlen*'
Leipzig, 27.04.2006

Workshop der DFG-Forschergruppe 522 '*Architektur von nano- und mikro-dimensionalen Strukturelementen*'
Naumburg, 29.09.2006

XIV. Workshop '*Oberflächentechnologien mit Plasma- und Ionenstrahlprozessen*'
Mühlleithen, 13.-15.03.2007

9th International Workshop on '*Plasma-Based Ion Implantation & Deposition*' - PBII&D
Leipzig, 02.-06.09.2007

Workshop der DFG Forschergruppe 845 '*Selbstorganisierte Nanostrukturen durch niederenergetische Ionenstrahlerosion*'
Leipzig, 18.12.2007

Institute Colloquia

J. Schneider (19.01.2006)
Rheinisch-Westfälische Technische Hochschule Aachen, Fakultät für Georessourcen und Materialtechnik, Aachen
Thin film growth in a high vacuum ambient

N. Hüsing (26.01.2006)
Universität Ulm, Institut für Anorganische Chemie I, Ulm
Vom dünnen Film zum Monolithen

H. Ritter (06.04.2006)
Heinrich-Heine-Universität Düsseldorf, Institut für Organische Chemie und Makromolekulare Chemie, Düsseldorf
Funktionalisierte Polymere mit Cyclodextrinen und Mikrowelle

Tapas Kumar Chini (20.04.2006)
Saha Institute of Nuclear Physics, Material Physics Group, Kolkata, India
Medium-keV Ar-ion-induced surface ripples in silicon: Experiment vs theory

S. Blechert (27.04.2006)
Technische Universität Berlin, Institut für Chemie, Berlin
Katalyse und Naturstoffe – eine starke Kombination

M. Moseler (04.05.2006)
Fraunhofer-Institut für Werkstoffmechanik, Freiburg
Multiskalensimulation der ionenstrahlinduzierten Glättung von Oberflächen

J. Reif (11.05.2006)
Brandenburgische Technische Universität Cottbus, Institut für Physik und Chemie, Cottbus
Bildung von selbstorganiserten Nanostrukturen aufgrund von Laser induzierter transienten Instabilitäten

S. Müller (19.05.2006)
Max-Planck-Institut für Mathematik in den Naturwissenschaften, Leipzig
Analysis of microstructures

R.R. Schrock (01.06.2006)
Massachusetts Institute of Technology (MIT), Cambridge, USA
Recent advances in high oxidation state alkylidene chemistry and applications to organic and polymer chemistry

B. Fuhrmann (15.06.2006)
Martin-Luther-Universität Halle-Wittenberg, Interdiziplinäres Zentrum für Materialwissenschaften, Halle
Nanosphere lithography

A. Müller (22.06.2006)
Universität Bayreuth, Fakultät für Biologie, Chemie und Geowissenschaften, Bayreuth
New amphiphilic and hybrid nanoparticles: Janus micelles, core-shell cylinders, nanomagnets, nanowires

O. Nuyken (29.06.2006)
Technische Universität München, Fakultät für Chemie, München
2-Oxazoline based amphiphile block copolymers and their application for micellar catalysis

A. Holländer (13.07.2006)
Fraunhofer-Institut für Angewandte Polymerforschung, Golm
Oberflächenchemie an Polymeren: Anwendungen und Analytik

Y. Sano (20.07.2006)
Osaka University, Department of Precision Science und Technology, Osaka, Japan
Plasma chemical vaporization machining (PCVM) - Ultraprecision machining method using atmospheric pressure plasma

M. Aziz (12.09.2006)
Harvard University, Division of Engineering and Applied Sciences, Cambridge, USA
Nanoscale morphology control using ion beams

C. Huber (05.10.2006)
Universität des Saarlandes, Naturwissenschaftlich-Technische Fakultät III, Saarbrücken
Monolithische Strukturen als hocheffiziente Trennmedien für biologische Makromoleküle

K.C. Hultzsch (12.10.2006)
Friedrich-Alexander-Universität Erlangen-Nürnberg, Institut für organische Chemie, Erlangen
Seltenerdmetall-katalysierte asymmetrische Hydroanimierung und Epoxid/Kohlendioxid-Copolymerisation

S. Spange (19.10.2006)
Technische Universität Chemnitz, Institut für Chemie, Chemnitz
Cationically induced twin-polymerization of novel furan monomers

S. Christiansen (26.10.2006)
Max-Planck-Institut für Mikrostrukturphysik, Halle
Zukünftiges Silizium: Materialdesign durch Verspannung und Nanostrukturierung

M. Wuttig (07.11.2006)
Rheinisch-Westfälische Technische Hochschule Aachen, I. Physikalisches Institut, Aachen
Ultraschnelle Datenspeicherung mit Phasenwechselmedien: Von der Kristallstruktur zur Kinetik

W. Mormann (16.11.2006)
Universität Siegen, Fachbereich für Chemie und Biologie, Siegen
Zellulosechemie in ionischen Flüssigkeiten

W. Ensinger (30.11.2006)
Technische Universität Darmstadt, Fachbereich für Material- und Geowissenschaften, Darmstadt
Von KeV bis GeV: Einsatz von Ionenstrahlen in der Materialmodifikation und Nanotechnologie

K. Müllen (07.12.2006)
Max-Planck-Institut für Polymerforschung, Mainz
Organische funktionale Nanoteilchen

R. Behrisch (14.12.2006)
Max-Planck-Institut für Plasmaforschung, Garching
Kontrollierte thermonukleare Fusion und das Problem der Plasma-Wand-Wechselwirkung

H.-J. Adler (11.01.2007)
Technische Universität Dresden, Fachrichtung Chemie und Lebensmittelchemie, Dresden
Anorganisch-organische Hybridpartikel: Von der Synthese bis zu Anwendungs-beispielen

V. Lavrentiev (25.01.2007)
Academy of Sciences of the Czech Republic, Nuclear Physics Institute, Rez, Czechia
From MICRO to NANO: Selective topics of ion beam application

K. Kern (30.01.2007)
Max-Planck-Institut für Festkörperforschung, Stuttgart
Atomare und molekulare Nanostrukturen: Kleiner Maßstab mit großem Potenzial

U. Schubert (22.03.2007)
Technische Universität Wien, Institut für Materialchemie, Wien, Austria
Funktionalisierte Metalloxid-Cluster als Bausteine für anorganisch-organische Hybridmaterialien

M. Beller (29..03.2007)
Leibniz-Institut für Katalyse, Rostock
Catalysis - A key technology for the synthesis of pharmaceuticals and new materials

T. Lippert (05.04.2007)
Paul Scherrer Institut, Villigen, Switzerland
Interactions of photons with surfaces: Structuring, modification, and applications

G. Dehm (12.04.2007)
Erich Schmid Institut für Materialwissenschaft und Department Materialphysik der Montanuniversität Leoben, Loeben, Austria
Plastizität und Versetzungsmechanismen in dünnen Metallschichten

J. Kressler (19.04.2007)
Martin-Luther-Universität Halle-Wittenberg, Institut für Physikalische Chemie, Halle
Synthese amphiphiler Polymere und deren Selbstorganisation in Wasser und an Oberflächen

C. Trautmann (26.04.2007)
Gesellschaft für Schwerionenforschung, Darmstadt
Materialmodifikation mit hochenergetischen Schwerionen

F. Cichos (03.05.2007)
Universität Leipzig, Institut für Experimentelle Physik I, Leipzig
Einzelne Moleküle als Nanosonden für Materialien

P.A. van Aken (10.05.200)
Max-Planck-Institut für Metallforschung, Stuttgart
Neue Entwicklungen in der Transmissionselektronenmikroskopie: Einblicke in die atomare Welt

R. Mühlhaupt (24.05.2007)
Albert-Ludwigs-Universität Freiburg, Institut für makromolekulare Chemie und Freiburger Materialforschungszentrum, Freiburg
Polymerisationskatalyse: Neue Materialien aus alten Bausteinen

J.A. Gladysz (14.06.2007)
Friedrich-Alexander-Universität Erlangen-Nürnberg, Institut für organische Chemie, Erlangen
Alkene metathesis in metal coordination spheres: The quest for molecular gyroscopes

D.M. Guldi (13.09.2007)
Friedrich-Alexander-Universität Erlangen-Nürnberg, Institut für physikalische Chemie, Erlangen
Carbon nanotube nanoconjugates and nanohybrids - Electron donor acceptor interactions

A. Tünnermann (18.10.2007)
Fraunhofer-Institut für Angewandte Optik und Feinmechanik, Jena
Moderne Optik - Neue Herausforderungen an die Mikro- und Nanostrukturtechnik

B. Bildstein (08.11.2007)
Leopold-Franzens-Universität Innsbruck, Institut für Allgemeine, Anorganische und Theoretische Chemie, Innsbruck, Austria
Design und Entwicklung neuer Olefin-Polymerisationskatalysatoren der frühen bis späten Übergangsmetalle

A. Greiner (15.11.2007)
Philips-Universität Marburg, Fachbereich Chemie, Marburg
Funktionalisierte Nanofaservliese durch Elektrospinnen

R. Grunwald (22.11.2007)
Max-Born-Institut für Nichtlineare Optik und Kurzzeitspektroskopie, Berlin
Ultraflat microoptics for pulse diagnostics and multichannel processing

G. Erker (29.11.2007)
Westfälische Wilhelms-Universität Münster, Organisch-Chemisches Institut, Münster
Konstruktion chemischer Brücken: Neue Wege zu ansa-Metallocenen und Metallocenophanen

A. Laschewsky (06.12.2007)
Fraunhofer-Institut für Angewandte Polymerforschung, Golm
Wege zu neuen Materialien mittels selbstorganisierender Polymere

Publications and Presentations

Publications in Journals and Books

Conference Proceedings

Presentations

Patent Applications and Patents

Publications and Presentations

Publications in Journals and Books

D.J. As, M. Schnietz, J. Schörmann, S. Potthast, J.W. Gerlach, J. Vogt and K. Lischka
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T. Bahners, K. Opwis, E. Schollmeyer, B. Marquardt and L. Prager
Selbstreinigende Lack-Topcoats auf Planen und Membranen durch photonisch induzierte Mikrofaltung
DTNW-Mitteilung **53** (2006) 1-52

R. Bandari, A. Prager-Duschke, C. Kühnel, U. Decker, B. Schlemmer and M.R. Buchmeiser
Tailored ring-opening metathesis polymerization derived monolithic media prepared from cyclooctene-basid monomers and cross-linkers
Macromolecules **39** (2006) 5222-5229

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Macromolecular Chemistry and Physics **208** (2007) 1428-1436

R. Bandari, W. Knolle, A. Prager-Duschke and M.R. Buchmeiser
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Macromolecular Rapid Communications **28** (2007) 2090-2094

R. Bandari, C. Elsner, W. Knolle, C. Kühnel, U. Decker and M.R. Buchmeiser
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Journal of Separation Science **30** (2007) 2821-2827

R. Bandari, W. Knolle and M.R. Buchmeiser
Preparation, characterization and application of electron-beam curing-derived monolithic materials
Macromolecular Symposia **254** (2007) 87-92

B. Bantu, K. Wurst and M.R. Buchmeiser
N-Acetyl-N, N-dipryrid-2yl (cyclooctadiene) rhodium (I) and iridium (I) complexes: Synthesis, X-ray structures, their use in hydroformylation and carbonyl hydrosilylation reactions and in the polymerization of diazocompounds
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Macromolecular Materials and Engineering **291** (2006) 493-498

F. Bauer
Functionalized inorganic/organic nanocomposites as new basic raw materials for adhesives and sealants
Pitture e vernice - European coatings **82** (2006) 35

F. Bauer and H.G. Karge
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In: Molecular Sieves - Science and Technology, Volume 5, Eds. H.G. Karge and J. Weitkamp, 249-364. Springer, Berlin, Germany (2006)

F. Bauer, E. Bilz and A. Freyer
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Czechoslovak Journal of Physics **56**, Supplement 4 (2006) D417-D424

F. Bauer, U. Decker, H. Ernst, M. Findeisen, H. Langguth, R. Mehnert, V. Sauerland and R. Hinterwaldner
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F. Bauer, E. Bilz, W.H. Chen, A. Freyer, V. Sauerland and S.B. Liu
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Studies in Surface Science and Catalysis **170** (2007) 1096-1103

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Nuclear Instruments and Methods in Physics Research B **265** (2007) 87-91

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R. Böhme and K. Zimmer
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R. Böhme, K. Zimmer and B. Rauschenbach
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Applied Physics A **82** (2006) 325-328

R. Böhme and K. Zimmer
Ultraglatt und hochpräzise: Laserabtrag an der Rückseite transparenter Dielektrika
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R. Böhme and K. Zimmer
Smooth and precise: Laser backside etching of transparent dielectrics
Photonik International (2007) 80-83

R. Böhme and K. Zimmer
Laser-induced writing of submicron surface relief gratings in fused silica on the fly
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R. Böhme, S. Pissadakis, M. Ehrhardt, T. Rudolph, D. Ruthe and K. Zimmer
Backside etching of fused silica with ultra-short laser pulses at the interface to absorbing liquid
Journal of Physics: Conference Series **59** (2007) 173-176

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The reduction of the threshold fluence at laser-induced backside wet etching due to a liquid-mediated photochemical mechanism
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Applied Surface Science **253** (2007) 8091-8096

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Nuclear Instruments and Methods in Physics Research B **257** (2007) 392-396

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Plasma Processes and Polymers **4** (2007) S584-S587

Y. Bohne, D. Manova, C. Blawert, M. Störmer, W. Dietzel and S. Mändl
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Y. Bohne, D. Manova, S. Mändl, H. Neumann and B. Rauschenbach
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Plasma Processes and Polymers **4** (2007) S660-S663

M. Braeckevelt, H. Rokadia, G. Mirschel, S. Weber, G. Imfeld, N. Stelzer, P. Kuschk, M. Kästner and H.H. Richnow
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M.R. Buchmeiser
Recent progress in ring-opening metathesis polymerization
Chimia Oggi - Chemistry Today **25** (2007) 78-81

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