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BIANNUAL REPORT 2008/2009

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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 2008/2009 and as documentation of publications, 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 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 successful cooperation with chemical, optical, and semiconductor industry was continued.

Scientific success and resulting innovations achieved in 2008/2009 as well as their transfer into commercial products are the result of long-term research and development, based knowledge gained by the IOM staff members in close cooperation with our research and industrial partners.

In this report the IOM presents its scientific activities and major achievements in the years 2008 and 2009. In this context, the Biannual Report 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 Institut 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 Institut for their active and excellent contributions to a successful development.

Leipzig, January 2010

Prof. Dr. Dr. h. c. Bernd Rauschenbach

A warm welcome to our new colleague Stefan G. Mayr. Since May 2009 he is Professor at the University of Leipzig and group leader at the Leibniz-Institut für Oberflächenmodifizierung e. V. We are glad that he joined us and wish him success and good luck for his future.

In 2009 Tom Scherzer received the prestigious Paul Dufour Award from RadTech Europe, the European Association promoting the use of UV/EB technology, at the RadTech Europe Conference in Nice for his contributions on continuous monitoring of process parameters in UV curing processes.
Congratulations!

The 16th International Conference on Ion Beam Modification of Materials (IBMM2008) was jointly organised in Dresden by the Leibniz-Institut für Oberflächenmodifizierung e. V. and the Forschungszentrum Dresden-Rossendorf in September 2008. It was the major international forum to present and discuss recent results in ion-related materials research, and to point into the future of the field.

The Leibniz-Institut für Oberflächenmodifizierung e. V. organised the 18th International Symposium on Olefin Metathesis and Related Chemistry (ISOM18) in Leipzig in September 2009. Recent developments in the area of olefin polymerization were emphasized, especially those aimed at controlling polymer structure and/or molecular weight through catalyst design.

Scientific and Technology Results

Reports and Selected Results

Polymer modification by radiation-induced processes for biological applications

C. Elsner, C. Ernst, S. Reichelt, A. Boulares-Pender, M. R. Buchmeiser

Introduction

In recent years, considerable interest focused on tailoring polymer surfaces for specific biological and biomedical applications, e.g. aiming on an increase in the performance by immobilization of biomolecules on the test supports [1]. Indeed, the inert nature of polymers makes polymer/surface functionalization for the consecutive binding of bioactive compounds inevitable. Moreover, spatial resolved immobilization techniques become increasingly important for miniaturized, integrated systems. Therefore, radiation-based methods have been established for surface activation and functionalization. However, final biological test systems consist of more than one component and the discrimination between unspecific adsorptive and specific binding effects as well as the preservation of bioactivity have to be managed, mostly on the first stage of polymer modification. Herein, we present results concerning the modification of polymeric surfaces by radiation-induced processes such as UV, electron beam, and plasma-mediated grafting processes for tailoring protein immobilization for several applications.

Excimer VUV-triggered photodegradation

Aminofunctional surfaces are widely used for the attachment of biomolecules, and suitable methods for their micropatterning are of special interest for localized immobilization techniques with high spatial resolution. Thus, the photodegradation of condensed 3-aminopropyltrimethoxysilane (APTMS) films by a Xe_2^*-excimer lamp set-up was elucidated [2]. Under atmospheric conditions APTMS undergoes an incomplete hydrolysis-condensation reaction leaving a large number of methoxy groups unreacted. High-energy VUV photons at 172 nm are able to cleave chemical bonds up to a dissociation energy of 7.2 eV, which enables the degradation of that loosely condensed APTMS-based films to a SiO_x-like network including the degradation of the amino functionality (Table 1). The addition of small amounts of oxygen into the nitrogen inert-rendered irradiation zone influences the efficiency and the rate of that transformation process. The optimal oxygen concentration was determined to be between 1000 and 5000 ppm, which is a balance between a reduced irradiation dose reaching the surface due to the absorption of photons by oxygen and the generation of reactive oxygen species which promote the surface transformation process (Figure 1).

Table 1: Chemical surface composition (XPS) of APTMS-based films irradiated under different oxygen concentrations at 20 minutes irradiation time

oxygen [ppm]	surface composition [atom-%]			
	C	N	O	Si
without irradiation	38	11	28	23
50	20	5	47	28
200	11	2	52	35
500	10	2	50	38
1000	10	2	49	39
5000	9	3	51	37

For micropatterning, an APTMS-coated silicon wafer was irradiated through a mask. During the process, amino groups in the exposed regions are degraded whereas those in the unexposed regions are retained. For visualization purposes, the irradiated wafer has been exposed to the fluorescent dye fluorescein isothiocyanate, which

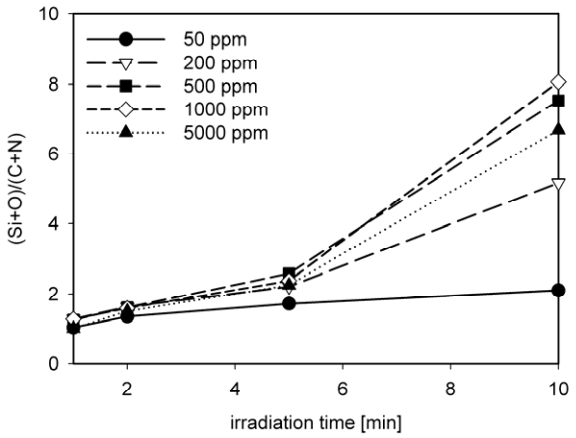
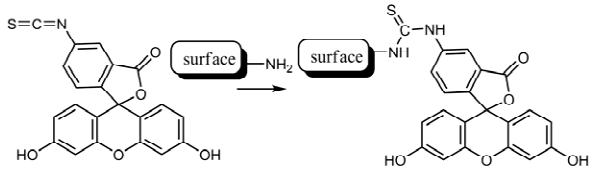


Figure 1: Changes in the ratio of inorganic to organic surface content as expressed by the (Si+O)/(C+N) ratio during the irradiation of APTMS-based films at different oxygen concentrations determined by XPS.



Scheme 1: Labeling of primary amino-groups with fluorescein isothiocyanate.

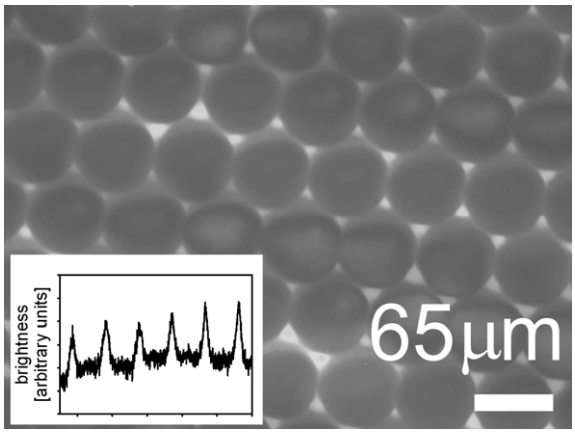


Figure 2: Fluorescence image of a micropatterned amino-functional surface modified with fluorescein isothiocyanate. Bright: Modification of the retained amino-groups with the fluorescent dye. Inset: Brightness profile through the third spot line. The distance between the maxima corresponds to 65 µm.

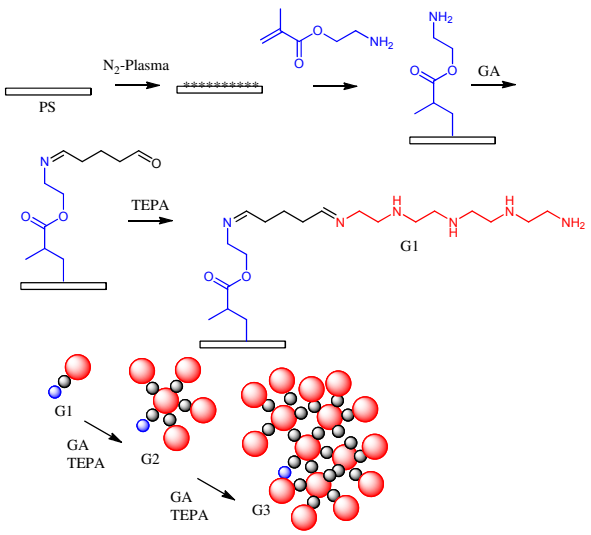
reacts with primary amino-groups as indicated in Scheme 1. Fluorescence microscopy indicates a micropatterned surface with high spatial resolution (Figure 2).

Modification of polymeric surfaces by plasma treatment

Besides wet chemical treatment with oxidizing agents radiation-induced processes are a versatile strategy for the primary modification of inert polymer surfaces. Indeed, after exposure of polystyrene to N_2 or O_2 plasma an increased atomic concentration of nitrogen or oxygen was found (Table 2). However, after such an initial activation, the functionalization density and the

Table 2: Chemical surface composition (XPS) of nitrogen and oxygen plasma treated polystyrene (PS)

	surface composition [atom-%]		
	C	**N**	**O**
PS	98.4	0	1.6
N_2 plasma treated PS	71.1	10.6	18.3
O_2 plasma	79.4	1.2	19.4



Scheme 2: Principle of the build up of hyperbranched structures on plasma treated polystyrene surfaces.

homogeneity of functional groups are rather low and the stability of the obtained super-hydrophilic surfaces is limited due to hydrophobic recovery effects. To increase functionality density at the surface, several types of polymeric structures can be added to or built onto the activated surface, e.g. polymeric brushes, dendritic and hyperbranched structures. For example, based on the alternating reaction of glutardialdehyde (GA) and tetraethylenepentamine (TEPA), a hyperbranched polymer with terminal amine functions for the covalent attachment of biomolecules, e.g. proteins, was built up on nitrogen-plasma-treated polystyrene surfaces [3].

The introduced amine functions were labeled with pentafluorobenzaldehyde and analyzed by XPS, confirming the successful attachment of each generation of branching (Figure 3). However, the exponential growth of the atomic N/F quotient with each generation of branching indicates a limitation of the binding sites even for small molecules. BSA and trypsin were covalently immobilized on the branched graft polymer. The amount of the immobilized proteins was analyzed by the BCA test and the hydrolysis of the trypsin substrate Bz-Arg-pNA, respectively. In both cases, an enhanced activity was found for the generated hyperbranched structures on nitrogen-plasma-treated surfaces (Figure 4).

By studying the feasibility to produce functional coatings on thermo-sensitive substrates by the use of a plasma-induced polymerization process the curing of more than 10 micrometer thick acrylate-based coatings without the addition of a

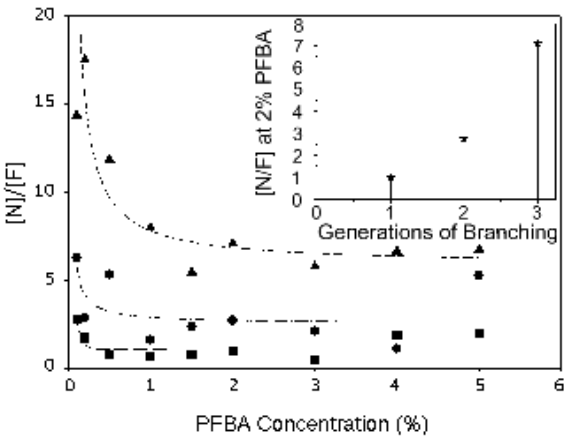


Figure 3: N/F atomic ratio versus PFBA concentration of PS-AEMA-(GA-TEPA)$_n$. Insert: Variation of the N/F ratio versus number of generation, at a given concentration of 2% PFBA in ethanol.

photoinitiator was observed [4]. The bulk properties (micro-hardness) of the obtained coatings were comparable to UV-cured coatings prepared by the use of a Hg-lamp whereas the surface properties (surface energies) differed extremely. The absence of the photoinitiator reduces the risk of the release of VOCs based on cleaved initiator fragments and may have a positive influence on the reduction of background fluorescence. Therefore, the process may be especially suited for the coating on substrates which are in contact with biological systems. ATR-IR spectroscopy revealed a nearly quantitative consumption of the acrylic double bound (810 and 1405 cm^{-1}) in selected cases (Figure 5). Optical emission spectroscopy in the range of 150-500 nm was applied for the characterization of the used nitrogen plasma to identify species which may initiate the photoinitiator-free curing. Indeed, emissions at approx. 150, 175, and

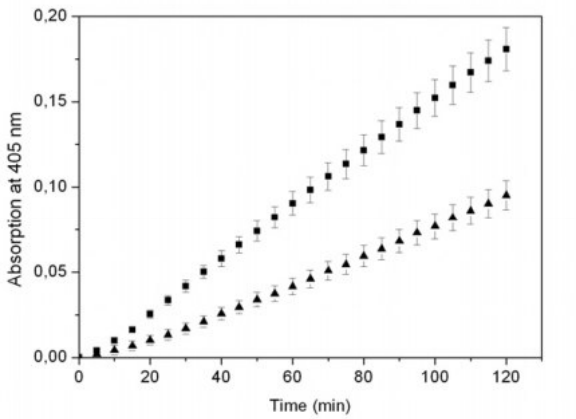


Figure 4: Activity of trypsin immobilized at the surface of non-plasma and nitrogen plasma treated PS-AEMA-(GA-TEPA)$_3$-surface monitored by the tryptic release of p-nitroaniline from Bz-Arg-pNA, measured at 405 nm.

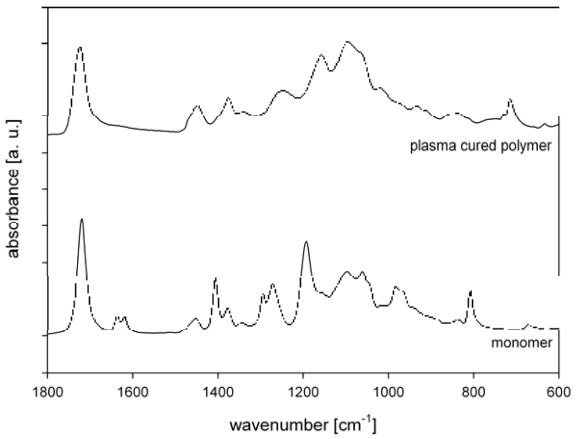


Figure 5: ATR-IR spectrum of tripropyleneglycol diacrylate in the monomeric and plasma cured form without the addition of a photoinitiator.

230-270 nm were observed as a source for photoinitiator-free curing (Figure 6). An emission line at 399.75 nm was indicative for the presence of N^+ ions as a result of the cleavage of molecular nitrogen. This process requires an energy of 9.8 eV, which may be raised from high-energy photons.

Monolithic materials

Monolithic polymer-based materials are increasingly important for separation science and heterogeneous catalysis but are also suitable for regenerative medicine [5]. Recently we have developed radiation-based polymerization techniques based on electron beam curing for the generation of monoliths in micro-dimensional systems which favors other radiation-based techniques due to several aspects. However, the introduction of functional units into the polymer backbone by the copolymerization of functional monomers seems not straightforward because

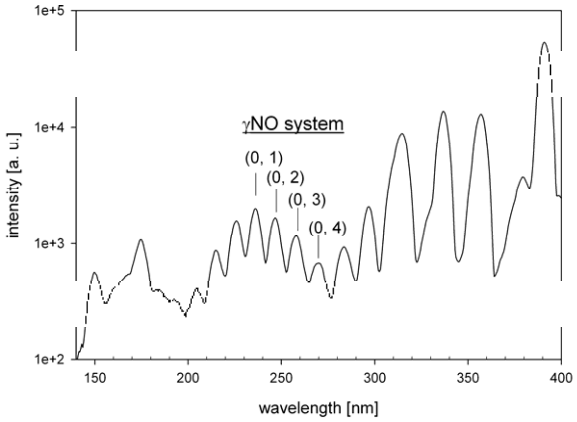
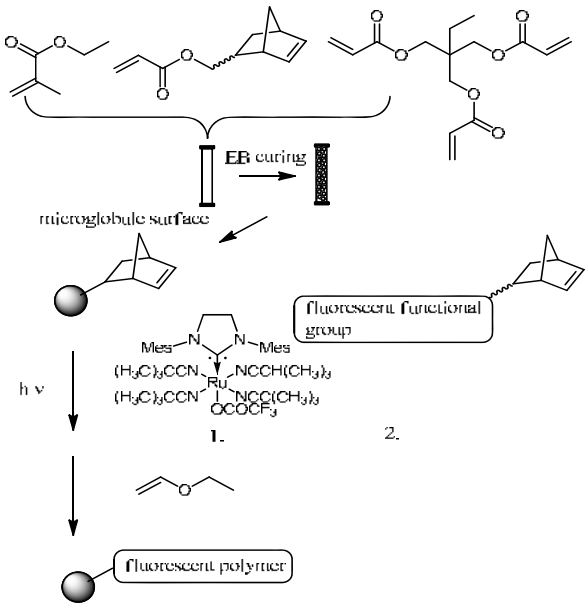


Figure 6: Optical emission spectrum of the nitrogen discharge.

Figure 7: Spatial resolved functionalized capillary monolith.

any change in the polymerization mixture strongly influences the phase separation behavior during the polymerization process and finally the pore structure of the monolith. Thus, only small amounts of functional monomers for the introduction of orthogonal functional groups can be copolymerized, followed by other polymerization techniques to enhance the functionality. This has been shown for post-cure "grafting from"-polymerization techniques, e.g. ROMP using Grubbs-type catalysts. In recent years a lot of efforts were devoted to the integration of multiple functionalities in miniaturized smart devices. In the case of monolithic media the spatial resolved functionalization becomes increasingly important, e.g. for the generation of reaction cascades for analytical applications [6]. In order to demonstrate a new method of spatial resolved functionalization of capillary monoliths, a recently developed UV-activable Grubbs-type initiator [7] for the ROM-polymerization of a fluorescent monomer for monitoring purposes during UV irradiation through a mask was used (Scheme 3). Further spatial resolved immobilization techniques were developed for UV and EB curing processes and ROMP-based on the grafting of functional acrylates and norbornenes (Figure 7) [8]. The obtained findings were used for the construction of miniaturized capillary and chip systems for biocatalytical and analytical purposes.



Schema 3: Principle of the spatial resolved functionalization of a capillary monolith by ROMP using a UV-activated initiator and a fluorescent monomer.

Conclusions

Different methods for tailoring protein immobilization to surfaces were developed on the basis of radiation-induced surface functionalization processes comprising the use of plasmas, electron beam and UV irradiation. Thereby, the work presented here focused on spatial resolved functionalization and immobilization techniques. Moreover, modification routes for polymeric materials were established, which allow for a discrimination of covalent protein binding and non-specific protein adsorption comprising the build-up of hyperbranched structures from simple monomers as well as the attachment of multifunctional polymers to activated surfaces. Based on the presented investigations, the current work focuses on the development of miniaturized analytical and preparative polymeric monolithic systems for affinity chromatography.

Acknowledgements

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Surface refinement of radiation-curable varnish coatings by 172 nm excimer VUV radiation

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in collaboration with
R. Mehnert, Cetelon Nanotechnik GmbH, Eilenburg, Germany
C. Riedel, Innovative Oberflächentechnologien GmbH, Leipzig, Germany

Introduction

Excimer UV lamps, which were developed about 20 years ago [1,2], were establishing new ways for acrylate-based radiation-curable coatings. In particular, the short-wavelength 172 nm Xe_2* VUV photons became highly important. Due to the low penetration depth of the high-energy photons into acrylates (typically 100 nm), only the surface layer of the coating is subject to polymerization. The concomitant volume contraction leads to the micro-wrinkling of the thin polymerized skin, which "swims" on a liquid layer. In a second step, these surface structures are fixed by through-curing of the subjacent uncured acrylate layer, either with the aid of UV light (e.g. with a medium-pressure mercury lamp or by use of a XeCl* excimer lamp with an emission at 308 nm), or by electron beam curing. The basic principle of the photochemical micro-folding technology is shown in Figure 1. The effect of this surface structuring is a deep matt appearance of the coating surface.

But there are still other advantages resulting from 172 nm photons treatment. The high photon energy (7.2 eV) allows curing without photoinitiator since acrylates are absorbing < 230 nm. Moreover, it leads to better efficiency in double bond conversion due to the generation of additional radicals, which results in enhanced surface properties of the coating [3]. Thus, an excellent surface refinement is achieved.

VUV-induced micro-folding creates random structures (Fig. 2) depending on a wide variety of chemical and technological parameters. Extensive investigations on the mechanism of the micro-folding process via elucidation of the curing and wrinkling kinetics, of the surface properties, and the development of a reliable excimer technology resulted in a successful introduction in different branches of the industry [4-7].

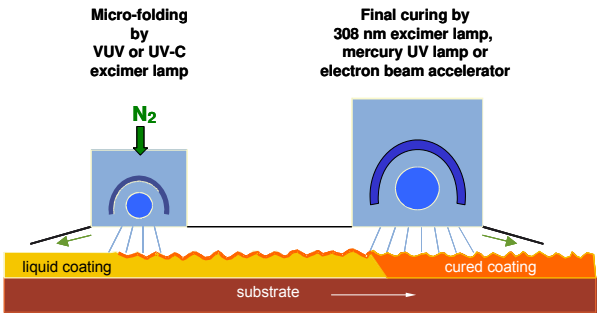

Figure 1: Technology of photochemical micro-folding.

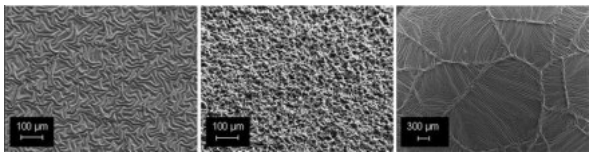

Figure 2: Examples of micro-wrinkled random structures.

Micro-folding kinetics

Materials for the investigation of the micro-folding behaviour of acrylic monomers and oligomers were chosen with the objective to cover a wide range of relevant properties, e.g. type, molecular weight, viscosity, functionality, glass transition temperature, surface and shrinking tension of the corresponding compounds. Acrylates and acrylate-based formulations used in this study were sponsored by CYTEC Surface Specialities (Belgium), SARTOMER (France), Bayer MaterialScience AG and Jäklechemie GmbH & Co KG (Germany), Rahn AG (Switzerland), Evonik – Goldschmidt Industrial Specialities (Germany), and Cetelon Nanotechnik GmbH (Germany).

For monitoring and recording of the folding kinetics of representative acrylate components and varnish formulations including UV powder coatings, a novel device for the real-time measurement of the folding kinetics (RT-FK device) was developed. It is based on the measurement of the decrease of the incident light intensity due to diffuse scattering of the light at a wrinkled surface generated during the micro-folding process. The progress and the extent of micro-folding were detected by the use of a gallium phosphite photodiode, whose response ranged from about 150 to 550 nm. Measurements are carried out either in transmission (for clear coats) or in reflection mode. In addition, a high-speed video

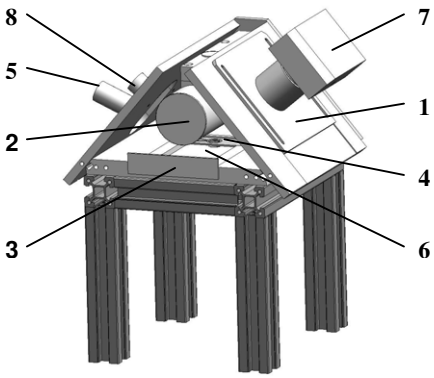

Figure 3: Layout of the real-time chamber for the investigation of the kinetics of micro-folding.

camera may be used. The basic set-up of the equipment is schematically shown in Figure 3.

It consisted of adjustable sidewalls (1) for an optimum adjustment of the camera. At the bottom of the chamber, an electrically heatable sample table (3) was installed on a slide mechanism, which permitted an easy change of the sample. A liquid acrylate sample was applied to a glass object plate which was placed on the sample table (4). The micro-folding process was initiated by a 172 nm XERADEX® excimer lamp (2) (Radium Lampenwerke, Wipperfürth, Germany), which had a length of 120 mm, equipped with a shutter element for variation of the exposure time. The maximum intensity at the surface of the sample, i.e. in a distance of 30 mm from the lamp, was determined to be 8 mW cm^{-2}. It could be gradually reduced to 1.25 mW cm^{-2} by metal meshes with different mesh sizes, mounted between the lamp and the sample.

Flushing the chamber with nitrogen allowed the irradiation of the samples with various VUV doses in an inert atmosphere while the oxygen content inside the chamber was controlled by a V6 oxygen measurement system (Metrotec, Germany). The intensity of the measuring light emitted by a green-light diode (5), which showed no emission in the UV range to prevent curing before micro-folding, was recorded after transmission through the sample by a VUV "FlatLog" sensor (6), which was placed under the sample. Both the diode and the VUV sensor were supplied by EPIGAP GmbH (Berlin, Germany). Alternatively, the VUV sensor could be used for measuring the dose and dose rate of the VUV radiation at the sample position. Video sequences were recorded by use of a high-speed camera (7) (model pco 1200 s; PCO AG, Germany). The final curing of the sample after micro-folding was carried out by a 365 nm UV-emitting LED from Dr. Groebel GmbH (Germany).

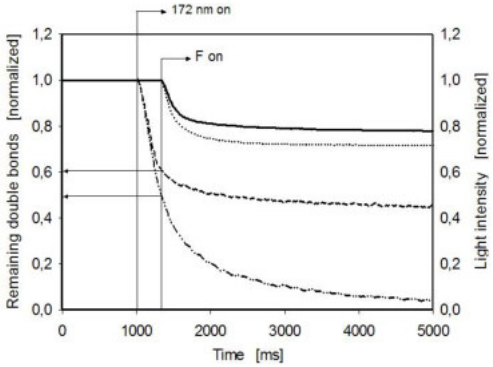

Figure 4: Correlation of micro-folding and curing kinetics of TPGDA induced by 172 nm photons

Folding ——— *0.9 mJ cm^{-2}* ····· *24.9 mJ cm^{-2}*

Curing – – – *0.9 mJ cm^{-2}* — · · *24.9 mJ cm^{-2}*

In a typical test procedure, the irradiation chamber was flushed with nitrogen in order to realize a final oxygen content < 10 ppm. After insertion of the sensor into the slide, a coated glass object plate was placed on the sample table at a temperature, which depended on the consistency of the formulation. The sensor recording cycle and the camera were started. After about 2 s, the excimer lamp was switched on, and the sample was irradiated with 172 nm photons for 0.2 to 20 s. After VUV exposure, the recording cycle of the sensor and the camera were stopped. Samples were either internally through-cured by the 365 nm LED or externally via EB curing. Then the data of the sensor were read out with the aid of a PC.

We commenced our investigations with the monitoring of the micro-folding of tripropylene glycol diacrylate (TPGDA) with the aid of the newly developed RT-FK chamber. The micro-folding kinetics of TPGDA in the absence of any photoinitiator is shown in Figure 4.

We tried to correlate the micro-folding speed with the rate of polymerization, which can be conveniently determined by RT-FTIR spectroscopy using a Digilab FTS 6000 spectrometer, a Golden Gate diamond ATR accessory (Specac, UK) and a XERADEX® excimer lamp similar to that in the RT-FK-chamber. In Figure 4, it is clearly demonstrated that a certain minimum double bound conversion is required to generate shrinking tensions in the cured skin before micro-wrinkling can start. Moreover, the influence of VUV dose on double bond conversion, folding speed, and coarseness of the surface structure is reflected in the different attenuation of light. The last conclusion is emphasised by the kinetics of two components with different surface topologies, which is shown in Figure 5.

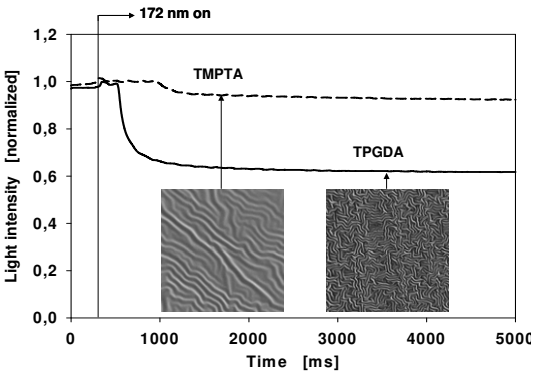

Figure 5: Influence of different surface structures on the attenuation of light.

Using the RT-FK-device for further investigations, the influence of temperature, viscosity, photoinitiators and their concentration, nano- and microparticles, and additives on the kinetics and the surface topology of the formulations was studied. Moreover, the micro-folding of UV powder coating could be investigated.

Thus, this device proves to be an excellent tool for designing of excimer VUV irradiation equipment and for the formulation of special radiation-curable varnishes.

Further effects of 172 nm irradiation on surface refinement

This kind of physical matting is advantageous because of the transparency of clear coats matted in this way. In particular, application of this technology to nanocomposite varnishes (Cetelon Nanotechnik) resulted in coatings with excellent scratch and abrasion resistance, which might be suited, e.g., for touch-screens since the sensitivity to fingerprints is extremely reduced as well.

Further beneficial effects are based on a better efficiency in double bond conversion in comparison to polychromatic UV light and in the higher energy (7.2 eV) of 172 nm photons generating additional free radicals, which is shown in Figure 6. This way, a higher micro-hardness is achieved on the surface of the coating (Fig. 7).

The increased polymerization and cross-linking leads to a higher gas and liquid barrier efficiency, better chemical and weathering resistance, as well as corrosion protection.

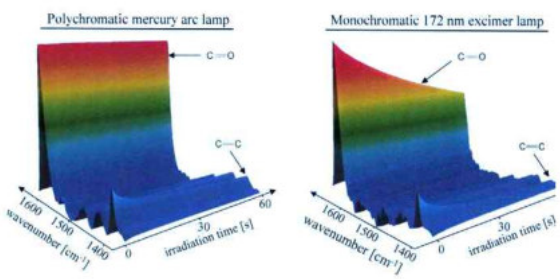

Figure 6: Comparison of C=C and C=O bond conversion.

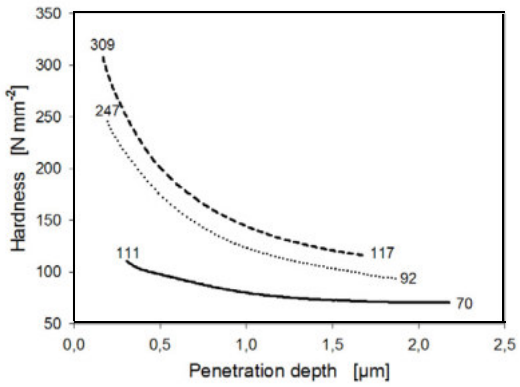

Figure 7: Micro-hardness of a nanocomposite varnish obtained by a Fischerscope H100C hardness tester.

UV 450 mJ cm^{-2}
VUV 10 mJ cm^{-2} + UV 450 mJ cm^{-2}
VUV 20 mJ cm^{-2} + UV 450 mJ cm^{-2}

Moreover, the surface friction can be varied. Figure 8 shows some results of the determination of this parameter using a TENSOR-SF device (INNOWEP GmbH, Würzburg, Germany).

Finally, the 172 nm treatment of the coating generates polar groups on the surface leading to a higher surface tension and, thus, to a better wetting. But if there is a large amount of free radicals in the system as a result of a high VUV dose application, a total curing of some ten micrometers coating without micro-folding is possible. This means that a special dose window exists for the micro-folding of each formulation.

Micro-folding technology

Only some time after the invention of the excimer lamps, the IOM and the Heraeus Noblelight

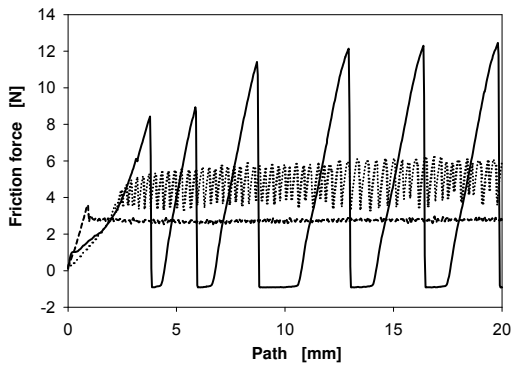

Figure 8: Different friction behaviour of smooth and micro-structured surfaces of the varnish.

smooth on smooth surface
micro-structured on smooth
micro-structured on micro-structured

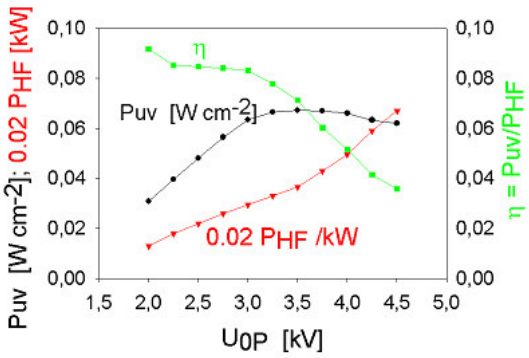

Figure 9: Influence of voltage U_{OP} on the UV output P_{UV} and the efficiency η.

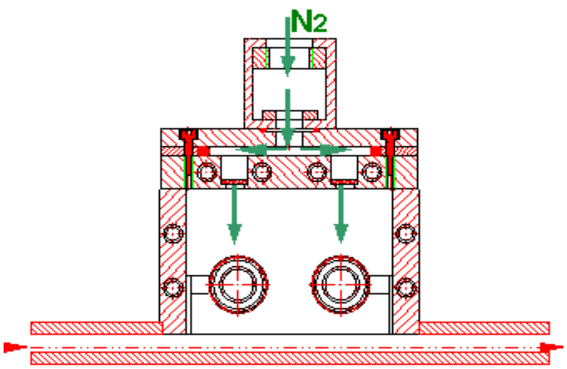

Figure 10: Scheme of a nitrogen-cooled 172 nm excimer double lamp of the Heraeus type.

GmbH (Hanau, Germany) intensified the further development of the lamps to be used for the micro-folding process. Since the efficiency of lamps, which are water-cooled only inside, decreases with rising temperature on the outer surface due to high electrical input (see Fig. 9), the lamps got a new design with direct injection of nitrogen for cooling, keeping the lamp clean, and inertization of the curing process (Fig. 10).

For two years, the Innovative Oberflächentechnologien GmbH (IOT) is manufacturing these systems and provides the industry with this technology successfully.

Figure 11: EXCIRAD 172 nm system of the IOT GmbH with a working width of about 2 m.

Figure 12: From glossy to matt cladding of buildings (Steni Norden a.s., Norway).

Meanwhile, an alternative 172 nm excimer lamp system, which is cooled by nitrogen only, is available from the Radium Lampenwerke GmbH (Wipperfürth, Germany).

Application of micro-folding

Six years ago, the first industrial excimer VUV matting plant for the production of matt cladding panels was installed (see Fig. 12).

Meanwhile, finish foils, decor paper, different flooring material, metal elements for the internal decor of cars are provided with physically matted scratch- and abrasion-resistant coatings.

Acknowledgment

The authors are indebted to the Sächsische Aufbaubank (SAB, Dresden) for funding and to the companies, which supported this project by providing materials. We also wish to thank the Heraeus Noblelight GmbH for successful longtime collaboration, the Radium Lampenwerke GmbH for the development of an excimer spot lamp and the EPIGAP GmbH for the joint development of the mobile FlatLog VUV dosimeter as well as for providing the LED light source. We are also grateful to the analytic group of the IOM.

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UV-induced conversion of polysilazanes into SiO_x networks: access to flexible, transparent barrier coatings

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Introduction

Thin inorganic films of about 100 nm thickness deposited onto polymeric foils as barrier coatings against water vapour and oxygen are of significant interest in many areas of material science. The successful realization of numerous high-tech applications such as flexible organic light-emitting diodes, thin-film photovoltaic modules, or multilayer arrangements for applications in electronics is dependent on the development of suitable barrier materials, which protect these devices against oxygen and moisture. Vacuum-deposited metal layers provide the highest barrier values associated with high flexibility. However, where high transparency is required, silicon and aluminium oxides still dominate. The state of the art for producing such layers is defined by techniques like physical vapour deposition, plasma-enhanced chemical vapour deposition, reactive sputtering, and sol-gel procedures. An alternative technique is the conversion of polysilazane layers in a process proceeding at ambient temperature and pressure.

The thermal conversion of perhydropolysilazane (PHPS), which consists of $-(SiH_2\text{-}NH)-$ units arranged in cyclic structures, into SiO_x in the presence of moisture has already been investigated and described as a consecutively proceeding hydrolysis/condensation process [1]. The thermally triggered condensation proceeds slowly on a time scale of some hours at the glass transition temperatures of polymers like PET and PEN (80 and 100 °C, respectively). Hence, efforts have been undertaken to accelerate this process by adding catalysts [2]. Regrettably, only partial success could be achieved. To overcome these limitations, the photochemical conversion of thin layers of PHPS in the presence of oxygen into a SiO_x network has been studied and described here. Research has been carried out in the framework of the research project „Herstellung von SiO_x-Barriereschichten aus Polysilazanen bei Normaldruck und niedriger Temperatur mittels VUV-Strahlung" (12/06–11/09, 01RI06007). Results have been published elsewhere [3-5].

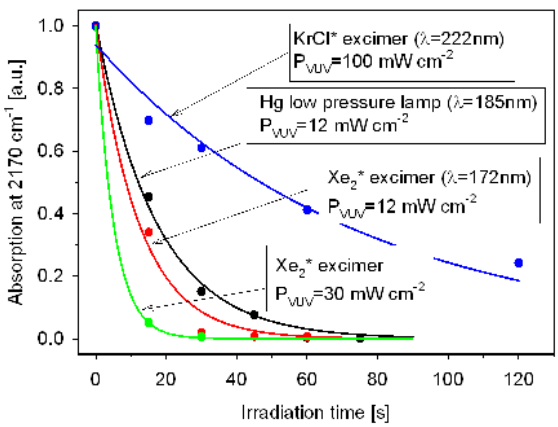


Figure 1: Kinetics of the Si-H bond decomposition.

Results

Quantum chemical calculations on the model molecule $NH_2\text{-}SiH_2\text{-}NH\text{-}SiH_3$ revealed that, as a result of a multi-stage process, the excitation by high-energy VUV photons leads to Si-N bond scission. The energy gap S1-S0 for the absorption threshold was calculated to be 5.63 eV (λ = 220 nm). Consequently, photons with wavelengths ≤ 220 nm should be able to trigger Si-N bond scission.

By means of time-resolved FTIR spectrometry the disappearance of Si-N bonds and the appearance of Si-O bonds could be investigated. Since N-centred radicals abstract H atoms from the Si atoms forming ammonia, the decomposition of the silazane can be followed by the Si-H decomposition which is evaluable more certainly than the Si-N band because of the isolated position of the Si-H band in the FTIR spectrum.

The kinetics of the Si-H decomposition (Fig. 1) has been characterized by a first-order law with apparent rate constants of $8.2 \times 10^{-2}\ s^{-1}$ and $5.8 \times 10^{-2}\ s^{-1}$ for λ = 172 nm and 185 nm, respectively. Irradiation by a KrCl* excimer lamp (λ = 222 nm) resulted in a more slowly degradation of the Si-H bond with an apparent rate constant of $1.4 \times 10^{-2}\ s^{-1}$. This result supports the finding of the quantum chemical calculations with an excitation threshold of about 220 nm. Subsequent to the Si-N bond scission the forma-

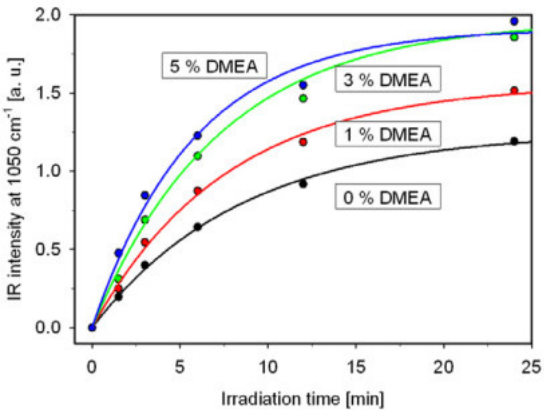


Figure 2: Kinetics of the Si-O-Si bond formation.

tion of the Si-O network proceeds with apparent rate constants of about 2×10^{-3} s^{-1}. Applying tertiary amines like dimethylethanolamine (DMEA), for example, as catalysts in the range of 1-5%, the rate constants can be increased up to 3×10^{-2} s^{-1}. Concomitantly, the absolute height of the Si-O-Si vibration peak at about 1050 cm^{-1} increases by about 50% (Fig. 2).

An important criterion for long-time stability and density of the SiO_x layers formed is the chemical composition of the layer. Using X-ray photoelectron spectroscopy (XPS) depth profiles of the composition could be obtained showing a nearly complete conversion of the polysilazane Si-N networks to SiO_x networks with an elemental formula $SiO_{1.7}N_{0.06}$, for instance (Fig. 3).

By use of X-ray reflectometry (XRR) the density of such layers produced on silicon wafers could be measured. The results shown in Table 1 illustrate the significant higher density of layers produced by using VUV irradiation compared with samples obtained by applying different thermal treatments ("var. 1 or 2").

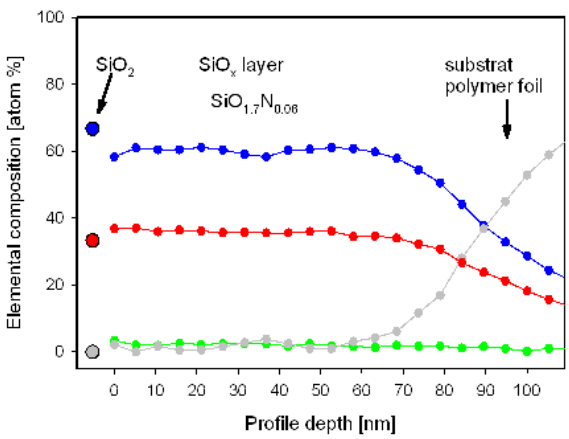


Figure 3: XPS depth profile of elemental composition of a polysilazane-derived SiO_x layer on polymer foil.

Table 1: Density and refraction index of PHPS-derived SiO_x layers compared with thermally grown SiO_x and fused silica.

Treatment	Density [g cm^{-3}]	Refraction Index (λ=550nm)
24 h 150 °C	1.60	1.4592
6 h 215 °C	1.58	1.4672
1 h 600 °C	1.87	1.4482
VUV, var. 1	2.05	1.4962
VUV, var. 2	2.15	1.4832
SiO_x therm.	2.18	1.4982
fused silica	2.203	1.4601

The results allowed for establishing both a continuous and a discontinuous process for the production of fully transparent, flexible barrier coatings.

Figure 4 shows a pilot plant for the production of such coatings on polymer foils (width 20 cm) from roll to roll at a speed up to 10 m min^{-1}. SiO_x layers of 80-120 nm thickness (Fig. 5) produced in this way show oxygen transmission rates (OTR) < 0.2 cm^3 m^{-2} d^{-1} bar^{-1}. Double layers prepared by this technique allowed for the realization of OTRs ≤ 0.1 cm^3 m^{-2} d^{-1} bar^{-1}. Water vapour transmission rates (WVTR) reach values down to 2.0 g m^{-2} d^{-1}.

Different UV sources in the wavelength range of 160-230 nm, i.e. 172 nm Xe_2* and 222 nm KrCl* excimer as well as 185 nm mercury low pressure (HgLP) lamps, are commercially available for these purposes and have been tested by evaluating UV spectra, radiant power, energy costs and others.

Figure 4: View of a pilot plant for the roll-to-roll production of barrier coatings on 20 cm wide polymer foil at a coating speed up to 10 m min^{-1}.

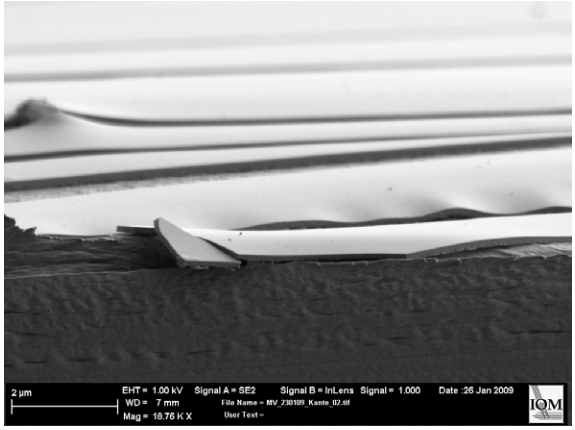


Figure 5: SEM image of a polysilazane-derived SiO_x monolayer.

For the set-up of the pilot plant, it was of significant importance to develop Xe* excimer lamps with high efficiency of about 40% for the conversion of electrical input power into VUV radiation power and with an absolute power output of > 35 mW cm^{-2} in the irradiation plane. The partner within the research project, the OSRAM GmbH, Wipperfürth, provided such lamps for installation in the VUV irradiation channel of the pilot plant.

By means of the successful development of a new tuneable electronic control gear together with the application of special VUV reflectors and with a computer-aided geometry optimization of the lamps the absolute VUV output power per unit area could be increased up to 38 mW cm^{-2}. i.e. up to 175% deposited VUV energy compared to the deposited energy on the surface at the beginning of the project (Fig. 6).

An important contribution of the Clariant Produkte (Deutschland) GmbH within the research project consisted in the development of a new technology for producing PHPS with the aim to replace the customary process, which uses a pyridine dichlorosilane adduct as starting material, by a cost-efficient process using dichlorosilane directly in the reaction with gaseous ammonia to form perhydropolysilazane, and its up-scaling from the laboratory into the pilot and the industrial scale. Thereby, emphasis was laid on an oxygen and water vapour free process to ensure a very pure product with a minimum content of silanols instead of Si-NH-Si (silazane) units.

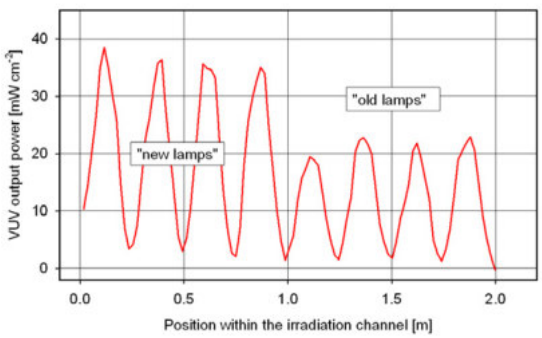


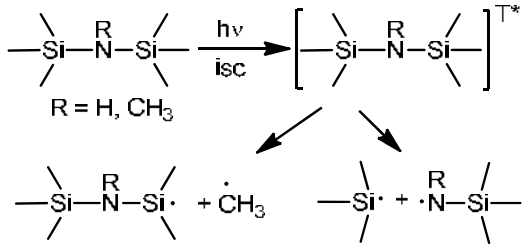
Figure 6: Comparison of the VUV output of Xe excimer lamps from OSRAM GmbH Wipperfürth at the beginning (right) and at the end (left) of the research project.*

Primary photoinduced reactions

Working on the research project a special challenge was the elucidation of the primary processes proceeding immediately after photoexcitation of the polysilazane matrix converting the Si-NH-Si network into a dense Si-O-Si network. Below the investigation on this topic is described in more detail.

Because of the extraordinary sensitivity of PHPS against hydrolysis these investigations were carried out on partially or completely methyl-substituted silazanes. A combined experimental approach on disilazane model compounds was chosen for the direct study of fast processes (kinetic measurements by laser flash photolysis), to investigate intermediate species by trapping (low-temperature matrix EPR), and to identify the final products by GC-MS following steady state photolysis [5]. Quantum chemical calculations support the assignment of UV and EPR spectra and are used to distinguish between different reaction pathways.

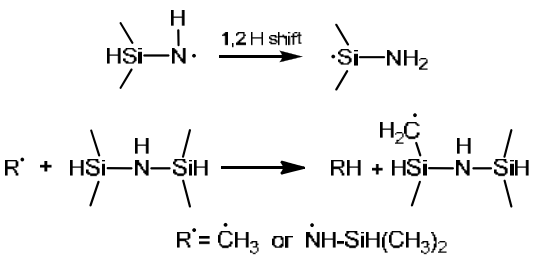
Summarizing the experiments, two main photolytically induced pathways are confirmed. i.e. the cleavage of the Si–N and of the Si–CH_3 bonds.



Si–CH_3 scission with subsequent formation of methane, which was found to be the favourable reaction pathway in the gas phase [6], takes place as well, as approved by the observation of $CH_3^{\bullet}$ radicals by low-temperature EPR, by the observation of small amounts of CH_4 (gas-phase IR), and by the decay of Si–CH_3 vibrations (ATR-FTIR) [4]. However, Si–NH bond scission dominates the overall process in the condensed phase, which can be rationalized by the favour-

able change of electron distribution along the Si–NH–Si bonds (weakening) upon excitation. The cleavage of the excited silazane was expected to be fast, as the excited triplet state was calculated to be dissociative. Indeed, only short-lived fluorescence spectra were observed. On the basis of the current results it cannot completely be excluded that the cleavage of the silazanes may also occur from the excited singlet state. Nevertheless, the origin of the primary radical pairs has no impact on the subsequent reactions discussed.

Aminyl radicals $^{\bullet}$NH–R could not be trapped in the matrix EPR experiments and there was no indication of stable products bearing the amino group in the GC-MS experiments. However, aminyl radicals were observed by laser flash photolysis and they disappear rapidly, most probably by H-transfer reactions leading to terminal amino groups R-NH_2. In case of tetramethyldisilazane (TMDSz) the decay of the aminyl radicals is faster, which can be explained by a facile intramolecular 1,2 H-shift (ΔG = -12 kcal mol^{-1}) leading to $^{\bullet}Si(CH_3)_2$–NH_2, and also by H-abstraction from another molecule ($\Delta G \sim$ -13 kcal mol^{-1}) with the formation of the $^{\bullet}Si(CH_3)_2$–NH–$SiH(CH_3)_2$ radical and diethylsilylamine. In case of hexamethyldisilazane (HMDSz) H-abstraction will occur from one of the six methyl groups (at a lower rate than from SiH in case of TMDSz), and indeed $^{\bullet}CH_2$–$Si(CH_3)_2$–NH–R radicals are observed as the dominating species in the EPR spectra of irradiated HMDSz.



Under continuous irradiation conditions, intermediate products with terminal NH_2 groups undergo further photolysis, and the cleavage of the NH_2–Si bond most likely occurs with the subsequent formation of NH_3 by H-abstraction. Ammonia was found to be a major volatile decomposition product in the VUV irradiation of PHPS [3] and polyorganosilazanes [4]. The present results suggest that the rate of photolytical decomposition of oligosilazanes should depend on the amount of labile (reactive) H available in the matrix (i.e. H bound to Si). This conclusion at least qualitatively explains the experimental finding that PHPS is much faster converted into a Si–O–Si network than the corresponding (P(DMS*co*MS)) into a methyl-Si–O–Si network.

The main product identified by GC-MS after photolysis of TMDSz is a "dimer" of the silazane, most probably formed by recombination of two Si–N–Si$^{\bullet}$ fragments. This dimer is also formed in the presence of oxygen, and this clearly shows that such radical recombination is fast enough to compete with the reaction of O_2 with the Si–N–Si$^{\bullet}$ radical. In the presence of O_2, siloxazane compounds are the main irradiation products, formed after several reaction steps from the secondary silylperoxyl species. The identification of oligomers with subsequently increasing number of –$Si(CH_3)_2$-O– units strongly points to a reaction mechanism for the conversion based on building of linear R–$(Si–O)_n$–chains, which in turn may later crosslink by further cleavage of Si-CH_3 bonds along the backbone.

The present investigations show that the reactivity of organosilazanes does not depend only on the quantum yield of radical pair formation, but also on structural parameters such as the availability of reactive hydrogens.

Conclusion

In summary, the VUV-triggered process of converting PHPS into SiO_x in the presence of oxygen has been elucidated. The investigations open up the possibility for the continuous production of barrier layers on polymeric substrates at ambient temperature and pressure. Further investigation will focus on the improvement of the barrier properties against water vapour and on the development of multi-layers suitable for the application as a transparent top layer for encapsulation of thin-film photovoltaic modules.

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Ion beam assisted epitaxy of nitride thin films

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Introduction

Conventional ion beam assisted deposition (IBAD) of binary nitrides is usually realized by evaporation of the metal component and simultaneous nitrogen ion irradiation of the growing film with ion energies in the range of several 100 eV to several keV (see schematic in Fig. 1). In the literature, many consequences of this ion irradiation during film growth are reported like film densification, enlargement of the crystallite size, change of the preferred crystallite orientation, biaxial texturing of the films, and others.

For the ion beam assisted molecular beam epitaxy (IBA-MBE) of wurtzitic (hexagonal) GaN films using irradiation with nitrogen ions possessing hyperthermal energies of several tens of electron volts, a further beneficial effect on the film growth and the resulting film properties was found [1]. There, the hyperthermal nitrogen ion irradiation leads to a ballistic enhancement of the adatom mobility by producing ion-induced point defects only at the film surface but not in the bulk below the surface. Due to this enhanced surface mobility, adatoms can reach energetically favourable lattice sites and can overcome energy barriers, e.g. the Ehrlich-Schwoebel barrier, leading to epitaxial films of high crystalline quality.

In the present report, the actual state of the basic research on IBA-MBE at the IOM is briefly reviewed. In the end, prospective directions of future investigation in this field are sketched.

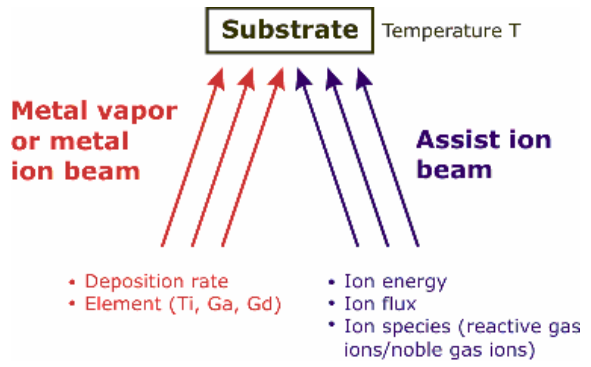

Figure 1: Schematic of ion beam assisted thin film deposition and relevant process parameters.

Experimental

The IBA-MBE set-up at the IOM consists of an ultra-high vacuum (UHV) four-chamber system: deposition chamber, analysis chamber, sample load-lock chamber, and scanning probe microscopy (SPM) chamber. Two effusion cells and a metal plasma source allow the evaporation of gallium and gadolinium, and the formation of a highly ionized titanium plasma beam, respectively. Hyperthermal nitrogen ions with a kinetic energy of less than 25 eV are produced by a hollow-anode constricted glow-discharge plasma beam source, developed at the Lawrence Berkeley National Laboratory, USA. The typically used single crystal substrates are heated to temperatures of up to 750 °C. Film growth can be monitored *in situ* using a quartz crystal thickness monitor as well as a 30 kV reflection high-energy electron diffraction (RHEED) system. Additional analysis methods situated in the analysis chamber are Auger electron spectrometry (AES) and low-energy electron diffraction (LEED).

Epitaxial GaN films with non-polar orientations

Compared to the huge progress in conventional group-III nitride technology, that led to, e.g., blue light emitting diodes and blue laser diodes, a relatively new research direction in this field is the investigation of group-III nitride films grown with non-polar orientations. The commercially used c-plane oriented wurtzitic group-III nitride films suffer from spontaneous and piezoelectric polarization fields that are inherent to the c-axis orientation of the wurtzite structure. In contrast, m-plane oriented films, where c-axis and a-axis lie in the growth plane, do not exhibit such fields along the growth direction, thus allowing higher achievable quantum yields and emitting polarized light.

Attempts to grow m-plane oriented GaN films on γ-$LiAlO_2$(100) substrates by IBA-MBE were successful and the results indicated that in order to avoid the formation of c-plane oriented GaN and to obtain m-plane oriented GaN of high crystal-

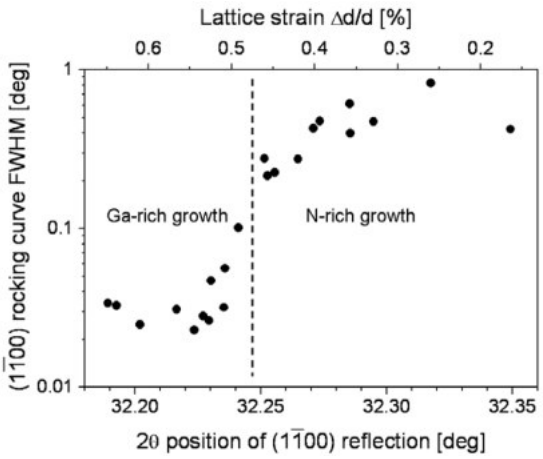

Figure 2: Correlation of crystalline quality (width of XRD rocking curves) and lattice strain along the growth direction for IBA-MBE of m-plane oriented hexagonal GaN on γ-$LiAlO_2$(100).

line quality, a specific growth regime – Ga-rich growth conditions – is necessary [2]. The higher N-ion-to-Ga-atom ratio, which is used for high quality c-plane oriented GaN films on Al_2O_3(0001) and 6H-SiC(0001) substrates [1], resulted for the films deposited on γ-$LiAlO_2$(100) in a mixture of m-plane and c-plane oriented crystallites, and consequently led to a low crystalline quality due to competitive growth, a rough surface topography as well as a weak and broad luminescence band. In contrast, the films deposited with Ga-rich conditions, i.e. a higher Ga atom than N ion flux, were purely m-plane oriented, of high crystalline quality, of small surface roughness, and exhibited an intense, sharp near-band gap luminescence line.

Despite the obvious advantages of Ga-rich growth conditions, several drawbacks appeared, namely a high density of basal plane stacking faults as well as huge, anisotropic mechanical stress in the films. Figure 2 shows the X-ray rocking curve width of the m-plane reflection of GaN, being a measure of the crystalline quality, as a function of the reflection shift in the 2θ diffraction diagram (lower abscissa) and recalculated as a function of the lattice strain along the growth direction (upper abscissa). A significant correlation can be observed, indicating that a high crystalline quality of m-plane oriented GaN films is interconnected with high mechanical stresses in the films. Origin of the stresses is not only the high thermal mismatch of film and substrate, resulting in compressive stress build-up during cooling down the sample to room temperature after deposition, but also compressive stress build-up already during the growth process. A combination of *in situ* RHEED-monitoring and *ex situ* X-ray diffraction analysis by recording reciprocal space maps is necessary to further investigate this correlation.

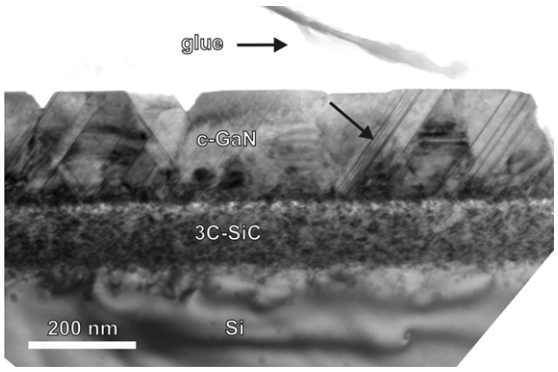

Figure 3: Cross-sectional bright-field TEM micrograph of a cubic GaN(100) IBA-MBE film on an ion beam synthesized 3C-SiC(100)/Si(100) substrate.

Another way of obtaining non-polar GaN is to deposit the zinc-blende (cubic) instead of the wurtzitic GaN polytype. For this purpose, 3C-SiC/Si substrates, prepared by ion beam synthesis at the Universität Augsburg, were used for GaN epitaxy by IBA-MBE, leading to the very first cubic IBA-MBE GaN films [3]. As a result, the necessity of Ga-rich growth conditions was shown also in this case, i.e. the meta-stable cubic polytype could only be stabilized in this way. For N-rich growth conditions, hexagonal GaN crystallites formed in addition to the cubic ones. These results proved the usability of such unconventional substrates for cubic GaN thin film epitaxy. However, for obtaining high quality films, both the crystalline and the surface quality of the 3C-SiC film as well as the GaN growth parameters would require further optimization. This can be derived from the cross-section TEM micrograph of such a film in Figure 3. The defect density within the SiC film is relatively high when compared to the Si substrate, but not all defects extend into the GaN film which exhibits mostly planar defects on {111} planes (black arrow).

TiN film epitaxy with hyperthermal Ti ions

It can be expected that, when in ion beam assisted epitaxy the metal component of the nitride compound is delivered with hyperthermal energies instead of or in addition to the energetic nitrogen ions, this will further enhance the thin-film epitaxy by ballistically enhancing the adatom mobility even more effectively. To prove this, the growth of TiN films by deposition of titanium ions, possessing hyperthermal energies of 70 ± 12 eV, in a nitrogen ambient was investigated [4]. The hyperthermal titanium ions

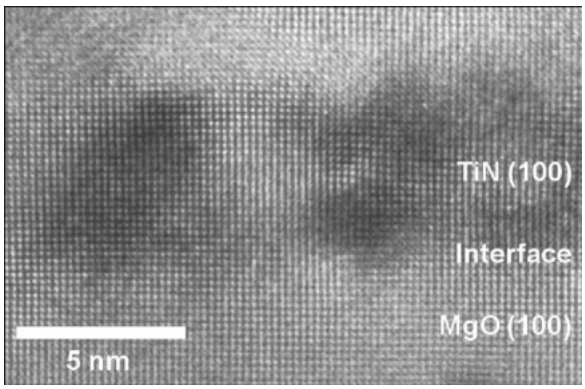

Figure 4: High-resolution cross-section TEM micrograph of an epitaxial TiN film deposited on MgO(100) by hyperthermal epitaxy at a substrate temperature of 700 °C.

were produced by a pulsed dc cathodic vacuum arc, delivering a highly ionized Ti plasma beam. The TiN films with thicknesses of up to 50 nm were deposited at substrate temperatures ranging from 700 °C down to room temperature on MgO(100) substrates, resulting in a small lattice mismatch of 0.7%. The crystalline surface structure of the films was monitored *in situ* by RHEED. High resolution TEM was used to examine the morphology and defect structure of the films. The results showed that all the ultra-thin TiN films deposited this way were epitaxial, exhibiting the well known 'cube-on-cube' epitaxy (see Fig. 4). The most interesting result was that in this way epitaxial growth could be achieved even at room temperature (see Figs. 5 and 6), while reported values for the critical temperature of epitaxial growth of TiN are in the range of 400 to 550 °C. This demonstrates the beneficial effect of the hyperthermal metal component involved in the deposition process on the film growth and the resulting film properties.

The crystalline quality of these TiN films could still be improved by optimizing the deposition parameters, in particular the nitrogen background pressure and the energy of the titanium ions.

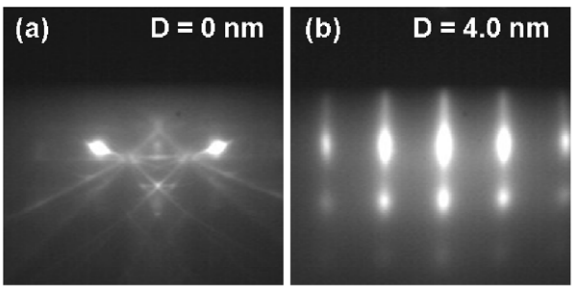

Figure 5: RHEED patterns obtained (a) before and (b) after hyperthermal TiN film epitaxy on MgO(100) at room temperature.

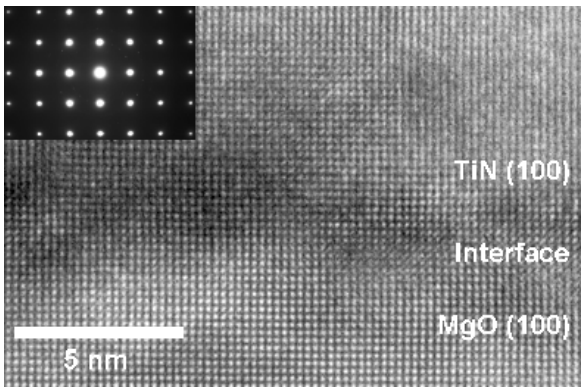

Figure 6: High-resolution cross-section TEM micrograph of an epitaxial TiN film deposited on MgO(100) by hyperthermal epitaxy at room temperature. The insert shows the corresponding selected-area electron diffraction pattern of the TiN film.

Kinetic model for IBA-MBE

Based on earlier experimental results of IBA-MBE of c-plane oriented GaN deposited on 6H-SiC(0001) [1], a film-growth model based on the kinetic theory of nucleation and film growth was developed in cooperation with a theory group in St. Petersburg, Russia [5]. The model compares the case of conventional MBE, where all involved particles are of low kinetic energy (< 1 eV), with the case of IBA-MBE, where nitrogen ions with hyperthermal energy are included. The crucial difference in the model

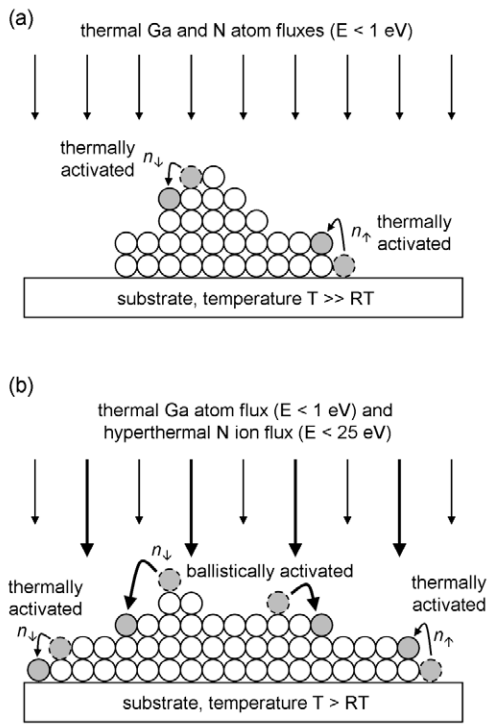

Figure 7: Schematic representation of the fundamental differences between (a) conventional MBE and (b) IBA-MBE growth for the example of GaN (not shown: adsorption, desorption and diffusion).

between MBE and IBA-MBE is schematically shown in Figure 7. The main assumption is that the energetic ions in IBA-MBE lead to a reduction of GaN cluster heights by allowing adatoms to overcome the Ehrlich-Schwoebel energy barrier at cluster edges.

This process was implemented in the set of kinetic equations by adding a term for ion-impact induced, i.e. ballistically activated adatom jumps from cluster tops downwards. Numerically solving the set of equations resulted in a cluster height *H* distribution function *f (H, t)* depending on the deposition time *t*. The abort criterion for the calculations was when the condition of fully completed coalescence of the GaN clusters was reached, i.e. when the substrate was completely covered with a compact GaN film. The resulting height distribution functions for the case of conventional MBE in Figure 8(a) show that coalescence is reached after a deposition time of nearly 700 s with a peak in height distribution function at cluster heights of about 40 nm. This corresponds to three-dimensional growth. Contrary for the IBA-MBE case depicted in Figure 8(b), coalescence is reached already after about 30 s of deposition time with a cluster height of only a few monolayers of GaN, corresponding to two-dimensional growth. Thus, the consequence of inserting ion beam irradiation induced surface processes into the model was a change in growth mode from three-dimensional to two-dimensional growth which is a prerequisite for epitaxial growth of high-quality films.

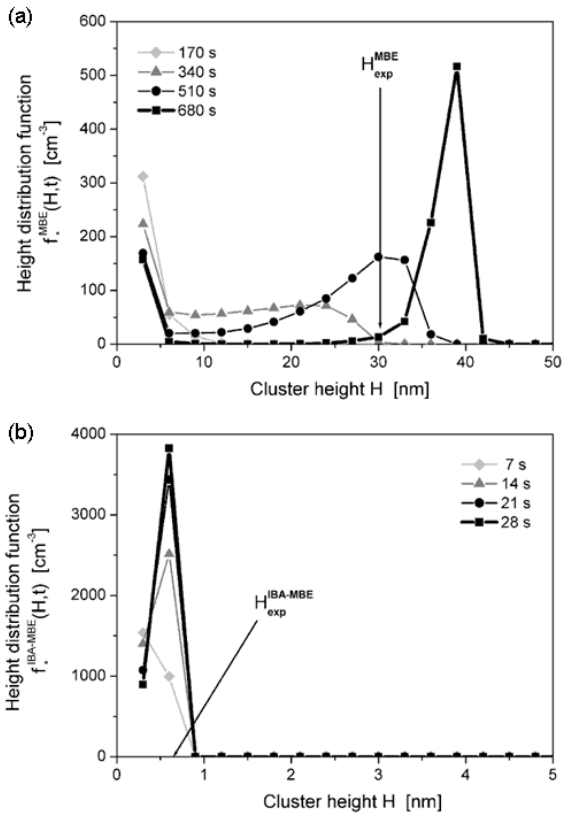

Figure 8: Calculated GaN cluster height distribution functions for different deposition times up to completed film coalescence (thick curves): (a) Conventional MBE, (b) IBA-MBE.

Outlook

As many of the obtained results on thin-film epitaxy generally depend on the first few nanometers of deposited film material, the very first stages of film growth like nucleation, island formation, coalescence of islands, etc. are of particular interest. Therefore, the IBA-MBE system was recently extended by an UHV-SPM system, comprising scanning tunnelling microscopy (STM) and atomic force microscopy (AFM) options, in order to investigate the crucial first stages of ion beam assisted epitaxial film growth. The surface topography can now be recorded before, during and after the deposition process by transferring the samples *in vacuo* to the SPM chamber. It is expected that the results to be obtained in future with the SPM will give much deeper insights into the influence of ion beam irradiation during thin-film epitaxy, allowing for refinement of existing growth models.

Acknowledgement

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Plasma jet machining (PJM) of off-axis collimating mirrors made from silicon carbide for space applications

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Introduction

Plasma jet machining (PJM) is a non-conventional machining method for the figuring and the correction of optical elements, which has been developed in IOM before more than one decade. PJM is based on a chemically reactive microwave or RF excited plasma jet discharge that interacts with a substrate surface to remove material. It has been shown [1,2] that reactive plasma jet machining technology working at atmospheric pressure or in rough vacuum is a very efficient tool for the treatment of optical surfaces made of silicon, silicon based materials like fused silica and silicon carbide, or ULE®. Different plasma jet sources have been developed to accomplish deterministic surface shaping and surface figure error correction over a wide spatial range with nanometer depth accuracy. The half-width of the removal functions reaches from about 0.1 mm to about 10 mm. Maximum volume removal rates of about 50 mm³/min have been achieved for fused silica and ULE™.

Surface machining is performed using the dwell-time algorithm on CNC controlled multi-axes systems. Extensively developed mathematical de-convolution routines are used for creating the machining files. Since the material is removed by plasma-assisted chemical etching during plasma jet treatment without any mechanical or physical contribution no sub-surface damage occurs in contrast to abrasive methods. This advantage makes the plasma jet technology very attractive for the precise manufacturing of especially aspherical and free-form optics. Plasma jets are predestined not only for pre-shaping with high machining depth and for deterministic shape correction with high spatial resolution, but also for efficient removal of surface contaminants like hydrocarbons or SiO_x. However, the chemical removal mechanism leads to an increase of surface roughness depending on the treated material and the removal depth. At the same time potential sub-surface damage is removed. In order to achieve both minimal form error and surface roughness a processing chain is necessary that includes at least one mechanical polishing step.

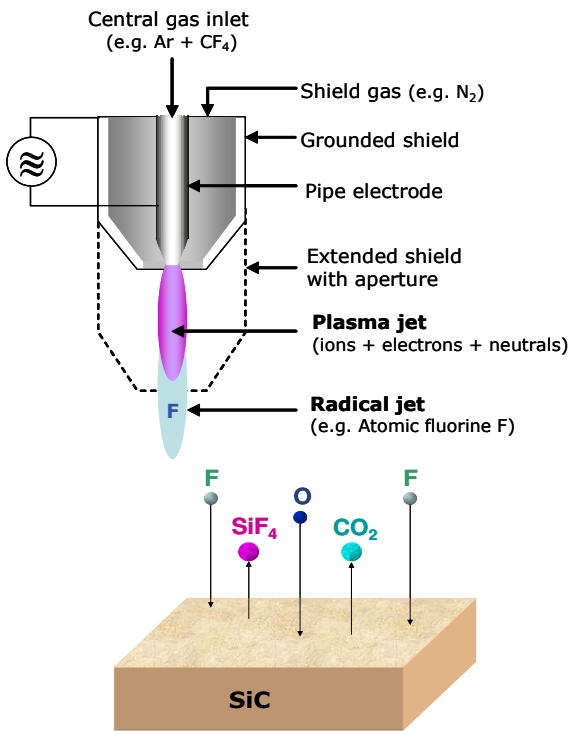

Figure 1: Principle of the plasma jet generation and the removal process by chemical reactions of radicals with the surface atoms.

In this report we summarize the results of basic investigations on SiC plasma jet machining for process optimization and stabilization as well as the results of the fabrication of three mirrors.

Basics of plasma jet etching of SiC

The principle of the plasma jet generation is schematically shown in Figure 1. A high flux of reactive species or radicals is flowing out of the plasma jet source. Material removal is obtained by chemical reactions between the radicals and the surface-atoms, whereas volatile products are formed, which can be exhausted. A small size RF (13.56 MHz) driven plasma jet source was used for the investigations of plasma-surface interactions. A mixture of CF_4 and peripherically injected O_2 has been applied as process gas.

For etching experiments it has to be taken into account that silicon carbide is a bilayer material. Its crystal structure exhibits two differently oriented faces – the C-face (000-1) and the Si-face (0001). Due to their distinct orientation the faces show slightly different properties in the etching behaviour. In the case of sintered silicon carbide which is mostly used for optical or me-

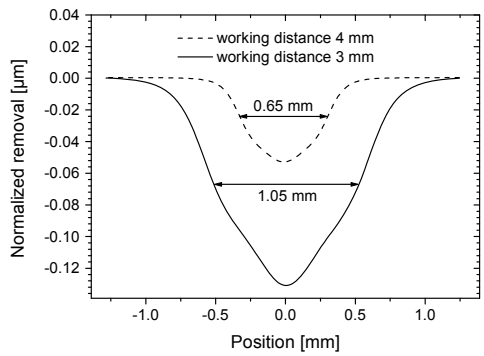

Figure 2: Typical tool function profiles for two different working distances.

chanical workpieces in technological applications, the material consists of non-oriented grains. Thus, the bilayer-related properties play an important role in surface reaction and roughness development. Furthermore, ions generated in the RF excited plasma jet discharge can interact with the surface. Sakiyama *et al.* [3] calculated the energy of energetic ions impinging the surface in a similar set-up to be approximately 10 eV. Thus, the interactions of energetic ions with the surface-enhancing desorption of volatile reaction products have to be considered. In order to isolate and clarify the different effects, all basic investigations have been carried out on mono-crystalline 4H-SiC wafers.

The plasma jet exhibits a more or less bell-shaped tool function, as shown in Figure 2. Depending on the specifications for the correction process, the volume removal rates and the half-width of the tool function can be varied by changing the work distance between nozzle and surface in the ranges of 0.001–0.01 mm³/min and 0.5–1.0 mm, respectively. It has been found that the SiC etching rate and the roughening effect is strongly influenced by the CF_4/O_2 ratio and the surface temperature. On the one hand, it is necessary to get a balanced removal of Si and C to minimize the roughening effect as well as to maximize the etching rate. Therefore, a certain mixture of F- and O-radicals is needed in the plasma near the surface to balance the reaction

$$SiC\ (s) + 4F + 2O \rightarrow SiF_4\ (g) + CO_2\ (g) \qquad (1)$$

On the other hand, deposition of CO_xF_y and SiO_x layers from non-volatile reaction products may occur, which competes with the removal process. The deposition rates depend on the concentration of the involved species but, additionally, there is a relation with the surface temperature. A significant dip of the etching rate occurs for surface temperatures around 150 °C. The Arrhenius-type plot shown in Figure 3 indicates that deposition effects are dominant for low temperatures, whereas beyond 150 °C etching becomes more efficient.

XPS measurements have been carried out to characterize deposition effects after etching with a CF_4/O_2 gas mixture and additional sample heating. It has been found that the amount of SiC and the total amount of oxygen at the surface change with surface temperature. The oxygen concentration shows a clear maximum at

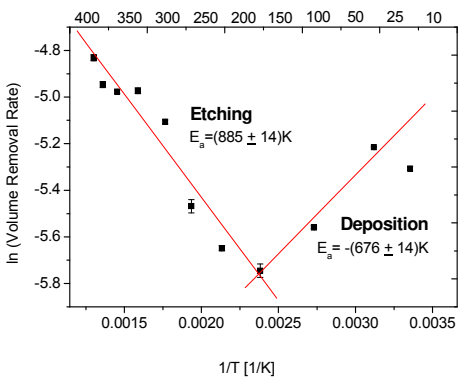

Figure 3: Logarithm of volume removal rate in mm³/min vs. 1/T, where T is the surface temperature during plasma jet etching.

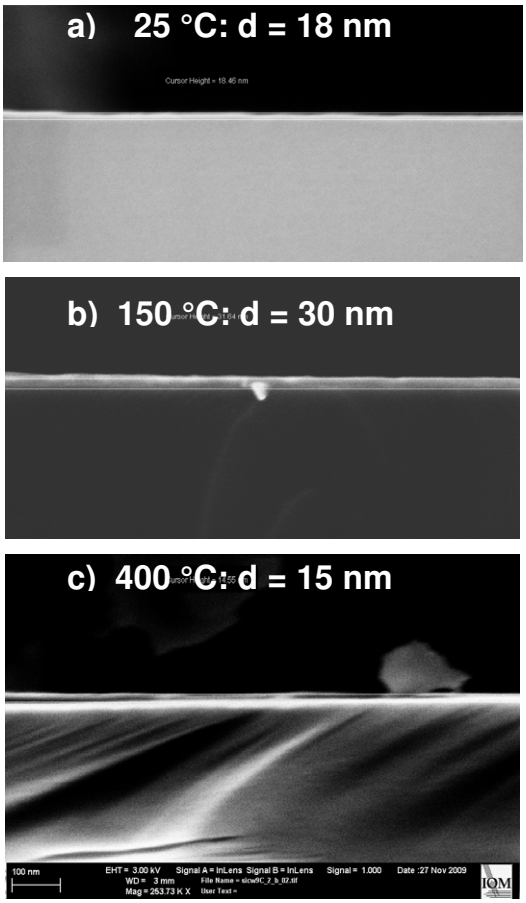

Figure 4: SEM cross sectional images of 4H-SiC wafer C-face with SiO_2 top layer after etching at different surface temperatures.

150 °C with a minimum for the SiC component. Furthermore, the ratio of the portion of silicon sub-oxides to the total amount of detected oxygen suggests the formation of SiO_2, assuming that all oxygen is bound to silicon. This indicates that the formation of silicon oxides is responsible for the minimum in the volume removal rate at 150 °C sample temperature. SEM images taken from a cross-sectional view of a plasma-jet treated C-face displayed in Figure 4 support this proposal. The highest thickness for the top layer is observed for 150 °C. The results obtained from XPS and SEM analysis lead to the conclusion that the minimum in the etching rate is caused by enhanced layer formation dominating over fluorine etching of the surface.

Fabrication of the collimator mirrors for Gaia

The IOM and TNO Science and Industry (NL) established a process chain consisting of 3D robot polishing and Plasma jet machining for finishing strongly curved off-axis parabolic silicon carbide (SiC) mirrors. The mirrors are a crucial part of the cryogenic fibre collimators for the Basic Angle Monitoring Opto-Mechanical Assembly (BAM OMA) of the Gaia satellite mission planned to be launched to space in 2012. The fibre collimator mirrors are off-axis parabolic mirrors which need a surface shape accuracy of less than 12.5 nm RMS and a surface micro-roughness of at least 6 nm RMS. The main difficulty in the fabrication of those mirrors is their small radius of curvature (R = 50.17 mm) over an effective aperture of 10 mm.

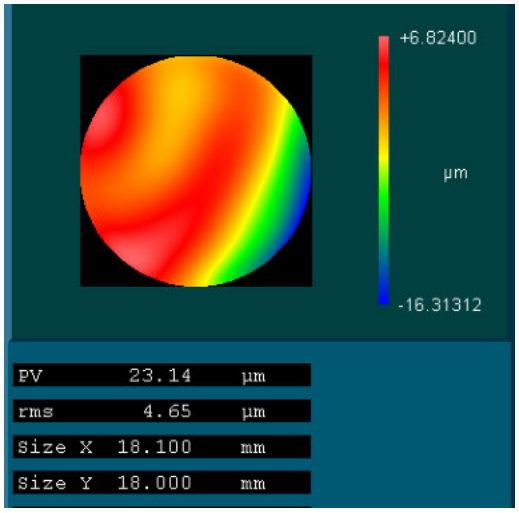

Figure 5: Mirror #2 initial state after grinding and pre-polishing (Zernike representation of the surface error measured by a tactile probe).

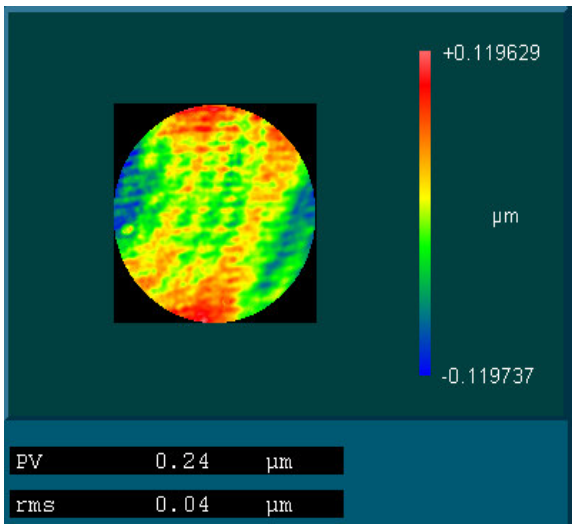

Figure 6: Status of mirror #2 after correction of the optical axis positions by corrective PJM and cleanup polishing

The three mirror pre-forms fixed in a special set-up were delivered with mechanically ground surface. Afterwards, a pre-polishing step was carried out by TNO separately on each mirror yielding surface shape errors of 15–25 µm PV and 3–5 µm RMS, respectively, over nearly the full aperture of 18 mm as shown in Figure 5. The corresponding measurements were carried out on an SPDT machine at TNO. Furthermore, displacements of the optical axes have been found, equivalent to a small tilt of the mirrors yielding additional errors in the order of some microns. Regardless of these large shape deviations, it was decided to start with PJM at this point since prior tests had shown a sufficiently high removal rate and a higher convergence of the PJM process compared to corrective polishing.

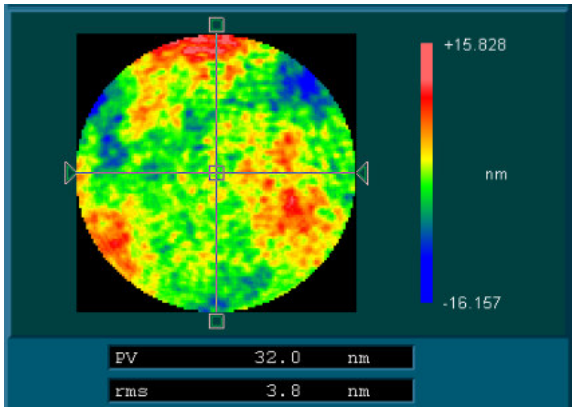

Figure 7: Final status of mirror #2 on the clear aperture of ∅ 10 mm. The shape RMS value is far below the specification of 12.5 nm RMS. Surface roughness has been determined by microinterferometer (20× magnification) to be < 6 nm RMS, which is also within the specificiation.

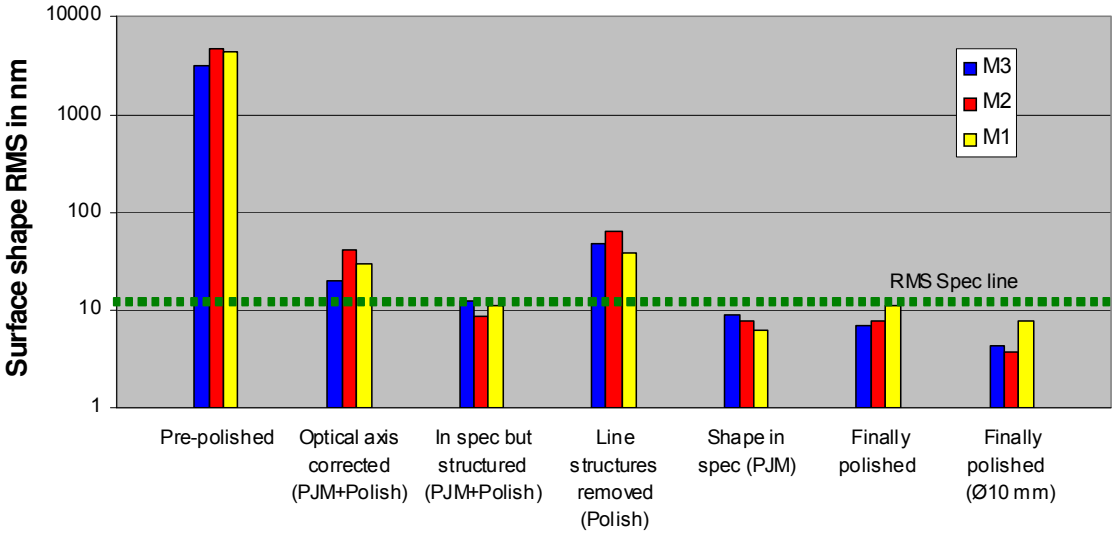

Figure 8: Surface shape RMS (log scale) on three SiC off-axis parabola collimating mirrors for the plasma jet machining/polishing processing steps.

To avoid the transfer of small structures originating from the tactile measurement into the surface if the millimetre PJM tool is utilized, it was necessary to use the Zernike expansion of the error data instead of raw data for correction. The high amount of material that had to be removed required long machining times of 2–4 h per PJM cycle. Hence, the workpiece was significantly heated by the plasma jet during the PJM process and, according to the results explained in the previous section, this led to a significant decrease of the etching rate. Thus, efficient sample cooling was necessary to maintain a stable and deterministic material removal.

After two PJM correction and clean-up polishing cycles the surface errors have been reduced by more than 90% to the sub-µm PV level. The optical axes of all mirrors have been shifted to their specified positions and the roughness has been reduced. Hence, the mirrors could be measured by interferometer. As an example, the corresponding surface error map of mirror #2 is given in Figure 6. In this measurement a horizontal periodic line structure occurs, which originates from the clean-up polishing step. The lowering of such error structures with PJM requires the application of the sub-millimetre tool. At the same time an excellent sample alignment is demanded. A further PJM step has reduced the error to near the specified RMS value. Unfortunately, strong periodic line structures resulting from an improper relation between feed and rotary speed of the polishing head during the clean-up polishing process occurred. Thus, a subsequent high-removal polishing step with modified parameters was necessary to remove these structures.

While having a sufficient surface roughness and low periodic mid-spatial structures, however, the form error of all three mirrors was increased to 40–60 nm RMS. The next PJM correction step with subsequent soft clean-up polishing reduced the form error by 82% on all three mirrors. This result demonstrates the excellent stability and reproducibility of the process. A final PJM/polishing cycle decreased the form error PV and RMS values of all mirrors well below the specification. Surface roughness met the specifications as well. The final surface error of mirror #2 on the clear aperture of Ø 10 mm is displayed in Figure 7. The diagram in Figure 8 summarizes the individual processing steps and gives an idea about the process convergence in terms of surface form RMS.

Acknowledgment

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Examples of problems solved with new analytical instrumentation

D. Hirsch, C. Elsner, U. Decker, J.W. Gerlach

Introduction

The institute obtained several state-of-the-art analytical instruments during the last two years, in addition to already sophisticated equipment. In this short report, the new instruments are presented, together with selective analytical problems which arose during the research and technology work at the Leibniz-Institut für Oberflächenmodifizierung. Beside the new systems for X-ray Photoelectron Spectroscopy (XPS) and Matrix Assisted Laser Desorption/Ionization Time-Of-Flight Mass Spectrometry (MALDI-TOF-MS/MS), the established Nuclear Magnetic Resonance Spectroscopy (NMR) and Secondary Ion Mass Spectrometry (SIMS) are discussed in detail.

KRATOS Axis Ultra XPS

X-ray photoelectron spectroscopy is a method to determine the composition of a surface-near volume in a qualitative and quantitative manner. The measurements done with photoelectron spectrometers yield a distribution of electrons over their kinetic energies which depends on the design and precision of the instruments manufacturers. They give specifications that the instrument can reach several pairs of count rates at a defined peak resolution from a signal of a noble metal under a defined X-ray flux. The transmission characteristics of the analyser system is not specified.

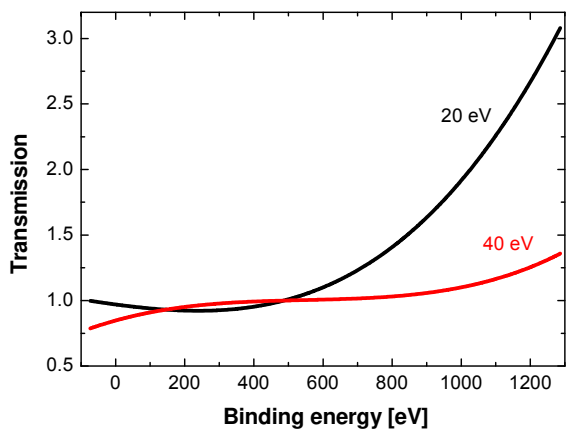

Figure 1: Transmission function comparison for 20 eV and 40 eV electron pass energy (full analysis area). The binding energy is in respect to the Al Kα line.

Whereas the precision of the energy scale can be easily checked by measuring the binding energies of the intense peaks of noble metals, the intensity behaviour over the energy range, the so-called instrument transmission function, should be investigated in detail. Seah summarized the known data and he emphasized that large errors can occur in XPS and AES due to inaccurate sensitivity factors [1]. Thus, estimated transmission functions should be used. Hesse *et al*. tested two algorithms to estimate the transmission functions of the instruments [2]. The first is a numerical fit of measured survey spectra of Au, Ag, and Cu taken at various analyser settings with published reference spectra. The second way is to measure the spectra of 15 atomic level signals of sputter-cleaned Au, Ag, Cu, and Ge samples for all analyser settings of interest using Al Kα and Mg Kα radiation.

Both measurement campaigns were performed with the recently installed Axis Ultra DLD XPS system. The fit of the survey spectra taken at higher electron pass energies using the UNIFIT program [3] failed. For all pass energies 80 eV and higher and magnetic lens on high fit errors occurred. Tests showed that the DL detector is not longer proportional to count rates larger than 250 kcps. Hence, the ratio between signal and background is not correct. The results for different analyser settings calculated from the results of the 15 level measurements (lower pass energies) have low error values. The transmission functions for the low electron pass energies (10 and 20 eV) show a strong dependence on the electron energy. Contrary, at the higher pass energy 40 eV (for full and reduced analysis area) the transmission functions depend only weak from the electron energy (see Fig. 1).

After data evaluation as proposed in the literature [1-2], the error in the XPS concentration estimation using general elemental sensitivity factors from other authors often exceeds 20% from the nominal values whereas the procedure using transmission functions is better than 8%. For the compound NaCl as an example one would use the signals Na 1s and Cl 2p. The transmission function ratios at 1072 (Na) and 200 eV (Cl) binding energy differ by 45%. Using correct general sensitivity factors which were estimated at one of these pass energies would

give a ratio of 50:50 for the corresponding pass energy, but 41:59 for the other one. This large error is not tolerable. After knowing the spectrometer transmission parameters we can improve the accuracy of our analysis.

MALDI-TOF-MS/MS with the new AUTOFLEX III mass spectrometer

Matrix-assisted laser desorption/ionization (MALDI) is a soft ionization technique which enables the sensitive ablation and detection of large, non-volatile and labile molecules by mass spectrometry (MS) [3]. Therefore, it has become a standard technique in the analysis of biomolecules, mostly in protein chemistry, proteomics and genomics [4,5]. Currently, the application of MALDI-MS is extended to the analysis of polymers, and also small molecules, by the use of special desorption/ionisation techniques for the latter one (surface-assisted desorption/ionization (SALDI)).

The most common type of analyzers for MALDI-generated ions is the time-of-flight mass spectrometer (TOF-MS) which can analyze, in principle, an unlimited mass range by measuring the flight time of ions in a field-free drift region. However, the broad kinetic energy distribution of initial MALDI ions causes limitations to mass resolution. One approach partly to compensate the initial velocity and the energy distribution is the use of an ion reflector (ion-mirror), which re-focuses energy distributed ions. Thus, the corrected ions hit the detector at the same time after passing a second field-free drift region. As a consequence, the mass resolution is significantly improved (for the AUTOFLEX III in our lab it is about 5-10 ppm). Due to the fast and sensitive process, MALDI-TOF-MS is suited for high throughput and imaging applications and off-line coupling techniques with micro/nano-liquid chromatography (LC). Both features are available in house, e.g. an LC-MS BRUKER PROTEINEER FC spotter is coupled to a DIONEX ULTIMATE 3000 micro/nano-LC system.

MALDI-MS is a powerful technique for polymer characterization and can produce rich chemical information for polymer structural analysis, e.g. the determination of polymer end groups and repetition units. In addition, it is very sensitive, allowing the detection and identification of minor polymer components, impurities and byproducts of the polymerisation as well as the characterization of copolymers and blends. Moreover, it can provide information about the molecular mass and molecular mass distribution of polymers with narrow polydispersity. Currently, the new MS technique is applied to the characterization of chemical modifications in proteins and polymers, the characterization of monolithic materials concerning her specificity for binding and repelling proteins, the characterization of polymeric blends, and for proteomic studies.

NMR Spectroscopy

New monomers, polymers, modified particles and compounds prepared in the IOM were comprehensively characterized by the analytical methods of the institute [6]. Especially the

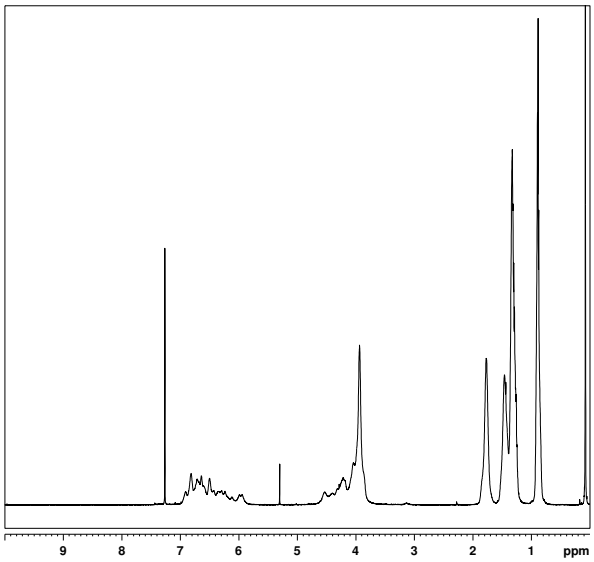

Figure 2: ^{1}H-NMR of newly synthesized polynorbornene at 393 K.

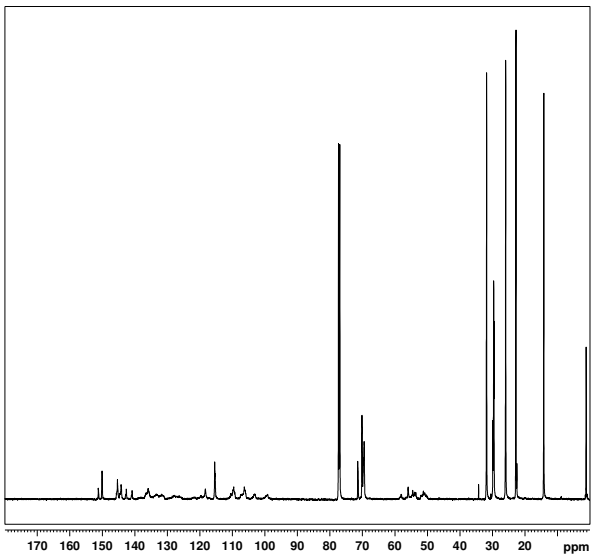

Figure 3: ^{13}C-NMR of newly synthesized polynorbornene at 393 K (overnight experiment).

600 MHz-NMR spectroscopy used for different nuclei (^{1}H-, ^{11}B-, ^{13}C-, ^{15}N-, ^{17}O-, ^{19}F-, ^{29}Si-, ^{31}P-, ^{51}V-, ^{117}Sn-) gives valuable information about structure and purity. Of interest is additionally the possibility of ^{19}F-NMR.

The higher field (14 T) in the new system in comparison to the previously used (250 MHz/ 6 T) gives better homogeneity of the magnetic field also by automatic shimming and so better resolution and sensitivity (also by automatic tuning and matching). Thus, smaller concentrations are detectable. The new equipment and software results in a significant time reduction in time consuming experiments (^{15}N-, ^{17}O-, ^{29}Si-). Furthermore, triple experiments such as magic angle spinning and temperature variations (223–423 K) are possible (see Figs. 4-5).

For structure elucidation and purity also such methods as IR spectroscopy, Gas Chromatography Mass Spectroscopy, High-Performance Liquid Chromatography were used. The reaction kinetics were followed by Real-Time IR (see Fig. 6), RT-NIR, and coupled measurements of RT-NIR viscosity [7] also by GC-MS and HPLC.

Gel Permeation Chromatography in different solvents gives information about the molecular masses of polymers and their distributions. In tetrahydrofurane (room temperature) and trichlorobenzene (at high temperatures – e.g. at 418 K) absolute mass detection of polymers is possible by triple detection.

HPLC characterizes the extractable residues (especially photoinitiators were determined), while IR and Raman spectroscopy allow analysis of the residual reactive groups of the products.

Several mechanical tests can be performed, including micro-hardness, scratch-resistance, and pendulum hardness. Furthermore, thermal characteristation is possible by Differential-Scanning Calorimetry, Dynamic-Mechanical Analysis, Thermo-Gravimetric Analysis and coupled DSC-TGA-IR.

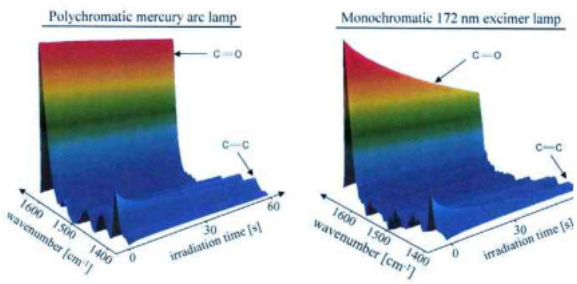

Figure 4: Comparison of the RT-IR kinetics of different light sources.

Traces of impurities can be analyzed by Inductively Coupled Plasma Optical Emission Spectroscopy. In addition, surface-sensitive tools as SEM and AFM are available.

SIMS

Epitaxial GdN films, deposited on MgO(100) and YSZ(100) substrates by ion beam assisted molecular beam epitaxy (IBA-MBE), were subject of investigations concerning their chemical composition. Besides the N/Gd concentration ratio, the contamination of the films with oxygen was a major question, as (i) oxygen has a high affinity to the rare-earth metal Gd and (ii) GdN films are hygroscopic and rapidly decompose in air by releasing nitrogen in favour of oxygen [8]. According to literature, oxygen contamination lowers the Curie temperature of the ferromagnetic GdN to values below 70 K and has a large impact on the electronic properties of the films [9]. To prevent oxidation in air, the GdN films were thus coated with protective Gd or GaN films.

While the existence of N and O in the films could be verified using time-of-flight secondary ion mass spectrometry (TOF-SIMS) by recording the ^{155}GdN and ^{160}GdO signals (see Fig. 7), a quantitative result was not obtainable. Thus, quantitative ion beam analysis methods were used to calibrate the TOF-SIMS measurements, namely Rutherford backscattering spectrometry (RBS) and elastic recoil detection analysis (ERDA).

With RBS, due to the quadratic dependence of the scattering cross section on the atomic number of the element to detect, the sensitivity for N is nearly 84 times lower than that for Gd,

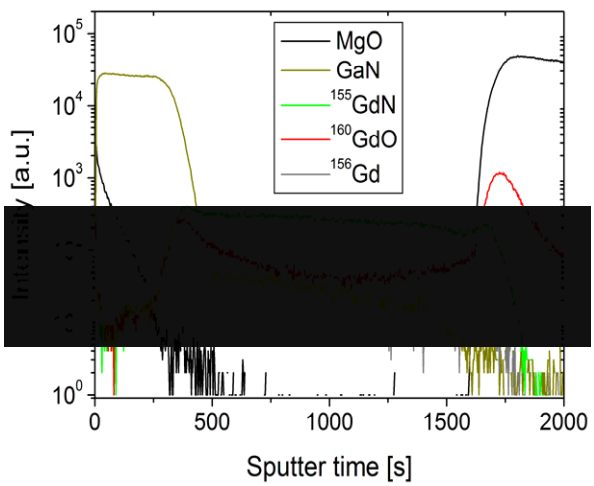

Figure 5: TOF-SIMS depth profile of an epitaxial GdN film on MgO(100) with a GaN protective coating.

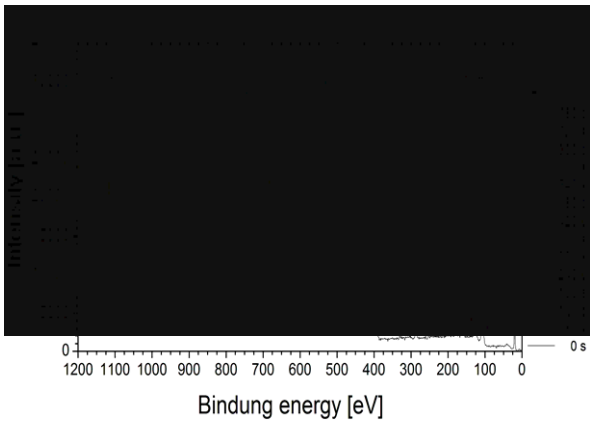

Figure 6: Depth-resolved XPS spectra of a GdN film deposited on a YSZ(100) substrate with a GaN protective coating (right: sputter time).

while the sensitivity for O is 64 times lower than that for Gd. Thus, the large imbalance of signal heights in RBS does not allow a correct quantification of the O and N concentrations in the GdN films. The only value derivable from these measurements was the total amount of Gd atoms in the film per area.

ERDA, which is in particular sensitive for the lighter elements, delivered reliable N/O ratio values, but due to overlaps of the signals related to Gd, Ga, Zr, Y and forward scattered Au projectiles the N/Gd ratio could not be obtained. From comparison with quantitative values of the N/O concentration ratio obtained by ERDA measurements, it was derived that in the TOF-SIMS measurements the sensitivity for the ^{160}GdO signal was about ten times higher than for the ^{155}GdN signal.

Attempts to gain quantified chemical composition values of GdN films by using spectroscopy of X-ray excited photo electrons (XPS) revealed another issue: during Ar ion sputtering for depth profiling a large fraction of N atoms was released from the surface of the GdN films, seriously diminishing the N 1s signal height, i.e. falsifying the XPS analysis (as shown in Figure 8). Nonetheless, XPS investigations revealed information on the oxygen chemistry in partly or fully oxidized GdN films, resulting in cubic Gd_2O_3 films.

Acknowledgement

Prof. Dr. T. Butz and Dr. J. Vogt, Universität Leipzig, are thanked for enabling RBS measurements, PD Dr. W. Assmann, LMU München, for providing ERDA beamtime, and Dipl.-Phys. J. Mennig, former IOM Leipzig, for film preparation.

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Comparative DFT study on the role of conformers in the ruthenium alkylidene-catalyzed ROMP of norborn-2-ene

S. Naumov, M. R. Buchmeiser

Comparative quantum chemical calculations on the reaction pathways for the formation of ruthena(IV)cyclobutanes from both 1st- and 2nd-generation Grubbs catalysts of the general formula $RuX_2(L)(L')(=CH_2)$ (L=PCy_3 or 1,3-dimesityl-4,5-dihydroimidazolin-2-ylidene, L'=PCy_3) and norborn-2-ene (NBE) were carried out on the B3LYP/LACVP** level in dependence on the ligand X=I, Br, Cl and F [1]. As depicted in Figure 1, the structures of the most stable complexes $RuX_2(PCy_3)(H_2IMes)(=CH_2)$ and $Ru(PCy_3)_2X_2(=CH_2)$ show different rotational conformations of the Ru-carbene unit ($Ru=CH_2$) relative to the C(1)-Ru-P plane.

The mechanism proposed by Straub [2] for the formation of (one) active and (three) inactive NBE-Ru-carbene π-complexes for non-cyclic alkenes was applied to the cyclic alkene NBE. In $RuX_2(PCy_3)_2(=CH_2)$, the inactive NBE-Ru-carbene complex is energetically more stable than the active one, however, in $RuX_2(IMesH_2)(PCy_3)(=CH_2)$, the active NBE-Ru-carbene complex is more stable than the inactive one. In consequence, the possible rate limiting barrier for the conversion of the NBE-Ru-carbene complex into the corresponding metallocyclobuten (MCB) is systematically larger in the case of 1st-generation Grubbs catalysts than in the case of 2nd-generation Grubbs catalysts due to an additional rearrangement for the formation of an active π-complex from the more stable (inactive) conformer (Fig. 2).

This correlates with the observed reactivity of both types of initiators. There is a strong influence of the ligands L and X on the conformational properties and relative stabilities of the

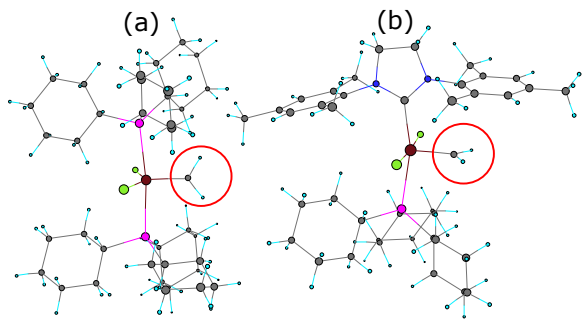

Figure 1: The most stable structures of (a) $RuX_2(PCy_3)_2(=CH_2)$ (b) $RuX_2(PCy_3)(H_2IMes)(=CH_2)$.

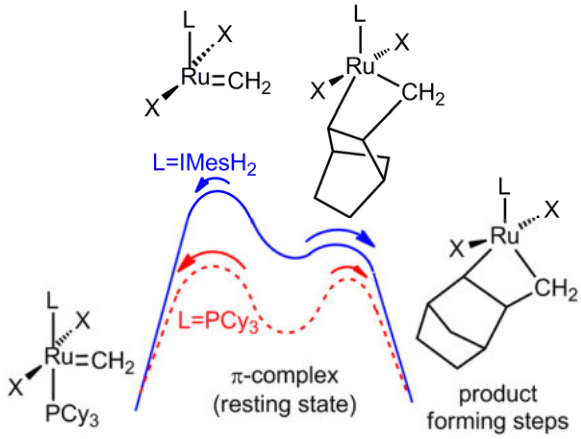

Figure 2: Most favorable reaction pathways of MCB formation in 1st- generation (dashed line) and 2nd-generation Grubbs catalysts.

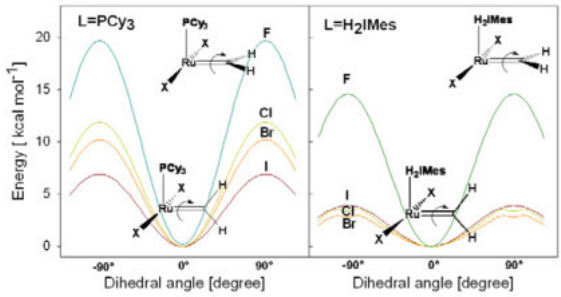

Figure 3: Potential energy for the rotation of the carbene unit in dependence on the ligands L and X.

14-electron intermediates (Fig. 3), which has a direct effect on the distribution of the inactive and active conformations of the corresponding Ru-carbene-NBE complexes.

Important findings are that (i) the active complex is energetically more stable than the inactive in the case of 2nd-generation catalysts, (ii) the possible rate limiting barriers for the conversion of the π-complex into the corresponding MCB are systematically larger in the case of 1st-generation catalysts due to a necessary additional step for the formation of the active complex from the more stable inactive one.

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Surface modification of nano-sized zeolites

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Due to the inevitable shortage of crude oil in the foreseeable future, the production of liquid fuel from various sources like coal, biomass or natural gas via the Fischer-Tropsch (FT) Process has lived a renewed commercial interest. FT waxes, i.e. long-chain n-alkanes, as one of the FT by-products can be profitably converted into lubricating oil by hydroisomerization on bifunctional zeolite catalysts.

To reduce the effects of diffusion on the liquid-phase hydroisomerization reaction, nano-sized zeolite crystallites can be applied facilitating the accessibility of molecules to the internal zeolite surface and improving their catalytic performance. The aim of the studies reported here is to increase the isomerization selectivity of nano-sized Pt/HBEA zeolite catalysts by surface modification.

For nano-sized zeolites, their large external surface may provoke non-selective reactions reducing the intrinsic zeolitic selectivity. In general, surface modifications should substantially suppress the undesired reactions occurring on the external crystallite surface. For the hydroisomerization of long-chain alkanes on Pt/HBEA catalysts, the effects of chemical liquid deposition of organosilicon compounds (tetraethoxysilane (TEOS) and aminopropyltriethoxysilane (AMEO)) on the zeolite properties have been studied and compared to the surface modification by pre-coking. Both techniques have already been used for the modification of nano-sized zeolites HZSM-5 and HFER catalysts [1]. Nitrogen sorption measurements revealed that silanization treatment leads to a reduction of pore size whereas the pre-coking treatment hardly affected the pore size distribution (Fig. 1). These findings can be explained by the formation of small $Si(OH)_x(OC_2H_5)_{4-x}$ species due to hydrolysis reactions in the case of TEOS silanization, which can penetrate into the zeolite channels.

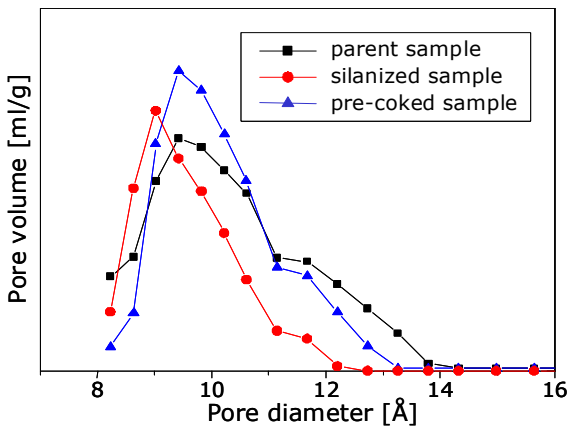

Figure 1: Pore size distribution before and after surface modification of HBEA by applying the NLDFT method.

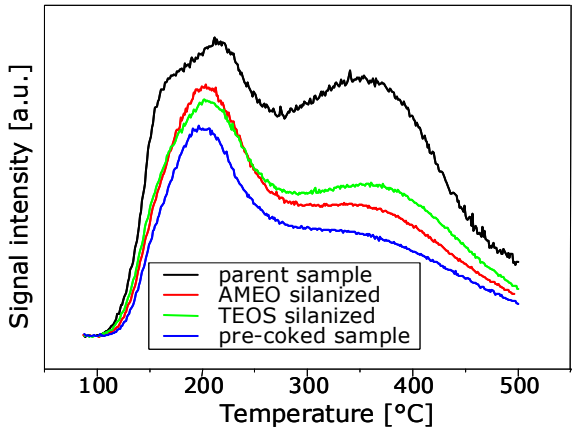

Figure 2: Temperature-programmed desorption of ammonia on fresh, silanized, and pre-coked samples of HBEA.

The acidity of the modified zeolite samples was determined by temperature-programmed desorption of ammonia (Fig. 2). The NH_3-desorption peaks revealed no temperature shift, i.e. surface modification resulted only in a decrease in the number of acid sites.

In addition to ongoing isotopic studies on hydroisomerization over modified Pt/HBEA zeolite, deuterium labeling experiments have been used for elucidating the mechanism of the dehydroalkylation of aromatics with alkanes on Pt/HZSM-5 [2]. Rapid H/D exchange leading to the formation of deuterated ethane proves that adsorption and desorption of reactants are significantly faster than dehydroalkylation and aromatic transformations, i.e. both reactions under study are not diffusion-controlled [3].

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Simultaneous monitoring of process parameters by near-infrared spectroscopy assisted by multivariate data analysis

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Near-infrared (NIR) spectroscopy is widely used for process control. In particular, it has already been used for in-line monitoring of various polymerization processes including UV photopolymerization reactions. In the past, analysis of the spectral data was mainly based on band integration or simple multivariate evaluation methods on the basis of partial least squares (PLS1) regression [1,2]. In case of monitoring the conversion, such methods only lead to correct results if the thickness of the layer permanently remains constant. However, it is well-known in roll coating technology that the thickness of coatings may vary with increasing line speed, which leads to incorrect predictions. Figure 1 shows the effect of the web speed on the thickness of an acrylate coating as well as on the prediction of the conversion from in-line data. For comparison, conversion data, which were determined off-line by a reference method (FTIR spectroscopy), are shown. With increasing line speed, the coating thickness more and more increases, which leads to an increasing divergence between the predicted and the actual conversion.

This problem can be overcome by use of two probe heads, which record separate spectra before and after UV irradiation. The conversion can be determined from the ratio of the integrals of the acrylate band. This way, the influence of thickness changes is compensated [3].
Alternatively, a more sophisticated solution using multivariate data analysis was proposed [4], which requires one probe head only. It is based on the application of the PLS2 algorithm, which is able to predict more than one parameter from the same set of input data. Figure 2 shows the use of a PLS2 calibration model for the prediction of coating thickness and conversion during a roll coating trial.

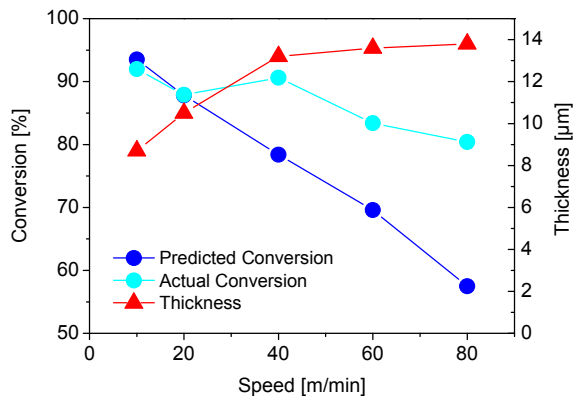

Figure 1: Effect of the line speed on the thickness of a coating and its impact on the in-line determination of the acrylate conversion. Actual conversion data (from FTIR spectroscopy) are given for comparison.

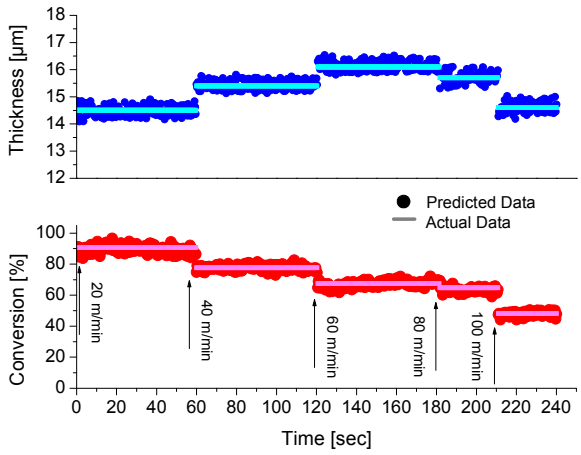

Figure 2: In-line monitoring of conversion and coating thickness of an acrylate coating at various line speeds. Moreover, reference values are shown.

Both parameters can be determined simultaneously. It is obvious that the coating thickness increases or decreases, when the line speed is raised. Nevertheless, the conversion after UV curing can be predicted with high precision (i.e. ± 2-3%), which is confirmed by reference data obtained off-line by FTIR spectroscopy. The error in the measurement of the thickness was found to be about 0.5-1 µm.

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ZnO-based nanocomposites for the UV protection of wood for outdoor applications

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It is well-established that bright wood species turn brown and brittle during the time when exposed to natural weathering. UV light, e.g. as part of the sun light, causes a photochemical decomposition of lignin, one of the main components of wood [1]. Oxygen and moisture can further accelerate this degradation process so that an ideal protective coating for the UV protection of wood must possess sufficient filter efficiency for UV light, a high barrier for water vapour and oxygen permeation, and high transparency. Coatings with commonly used UV-protective additives are, however, often only semi- or even non-transparent. Zinc oxide (ZnO) is a low-cost and easily synthesised material, which shows an excellent UV and IR adsorption as well as a high transparency in the visible range, making it a promising candidate for the UV protection of bright wood species [2].

In the framework of the AiF research project *'Weatherproof and wear resistant coatings for wood and WPC based on semi-/transparent acrylate-silica-ZnO nanocomposites with a low solvent content'* (09/07-02/09), commercially available as well as self-synthesised ZnO species were investigated with regard to their physicochemical properties in the coatings.

According to the directive ift-R5 set as standard by the Institut für Fenstertechnik (ift) Rosenheim, the UV transmittance and the Vis transparency of coatings can be quantitatively calculated (Fig. 1). Low values for the UV transmittance as well as high values for the Vis transparency of the coatings are desirable. Compared

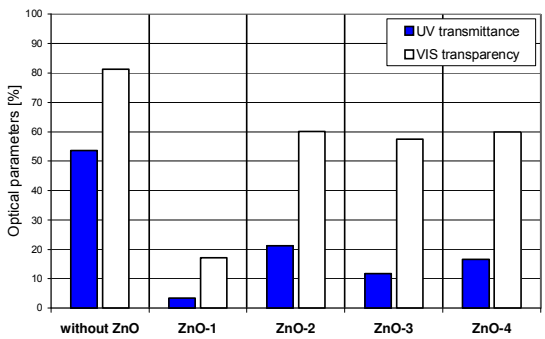

Figure 1: Calculated values of the UV transmittance and the VIS transparency of the investigated coatings.

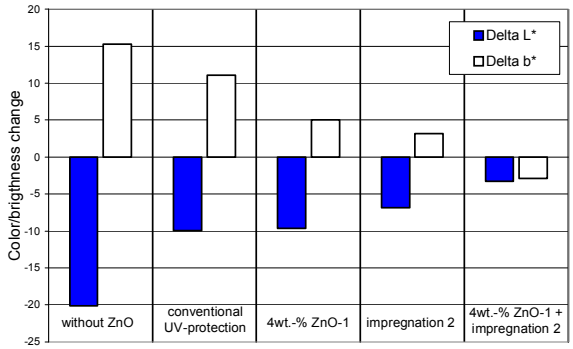

Figure 2: Changes of brightness (Delta L) and color (Delta b*) of coated spruce wood samples after 1500 hours of artificial weathering.*

to a coating without ZnO, this was fulfilled for most of the investigated samples. The coatings were mostly fully transparent and showed a high filter efficiency for UV light. In order to study the effect of ZnO on the hydrophobicity of the coatings, coated polycarbonate plates have been exposed to artificial weathering (XENON test) for at least 1000 hours. Though a decrease of the contact angle was observed for all samples after weathering, higher contact angles were found for the ZnO-based coatings compared to a coating without ZnO. Therefore, the water repellency (hydrophobicity) of the coatings could be improved when ZnO was used. In addition, the UV resistance of coated wood samples was investigated after exposure to artificial weathering (Fig. 2). Wood samples for outdoor applications could efficiently be protected from discoloration using nano-sized ZnO-based coatings. The investigated coatings showed a higher stabilization efficiency compared to conventional UV-stabilizing formulations. The best results were achieved if the wood was additionally pretreated with a water-based wood impregnation containing a special lignin-protector [3].

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Electron beam-based functionalization of polymer membranes

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Membrane separation systems have gained increasing importance for many different applications, e.g., in hemodialysis, water treatment, sterilizing filtration, or proton conduction in fuel cells. Today, micro- and ultrafiltration membranes are predominantly fabricated from synthetic membrane materials such as polyethersulfone (PES), polysulfone (PSf), polyvinylidene fluoride (PVDF), or polyacrylonitrile (PAN). The diverse applications necessitate the modification of the polymer in order to avoid any fouling of the hydrophobic membrane surfaces or to achieve a functionalized membrane for specific applications. Especially for the improvement of the antifouling properties of micro- and ultrafiltration membranes, commonly used hydrophilization methods are characterized by serious disadvantages as they risk the contamination of the eluent by non-permanently immobilized compounds or by the initiators/catalysts used. Furthermore, they require a substantial number of process steps (e.g. activation of the scaffold polymer for subsequent grafting and additional purification steps), and finally, the use of harmful or even toxic compounds.

A new method for the straightforward and permanent functionalization of polymer membranes using small molecules bearing diverse hydrophilic functionalities (Fig. 1) was developed [1]. The approach combines surface activation of the matrix polymer and the simultaneous immobilization of small molecules by use of low-energy electron beam in an aqueous system. The procedure guarantees for the permanent immobilization of hydrophilic small molecules on the membrane polymer. It neither requires any preceding surface functionalization nor the use of catalysts/photoinitiators or other toxic reagents. In addition, it avoids the synthesis of hydrophilic monomers/polymers, thus avoiding additional synthetic and purification steps as well as the use of organic solvents.

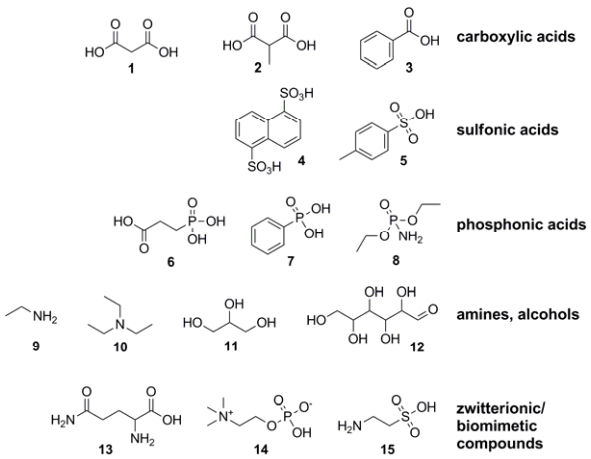

Figure 1: Functional molecules used for the membrane modification.

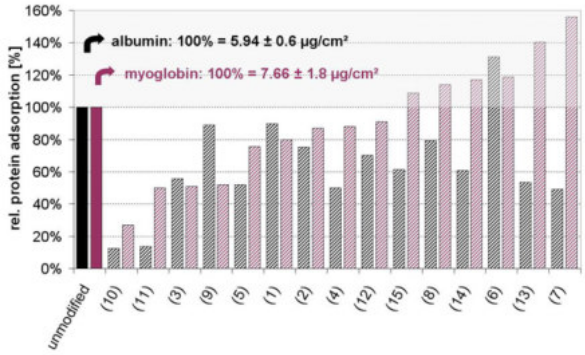

*Figure 2: Protein adsorption (albumin and myoglobin) after PES membrane modification with **1**-**15**.*

Since the use of electron beam activation allows for operating independently from specific functionalities present at the matrix polymer's surface, the method can be adopted to a vast variety of base membrane polymers, e.g., PES, PSf, PAN, and PVDF. Furthermore, in contrast to UV irradiation, electron beam is capable of interpenetrating polymeric membranes. This way, also the inner surface of the membrane is activated for the desired modification reactions.

For this purpose, the membranes were soaked with aqueous solutions of different low-molecular weight molecules bearing diverse hydrophilic functionalities and subject to electron beam treatment.

With selected functional molecules, this simple modification procedure resulted in a significantly increased hydrophilicity accompanied by a decreased protein adsorption at the membrane surface as demonstrated for different proteins (Fig. 2). Apparently, a permanent functionalization of the membrane polymer is achieved.

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Cyclopolymerization: new monomers, catalysts and insights

C. Schmidt, M. R. Buchmeiser

The cyclopolymerization of 1,6-heptadiyns is part of the metathesis-based polymerizations like ring opening metathesis polymerization (ROMP) or acyclic diene metathesis (ADMET). It is an alternative for established techniques that are normally used for the preparation of poly(acetylene)s [1]. The resulting polymers possess better properties like solubility due the substitution in 4-position of the 1,6-heptadiyne, especially hetero-atoms and related groups. The conjugated double bonds result from the polymerization mechanism (Figure 1) that forms ring units during the polymerization.

In the area of mechanistic investigations concerning the polymerization of monomers with hetero-atoms in the 4-position, e.g. nitrogen and sulphur, significant results and insights could be obtained. Especially in the field of nitrogen-bearing 1,6-heptadiynes, the influence of coordinating electron pairs at the 4-position of the monomer could be investigated and confirmed [2], leading to a modified mechanism for this type of monomers. Because of that, the activity of the initiator was changed, leading to a different reaction mechanism for ruthenium-based initiators. As a result, polymer structures with six-membered ring units in the polymer chain were obtained [3]. This is a significant difference because normally ruthenium-based systems form only polymers based on five-membered ring units. In addition, the free electron pair leads to backbiting of the propagating polymer chain resulting in low polymer weights [2]. For sulphur-based monomers, the coordination effectively disabled the polymerization with Grubbs-type initiators [4].

In the area of initiator design, a new family of Grubbs-type initiators based on pseudo-halide derivatives of the Grubbs catalyst was synthesised, characterized and successfully used in different metathesis reactions (Fig. 2) [5]. They showed a high reactivity in cyclopolymerization, ROMP and cross metathesis (CM), but only low reactivity in ring closing metathesis (RCM). The

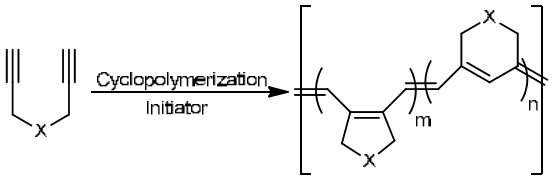

Figure 1: Scheme of cyclopolymerization.

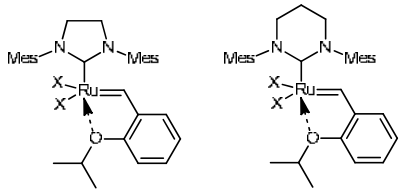

Figure 2: New Inititators (X = OCN or SCN).

reactivity depended on the selected metathesis reaction. For example, aniline derivatives were polymerized in quantitative yields using the isocyanate derivatives. The observed structures also differed significantly from polymers generated by other ruthenium-based initiator systems, because the resulting polymers had a regioregular structure selectively consisting of cyclo-1-pentene-vinylene units without the presence of any six-membered ring structures [4].

Table 1: Effect of oxidation.

Monomer	Yield [%]
Dipropargyle thioether	10
Dipropargylsulfoxide	70

In addition, monomers that form thiophene-like poly(acetylene)s were investigated. Herein, the role of the substituents in the 2-position and the effect of the oxidation state of the sulphur atom in the 1,6-heptadiyne were of special interest. It could be shown that the oxidation of a thioether into sulfoxide and sulfon analogues significantly increases the polymer yield (Table 1). The resulting polymers were insoluble until a substituent in 2-position was introduced. This was realized with a sulphur derivative of 3-(prop-2-yne-1-yloxy)oct-1-yne. The resulting monomer could be polymerized using Schrock-type initiators. The obtained polymers possessed no regioregular structure and consisted of a mixture of five- and six-membered ring units [4].

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Self-organized nanostructures by low-energy ion beam erosion

F. Frost, B. Ziberi, M. Cornejo, J. Völlner, D. Hirsch, B. Rauschenbach

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.

Within the DFG research unit 845 "Self-organized nanostructures by low-energy ion beam erosion", started in 2007, the IOM activities focus on the experimental exploration of fundamental processes, the combination of conventional lithographic techniques with patterning by self-organization, and the application of nanostructured optical and functional surfaces. Thus the ion beam induced pattern formation on Si and Ge model surfaces is considered, including the classification of erosion patterns as well as the detailed investigation of the transition between the different patterns and the underlying mechanisms. In a further sub-project guided self-organization processes are addressed by the combination of ion beam induced self-organization with conventional lithographic techniques, e.g. phase mask projection laser ablation or E-beam lithography. 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 is evaluated.

In the first funding period of the research group, a lot of substantial progress has been achieved for all IOM projects and in close collaboration with the other sub-projects in FOR 845 [2-6]. In the first sub-project it has been shown that ballistic drift processes are essential for the smoothing and stabilization of Si surfaces during erosion. Furthermore, evidence was given that secondary sputter events by highly energetic sputtered target atoms as well as backscattered projectile ions are important and might contribute to additional smoothing mechanism and an additional surface stabilization. Additionally, the important role of co-deposited metals, especially iron, for the pattern formation under near normal ion incidence conditions are highlighted which offers now a new degree of freedom for pattern control. With regard to the project of guided self-organization it can be summarized that the main parameters determining this pattern formation are the local incidence angle of ions, the orientation of the local surface with respect to the ion beam direction, and the local surface curvature. In combination with appropriate pre-pattern the approach facilitates the formation of a new type of nanostructures not possible on planar surfaces and a hierarchical nanostructuring across different length scales (Fig. 1). Finally, the last project related to pattern formation on quartz glass surfaces shows that gradient-dependent sputtering is an important process responsible for pattern formation at microscopic length scales and even below, which can be used, e.g., for the precise adjustment of ripple facet angles.

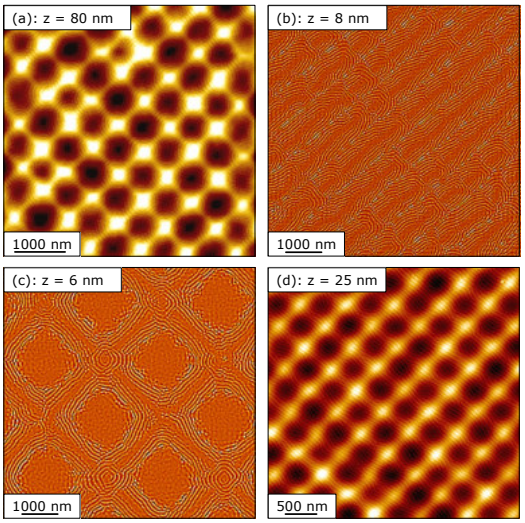

Figure 1: Novel surface pattern produced by combination of ion beam induced self-organization with conventional lithographic techniques. The larger scale structures originate from the lithographically defined pre-pattern.

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Glancing angle deposition and characterization of Si nanostructures

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Nanostructures with customized morphologies promise a high application potential, for instance as optical and photonic materials, magnetic storage devices, or sensors. Glancing angle deposition (GLAD) in combination with a computer-controlled substrate rotation is a sophisticated method to customize manifold nanostructure varieties. Here, the ion-beam-induced sputter deposition of self-organized Si nanostructures (columns, screws, spirals or zigzag-structures) was investigated. In particular, the nanostructure morphology and area density were examined with respect to the deposition parameters (rotation frequency, substrate temperature, deposition angle) [1,2].

In the course of deposition a structure broadening with a critical height was observed at which neighbouring nanostructures merge. At elevated substrate temperatures (300–360 °C) the nanostructure formation was found to be determined by adatom diffusion. Additionally, the deposition process was successfully simulated by the three-dimensional Monte Carlo (MC) method, yielding good agreement with the experiments [2].

The GLAD deposition of ordered Si nanostructures was achieved by substrate preparation via nanosphere lithography [2]. Honeycomb and hexagonal patterns of extremely uniform Si nanostructures of different shape were obtained.

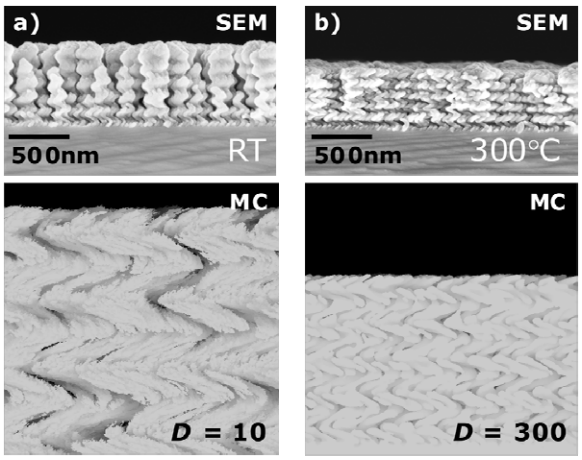

Figure 1: Si nanospirals deposited at a) room-temperature and b) 300 °C. The MC simulations show a good qualitative agreement with the SEM images.

A special electron-beam lithographical procedure allowed the defined positioning of Si nanocolumns [3]. In particular, for the deposition of Si on honeycomb templates at different deposition angles Θ it was shown that the structure of the growing film changes drastically from 1) a continuous film with honeycomb-like arranged hillocks on top at normal incidence (Θ = 0°), 2) a dense film with a mesh of hexagonally arranged pores at Θ = 70° to 3) separated rod-like structures with a triangular cross-section at Θ = 85°.

The mechanical properties of Si nanosprings were examined by force-distance spectroscopy [4]. The stiffness could be improved by a factor of 3.5 by post-deposition treatment with 1.2 MeV Ar^+ ions at liquid nitrogen temperature. The improved mechanical strength of the post-irradiated Si nanospring coatings indicates that these coatings are capable of withstanding higher loads without getting delaminated.

Additionally, the surface-enhanced fluorescence of nanostructured Si, Ag and Cu GLAD films was investigated [5]. Prospective applications are in device photonics, respective optical biosensors in general and in the field of water quality in particular. Inclined Ag nanoneedles on fused silica substrate exhibited the highest enhancement factor of about 14. The fluorescence experiments were applied for sensing of bacteria in water. Furthermore, surface plasmon resonance experiments showed a high sensitivity in air and in water.

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Interface-controlled nucleation and crystallisation

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Within this collaborative research project – financed by the EU within the 7th Framework Programme (NMP3-CT-2006-033200) – nucleation and crystal growth in non-isochemical glass systems was studied. The effect of precipitating crystals with a composition different from the surrounding matrix was thoroughly investigated with the aim to create novel photonic nanomaterials.

Besides the formation of application-relevant nanocrystals such as BaF_2, LaF_3, $NaLaF_4$, and CaF_2 [1-3], the perhaps most challenging task to deal with was the TEM study of lithium aluminosilicate glasses. The 3.5 Li_2O – 0.15 Na_2O – 0.2 K_2O – 1.15 MgO – 0.8 BaO – 1.5 ZnO – 20 Al_2O_3 – 67.2 SiO_2 – 2.6 TiO_2 – 1.7 ZrO_2 – 1.2 As_2O_3 model glass provided by Schott is similar to the low-thermal expansion glass Robax®. The conceptual idea behind this composition is to utilize nucleation agents (TiO_2 and ZrO_2) for a very homogeneous precipitation of high-quartz solid solution crystals possessing negative coefficients of thermal expansion.

TEM imaging or even analyses at lithia-containing glass-ceramics is handicapped by pronounced radiation damage. However, it turned out that electrons of 75 or 80 keV can be used to image and even micro-probe these samples. Aberration-corrected TEM is capable of resolving 0.1 nm spacings even at 80 keV. As an example, two $ZrTiO_4$ nanocrystals of just about 4-5 nm diameter are shown in Figure 1.

The investigation of the spatial distribution of majority elements in the lithium aluminosilicate glass proofed $ZrTiO_4$ nanocrystals to be surrounded by a shell significantly enriched in alumina (Fig. 2). This shell acts as a diffusion barrier for the growing crystals and inherently limits their growth, leading to a very homogeneous size distribution. Contrary to the assumption of epitaxial overgrowth, the evolving compositional gradient around the $ZrTiO_4$ nanocrystals is proposed to provide optimum preconditions for nucleation of the secondary high-quartz phase.

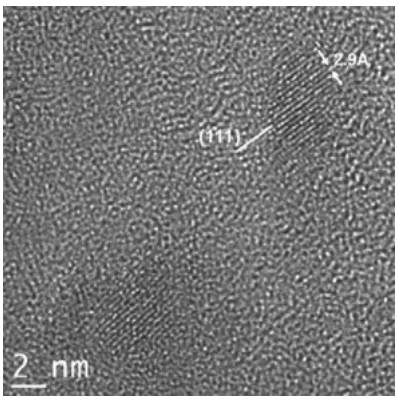

Figure 1: HRTEM micrograph of $ZrTiO_4$ nanocrystals (glass detailed above heat-treated at 750 °C for 1 h).

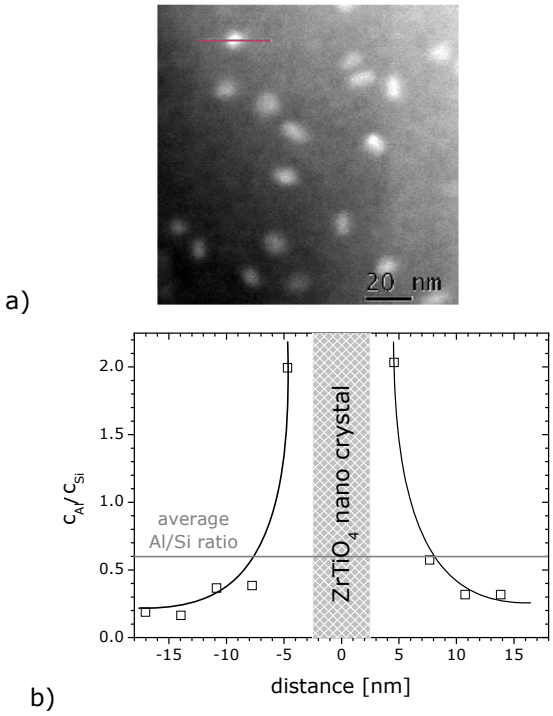

Figure 2: (a) Dark-field STEM image of the lithia aluminosilicate glass annealed for 1 h at 750 °C (position of line scan indicated), (b) Composition ratio of Al/Si along the line scan across the crystal.

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Killing of adherent microbes by an atmospheric plasma jet

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Plasma jets are the field of intensive research and development in the IOM since about 15 years. The main application field investigated is ultra precision surface machining by plasma assisted chemical etching (see T. Arnold *et al.*, p.26, this Biannual Report). After the development of a low mean power pulsed miniaturized atmospheric plasma jet source with low plasma temperatures down to human body temperature a joint BMBF-funded project with dentists from the Saarland University was started in 2007 to verify the applicability of the plasma jet in dentistry. In an *in vitro* study we tested a microwave-pulse-powered non-thermal atmospheric plasma jet for its antimicrobial efficacy against adherent oral microorganisms.

Agar plates and dentin slices were inoculated with 6 log10 colony forming units (CFUs)/cm² of the caries associated bacteria *Lactobacillus casei* and *Streptococcus mutans* as well as *Candida albicans*, which is frequently isolated from dentin caries lesions including *Escherichia coli* as a control. Areas of 1 cm² on the agar plates or complete dentin slices were irradiated with a helium plasma jet for 30, 60 or 90 s, respectively. The agar plates were incubated at 37 °C, dentin slices were vortexed in liquid media, and suspensions were placed on agar plates. The killing efficacy of the plasma jet was assessed by CFU-counting on the irradiated areas of agar plates, as well as by determination of the number of recovered CFUs from dentin slices.

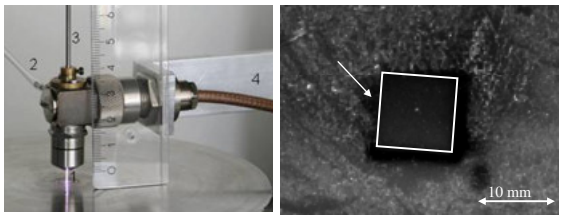

Figure 1: left: Experimental type plasma source with plasma jet (1), gas supply with outer (2) and inner gas inlet (3) and co-axial cable (4) for the 2,45 GHz pulsed microwave; right: 1 cm² plasma scan treated area for 90 s of Strep. mutans on an agar plate after 48 h incubation at 37 °C. The colony number in the treated area was 1 log10 CFUs. Also an expanded growth inhibition zone of 1 to 2 mm surrounding the irradiation raster scan area is also visible (arrow).

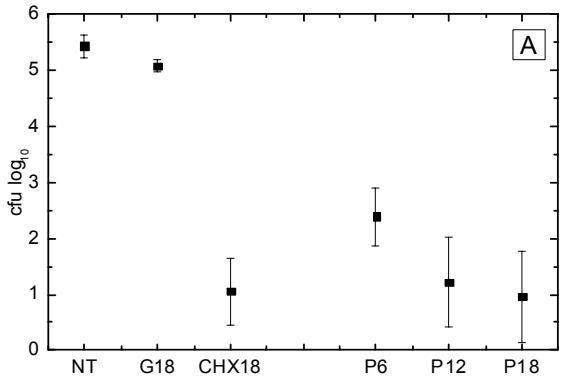

Figure 2: Colony forming units (CFUs) given in log10 of Escherichia coli *recovered after plasma jet raster scanning for 18, 12 and 6 s cumulative exposition times (P 18, P 12, P 6) using scan line speeds of 11, 16, and 3 mm/s, respectively. Three controls: (i) gas irradiation without plasma ignition for 18 s, with 1 mm/s scan line speed, (G 18), (ii) exposition to chlorhexidine for 18 s (CHX 18), and (iii) no treatment (NT). In the diagrams mean values with 95% confidence intervals (CI) are given, calculated of series of five for plasma jet treated samples and of three for controls.*

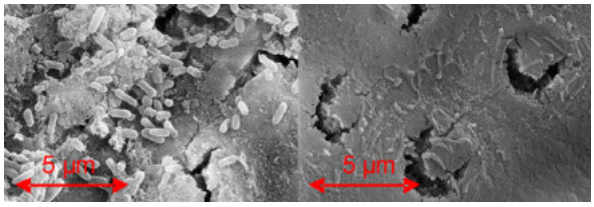

Figure 3: SEM micrographs of adherent Escherichia coli *on dentin slices after plasma jet scan treatment - 11 s/mm scan line speed, 18 s cumulated treatment time for 1 cm² (right) and controls without plasma treatment (left).*

A microbe-killing effect was found on irradiated parts of agar plates for *L. casei, S. mutans, C. albicans and E. coli.* The plasma jet treatment reduced the CFUs by 3–4 log10 intervals on dentin slices in comparison to recovery rates from untreated controls. The microbe-killing effect was correlated to increasing irradiation times.

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Nitriding of austenitic stainless steel by pulsed low-energy ion implantation

D. Manova, J. W. Gerlach, F. Scholze, S. Mändl, H. Neumann

Nitriding of stainless steel by energetic ions is a well-established laboratory technology, which is now ready to be employed by industry. The obtained layers exhibit very high hardness and wear resistance, while maintaining the excellent corrosion resistance of the base material. Among the different methods for nitriding of steel as gas and plasma nitriding, plasma immersion ion implantation, and ion beam implantation (PIII), broadbeam LEII is one of the most industry-friendly methods due to rather low voltages and a simple experimental set-up, together with a very high ion flux density across large areas, which can be delivered by modern ion sources [1].

However, for a detailed investigation of the dependencies on ion beam parameters, a large variation of time, ion energy, and ion current density is necessary. At the same time, the thermal energy balance of the substrate has to be taken into account where an additional external heating source with a fast control loop is required to reach and maintain the desired substrate surface temperature independent from – but in reaction to – the ion beam bombardment. In this short report the results from low energy ion beam nitriding of austenitic stainless steel using a novel electronic beam switch developed at IOM in Leipzig [2], allowing for decoupling of ion energy and average ion current density on a linear scale, are presented.

On the one hand, it could be shown that the nitriding depth is independent on the ion energy as long as the process temperature and temperature evolution is identical (see Fig. 1). As a

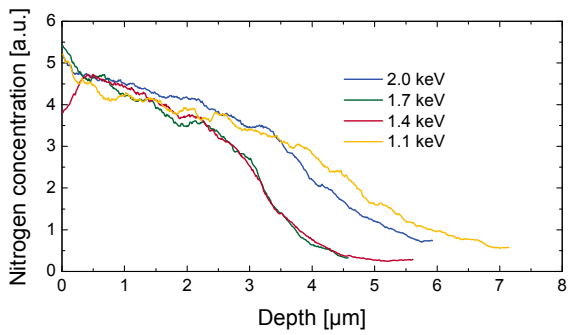

Figure 1: Nitrogen depth distribution as obtained from SIMS measurements for samples nitrided at four different energies at a duty cycle of 30%.

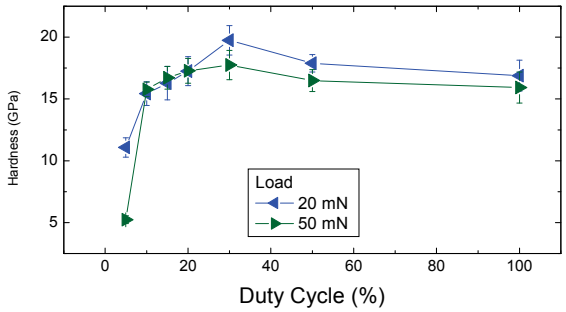

Figure 2: Nanohardnes of samples after low energy nitrogen ion implantation for different dutycycles at constant ion energy.

corollary, also no influence of the ion current density on the layer thickness was found, as long as a minimum ion current is available [3]. For the samples implanted at 1.1 and 2.0 keV a slightly higher temperature was measured, explaining the higher thickness.

When varying the duty cycle using the electronic beam switch, an optimum of the resulting nanohardness was found near duty cycles of 20–40% (see Fig. 2). For lower duty cycles, not enough nitrogen was incorporated to allow the formation of expanded austenite while the hardness remained close to the value of the base material of around 5 GPa. Interestingly, for higher duty cycles a reduction of the hardness was observed, which is in conjunction with a reduced absolute nitrogen content in the surface area for these layers (Sg. oder Pl.?). At the same time, a slight decrease of the layer thickness is caused by excessive sputtering at higher duty cycles.

Using pulsed low energy ion nitriding, an efficient industrial nitriding process is possible using lower ion energies, less current density and avoiding radiation safety issues associated with PIII.

Literature

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[2] J. Dienelt, K. Zimmer, F. Scholze, B. Dathe, H. Neumann, Plasma Sources Sci. Technol. 12 (2003) 489.
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Nucleation and phase formation of photocatalytically active TiO_2 films

A. Gjevori, J. W. Gerlach, D. Hirsch, D. Manova, S. Mändl

Photoactive thin films have a large potential for neutralizing air pollutants. Beside photocatalytic behaviour, superhydrophilicity can be induced by UV irradiation, leading to a reduction of the contact angle to values between 10° and 0° under illumination, thus allowing self-cleaning or anti-fogging applications. By increasing the average energy per incoming particle, it is possible to reduce the substrate temperature while keeping an identical morphology.

In the case of TiO_2, the phase composition can be additionally adjusted in this way. According to the literature [1], particle energies around 30 eV correspond to a substrate temperature of 800 °C with pure rutile films obtained in both cases. However, a direct correlation between the phase composition and the morphology does not exist: much higher ion energies are necessary to ascertain the formation of the rutile phase than to establish a columnar structure. Furthermore, radiation damage during the film deposition may lead to structural and electronic defects, which in turn degrade the photoactivity. Here, results on TiO_2 thin films formed by cathodic-arc metal plasma based ion implantation and deposition (MePBIID) with additional substrate heating are presented.

The films obtained by this procedure are slightly substoichiometric titania films with an O-Ti-ratio of 1.90–1.95, as determined by elastic recoil detection analysis. The film thickness was always close to 300 nm, indicating a growth rate of about 1 nm/s and no strong influence of the varying ion bombardment, especially the concomitant sputtering, on the growth rate. However, ion bombardment and substrate temperature strongly influence the phase formation [2]. No phase formation could be detected at RT for the pulse voltage range from 0 to 5 kV, indicating either an amorphous structure or nanocrystallites with a size of less than 5 nm.

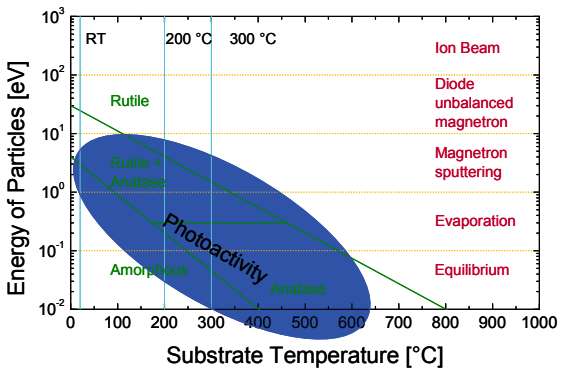

Figure 1: Identification of process window to obtain photoactive thin films using the correlation between phase composition and temperature/energy from Ref. 1.

A summary of these results on the photoactivity is schematically presented in Figure 6. As can be seen, the phase composition is more important than the microstructure for the photoactivity [3]. The nanocrystalline or amorphous film obtained at 5 kV and RT is photoactive, similar to the columnar structured film at 5 kV and 200 °C. In contrast, the film deposited with 5 kV pulse voltage at 300 °C, which is structurally nearly identical, except for slightly smaller grains, is not photoactive at all. A good agreement between the photoactive thin films and a phase composition of anatase or an anatase/rutile mixture is observed.

However, the absolute quantification of the particle energies in the present MePBIID experiments is still unfinished. Secondly, the published model [1] is purely empirical and only summarizes experimental observations. The replacement of substrate temperature with energy roughly implies an increase in the energy by a factor of 10 for a decrease of 300 °C. Albeit, this logarithmic scale is counter-intuitive.

The replacement of substrate heating with ion energy is possible in a restricted process window, thus enabling the formation of photoactive TiO_2 thin films on temperature-sensitive substrates. However, no exact correlation between microstructure or phase composition with the photoactivity was found.

Literature

[1] P. Löbl,. M. Huppertz, D. Mergel, Thin Solid Films 251 (1994) 72.
[2] D. Manova, A. Gjevori, F. Haberkorn, J. Lutz, S. Dimitrov, J. W. Gerlach, E. Valcheva, S. Mändl, Phys. Stat. Sol. a 206 (2009) 71.
[3] A. Gjevori, K. Nonnenmacher, B. Ziberi, D. Hirsch, J. W. Gerlach, T. Höche, D. Manova, S. Mändl, Nucl. Instrum. Meth. B 267 (2009) 1658.

Personal Activities and Scientific Events

Habilitations, Doctoral and Diploma Theses

Activities in Scientific Organisations

Honours and Awards

Lectures and Seminars

Scientific Meetings and Institute Colloquia

Personal Activities

Habilitations, Doctoral and Diploma Theses

Habilitations

Roman Flynt
Ionizing radiation and ozone in environmental studies: intermediates, stable products and mechanistic concepts
Adam Mickiewicz University Poznan/Polen, 2008

Doctoral Theses

Vikas Baranwal
Synthesis and modification of GaN by ion beams
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2008

Bhasker Bantu
Geschützte N-Heterozyklische Carbene (NHCs) und NHC-Metall Komplexe als (latente) Katalysatoren für die PUR Synthese, sowie für Carbony-, Hydrosilylierungs, Cyanosilylierungs- und Hydroformylierungsreaktionen
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2008

Ismaiel Mohamed Raaif
CO_2-laser nitriding of titanium
Sohang University, Egypt, 2008

Nicolas Imlinger
Multivariate modeling of reaction kinetics using spectroscopy and calorimetry – a chemometric approach
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2008

Christian Patzig
Glancing angle deposition of silicon nanostructures by ion beam sputtering
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Marisa Mäder
Nanostructures by diffraction mask projection laser ablation
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Rajendar Bandari
Preparation, characterization and application of monolithic materials to high-performance liquid chromatography and heterogeneous catalysis
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2009

Falk Brustmann
Funktionale Blockcopolymere – Synthese, Charakterisierung und Anwendungen in der mizellaren Katalyse
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2009

Diploma and Master Theses

Andre Mießler, diploma thesis
Mechanical properties of chiral nanostructures
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2008

Tina Hofmann, diploma thesis
In-situ-Untersuchungen an einem atmosphärischen Plasmajet zur Behandlung organischer Filme
Fachhochschule Osnabrück, Fakultät Ingenieurwissenschaften und Informatik, 2008

Katharina Nonnenmacher, master thesis
Abscheidung photoaktiver Titanoxid-Dünnschichten durch Metall-Plasma-Ionen-Immersions-Implantation
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2008

Matthias Schlegel, diploma thesis
Silber–Beschichtung und Implantation für Implantatschrauben
Fachhochschule Jena, Fachbereiches Medizintechnik und Biotechnologie, 2009

Markus Lippmann, diploma thesis
Musterübertragung mittels Phasenmasken für die Diffraktions-Maskenprojektions-laserablation
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Claudia Ernst, master thesis
Ortsaufgelöste Funktionalisierung monolithischer Materialien mittels strahlen-chemischer und Übergangsmetall-katalysierter Polymerisation
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2009

Anja Wehrmann, diploma thesis
Monolithische Verschaltung von flexiblen Dünnschichtsolarzellen
Fachhochschule Mittweida, Fakultät Elektro- und Informationstechnik, 2009

Susanne Perlt, diploma thesis
Gold-Nanostrukturmatrizen auf dünnen AlO_x-Schichten
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Bachelor Theses

Janett Teufert
Oberflächenanalytik an lasermodifizierten Dünnschichtsystemen
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Thomas Heyn
Investigation of sputter yields of isolating materials
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2009

Richard Bellmann
Laserabtrag polymerer Schichten zur Rückseitenkontaktierung flexiber Solarzellen
Fachhochschule Jena, Studiengang Laser- und Optotechnologien, 2009

Activities in Scientific Organisations

F. Bauer

- Member of the Steering Committee of the 'International Symposium on Ionizing Radiation and Polymers' (up to 2009)

M. R. Buchmeiser

- Chairman of the Scientific Advisory Board of the Institut für Nichtklassische Chemie, University of Leipzig
- Program Chairman of the '18th Intern. Symposium on Olefin Metathesis and Related Chemistry', Leipzig 2009
- Member of the Advisory Board of 'Molekulare Chemie' of the Association of German Chemists

W. Knolle

- Member of the Scientific Committee of the 'International Symposium on Ionizing Radiation and Polymers'
- 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'
- Member of the International Advisory Committee of the International Conference 'Recent Advances in Electrochemistry, Science, and Technology'

B. Rauschenbach

- Member of the Council 'Condensed Mater' of the German Physical Society (up to 2008)
- Member of the Advisory Board of the International Union of Vacuum Science, Technology and Application (IUVSTA)
- Member of the Curatorship for 'Innovation and Science'
- Berufung zum Mitglied des Leipziger Forschungsforums an der Universität Leipzig 2008
- Chairmen of '16th International Conference Ion Beam Modification of Materials' Dresden 2008

- Member of the Advisory Board '11th and 12th International Conference on Plasma Surface Engineering', Garmisch-Partenkirchen 2008 and 2010
- Member of the International Advisory Committee '14th International Conference of Thin Films', Ghent 2008
- Sprecher der DFG-Forschergruppe 845 'Selbstorganisierte Nanostrukturen durch niederenergetische Ionenstrahlerosion'
- 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

B. Ziberi

- Ruf als Professor und Prorektor der Universität, Universität Tetova/ Mazedonien

M. R. Buchmeiser

- Ruf als W3-Professor für Makromolekulare Chemie, Universität Stuttgart, Fakultät für Chemie

T. Höche

- Ruf als Außerplanmäßiger Professor für Experimentelle Physik, Universität Leipzig, Fakultät für Physik und Geowissenschaften

B. Rauschenbach

- Ruf als ständiger Gastprofessor, Wuhan University/China, Center for Nanosience and Nanotechnology

S. G. Mayr

- Ruf als W2-Professor (akzeptiert), TRM / IOM / Fakultät für Physik und Geowissenschaften, Universität Leipzig

- Ruf als Professor in Materials Science (abgelehnt), University of Pittsburgh, PA, USA

Lectures and Seminars

Lectures

F. Bauer

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

- *Radioaktivität, Kernenergie und Strahlenschutz*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 08/09

- *Radioaktivität, Kernenergie und Strahlenschutz*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 09/10

M. R. Buchmeiser

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

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

- *Molekulare Heterogenkatalyse*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 08

- *Nanotechnologie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 08

- *Molekulare heterogene Katalyse*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 09

- *Nanotechnologie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 09

- *Makromolekulare Chemie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 08/09

- *Spezial- und Funktionspolymere*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 08/09

T. Höche

- *Mikro- und Nanostrukturcharakterisierung mit elektronen-mikroskopischen Techniken*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 07/08

- *Mikro- u. Nanokosmos: Abbildung und Analyse mit Elektronen*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 08/09

- *Abbildung und Analysen mit Elektronen*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 09/10

J. W. Gerlach

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

- *Beschichtung u. Beschichtungsverfahren*
  Hochschule für Technik, Wirtschaft und Kultur Leipzig, Fachbereich für Maschinen- und Energietechnik
  winter 08/09

S. Mändl

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

- *Oberflächenmodifizierung von moderen Leichtmetallen*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 08

- *Plasmaphysik I: Plasmatechnologie*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 08/09

- *Plasmaphysik II: Kollektive Plasmen*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 09

- *Plasmaphysik*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 09/10

S. G. Mayr

- *Struktur der Materie*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  summer 09

- *Oberflächenphysik*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 09/10

B. Rauschenbach

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

- *Ionen-Festkörper-Wechselwirkung*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 08/09

- *Physik dünner Schichten*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  summer 09

- *Oberflächen- und Dünnschichtanalyse*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 09/10

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

A. Schindler

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

Seminars

M. R. Buchmeiser

- *Seminar für Diplomanden/Dissertanten*
  Universität Leipzig, Fakultät für Chemie und Mineralogie
  winter 07/08, summer 08, winter 08/09

S. G. Mayr

- *Seminar zur Oberflächenphysik*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften
  winter 09/10

S. G. Mayr and B. Rauschenbach

- *Materialwissenschaftliches Seminar*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften und Leibniz-Institut für Oberflächenmodifizierung e. V. Leipzig
  winter 09/10

B. Rauschenbach

- *Materialwissenschaftliches Seminar*
  Universität Leipzig, Fakultät für Physik und Geowissenschaften und Leibniz-Institut für Oberflächenmodifizierung e. V. Leipzig
  winter 07/08, winter 08/09, summer 09,

Scientific Events

Scientific Meetings and Institute Colloquia

Scientific Meetings

1st Scientific Symposium of Graduate School BuildMoNa, Leipzig, Germany, 07.-08.02.2008

XV. Workshop 'Oberflächentechnologie mit Plasma- und Ionenstrahlprozessen', Mühlleithen, Germany, 04.-06.03.2008

16th International Conference on Ion Beam Modification of Materials, Dresden, Germany, 31.08.-05.09.2008

1st Workshop for Doctoral Candidates of Graduate School BuildMoNa, Leipzig, Germany, 16.-17.10.2008

XVI. Workshop 'Oberflächentechnologie mit Plasma- und Ionenstrahlprozessen', Mühlleithen, Germany, 10.-12.03.2009

2nd Scientific Symposium of Graduate School BuildMoNa, Leipzig, Germany, 02.-03.04.2009

18th International Symposium on Olefin Metathesis and Related Chemistry, Leipzig, Germany, 02.-07.08.2009

Workshop 'Nanoscale Modification of Surfaces and Thin Films', Rathen, Germany, 30.08.-03.09.2009

2nd Workshop for Doctoral Candidates of Graduate School BuildMoNa, Leipzig, Germany, 08.-09.10.2009

Institute Colloquia

P. Sigmund (09.01.2008)
University Southern Denmark, Odense Department Physics & Chemistry, Odense, Denmark
Abbremsung schwerer Ionen im KeV- und MeV-Bereich

P. Schaaf (17.01.2008)
Universität Göttingen, Physikalisches Institut, Göttingen, Germany
Moderne Werkstoffe und Oberflächen: Herausforderung für Herstellung, Analyse und Anwendung

M. Antonietti (24.01.2008)
Max-Planck-Institut für Kolloid- und Grenzflächenforschung, Potsdam, Germany
New polymers and materials for energy applications and the raw material change

F. Svec (14.03.2008)
Lawrence Berkeley National Laboratory, Berkeley, CA, USA
Monolithic columns: present state-of-the-art and future trends

G. Kickelbick (26.03.2008)
Technische Universität Wien, Institut für Synthesechemie, Wien, Austria
Metalloxid-Polymer-Hybridmaterialien – Einblicke in das chemische Design der Grenzfläche

B. Rieger (10.04.2008)
Technische Universität München, Lehrstuhl für Makromolekulare Stoffe, München, Germany
Nachhaltigkeit in der Materialsynthese: Kohlenstoffoxide als Basis polarer Copolymerarchitekturen

R. Wehrspohn (24.04.2008)
Fraunhofer-Institut für Werkstoffmechanik, Halle/Saale
Licht – gestalten

H. M. Urbassek (08.05.2008)
Technische Universität Kaiserslautern, Fachbereich Physik, Kaiserslautern, Germany
Zerstäubung durch Atom- und Clusterbeschuss: Einsichten aus Molekulardynamik-simulation

A. Wucher (15.05.2008)
Universität Duisburg–Essen, Institut für Physik, Duisburg, Germany
Billard auf atomaren Dimensionen: Ionenstrahlen in der Oberflächenanalytik

K. Nielsch (22.05.2009)
Universität Hamburg, Institut für Angewandte Physik, Hamburg, Germany
Atomic layer deposition

P. Regenfuß (29.05.2008)
Laserinstitut Mittelsachsen, Mittweida, Germany
Models of specific material conversion on the micro and nano scale during laser irradiation

D. Gerlich (05.06.2008)
Technische Universität Chemnitz, Institut für Physik, Chemnitz, Germany
The production and study of cold and slow molecular ions using RF traps

U. S. Schubert (12.06.2008)
Friedrich-Schiller-Universität Jena, Institut für Organische Chemie und Makromolekulare Chemie, Jena, Germany
Inkjet printing of functional polymers and materials

S. Schulz (19.06.2008)
Fraunhofer Institut für Zuverlässigkeit und Mikrointegration, Chemnitz, Germany
Deposition and characterization of porous ultra-low-k dielectrics films

R. Micura (26.06.2008)
Universität Innsbruck, Institut für Organische Chemie, Innsbruck, Austria
Chemical synthesis in ribonucleic acid (RNA) research: on riboswitch and ribosome function

R. Hergenröder (03.07.2008)
Leibniz-Institut für Analytische Wissenschaften, Dortmund, Germany
Short and ultrashort pulse laser: perspectives in analytics and material processing

H. Frey (10.07.2008)
Johannes Gutenberg-Universität Mainz, Institut für Organische Chemie, Mainz, Germany
Von der faszinierenden Vielfalt der Polyether zur Polymerchemie in Mikroreaktoren

J. Bliedtner (17.07.2008)
Fachhochschule Jena, Fachbereich SciTec, Jena, Germany
UP-Bearbeitung mit geometrisch bestimmter Schneide

I. Avramov (24.07.2008)
Bulgarian Academy of Sciences, Sofia, Bulgaria
Photoacoustics, or can we listen how the molecules move

N. Moszner (23.09.2008)
Ivoclar Vivadent AG, Research & Development, Schaan, Liechtenstein
Neue Komponenten für dentale Füllungsmaterialien

K. Landfester (16.10.2008)
Max-Planck-Institut für Polymerforschung, Mainz, Germany
Von Nanopartikeln zu komplexen Nanokapseln

M. Rehahn (23.10.2008)
Technische Universität Darmstadt, Ernst-Berl-Institut für Technische und Makromolekulare Chemie, Darmstadt, Germany
Elektrisch leitfähige Polymere

J. Krug (28.10.2008)
Universität zu Köln, Institut für Theoretische Physik, Köln, Germany
Strukturbildung und nichtlineare Dynamik an Kristalloberflächen

P. Fratzl (30.10.2008)
Max-Planck-Institut für Kolloid- und Grenzflächenforschung, Potsdam, Germany
Nature's hierarchical materials

M. Ballauff (06.11.2008)
Universität Bayreuth, Physikalische Chemie I, Bayreuth, Germany
Metallic nanoparticles immobilized in polymer colloids: synthesis and catalytic properties

P. Hess (13.11.2008)
Universität Heidelberg, Institut für Physikalische Chemie, Heidelberg, Germany
Grundlagen und Anwendungen der Photochemie an Siliziumoberflächen

S. Förster (20.11.2008)
Universität Hamburg, Physikalische und Makromolekulare Chemie, Hamburg, Germany
0-3-dimensionale Nanocomposites

F. Bisio (28.11.2008)
Università di Genova, Dipartimento di Fisica, Genova, Italy
Tailoring the magnetic properties of ultrathin films and multilayers by ion sputtering

H.-J. Möller (04.12.2008)
Technische Universität Bergakademie Freiberg, Experimentelle Physik, Freiberg, Germany
Defekte in Solarsilizium

A. Fery (10.12.2008)
Universität Bayreuth, Physikalische Chemie II, Bayreuth, Germany
Nano- and microstructured polymeric coatings

S. Mecking (11.12.2008)
Universität Konstanz, Lehrstuhl für Chemische Materialwissenschaft, Konstanz, Germany
Kontrolle der molekularen und der partikulären Struktur bei katalytischen Polymerisationen (vgl. http://www.chemie.uni-konstanz.de/agmeck/PUBLICATIONS.HTML)

A. Jungbauer (08.01.2009)
Universität für Bodenkultur Wien, Institut für Angewandte Mikrobiologie, Wien, Austria
Separation of large biomolecular assemblies by monoliths

J. Hormes (22.01.2009)
University of Saskatchewan, Canadian Light Source Inc. Saskatoon, Canada
Innerschalen-Spektroskopie: von der theoretischen 'Kuriosität' zum Werkzeug der spektroskopischen Strukturanalyse

J.-F. Lutz (19.03.2009)
Fraunhofer-Institut für Angewandte Polymerforschung, Potsdam, Germany
Design of PEG-based stimuli-responsive materials

B. Kräutler (03.04.2009)
Universität Innsbruck, Institut für Organische Chemie und Zentrum für Molekulare Biowissenschaften, Innsbruck, Austria
On Balls and Bowls

K. Kroy (16.04.2009)
Universität Leipzig, Institut für Theoretische Physik, Leipzig, Germany
Die Physik der Wanderdünen – warum ist die Wüste nicht flach?

B. Abel (23.04.2009)
Universität Leipzig, Wilhelm-Ostwald-Institut für Physikalische und Theoretische Chemie, Leipzig, Germany
Dynamics and spectroscopy near interfaces

F. Mücklich (30.04.2009)
Universität des Saarlandes, Lehrstuhl für Funktionswerkstoffe, Saarbrücken, Germany
Nano-Tomographie mit Ionen- und Elektronenstrahlen – ein Werkzeug zur Aufklärung komplexer Mikrostrukturen und lokaler Degradation

R. Haag (07.05.2009)
Freie Universität Berlin, Institut für Chemie und Biochemie, Berlin, Germany
Functional Dendritic Architectures

J. Krüger (07.05.2009)
Bundesanstalt für Materialforschung und –prüfung, Berlin, Germany
Modifikation und Zerstörung von optischen Komponenten als Limit für die Anwendung ultrakurzer Laserimpulse

G. Fink (14.05.2009)
Max-Planck-Institut für Kohlenforschung, Mülheim, Germany
Arbeiten zur Ziegler-Natta-Katalyse: eine Anthologie

C. Ronning (28.05.2009)
Universität Jena, Institut für Festkörperphysik, Jena, Germany
Ion beam doping of semiconducter nanowires

J. Spatz (25.06.2009)
Max-Planck-Institut für Metallforschung, Stuttgart, Germany
Molecular engineering of cellular environments: cell adhesion to nano-digital surfaces

D. As (02.07.2009)
Universität Paderborn, Department Physik, Paderborn, Germany
Bauelemente basierend auf breitbandigen Halbleitern für optoelektrische und elektrische Anwendungen

S. Ludwigs (06.07.2009)
Albert-Ludwigs-Universität Freiburg, Freiburger Materialforschungszentrum, Freiburg, Germany
Optoelectronic Polymer-Nanohybrid Materials

D. Hesse (09.07.2009)
Max-Planck-Institut für Mikrostrukturforschung, Halle/Saale, Germany
Herstellung, Mikrostruktur und Eigenschaften von Nanokondensatoren

Y. Sano (19.10.09)
Osaka University, Department of Precision Science and Technology, Osaka, Japan
Machining of semiconductor substrates using atmospheric pressure plasma

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