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für
Oberflächenmodifizierung e.V.

BIANNUAL REPORT 2004/2005

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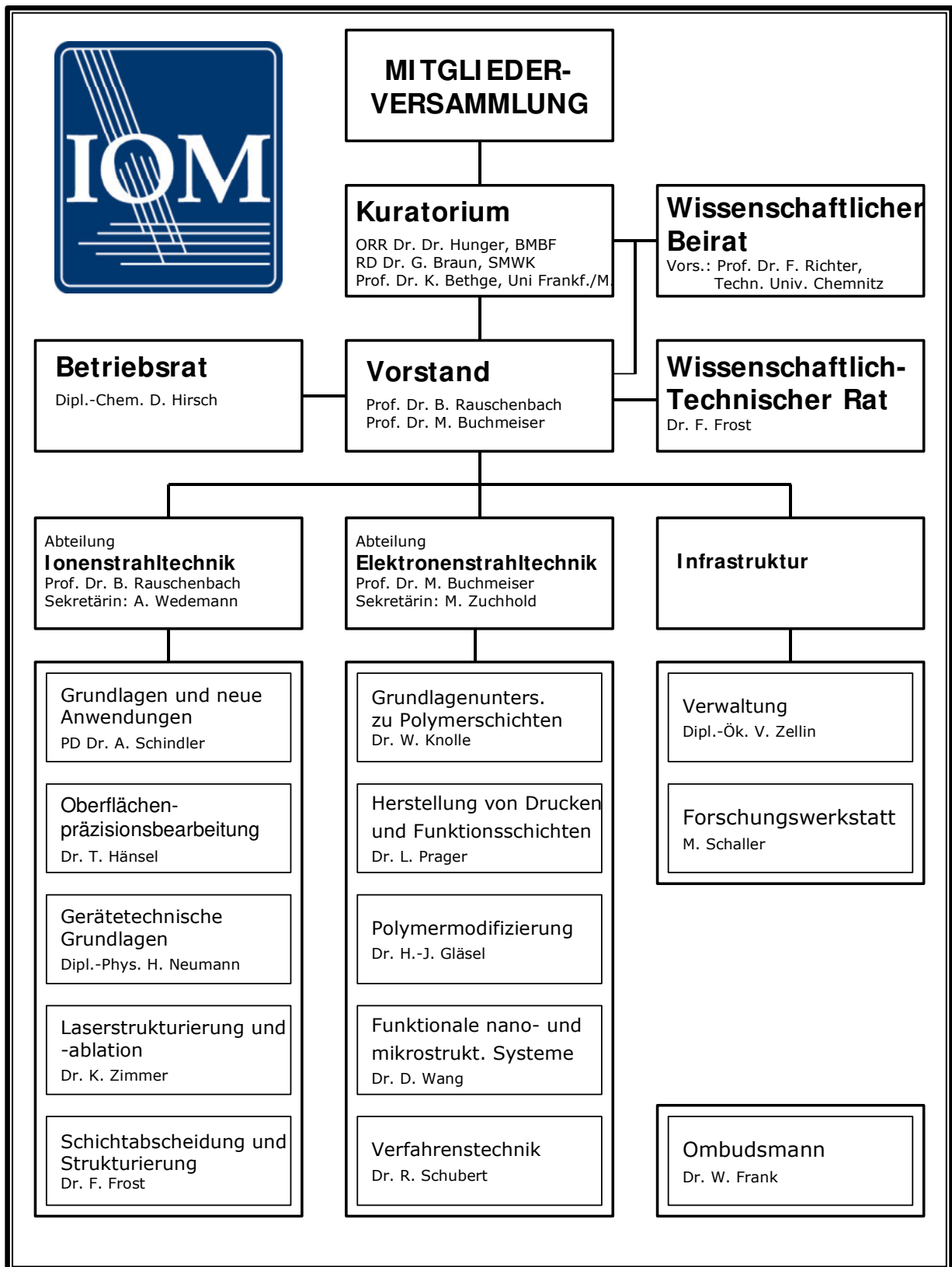
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Preface

The Leibniz-Institut für Oberflächenmodifizierung e.V. (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.

The IOM has 47.5 permanent positions, including scientific, technical, and administrative personal. In average about 90 additional employees are funded from special programs as well as governmental and industrial projects. The IOM is a member of the Leibniz Association.

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

In this report the IOM presents its scientific activities and major achievements in the years 2004 and 2005. In this context, the Biannual Report 2004/2005 presented here gives a comprehensive summary of our results. In the first part, overviews on selected projects are given, arranged according to the structure of the IOM research program. These overviews are supplemented by feature articles on selected topical highlights. Finally, the appendices give a full list of publications, talks, teaching activities, and other achievements of the IOM staff.

The Institute would like to thank all friends and organisations who supported its progress in the last two years. Special thank is due to our Board of Trustees and Scientific Advisory Board. Our partners from industry and other research institutes play an essential role for the IOM. The Board of the Institute would like to thank all members and guests of the institute for their active and excellent contributions to a successful development.

Leipzig, January 2006

Prof. Dr. Bernd Rauschenbach
Prof. Dr. Michael Buchmeiser



In January 2005 Rainer Mehnert has finished his professional activities in our Institute. He was a member of the Board of the IOM and the Head of the Electron Beam Department. Very many thanks for his work at the IOM and our best wishes.

A warm welcome to our new colleague Michael Buchmeiser. Since January 2006 he is a member of the Board of the IOM and the Head of the Electron Beam Department. We are glad that he joined us and wish him success and good luck for his future.



In 2005 Axel Schindler received the Manfred von Ardenne Award from the Europäische Fördergemeinschaft für Dünne Schichten for his contributions to the transfer of the ultra-precision surface treatment into the industry.

Bashkim Ziberi received the Young Scientist Award of the European Material Research Society in the year 2004 for his studies to self-assembly processes on surfaces by ion beam erosion.



Scientific and Technology Results

Reports and Selected Results

Ultra-precision surface finishing by ion beam techniques

T. Hänsel, F. Frost, R. Fechner, A. Schindler

Introduction

Ion beam etching (IBE) and ion beam sputter deposition (IBSD) become more and more established in high precision surface processing for advanced optical, electronic, and mechanical products. In optics fabrication ion beam figuring (IBF) and ion beam smoothing (IBS) are able to overcome physical constraints of the conventional full lap and small tool abrasive polishing processes. This together with an accurate control of the removal rates and the high stability in time and shape of the ion beam of different size result in an efficient correction of low, mid, and high spatial frequency surface errors down to the sub-nanometer amplitude level with almost no or minimum surface or subsurface damage, respectively due to the gentle beam surface interaction. Reactive ion beam etching (RIBE) is an effective technique to transfer 3D resist mask micro- and nano-structures proportionally into hard optical materials, e.g. for nano-optics or diffractive optical elements.

Ion beam figuring

Present ion beam figuring technology development in the IOM [1,2] is aimed to correct surface errors of millimetre spatial size range and below nanometer dimension in height to meet the demanding requirements especially for lithography DUV, EUV, and synchrotron optics. Therefore we concentrate on the development of ion sources for stable and reliable long-term operation with rotationally symmetric Gaussian beam shape. We operate a 13.56 MHz RF-source with an 8 mm FWHM beam. This beam is reduced by diaphragms of different sizes to 2 mm, 1 mm and 0.5 mm FWHM, respectively. We used this source for the surface figuring of advanced synchrotron beam line optics very successfully [3].

IBF is performed by controlled scanning line by line across the surface with a dwell time distribution calculated by a deconvolution program proportional to the desired material removal depth distribution. For the adequate surface topology

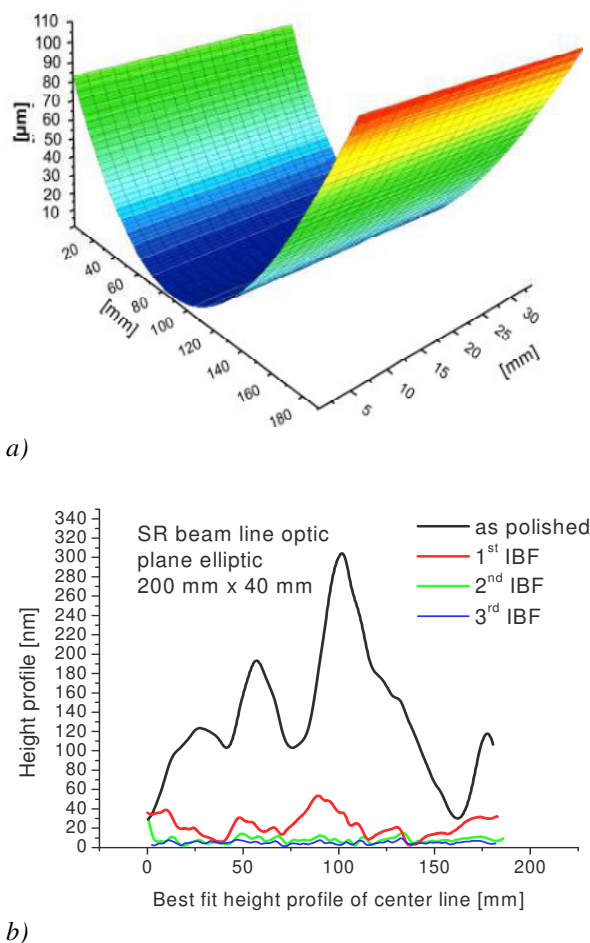


Figure 1: a) Pseudo 3D presentation of a plane-elliptical surface of a synchrotron-beamline optic stitched from seven adjacent sub-apertures with a 30 % overlap in one direction. Each sub-aperture interferogram was measured with a pixel distance of 0.082 mm, b) best fit height profiles of the plane elliptical mirror.

measurements of the asphere surfaces we developed a stitching interferometry method [4].

Figures 1 and 2 show recent results of ion beam polishing error correction of synchrotron beam line optics surfaces [3]. We used small spot ion beams down to a size of 2 mm FWHM to improve the surface figure in a sequence of IBF processing steps.

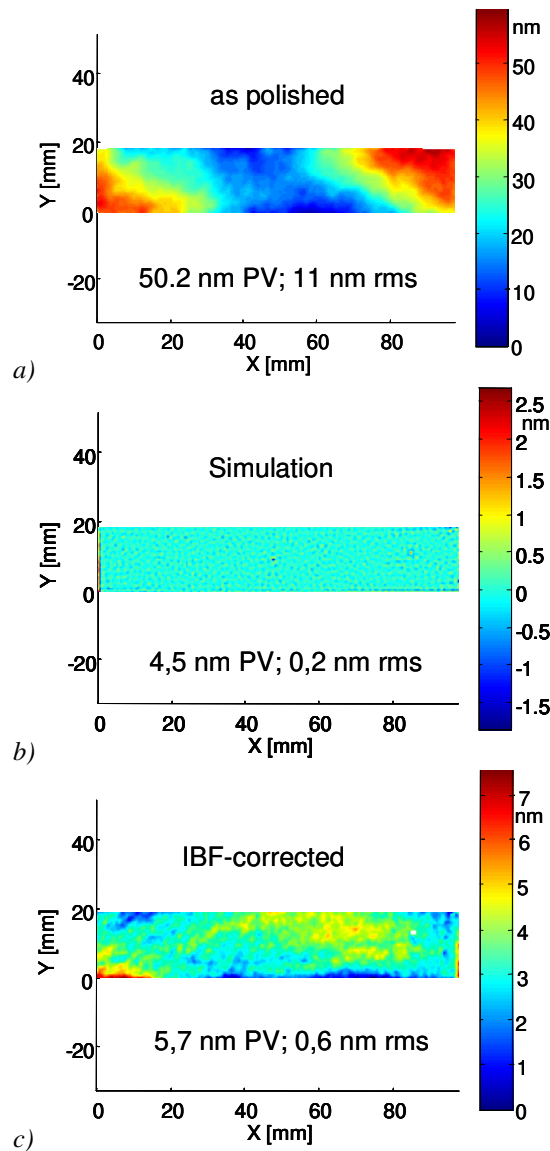


Figure 2: a) Interferometer surface topology measurements of a 100 mm x 20 mm Si plane substrate for a synchrotron beam-line grating as polished, b) computer simulation result for the IBF surface error correction, c) interferometer surface topology measurement of the surface after IBF correction.

Ion beam smoothing

Detailed investigations have been conducted in the IOM for the use of the planarising film technique and the near normal incidence ion beam etching direct smoothing as single step techniques or both methods combined for optical surfaces smoothing [5].

Normal incidence ion beam etching direct smoothing is illustrated in Figure 3, where the surface of an as-received quartz wafer was

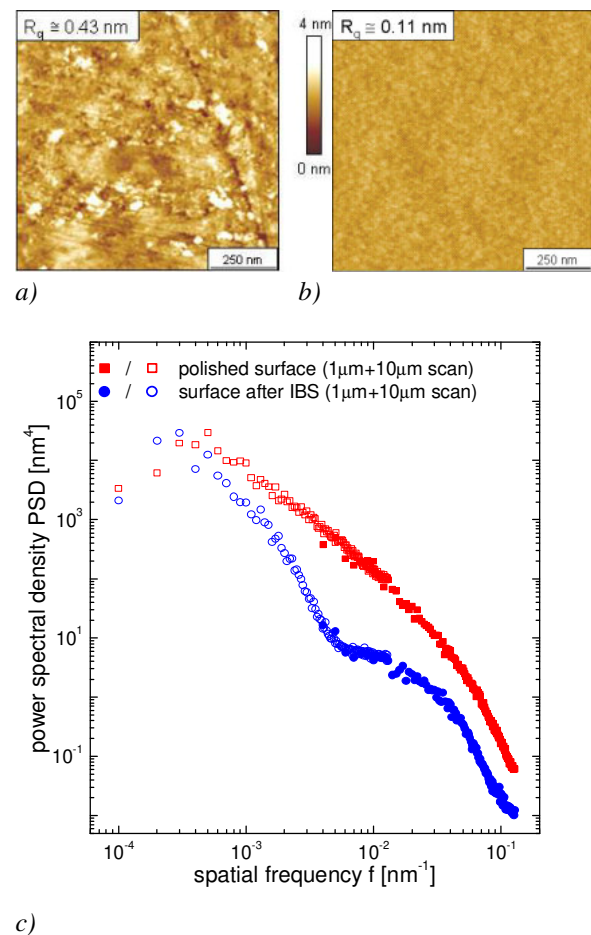


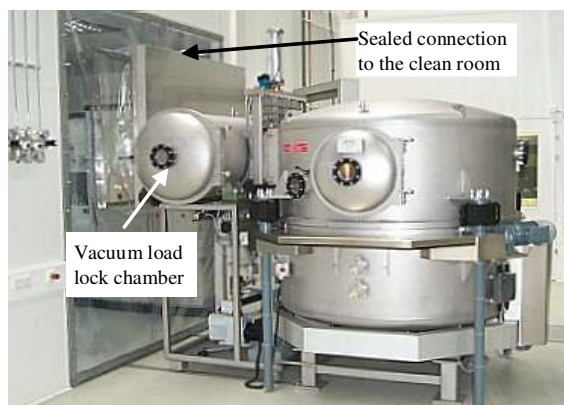
Figure 3: Ion beam direct smoothing of fused silica; a) optically polished; b) after Ar IBE (E_{ion} : 600 eV, α_{ion} : 0°, 300 nm material removal); the roughness has been reduced from 0.43 nm to 0.11 nm rms; c) angular averaged power spectral density for the surfaces shown gives evidence of smoothing for all spatial wavelengths grasped by the AFM (10^{-4} - 0.128 nm⁻¹).

smoothed under optimised ion beam sputter conditions down to 0.1 nm rms roughness. Figure 3c shows that smoothing occurs over all spatial frequencies covered by the AFM measurements. We have demonstrated the smoothing of surface features resulting from magneto-rheological finishing on silicon surfaces [6] and of ion beam etched ZERODUR® [7].

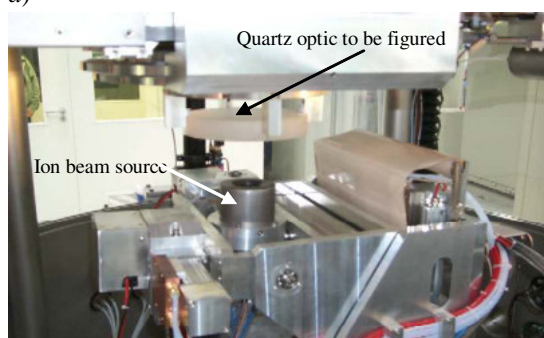
Ion beam figuring plant

During the last three years we have developed and qualified IBF technology for the final machining of high performance optics components mature for production requirements.

The NTG/NTGL GmbH has developed a modular machine system by comprising three standard



a)



b)

Figure 4: a) Ion beam figuring plant UPFA1 at the class 100,000 clean room lab in the IOM, b) upper part of the process chamber has been lifted (for easy maintenance work); the five axis system with the ion source and the face down mounted optic on the carrier are shown inside the chamber.

units for the IBF and ion beam smoothing of optics up to 300, 450, and 700 mm in diameter, respectively [8]. The main components of the systems are (i) a stainless steel processing chamber with a base vacuum of 10^{-4} Pa, (ii) a vacuum pumping system including a turbo molecular pump and a mechanical pump, (iii) a computer controlled precision five axes system (x, y, z, tilt A and tilt B) for the scanning path movement of the ion source, (iv) an RF-ion source with an automated Faraday cup system for measuring the beam profile and the beam position, (v) a work-piece handling system comprising lens carrier, automatic transport system, and self-adjusting mechanical fixing in the processing position of the lens, (vi) a vacuum load lock chamber enabling uninterrupted operation of IBF with handling time of the workpiece within a minute, (vii) a power supply cabinet for all machine parts and the computer hardware, and (viii) a process gas handling system. The machine is operated from a laptop computer.

The plant type for optics up to 700 mm in diameter has as an option a lens carrier lifting and turning device for easy handling the heavy weight parts. Figure 4 shows the IBF unit for up to 450 mm parts installed in the IOM test lab. The machine is operated from a class 100,000 clean room. In Figure 4b where the upper part of the process chamber has been lifted (for easy maintenance work) the five axis system with the ion source and the face down mounted optic on the carrier are shown inside the chamber.

Ion beam source

The ion source is an RF-type (13.56 MHz) developed by the IOM (Figure 5). The source is equipped with an RF-matching network to adjust the characteristic impedance. This allows the stable operation during moving the source by the five-axes motion system. The source has been optimised for long-term stability with respect to minimise downtime and to provide for stable and reliable beam removal function over hours. At the same time we improved the versatile use of the source by enabling different beam sizes with reasonable working distances by means of different extraction grid designs and additional diaphragm mounted in front of the source for getting reduced beam sizes down to 0.5 mm FWHM. A hidden hot filament neutraliser mounted outside of the beam area allows a more than 100 hours maintenance free operation of the source. In Figure 5 results of the beam shape diagnostics are shown.

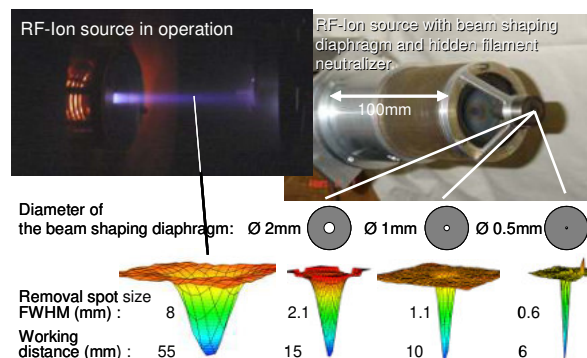


Figure 5: Measured sizes of the footprints of the beam by interferometry of an etched depletion on fused silica for different operation conditions of the RF ion source without and with beam shaping diaphragms; the etch depths has been normalised in this presentation with respect to the 8 mm spot.

RIBE proportional transfer of 3D resist masks

With the help of reactive ion beam etching (RIBE) we increase the profile depth of 3D resist mask, fabricated by holographic lithography [9], necessary for high efficiency diffractive optical elements like Fresnel lenses or diffraction gratings, and we transfer the diffractive structure from the soft resist into the hard fused silica or glass material in a single step. We use a standard ion beam etching plant with a six-inch broad beam Kaufman-type ion source. To optimise the etch rate ratio between the resist mask and the optical material we vary the gas flow rates of the etch gases of $\text{CF}_3\text{H}/\text{O}_2$ or CF_4/O_2 , respectively. In this way, we adjust the desired grating depth very precisely. Figure 6 shows examples of a Fresnel lens for DUV application and of Raman monochromator gratings both made of fused silica.

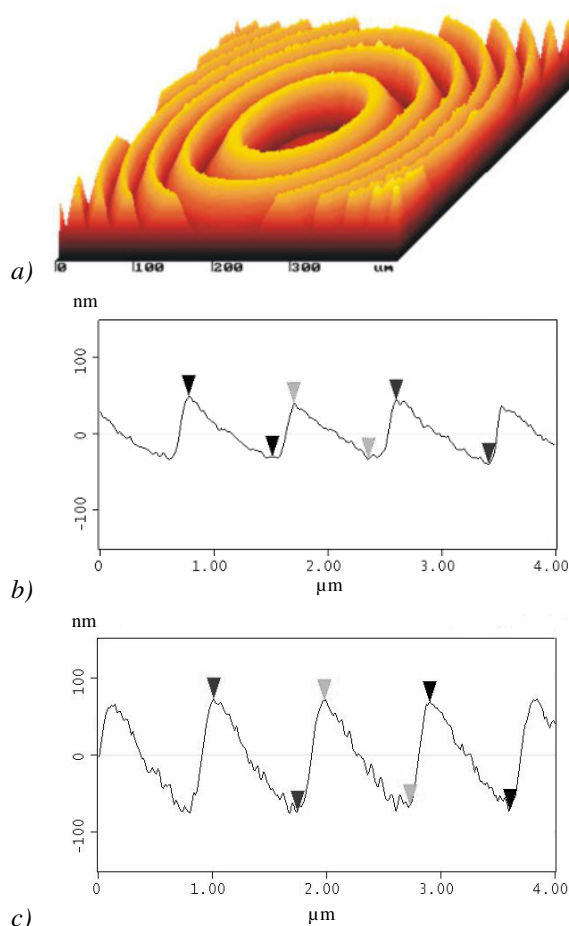


Figure 6: AFM measurements of a) a Fresnel lens transferred into fused silica and b), c) of a master grating structure for a Raman monochromator with a line density of 1180 L/mm; b) resist mask, c) topology transferred by RIBE into a fused silica surface.

Conclusion

Ion beam etching is a versatile technology with a high degree of predictability due to the high stability of state of the art ion sources and the acquired knowledge of the physics of beam surface interaction. The independent control of the ion energy and the ion current density over wide ranges and the possible additional use of chemical reactive species in combination with physical sputter removal allow solving tasks in a wide variety of applications.

The developed techniques, the productive machines and the fabrication results show that ion beam etching technologies are advanced and powerful tools for present and future surface processing and finishing in precision optics technology.

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Broad beam ion source development

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Introduction

Modern thin film technologies as well as surface modification processes require high standards in either thin film quality or bulk surface properties, which became especially apparent since industry utilizes these techniques in broad varieties of different products. Ion broad beam techniques offer more and more solutions mastering superior requests in research as well as in industrial high end applications.

To obtain a satisfying source performance, which is mainly determined by the chosen grid system, detailed knowledge about the basic beam parameters like beam composition, beam profile, and ion energy distribution is necessary and obtained by various ion beam diagnostic methods.

On the basis of this knowledge together with a validated computer simulation of the broad ion beam performance the design of process adapted grid systems is feasible.

In close co-operation with partners from industry our ion source concept was successfully adapted and utilized in different ion beam process equipments. Lately, the advantage of broad ion beam techniques is demonstrated e.g. during deposition of multilayer for mask blanks to be used in soft x-ray lithography (EUV), thin film head production processes, ion beam figuring applications, surface smoothing of fused silica, self organized pattern formation during ion erosion (e.g. on silicon) and hardening of stainless steels without loss of its good corrosion resistivity.

Ion source concept

Different types of broad beam ion sources have been established, since the time ion sources are applied in material processing [1,2,3]. Beside of specific grid system and the choice of source materials, broad beam ion sources can be classified according to the plasma excitation principle.

But which scheme of plasma excitation is best? Sources with hot filament excitation (Kaufman

type) are easily to handle, but require frequent maintenance cycles and are limited in reactive gas operation. For that reason, sources with electrodeless excitation principles were developed, giving longer operation cycles and better performance in reactive gas operation.

A well introduced electrode-less technique for plasma heating is the so called transformer coupled plasma (TCP) with a RF driven primary coil and the plasma acting as the secondary one. A TCP configuration requires a high RF power at the primary coil, and, especially for plasma etching equipment, a faraday shield is helpful to eliminate a direct capacitive coupling between the primary electrical field at the coil and the plasma. However, most of the really established configurations are running by the unwanted capacitive coupling, resulting in higher plasma sheath potentials within the source and consequently high sputter erosion of the source components assembled in the discharge chamber.

A very effective electrode-less excitation principle is the electron cyclotron resonance heating (ECR) in a microwave field. The introduction of a steady magnetic field, which establishes resonance between the applied microwave frequency ω_{MW} and the electron cyclotron frequency $\omega_{ce} = eB/m_e$, allows for the operation with high plasma densities as well as no cavity resonance is required for the plasma performance. Because of the cyclotron resonance, the gyrating electrons rotate in phase with the right hand polarised wave, seeing a steady electric field over many gyro orbits. With the electron mean free path large enough, the ECR heating provides for the most effective energy transfer, very low plasma sheath potentials, and low source parts erosion.

Beside the plasma excitation, the source performance is strongly affected by the grid system. Different shapes of the grid design as well as the

geometry of the grid holes realise specific beam profiles. Typically the ion sources are equipped with a double or alternatively triple grid system. Double grid systems are used as the standard solution, whereas triple grid systems deliver clearly a better beam performance for high end applications. The third grid, mostly driven at ground potential, realises defined potential conditions at the grid beam side and delivers well defined beam divergence angles. Finally, the accelerator grid gets protected from redeposition especially for reactive gas operation, which also reduces the risk of arcing.

The grid material, it's mechanical, electrical, thermal and chemical properties and on the other hand it's sputter yield under different conditions plays an important role in the grid system design, life time and stability. For grid system design the sputter yields in dependence on the applied ion energy and incidence angle are to be known, however, for the relevant energy range and ion-grid material combinations a lack of literature data was found. Figure 1 exemplifies our results for measured and calculated sputter yields of molybdenum (Mo) under xenon (Xe) ion bombardment [4, 5].

Since many years different types of ion sources with hot filament, RF and ECR excitation are

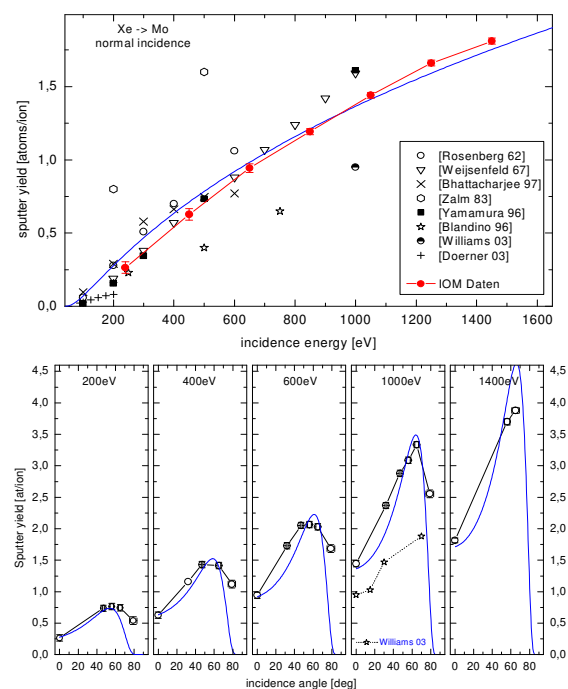


Figure 1: Experimental (squares) and calculated (blue lines) sputter yields for Xe sputtered Mo.

developed at the IOM in Leipzig. These ion sources are designed in a modular concept with beam diameters between 40...350 mm for circular sources and up to several meters for sources with a rectangular design, all equipped by technology-specified grid systems. All types of plasma discharges used here, are running within a ceramics housing providing for full grid support, electrical contacts and gas supply.

Ion beam diagnostics

A 4000 l/sec turbo pumped HV chamber is used for the characterisation of the ion sources. A chamber pressure p_{tot} between 1×10^{-6} mbar and 5×10^{-4} mbar is set by the process gases flow into the source. Because of the gas flow conductance of the grid system, the pressure in the discharge chamber and in the vacuum vessel differs by about one order of magnitude.

An in-house developed 256 Faraday cup array provides information on the beam current density profile across an area of 20 cm x 30 cm. Additionally, an in situ movable single Faraday probe is used for ion density measurements near the grid system, because the large faraday array located

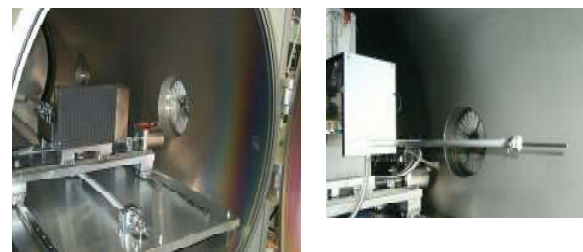


Figure 2: Equipment for Faraday and thermo cup measurements: Computer controlled positioning system-3 linear axis, 1 rotation axis (360 degree), Positioning accuracy < 0,1 mm; vacuum chamber length: 3 m, diameter: 1,2 m; pumping system: 2 turbo pumps ~ 4000 l/s, base pressure: < $1 \cdot 10^{-6}$ mbar.

close to the grids (2 mm-5 mm) hinders the gas flow through the ion source. Figure 2 shows the equipments.

For description of the plasma density profile at the sheath an indirect method is used too. By measuring beam and accelerator currents in dependency of the applied total grid system voltage and fitting this current functions by using a zero order Bessel

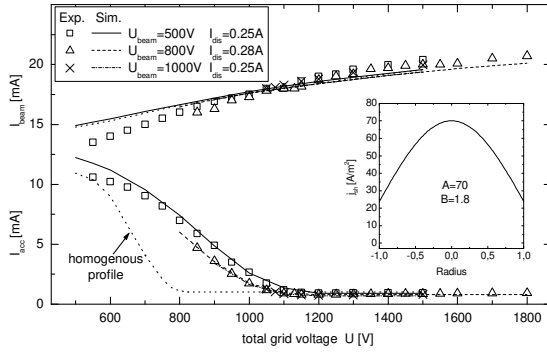


Figure 3: Measurement of beam and accelerator currents in a 3 cm Kaufman-type ion beam source; lines are simulated currents with a zero-order Bessel function (plasma sheath profile in the insert).

function, the plasma profile at the sheath is approximated (Figure 3).

An energy selective mass spectrometer (Hidden Analytical Ltd., EQP 300) is used to observe the beam composition, the ion energy distribution and the divergence angle of the ion beam in different distances. Using the differentially pumped mass spectrometer, a mass and energy resolved analysis of positive beam ions is obtained by sampling through a grounded $200\text{ }\mu\text{m}$ aperture. The ion analysis is provided by an electrostatic sector field analyser (1000 eV), a quadrupole mass filter (300 amu) and finally a channeltron secondary electron multiplier with counting electronics responsible for ion detection. Energy and mass dependent effects have to be taken into consideration of the quantitative discussion of the experimental results [7] and for adaptation of the ion source on the process demands.

Ion beam simulation

Computer simulations are another important feature in the broad beam source development for validation of the experimental results. The ion beam from a broad-beam ion source is extracted by a multi-aperture grid system of two or three grids. A single beamlet is extracted through each aperture and the superposition of the beamlets delivers the broad beam. The ion beam parameters are determined by the geometrical parameters of the extractions system, the applied voltages and the plasma parameters. Dishing the grids increases the stability during operation and focuses (inward dished) or defocuses (outward dished) the

ion beam, offering additional parameters to design the beam.

A simulation strategy has been developed at IOM to model the beam parameters. Single beamlets are modelled in two-dimensions (axial symmetry) using the well-approved ion trajectory code IGUN. Additional modules treat secondary effects to model the beam contamination and the lifetime of the grids affected by charge-exchange and other processes. The broad beam is modelled by a superposition of simulated beamlets at the target position taking into account an inhomogeneous plasma profile (using diagnostic methods described before and PIC-simulation results[6]), the eventually dishing of the grids and other beam shaping elements. The results are the current-density distribution and beam-shape parameters like FWHM and divergence.

Finally, the ion beam profile can be visualized by the interaction of the ions with a micro-disperse particle cloud which has been charged and confined in an additional rf-plasma. By this method, the interaction of the ions (momentum transfer, ion drag) as well as inhomogeneities in the beam can be realistically observed and estimated [8]

Ion beam profile control

Different technologies require specially adapted ion beam density profiles. Taken this into account, three different methods for ion beam profile control are investigated in combination with the ECR plasma excitation.

Electrical beam profile control

The 1D electrical beam profile control of a linear ECR- ion beam source by a segmented accelerator grid (30 segments on 600 mm) and a 30 times beam switch on the basis of a pulse length modulation for switching this segments between positive (blocking) and negative (accelerating) potentials could be successful demonstrated. [9,10]

Grid geometry method

Second, an ion beam profile control by changing the geometry of the grid hole diameters together with the transparency of the whole grid system is shown on measured profile density plots (Figure 4).

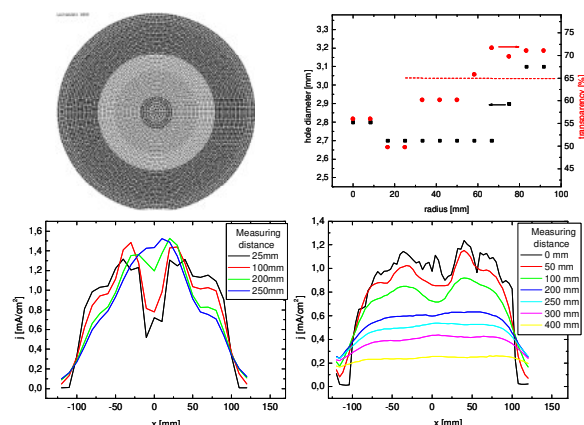


Figure 4: Modelling (top) on the basis of experimental sheath density distribution measurements (bottom left); grid system with homogenous divergence angle by hole diameter and transparency variation; goal (bottom right): homogenous ion beam density profile at 200 mm distance

gated method for ion beam profile control. Using 7 sources with special grid systems in the cluster an ion beam of 400 mm in diameter could be produced with homogeneity of less than $\pm 5\%$. On the other hand a high dense ion beam of about 25 mA/cm^2 can be produced by the alignment of 7 sources with focussed grid systems, whereby the focus of each ion beam is located in the same position approximately 300 mm in distance from the decelerator grid of the centre source.

Conclusions

Broad beam ion source development strategies are presented which are especially suited to meet the current requirements in high end film deposition, surface modification and structuring. The sources are designed with a modular concept independent from the excitation principle. A discharge lining from Al_2O_3 provides for full grid support and all electrical and gas supplies. Additionally, the lining guarantees a full reactive gas compatibility for all ion source types.

The grid material investigations are exemplified shortly. Especially for grid diameters more than 40 mm the mechanical properties play an important role and require intensive developments in the field of grid building technologies.

The report of different diagnostic techniques for the plasma discharge and the ion beam is suitable to demonstrate the development basics for source adaptation on technological demands. The mass

spectrometric results allow conclusions about processes occurring in the plasma discharge. Especially the information about the beam energy and mass composition could provide important correlations about the process performance. The methods for direct and indirect plasma sheath description are stringent necessary as the basis of a validated broad beam modelling.

A validated modelling of beam properties with help of analytical data designing grid system configurations is one of the most important tools in source adaptation. The described strategy is well suited for both, grid life time evaluation and for ion beam shape prediction.

The three methods for a beam profile control were shortly outlined. The electrical method is described more in detail in a further short paper of this report. The use of the “zone grids” method was demonstrated on an example of a 7 zone grid system for an ECR source with 200 mm ion beam diameter. The advantage of this method is the production of nearly the same beamlet divergence over the whole ion beam diameter. The possibility of source clustering was demonstrated on the basis of two main existing single source properties—compact design and interference free and independent excitation control.

Finally, the interplay of ion source hardware development and process technology development in a good co-operation is the key to further success and the basis of modern ion beam technology performance.

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Direct processing of surfaces and thin films with micron and sub-micron precision using ultraviolet and ultrashort pulse lasers

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Introduction

In addition to applications in engineering laser material processing is as well of increasing industrial interest in the field of micron technologies for, e.g., scribing, drilling, welding, and surface patterning of bulk materials and thin films. However, regarding the materials used, the aim of processing, and lateral dimensions this applications call for laser processing techniques with a high precision, a high reliability, and minimal material alterations. Of course industrial applications require a high flexibility, high speed, and the easy control of the processes, too.

According to the current developments high precision, low damage, and high throughput laser processing can be achieved in particular with short wavelength (UV) and ultrashort pulse (fs) laser radiation. For the intended application both the machining tool—the laser beam—and the interaction processes with the material have to be controlled and optimised. However, material processing can be achieved not only by etching but also by modifying the near surface region.

Accurate pulsed laser processing requires for high-precision lateral and vertical resolution the confinement of the laser-matter interaction to the near surface region. Therefore the requirement of a high material absorption coefficient that is essential for material patterning using laser ablation processes is regularly achieved with UV photons by linear absorption or with fs laser pulses by nonlinear processes. In addition high surface absorption causes reduced pulse energies and therefore assists low damage material processing.

Since some of interesting materials, e.g., oxides and fluorides, are highly transparent at usual laser wavelengths different approaches for enhancing the laser absorption near the surface by means of additional irradiation (VUV light) or by means of additional material is currently under development. Especially the indirect laser processing techniques using additional materials allow low-

energy material processing with established lasers.

Micron and sub-micron structures

Laser-induced bumps

Regularly laser modification of absorbing materials causes alterations of composition, morphology, or structure of a near surface material volume. One example of the topographic effect of a laser modification process is the “swelling” of glass in consequence of laser irradiation forming bumps. The heights of such laser-induced bumps

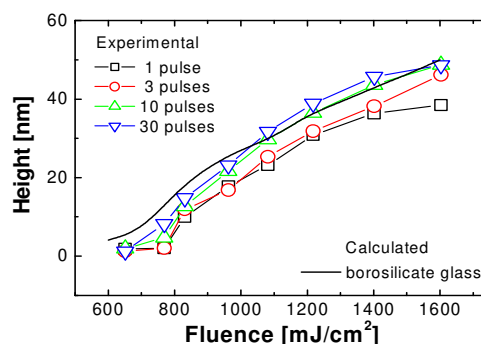


Figure 1: Measured height of laser-induced bumps onto borosilicate glass in dependence on the laser fluence for different pulse numbers and the expected height calculated from a thermo-mechanical model.

are shown in Figure 1 in dependence on the applied laser fluence for borosilicate glass using a KrF excimer laser ($\tau_p=25$ ns, $\lambda=248$ nm). The height of the bumps increases with the laser fluence up to the onset of laser ablation and can achieve 45 nm. The laser irradiation of the glass causes the heating of a near surface material above the softening point and the material expansion. After the pulse the fast cooling rates do not allow the complete relaxation of the glass and cause the freezing in the material expansion approximately at the glass transition point. The result of numerical calculations of the material swelling taking into account the laser heating, the thermal material expansion and the softening, and the phase changes of the glass is shown in Fig-

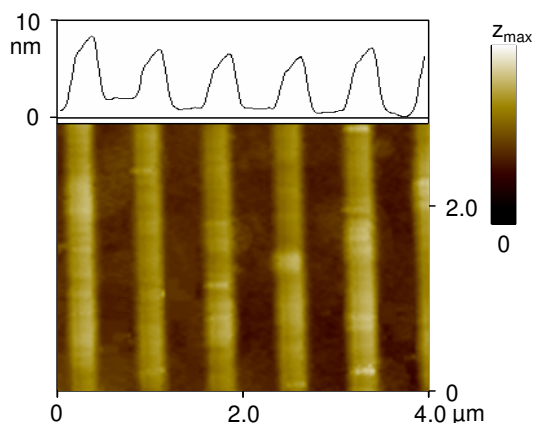


Figure 2: Sub-micron line gratings on borosilicate glass measured by AFM using tapping mode.

ure 1 in addition to the experimental values. The well agreement of the calculated and the experimental bump height confirms the validity of the model.

This laser-induced swelling process has been used for the fabrication of surface patterns on glass with only one laser pulse. In addition to micron-sized bumps sub-micron gratings with a grating period of ≈ 760 nm were generated as shown in Figure 2. The line grating features sharp line edges regardless a sinusoidal intensity distribution due to the interfering laser beams was used. Therefore the bump formation is a high contrast process that apparently improves the resolution of the whole laser patterning process.

Laser processing for nanostructures

Nanostructured surfaces and materials with their unique and extraordinary properties are of high interest for science and technology but can not be obtained directly by laser processing due to the

limited optical resolution achievable even with UV wavelengths. On the other hand the connection of nanostructures with microstructured surfaces is usually necessary to realise, e.g., electrical interconnections to micron and sub-micron patterns. With this goal laser-induced surface modification is studied in micron-sized areas for local generation of nanostructures.

The irradiation of surface coated silicon substrates with UV laser pulses of different wavelengths causes alterations of the surface morphology below the ablation threshold of the substrate. The example depicted in Figure 3 a) demonstrates the formation of nanoparticles upon ns UV laser irradiation of an iron nitride coated silicon surface. The lateral size of the particles formed inside of the laser exposed area depends on the laser fluence and the pulse number and ranges from 5 to 25 nm. Increasing the fluence above the ablation threshold either the film or the already formed nanoparticles can be removed in order to increase the patterns complexity further.

Such local excimer laser modified substrates were exploited for the stimulation of carbon nanotubes (CNT) growth in a thermal CVD process using an acetylene/nitrogen gas mixture. In Figure 3 b) a bundle of vertical aligned carbon nanotubes is shown. The well-defined CNT bundles have a base area according to the size of the laser spot ($100 \times 100 \mu\text{m}^2$) and feature very sharp edges. The inset of Figure 3 b) shows an enlarged view of the side wall of the bundle that proves the alignment of the nanotubes perpendicular to the surface. Because the individual nanotubes have certain chirality they touch and probably stick

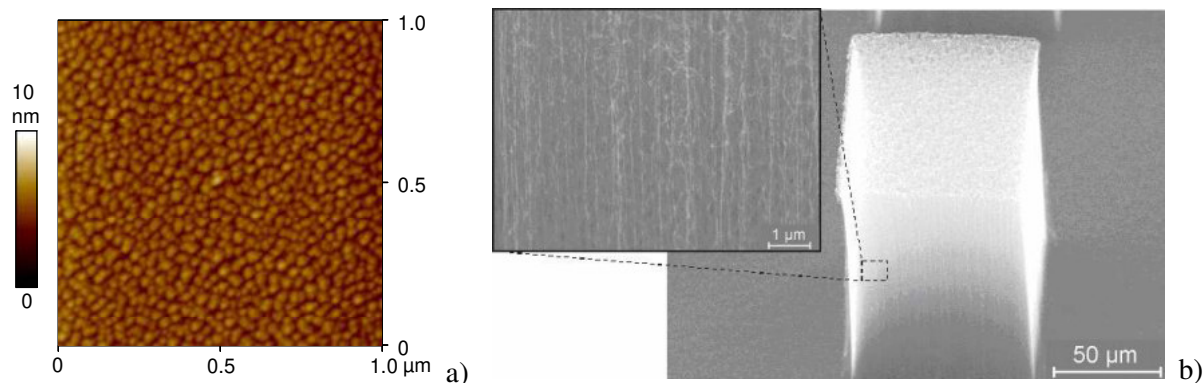


Figure 3: a) AFM image of a laser-modified surface having a dense film of nanoparticles. b) SEM image of a bundle of vertical grown carbon nanotubes. The local growth of the aligned nanotubes was attained only in the laser irradiated surface areas. Increasing the fluences beyond $\approx 1 \text{ J/cm}^2$ the particles are removed by ablation.

together due to van der Waals forces between adjacent CNT's. Hence, the individual nanotubes that prop up each other are forming a CNT network that stabilises the long nanotubes of the CNT bundles and therefore allows the growth of bundles with a height up to 150 μm within a growth time of about 5 min. The bundles consist of multi-walled CNT's with a diameter in the range from 10 to 30 nm as Raman and TEM investigations have shown. As known, the nanoparticles together with the growth conditions determine the properties of the nanostructures, for instance the diameter of CNT. Therefore, the variation of the nanoparticles properties by selecting appropriate laser processing parameters at different substrate areas might be used to adjust the nanostructures to the needs of applications locally and therefore probably allows the fabrication of arrays with specific CNT bundles.

Applications of laser processing

Etching of surfaces for micro-optical applications

Apart from the work on machining of refractive micro-optical elements using small laser spot direct writing techniques, grey scale and contour mask technique [1] here the laser machining of diffractive surface pattern is presented. The challenging requirements of micro-optical surface processing can be fulfilled to a large extent by indirect laser machining due to the low etch rates, the smooth etched surface, and the low contaminations achievable with this technique [2]. Because low roughness etching is achieved only in a

narrow fluence window the usage of grey scale techniques currently is ruled out and the etching of variable depth phase gratings for a HeNe laser beam shaping was performed by the more flexible direct writing technique. After process optimisation a reproducibility of the etch depth of better than 15 nm and roughness of less than 10 nm rms at multilevel test patterns were achieved. An example of a grating beam homogeniser and its optical effect is shown in Figure 4. The good consistence of the calculated and the measured grating depth distribution (0 to 350 nm) across the etched grating is shown in the upper part whereas the laser beam homogenisation due to the locally different grating depths that cause an equivalent phase shift to the laser beam is shown in the lower part. The remaining roughness of the grooves bottoms (≈ 10 nm rms) results mainly from the overlapping of adjacent laser pulses and can further be improved by exploiting specific projection masks with a partially gray scale design. Consequently backside etching is suited for direct writing of diffractive surface structures [3].

Laser scribing for electronic applications

The basic investigations on the low damage ablation of thermal sensitive semiconductors were continued for the interconnection of thin film CIS solar cells. Both the external and the integrated interconnection were achieved by exploitation the recent developed low-defect laser scribing technique. The current efforts aim to raise the speed and the precision of the scribing for highly efficient thin film solar cell modules [4].

Encouraged by the excellent quality of the scribing results, the trimming of resistors made by thick film technique was investigated. The scribing shows a high quality such as clean grooves, high stability, and low aging of the resistively. However, by laser irradiation with laser fluences below the ablation threshold a laser-induced modification of the films was observed which leads to a reduction of the resistance and can be used for trimming, too, but now towards lower resistance values.

Figure 5 shows the reduction of the resistance on the number of trimming scans onto the area of a thick film resistor. Both optical microscopy and

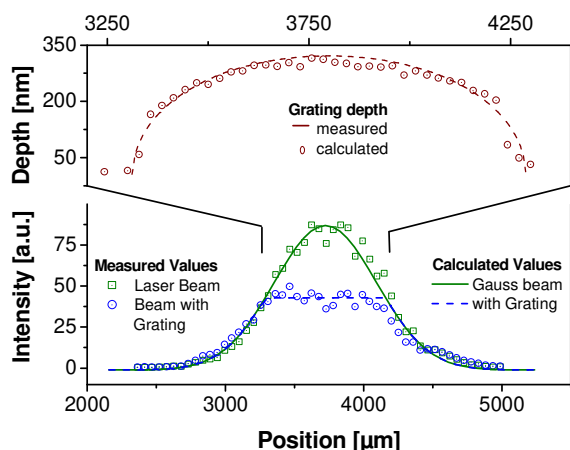


Figure 4: Depth distribution of a laser written variable depth fused silica grating and the optical effect for beam homogenisation.

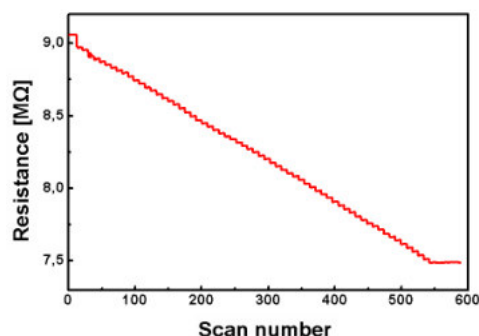


Figure 5: Reduction of the resistance of a thick film resistor on the number of laser exposures with a fluency below the ablation threshold.

SEM imaging show no visible alteration of the laser-processed surface. Therefore the trimming can be achieved in addition to ablative scribing by material modification without ablation and therewith coupled adverse processes such as crack formation or debris deposition. Using this new approach both the increasing and the reduction of the resistance upon laser irradiation can be achieved and allow new strategies in device trimming in the microelectronic technology.

Biomedical applications

Recently small sample and high throughput analytics of chemical and biological substances for lab-on-the-chip applications become reality. For these applications the surfaces have to be micro-machined as well as functionalised. The local surface functionalisation by means of laser processing for bioanalytical applications has been shown. The approach combines the large area functionalisation by means of silane chemistry (SAM), plasma polymerisation, or coating techniques with the local processing capabilities of laser machining. Additionally, such functionalized patterns can be used for the local and guided

growth of cells, respectively, that can be exploited for the growth of artificial tissue, the study of cell-drug interactions, or the investigation of nerve cell interactions in a controlled manner.

Due to the strict separation of functionalisation and micro patterning both steps can be optimised for the required functionalisation chemistry, the needed topography, and the used substrate material. Because the SAM functionalisation and its laser patterning can be applied repeatedly patterned surfaces with different chemical functionalities can be obtained. In the example shown in Figure 6 the degradation and therefore the patterning of UV transparent organosilanes films on borosilicate glass by excimer laser irradiation has been accomplished with only one laser pulse below the ablation threshold fluence. Therefore, a fast, reliable, and clean processing was achieved. Evidences for the substrate mediated, thermophysical nature of the patterning process has been deduced from the theoretical and experimental investigations of the laser-induced degradation process using different substrate materials as well as dissimilar laser wavelengths. Sub-micron resolution can be achieved because the optical resolution of the UV laser is practical not reduced due to the low thermal diffusivity of the glass.

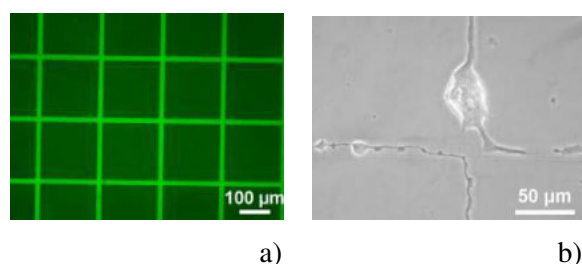


Figure 6: Fluorescence and phase contrast microscope image of a functionalized and laser microstructured sample and the local and guided growth of HB-12317 hybrid cells along the surface patterns.

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Novel aluminium carboxylate nanofillers for radiation-cured protective polymeric coatings

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Introduction

Early studies of the modifying effect of nanopowders on radiation-cured polymeric coatings were based on organometallic nanoparticles. Notwithstanding their pronounced modifying effect on viscoelastic data, inorganic nanopowders such as silica and alumina were used for practical extensions. By functionalising the latter nanoparticles in a heterogeneous hydrolytic condensation (HHC), an enhancement of surface mechanical properties was attained. In order to evade toxicological by-products and to improve the rheological application behaviour, the organometallic nanopowders were revisited. The latter are applicable to radiation curing of (meth)acrylic dispersions and also to thermal curing of two-component systems (epoxide/polyol, isocyanate/polyol).

In previous studies of the modifying effect of nanopowders on polymeric coatings and foils, organometallic species were employed such as cerium(IV)sorbate of obviously low practical relevance, and the focus was set on viscoelastic rather than on surface mechanical data [1]. For an enhancement of surface mechanical properties commercial inorganic nanopowders were used (first of all amorphous SiO_2 ; trade name AEROSIL). A polymerisation activity was imposed on the surface of these nanoparticles by grafting alkoxysilanes (trade name DYNASYLAN) onto them in a heterogeneous hydrolytic condensation (HHC) [2]. Notwithstanding the successful practical applications of the HHC route [3], its further development was urgently motivated: (i) The HHC nanodispersion could be processed by heated (typically 40 °C) roll application, however eluding application at room temperature incl. dipping and spraying. (ii) In addition to that, in the course of the acid catalyzed HHC detrimental alcohols are formed bringing about a marked transesterification of the respec-

tive monomer/oligomer nanodispersion. Combined autocatalytic HHC and Michael addition (MA, [4]²) with small amounts of aminosilanes (typically 2 wt.-% instead of 15 wt.-%) may largely remove these complications. Furthermore, an adsorptive particle organophilation (APO) also proved feasible on the presupposition that a monomer/oligomer fraction of adequate molecular mass (around 600 g/mol) forms part of the nanodispersion. The simplification to evade the formation of toxic by-products is potentially compensated by surface-active impurities which may override the APO principle [4]³. In addition, finer powders ($d_m < 20$ nm) are inapplicable in the APO route.

These environmental and rheological aspects led us to revisit organometallic nanopowders because the latter are organophil by preparation. In doing so, a literature concept [4]¹ is of relevance which bases on boehmite or pseudo-boehmite nanoparticles. After surface modification by carbonic acids, these carboxylate alumoxanes (together with counter-reactive groups) were dispersed in polymerisation-active substrates and subsequently thermally cured. The obtained nanocomposites revealed an enhanced flexural strength but a remarkable mechanical reinforcement did not occur. This can be traced back to the high concentration of the carbonic acids which involves a likewise high ligand concentration on the particles' surfaces. Obviously these both high concentrations have a softening effect on the polymeric surroundings. Thus a more intrinsic chemical structure of the organometallic nanoparticles appears highly desirable. In our route, a one-step precipitation furnishes the more intrinsic organometallic nanopowders in a quite direct and cost-efficient way with nearly quantitative yield.

An assessment of our novel organometallic nanofiller [4]⁴ was based on comparative preparations of nanocomposites using AEROSIL OX50 (SiO_2)

[3] and the non-intrinsic organometallic nanopowders [4]¹.

Preparations

Organometallic nanopowders

A basic aluminium maleate (Almal) was obtained nearly quantitatively in a one-step precipitation reaction from dissolved aluminium isopropylate with aqueous maleic acid solution (molecular ratio 2:1). Thermogravimetric analysis testified the final product to bis-aluminiumdihydroxymaleate. Scanning electron micrographs revealed on an average 100 nm particle aggregates of about 30 nm primary particles where the latter measure was derived by means of combined ultracentrifugation and dynamic light scattering. The ²⁷Al MAS NMR spectra (with the main line at 2.4 ppm) were typical of an octahedral surrounding. The vibration-spectroscopic characterisation of the Almal nanopowder as prepared (cf. Figure 1) is exceedingly informative: The respective FTIR spectrum reveals a wavenumber difference between the asymmetric and symmetric OCO stretching vibration bands of 118 cm⁻¹. This value speaks of a bidental ligand coordination. Furthermore, the corresponding FT-Raman spectrum in Figure 1 comprises the C=C stretching vibration band at 1660 cm⁻¹ which markedly differs from the corresponding acrylate value (1634 cm⁻¹), thus enabling a spectroscopic determination of the acrylic and maleic C=C conver-

sions.

In addition to Almal, analogous nanoparticles were obtained with ligands such as D,L-maleate (Almalat), citrate (Alcit), oxalate (Aloxal), and stearate (Alstear). For comparison, the corresponding FTIR studies on Aloxal and Almalat brought out much greater wavelength differences (277 cm⁻¹, 203 cm⁻¹, respectively) clearly evidencing monodental coordinations in the latter both cases. While the Almal nanofiller proved optimal for acrylates and epoxides, Almalat, Alcit, and Aloxal could readily modify the isocyanate/polyol system. Alstear proved advantageous for bulk modification of nonpolar polymers, e.g. polypropylene from an extrusion procedure. In the outlook, preparation of basic aluminium phosphates and phosphonates is envisaged to impose flame retardance on polyurethane foams.

The performance of all the intrinsic organometallic nanopowders could be further enhanced by the application of a mild tempering (24 h, 120 °C). This is probably thanks to a removal of adsorbed water from the nanoparticles and concomitant reduction of polarity and hardness increase.

The possibility is worth mentioning to transfer all the various aluminium carboxylate nanopowders as specific precursors to a more brute thermal treatment (oxidative atmosphere, 10 h, 1100 °C), yielding corundum nanopowders. Quite astonishing, macroscopic properties such as the mean density are specifically influenced by the ligands of the precursor nanoparticles. While the aluminium carboxylate nanoparticles proved amorphous in X-ray diffraction (XRD), the resulting nanocorundum particles revealed a rather perfect crystallinity and a smooth spherical surface morphology. In advance, we expect from these nanocorundum powders a very high synergetic action in nearly all nanomodification schemes.

The preparation of the non-intrinsic Almal reference compound according to [4]¹ (Almal-Cook) was done by a synthesis of nano-boehmite according to example 1 of the aforementioned USP and subsequent reaction with equimolar amount of maleic acid (instead of 4-hydroxybenzoic acid in example 2). While our Almal proved amorphous in XRD and reveals an intrinsic structure (homogeneous ligand distribution over the whole

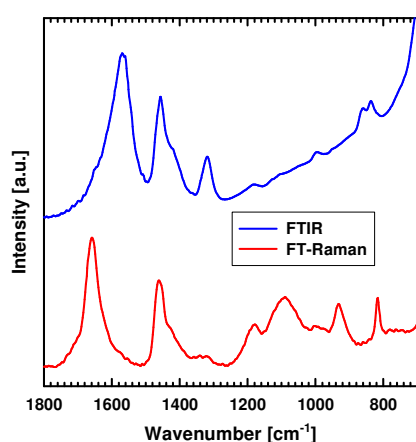


Figure 1: FTIR and FT-Raman spectra of the Almal nanopowder as prepared.

nanoparticle), Almal-Cook consists of the crystalline boehmite nucleus surrounded by the carboxylate ligand shell. The latter non-intrinsic structure with the exceedingly high organics concentration at the surface readily provides for organophil features but unfortunately also for a softening effect in the pertinent polymeric nanocomposites.

Reference nanodispersions

An acrylate mixture of hexafunctional aliphatic urethane acrylate (EB5129) and trifunctional polyether acrylate (SR454, mass ratio 1:2) was filled by 30 wt.-% Almal, using a dispermate (yielding Almal 01). Furthermore, a 1 h application of a high-energy attrition ball mill could readily implement perfect optical transparency. One can expect that the latter property can be achieved in a direct way by further optimising the original particle preparation route. Although dispensable in an Almal modification, small amounts (2 wt.-%) vinyl trimethoxysilane (VTMO) were added which compare to the much larger proportions in the SiO_2/HHC route (> 10 wt.-%). A gentle thermal treatment (5 min, 100°C) as accomplishable in curing lines by IR lamp arrays further improves the surface mechanical data through thermal post-condensation processes (both between particle surfaces and polysiloxane shell and within the latter shell itself). By just varying the organic matrix, five dedicated Almal-dispersions were prepared bringing about adapted rheology (roll application, dipping, and spraying), enhanced adhesion, and exterior durability. The components 30 wt.-% Almal-Cook / 2 wt.-% VTMO and

25 wt.-% AEROSIL OX50 / 12.5 wt.-% VTMO were admixed to the abovementioned acrylate system to obtain the reference dispersions Almal-Cook 01 and Carat (SiO_2/HHC).

Nanocomposite coatings on acrylate basis

The nanodispersion films were applied on paper utilising a doctor blade (slit width $50\ \mu\text{m}$) in the doctor blade coater Simex AF-3. In UV curing a Hg middle-pressure lamp was used ($120\ \text{W}/\text{cm}^2$; O_2 concentration < 200 ppm).

Characterisation

Acrylate-based nanodispersions

The organometallic lacquers reveal largely shear-independent low viscosities (cf. Figure 2), thus enabling roll (Almal 01, 02, 06), spray (Almal 07, 08), and dip applications at room temperature.

The rheology adaptation by just choosing an appropriate acrylic component forms an outstanding result which becomes exceedingly obvious in the light of the Carat data. Anticipating the poor surface mechanical parameters mediated by the non-intrinsic Almal-Cook filler, the pertinent rheological characteristics were omitted here.

Acrylate-based nanocomposite coatings

Abrasion tests (Taber Abraser, 2 CS-0 friction rolls covered by emery paper S-42, each friction roll charged by 500 g weights) furnish most significant surface mechanical data. Figures 3 and 4 clearly prove the superiority of the Almal route.

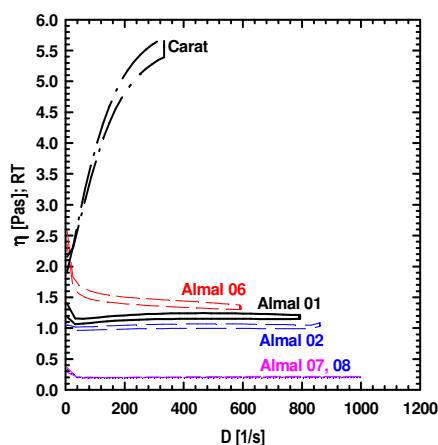


Figure 2: Rheological characteristics of Carat and various Almal dispersions at room temperature.

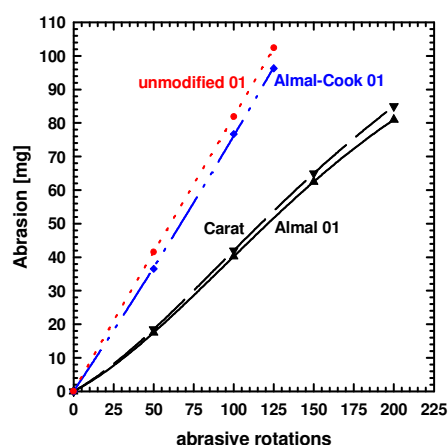


Figure 3: Abrasions for organometallic (intrinsic Almal 01, extrinsic Almal-Cook) and SiO_2 (*Carat) routes.

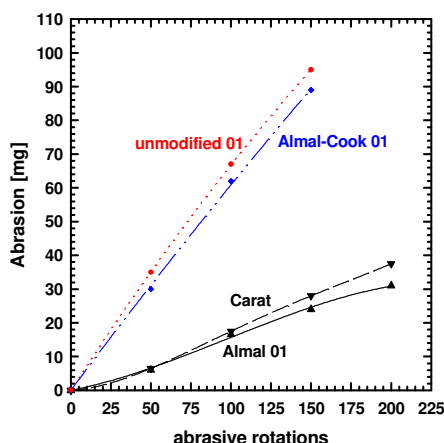


Figure 4: The same as in Figure 3 but now demonstrating the effect of post-condensation.

By the way, the synergetic nano/micro effect is worth mentioning if a microcorundum powder (e.g. 10 wt.-% round milled alumina L9 or the sharp-edged ZWSK F 600) is added to a respective nanoformulation. Of course, the decrease of abrasion owing to nano/micro modification (typically by a factor > 5) is highly appreciated but due consideration must also be given to the abrasive action of the micro-component on parts of the application plant. Here, however, besides round milled alumina the Almal-derived nanocorundum powder yields a positive alternative thereby even maintaining optical transparency.

The Almal enforcement becomes also manifest in pertinent findings obtained by related characterisation techniques. Thus, as compared with unmodified reference coating, the diamond scratching hardness increases by typically 2.0 N, the Martens indentation hardness by 70 N/mm², the storage modulus by 1.5 Pa·s, and, finally, glass transition temperature shifted by +30 K.

Summary

Novel organometallic nanopowders were obtained from an efficient precipitation route. These metal carboxylates tend to reveal open architectures [5] and, therefore, it appears hardly conceivable to attribute a greater hardness to them. Therefore, from the very beginning the active enforcement principle must be reconsidered.

The peculiarities of this innovative modification route also comprise the synergetic effects of a micropowder additive and a post-condensation, as expected. Surprisingly, these organometallic

nano-fillers are also amenable to nanomodification of two-component systems (epoxide/polyole and isocyanate/polyol) – corresponding work is under way (cf. the pertinent brief report by Wennrich et al.). As for polypropylene bulk systems filled by basic aluminium stearate nanopowder (Alstear) through extrusion (filler content below 5 wt.-%) we confine ourselves to just quoting an enhanced notched impact strength by as much as 40 % with the tensile strength kept unchanged. The exceedingly broad application field does not only comprise mechanical enforcement of acrylic and two-component coatings as well as bulk systems but, in addition to that, also focuses on quite different modification goals like flame retardance when utilising basic aluminium phosphates or phosphonates.

Acknowledgements

These investigations were supported by *Federal Ministry of Education and Research (BMBF)* within the projects 03N8015B and 01RI05026 as well as two feasibility studies within the centre of competence *CC UFS Dresden/Chemnitz*. The latter studies were co-financed by *Cetelon Nanotechnik GmbH & Co. KG Leipzig*.

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The transformation of PHPS to SiO_x was monitored by ATR-FTIR spectroscopy. Figure 1 shows some typical spectra. Figure 2 shows a REM photograph of a layer prepared by this method on a PET foil. It can be seen that the SiO_x layer formed consists of packed grains.

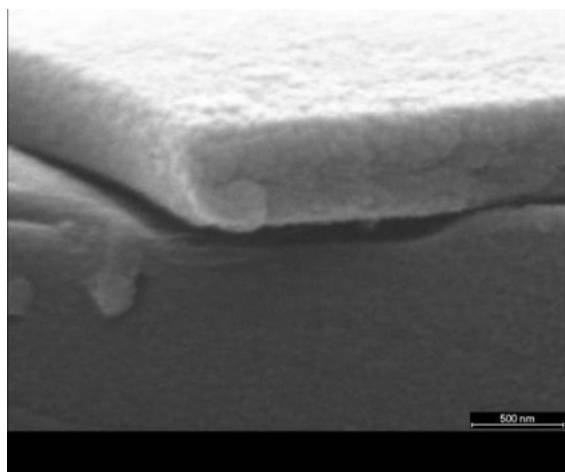


Figure 2: REM photograph of a PHPS coating transformed to silica by thermal treatment.

Using TOF-SIMS spectroscopy depth profiles of elements were gained. The diagram (Figure 3) shows largely homogeneous depth profiles of several ions consisting of the relevant elements. It should be mentioned that sensitivity differs between different ions and the detected ion currents do not allow direct inference with the concentration of the elements.

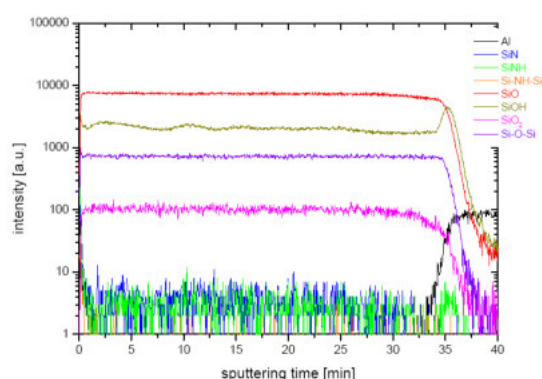


Figure 3: TOF-SIMS diagram of a SiO_x -layer generated by thermal treatment (2h 180 °C) of a PHPS (NP110) layer on aluminium foil.

ESCA measurements show that the percentages of elements present on the surface are: oxygen 57.7 %, silicon 31.8 %, nitrogen 5.0 %, and carbon 5.5 %. Apart from carbon which probably

originated from the added catalyst, the empirical formula SiO_xN_y with $x+y=2$ is fulfilled, however indicates some remaining polysilazane structures.

Applying differential scanning calorimetry (DSC), one can see that after 3 hour treatment at 60 °C the transformation process is not completed and even after 3 hours at 180 °C some reaction enthalpy is detectable (Figure 4). This is in accordance with the ESCA measurement where a remaining quantity of nitrogen was detected.

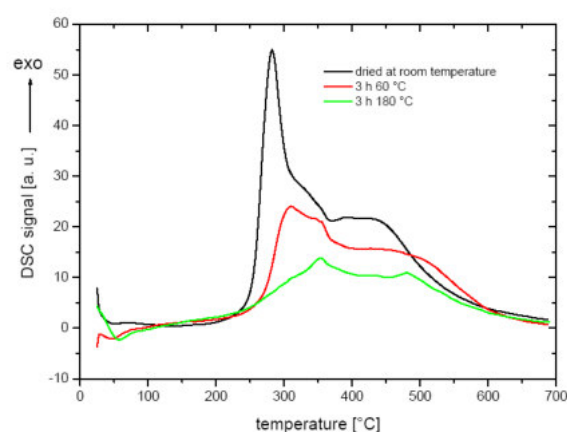


Figure 4: DSC diagram of powder obtained from 3% solution NP110 on glass dried and optionally heated.

By coating such solutions onto 36 μm PET foils the oxygen transmission rate (OTR) and the water vapour transmission rate (WVTR) were improved significantly as shown in Table 1 for OTR.

Table 1: Transmission rates for oxygen and water vapour of PHPS based coatings.

sample/treatment	OTR [cm ³ m ⁻² d ⁻¹ bar ⁻¹]	
	80 °C	180 °C
36 μm PET	34	
3 % NP110 1 week at 20 °C	17.8	
3 % NP110 1 h at T	1.7	0.7

However, extensive research on the effect of a variety of catalysts focussing on the acceleration of the transformation process of PHPS and on decreasing temperature was not really successful. Even at high temperatures of about 200 °C

approx. 10 minutes were necessary in order to achieve the complete transformation of PHPS into SiO_x [1, 2].

Large scale production processes require processes that are, as a minimum, one order of magnitude faster. In addition, coating of thermally sensitive substrates needs to be accomplished in both packaging and electronic industries. This leads to a demand for process temperatures below 80 °C.

A potential solution has been proposed by quantum chemical calculations suggesting the possibility of photolytic Si-N bond scission schematically shown in Figure 5.

The calculated threshold energy of 5.66 eV is related to a UV photon with a wavelength of

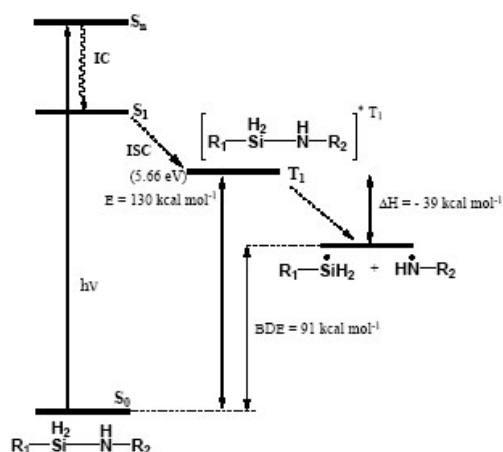


Figure 5: Energy scheme of the hypothetical molecule $\text{R1-SiH}_2\text{-NH-R2}$ after photoexcitation.

220 nm. Accordingly, irradiation with UV light with a wavelength below 220 nm should be able to initiate the transformation process of PHPS.

The result of transmission measurements on 100 nm layers of PHPS before and after thermal treatment in the deep UV/VUV region using synchrotron radiation (PTB-BESSY) is in good agreement with calculations. Radiation about 200 nm is mainly absorbed by the layer. Hence, the irradiation energy is not wasted into the substrate. Otherwise, the penetration depth of the photons is in the order of the layer thickness and the absorption occurs not only in a thin surface layer. Industrially available radiation sources in this range are low pressure mercury lamps with an emission of 185 nm and excimer lamps with emissions of about 172 nm and 222 nm.

Based on these considerations, research in collaboration with Clariant GmbH on the VUV stimulated oxidation of PHPS films into a thin flexible silica layer has been started focussing on the production of coated PET foils from roll to roll in the speed range of 10 m per minute.

UV-curable coatings with layered silicates

Dust-based defects and micro cracks in barrier coatings based on vacuum and hydrolysis/condensation processes are one of the most significant drawbacks [3]. Therefore, the filling of

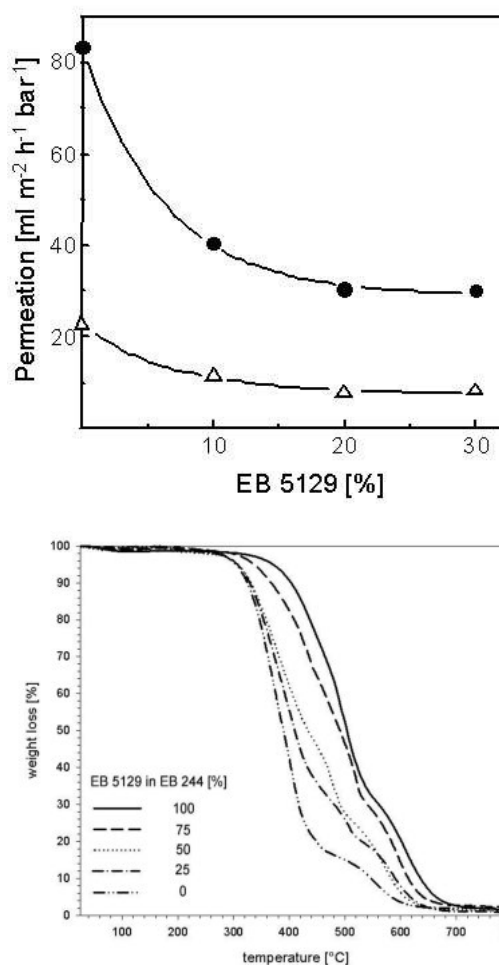


Figure 6: Top: Permeation of oxygen (●) and nitrogen (Δ) through a crosslinked acrylate layer on PE in dependence of the amount of EB 5129 in an aliphatic bifunctional acrylate EB 244. OTR at 0 % relative humidity. Bottom: Thermogravimetric data for different blends of EB 244 and EB 5129.

these dust-based defects with a suitable coating is a promising strategy for the further reduction of gas permeability. Encouraged by previous studies based on EB-cured barrier polymers [4] we have engineered the barrier properties of selected UV-

curable systems by the use of nano-scaled layered fillers. In this context, we focused on UV-curable acrylates and cycloaliphatic epoxides which were selected by the “Permachor”-principal [5].

Besides of specific functional groups, the polymer network density exemplary adjusted by different mixing ratios of the bifunctional acrylate EB 244 (UCB) and the hexafunctional acrylate EB 5129 (UCB) influences the barrier properties of the final films (Figure 6). Thermogravimetical data support these findings. However, a high content of EB 5129 resulted in high E-moduli. The coatings lost their flexibility and became brittle and unsuited for barrier applications.

It is well known that barrier properties of coatings may be improved by the addition of impermeable layered silicates where the extent in one dimension is in the nanometer range [6]. In addition, improved tensile properties, thermal stability, and resistance to swelling by solvents were observed in different layered silicate nanocomposite systems. Due to the chemical structure of both the flakes and the polymer and the processing conditions the arrangement of the layered silicates in the polymer matrix ranges from tactid over intercalated to delaminated structures. Moreover, the size and the shape of the silicates as well as the content of impurities differs which influences the final properties of the film. Therefore, the screening and adjustment of a matching polymer-nanocomposite system was necessary for achieving the desired properties. This has already been demonstrated for many polymer-nanocomposite systems but was rather poorly investigated for UV-curable systems. In order to generate intercalated or exfoliated polymer-silicate structures a general approach was found in the substitution of the inorganic cations between the silicate-layers by organic alkylammonium ions. This procedure adapted the clay surface polarity to the polarity of the polymer and thus expanded the gallery between the sheets which enabled the polymer to penetrate between the sheets of the silicate. However, the use of these so called organoclays resulted in an inhibition of cationic UV curing. In addition, viscosities increased dramatically already at low loading levels which complicated the application of these formulations. A positive

influence on the barrier properties at an applicable loading level was not observed.

In order to circumvent these problems layered silicates, particularly muscovites, and talcum were used for the reduction of the permeability of coatings for oxygen without substituting the inorganic cations between the layered sheets. Using these systems the permeation of oxygen was successfully reduced by a factor of 3-5.

Finally, based on these principal investigations an oxygen transmission value of an UV curable system below $10 \text{ cm}^3/(\text{m}^2 \text{ d})$ for an applicable film thickness was attained. The arrangement of the layered silicate in such a coating is shown in Fig-

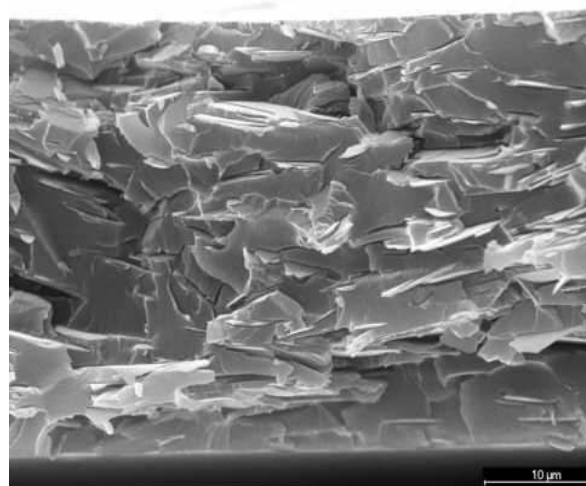


Figure 7: Scanning electron micrograph of the arrangement of MICA-sheets in a UV cured formulation.

ure 7. The low viscosity as well as the good adhesion to plastics enables an application of these formulations in form of spray coatings for 3-dimensional work pieces as well as for barrier adhesives in laminates [7].

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Production of functional coatings: Solid-phase extraction materials for trace analysis

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Introduction

Functional coatings comprise a wealth of applications, from mechanical protection against scratches and abrasion to chemical protection like stain resistance and corrosion protection. In recent time, the tasks assigned to and tackled by functional coatings grow ever further: transparent gas barriers are sought-after, optical properties such as gloss or refractive index must be met.

Solid-phase extraction

Solid-phase extraction (SPE) is a versatile method of sample preparation for trace analysis. The method is derived from classical solvent extraction using a polymer as solvent. The polymer is stirred in the aqueous sample, in the example shown later from a polluted creek, and the pollutants (analytes) are equilibrated between the aqueous phase and the polymer depending on their relative solubility. The diffusion of molecules into polymers is not very fast so that the diffusion lengths achieved within the time span of a laboratory extraction (in the order of minutes to hours) are limited to a fraction of a millimetre. For this reason it is favourable to use a coating on an inert support instead of a chunk of material.

Solid-Phase Micro Extraction SPME was developed in the late 1980es [1]. This method uses coated fibres or capillaries quite similar to GC-capillaries as enrichment phase. The rather straight-forward adaptation of GC-phase knowledge to SPME has fostered the rise of this method. The very small active phase volumes though result in limited performance in trace analysis. As a consequence, around the turn of the century the Stir Bar Sorptive Extraction SBSE was developed [2] increasing the active volume from 0.5 μL for SPME to 25-250 μL in SBSE.

While in SPME a range of fibres are available covering unpolar and polar analytes, in SPE only PDMS-phases are state of the art. PDMS works extremely well with unpolar analytes, but fails with more polar ones which in SPME are covered by polyacrylate phases.

Requirements for SPE-materials

The coatings are envisaged for use in thermodesorption-GC/MS. In this method analytes are desorbed from the loaded coating by means of a nitrogen stream at elevated temperatures, typically 250 °C. The coating thus has to fulfil the following requirements:

- high extraction yields at room temperature,
- stability against thermal decomposition at 250 °C (in inert atmosphere).

High extraction yields are correlated with low chain-chain interactions (ease of diffusion), while thermal stability requires strong interactions. Thus, classical thermoplastic materials cannot fulfil both requirements. The commercially used SPE material, PDMS, shows the way: A strong, moderately cross-linked main-chain for temperature stability combined with weak side-chain interactions for ease of solute inclusion.

Production of Coatings

In the science of coatings technology and chemistry are intertwined, superior results can only be obtained by mastering both. Levelling is an example for this interweavement: perfectly smooth surfaces are only obtained by using a formulation that is adapted to the substrate and to the application/cure technology. Quite trivially do different sample sizes require different technologies. At the IOM various technologies are established covering the entire scale-up chain from microscope slide size for expensive coatings via beer mat-

sized lab scale coatings for the determination of haze, scratch, and abrasion resistance to pilot scale roller coaters.

Surface magnification by micro folding

In contrast to the conditions encountered with PDMS recoveries obtained with polyacrylate SPE materials do not scale with the phase volume but rather with the surface area. The effect may be caused by slow diffusion in the polar material. Two methods for surface magnification were tested: the manufacture and use of flat strips and the application of micro wrinkles produced via monochromatic VUV-irradiation. 172 nm-photons selectively generate radicals in the top layer only. The thin cured layer swims on its own liquid precursor and the interplay of shrinking tension vs. viscous drag creates a unique wrinkle pattern (Figure 1).

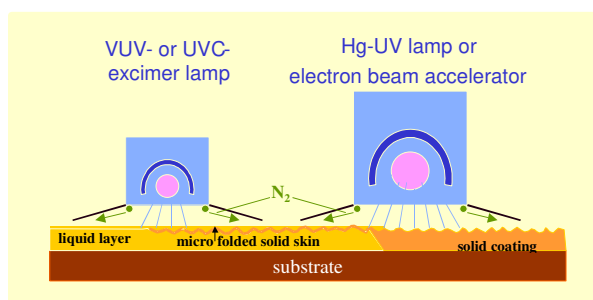


Figure 1 Principle of surface magnification via micro folding. The wrinkled surface is fixed with a second irradiation step (depth cure).

The method is described in depth in another article in this publication and in [3].

Coating machines

The efforts put into validation and verification of a novel material would be futile unless there is a method of assuring a constant quality production. A long term focus of the IOM is therefore the scale-up of coating processes using reel-to-reel coaters. A Krönert machine (“Labcoater I”) is available running web speeds from 5 to 200 m/min at a working width of up to 450 mm, equipped with a corona pre-treatment, a three-reel coater (gravure and flexo), a 150 kV, 50 mA linear electron accelerator LEA, a 120 W/cm Hg-Arc, 308 nm-, 222 nm-, and 172 nm-excimer lamps, each producing 50 W/cm. All curing devices can be flushed with N₂ (< 50 ppm O₂). The

machine is also equipped with an infrared heater and a lamination station, but for drying solvent or water based coatings the second “Labcoater II”, a five-roll EHA machine, is used.



Figure 2: Thick acrylate coatings crack after heating to 250 °C when applied onto a glass rod due to differing thermal expansion coefficients. The magnet inside the glass rod is left with some expansion space for the same reason.

Special Coating Procedures used for SBSE

The thermodesorption units in trade are manufactured to suit the format of SBSE, i.e. comparatively short, compact stir bars. A typical unit uses glass tubes with an inner diameter of 4 mm and a 60 mm long heat zone. In order to produce coatings adapted to these conditions glass-encapsulated magnets were fixed in the centre of a silicone hose and the remaining rim filled with acrylate. After curing the magnet has an 0.5 mm thick acrylate jacket much like the PDMS-jacket of the commercial SBSE-Twisters®. Figure 2 shows their appearance after heat treatment.

In order to overcome the thermal expansion problem and yet provide the acrylate with some mechanical stability at 250 °C glass fibre fabrics were chosen as skeleton. In this case, the fabric was laid on a polyethylene foil, soaked with the acrylate monomer formulation, covered with another polyethylene foil and the superfluous liquid squeezed out by means of a cylindrical steel bar. After curing the sandwich in the UV tunnel the polyethylene foils can be pulled off without damaging the composite leaving a smooth surface. By using structured lamination foils structures could be replicated into the glass fibre composite. In order to obtain more rigid samples polyimide foils

can be used as support with the acrylate being coated on both sides.

Analytical Procedures

Reagents and materials

An EPA phenolic standard consisting of seven phenols (phenol, 2-chlorophenol, 2,4,-dimethylphenol, 2,4-dichlorophenol, 4-chloro-3-methylphenol, 2,4,6-trichlorophenol, pentachlorophenol) with a concentration of 500 mg/dm³ of each phenol and alpha-hexachlorocyclohexane, delta-hexachlorocyclohexane, and hexachlorobenzene standards were obtained from Supelco (Bellefonte, PA, USA). Methanol, acetone, and sodium chloride were purchased from Merck (Darmstadt, Germany). The commercial stir bar Twister[®] for sorptive extraction was provided by Gerstel. It consists of a 10 mm length glass-encapsulated magnetic stir bar onto which a 0.5 mm thick PDMS tube (22 µg = 25 mm³) is mounted. Prior to first use, the stir bar was conditioned 16 h at 250 °C with a nitrogen stream of 30 cm³/min.

Extraction Procedure

All the studies to optimize the extraction procedure were performed using 50 cm³ aqueous sample spiked at a concentration level of 10 ng/cm³ of each compound. To perform the extraction under the optimal conditions the sorbent media was introduced into a 50 cm³ flask containing 50 cm³ of a water sample saturated with NaCl and submitted to a stirring speed of 1500 rpm for PDMS Twister or to a shaking speed of 500 min⁻¹ for PA strip for an extraction time of 4 h at room temperature. After that, the Twister or PA strip was removed from the aqueous solution with tweezers, rinsed with purified water, dried with a lint-free tissue, and inserted into an appropriate Gerstel thermal desorption glass tube (187 mm length x 4 mm inner diameter).

For determination of recoveries, a plug of pesticide-grade glass wool (Supelco, Bellefonte, PA, USA) was placed inside of an empty Gerstel thermal desorption glass tube. One end of the plug was sealed with a metallic gauge stopper for thermal desorption tubes (Gerstel). The tube was then spiked with 1 mm³ of a standard solution containing the selected compounds and connected

to a cold nitrogen stream (30 cm³/min) for 1 min to allow evaporation of the solvent. The tube was immediately transferred to the thermal desorption device for subsequent analysis.

Instrumental

Thermal desorption GC-MS of the selected compounds sorbed on the Twisters[®] and strips was performed on an Agilent system (Agilent Technologies, Palo Alto, CA, USA) coupled to a Gerstel TDS A thermal desorption device. A cold injection system (CIS) using liquid nitrogen as a coolant consisted of an empty liner for cryofocusing the analytes prior to introduction into the capillary column.

The optimized conditions utilized for the thermal desorption system were as follows: desorption temperature, 250 °C; desorption time, 5 min; and helium flow rate, 100 cm³/min (solvent vent mode). Both transfer lines, situated between the thermal desorption system and the CIS, and between the GC and the MS detector, were set at 250 °C.

The method utilized for the cold injection system was as follows: during thermal desorption, temperature set at -20 °C; heating at a rate of 10 K/s to 250 °C (hold for 2 min); the injector was used in splitless mode with a splitless time of 1.5 min.

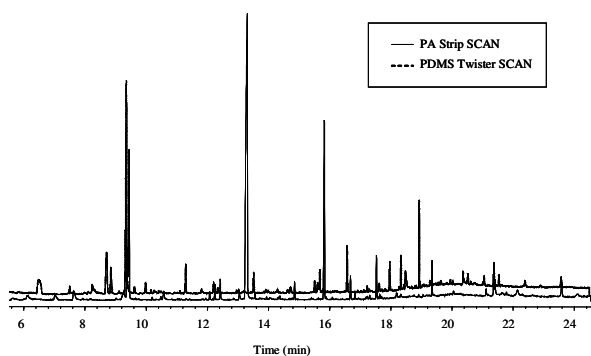


Figure 3: Background scan of a PDMS Twister[®] vs. a PA strip. In both cases a flat background is obtained spiked with some characteristic deterioration products. An HP-5ms capillary column (30 m x 250 µm i.d., 0.25 µm film thickness) was used with a GC oven program from 50 °C (2 min) to 200 °C at 10 K/min, and to 270 °C (5 min) at 25 K/min. Helium was used as carrier gas with a flow of 1 cm³/min. A detection method using single ion monitoring (SIM) mode which considered two characteristic ions for each compound was estab-

lished for detection. The characteristic ions for each studied compound are shown in Table 1.

Table 1: Octanol/water partition coefficients (K_{ow}) and m/z of the analytes.

Compound	Ions (m/z)	log K_{ow}
Phenol	66, 94	1.46
2-Chlorophenol	64, 128	2.15
2,4-Di-Me-Phenol	107, 122	2.3
2,4-Di-Cl-Phenol	63, 162	3.06
4-Cl,3-Me-Phenol	107, 142	3.1
2,4,6-Tri-Cl-Phenol	196, 198	3.69
α -Hexa-Cl- C_6H_6	181, 219	3.8
Hexachlorobenzene	284, 286	5.73
Pentachlorophenol	266, 268	5.12
δ -Hexa-Cl- C_6H_6	181, 219	4.14

Analytical Results

Qualifying a novel material for use in trace analysis requires a thorough testing, in the following only the most important achievements are reported. Details will be published soon [4]. First we will report the results with the commercial Twister[®] as reference (Table 2) and compare them to the results with our PA strips (Table 3). Extraction efficiencies were determined vs. analytes directly desorbed from glass wool.

Table 2: Figure of merit for the compounds studied using PDMS Twisters[®]. The repeatability is given as relative standard deviation $\sigma_{n-1}/\bar{x}$.

Compound	σ_{n-1} , %	LOD ng/dm ³	EE (%)
Phenol	10.5	435	1.3
2-Chlorophenol	5.2	196	3.8
2,4-Di-Me-Phenol	2.8	41	2.7
2,4-Di-Cl-Phenol	1.8	9.7	11.6
4-Cl,3-Me-Phenol	4.6	107	2.2
2,4,6-Tri-Cl-Phenol	2.2	7.0	40.7
α -Hexa-Cl- C_6H_6	3.8	1.4	95.1
Hexachlorobenzene	4.9	0.2	83.7
Pentachlorophenol	4.0	14	84.5
δ -Hexa-Cl- C_6H_6	3.7	2.0	86.0

As evident from Table 3, the PA strips outperform the PDMS Twisters[®] in enrichment of polar compounds and close up to them in unpolar compounds, thus being a true alternative.

Table 3: Figure of merit for the compounds studied using the PA strips. The repeatability is given as relative standard deviation $\sigma_{n-1}/\bar{x}$.

Compound	σ_{n-1} , %	LOD ng/L	EE (%)
Phenol	19.2	25.4	6.9
2-Chlorophenol	6.4	11.2	7.7
2,4-Di-Me-Phenol	5.9	3.5	13.1
2,4-Di-Cl-Phenol	6.0	0.54	39.8
4-Cl,3-Me-Phenol	7.7	15.3	23.9
2,4,6-Tri-Cl-Phenol	5.8	0.26	69.3
α -Hexa-Cl- C_6H_6	8.3	4.00	98.5
Hexachlorobenzene	6.2	0.11	96.9
Pentachlorophenol	11.7	1.9	91.3
δ -Hexa-Cl- C_6H_6	17.9	14.3	86.1

Furthermore, and somewhat to our surprise, as monomers in technical quality were used without further purification, the pretreatment chosen (16 h at 250 °C in a nitrogen stream) effectively removes residues that could bleed during thermodesorption. The background noise is as low as the one from PDMS.

Conclusion

The results shown here pave the way to introducing polyacrylate materials into the realm of SBSE in the near future. A first manuscript is in preparation [4], a grant application is filed together with two industrial partners.

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Influence of deposited layer at laser backside etching of fused silica

R. Böhme, K. Zimmer

The indirect laser processing of transparent materials offers a number of advantages compared to laser ablation like low threshold fluences, etch rates in the nanometer range, minimal material alterations, and low roughness. Due to this attributes high accurate surface patterns for, e.g., applications in micro-optics, are attainable but the underlying interaction processes are complex and not well understood. The indirect laser processing techniques LIBWE (*laser-induced backside wet etching*) [1] and LESAL (*laser etching at a surface adsorbed layer*) [2] make use of the laser absorption at the materials backside by means of organic liquids and adsorbed hydrocarbon layers, respectively. This intensive energy deposition near the interface of the solid and the absorbing medium causes the observed etch processes.

The proposed etch mechanism comprises a sequence of fast surface heating by the hot additive up to the melting/softening point followed by mechanical removing of the heated/softened surface region due to high pressures/stresses. On the other hand the regularly observed incubation effects and the analytically measured surface alterations give evidences for laser-induced surface modifications. Especially the deposition of hydrocarbon layers due to decomposition of the used organic absorbers has been detected for LIBWE as well as for LESAL [1-3].

This carbon-containing modified surface probably

enhances the laser absorption, affects the materials interaction at subsequent laser pulses, and alters the etch mechanism but in a different manner for LIBWE and LESAL.

Therefore the influence of the confinement to the backside etching was studied by backside ablation rate measurements of the of a 20 nm thick carbon layer on fused silica at two different ambient inert media without absorption that are gas (air) and liquid (water). In Figure 1 the measured etch depth after one laser pulse is depicted in dependence on the laser fluence in comparison for water and air. The confinement influences the etch rate and the laser fluence dependence of the etch rate significantly while the threshold fluence is similar. In the case of air rate saturation similar to LESAL [2] occurs whereas with liquid as backside medium a linear growing rate typical for LIBWE at moderate fluences [1] can be observed.

The less dense air permits a faster expansion of the ablation plume than in the case of water confinement and consequently affect the interaction time and interaction strength of the hot plume with the fused silica surface. The differences in the laser-plume-substrate interaction [4] cause the observed rate saturation at weak interaction (air) and the linear growing etch rate at strong interaction (water). Thus, the different backside media affect the thermal and the mechanical confinement of the etching processes that influence the dynamics of the interaction process and must be considered in developing a suited model for backside etching processes.

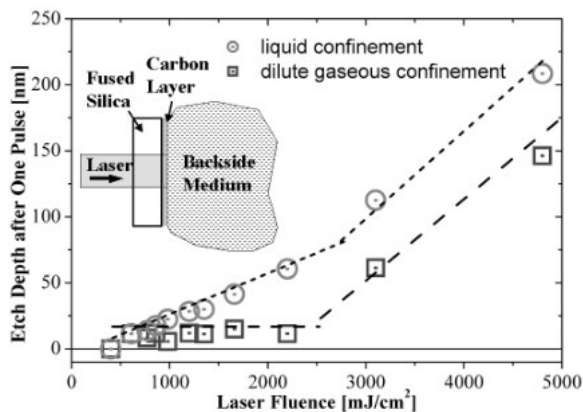


Figure 1: Etch depth of fused silica in dependence on the laser fluence after one-pulse treatment of solid/layer interface in liquid and air confinement.

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Ion beam assisted molecular beam epitaxy of high-quality m-plane oriented gallium nitride thin films on lithium aluminate substrates

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Recent research at the IOM proved that ion beam assisted molecular beam epitaxy (IBA-MBE) is well suited to deposit thin epitaxial wurtzitic gallium nitride (w-GaN) films of high crystalline and optical quality on 6H-SiC(0001) [1]. This originates in the additional energy input of the hyper-thermal nitrogen ions into the film surface during deposition, leading to a ballistic enhancement of the adatom surface mobility.

C-plane oriented w-GaN, that became prominent in the last years as base for highly efficient blue light emitting diodes and laser diodes, is polar along the growth direction. This may lead to electric polarisation effects interfering with the electronic device performance. Thus, w-GaN films with a non-polar growth direction are of large interest. Non-polar, typically a-plane oriented w-GaN films deposited on r-plane Al_2O_3 exhibited the problem that a high nitrogen ion/gallium atom arrival ratio (I/A ratio) resulted in heavily tilted c-axis oriented w-GaN domains [2]. A higher film quality due to lower lattice misfit was shown for m-plane oriented GaN grown on the (100) plane of lithium aluminate ($\gamma\text{-LiAlO}_2$) [3].

Optimisation of the quality of non-polar m-plane oriented w-GaN films deposited by IBA-MBE at the IOM was accomplished by variation of the I/A ratio in particular. A substrate temperature of

700 °C was found to be optimal. The comparison of N-rich ($I/A > 1$) and Ga-rich ($I/A < 1$) deposition showed that the crystalline and optical quality of films deposited under slightly Ga-rich conditions is higher than under N-rich conditions. $I/A \gg 1$ resulted in the presence of polar, c-plane oriented w-GaN domains, $I/A \ll 1$ resulted in droplets of surplus gallium at the film surface.

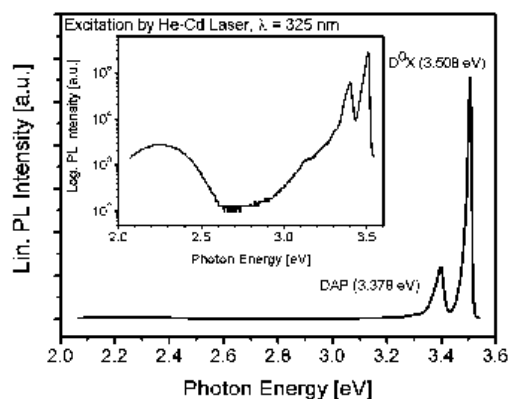


Figure 2: Photoluminescence spectrum of an m-plane oriented IBA-MBE GaN film on LiAlO_2 . Note the strong and narrow (20 meV) near-bandgap transition signal at 3.508 eV.

X-ray diffraction measurements and high resolution electron microscopy demonstrated the high structural quality of the optimised films (Figure 1), whereas the high optical quality of the films was revealed by photoluminescence spectroscopy (Figure 2) [4]. However, these films are characterised by high, anisotropic, compressive mechanical stress of several GPa due to largely different thermal expansion coefficients of film and substrate [5].

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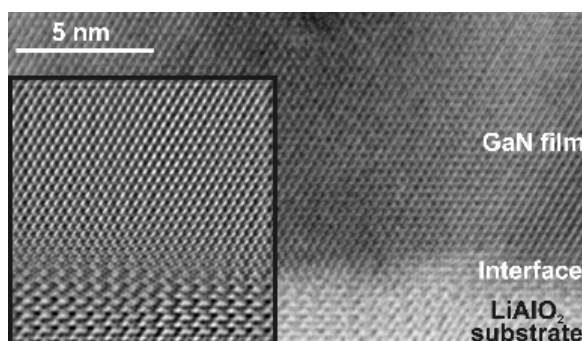


Figure 1: High resolution electron micrograph with zone axis w-GaN[0001] of an m-plane oriented IBA-MBE w-GaN film on LiAlO_2 . The framed region was filtered to emphasise the high crystalline order.

Self-organised nanostructures by ion beam erosion

B. Ziberi, F. Frost

Particle beams of low-energy ions or atoms are particularly suitable tools for ultraprecise processing of any arbitrary surface. Due to the ion bombardment material is eroded from the surface reminiscent of the sandblasting of surfaces or the erosion by water and wind. Beside the intended removal of material the surface topography is modified by the ion bombardment, as well. The surface evolution during ion erosion is effected by numerous atomistic mechanisms. Depending on the dominating process a large variety of erosion forms and patterns can be developed. Thus the interplay of surface erosion and surface diffusion give rise to self-organization, i.e. pattern formation for conditions far away from equilibrium, and causes well-ordered nanometer-sized structures. Recently, it was observed that self-organised, ordered hexagonal or square dot patterns can evolve from low-energy ion sputtering of III/V semiconductor surfaces under normal incidence, or under oblique incidence and simultaneous sample rotation initiating new interest in this alternative approach for surface nanostructuring [1].

In the last two years, our main focus was set on the pattern formation on Si and Ge surfaces during low-energy noble gas ion beam erosion [2-4]. In particular, it is demonstrated that (i) complex pattern formation processes do arise during erosion of Si and Ge surfaces and (ii) remarkably high ordered dots as well as ripple patterns with structure sizes below 50 nm can be obtained by

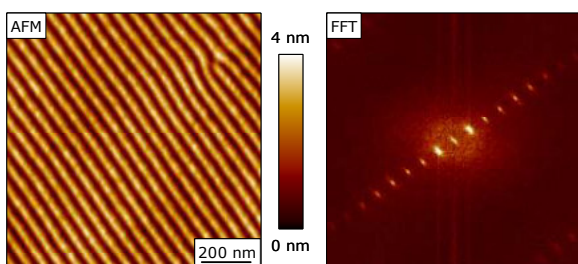


Figure 1: Nano ripples on Si with a periodicity of 50 nm generated by 1200 eV Kr^+ ion beam erosion under 15 deg ion incidence. Left: surface topography measured by AFM; right: Fourier transformed height profile.

choosing appropriate ion beam conditions for both surfaces.

Two examples of generated nanostructures are shown in the figures. In the first example small wave-like structures were produced by oblique ion bombardment of Si wafer surfaces. In the second example ordered nanometer-sized dots also on Si surfaces were formed. The ordering of these structures increases with ion fluence, leading to remarkably well ordered patterns. The size and the geometrical shape of this structures can be adjusted by the ion beam and sample parameters.

Due to the application of qualified broad-beam ion sources, homogeneously patterned, large-area surface modification can be accomplished. Up to now, Si wafers with diameters up to 200 mm were processed. The simple and cost efficient one-step fabrication process ensures an extraordinary application potential for the generation of sub-wavelength nano-optic components or as templates for the growth of nanostructured functional thin films.

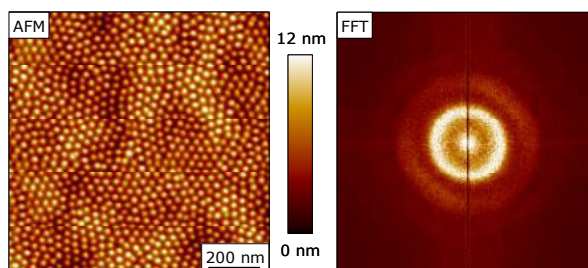


Figure 2: Hexagonal ordered nanodots on Si with a periodicity of 30 nm generated by 500 eV Ar^+ ion beam erosion under 75 deg ion incidence with simultaneous sample rotation. Left: surface topography measured by AFM; right: Fourier transformed height profile.

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Ion beam assisted deposition of chiral sculptured thin films

E. Schubert, B. Rauschenbach

Nanostructures with complex geometries 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 [1].

In our work the particle flux is provided by ion beam sputtering and reaches the substrate under an extremely oblique angle-of-incidence (typically 85 deg respective to the normal). In this deposition configuration, highly porous sculptured thin films (STF) are created, which consist of slanted amorphous silicon needles with a diameter from 20 nm to 50 nm, whereas competitive growth mechanism due to geometric shadowing determine the nucleation and growth processes (Figure 1). By applying an appropriate substrate rotation during growth the nanostructure geometry can be tailored. Chevrons and square spirals (Figures 2b and 2c) are created with a symmetric stepwise substrate rotation of 180 deg and 90 deg, respectively. The fabrication of circular spirals, screws (Figure 2a) and vertical posts is realised by a constant substrate rotation, and the nanostructure geometry depends on the ratio from deposition rate to substrate rotation speed [2].

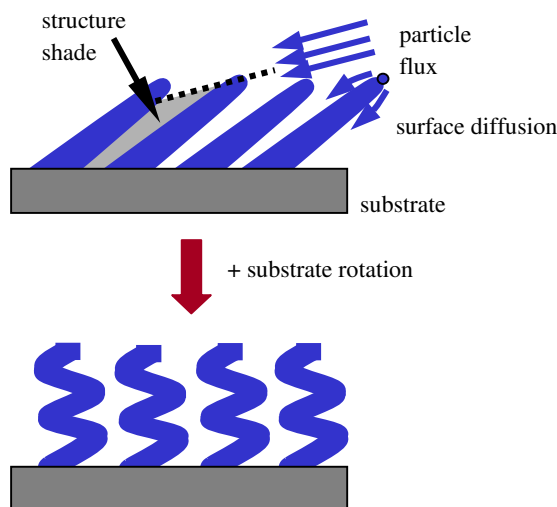


Figure 1: Principle of GLAD

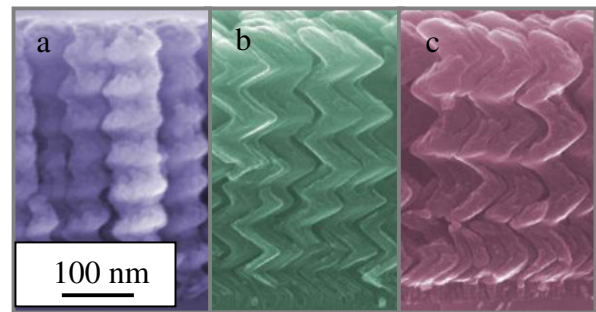


Figure 2: Chiral nanostructures from Si grown by GLAD: screws (a), chevrons (b), and spirals (c).

Chiral nanostructures exhibit a fibre-like fine structure, where each fibre exhibits a diameter of roughly 20 nm. A single chiral nanostructure is composed of a fibre bundle and the amount of fibres within one bundle determines the final diameter of the structure.

Sculptured thin film growth on unseeded substrates is determined by self-ordering phenomena yielding to periodic arrangements of chiral nanostructures across the substrate. Upon interaction with visible light the nanostructure arrangement supports directional diffraction making sculptured thin film acting like nanogratings. Periodic sculptured thin films can be also achieved by using prepatterned substrate templates (Figure 3).

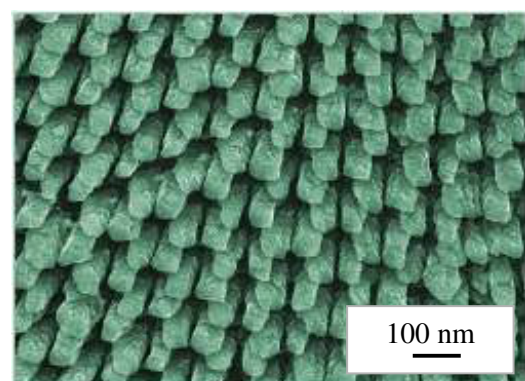


Figure 3: STF growth on substrate templates.

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Low-power plasma jet treatment of small size optics

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Low-power reactive plasma jets working at atmospheric pressure and with mm or sub-mm spot sizes are promising tools for highly deterministic local surface processing [1-5]. Especially, exploiting fluorine chemistry for etching silicon and silicon based materials like quartz, silicon nitride or silicon carbide and some metals like tungsten, molybdenum, or titanium as well as material combinations like the low thermal expansion material ULETM can be removed effectively. Thus, the plasma jet is well suited for the figuring and figure error correction of small size optics as well as the correction of mid spatial frequency errors of large and medium size optics.

For that purpose a compact microwave driven plasma jet source has been developed and tested. Depending on the material under consideration Gaussian like etch spots with a FWHM between 0.3 mm and 0.7 mm have been achieved (see Figure 2).

The typical working distance is about 3 mm which allows the treatment of small size and strongly curved workpieces.

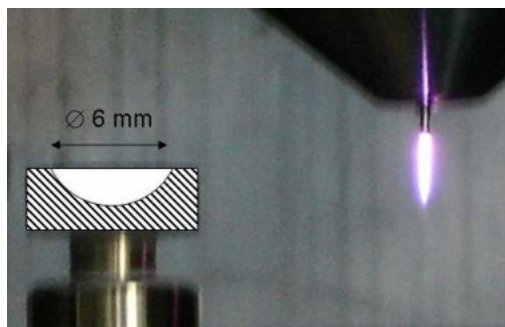


Figure 1: Plasma jet together with small size concave quartz optical element.

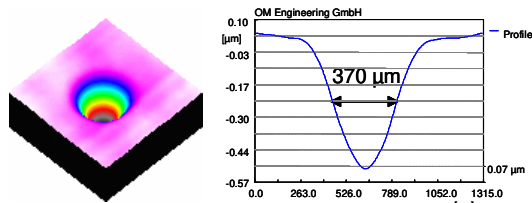


Figure 2: Removal spot of the plasma jet on silicon.

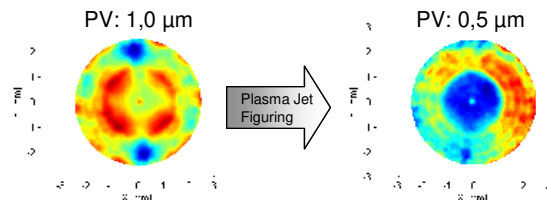


Figure 3: Shape error improvement of a small size and strongly curved concave lens with aspheric shape after single step scanning dwell time plasma jet processing.

The removal rate can be adjusted by varying microwave power and reactive gas flow (typical etch rates for quartz substrates lie between 10 nm/s and 100 nm/s).

Figure 1 shows the plasma jet together with a small size concave quartz optical element which has an aspheric shape. The edge angle of this element was more than 60 degrees. In a single step dwell time line by line scanning procedure the initial shape error of about 1 μm PV was reduced by nearly 50 % to about 500 nm PV as shown in Figure 3. In this case the plasma jet source had been mounted to a 3-axis moving system to keep the working distance constant during the figuring process. Work is continued using a 5-axis moving system that makes it possible to position the plasma jet always normal to the surface. Thus, a significant improvement of the figuring result is assumed due to the fact, that the angle dependence of the etch rate and the shape deformation of the etch spot near the edges are avoided.

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A new EUVL mask blank deposition tool

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The internationally favourable technology to achieve the 45 nm node is the extreme ultra-violet lithography (EUVL) with an exposure wavelength at 13.4 nm. Extremely high demands will be made on the deposition technology for the reflection masks [1,2]: (i) The relative deviation of reflectance over an area of 150 mm×150 mm of less than 1 % is required. (ii) The particle or the defect density for masks should be less than 10^{-3} cm⁻² for 6 inch square. (iii) The deposition rates of Mo and Si have to be as high as possible to process an EUV mask blank within one hour.

A schematic view of the new EUV mask blank deposition tool is shown in Figure 1. The Mo and Si layer deposition is performed with a segment controlled linear electron cyclotron resonance

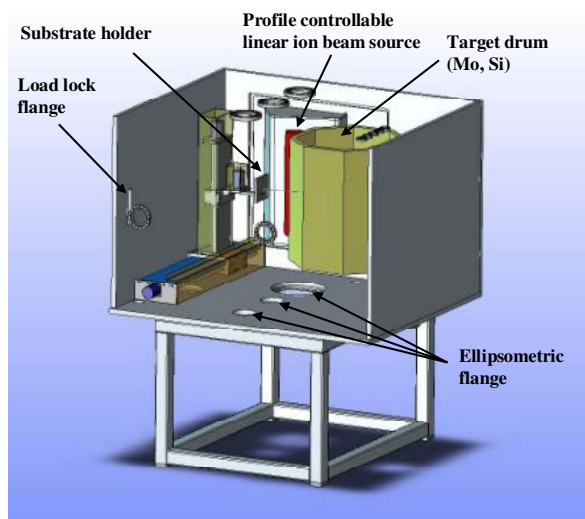


Figure 1: Schematic view of the process chamber of the ion beam sputter deposition tool.

(ECR) ion beam source. 8 Mo and 8 Si targets are mounted on a rotating target drum where the target surface is positioned in the ion beam focus. The base pressure of the process chamber is less than 5×10^{-8} mbar using a combination of a turbo molecular and a cryo pump. The particle minimisation is realised by: 1) huge process chamber dimension, 2) permanently rotating of the target drum, 3) vertical handling and deposition of the mask blank, 4) exclusive material selection for

contact pads and chamber lining, 5) electrical beam profile control by the ion beam source (no substrate rotation necessary), and 6) ion source material and geometry optimisation. A handler chamber is modular designed to mount an additionally process chamber as well as a further load lock. The transfer of the mask blanks from the load lock to the process chamber is carried by a particle specified (<0.1 particle/cycle) linear pneumatic rod.

The deposited Mo/Si multilayers were studied by TEM, AFM, ellipsometry, X-ray reflectometry, etc. As example, in Figure 2, a cross section taken

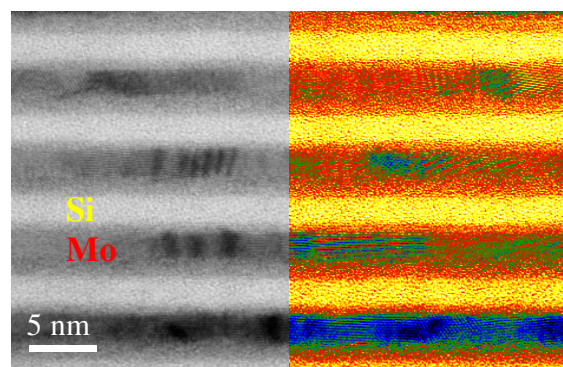


Figure 2: XTEM micrograph and the corresponding element mapping by EEL of a Mo/Si multilayer on the mask blank.

from an as-deposited Mo/Si multilayer sample prepared by ion-beam thinning is shown together with the corresponding element mapping across five periods of the layer stack (details see refs. [3-5]).

This industrial suitable deposition tool is ready for use for application of high-quality EUV mask blanks.

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Plasma immersion ion implantation of Ti alloys for medical application

S. Mändl, D. Manova

Biocompatibility itself is a concept easy to understand and difficult to define, thus necessitating further fundamental research on surface interactions. Several modes can be identified, albeit it is still not possible to give a succinct description of their respective mechanisms: (i) surface topography on the μm - and nm -scale; (ii) electronic density of state at the surface; (iii) outdiffusion of metallic cations; (iv) generation of wear particles and their transport, e.g. in macrophages towards a final agglomeration in the lung and spleen.

Plasma immersion ion implantation (PIII) is a powerful method to obtain hard and wear resistant surface on Ti alloys and NiTi by oxygen or nitrogen implantation [1,2]. By adjusting the temperature, treatment time, and heating regime, different phase compositions and layer thickness can be obtained. Depending on the specific system, a strong influence of the resulting microstructure on the wear and fatigue properties was observed.

In addition to the microstructure, the phase formation is also strongly influenced by the temperature and heating regime. Oxygen implantation into NiTi and pure Ti always leads to the formation of rutile, independent of the temperature and the heating regime. $\text{Ti}_6\text{Al}_4\text{V}$ shows a mixture of anatase and rutile, together with the formation of alumina at temperatures beyond 600°C with anatase, the metastable low temperature phase, favored against rutile when the samples are pre-heated to 400°C before starting the ion implantation.

A similar effect is observed during nitrogen implantation into pure Ti [3]. Starting the implantation at room temperature leads to the parallel formation of $\delta\text{-TiN}$ and $\epsilon\text{-Ti}_2\text{N}$, as observed by X-ray diffraction (XRD). In contrast, additional external heating with a start of the ion implantation at 350°C results in the dominance of Ti_2N .

Correspondingly, the treatment time, temperature, and heating regime translate into different mechanical properties as mediated by the layer thickness, phase composition, and microstructure. Using a rotating ball-on-disc test, no significant

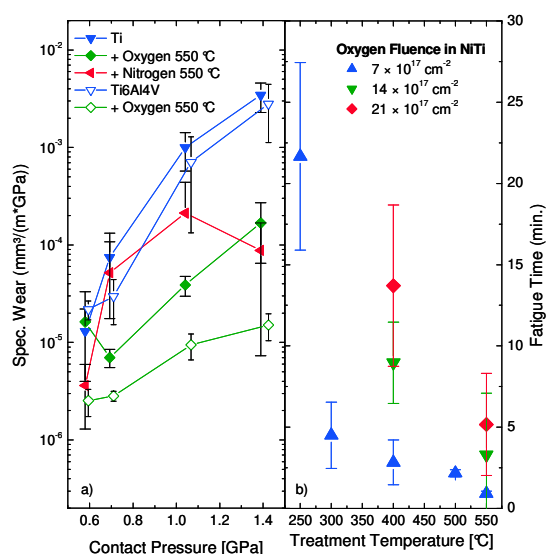


Figure 1: a) specific wear and b) fatigue time for oxygen implantation at different contact pressures, implantation temperatures and ion fluxes.

difference in the wear rate of untreated Ti and $\text{Ti}_6\text{Al}_4\text{V}$ was found (see Figure 1a), whereas oxygen implantation leads to a wear reduction of two orders of magnitude for the former material, increasing to 2.5 orders of magnitude for the latter one.

As the fatigue time of oxygen implanted NiTi, as depicted in Figure 1b, actually increases with decreasing implantation temperature and increasing ion fluence, a complex interplay of adhesion problems due to the sharp interface between the rutile surface and the NiTi intermetallic base material, coupled with thermal stress annealing can be proposed in this system.

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UV/VIS absorption spectra of alkyl-, vinyl-, aryl- and thiylperoxyl, and some related radicals in aqueous solution. A quantum-chemical study

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Alkylperoxyl and alkoxy radicals only absorb in the UV, while vinylperoxyl, phenylperoxyl, alkylthiylperoxyl and benzyloxyl radicals have strong absorptions in the visible. Using Time Dependent Density Functional Theory (TD DFT) the UTD/B3LYP/6-31G+(d,p) method we have calculated the long-wavelength absorption maxima of these and related radicals in vacuo and in aqueous solution [1]. The latter has been accounted for by a dielectric continuum model (SCRF=PCM). The vinyl peroxyl radical long-wavelength absorption band is due to a $-2\beta[\pi] \rightarrow 0\beta[n(O)_z]$ transition. The $n(O)_y$ orbital lies above the π orbital, but since it is orthogonal to the $n(O)_z$ orbital the oscillator strength of the $-1\beta[n(O)_y] \rightarrow 0\beta[n(O)_z]$ transition is close to zero (Figure 1).

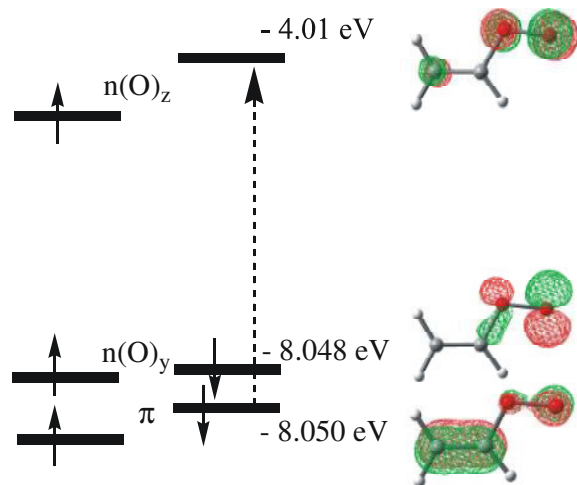


Figure 1: MO schemes for transitions of the vinylperoxyl radical in water.

Upon chlorine substitution, the first absorption band is red-shifted. The π orbital is now raised above the $n(O)_y$ orbital, and the transition is denoted as $-1\beta[\pi] \rightarrow 0\beta[n(O)_z]$ (Figure 2).

The absorption of the phenylperoxyl radical in the visible is accounted by two nearby transitions of the same type, i.e. $-2\beta[\pi] \rightarrow 0\beta[n(O)_z]$ and $-1\beta[\pi] \rightarrow 0\beta[n(O)_z]$. Due to the charge transfer character of these transitions there is a marked red-shift upon going from the gas phase to aqueous solutions. The benzyloxyl and phenoxymethyl radicals are related to the phenylperoxyl

radicals in so far as one of the peroxyl oxygens is replaced by a methylene group.

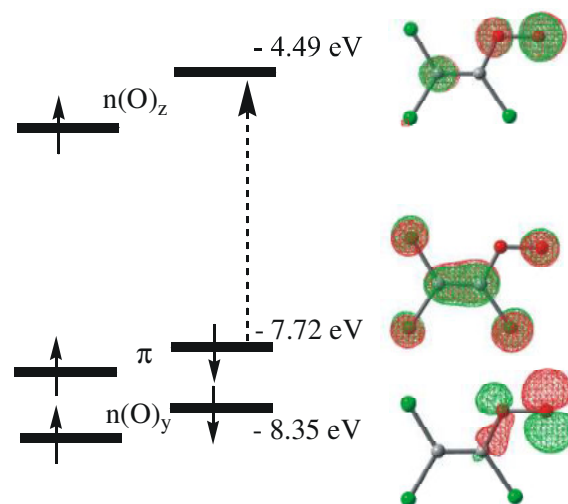


Figure 2: MO schemes for transitions of the chlorine substituted vinylperoxyl radical in water.

The benzyloxyl radical also absorbs in the visible and the transition is of the $-2\beta[\pi] \rightarrow 0\beta[n(O)_z]$ plus $-1\beta[\pi] \rightarrow 0\beta[n(O)_z]$ type, i.e. it is closely related to that of the phenylperoxyl radical. The phenoxymethyl radical only absorbs in the UV ($\lambda_{\max} = 320$ nm; spectrum obtained by pulse radiolysis) and this absorption band is due to $0\alpha[\pi] \rightarrow +2\alpha[\pi^*]$ plus $-1\beta[\pi] \rightarrow 0\beta[\pi]$ transitions. The absorption in the visible of the alkylthiylperoxyl radical is a $-1\beta[n(S)_z] \rightarrow 0\beta[n(O)_z]$ transition. Alkylthiyl and some other sulfur- and carbon-centered radicals react reversibly with O_2 . The energetics of these reactions have been addressed by DFT quantum-chemical calculations.

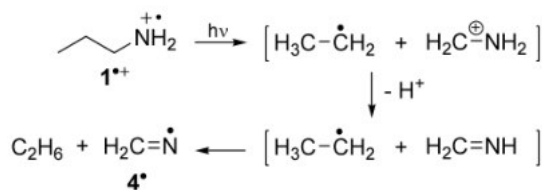
The programmes for calculations of absorption spectra of free radicals can be used now to predict the positions of the absorption maxima which allows for the theory-based optimization of irradiation sources. With radicals whose transitions have CT character the dielectric constant of the medium has to be taken into account.

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Löffler-Freytag reaction. The rates of these reactions are found to be in accord with theoretical calculations that predict a much lower barrier for the 1,5-shift in the *n*-butylamine radical cation than for the 1,4-shift in the *n*-propylamine radical cation. For **1⁺**, the 1,4-H shift is also brought about directly at 77 K by exposure to ~ 350 nm light, although there is also evidence for the 1,3-H shift requiring a higher energy. A surprising result of the present work is the formation of the methylene imino radical H₂C=N[•] (**4[•]**) after photoexcitation. It is suggested that this occurs as a consequence of the β-fragmentation of **1⁺** to the ethyl radical and the CH₂=NH₂⁺ cation, followed by consecutive cage reactions of deprotonation and hydrogen transfer from the iminium group.



Additionally, at high substrate concentrations the propane-1-iminyl radical $\text{CH}_3\text{CH}_2\text{CH}=\text{N}^\bullet$ was detected. Its formation is attributed to a modified reaction sequence in which $\mathbf{1}^{+\bullet}$ first undergoes a proton transfer within a cluster of amine molecules to yield the aminyl radical $\text{CH}_3\text{CH}_2\text{CH}_2\text{N}^\bullet\text{H}$. A subsequent disproportionation of these radicals can then yield the propane-1-imine precursor $\text{CH}_3\text{CH}_2\text{CH}=\text{NH}$ which is known to easily undergo hydrogen abstraction from the nitrogen.

Both cases highlight the great stability of $\text{RCH}=\text{N}^\bullet$ radicals due to a strong hyperconjugation between the C–H σ -orbital(s) and the nitrogen 2p orbital of the unpaired electron. Iminyl radicals are important reaction intermediates and are often detected under circumstances where normally their formation would not be expected.

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Surface modification of nano-sized zeolites

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Academia Sinica

Selectivity enhancement of zeolite catalysts for hydrocarbon processing often invokes inactivation of unselective sites by post-synthesis modifications. This issue, which is closely related to the external surface area of zeolite particles, is more crucial for catalysts consisting of nano-size crystallites. Surface modifications by chemical liquid deposition of organosilicon compounds or pre-coking treatment have been used to deactivate non-selective acid sites present on the external surfaces of zeolite H-ZSM-5 and H-FER.

Solid-state ^{31}P MAS NMR of adsorbed tributylphosphine oxide (TBPO) probe molecules has been shown to be an excellent technique for the characterisation of external acid sites on zeolite crystallites [1]. As shown in Figure 1, a significant decrease in the amount of strongest acid site (i.e., resonance peak at 90 ppm) on H-FER has been preferentially obtained after pre-coking.

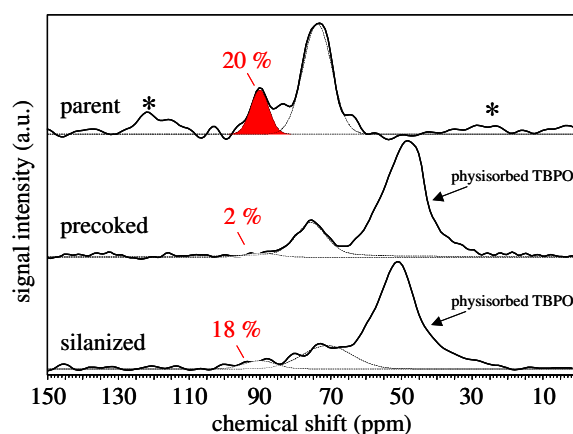


Figure 1: ^{31}P MAS NMR spectra of TBPO adsorbed on H-FER samples before and after surface modification. The asterisks indicate spinning sidebands.

To determine the genuine distribution of coke species formed during the pre-coking procedure, carbonaceous deposits have been released by HF dissolution of the zeolite framework. After extraction by CH_2Cl_2 a huge amount of "insoluble coke" remained in form of black particles. The MALDI-TOF mass spectrum revealed a broad molecular weight distribution up to 1300 Da corresponding to coke species with $n_{\text{C}} < 100$ (Figure 2). Obvi-

ously, such large carbon entities are preferentially deposited on the external surface and/or in the pore-mouth region. The MS patterns with repeating mass increments of 24, 37, and 50 Da point to a belt-like topology of polyaromatic deposits.

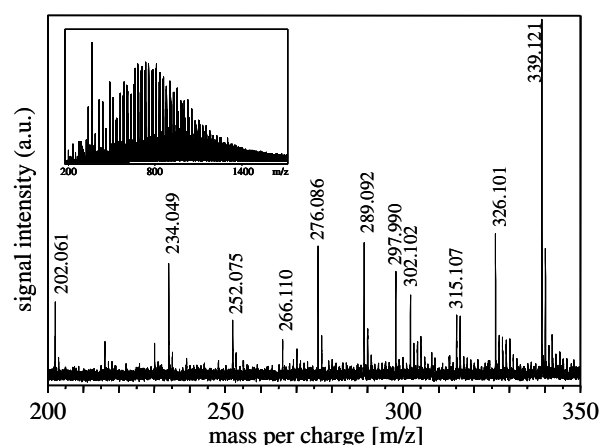


Figure 2: Low-molecular part of MALDI-TOF mass spectrum of CH_2Cl_2 -insoluble coke prepared by pre-coking treatment at 450 °C (insert: full mass spectrum).

Effects of surface modification by chemical liquid deposition of organosilicon compounds and the pre-coking technique on selectivity during xylene isomerisation and skeletal isomerisation of n-butene have been studied over Pt/H-ZSM-5 and H-FER, respectively.

A significant reduction of undesired xylene disproportionation reactions was only found after pre-coking treatment of H-ZSM-5. Thus, the effective inactivation of strong acids site on the external surfaces of nano-sized crystallites allows decreasing the xylene loss during xylene isomerisation. Liquid phase deposition of organosilicons, however, resulted in pore narrowing of both zeolites revealed by sorption measurements. These findings are in agreement with the observed enhancement of para-selectivity [2].

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Polymeric nanocomposite coatings based on isocyanate/polyol systems reinforced by aluminium carboxylate nanofillers

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Novel aluminium carboxylate nanopowders have been developed [1]. Owing to their organophilic nature, the aluminium-organic particles are promising fillers for scratch and abrasion resistant nanocomposites. The efficiency of these nanopowders was proven in radiation-cured (meth)acrylic systems. The aim of this study was the development of transparent aluminium-organic nanoparticles-reinforced isocyanate/polyol (two-component) systems with enhanced scratch and abrasion resistance.

Aluminium carboxylates were synthesized through precipitation reaction from dissolved aluminium isopropylate in aqueous solutions of carbonic acids [1]. A series of analogous nanomaterials was obtained by utilizing different acids: maleic (*Almal*), D,L-malic (*Almalat*), L-(+)-tartaric (*Altar*), mucic (*Almuc*), oxalic (*Aloxal*), citric (*Alcit*), 2,2-di(hydroxymethyl)propionic (*Aldihyp*) and L-glutamic (*Alglutam*) acid. Polyol (110/012464/00) and isocyanate (065/000103/00) with *Cetelon* charge codes in parentheses were chosen for the preparation of the two-component systems. The nanopowders were dispersed in the polyol component (2 h, 60 °C, 3000 rpm) and subsequently subjected to a bead mill treatment (ZrO₂, 0.4-0.7 mm, 4 h, 30 °C, 5000 rpm). After addition of isocyanate (mixing ratio polyol/isocyanate 10/3) and subsequent dilution in butyl acetate, application of the substrates was realized by spraying. The nanocomposite coatings were thermally cured at 120 °C for 90 min (aluminium) and 90 °C for 10 h (PMMA), respectively.

For the characterisation of the nanocomposite coatings microindenter and abrasion tests as well as haze measurements were performed. The modification of the two-component lacquer with the relevant nanofillers results generally in significantly enhanced hardness, e.g. to 175 % for *Almal* modified coatings (Figure 1). The abrasion tests

(Taber Abraser CS-0/S-42, two 500 g weights) show in all cases a slightly decreased abrasion of the nanocomposite coatings compared to the unmodified material (e.g. to 80 % for *Almalat* addition).

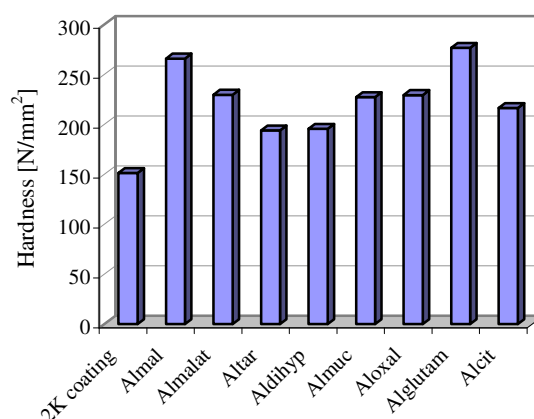


Figure 1: Martens hardness (100 mN; 20 s) of the nanocomposite coatings (35 wt.-%; ~50 µm on PMMA) compared to the unmodified reference coating.

In most cases the admixture of the nanocomponent markedly reduces transparency (e.g. haze values of 3.8 % and 5.0 % for *Aloxal* und *Alcit* addition, respectively). This unsatisfying feature is probably due to particle agglomerates and is currently under investigation.

Summarising, aluminium-organic nanofillers can readily enhance hardness and abrasion resistance of the two-component (isocyanate/polyol) system. However, this particular modification tends to reduce transparency. This disadvantage can be removed by the application of high-energy attrition ball milling or, alternatively, by precluding agglomeration in an optimised particle preparation route.

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In-line monitoring of the conversion in UV-cured coatings by near-infrared spectroscopy

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Most functional properties of UV-cured coatings strongly depend on the conversion which is achieved during irradiation. Therefore, continuous control of the conversion is required in order to ensure a constantly high quality of the product. However, in the past no analytical method had been proven to be suited for use in a production environment. Recently, it could be shown that near-infrared (NIR) reflection spectroscopy can be used for process control [1].

At first, NIR spectra have to be calibrated to calibration samples with well-known properties. Sophisticated chemometric evaluation methods (e.g. PLS) are used for the calibration procedure [2]. A typical PLS-based calibration using FTIR transmission spectroscopy as independent reference method is shown in Figure 1.

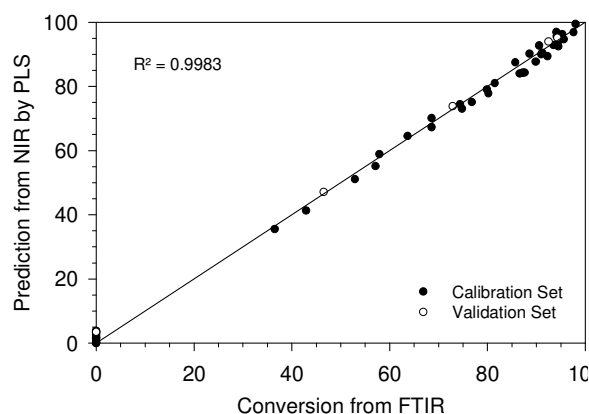


Figure 1 : PLS calibration of NIR reflection spectra of UV-cured acrylate coatings to the conversion determined by FTIR spectroscopy.

Alternatively, quantification can be carried out according to Beer-Lambert using the absorption band of the acrylic double bonds at 1620 nm [1].

For in-line monitoring of the conversion the probe head of the spectrometer is mounted on a coating machine. The conversion after UV or EB curing can be determined in any coating system based on (meth)acrylates and on almost any substrate. Ty-

pical examples are clear and pigmented coatings on polymer foils, paper, or fibreboard [3]. A special application are UV-curable pressure-sensitive adhesives. Their adhesive properties were found to respond extremely sensitive even to minor changes of the conversion [4] which requires permanent and reliable control of the conversion. An example is shown in Figure 2.

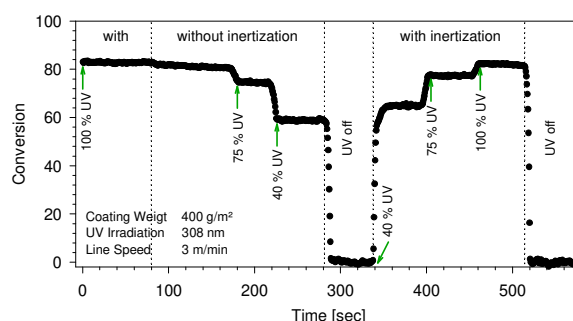


Figure 2 : In-line monitoring of the conversion in an acrylic hot-melt adhesive after UV irradiation with variable irradiance.

Cycloaliphatic epoxies which are the main component in cationic UV formulations do not show a specific absorption band which originates from their functional groups. Nevertheless, it could be shown that cationic systems can be characterised by NIR spectroscopy as well if the formulation contains vinyl ethers which are often used for dilution. The absorption of the vinyl groups at 1612 nm allows at least an indirect determination of the conversion and enables in this way a process control for such coatings.

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Contributions to the improvement of UV-technology for the UV-curing of functional coatings

R. Schubert, M. Hinkefuß, R. Konieczny, M.R. Buchmeiser

About 95 % of radiation curable printing inks and varnishes are polymerised via free radical polymerisation. However, this reaction is very sensitive to O_2 . Its presence results in incomplete curing and consequently the desired application properties of the final coating will not be reached. In principle, there exist two ways of reducing or compensating the influence of O_2 . One is the use of high photoinitiator concentrations which entails a large number of disadvantages. Alternatively, an efficient rinsing of the UV-irradiation zone by inert gas, preferably N_2 —termed “inertisation”—may be applied [1].

Meanwhile there exist several types of inertisation systems [2,3] both for reel to reel and panel coating, which aim on realizing low levels of O_2 in the irradiation zone by the economical use of nitrogen in an open channel. This is in principle applicable, however, it does not guarantee the removal of O_2 out of the coating layer since this process needs time and depends on mass transfer conditions. In this context, one important factor among others is the diffusion coefficient of O_2 inside the liquid layer especially depending on viscosity.

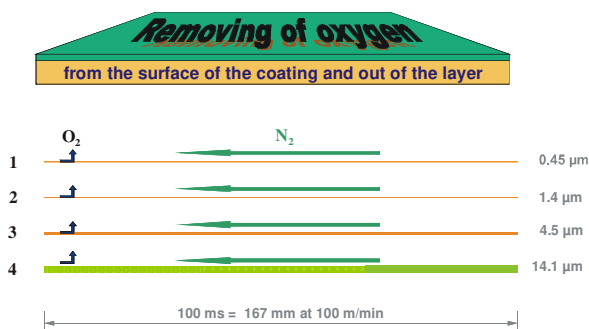


Figure 1: Diffusion coefficient of oxygen in:

1	solid coatings	$10^{-8} \text{ cm}^2 \text{ s}^{-1}$
2	highly viscous coatings	$10^{-7} \text{ cm}^2 \text{ s}^{-1}$
3	medium viscous coatings	$10^{-6} \text{ cm}^2 \text{ s}^{-1}$
4	low viscous coatings	$10^{-5} \text{ cm}^2 \text{ s}^{-1}$

Of additional importance is the mass transfer coefficient in the border layer. A high relative speed between web surface and N_2 is required to intensify mass transfer. This will be reached by using a low irradiation channel and an air knife nozzle.

In each case a long inerted channel, possibly placed in front of the UV-lamp, should be chosen.

Considering this know-how, a “high-speed” inerting system for UV-curing in flexo-graphic printing has been constructed and tested under production conditions up to 400 m/min.

This inertisation system consists of a low channel with an air knife, a volume nozzle on the inlet side, and a contactless turbulence barrier on the outlet side. The total nitrogen consumption was low.

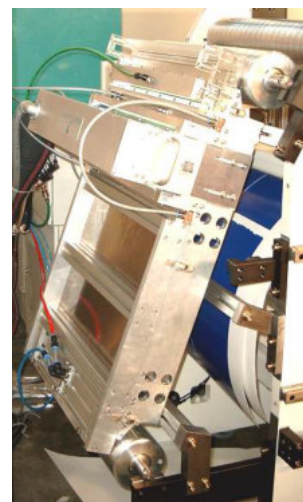


Figure 2: “High-speed” inertisation system with mercury-UV-lamp.

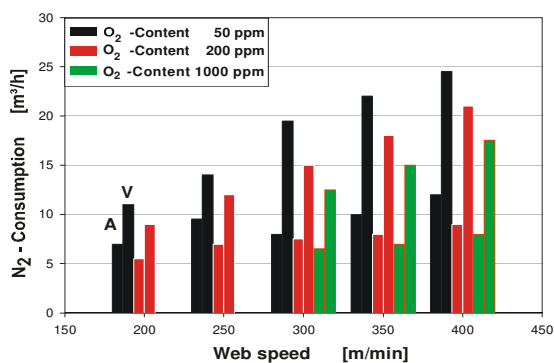


Figure 3: N_2 consumption of “high-speed” inertisation. A: air knife nozzle, V: volume nozzle

SPEED [m/min]	O_2 [ppm]	N_2 total [l/m²]
200	50	3.0
400	50	3.0
400	1000	2.2

Literature

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PTS-Manuskript: PTS-MS 479 p. C 14b 1-12
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- [3] EP 0 830 217 B1

Development environment - friendly and waterbased flexo printing inks

P. Klenert

Today solvent-based printing inks are worldwide used in flexo printing and still dominate the market for non-absorbing substrates. Environmental, health, and safety legislation have put strain on users exposed to solvents. Therefore, the goal of the project is the substitution of volatile organic compounds (VOC)-based flexo printing inks by water based ones. However, water based inks are more difficult to dry and impose problems in adhesion and wetting since they contain about 75 % water in binding which must be removed in well aligned drying processes. In film packaging applications, different foils (e.g. PP, PE, PET, PA) are used with strong diverse surface properties requiring well aligned adhesion properties of the corresponding ink. In collaboration with an ink producer, the IOM has started a project with two main targets, i.e. (i) the development of water based ink systems with high solid contents in formulation for use with broad range of foils, (ii) the development of inks for lamination in course of adapted laminating process.

For our studies, a combined thermal and infrared drying system was developed. This drying application was installed between the presses to avoid back fission of inks. Using existing dryers the



Figure 1: Drying system between the presses.

optimum printing conditions necessary to achieve optimum print quality have been determined.

Finally, changes in ink formulation led to homogeneous ink density at different printing speeds.

The inks demand a well adapted adhesive.

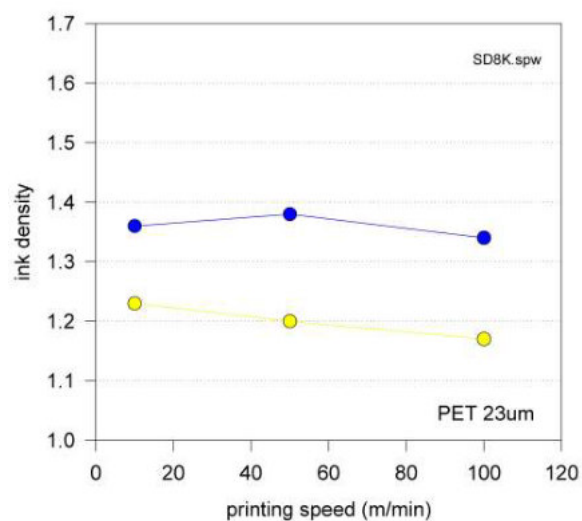


Figure 2 : Ink density versus printing speed.

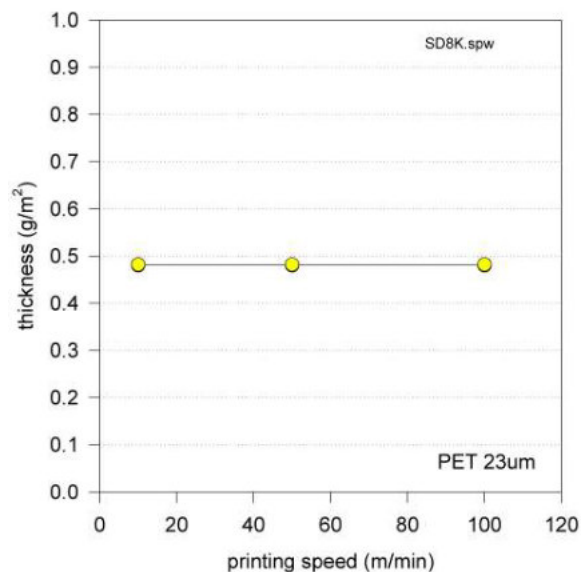


Figure 3: Ink thickness versus printing speed.

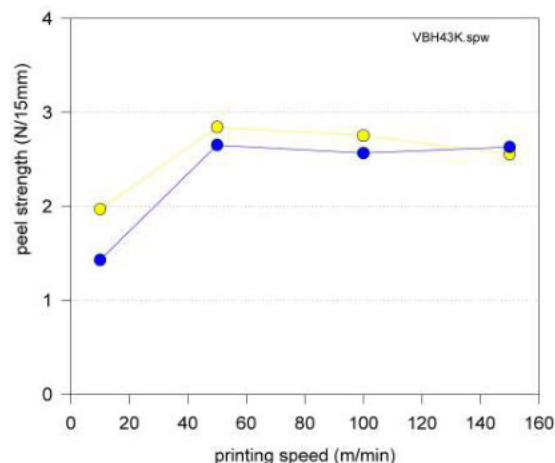


Figure 4: Peel strength versus printing speed.

Personal Activities and Scientific Events

Habilitations, Doctoral and Diploma Theses

Activities in Scientific Organisations

Honours and Awards

Scientific Meetings and Institute Colloquia

Lectures and Seminars

Personal Activities

Habilitations, Doctoral and Diploma Theses

Habilitations

Thomas Höche
Incommensurate Structural Modulations in Fresnoite Framework Structures
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2005

Doctoral Theses

Jens Dienelt
Chemisch unterstütztes Ionenstrahlätzen von Galliumarsenid: Prozessaufklärung und Anwendungen
Technische Universität Ilmenau, Fakultät für Mathematik und Naturwissenschaften, 2004

Stefan Sienz
Ionenstrahlgestützte Synthese von epitaktischen Galliumnitrid-Schichten auf Siliziumkarbid
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2004

Thomas Arnold
Untersuchungen zur Plasma-Oberflächen-Wechselwirkung beim Ätzen von Silizium mit einem Ar/SF₆/O₂-Plasmajet
Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2005

Dirk Schumann
Lösungsmittelfreie Systeme aus biogenem Polyhydroxybutyrat und synthetischen Polymeren - Beiträge zu biologisch abbaubaren Beschichtungen
Universität Leipzig, Fakultät für Chemie und Mineralogie, 2005

Diploma Theses

Tina Otto
Zeitaufgelöste Untersuchungen zur Prozessaufklärung beim Laserrückseitenätzen
Hochschule Mittweida (FH), Fachbereich Mathematik, Physik und Informatik, 2004

Mirko Kramer
Konstruktion von Probenaufnahmen für LEII
Hochschule für Technik, Wirtschaft und Kultur Leipzig (FH), Fachbereich Maschinen- und Energietechnik, 2004

Claudia Fleischmann
Mikrostrukturierung von Festkörperoberflächen durch Ultrakurzpulslaser
Westfälische Hochschule Zwickau (FH), Fachbereich Physikalische Technik und Informatik, 2004

Alexander Fleischer

Mechanische Eigenschaften von oberflächenmodifizierten Ti- und NiTi-Legierungen für biomedizinische Anwendungen

Hochschule für angewandte Wissenschaften Anhalt/Köthen (FH), Fachbereich Elektrotechnik, 2004

Marlen Ducherow

Wechselwirkung von modifizierten Titanoberflächen in simulierter Körperflüssigkeit

Hochschule für angewandte Wissenschaften Anhalt/Köthen (FH), Fachbereich Elektrotechnik, 2005

Steffen Müller

Anwendungserweiterung von Interferometermessungen mittels der Stitching-Methode – Mathematische Grundlagen und Programmierung

Hochschule für Technik, Wirtschaft und Kultur Leipzig (FH), Fachbereich Informatik und Mathematik, 2004

Markus Reinhardt

Auswertung von Verweilzeitätzungen zur Bestimmung der Werkzeugfunktion des Ionenstrahl- und des PACE-Verfahrens

Hochschule für Technik, Wirtschaft und Kultur Leipzig (FH), Fachbereich Informatik und Mathematik, 2005

Melanie Kitzing

Segregation in Nickel Titanium after Oxygen Plasma Immersion Ion Implantation

Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2005

Martin Ehrhardt

Laserinduziertes Rückseitenätzen transparenter Materialien mit ultrakurzen Pulsen

Technische Fachhochschule Wildau, Fachbereich Physikalische Technik, 2005

Anja Hänchen

Untersuchung der Phasenbildung mittels in situ Röntgendiffraktometrie während der Synthese von vergrabenen Übergangsmetalloxidschichten durch Hochenergie-Ionenimplantation

Hochschule Zittau/Görlitz (FH), Fachbereich Mathematik und Naturwissenschaften, 2005

John Fahlteich

Darstellung chiraler Nanostrukturen mit der GLAD

Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2005

Frank Ulmer

Synthese dünner Metallfilme mittels ultrakurz gepulster Laserablation

Universität Leipzig, Fakultät für Physik und Geowissenschaften, 2005

Activities in Scientific Organisations

M.R. Buchmeiser

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

B. Rauschenbach

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

Honours and Awards

A. Schindler

Manfred-von-Ardenne-Preis für Angewandte Physik, 2005

B. Ziberi

Young Scientist Award of the European Material Research Society, 2004

Arbeitsgruppe unter Leitung von Dr. A. Schindler

Finanzielle Zuwendung der Carl-Zeiss-Stiftung für die Entwicklung der Strahlbearbeitungsverfahren, 2004

Scientific Events

Scientific Meetings and Institute Colloquia

Scientific Meetings

Workshop '*Ionenstrahlphysik und -technologie*', Leipzig, 11-13.04.2005

VI. Workshop der DFG-Forschergruppe '*Teilchenstrahlen-stimulierte Ultra-präzisions-Oberflächenbearbeitung*', Leipzig, 03.05.2005

XII. Workshop '*Oberflächentechnologien mit Plasma- und Ionenstrahlprozessen*', Mühlleithen, 14-16.03.2005

XI. Workshop '*Oberflächentechnologien mit Plasma- und Ionenstrahlprozessen*', Mühlleithen, 14-17.03.2004

V. Workshop der DFG-Forschergruppe '*Teilchenstrahlen-stimulierte Ultrapräzisions-Oberflächenbearbeitung*', Großbothen, 14-15.05.2004

Institute Colloquia

S. Mayr (22.01.2004)
Universität Göttingen, I. Institut für Experimentelle Physik, Göttingen
Ionenstrahlinduziertes viskoses Fließen: Wie harte Materie weich wird

J. Keckes (05.02.2004)
Erich Schmid Institut für Materialwissenschaft, Österreichische Akademie der Wissenschaften, Leoben, Austria
Elevated-temperature studies of residual stresses in thin films using X-ray diffraction

A. Duparré (25.02.2004)
Fraunhofer-Institut für Angewandte Optik und Feinmechanik, Jena
Oberflächencharakterisierung im Nanometerbereich

H.-F. Zeilhofer (20.04.2004)
Kantonsspital Basel, Universitätsklinik für Wiederherstellende Chirurgie, Basel, Switzerland
Neue Materialien in der Chirurgie

G. Bräuer (06.05.2004)
Fraunhofer-Institut für Schicht- und Oberflächentechnik Braunschweig und Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik Dresden
Aktuelle Entwicklungen der Magnetron-Sputtertechnik

W. Neumann (13.05.2004)

Humboldt-Universität Berlin, Institut für Physik, Berlin

Elektronenmikroskopie nanostrukturierter Halbleitermaterialien

J.-H. Peters (27.05.2004)

Advanced Mask Technology Center GmbH & Co. KG, Dresden

Herausforderungen bei der Maskenherstellung für die EUV-Lithographie

G. Schiwietz (22.01.2004)

Hahn-Meitner-Institut Berlin, Berlin

Ionenspuren in Festkörpern: Materie am Rande der Stabilität

V. Liebig (22.06.2004)

Deutsches Zentrum für Luft- und Raumfahrt Bonn-Oberkassel, Bonn

Das deutsche Raumfahrtprogramm

G.K. Wolf (13.07.2004)

Universität Heidelberg, Physikalisch-Chemisches Institut, Heidelberg

Großflächige ionenstrahlgestützte Abscheidung von Zinklegierungen auf Stahl

A. Robitzki (15.07.2004)

Universität Leipzig, Biotechnologisch-Biomedizinisches Zentrum, Leipzig

Funktionelles Biomonitoring in Echtzeit: Zell- und Gewebe-basierte Biochips

O. Anisimov (08.09.2004)

Institute of Chemical Kinetics and Combustion, Russian Academy of Sciences, Novosibirsk, Russia

Modulation of delayed fluorescence decay as the method of studying of radical ions and their reactions in solution

F.-J. Hormes (07.10.2004)

Center for Advanced Microstructures and Devices, Louisiana State University Baton Rouge, USA

Materialwissenschaftliche Untersuchungen am CAMD Baton Rouge

L. Chang (14.10.2004)

University Wuhan, School of Electrical and Microelectronic Materials, Wuhan, China

Preparation and Characterisation of Nanocrystalline Titanium and Zinc Oxides

I. Gurrappa (28.10.2004)

Defence Metallurgical Research Laboratory, Hyderabad, India

Development of high performance coatings for titanium alloys

K. Bewilogua (04.11.2004)

Fraunhofer-Institut für Schicht- und Oberflächentechnik, Braunschweig

PVD- und PACVD-Abscheidung harter, verschleiss- und reibungsarmer Schichten

H. Grünwald (04.11.2004)

Tetra Pak, Plastic Packaging, Darmstadt

Barrierschichten in Flaschen

K. Yamauchi (10.11.2004)

Osaka University, Department of Precision Science & Technology, Osaka, Japan

Fabrication and figure testing methods for hard X-ray optics at Osaka University

K. Helming (09.12.2004)

Bruker-AXS GmbH, Karlsruhe und Madison/Wisconsin

Textur und Anisotropie kristalliner Materialien

E. Zschech (20.01.2005)

AMD Saxony LLC & Co KG Dresden, Dresden

Anforderungen an die physikalische Analytik für Prozesskontrolle und Fehleranalyse in der Halbleiterindustrie

U. Gösele (01.02.2005)

Max-Planck-Institut für Mikrostrukturphysik, Halle

Nano-Silizium à la carte

F. Heyroth (07.04.2004)

Martin-Luther-Universität Halle-Wittenberg, Interdisziplinäres Zentrum für Materialwissenschaften, Halle

Atmosphärische Rasterelektronenmikroskopie: Möglichkeiten - Anwendung - Grenzen

J. Meijer (12.05.2005)

Ruhr-Universität Bochum, Central Laboratories of Ion beams and Radioisotopes, Bochum

Einzelionenimplantation zur Nanostrukturierung

M. Posselt (19.05.2005)

Forschungszentrum Rossendorf, Institut für Ionenstrahlphysik und Materialforschung, Rossendorf

Atomistische Computersimulation ionenstrahlinduzierter Prozesse: Implantation, Defektbildung, Defektmigration

G. Andersson (26.05.2005)

Universität Leipzig, Fakultät für Chemie und Mineralogie, Leipzig

Struktur von Oberflächen weicher Materie: Untersuchungen mit Elektronen- und Ionenstreu-spektroskopie

D.K. Avasthi (01.06.2005)

Nuclear Science Center New Delhi, New Delhi, India

Nanostructuring with ion beams at Nuclear Science Center

S. Guder (09.06.2005)

Technische Universität München, Institut für Werkstoffkunde und Werkstoffmechanik, München-Garching

Schädigungsvorgänge an zementierten Hüftendoprothesenstielen

U. De (30.06.2005)

Variable Energy Cyclotron Centre, Kolkata, India

Rough oxide surfaces studied by Rutherford backscattering

T. Michely (07.07.2005)

RWTH Aachen, 1. Physikalisches Institut, Aachen

Atomarer Beschussschaden und Musterbildung durch Ionenbeschuss auf Metalloberflächen

S. Pissadakis (08.09.2005)

Institute of Electronic Structure and Laser, Foundation for Research and Technology, Hellas, Greece

Laser Induced Periodic Structures in Optical Bulks and Fibres: Photosensitivity and Surface Engineering Processes

B. Voit (06.10.2005)

Leibniz-Institut für Polymerforschung, Dresden

Hyperbranched polymers in thin film and coating applications

T. Oates and M. Vinnichenko (20.10.2005)

Forschungszentrum Rossendorf, Institut für Ionenstrahlphysik und Materialforschung, Rossendorf

In situ spectroscopic ellipsometry of metallic nanoparticulate thin films

W. Binder (27.10.2005)

Technische Universität Wien, Institut für Synthesechemie, Wien, Austria

Supramolekulare Chemie mit Polymeren: Neue Zugänge zu funktionalen Materialien und Oberflächen

M. Möller (01.12.2005)

Deutsches Wollforschungsinstitut, Aachen

Biofunktionale Oberflächen

A. von Keudell (06.12.2005)

Ruhr-Universität Bochum, Institut für Experimentalphysik II, Bochum

Strukturbildung in reaktiven Plasmen: von Nanoteilchen zur kontrollierten Rauigkeit

C. Hollenstein (08.12.2005)

Ecole Polytechnique Fédérale de Lausanne, Centre de Recherches en Physique des Plasma, Lausanne, Switzerland

Industrielle Plasmaphysik an der Universität: Einige Beispiele

Lectures and Seminars

Lectures

F. Bauer

- *Radioaktivität, Kernenergie und Strahlenschutz*
HTWK Leipzig, Fakultät für Maschinen- und Energietechnik
winter 03/04
- *Radioaktivität, Kernenergie und Strahlenschutz*
HTWK Leipzig, Fakultät für Maschinen- und Energietechnik
winter 04/05
- *Radioaktivität, Kernenergie und Strahlenschutz*
HTWK Leipzig, Fakultät für Maschinen- und Energietechnik
winter 05/06

M.R. Buchmeiser

- *Makromolekulare Chemie*
Universität Leipzig, Fakultät für Chemie und Mineralogie
summer 05
- *Makromolekulare Chemie, Spezial- und Funktionspolymere*
Universität Leipzig, Fakultät für Chemie und Mineralogie
winter 05/06

T. Höche

- *Elektronenmikroskopie*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 05/06

S. Mändl

- *Physikalische Oberflächenmodifizierung von Werkstoffen der Medizintechnik*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 03/04
- *Lichtbogen und Laserablation zur Schichtabscheidung*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
summer 04
- *Oberflächenanalytik in Astronomie, Archäologie und Kunstgeschichte*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 04/05
- *Oberflächenflächenmodifizierung von modernen Leichtmetallen*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
summer 05
- *Plasmaphysik I: Plasmatechnologie*

Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 05/06

B. Rauschenbach

- *Physik dünner Schichten: Wachstum, Epitaxie, Struktur, Eigenschaften*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 03/04
- *Ionen-Festkörper-Wechselwirkung*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
summer 04

Einführung in Nanophysik und Nanotechnologie - Teil I
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 04/05

- *Fundamentals of the Ion-Solid-Interaction*
Nucl. Research Center New Delhi, India (compact lecture series)
winter 05
- *Oberflächen- und Dünnschichtanalytik*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
summer 05
- *Physik dünner Schichten: Wachstum, Epitaxie, Struktur, Eigenschaften*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 05/06

E. Schubert

- *Beschichtung und Beschichtungstechnologien*
HTWK Leipzig, Fakultät für Maschinenbau
winter 05/06

Seminars

T. Höche and B. Rauschenbach

- *Materialwissenschaftliches Seminar*
Universität Leipzig, Fakultät für Physik und Geowissenschaften
winter 03/04, summer 04, winter 04/05, summer 05, winter 05/06

M.R. Buchmeiser

- *Seminar für Diplomanden/Dissertanten*
Universität Leipzig, Fakultät für Chemie und Mineralogie
summer 05, winter 05/06

Publications and Presentations

Publications in Journals and Books

Conference Proceedings

Contributed Presentations

Patent Applications and Patents

Publications and Presentations

Publications in Journals and Books

T. Arnold, S. Grabovski, A. Schindler and H.-E. Wagner

Spatially resolved mass spectrometry of reactive Ar/SF₆/N₂ plasma jets
Surface and Coatings Technology **200** (2005) 818-821

T. Arnold, S. Grabovsky, A. Schindler and H.-E. Wagner

Spatially resolved emission spectroscopic investigation of microwave-induced reactive low-power plasma jets
Plasma Sources Science and Technology **13** (2004) 309-314

B. Bantu, D. Wang, K. Wurst and M.R. Buchmeiser

Copper (I) 1,3-R²-3,4,5,6-Tetrahydropyrimidin-2ylidenes (R=Mesityl, 2-Propyl): synthesis, X-ray structures, immobilization and catalytic activity
Tetrahedron Symposium **61** (2005) 12145-12152

F. Bauer and R. Mehnert

UV curable acrylate nanocomposites: Properties and applications
Journal of Polymer Research **12** (2005) 483-491

F. Bauer, U. Decker, A. Dierdorf, H. Ernst, R. Heller, H. Liebe and R. Mehnert Preparation of moisture curable polysilazane coatings. Part I. Elucidation of low temperature curing kinetics by FT-IR spectroscopy
Progress in Organic Coatings **53** (2005) 183-190

F. Bauer, E. Bilz and A. Freyer

C-14 studies in xylene isomerization on modified H-ZSM-5
Applied Catalysis A **289** (2005) 2-9

F. Bauer, E. Bilz and A. Freyer

Selectivity improvement in xylene isomerization
Studies in Surface Science and Catalysis **154** (2004) 2169-2178

F. Bauer, H. Ernst, D. Hirsch, S. Naumov, M. Pelzing, V. Sauerland and R. Mehnert Preparation of scratch and abrasion resistant polymeric nanocomposites by monomer grafting onto nanoparticles. Part V. Application of mass spectroscopy and atomic force microscopy to the characterization of silane-modified silica surface
Macromolecular Chemistry and Physics **205** (2004) 1587-1593

F. Bauer, W.H. Chen, H. Ernst, S.J. Huang, A. Freyer and S.B. Liu

Selectivity improvement in xylene isomerization
Microporous and Mesoporous Materials **72** (2004) 81-89

F. Bauer, H.-J. Gläsel, E. Hartmann, H. Langguth and R. Hinterwaldner Functionalized inorganic/organic nanocomposites as new basic raw materials for adhesives and sealants

International Journal of Adhesion and Adhesives **24** (2004) 519-522

N. Bogdanski, H. Schulz, M. Wissen, H.-C. Scheer, J. Zajadacz and K. Zimmer
3D-Hot embossing of undercut structures - an approach to micro-zippers
Microelectronic Engineering **73-74** (2004) 190-195

R. Böhme, J. Zajadacz, K. Zimmer and B. Rauschenbach
Topography and roughness evolution of microstructured surfaces at laser-induced backside wet etching
Applied Physics A **80** (2005) 433-438

R. Böhme and K. Zimmer
The influence of the laser spot size and the pulse number on backside wet etching
Applied Surface Science **247** (2005) 256-261

R. Böhme and K. Zimmer
Low roughness etching of fused silica using an adsorbed layer
Applied Surface Science **239** (2004) 109-116

R. Böhme and K. Zimmer
Rapid Prototyping von diffraktiven und refraktiven mikrooptischen Elementen
Photonik **03** (2004) 58-61

R. Böhme, K. Zimmer and B. Rauschenbach
Laser backside etching of fused silica due to carbon layer ablation
Applied Physics A **82** (2005) 325-328

R. Böhme, D. Spemann and K. Zimmer
Surface characterization of backside-etched transparent dielectrics
Thin Solid Films **453** (2004) 127-132

Y. Bohne, N. Shevchenko, F. Prokert, J. von Borany, B. Rauschenbach and W. Möller
In situ characterization of phase formation during high-energy oxygen ion implantation in molybdenum
Nuclear Instruments and Methods in Physics Research B **240** (2005) 157-161

Y. Bohne, N. Shevchenko, F. Prokert, J. von Borany, B. Rauschenbach and W. Möller
In situ X-ray diffraction investigations during high-energy oxygen ion implantation in transition metals
Vacuum **76** (2004) 281-285

M. Bortenschlager, M. Mayr, O. Nuyken and M.R. Buchmeiser
Hydroformylation of 1-octene using rhodium-1,3-R²-3,4,5,6-tetrahydropyrimidin-2-ylidenes (R = 2-Pr, mesityl)
Journal of Molecular Catalysis A **233** (2005) 67-71

O. Brede, R. Hermann, N. Karakostas and S. Naumov
Ionization of the three isomeric hydroxybenzoates by free electron transfer: product distribution depends on the mobility of the phenoxyl group
Physical Chemistry Chemical Physics **6** (2004) 5184-5188

O. Brede, A. Maroz, R. Hermann and S. Naumov

Ionization of cyclic aromatic amines by free electron transfer: products are governed by femtosecond molecule dynamics

Journal of Physical Chemistry A **109** (2005) 8081-8087

O. Brede, R. Hermann, S. Naumov, A.K. Zarkadis, G.P. Perdikomatis and M.G. Siskos

Free electron transfer reflects rotational conformers of substituted aromatics: Reaction of benzyltrimethylsilanes with n-Butyl chloride parent radical cations

Physical Chemistry Chemical Physics **6** (2004) 2267-2275

M.R. Buchmeiser

Zukunftstechnologie: Oberflächenveredelung mittels UV- und Elektronenstrahlhärtung

Kunststoff-Cluster-aktuell **5** (2005)

M.R. Buchmeiser

A Commentary on 'Bis(cyclopentadienyl)zirkon-Verbindungen und Aluminoxan als Ziegler-Katalysatoren für die Polymerisation und Copolymerisation von Olefinen' by W. Kaminsky, M. Miri, H. Sinn, R. Woldt (Makromol. Chem., Rapid Commun. 1983, 4, 417-421)

Macromolecular Rapid Communications **26** (2005) 1201-1207

M.R. Buchmeiser

Metathesis Polymerisation

Advances in Polymer Science, Volume 176, Ed. M.R. Buchmeiser, Springer, Berlin, Germany (2005)

M.R. Buchmeiser

Regioselective Polymerization of 1-Alkynes and Stereoselective Cyclopolymerization of α,ω -Heptadiynes

In: *Advances in Polymer Science*, Volume 176, Ed. M.R. Buchmeiser, 89-120. Springer, Berlin, Germany (2005)

M.R. Buchmeiser

Design and synthesis of supported catalysts on a molecular base

Catalysis Today **105** (2005) 612-617

M.R. Buchmeiser

In honor of Prof. Richard (Dick) R. Schrock on the occasion of his 60th birthday

Advanced Synthesis & Catalysis **347** (2005) 321

W.H. Chen, F. Bauer, E. Bilz, A. Freyer, S.J. Huang, C.S. Lai and S.B. Liu

Acidity characterization of H-ZSM-5 catalysts modified by pre-coking and silylation

Studies in Surface Science and Catalysis **154** (2004) 2269-2274

J. Dienelt, J. von Sonntag, K. Zimmer and B. Rauschenbach

Structuring of GaAs. I. Chemical dry etching: Temperature und chlorine pressure dependence of etch rates

Journal of Vacuum Science and Technology B **22** (2004) 1964-1969

- J. Dienelt, K. Zimmer, J. von Sonntag, B. Rauschenbach and C. Bundesmann*
Roughness and damage of a GaAs surface after chemically assisted ion beam etching with Cl_2/Ar^+
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