MI 8: Poster – Microanalysis and microscopy

Chair: H. S. Leipner and E. Langer

Time: Wednesday 15:00–17:30 Location: Poster E

MI 8.1 Wed 15:00 Poster E

XRF-instrumentation for polarization-dependent and reference-free nano-analysis as well as design study for 450 mm wafer-analysis — Jan Weser, Janin Lubeck, and •Ina Holfelder — Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin

New instrumentation for polarization-dependent measurements became recently available at PTB and will be presented. The new analytical chamber allows for polarization-dependent fluorescence measurements on e.g. functional nano-layers. The maximum sample size is 4 inch in diameter. By means of two orthogonally arranged Silicon Drift Detectors, the measurements can be realized spatially resolved and depths sensitive with a large angular range. This new instrumentation increases the measurement options for the plane-grating and four-crystal monochromator beamlines of the PTB at BESSYII, and makes it possible to measure layer thickness and composition, depth-dependent element profiles, and bonding states. Furthermore, in the framework of European project EEMI450, an advanced metrology chamber for 450 mm wafer is being designed. The current instrumentation of the PTB should be transferred for the 450 mm wafer application and completed with optical analytical methods. The current status of this virtual design studies will be presented too.

MI 8.2 Wed 15:00 Poster E

Alternative thin film and ion beam lithographic processing approaches for the fabrication of Fresnel zone plates — •Kahraman Keskinbora[1], Corinne Grévent[1], Markus Weigand[1], Mato Knez[2], Adriana Szeghalmi[2], Nadzeyka Achim[3], Lloyd Peto[3], Anatoly Snigirev[4], and Gisela Schütz[1] — [1]Max Planck Institute for Intelligent Systems, 70569 Stuttgart, Germany — [2]Max Planck Institute for Microstructure Physics, 06120 Halle, Germany — [3]Raith GmbH, 44263 Dortmund, Germany — [4]European Synchrotron Radiation Facility (ESRF), 38043 Grenoble, France

Fresnel zone plates (FZPs) are diffractive optics which are widely used for focusing X-rays, especially in X-ray microscopes. Their resolutions are essentially determined by the width of their outermost zone. Electron beam lithography (EBL) is the usual method of fabrication; it delivers FZPs with very fine outermost zone widths. However, the technique requires several steps of fabrication and the achievable aspect ratio which is critical for an effective focusing of hard X-rays is limited. In this work, new alternative fabrication methods are reported. One alternative method is ion beam lithography (IBL). It advantageously allows the fabrication of FZPs in a single step. Another method involves the deposition of a multilayer on a glass fiber with atomic layer deposition followed by its sectioning to deliver a multilayer FZP (ML-FZP) without any limitation in aspect ratio. FZPs fabricated according to both methods have been successfully tested in a soft X-ray scanning microscope (STXM) to perform imaging.

MI 8.3 Wed 15:00 Poster E

Untersuchungen zur Biokorrosion an mittelalterlichen Kirchenfenstern der Kathedrale von Tours (Frankreich) und des Meißner Doms (Deutschland) mittels REM/EDX — Evelyn Krawczyk-Bärsch[1] und •Siegfried Däbritz[2] — [1]Helmholtz Zentrum Dresden-Rossendorf, Institut für Ressourcenökologie, 01328 Dresden — [2]Technische Universität Dresden, Institut für Festkörperphysik, 01062 Dresden

Der Verfall mittelalterlicher Gläser in Domen und Kirchen schreitet infolge von Schadstoffbelastungen der Umwelt immer weiter fort. An bemalten mittelalterlichen Kirchenfensterfragmenten der Kathedrale St. Gatien in Tours (Frankreich) und der Allerheiligenkapelle des Meißner Doms (Deutschland) wurden nach Anwendung des Ionenstrahl-Böschungsschnittverfahrens Untersuchungen mittels REM und EDX durchgeführt. Diese erlaubten Aussagen über Verwitterungsphänomene, die vor allem in Form von Rissen und kraterförmigen Korrosionen (Lochfraß) zu erkennen waren. Da mittelalterliche Gläser aufgrund ihres niedrigen Anteiles an stabilisierenden Glaskomponenten, wie P_2O_5, MgO instabil sind, kommt es sehr häufig zu vertikalen und horizontalen Rissen. Durch unsere Untersuchungen konnte festgestellt werden, dass diese Risse stets von kleinen Löchern ausgehen. Da diese Löcher auf Ausscheidungsprodukte von Pilzen zurückgeführt werden können, wird dieses Phänomen auch "Lochfraß" genannt. Durch EDX-Mikroanalysen war es möglich, eine Abreicherung von glasstabilisierenden Verbindungen, wie CaO und K_2O, entlang dieser Risse nachzuweisen.

MI 8.4 Wed 15:00 Poster E

Precision of high-resolution EBSD strain determination using cross-correlation and phase-only correlation — •Thomas Riedl[1], Horst Wendrock[2], and Stefan Wege[2,3] — [1]Institut für Werkstoffwissenschaft, TU Dresden, 01062 Dresden, Germany — [2]IFW Dresden, P.O. Box 270116, 01171 Dresden, Germany — [3]now at: MPI für Eisenforschung, 40237 Düsseldorf, Germany

The present contribution analyses the precision of EBSD elastic strain measurement based on cross-correlation or phase-only correlation of regions of interest distributed over experimental patterns [1]. By means of correlation peak fitting and least-squares minimisation the procedure allows to obtain the eight independent components of the displacement gradient tensor with an average standard deviation of $1.2*10^{-4}$ under best conditions. It is shown that the cross-correlation version provides a better angular resolution compared to its phase-only counterpart. Moreover, optimum parameters such as Fourier filter widths and fit region sizes are determined as a function of the pattern signal-to-noise ratio. The achievable precision is also evaluated for specimen rotation and bending experiments. Finally, the application of the strain measurement method to diffusive-displacive phase transformations is discussed [2].

[1] A.J. Wilkinson et al.: Ultramicr. 106 (2006) 307
[2] G. Miyamoto et al.: Acta Mater. 57 (2009) 1120

MI 8.5 Wed 15:00 Poster E

Refinement of diffraction contrast tomography data by EBSD measurements at selected cross sections — •Melanie Syha[1], Andreas Graff[2], Frank Altmann[2], Daniel Weygand[1], and Peter Gumbsch[1,2] — [1]Karlsruhe Institute of Technology, Institute for Applied Materials, Kaiserstr. 12, 76131 Karlsruhe, Germany — [2]Fraunhofer Institute for Mechanics of Materials IWM, Freiburg and Halle, Germany

The evolution of the 3D microstructure in $SrTiO_3$ ceramic during annealing was studied by diffraction contrast tomography (DCT).

Since 3D orientation and microstructure data are analyzed with special emphasis on the local interface orientation, a good spacial resolution at the grain boundaries is of particular importance. For the non destructive DCT measurements of $SrTiO_3$ the sample size is about $0.2mm^3$. The precision of the grain shape measurements is a few microns. Grain boundaries are reconstructed by expanding the grains up to contact. To check the validity of these process EBSD measurements were performed on selected cross sections. Therefore cross sections at defined positions were produced by grinding and polishing. Due to the simple cubic perowscite structure fast EBSD measurements could be done on the whole cross section with one micron resolution.

The higher resolution makes smaller grains detectable. Also the shape of the grains and the pores is better defined. The EBSD microstructure data allow a classification of the grain boundaries at the selected cross sections. The spatial resolution of the EBSD data can be used to improve the tomography data analysis.

MI 8.6 Wed 15:00 Poster E

Spectroscopic investigation of silicon polymorphs formed by indentation — •Martin Schade[1], Bianca Haberl[2], and Hartmut S. Leipner[1] — [1]Interdisziplinäres Zentrum für Materialwissenschaften, Martin-Luther-Universität Halle-Wittenberg, D-06099 Halle — [2]Department of Electronic Materials Engineering, Research School of Physics and Engineering, The Australian National University, Canberra ACT 0200, Australia

Silicon polymorphs have been prepared by means of indentation of Si(100) surfaces. An indenter with a spherical tip has been used at a load of around 700 mN. A slow unloading rate of around 1.5 mN/s was used in order to form silicon polymorphs in the area indented and avoid amorphization. The formation of silicon polymorphs was verified subsequently by Raman spectroscopy. Related to the load applied only the formation of the meta-stable silicon phases Si-III, Si-IV and Si-XII was possible. Furthermore, electron transparent samples have been prepared in order to apply transmission electron microscopy and electron energy loss spectroscopy. A comparison of the Si $L_{2,3}$ edges of c-Si (Si-I) and Si-III/Si-XII will be presented. In addition, the spectra acquired are compared to simulations.

MI 8.7 Wed 15:00 Poster E

Quantitative STEM of Sn-Pd Nanoparticles with Core-Shell Structures — •Dietrich Häussler[1], Bernhard Schaffer[2,3], Ferdinand Hofer[3], and Wolfgang Jäger[1] — [1]Microanalysis of Materials, Christian-Albrechts-University Kiel, 24143 Kiel, Germany — [2]SuperSTEM Facility, Daresbury Laboratory, WA4 4AD, Warrington, UK — [3]Institute for Electron Microscopy, Graz University of Technology, 8010 Graz, Austria

Aberration-corrected high-resolution scanning TEM (STEM) and spectrum-imaging using X-ray (EDXS) and electron energy loss (EELS) signals are combined to quantitatively characterize crystalline Pd-Sn nanoparticles with a core-shell structure. The crystallographic structure of the particle core and shell was determined from the Fourier-filtered periodic back-transformations of STEM bright-field images and by applying a novel STEM diffraction-imaging (DI) technique. In an areal scan, convergent beam electron diffraction patterns are acquired, resulting in a set of data that contains the diffraction information for each image coordinate of the nanoparticle image. The evaluation of such '4D' datasets can be used to obtain by back-projection 2-dimensional dark-field maps that highlight regions of equivalent crystallographic structure and orientation. It is concluded that the approach can be used in monitoring chemical reactions or the degradation of composite nanoparticle materials. - B. Schaffer, now: Gatan GmbH, München, Germany. - We thank F. Liu and X. B. Zhang (Zhejiang University, Hangzhou, China) for provision of samples.

MI 8.8 Wed 15:00 Poster E

Cross-Section STEM Study of Bonding Concepts for Solar Cells — •Dietrich Häussler[1], Mert Kurttepeli[1], Stephanie Essig[2], Karen Derendorf[2], Frank Dimroth[2], and Wolfgang Jäger[1] — [1]Microanalysis of Materials, Christian-Albrechts-University Kiel, 24143 Kiel, Germany — [2]Fraunhofer Institute for Solar Energy Systems ISE, 79110 Freiburg, Germany

Crystalline silicon based multi-junction solar cells are a promising way to circumvent the conversion efficiency limits of conventional single-junction photovoltaic cells. In GaInP/GaAs/Si multi-junction solar cells, the visible and near infrared wavelength range of the solar spectrum is converted more efficiently when compared to solar cells produced conventionally.

As a decisive step of the technology a bonding process is aimed in which a GaInP/GaAs sub-cell is contacted with a Si substrate. The interface between GaAs and Si is of great importance for the total efficiency of this multi-junction cell.

Cross-section TEM samples are prepared from two types of GaInP/GaAs/Si multi-junction solar cell specimens. In order to investigate the bonding concepts, between the GaAs middle-cell and the Si bottom-cell, in the vicinity of the bonding interface, elemental distributions have been analyzed using STEM / EDXS and pictured using spectra and elemental maps. With the help of HRTEM micrographs an amorphous layer has been detected in the samples and gauged with high accuracy.

MI 8.9 Wed 15:00 Poster E

Dislocations and cracks in deformed GaN — •Ingmar Ratschinski[1], Hartmut S. Leipner[1], Jörg Haeberle[2], Reinhard Krause-Rehberg[2], Ludovic Thilly[3], Wolfgang Fränzel[2], Gunnar Leibiger[4], and Frank Habel[4] — [1]Interdisziplinäres Zentrum für Materialwissenschaften, Martin-Luther-Universität Halle-Wittenberg, 06099 Halle, Germany — [2]Institut für Physik, Martin-Luther-Universität Halle-Wittenberg, 06099 Halle, Germany — [3]Département de Physique et Mécanique des Matériaux, CNRS UPR 3346 Université de Poitiers, 86962 Futuroscope Chasseneuil Cedex, France — [4]Freiberger Compound Materials GmbH, 09599 Freiberg, Germany

Two inch (0001) GaN single crystals having a thickness of more than 3 mm and a density of in-grown dislocations in the magnitude of 10^6 cm^{-2} have been prepared for deformation experiments. GaN specimens of 3.0x3.0x10 mm^{-3} were fitted into iron cylinders. The samples were compressed more than 9 % at 700 °C and 800 °C. Furthermore, the as-grown (0001) surface was deformed at room temperature using a Vickers indenter. The samples were indented with loads in the range from 0.02 N to 4.90 N. Dislocations occur at all indentations whereas cracks are formed only at higher loads. The deformed samples were investigated by means of optical microscopy, scanning electron microscopy in secondary electron contrast and cathodoluminescence as well as positron annihilation. The results of compression experiments and indentation tests are compared.

MI 8.10 Wed 15:00 Poster E

Perturbed γ-γ angular correlation – applications in terms of elastic and plastic deformation in MAX phases — •Christoph Brüsewitz[1], Daniel Jürgens[1], Michael Uhrmacher[1], Ulrich Vetter[1], Hans Hofsäss[1], and Michel W. Barsoum[2] — [1]II. Physikalisches Institut, Georg-August-Universität Göttingen, Friedrich-Hund-Platz 1, D-37077 Göttingen, Germany — [2]Dep. Mat. Sci. & Eng., Drexel University, Philadelphia, PA 19104, USA

The perturbed γ-γ angular correlation (PAC) is used to investigate the neighbourhood of a probe atom at a scale of a few Ångström. The method utilizes the oscillating anisotropy of the depopulated intermediate state in the decay cascade of e. g. implanted ^{111}In. This state is split due to hyperfine interactions, originated by the local probe environment. This hyperfine interaction – here caused by an electric field gradient (EFG) at the Al-site of the MAX phase Ti_4AlN_3 – has been studied as a function of uniaxial load. The strength as well as the distribution of the EFG increase with pressure, which can be attributed to a reduction of the lattice spacing and an increase in dislocation density due to the deformation, respectively. Both aspects will be described theoretically by means of DFT calculations and a model concerning the stress field of dislocations. For a quantitative description of the distribution, one additionally has to take grain boundaries, point defects and inhomogeneous stresses into account. The analogy to high-resolution X-ray diffraction will be discussed as a supplemental issue. This work is supported by the DFG under contract HO 1125/19-1.

MI 8.11 Wed 15:00 Poster E

Precise determination of force microscopy cantilever stiffness from dimensions and eigenfrequencies — •Jannis Lübbe[1], Lutz Doering[2], and Michael Reichling[1] — [1]Fachbereich Physik, Universität Osnabrück, Barbarastraße 7, 49076 Osnabrück, Germany — [2]PTB, Nano- and Micrometrology, Bundesallee 100, 38116 Braunschweig, Germany

We demonstrate the non-destructive measurement of the stiffness of silicon cantilevers with tips as used for non-contact atomic force microscopy (NC-AFM) from the knowledge of cantilever dimensions and eigenfrequencies. The calculation of the stiffness is based on dimensions derived from scanning electron microscopy, optical microscopy and laser interferometry measurements. This yields stiffness values with an uncertainty of ±25% as the result critically depends on the thickness of the cantilever that is experimentally difficult to be determined. The uncertainty is reduced to ±7% when the measured fundamental eigenfrequency is included in the calculation and a tip mass correction is applied. The tip mass correction can be determined from the eigenfrequencies of the fundamental and first harmonic modes. Results are verified by tip destructive measurements of the stiffness with a precision instrument recording a force-bending curve yielding an uncertainty better than ±5%.

MI 8.12 Wed 15:00 Poster E

How to operate a non-contact atomic force microscope (NC-AFM) for ultra-high vacuum applications at the thermal noise limit — •Matthias Temmen[1], Jannis Lübbe[1], Sebastian Rode[2], Philipp Rahe[2], Angelika Kühnle[2], and Michael Reichling[1] — [1]Fachbereich Physik, Universität Osnabrück, Germany — [2]Institut für Physikalische Chemie, Johannes Gutenberg-Universität Mainz, Germany

The total noise in the frequency shift signal Δf of NC-AFM measurements consists of thermal noise, tip-surface interaction noise and instrumental noise from the detection and signal processing systems. We investigate the deflection noise spectral density (d^z) at the input of the frequence demodulator (PLL) as well as the frequency noise spectral density ($d^{\Delta f}$) at the output depending on cantilever properties and settings of the signal processing electronics for the case of negligible tip-surface interaction. For a quantification of noise figures we calibrate the cantilever deflection signal and determine the signal processing electronics transfer function to we derive predictions for the frequency noise spectral density for various filter settings and different levels of detection system noise (d^z_{ds}). We demonstrate that an optimised system with low noise signal detection operated with appropriate settings allows room temperature operation at the thermal noise limit of the NC-AFM with a significant bandwidth.

MI 8.13 Wed 15:00 Poster E

Test-objects for emission electron microscopy — •Sergej A. Nepijko and Gerd Schönhense — Institute of Physics, University of Mainz, 55099 Mainz, Germany

The resolution of emission electron microscopes approaches some nanometers which rises the need for new test-objects. Microfields due to a work function difference $\Delta\phi$ deform the trajectories of electrons forming the image which leads to a distortion of the emission electron microscopy image and a decrease of lateral resolution. We discuss three possibilities to fabricate test-objects, avoiding microfields: (i) Application of bias voltage $V_b = \Delta\phi$ between substrate and film. (ii) $\Delta\phi$ can be compensated by a relief h being equivalent to a smooth surface with distribution of potential $V(x,y)=-E_{ext}\cdot h(x,y)$, here E_{ext} is extractor voltage. The maximal lateral resolution is realized close to the centre of such a test-object. (iii) The third possibility is that the work function of a semiconducting substrate is adjusted by ion-implantation to the work function of a structured metal film.

MI 8.14 Wed 15:00 Poster E

Towards a deeper understanding of the dynamic properties of cantilever probes — •Maria-Astrid Schröter[1], Christiane Weimann[1], and Heinz Sturm[1,2] — [1]BAM - Federal Institute for Materials Research and Testing, Division 6.2, Unter den Eichen 87, 12205 Berlin, Germany — [2]Technische Universität Berlin, Pascalstraße 8-9, 10587 Berlin, Germany

In this poster a first step to measure and understand the dynamics of small and weak mechanical structures with ultra-high precision and sensitivity is presented. For this reason we analyze different types and geometries of silicon cantilevers with a special focus on T-shaped cantilevers. These cantilevers have an off-axis tip, so that tip-sample forces excite torsional vibrations.

For dynamic measurement of very small vibrating structures a hybrid of a Scanning Electron Microscope (SEM) and a Scanning Force Microscope (SFM) is used. One of the most obvious advantages using a combined system is that complementary analysis can be made at exactly the same sample position.

With the setup used here images of vibrating SFM cantilevers are presented to demonstrate the technique and to show, that torsional and flexural resonances can be distinguished. Beside DC-Type SE-signal, images of the superimposed AC-modulation as amplitude/phase shift and real/imaginary part amplitudes can be obtained using a lock-in amplifier synchronized to the excitation frequency. The analysis of vibrating structures includes several modes in the normal and torsional direction.

MI 8.15 Wed 15:00 Poster E

Beams of Highly Charged Ions for Micrometer Surface Structuring and Analysis — •Mike Schmidt[1], Günter Zschornack[2], Vladimir Ovsyannikov[1], and Jacques Gierak[3] — [1]DREEBIT GmbH, Zur Wetterwarte 50, 01109 Dresden — [2]TU Dresden, Helmholtzstr. 10, 01069 Dresden — [3]LPN/CNRS, 91460 Marcoussis, France

The particular properties of highly charged ions yield an interesting application potential in the field of micrometer surface structuring and analysis. Investigating these properties a demonstration setup was commissioned and is operated by a consortium of the Technische Universität Dresden and the DREEBIT GmbH in cooperation with the Helmholtz Zentrum Dresden-Rossendorf e.V. The setup features an ion source for highly charged ions (DRESDEN EBIS) on a Focused Ion Beam column which is connected to a target chamber with target transfer and positioning system for ion irradiation experiments. Accomplished investigations and experiments are presented. The work is supported by the EFRE fund of the EU and by the Freistaat Sachsen (Projects 100074113 and 100074115).

MI 8.16 Wed 15:00 Poster E

Proton Beam Writing in semiconductors: A new approach towards MEMS devices — Martina Schulte-Borchers, •Ulrich Vetter, and Hans Hofsäss — 2. Physikalisches Institut, Georg-August-Universität Göttingen, Friedrich-Hund-Platz 1, 37077 Göttingen, Germany

Proton Beam Writing is a fast direct-write method which allows for the fabrication of structures in bulk semiconductors as well as in photoresists in the micrometer range and with high aspect ratios. The structuring of semiconductors is enabled simply by scanning the proton beam over the semiconductor surface in the desired pattern. After irradiation of selected areas of a near surface region with protons in the energy range of a few MeV, structures can be revealed by etching under appropoate conditions, e.g. electrochemical etching for GaAs.
In this work, we will discuss the suitability of proton beam writing for MEMS fabrication on the example of GaAs. We will show our new results of three-dimensional structuring in only one lithography and irradiation step which employes the usage of different proton fluences instead of varying beam energies [1]. This fast and easy 3D production method and the smooth sidewalls as well as good structure quality make Proton Beam Writing an interesting tool for the manufacturing of prototypes in the field of MEMS devices.

[1] M. Schulte-Borchers, U. Vetter, T. Koppe, H. Hofsaess: "3D microstructuring in p-GaAs with Proton Beam Writing using multiple ion fluences", *provisionally accepted for publication in J. microeng. micromech.*