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Overview of Invited Tutorials, Invited Talks and Sessions

(lecture rooms EW(PN) 202, EMH 225 and TA 201; Poster E)

Invited Tutorials

MI 1.1	Sun	16:00–16:45	EW 202	**2D und 3D Röntgenbildgebung mit Synchrotronstrahlung** — •Tilo Baumbach
MI 1.2	Sun	16:45–17:30	EW 202	**Hochaufgelöste Röntgenbeugung an niedrigdimensionalen Halbleiterstrukturen** — •Michael Hanke
MI 1.3	Sun	17:30–18:15	EW 202	**New Lightsources and New Opportunities in Time-resolved Soft X-ray Spectroscopy** — •Martin Beye

Invited Talks

MI 2.1	Mon	9:30–10:15	EMH 225	**Chemical mapping at the atomic level using energy dispersive X-ray spectroscopy** — Peter Schlossmacher, •Bert Freitag, Dmitri Klenov, Adrian D Alfonso, Les Allen
MI 2.2	Mon	10:15–11:00	EMH 225	**Physical foundations of electron diffraction methods in the scanning electron microscope** — •Aimo Winkelmann
MI 5.1	Wed	9:30–10:15	TA 201	**Das Arkanum von Meissener Porzellan: Beharrung oder Wandel?** — •Christian Neelmeijer, Ulrich Pietsch, Heike Ulbricht
MI 5.2	Wed	10:15–11:00	TA 201	**From micro to nano - new X-ray techniques as depth sensors for material characterization** — •Ioanna Mantouvalou, Lars Lühl, Marcel Pagels, Wolfgang Malzer, Birgit Kanngiesser

Invited talk of the joint session "100 years since the Laue experiment: Topical aspects of diffraction and scattering" (Joint Session KR, BP, DF, MA, MI, MM, GP; related to SYXD)

MI 4.1	Tue	9:30–10:00	EMH 225	**The discovery of X-ray interferences, the role of characteristic radiation therein and potential applications of the Laue method in modern engineering.** — •Hans-Jürgen Ullrich, Siegfried Däbritz, Enrico Langer, Jürgen Bauch, Andreas Danilewsky, Peter Paufler

The symposium "100 years of X-ray diffraction: from the Laue-experiment to new frontiers" (SYXD) related to the joint sessions MI 4 and MI 7 takes place on Monday afternoon 15:00–17:30 in H 0105. See SYXD for details of the program.

Sessions

MI 1.1–1.3	Sun	16:00–18:15	EW 202	**Innovations in synchrotron X-ray studies**
MI 2.1–2.7	Mon	9:30–12:15	EMH 225	**TEM- and SEM-based material analysis**
MI 3.1–3.4	Mon	12:45–13:45	EMH 225	**Scanning probe microscopy**
MI 4.1–4.11	Tue	9:30–12:45	EMH 225	**100 years since the Laue experiment: Topical aspects of diffraction and scattering (Joint Session KR, BP, DF, GP, MA, MI, MM; related to SYXD)**
MI 5.1–5.7	Wed	9:30–12:15	TA 201	**X-ray spectrometry and analysis of works of art**
MI 6.1–6.8	Wed	12:30–14:30	TA 201	**X-ray imaging, holography and tomography**
MI 7.1–7.4	Wed	15:00–17:30	Poster E	**Poster – 100 years since the Laue experiment: Topical aspects of diffraction and scattering (Joint Session KR, BP, DF, GP, MA, MI, MM; related SYXD)**
MI 8.1–8.16	Wed	15:00–17:30	Poster E	**Poster – Microanalysis and microscopy**

Mitgliederversammlung des Fachverbandes Mikrosonden

Montag 14:00 EMH 225

- Bericht des Fachverbandsvorsitzenden
- Planung der DPG-Tagung 2013
- Verschiedenes

MI 1: Innovations in synchrotron X-ray studies

This tutorial is related to the centenary of the famous Laue experiment of X-ray diffraction. It deals with various topical aspects of the application of synchrotron radiation covering a broad range of materials and methods.

Chair: E. Langer

Time: Sunday 16:00–18:15 Location: EW 202

Tutorial MI 1.1 Sun 16:00 EW 202

2D und 3D Röntgenbildgebung mit Synchrotronstrahlung — •Tilo Baumbach — Karlsruher Institut für Technologie, Institut für Synchrotronstrahlung, 76344 Eggenstein-Leopoldshafen

Illustriert durch Materialforschung und Lebenswissenschaften entwickelt dieser Einführungsvortrag einen Überblick zu den wichtigsten physikalischen Prinzipien der Bildentstehung und Rekonstruktion. Das heute verfügbare methodische Ensemble erlaubt die weitgehend zerstörungsfreie Abbildung von Strukturen im Realraum mit Auflösung bis zur Nanoskala. Die besonderen Eigenschaften der Synchrotronstrahlung und ein rasanter Fortschritt bei abbildenden Röntgenoptiken und Detektoren, präziser Manipulatorik und leistungsfähigen Algorithmen ermöglicht die Verwendung und Kombination vielfältiger physikalischer Kontrastmechanismen wie z.B. Absorption, kohärente und inkohärente Streuung und Spektroskopie zur Abbildung von Realstrukturen inklusive der 2D und 3D Verteilung statistischer Struktureigenschaften. Variable örtliche Auflösung und die Kombination komplementärer Abbildungsmethoden ermöglichen korrelierte und hierarchische Bildgebung z.B. biologischer Systeme - vom Organismus zu einzelnen Organen, Gewebe, Zell- und subzellularer Strukturen. Zeitlich und örtlich hochauflösende abbildende Röntgentechniken sind wichtige Werkzeuge zur Erforschung der Mikro- und Nanowelt und deren Veränderung in Abhängigkeit von äußeren und inneren Bedingungen. Das Verständnis technischer oder biologischer Systeme wiederum fördert unsere Fähigkeit der gezielten Beeinflussung von makroskopischen Eigenschaften und Funktionalitäten.

Tutorial MI 1.2 Sun 16:45 EW 202

Hochaufgelöste Röntgenbeugung an niedrigdimensionalen Halbleiterstrukturen — •Michael Hanke — Paul-Drude-Institut für Festkörperelektronik

Hochaufgelöste diffuse Röntgenbeugung ist eine weitestgehend etablierte Methode, um geringste mechanische Deformationen in Nanostrukturen nachzuweisen. So lässt sich neben der Form und Positionskorrelation sehr genau die Variation des lokalen Gitterparameters bestimmen. Eine zentrale Ursache für diese mechanischen Deformationen stellt die sich beim Wachstum einstellende chemische Zusammensetzung dar. Fern von Absorptionskanten verschwindet jedoch deren direkter Einfluss auf die diffuse Röntgenstreuung nahezu vollständig, während die mechanische Deformation als Kontrastmechanismus dominiert. Insofern lässt sich indirekt von der diffusen Streuung ausgehend, über den Umweg der Deformation, auf die chemischen Profile schließen. Ohne deren detaillierte Kenntnis lassen sich die zugrunde liegenden, oft hochkomplexen Wachstums- und Selbstorganisationsphänomene kaum verstehen.

Der Vortrag gibt zunächst einen Überblick über verschiedene Verfahren der hochaufgelösten Röntgenbeugung und die Simulation diffuser Streuintensitäten im Rahmen der kinematischen Näherung. Beispielhaft wird die Strukturaufklärung an Objekten wie Nanowires, Einzelquantenpunkten, Quantenpunktmolekülen, deren Entwicklungsstadien sowie an freistehenden und vergrabenen Doppelringstrukturen im Nanometerbereich diskutiert.

Tutorial MI 1.3 Sun 17:30 EW 202

New Lightsources and New Opportunities in Time-resolved Soft X-ray Spectroscopy — •Martin Beye — Helmholtz-Zentrum Berlin, Berlin, Germany

In this tutorial, I will quickly introduce novel pulsed X-ray sources (especially free-electron lasers like FLASH in Hamburg and LCLS in Stanford, USA) that enable soft X-ray spectroscopy on the ultrafast timescale relevant for electron dynamics and chemical bond making and breaking. After showing how we solve the experimental challenges connected with these new sources, I will feature some results from spectroscopy on correlated materials, where the ultrafast timescale allows to disentangle electronic and lattice degrees of freedom. We study how the excitation with a femtosecond laser destroys orbital and/or charge ordering phenomena. I will close with results on spectroscopy of chemical reactions where we can make full use of the element specificity of soft X-rays to study the dynamics of molecular bonds during chemical reactions. I will show results from reactions in the liquid phase as well as catalytic reactions on surfaces. A fundamental understanding of the ultrafast processes is relevant for applications in the chemical industry. This might allow to specifically tailor active surfaces, for example new catalytic converters in car exhausts.

MI 2: TEM- and SEM-based material analysis

Chair: E. Langer

Time: Monday 9:30–12:15 Location: EMH 225

Invited Talk MI 2.1 Mon 9:30 EMH 225

Chemical mapping at the atomic level using energy dispersive X-ray spectroscopy — Peter Schlossmacher[1], •Bert Freitag[1], Dmitri Klenov[1], Adrian D Alfonso[2], and Les Allen[2] — [1]FEI Company, Building AAE, Achtseweg Noord 5, Eindhoven, The Netherlands — [2]School of Physics, University of Melbourne, Parkville, Victoria 3010, Australia

We demonstrate chemical mapping at the atomic level using energy dispersive X-ray spectroscopy (EDS) in Cs-corrected scanning transmission electron microscopy (STEM). The combination of the increase in current in an atomic sized probe by probe Cs-correction and the increase in sensitivity of the Super-X detector allows acquisition of such results fast and reliable. Atomic chemical maps of the strontium and titanium sub lattice in SrTiO3 are obtained in minutes with a high sampling (256*256 pixels). The signal strength is much higher when compared to previously reported results using conventional EDS detectors. Even the GaAs dumbbell of 141 pm distance can be resolved in the chemical EDS map using the Ga and As L and K-edges. Examples on interfaces and complex oxide materials are discussed.

Theoretical Bloch wave simulations are compared with the experimental GaAs results.The simulations for EDS mapping assume that the cross section for X-ray emission is proportional to the total cross section for K or L shell ionization for a detector which samples the full solid angle and all possible energy losses above the ionization threshold.

Invited Talk MI 2.2 Mon 10:15 EMH 225

Physical foundations of electron diffraction methods in the scanning electron microscope — •Aimo Winkelmann — Max-Planck-Institut für Mikrostrukturphysik, Halle (Saale)

Electron diffraction techniques like Electron Backscatter Diffraction (EBSD) and Electron Channeling Patterns (ECP) are powerful tools for the crystallographic analysis of materials in the scanning electron microscope (SEM). Pronounced improvements in applications of these methods can be expected with quantitative theoretical and experimental access to the physics of the underlying diffraction processes.

It is demonstrated how the Bloch-wave approach of dynamical electron diffraction theory can be applied for the quantitative simulation of the Kikuchi diffraction patterns observed in EBSD and ECP in the SEM. Analogies are drawn to Kikuchi pattern formation in transmission electron microscopy (TEM). By energy-resolved experimental measurements, the correlations between the energy of the elastically and inelastically scattered electrons and their diffraction effects were

investigated. It is shown that quasi-elastic nuclear recoil is a central incoherent process relevant for the formation of Kikuchi patterns. Also, under certain conditions, inelastically scattered electrons in the SEM can show more pronounced diffraction effects than the elastic electrons.

MI 2.3 Mon 11:00 EMH 225

Characterization of thin-film solar cells by means of various scanning electron microscopy methods — •Daniel Abou-Ras — Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin, Germany

This contribution gives an overview of the various analysis techniques available in scanning electron microscopy, applied to thin-film solar cells as important contribution for their optimization. Specifically, electron backscatter diffraction (EBSD) and energy-dispersive X-ray spectrometry (EDX) give information on microstructure and elemental distribution, while electron-beam-induced current (EBIC) and cathodoluminescence (CL) measurements provide access to electrical and optoelectronic properties. The combination of EBSD, EDX, EBIC, and CL on identical sample positions enhances the information content of the analysis results substantially. Various examples of such analyses will be given.

MI 2.4 Mon 11:15 EMH 225

Aktuelle Ergebnisse mit den HRTEM JEOL JEM-ARM 200F — •Jürgen Heindl — JEOL (Germany) GmbH, Oskar-von-Miller-Str. 1a, 85386 Eching

Das JEOL JEM-ARM 200F ist das erste Transmissions-Elektronen-Mikroskop das von Grund auf ausschließlich für den Betrieb mit Korrektoren für die Aberration der Linsen entwickelt wurde. Es können sowohl die limitierenden Aberrationen im STEM (Scanning Transmission Electron Microscopy) Betrieb (CESCOR) als auch die des Objektivs in der hochauflösenden Transmissionselektronenmikroskopie (HREM, CETCOR) bzw. beide ausgeglichen werden. Ergänzend kann das System an Stelle der Schottky-Feldemissionskathode mit einer völlig neuartigen kalten Feldemissionsquelle (ColdFEG) mit sehr geringer Energiebreite mit hoher Intensität ausgerüstet werden. Im STEM-Betrieb zeigt die ColdFEG deutlich verbesserte Abbildungsleistungen gegenüber einer Schottky-Quelle, was bei der direkten Abbildung der H-Atome in Yttriumhydrid gezeigt wird. Im HREM-Betrieb ist die ColdFEG anderen Lösungen überlegen, weil die geringe Energiebreite der Primärelektronen unmittelbar auf die Auflösung verbessert. Der Nachteil einer polychromen Beleuchtung entfällt; die Bildergebnisse sind vollumfänglich simulierbar. Ein vollanalytisches JEM-ARM200F zeichnet sich durch den neuen JEOL Centurio-EDX- Detektor aus. Der Centurio-Detektor erreicht seine sehr hohe Empfindlichkeit durch eine aktive Fläche von 100 mm2 und einen Raumwinkel von 1 sr. Mit der neuen ColdFEG wird ein Echtzeit-EDX-Mapping an SrTi03 und GaAs gezeigt.

MI 2.5 Mon 11:30 EMH 225

Chemical analysis between the mm and atomic scale using silicon drift detectors — •Meiken Falke, Ralf Terborg, Tobias Salge, Samuel Scheller, Daniel Goran, Ulrich Waldschläger, and Martin Rohde — Bruker Nano GmbH, Schwarzschildstr.12, 12489 Berlin, Germany

Silicon drift detectors (SDDs) were originally developed to avoid the use of liquid nitrogen for cooling X-ray spectrometers during space missions. Now SDDs are about to revolutionize energy dispersive X-ray spectroscopy (EDS), since they provide fast and robust readout of high X-ray input count rates with superb energy resolution, particularly at the low energy end, enabling the detection and quantification of even light elements such as Boron. SDDs are used for chemical analysis in integrated multiple detector solutions as well as in powerful stand- alone systems for standard and aberration corrected transmission (TEM) and scanning electron microscopy (SEM). The speed of data acquisition and analysis can be combined with other techniques giving crystallographic information or higher energy resolution, such as EBSD on SEM and EELS on TEM. Furthermore, fast EDS data acquisition for tomography and the respective reconstruction of element compositions in 3D are reality now. We want to explain the SDD technology and show data achieved so far, which provide a vision for future possibilities.

MI 2.6 Mon 11:45 EMH 225

Characterization of novel capacitors for energy storage on the basis of 0-3 composites — •Jens Glenneberg[1], Alexandra Buchsteiner[1], Mandy Zenkner[1], Thomas Grossmann[1], Claudia Erhardt[1], Stefan G. Ebbinghaus[1], Martin Diestelhorst[1], Sebastian Lemm[1], Wolfram Münchgesang[1], Horst Beige[1], Gerald Wagner[1,2], and Hartmut S. Leipner[1] — [1]Martin-Luther-Universität Halle-Wittenberg — [2]Universität Leipzig

Thin film capacitors with high energy densities represent an alternative solution for energy storage issues. Their advantages are quick charging and discharging times, combined with long lifetimes as well as high robustness associated with nearly no maintenance. The capacitors under development are realized by embedding nano-particles of different perovskites ($BaTiO_3$, $Ba(Ti,Ge)O_3$, $CaCu_3Ti_4O_{12}$) into either an organic or inorganic matrix (0-3-composite). Depending on the applied matrix, the perovskite particles are coated with a specific surfactant in order to achieve a uniform distribution. The distribution as well as the size of the perovskites-particles determines the electrical properties of the capacitor dielectrics. In order to investigate the microstructure, the nano-particles and the surrounding matrix are imaged by environmental scanning electron microscopy (ESEM) in both secondary electron contrast (SE) and backscattered electron contrast (BSE) as well as by transmission electron microscopy (TEM). To acquire compositional information, additional energy-dispersive X-ray spectroscopy (EDS) has been carried out.

MI 2.7 Mon 12:00 EMH 225

Oberflächenanalytik für die Untersuchung tribologischer Prozesse wie Verschleiß, Grenzschichtformierung und Schmierstoffdegradation molekularer Schmierstoffe mittels ToF-SIMS an realen tribologischen Systemen — •Ullrich Gunst — Westfälische Wilhelms-Universität Münster

Die Eigenschaften tribologischer Systeme werden zu einem wesentlichen Teil durch Struktur und Zusammensetzung ihrer Systembestandteile bestimmt, insbesondere durch die Eigenschaften der wechselwirkenden Oberflächen, molekularen Strukturen und Grenzflächen. Die Gebrauchsdauer und die Entwicklung des Betriebsverhaltens von tribologischen Systemen hängen stark von den Änderungen dieser Zusammensetzungen und Strukturen ab. Die oberflächenanalytische Methode der Time-of-Flight SIMS verfügt über eine ganze Anzahl von Betriebsmoden, um Zugang zu diesen Zusammensetzungen und Strukturen zu erhalten sowie die Änderung tribologischer Systemeigenschaften und die verknüpften tribologischen Prozesse zu erforschen. Der Einsatz komplementärer oberflächenanalytischer Methoden (XPS, TEM etc.) liefert weitere wertvolle analytische Informationen zum Status und zur Entwicklung von atomaren und molekularen Zusammensetzungen als auch Strukturen tribologischer Systemoberflächen mit deren Betriebszeit. Der Beitrag zeigt den Einsatz und Ergebnisse oberflächenanalytischer Methoden - insbesondere der ToF-SIMS - zur Charakterisierung von Systemoberflächen, tribologischer Vorgänge und der Korrelation dieser zum Betriebsverhalten am Beispiel fettgeschmierter schnelllaufender Wälzlager bei Variation der verwendeten Schmierfette auf.

MI 3: Scanning probe microscopy

Chair: H. S. Leipner

Time: Monday 12:45–13:45 Location: EMH 225

MI 3.1 Mon 12:45 EMH 225

Chemical nano-imaging of thin surface layers by combining AFM with tip-localized infrared spectroscopy — Sergiu Amarie and •Fritz Keilmann — MPI für Quantenoptik, Garching

We have extended the scattering near-field microscope (s-SNOM) that returns an optical image together with topography, both at <20 nm resolution, by operating with broadband infrared illumination. Thus a continuous infrared spectrum from 4 to 15 micron wavelength can be recorded at each scanned pixel. As this covers the "molecular fingerprint" region one can determine the local chemical composition.

The probing depth is about equal to the spatial resolution, of the order of the tip radius of typically 20 nm. Our new method should find many applications requiring a quantitative material analysis at the nanoscale, be it in general analytical chemistry, nanofabrication, mineralogy or condensed matter physics.

Results of imaging hard biological matter will be presented, where phosphate and carbonate nanocrystals evoke especially bright contrasts due to infrared phonon resonance, and where new insights into biomineral growth and decay mechanisms of biological and even medical interest can be expected.

MI 3.2 Mon 13:00 EMH 225

New generation micro vacuum gauge for ultra high vacuum measurements using modified AFM tips — •Amra Avdic, Anna-Maria Lausch, Alois Lugstein, and Emmerich Bertagnolli — Solid State Electronics Institute, Vienna University of Technology, Floragasse 7, 1040 Vienna, Austria.

Every modern high vacuum and ultrahigh vacuum system relies on ionization gauges for pressure measurements under 10E-3 Torr. For the first time we present the micro vacuum gauge (MVG) fabricated by modification of commercial available metall-coated Si Atomic Force Microscopy (AFM) tips with the lateral resolution $<$ 1um. As any cold-cathode based vacuum gauge, our MVG consist of two properly insulated electrodes integrated on the AFM tip, forming coaxial embodiment. The conductive AFM probes are insulated with a Si3N4 layer using plasma enhanced chemical vapour deposition and subsequently coated by a second metallic layer, which later forms a ring shaped collector around the tip. Our self aligned approach for MVG formation comprises focused ion beam (FIB) machining and isotropic Reactive Ion Etching (RIE). The RIE is used to selectively etch the insulation and expose the conductive tip, thereby forming the cavity between the immediate tip (emitter) of about 60 nm in diameter and the ring (collector) separated by a cavity of about 600nm. After the modification the MVG is mounted into the Crossbeam Neon40EsB and tested upon the gas injection available at this system.

MI 3.3 Mon 13:15 EMH 225

All-Metal Cantilevers for Kelvin Force Microscopy — •Raul D. Rodriguez, Franziska Lüttich, Susanne Müller, Daniel Lehmann, and Dietrich R. T. Zahn — Semiconductor Physics, Chemnitz University of Technology, Reichenhainer Str. 70. Chemnitz, 09126, Germany

Kelvin force microscopy (KFM) has become a key analytical technique in several research areas from organic optoelectronic devices to single biomolecular interactions. Ultra sharp, wear-proof, electrical conductors and tapping-mode compatible are key characteristics of cantilevers for KFM. In this work we introduce a new class of all-metal cantilevers that meet all the requirements mentioned above. From metallic microwires as precursors gold and silver probes have been fabricated using a dedicated device for tip fabrication which enhances reproducibility and control of the cantilever dimensions. The resonance frequency of the cantilevers could be tuned in a range from 10 to 400 kHz in air. Such probes were characterized by scanning electron microscopy and their performance was evaluated by analyzing organic semiconductor devices and commercial probe characterizers. The advantages and limitations of these novel probes are discussed and compared to conventional metal-coated silicon and silicon nitride cantilevers.

MI 3.4 Mon 13:30 EMH 225

Extracting intrinsic cantilever properties from thermal noise — •Jannis Lübbe[1], Matthias Temmen[1], Philipp Rahe[2], Angelika Kühnle[2], and Michael Reichling[1] — [1]Fachbereich Physik, Universität Osnabrück, Barbarastraße 7, 49076 Osnabrück, Germany — [2]Institut für Physikalische Chemie, Johannes Gutenberg-Universität Mainz, Jakob-Welder-Weg 11, 55099 Mainz, Germany

We describe a method to determine the eigenfrequency f_0, the intrinsic quality factor Q_0 and the stiffness k from the cantilever deflection noise spectral density $d^z_{\mathrm{th}}(f)$ of a thermally excited cantilever in an ultra-high vacuum environment. The precision of the f_0 value as derived from the peak position is of the order of 0.1% and solely limited by thermal drift. The Q_0 value can be determined from a fit with a precision of 10%. Most delicate is the determination of k derived from the fit of the $d^z_{\mathrm{th}}(f)$ curve. This measurement requires a calibration of the deflection measurement and utmost care has to be taken to avoid any excitation of the cantilever other than by thermal noise. An alternative approach is to determine dynamic stiffness from a measurement of the spectral density $d^{\Delta f}_{\mathrm{th}}(f)$ of the frequency demodulated thermal noise signal. This approach can easily be extended to a measurement of higher order eigenmodes as the spectral analysis of $d^{\Delta f}_{\mathrm{th}}(f)$ can mostly be restricted to frequencies below 100 Hz and does not require high spectral resolution. Results obtained from the two methods are in good agreement with each other.

MI 4: 100 years since the Laue experiment: Topical aspects of diffraction and scattering (Joint Session KR, BP, DF, GP, MA, MI, MM; related to SYXD)

Time: Tuesday 9:30–12:45 Location: EMH 225

Invited Talk MI 4.1 Tue 9:30 EMH 225

The discovery of X-ray interferences, the role of characteristic radiation therein and potential applications of the Laue method in modern engineering. — •Hans-Jürgen Ullrich[1], Siegfried Däbritz[2], Enrico Langer[2], Jürgen Bauch[1], Andreas Danilewsky[3], and Peter Paufler[4] — [1]Institute of Materials Science at the TU Dresden — [2]Institute for Solid State Physics at the TU Dresden — [3]Institute of Crystallography at the University of Freiberg — [4]Institute of Structural Physics at the TU Dresden

It was during a noteworthy conversation between Paul Peter Ewald and Max von Laue in the English Garden in Munich in early 1912 that the foundation for the discovery of X-ray interferences was laid. They were debating which effects can be expected when short-wave electromagnetic radiation is allowed to impinge on crystals, and in a eureka moment Max von Laue theorized that interference phenomena are caused.

The first diffraction experiments were founded on the notion that the interferences in question might be characteristic radiation. The effect subsequently searched for was eventually discovered by Walther Kossel and his colleagues in 1934 (interferences from lattice sources, Kossel interferences). It is with this fact in mind that our lecture will look at the significance of Laue and Kossel diffraction patterns during the initial research into X-ray physics and their influence on all further academic work in this area. In the past, Laue's discovery was mainly applied within the natural sciences, but more recently the Laue method has also been employed successfully in engineering, for example:

- for quality assessment procedures used within the framework of semiconductor chip production
- as diagnostic techniques for gas turbine blades.

MI 4.2 Tue 10:00 EMH 225

Thermal diffuse scattering as a complementary tool in the study of lattice dynamics — Björn Wehinger, Alexeï Bosak, and •Michael Krisch — ESRF, 6 Rue Jules Horowitz, BP 220, 38043 Grenoble, France

Thermal diffuse scattering (TDS) in combination with inelastic x-ray scattering (IXS) and lattice dynamics calculations allows the reconstruction of the lattice dynamics in the entire Brillouin zone. X-ray scattering by thermally populated phonons in crystals reduces the intensity of Bragg spots and substantially increases the intensity of the diffuse scattering which has a rich structure in reciprocal space [1,2]. In combination the two techniques can serve as a rigorous benchmark for parameter free lattice dynamics calculations [3]. The proposed method can be used for the precise detection of mode softening, for the study of lattice dynamics under extreme conditions and for time resolved measurements. In metallic systems it is possible to map the Fermi surface in tree dimensions by directional tracing of Kohn anomalies [4]. The presented results on β-tin illustrate the functionality of the proposed combined approach with new insights into the dynamical properties on this system.

[1] Wooster, Diffuse X-ray reflections from crystals, Clarendon Press, Oxford (1962)

[2] Xu RQ, Chiang TC, Z. Kristallogr. 220, 1009 (2005)

[3] A. Bosak, et al., Z. Kristallogr. preprint: doi: 10.1524/zkri.2012.1432
[4] A. Bosak, et al., PRL, 103, 076403 (2009)

MI 4.3 Tue 10:15 EMH 225

Brillouin scattering of ultrashort optical and x-ray pulses from quasi-monochromatic phonon wavepackets — •Marc Herzog[1], André Bojahr[1], Jevgenij Goldshteyn[2], Steffen Mitscherling[1], Wolfram Leitenberger[1], Dmitry Khakhulin[3], Michael Wulff[3], Ionela Vrejoiu[4], Roman Shayduk[2], Peter Gaal[1], and Matias Bargheer[1,2] — [1]Institut für Physik und Astronomie, Universität Potsdam, Potsdam, Germany — [2]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin, Germany — [3]Max-Planck-Institut für Mikrostrukturphysik, Halle, Germany — [4]European Synchrotron Radiation Facility, Grenoble, France

We excite a $SrRuO_3$ thin film transducer epitaxially grown on a $SrTiO_3$ substrate with pulse trains of ultrashort laser pulses. Each laser pulse launches single bipolar strain pulses of broad bandwidth into the substrate [1] which coherently add up to form a quasi-monochromatic sub-THz phonon wavepacket. The generation and dynamics of these phonon pulses is investigated by Brillouin scattering using visible and hard x-ray photons. The combination of both methods reveals the excited narrow phonon spectrum as well as the phonon lifetime which is on the order of a few 100 ps in the considered frequency range. This lifetime is explained by anharmonic phonon interactions.

[1] Thomsen *et al.*, Phys. Rev. B 34, 4129 (1986).

MI 4.4 Tue 10:30 EMH 225

Following Strain-Induced Mosaicity Changes of $PbZr_{0.2}Ti_{0.8}O_3$ Thin Films by Ultrafast Reciprocal Space Mapping — •Daniel Schick, André Bojahr, Marc Herzog, Peter Gaal, and Matias Bargheer — Institut für Physik & Astronomie, Universität Potsdam, Karl-Liebknecht-Str. 24-25, 14476 Potsdam/Golm

We studied the propagation of coherent sound waves in a $PbZr_{0.2}Ti_{0.8}O_3$ (PZT) - $SrRuO_3$ (SRO) bilayer sample after optical excitation of the metallic SRO layer. We observed changes of the out-of-plane lattice constant and structure factor of the ferroelectric PZT layer that can be exclusively attributed to the transient strain wave launched from within the SRO layer. In addition to this we are also able to follow in-plane structural dynamics simultaneously utilizing a new ultrafast reciprocal space mapping technique. Thereby we observed a transient change of the mosaicity of the PZT layer on a ps timescale which is again directly coupled to the coherent sound wave travelling through the layer.

MI 4.5 Tue 10:45 EMH 225

Analysis of the size and shape of colloidally prepared nanocrystals by Rietveld refinement — •Holger Borchert, Xiaodong Wang, Marta Kruszynska, Joanna Kolny-Olesiak, and Jürgen Parisi — University of Oldenburg, Department of Physics, Energy and Semiconductor Research Laboratory, Carl-von-Ossietzky Str. 9-11, 26129 Oldenburg, Germany

Many properties of colloidal nanocrystals can be tuned by controlling the crystal size and shape. Examples are the quantum size effect in the case of semiconductors or size-dependent catalytic properties in the case of metals. Establishing correlations between the structure and other properties relevant for applications requires suitable methods to characterize the size and shape of nanocrystals. Most evident are imaging techniques like transmission electron microscopy (TEM). However, as a disadvantage only a limited number of particles can be evaluated. Powder X-ray diffraction (XRD), in contrast, probes a large ensemble of nanocrystals, but it remains a challenge to reliably extract information on the crystallite size and shape from XRD data. In this work, colloidal chemistry was used to prepare mono- and bimetallic Pt and Pt/Sn nanocrystals as well as semiconductor nanocrystals of ZnO, CuInS2 and composite particles consisting of CuInS2 and Cu2S. The samples were analyzed by TEM and XRD. Rietveld refinement of XRD data was done with a program enabling to simulate also anisotropic crystallite shapes. This approach turned out to be suitable for the determination of the average size and shape, in particular also in the case of nanorods and composite nanomaterials.

MI 4.6 Tue 11:00 EMH 225

Strain measurement in semiconductor nanostructures by convergent electron nanoprobe diffraction — •Knut Müller[1], Andreas Rosenauer[1], Marco Schowalter[1], Josef Zweck[2], Rafael Fritz[3], and Kerstin Volz[3] — [1]Universität Bremen, Germany — [2]Universität Regensburg, Germany — [3]Universität Marburg, Germany

The fundamental but simple Bragg law is exploited to measure lattice strain with a precision of $7 \cdot 10^{-4}$ and a spatial resolution of $0.5-0.7$ nm directly from convergent beam electron diffraction (CBED) patterns. In particular, we present 3 different algorithms for pattern recognition to measure CBED reflection positions accurately: The first detects edges in a patch around each CBED disc and iteratively finds all edge points which lie on the disc boundary by circle fitting. The second takes a rotational average in the patch and maximises the gradient in radial direction by optimising the centre of the rotational average. The third and fastest method exploits cross-correlations between each reflection patch and different types of masks. Besides results for a 350 nm wide $In_xGa_{1-x}N_yAs_{1-y}/GaAs$ highly strained quantum layer stack with alternating compressive/tensile strain, we present prospects for the operation and acquisition hardware of a TEM, directly deduced from the three algorithms above to allow for a fast strain map acquisition directly at the microscope in future. For the present study we operated an FEI Titan (S)TEM microscope in STEM mode to record a series of energy filtered CBED patterns on CCD.

15 min. break

MI 4.7 Tue 11:30 EMH 225

Theory of Electron Magnetic Circular Dichroism — •Jan Rusz — Dept. of Physics and Astronomy, Uppsala University, Sweden

Electron magnetic circular dichroism (EMCD) is an electron microscopy analogue of the established x-ray magnetic circular dichroism, that can provide atom-specific spin and orbital moments. EMCD, compared to its x-ray counterpart, offers a potential of significantly better spatial resolution, potentially in the Angstrom range. Presently, the technique is limited by difficulties of reaching sufficient signal to noise ratio and complexity of the accompanying dynamical diffraction effects, both of which make quantitative analysis demanding and prone to systematic errors.

We present recent theoretical developments in the field of EMCD, namely, 1) influence of plural scattering and associated spectral post-processing corrections; 2) convergence of the dynamical diffraction calculations in electron energy loss spectroscopy (ELNES), and 3) decomposition of the signal in diffraction plane to maps of various irreducible operators, such as orbital and spin magnetic moments, number of holes, orbital and spin-orbital anisotropy tensors.

These developments improve our understanding of deviations of recent quantitative EMCD experiments from expected values, allow more accurate predictions of the signal distribution, and uncover the wealth of information contained in electron energy loss spectra and thus aid in improving the methods of extraction of the magnetic signal from experimental datasets.

MI 4.8 Tue 11:45 EMH 225

Magnetic structure of magnetoelectric $NdFe_3(BO_3)_4$ under applied magnetic fields — •Jorge E. Hamann-Borrero[1], Sven Partzsch[1], Sergio Valencia[2], Claudio Mazzoli[3], Christian Hess[1], A. Vasiliev[4], L. Bezmaternikh[5], Bernd Büchner[1], and Jochen Geck[1] — [1]IFW-Dresden — [2]Helmholtz-Zentrum-Berlin — [3]ESRF, Grenoble, France — [4]Moscow State University, Russia — [5]L. V. Kirensky Institute of Physics, Russian Academy of Sciences, Krasnoyarsk, Russia

The magnetic structure of the magneto-electric $NdFe_3(BO_3)_4$ is studied by means of Resonant X-ray Magnetic Scattering (RXS) at the Nd $L_{2,3}$ and Fe K edges. The temperature dependent experiments show below $T_N = 30$ K the appearance of commensurate (CM) magnetic superlattice reflections with Miller indices $(0, 0, l \pm 3/2)$ (where $l = 3n$ and n = integer). By further cooling, at $T_{ICM} \sim 16$ K, a transition into an incommensurate (ICM) spin helix structure is observed in agreement with recent neutron experiments[1, 2]. Detailed mean field based analysis of the x-ray diffracted intensities show, that the Nd and Fe magnetic sublattices behave differently. In fact the magnetization of the Nd sublattice is induced by the Fe moments. At $T<T_{ICM}$, by applying an external magnetic field **B** parallel to the *ab*-plane, the magnetic structure suffers a reorientation transition from a spin helix configuration to a collinear structure where all the moments align perpendicular to **B** in the basal plane.

[1] M. Janoschek et al. Phys. Rev. B, 2010, 81, 094429
[2] P. Fischer et al. Jour. Phys. Cond. Matt., 2006, 18, 7975-7989(15)

MI 4.9 Tue 12:00 EMH 225

Monoclinic Symmetry in Barium Titanate — Christian Eisenschmidt, •Hans Theo Langhammer, and Günther Schmidt — Martin-Luther-Universität Halle-Wittenberg, Institut für Physik

The tetragonal-orthorhombic phase transition of barium titanate crystals has been investigated by XRD measurements during slow cooling. Additional diffuse scattering intensity between the (002) and (200) reflexes as well as a shift of the (200) reflex towards higher 2Θ values develop with decreasing temperature and time. The tetragonal-orthorhombic phase transition takes place obviously via a monoclinic intermediate stage. This can be understood by assuming this order-disorder phase transition is initiated by increasing short-range order (SRO) of Ti ions followed by rearranging of Ba ions similar to the 'tetragonal' SRO below the Burns temperature above the transition cubic-tetragonal. This results, finally, in nucleation and transition to the long-range ordered orthorhombic phase. Conclusions of the proposed mechanism in compositionally disordered systems like Ba(Ti,Sn)O3, PMN-PT et al. are discussed.

MI 4.10 Tue 12:15 EMH 225

Multilayer Optics for Modern X-ray Analytical Equipment — •Andreas Kleine, Jörg Wiesmann, Bernd Hasse, Jürgen Graf, Uwe Heidorn, Steffen Kroth, Frank Hertlein, and Carsten Michaelsen — Incoatec GmbH, Max-Planck-Str. 2, 21502 Geesthacht, Germany

Even 100 years after the first publication of the Bragg equation, there are current developments which are still mainly based on this fundamental law. One of these developments are multilayer optics which are used for beam shaping of X-rays e.g. for focusing the X-rays onto the sample. The multilayer optics simulate an artificial crystal with the typical distance d of the Bragg equation. It is advantageous that this distance can be changed and thus adapted to the specific application and setup. The development of multilayer optics allowed a performance increase of modern diffractometers by more than one order of magnitude.

In this contribution, we will give an overview of current developments of multilayer optics. We will explain the design and the manufacturing process of the optics and give some examples of typical applications which benefit from the new possibilities, especially in combination with modern microfocus X-ray sources. Applications like GISAXS, high-pressure XRD or micro-diffraction known from synchrotrons, can be realized now in the home-lab.

MI 4.11 Tue 12:30 EMH 225

Reconstruction phenomena at the interfaces of $LaCoO_3$ single films: A resonant x-ray reflectivity study — •Jorge E. Hamann-Borrero[1,2], Abdullah Radi[2], Woo Seok Choi[3], Sebastian Macke[4], Ronny Sutarto[5], Feizhou He[5], George A. Sawatzky[2], Ho Nyung Lee[3], and Vladimir Hinkov[4] — [1]IFW-Dresden — [2]University of British Columbia, Vancouver, Canada — [3]Oak Ridge National Laboratory, Materials Science and Technology Division, USA. — [4]Max Planck-UBC Centre for Quantum Materials, Vancouver, Canada — [5]Canadian Light Source, Saskatoon, Canada

A series of $LaCoO_3$ (LCO) single films grown on polar $NdGaO_3$ (NGO) and non polar $SrTiO_3$ (STO) substrates were studied by means of Resonant Soft X-ray Reflectivity (RXRR) and X-ray Absorption Spectroscopy (XAS). The RXRR measurements were performed at photon energies close to the Co $L_{2,3}$ edges. The detailed analysis of the energy dependent measurements at fixed Q values corresponding to maxima and minima of the RXRR Kiessig fringes reveals a strong signal contribution to the line-shapes which can not be attributed to pure Co^{3+}. By considering the polar nature of the LCO structure we find that, at interfaces with polar discontinuity, e.g., at LCO/STO and LCO/Vaccum, reconstruction phenomena take place.

The work at Oak Rigde National Laboratory was supported by the U.S. Department of Energy, Basic Energy Sciences, Materials Sciences and Engineering Division.

MI 5: X-ray spectrometry and analysis of works of art

Chair: B. Kanngießer

Time: Wednesday 9:30–12:15 Location: TA 201

Invited Talk MI 5.1 Wed 9:30 TA 201

Das Arkanum von Meissener Porzellan: Beharrung oder Wandel? — •Christian Neelmeijer[1], Ulrich Pietsch[2] und Heike Ulbricht[2] — [1]Helmholtz-Zentrum Dresden-Rossendorf, PF 510119, 01314 Dresden, Germany — [2]Staatliche Kunstsammlungen Dresden, Porzellansammlung, Sophienstraße, 01067 Dresden, Germany

Das Arkanum zur Herstellung von Hartporzellan in Europa geht auf ein Patent von Johann Friedrich Böttger im Jahre 1708 zurück. Seitdem ist die Porzellanmanufaktur Meissen Hersteller und Vertreiber des "Weißen Goldes". Wie streng wird auf dem dort hinterlegten Geheimnis seiner Rezeptur beharrt? Gibt es durch Variationen in den Einsatzstoffen entscheidende Modifikationen in der chemischen Zusammensetzung? Die zerstörungsfreie Materialanalyse mittels hochenergetischen Protonen an Luft gestattet es, diesen Fragen nachzugehen. Vor der Restaurierung war die nichtglasierte Porzellanmasse an Defekten von 34 gesicherten Originalen aus dem 18. Jahrhundert für den Rossendorfer 4-MeV-Protonenstrahl und das Arrangement verschiedener, simultan arbeitender Nachweisgeräte zugänglich. Über den erfassten Zeitraum von knapp 100 Jahren erweist sich die Grundzusammensetzung der Porzellanmasse (Oxide von Silizium und Aluminium) als nahezu gleichbleibend. Größere Schwankungen in den Konzentrationen der Begleitsubstanzen und der Spurenelemente verweisen auf unterschiedliche Fundorte von Feldspat, Kaolin und Quarz. Unikate von höchstem Wert sind in die Untersuchungen eingebunden, auch deren Glasuren und Malfarben.

Invited Talk MI 5.2 Wed 10:15 TA 201

From micro to nano - new X-ray techniques as depth sensors for material characterization — •Ioanna Mantouvalou[1], Lars Lühl[1,2], Marcel Pagels[1], Wolfgang Malzer[1], and Birgit Kanngiesser[1] — [1]Technische Universität Berlin — [2]Bundesanstalt für Materialforschung und -prüfung, Berlin

Due to their high penetration depth in matter X-rays render the analysis of the inside of material feasible. While for hard X-rays information can be gathered from a few tens of micrometers to even millimeters, soft X-rays are sensitive for nano-scale features. In both regimes new technological developments offer additional techniques which can satisfy the growing demand for novel analytical methods.

While soft X-ray spectroscopy is used widely at large scale facilities like synchrotrons, laboratory based investigations have up until now been hindered due to a lack of suitable radiations sources with sufficient brilliance. Laser-produced plasma sources can fill this gap. With the right choice of laser and target parameters the optimal emission energy can be tailored according to the experiment at hand.

In this presentation examples for depth resolved investigations on the micro- and nano-meter scale will be given. The application of 3D Micro- X-ray fluorescence spectroscopy (XRF) in the field of cultural heritage will be demonstrated on reverse glass paintings. Grazing incidence XRF is shown to be very well suited for nanometer depth resolved characterization of thin film solar cells. The spectroscopic possibilities of laser plasma sources as new X-ray sources are discussed with these investigations at hand.

MI 5.3 Wed 11:00 TA 201

Investigation of the manufacturing process of attic shards by X-ray absorption spectroscopy — •Lars Lühl[1,2], Sammia Mahlkow[1], Eleni Aloupi[3], and Birgit Kanngiesser[1] — [1]Institut für Optik und Atomare Physik, Technische Universität Berlin, Hardenbergstr. 36, 10623 Berlin, German — [2]Bundesanstalt für Materialforschung und -prüfung, Unter den Eichen 44-46, 12203 Berlin, German — [3]NCSR Demokritos, Gr-15310 Aghia Paraskevi, Greece

Ceramics in general are of great historical importance because they are among the earliest evidence of human settlements. Primitive handmade ceramics that were made of coarse clay and burned at low temperatures are considered to be the first signs of tribes to settle. Up to the masterful and artistic ceramics of Greek and especially of Attic vases, the method has been refined over the centuries by polishing the surface, carving notches and especially by the use of wheels.

Attic vases are unique in its production technology due to the three-stage firing process. During the firing process the ornamentation applied on the leather dried ceramic is given its intense, glassy black colour, while the underlying ceramic coloured reddish-brown.

In order to gain knowledge about the manufacturing process, modern replica of attic shards, produced with different coarsenesses of suspension and fired with different temperatures, were investigated. The iron oxidation state was determined as an indicator for the different manufacturing procedures. The results are compared with results from historic attic shards which gave a deeper insight into the manufacturing process.

MI 5.4 Wed 11:15 TA 201

Limits of detection of μ-XRF with the SEM/EDS for RoHS relevant elements — •Vanessa Rackwitz[1], Markus Ostermann[1], Ulrich Panne[1,2], and Vasile-Dan Hodoroaba[1] — [1]BAM Federal Institute for Materials Research and Testing, 12200 Berlin, Germany — [2]Humboldt-Universität zu Berlin, 10099 Berlin, Germany

For the determination of the specimen elemental composition most modern Scanning Electron Microscopes (SEM) are equipped with an energy dispersive X-ray spectrometer (EDS). Based on the technological improvement of low-power X-ray tubes and X-ray polycapillary optics in the last few years a new analytical tool for SEM has been developed: micro-focus X-ray fluorescence μ-XRF. In order to evaluate the performance of the new μ-XRF with SEM/EDS systematically, the limits of detection (LoD) for trace element concentration in a set of ten plastic reference materials were under examination. The reference materials consist of acrylonitrile-butadiene-styrene terpolymer (ABS) and have been doped with different contents of the elements Br, Cd, Cr, Hg and Pb. The interest for this kind of samples has its roots in the European Directive 2002/95/EC on the restriction of the use of certain hazardous substances in electrical and electronic equipment, RoHS. Additionally, the LoD for defined conditions can also be predicted by means of forward calculations of μ-XRF spectra based on a physical model.

MI 5.5 Wed 11:30 TA 201

Mikro-Röntgenfluoreszenz für die schnelle und empfindliche ortsabhängige Elementanalytik — •Michael Haschke, Ulrich Waldschläger, Roald Tagle und Uwe Rossek — Bruker Nano GmbH

Die Mikro-Röntgenfluoreszenz-Spektroskopie hat sich in den letzten Jahren zu einer oft genutzten Methode für die ortsempfindliche Elementanalytik entwickelt. Die Verfügbarkeit von neuen leistungsfähigen Baugruppen etwa hochbrillante Röntgenröhren, Kapillaroptiken mit hoher Transmissions-Effektivität und Röntgendetektoren mit hoher Pulsverarbeitungskapazität eröffnen neue analytische Möglichkeiten.

Der Einfluss dieser die Eigenschaften eines Mikro-Röntgenfluoreszenz-Spektrometers bestimmenden Baugruppen insbesondere auf die Signalintensität wird diskutiert. Davon ausgehend werden die Auswirkungen dieser Verbesserung sowohl auf die Präzision einer Punktmessung als auch auf die Bestimmung von Elementverteilungen untersucht und anhand von konkreten Applikationsbeispielen dargestellt.

MI 5.6 Wed 11:45 TA 201

Quantitative 3D micro-XRF analysis with laboratory setup — •Timo Wolff, Wolfgang Malzer, Christian Herzog, and Birgit Kanngiesser — Institut für Optik und Atomare Physik, TU-Berlin, Hardenbergstr. 36, 10623 Berlin

X-ray fluorescence (XRF) is a wide spread analytical tool for elemental analysis. Due to its non destructive character and a relative high information depth it is frequently used various fields of science. The development of thermoelectric cooled silicon drift chamber detectors (SDD) and of X-ray optics, enabling a spatial resolution in the micrometer range, even strengthened this trend. Using a confocal setup 3D micro-XRF is possible. In this technique a second optic is mounted in front of the detector entrance to limit the field of view. The optics form a probing volume with dimensions in the micrometer range from which fluorescence is detected only. By moving the sample through this volume depth resolved information can be obtained from the sample in a non destructive way. This confocal geometry influences another aspect of XRF, making this technique to an interesting tool: the possibility of quantitative analysis without reference samples, based on the fundamental parameter (FP) method. A new analytical description was developed and successfully tested with 3D micro XRF measurements with synchrotron radiation on standard reference samples and on archaeomtric objects in the last years. The procedure was now extended to setups with polychromatic tube excitation. Thus, for the first time quantitative 3D micro-XRF in the laboratory is possible.

MI 5.7 Wed 12:00 TA 201

Advanced light element and low energy X-ray analysis of ceramics and ceramic-metal joints using SEM/EDX with Silicon Drift Detectors (SDD) — •Tobias Salge[1], Orkun Tunckan[2], Ralf Terborg[1], Vasile-Dan Hodoroaba[3], and Servet Turan[4] — [1]Bruker Nano GmbH, Berlin, Germany — [2]Civil Aviation School, Anadolu Univ., Turkey — [3]BAM, Berlin, Germany — [4]Materials Science and Engineering Dept., Anadolu Univ., Turkey

An analytical aspect in EDX analysis of massive samples is to lower the spatial resolution. This makes it necessary to work with low excitation voltages to decrease the excitation volume for generated X-rays and evaluate low energy X-ray lines. In order to separate overlapping peaks, the line deconvolution algorithms incorporated into the EDX software are of crucial importance. Two representative samples will be discussed: (1) a sintered hard ceramic composed of three major phases TiB2, TiC, and SiC and (2) a Si3N4-Ti joint with a heterogeneously composed reaction layer [1]. This study demonstrates that state-of-the-art hardware in combination with modern data processing allows advanced spectrum imaging of light elements and low energy X-ray lines. Element distribution of overlapping lines at the low energy range (e.g. Ti-L, N-K) can be analyzed on the sub-micro scale by EDX in a short time by peak deconvolution. Even at low impulse statistics, spectra containing low energy lines can be accurately quantified.

References: [1] O. Tunckan (2010) Ph.D thesis. Anadolu University, Eskişehir, Turkey, 176 p. [2] Dr. R. Wäsche (BAM) is gratefully acknowledged for producing the hard ceramic sample.

MI 6: X-ray imaging, holography and tomography

Chair: H. S. Leipner

Time: Wednesday 12:30–14:30 Location: TA 201

MI 6.1 Wed 12:30 TA 201

Confocal STXM - a novel approach to 3D X-ray microscopy — •Andreas Späth[1], Jörg Raabe[2], Christian Riess[3], Simon Schöll[3], Joachim Hornegger[3], and Rainer H. Fink[1] — [1]Friedrich-Alexander Universität Erlangen-Nürnberg, Physical Chemistry II, Erlangen, Germany — [2]Swiss Light Source (SLS), Paul Scherrer Institut, Villigen Switzerland — [3]Friedrich-Alexander Universität Erlangen-Nürnberg, Computer Science 5, Erlangen, Germany

Common approaches to 3D microscopy with soft X-rays (e.g. tomography, holography, etc.) are often disadvantaged by a high experimental or computational effort. We will present a novel procedure to obtain 3D images of nanostructured soft matter based on the PolLux scanning transmission X-ray microspectroscope installed at a bending magnet beamline of the Swiss Light Source [1]. Using latest zone-plate technonology, imaging is not only achieved with very high lateral resolution (limit 12 nm) [2], but also with a depth of focus of better than 550 nm. Image stacks from the variation of the sample position along the X-ray propagation axis offers the opportunity to confocal imaging. Thus, we are able to image nanoscopic inhomogeneities of the sample (e.g. nanoparticles) within each confocal plane and recheive 3-dimensional reconstructions by determining the focus plane of these features. The required analysis tools are provided by computational sciences and comfortable in application and computing time. We will discuss the opportunities and limitations in conventional STXM imaging.

1. J. Raabe, et al., Rev. Sci. Instrum. 79, 2008, 113704.
2. J. Vila-Comamala, et al., Ultramicroscopy 109, 2009, 1360.

MI 6.2 Wed 12:45 TA 201

Hard x-ray scanning microscope with chemical, elemental and structural contrast — •Jens Patommel[1], Christian G. Schroer[1], Manfred Burghammer[2], and Gerald Falkenberg[3] — [1]Institute of Structural Physics, Technische Universität Dresden, D-01062 Dresden, Germany — [2]European Synchrotron Radiation Facility ESRF, B. P. 220, F-38043 Grenoble Cedex, France — [3]HASYLAB at DESY, Notkestr. 85, D-22607, Hamburg, Germany

We designed, built and are now operating a scanning microscope for hard x rays at the synchrotron radiation facility PETRA III at DESY. The instrument uses nanofocusing refractive lenses (NFLs) to generate an intense x-ray nanobeam with a size of between 50 nm and 100 nm. The nanoprobe supports a variety of contrast mechanisms like x-ray absorption spectroscopy, fluorescence spectroscopy, small/wide angle x-ray scattering, and coherent diffraction contrast, retrieving chemical, elemental and structural information from inside the specimen. Although the spatial resolution in direct measurement is limited by the beam size, a much better resolution is achieved with ptychography, combining coherent x-ray diffraction imaging with scanning techniques. All these contrast mechanisms can be performed in tomography mode to retrieve the three-dimensional contrast distribution.

In my talk I will address theoretical considerations of hard x-ray scanning microscopy as well as instrumental aspects of the nanoprobe at PETRA III. I will also present examples for each of the contrast mechanisms.

MI 6.3 Wed 13:00 TA 201

Investigation of biological specimens with a 30 nm resolution table top X-ray microscope based on a high repetition laser plasma source — •Christian Seim[1], Christoph Redlich[1], Jonas Baumann[1], Gernot Blobel[2], Herbert Legall[2], Bernhard Hesse[3], Holger Stiel[2,4], and Birgit Kanngiesser[1,4] — [1]TU Berlin, Germany — [2]Max-Born-Institute, Germany — [3]Chartité Berlin, Germany — [4]Berlin Laboratory for innovative X-ray Technologies (BLiX), Germany

This talk describes a table top X-ray microscope based on a high repetition rate laser plasma source which enables investigations of biological specimens at 30 nm resolution. The laboratory soft X-ray microscope works at the nitrogen Ly$_\alpha$ at 2.478 nm in the so called water window, to gain a high contrast when investigating biological samples with thickness up to 10 μm. A detailed description of the instrument is given and the feasibility of X-ray tomography of aqueous samples will be discussed. First images of reference biological samples, diatoms, with structures in the 30 nm region will be shown. It is planned to apply the instrument for investigations of the BRONJ disease. BRONJ is a rare disease, in which the patient's jaw bone suffers osteonecrosis, induced by bisphosphonates (Salvatore L. Ruggiero et al., 2009). One theory is that the canaliculi system that connects and supplies the osteocytes (bone cells) becomes clogged. The canaliculi diameter is approximately 300 nm. The feasibility of capturing a 3D image of the canaliculi network using X-ray tomography will be discussed.

MI 6.4 Wed 13:15 TA 201

Ptychographie: Rastermikroskopie mit kohärentem Röntgenbeugungskontrast — •Robert Hoppe[1], Manfred Burghammer[2], Gerald Falkenberg[3] und Christian G. Schroer[1] — [1]TU Dresden, Dresden, Deutschland — [2]ESRF, Grenoble, Fankreich — [3]DESY, Hamburg, Deutschland

Ptychographie kann zur ortsaufgelösten Bestimmung der Absorption und des Phasenschubs einer Probe verwendet werden. Die bisher erreichte laterale Auflösung ist dabei besser als 10 nm [1]. Ptychographische Röntgenrastermikroskopie nutzt den Fernfeldbeugungskontrast zur Abbildung. Parallel zum Objekt werden die Amplitude und die Phase der Beleuchtung mit rekonstruiert. Diese Trennung der Beleuchtung vom Objekt erlaubt einzigartige Einblicke in die Intensitätsverteilung und Phasenlage verschiedener Teilbereiche des fokussierten Röntgenstrahls. Das bedeutet, dass über den Röntgenstrahl die vollständigen Informationen vorliegen. Diese einzigartigen Information werden unter anderem zur Weiterentwicklung von Röntgenoptiken wie zum Beispiel von Nanofokussierenden Linsen (NFLs) [2] verwendet. Mit NFLs wurde der Röntgenstrahl bisher bis auf eine Größe von $46 \times 63\,\mathrm{nm}^2$ (FWHM) fokussiert [3]. Der fokussierte Strahl erlaubt es, simultan zur ptychographischen Bildgebung die Elementverteilung mittels Fluoreszenzanalyse im Objekt ortsaufgelöst zu untersuchen. [1] A. Schropp et. al., Appl. Phys. Lett. 96, 091102(2010) [2] C. G. Schroer et.al., Appl. Phys. Lett. 87, 124103(2005) [3] C. G. Schroer et. al. AIP Conference Proceedings, P227-230,(2011)

MI 6.5 Wed 13:30 TA 201

Pink Beam Ptychography — •Björn Enders, Pierre Thibault, Martin Dierolf, Andreas Fehringer, Marco Stockmar, Irene Zanette, and Franz Pfeiffer — Department of Physics (E17) and Institute of Medical Engineering (IMETUM)

In the field of X-ray Coherent Diffraction Imaging we use a scanning technique called ptychography to image 2D and 3D specimen at the nanoscale. From the ptychographically reconstructed sample projections we use computed tomography to obtain fully quantitative 3d electron density maps of bone or other material science specimen. We report on latest results in ptychographic tomography on bone and on its applications to less coherent synchrotron x-ray sources of wider spectral bandwidth (pink beam sources). Providing higher flux, those sources promise a significant speed increase that would facilitate the usage of ptychography tomography for a broader application spectrum.

MI 6.6 Wed 13:45 TA 201

Progress in X-Ray Inline Holography Phase Retrieval — •Marco Stockmar[1], Pierre Thibault[1], Martin Dierolf[1], Björn Enders[1], Andreas Fehringer[1], Julia Herzen[1], Irene Zanette[1], Peter Cloetens[2], and Franz Pfeiffer[1] — [1]Department of Physics (E17) and Institute of Medical Engineering (IMETUM), Technische Universität München, Germany — [2]European Synchrotron Radiation Facility (ESRF), Grenoble, France

We present first numerical and experimental results obtained at a Synchrotron light source of an advanced X-ray inline holography phase retrieval method for high resolution nondestructive 2D and 3D imaging. Unlike existing methods, which are based on limiting constraints such as weak phase or absorption, our new method retrieves the full complex-valued transmission function of any object while eliminating reconstruction artefacts from imperfect wavefronts. This allows for imaging of a wide range of challenging samples inaccessible by existing methods.

MI 6.7 Wed 14:00 TA 201

Soft X-ray tomoholography — •Erik Guehrs[1,2], Andreas M. Stadler[2], Sam Flewett[1], Stefanie Frömmel[1], Jan Geilhufe[3], Bastian Pfau[1], Torbjörn Rander[1], Stefan Schaffert[1], Georg Büldt[2,4], and Stefan Eisebitt[1,3] — [1]Institut für Optik und Atomare Physik, Technische Universität Berlin, Hardenbergstr. 36, 10623 Berlin — [2]Institute of Complex Systems, Forschungszentrum Jülich, 52428 Jülich — [3]Helmholtz-Zentrum Berlin für Materialien und Energie, Hahn-Meitner-Platz 1, 14109 Berlin — [4]Research-Educational Centre Bionanophysics, Moscow Institute of Physics and Technology, 141700 Dolgoprudniy, Russia

Fourier transform holography (FTH) is a well established imaging method in the soft X-ray regime. In common mask-based FTH the reference wave is produced by a small pinhole which is physically connected to the sample. Due to the high aspect ratio (1:10) of the pinhole it is not possible to record a tomographic dataset as the required rotation of the sample blocks the transmission through the pinhole at small angles. Therefore, only 2D projections have been imaged until now using soft X-ray FTH. However, the determination of the three-dimensional structure of samples is of high interest in many scientific areas. We demonstrate an X-ray imaging method which combines Fourier transform holography with tomography ("tomoholography") for 3D microscopic imaging. A 3D image of a diatom shell with a spatial resolution of 140 nm is presented. The experiment is realized by using a small gold sphere as reference wave source for holographic imaging.

MI 6.8 Wed 14:15 TA 201

Three-dimensional X-ray Fourier transform holography — •Jan Geilhufe[1], Carsten Tieg[1], Christian Günther[2], Erik Gührs[2], Bastian Pfau[2], Stefan Schaffert[2], and Stefan Eisebitt[1,2] — [1]Helmholtz-Zentrum für Materialien und Energie GmbH — [2]Institut für Optik und Atomare Physik, Technische Universität Berlin

Three-dimensional topography information is successfully extracted from a single soft X-ray Fourier transform hologram. The feature height is retrieved by propagating the focal plane through the object reconstruction. An artificial test specimen with features sizes down to 20 nm and maximum height differences of 10 μm was prepared by focused ion beam assisted deposition of platinum. In order to acquire

a small depth of focus, the hologram was recorded with a large numerical aperture. The small depth of field is exploited to acquire depth information with 500 nm resolution to model a two-dimensional height map with a lateral resolution of 50 nm.

MI 7: Poster – 100 years since the Laue experiment: Topical aspects of diffraction and scattering (Joint Session KR, BP, DF, GP, MA, MI, MM; related SYXD)

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

MI 7.1 Wed 15:00 Poster E

Clip - The Cologne Laue Indexation Program — •Olaf J. Schumann — Fraunhofer-Institut für Naturwissenschaftlich-Technische Trendanalysen, Euskirchen, Deutschland — II. Physikalisches Institut, Universität zu Köln, Germany

The Cologne Laue Indexation Program is a software for the analysis and simulation of Laue images.

Clip features a modern graphical user interface, could read a large variety of image formats and allows to mark spots *and* zones in a recorded image. These could be used for automatic indexation of the image for arbitrary crystal symmetries and refinement of lattice constants and projection plane parameters. Clip helps with the alignment of the crystal to a desired orientation. It is an open source software (GPL) written in C++ and the cross platform toolkit Qt and runs on Windows, Linux and Mac OS X.

MI 7.2 Wed 15:00 Poster E

A new access to extinction corrections — •Anne K. Hüsecken and Ullrich Pietsch — Naturwissenschaftlich Technische Fakultät, Fachbereich Physik, Universität Siegen, D-57068 Siegen, Germany

In x-ray crystal structure analysis a problem, called extinction, occurs due to multiple scattering in crystals. Over the years several extinction correction theorems have been formulated, but the used parameters have never been proved to be valid for a certain crystal under investigation. Perfect crystals scatter according to the dynamical theory (I~|F|) and imperfect crystals or ideal mosaic crystals due to the kinematical theory (I~|F|*). In most cases, the measured intensities of real crystals are in between both cases and an extinction correction is needed to fulfil the kinematic approach. Present theories dealing with extinction corrections are based on the approach of a mosaic crystal and describe x-ray scattering in terms of the kinematic approach using certain "correction terms" to implement the structure of a real crystal. The mosaic blocs within a real crystal are misorientated to each other and are affected by lattice strain. In addition both 3D shape and size of the blocs are not known. All these parameters can be determined by high-resolution x-ray diffraction techniques performing ω- and ω-2θ-scans through certain reciprocal lattice points. The measured parameters can be used to determine extinction. Our aim for crystallography is to perform these scans only for a few reflections, make a short analysis, to get the size, misorientation and lattice strain of the mosaic blocs. With these parameters it should then be possible to decide which one is the best extinction correction to use.

MI 7.3 Wed 15:00 Poster E

Evaluation of interfacial orientation information from 3D X-Ray diffraction contrast tomography in and its application in a mesoscale grain coasening model — •Melanie Syha, Fabian Sehn, Andreas Trenkle, and Daniel Weygand — Karlsruher Institut für Technologie, IAM

The orientation information from 3D X-Ray diffraction contrast tomography investigations in polycrystalline $SrTiO_3$ ceramics was evaluated before and after annealing. Special emphasis was put on local interface orientations, showing a preference for <100> orientated interfaces that increases during microstructural evolution. Moreover the data was used to investigate orientation dependent relative interface mobilities. The results are discussed in the context of the abnormal growth behavior found in $SrTiO_3$ and used to adapt a mesoscale grain coarsening model to more realistic simulations of microstructure evolution in this material.

MI 7.4 Wed 15:00 Poster E

Inter-layer disorder in sodium cobaltate — •David Jonathan Pryce Morris[1], Alan Tennant[1,2], Klaus Seiffert[1,3], Esther Dudzik[1], Dharmalingam Prabhakaran[4], Jon Goff[5], Michel Roger[6], and Jon Wright[7] — [1]Helmholtz-Zentrum Berlin, Germany — [2]TU-Berlin, Germany — [3]Kiel University, Germany — [4]Oxford University, UK — [5]Royal Holloway, University of London, UK — [6]CEA-Saclay, France — [7]ESRF, France

Sodium Cobaltate is a layered material which has been studied as a potential battery material, has shown good thermoelectric properties and becomes superconducting when hydrated. The physical properties are dependent on sodium content and the ordering of sodium ions. Sodium ordering in NaxCoO2 has previously been observed to have long-range order. Using x-ray diffraction we have observed a phase with long-range in-plane order and inter-layer disorder. Here we will present the data giving a possible structural interpretation.

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 Struc-

turing 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.*