MI 7: Poster: Microanalysis and Microscopy

Chair: Enrico Langer (TU Dresden) and Hartmut S. Leipner (Martin-Luther-Universität Halle-Wittenberg)

Time: Wednesday 18:00–20:00 Location: Poster E

MI 7.1 Wed 18:00 Poster E

Micro-XRF studies on the colour brilliance in ancient wool carpets — •Andreas Späth[1], Markus Meyer[1], Camelia Borca[2], Karl Messlinger[3], Manfred Bieber[4], and Rainer H. Fink[1] — [1]FAU Erlangen-Nürnberg, Physical Chemistry II & Interdisciplinary Center for Molecular Materials (ICMM), Erlangen, Germany — [2]Swiss Light Source (SLS), Paul Scherrer Institute, Villigen, Switzerland — [3]FAU Erlangen-Nürnberg, Physiology and Pathophysiology, Erlangen, Germany — [4]Ex Oriente, Waldbrunn, Germany

Chemical colouring of natural fibers is one of the oldest crafts in mankind and still a highly relevant market. Experimental archaeologists revived an ancient Anatolian dyeing method based on previous fermentation of the wool fibers resulting in remarkable colour brilliance and persistence. Mordants used during the bating process are typically potassium alum or iron(II)sulfate. Electron microscopic studies show morphological changes of the fibers due to fermentation, suggesting an easier and deeper penetration of mordants into the hair fiber. We present micro-XRF studies on cross-sections of such prepared recent wool fibers and regional carpet fibers from the 18th century showing the distribution of mordant metals. A comparison with undyed fibers proves a significantly increased metal content also in the central region of the recently fermented and ancient wool fibers. These findings provide a conclusive explanation for persistent colour brilliance and prove the suggested crafting of the ancient specimen. Further studies focus on the oxidation state of the Fe mordant to clarify the typically strong modification of dye colour that is not apparent for redox-inactive Al.

MI 7.2 Wed 18:00 Poster E

The estimation of thickness and composition of thin films using EDX — •Steffen Schulze[1], Sandra Hahn[2], Wolfgang Baumann[3], and Hans-Jörg Hunger[3] — [1]TU Chemnitz, Physik, Analytik an Festkörperoberflächen — [2]TU Chemnitz, Maschinenbau, Werkstoffwissenschaft — [3]TU Karl-Marx-Stadt, Physik/EB

EDX is capable of estimating thickness and composition of thin films on substrates in the 10 nm range. The intensity ratio of X-rays from the coating to that of the substrate depends sensitively on film thickness. For taking into account the alterations of X-ray generation by substrate backscattering and of the absorption in the coating the depth distribution function of X-ray production as given by Packwood and Brown [1] has been slightly modified to take account of the thin film situation. Examples are given demonstrating successfull quantification and thickness determination by use of the according correction procedures.

[1] Packwood, Brown: X-ray spectroscopy, Vol. 10, 3 (1981).

MI 7.3 Wed 18:00 Poster E

Generation of electron vortex beams and OAM selection using aberration-corrected probes for local EMCD measurements — Darius Pohl[1], •Sebastian Schneider[1,2], Pascal Gerhards[1,2], Jan Rusz[3], Jakob Spiegelberg[3], Peter Tiemeijer[4], and Bernd Rellinghaus[1] — [1]IFW Dresden, P.O. Box 270116, D-01171 Dresden, Germany — [2]TU Dresden, Institute for Solid State Physics, D-01062 Dresden, Germany — [3]Uppsala University, Department of Physics and Astronomy, SE-752 37 Uppsala, Sweden — [4]FEI Company, PO Box 8066, 5600 KA Eindhoven, The Netherlands

Electron vortex beams (EVBs) carry a discrete orbital angular momentum (OAM), L, and are predicted to reveal magnetic dichroic in electron energy loss spectra upon interacting with magnetic samples. Since electron beams can be easily focused down to sub-nanometer diameters, this novel technique provides the possibility to quantitatively determine local magnetic properties with unrivalled lateral resolution. The generation of EVBs in the double aberration-corrected FEI Titan^3 80-300 transmission electron microscope (TEM) is achieved by the implementation of spiral and dislocation-type apertures into the condenser lens system. The setup allows for scanning TEM investigations (STEM) with EVBs, whose OAM is selected by means of an additional discriminator aperture. New FIB cutting strategies facilitate the production for 50 μm wide and 1 μm thick high quality vortex apertures. The status quo of both experiments and simulations of the interaction of the EVB with $L1_0$-FePt nanomagnets will be presented.

MI 7.4 Wed 18:00 Poster E

Nanostructures from electron beam assisted decomposition of gold nitrates — •Daniel Zink, Torsten Henning, and Peter J. Klar — Justus-Liebig-Universität Gießen, I. Physikalisches Institut, Heinrich-Buff-Ring 16, 35392 Gießen

To produce elemental gold in defined structures on substrates is of interest for a wide range of applications. It may be useful for optical and electrical devices, plasmonic structures or gold nanoparticles. The electron beam assisted decomposition of $(NO_2)[Au(NO_3)_4]$ and $(NO)[Au(NO_3)_4]$ has advantages compared with common methods for depositing gold nanostructures (including MOCVD or organic resists as PMMA) such as avoiding carbon contamination. The decomposition of gold precursor takes place by interaction with the electron beam. As in conventional e-beam lithography proximity effects need to be controlled in order to optimize the lateral resolution. We will present some first experiments using this novel method.

MI 7.5 Wed 18:00 Poster E

First results of ptychographic imaging at MAXYMUS X-ray microscope — •Iuliia Bykova, Markus Weigand, Michael Bechtel, Joachim Gräfe, Eberhard Goering, and Gisela Schütz — Max-Planck-Institut für Intelligente Systeme, Heisenbergstraße 3, 70569 Stuttgart, Germany

Ptychography is a recently established and actively developing technique for producing highly resolved images. This method is a combination of x-ray diffraction imaging with scanning microscopy which aids to visualize objects with spatial resolution not limited by the properties of the used optics. MAXYMUS is an Ultra-High Vacuum Scanning Transmission X-ray Microscope (STXM) operated by the MPI for Intelligent Systems at the Bessy II synchrotron (Berlin, Germany). The feasibility of high brightness and selectable polarization measurements make it a unique tool for studying magnetic materials in an element specific manner. To allow high resolution ptychographic imaging MAXYMUS was upgraded with a new fast in-vacuum CCD camera with high readout speed up to 450 Hz, quantum efficiency >70% (for E>300 eV) and RMS noise per pixel less than 3e-. We will present the results of commissioning of the new in-vacuum CCD camera and the implementation of ptychographic imaging at MAXYMUS. We will show the first ptychographic reconstructions made at MAXYMUS which have significant advantage in resolution over images done using conventional STXM methods.

MI 7.6 Wed 18:00 Poster E

Ultra-narrow germanium-slits for laboratory and synchrotron x-ray applications — •F. Döring, S. Hoffmann, M. Kanbach, and Tim Salditt — Institute for X-Ray Physics, University of Göttingen

Modern nano-beam x-ray diffraction and imaging applications rely on precise optical elements for beam focusing and shaping. We have recently developed x-ray waveguide optics to deliver coherence and wavefront filtered beams confined down to below 20 nm. While these optics are well suited for coherent imaging applications with synchrotron radiation, the transmission is too low for laboratory applications. Here we investigate the transition from a x-ray waveguide to a nanoscale slit suitable for laboratory and synchrotron applications in a setting of low and medium brilliance. Therefore, we aim at a larger angular acceptance of the beam, requiring a smaller optical thickness so that the mode structure is not yet fully developed, while the absorption must be sufficiently high. So, we replace silicon by germanium and adapt the processes of silicon based waveguide fabrication to germanium slit production. We use a lithographic manufacturing process at 400 nm in combination with reactive ion etching (RIE) and germanium wafer bonding. Along the vertical direction, the slit size shall be varied from 100 nm up to 1 μm as controlled by RIE parameters, while the horizontal size is controlled by UV exposure and varied in the range between 1 μm and 1.5 mm. The slit length is selected according to the required photon energy, ranging from 200 μm to 1 mm, for 8-20 keV photon energy, respectively. The fabrication is guided by finite differences simulation.

MI 7.7 Wed 18:00 Poster E

Acto-myosin in cardiac muscle cells by scanning x-ray nano-diffraction — •Jan-David Nicolas, Marten Bernhard, and Tim Salditt — Institut für Röntgenphysik, Göttingen, Deutschland

Owing to the highly oriented molecular structure of the actin-myosin cortex in muscle cells, diffraction techniques are well-suited to study the geometry of this filament assembly down to nanometer resolution. In particular, classical x-ray diffraction studies on muscular tissue were the first to unravel the detailed structure of the sarcomere. In these experiments, however, structural information is averaged over macroscopically large volumes of the tissue, with diffraction volumes containing a vast ensemble of muscle cells. Contrarily, recent progress in x-ray optics has enabled diffraction experiments with spot sizes in the sub-micron range, well-suited to illuminate only selected organelles of a single cell.

We report on recent experiments analyzing the micro-structure of acto-myosin complexes in individual cardiomyocytes which make up the striated muscular tissue of the heart. We performed experiments on (initially) alive, chemically fixed as well as freeze-dried cell preparations. Scanning the sample through the nano-focused beam, SAXS data were recorded and analyzed to generate mappings of different structural parameters. Scanning SAXS mappings are complemented by holographic reconstructions, extending the covered frequency range by two orders of magnitude. By means of x-ray holography, samples could also be immediately checked for radiation damage.

MI 7.8 Wed 18:00 Poster E

SIMS based in-situ correlative microscopy: HIM-, TEM- and SPM-SIMS — David Dowsett, Santhana Eswara, •Florian Vollnhals, and Tom Wirtz — Advanced Instrumentation for Ion Nano-Analytics (AINA), MRT Department, Luxembourg Institute of Science and Technology (LIST), 41 rue du Brill, L-4422 Belvaux, Luxembourg

While Secondary Ion Mass Spectrometry (SIMS) is among the most sensitive surface analysis techniques, like all techniques, it has its limitations. For example, the lateral resolution in commercial instruments is limited to several tens of nanometers with a fundamental limit of around 10-20 nanometers.

Fortunately, some of the limitations of SIMS can be overcome by combining it with complementary techniques in a correlative approach. Combining SIMS data with high resolution microscopy techniques such as Transmission Electron Microscopy (TEM) or Helium Ion Microscopy (HIM), we can take advantage of high spatial resolution and high chemical sensitivity. Correlation with other analysis techniques, like Energy Dispersive X-ray Spectroscopy (EDS), facilitates quantification. Additionally, SIMS combined with Atomic Force Microscopy (AFM) provides a path to artifact free 3D reconstruction even for complex samples with widely varying sputter yields.

In order to apply these techniques for in-situ correlative microscopy, we developed integrated instruments and methods, which will be presented in this contribution [1].

[1] T. Wirtz et al., Nanotechnology 26, 434001 (2015).

MI 7.9 Wed 18:00 Poster E

Stress Analysis in Semiconductor Devices by Kelvin Probe Force Microscopy — •Evgeniya Sheremet[1], Florian Fuchs[1,3], Soumya D. Paul[1], Sven Haas[1], Dietmar Vogel[2], Raul D. Rodriguez[1], Andreas Zienert[1], Jörg Schuster[2], Danny Reuter[1], Thomas Gessner[1], Dietrich R.T. Zahn[1], and Michael Hietschold[1] — [1]Technische Universität Chemnitz, Chemnitz, Germany — [2]ENAS Fraunhofer, Chemnitz, Germany — [3]Helmholtz-Zentrum Dresden-Rossendorf and Center for Advancing Electronics Dresden (cfaed), Dresden, Germany

The determination of built-in strain in semiconductor devices with nanometer spatial resolution and high sensitivity is needed for the characterization of nanoscale electronic devices. Kelvin probe force microscopy (KPFM) is an atomic force microscopy-based method that provides the spatially resolved surface potential at the sample surface, fulfilling the requirements on resolution and sensitivity. The contrast observed in KPFM imaging is often attributed to stress, but there are only a few reports on the application of KPFM for quantitative stress analysis [1]. In this contribution we focus on the application of KPFM for analysis of stress in silicon devices, such as copper through silicon vias and silicon membranes. The experimental results are compared with density functional theory calculations of strained silicon. This work provides critical insights into the quantitative determination of stress at the nanoscale that so far has gone largely unnoticed in the scanning probe microscopy community.

[1] W. Li, D.Y. Li, J. Appl. Phys. 99, 073502 (2006).

MI 7.10 Wed 18:00 Poster E

Characterization of a UHV evaporator for the coating of W-tips for room-temperature SP-STM — •Kai Besocke, Hendrik Bettermann, and Mathias Getzlaff — Institute of Applied Physics, University Düsseldorf

Our research involves supported nanoparticles of 3d-metal alloys. Magnetic and electronic properties and temperature dependent behavior are interesting for fundamental research and possible technological applications. These properties depend strongly on the nanoparticles' size.

A possible approach to measure the magnetic properties and their magnetic interaction on different substrates with spatial resolution is spin-polarized scanning tunneling microscopy (SP-STM).

We focus on the in-situ coating of non-magnetic W-tips with a layer of Fe since this system is known to have an intrinsic in-plane magnetization and hence doesn't need external magnetic fields.

Self-etched and commercially available tips are used. Effects of heating on the Fe-layer that was prepared by rod and/or crucible evaporation were investigated. The MircoSPM by Omicron is operating at room temperature at a base pressure below $2 \cdot 10^{-10}$mbar.

Examination of prepared tips is focused on scanning tunneling spectroscopy (SPS). Ex-situ (Off-site) scanning electron microscopy (SEM) is also availabe.