TUT 4: State of the Art of X-Ray Microanalysis (MI)

Advanced microanalysis allows researchers to examine in detail the composition of materials. At present, the detection of elements ranging from Beryllium (4) to Americium (95) can be carried out in condensed matter with very high local and spectral resolution. Microanalytical methods are able to provide in situ information on chemical, crystallographic, and structural parameters. In this tutorial, current frontiers of X-ray microanalysis for cutting-edge research in physics, engineering, geology, biosciences, and materials research are described. Fundamental physical processes related to energy and wavelength dispersive X-ray microanalysis, to X-ray fluorescence and tomography, and to ion-beam-induced microanalysis are specified. Experts from industrial and academic research explain the basics and present recent developments in instrumentation and break-throughs in detection and quantification.

Organization/Chair: Enrico Langer

Time: Sunday 16:00–19:15 Location: HSZ 401

Tutorial TUT 4.1 Sun 16:00 HSZ 401 Energy dispersive X-ray spectroscopy, from the method to the instrumentation — •Jana Berlin — Bruker Nano GmbH, Berlin, Germany

One of the most important interactions of beam and sample in the electron microscope is the generation of element-specific X-ray radiation. Energy dispersive X-ray spectrometry (EDS) uses semiconductor detectors to collect this radiation from the sample. "Energy-dispersive" means that the detector measures the relative abundance of emitted X-rays versus their energy. The signal can be evaluated both qualitatively (element identification) and quantitatively (element concentration in mass% or atom%). Spot measurements as well as one, two and nowadays even three-dimensional data acquisition are possible.

The instrumentation used for detection and analysis has made big advances within the past decade. Thermoelectrically cooled silicon drift detectors (SDD) have become state of the art technology, replacing liquid nitrogen cooled Si(Li) detectors. Properties and applications of the SDD technology in the analysis of different sample categories will be discussed to round off this tutorial.

Tutorial TUT 4.2 Sun 16:45 HSZ 401 WDS technique - advanced analytical tool for the SEM — •Frank Bauer — Oxford Instruments, Otto von Guericke Ring 10, D-65205, Wiesbaden, Germany

The typical and very common micro analytical equipment on scanning electron microscopes are energy dispersive systems (EDS). An advanced method in analytical investigations for higher accuracy and sensitivity is the wavelength dispersive X-ray spectroscopy (WDS) in the normal practice the resolution of the detector and sensitivity for elements is in minimum ten times better. Compared to EDS the wavelength dispersive technique needs additional minimum requirements for the scanning electron microscope (SEM) in case of beam current, emission, geometry, etc.

The fundamental physical background of this technique will be shown. Examples of the advantages and also the limitations of WDS are discussed.

15 min. break

Tutorial TUT 4.3 Sun 17:45 HSZ 401 Hard X-ray scanning microscopy and tomography with elemental, chemical, and structural contrast — • Christian G. Schroer — Institut für Strukturphysik, TU Dresden, 01062 Dresden, Germany

In this tutorial, hard x-ray scanning microscopy and tomography is reviewed. There is a growing demand for these techniques in many fields of science, from physics and chemistry, to materials, earth, and envrionmental science, biology and nanotechnology. The large penetration depth of hard x-rays in matter allows one to investigate the inside of a specimen without destructive sample preparation or inside a special sample environment, such as a chemical reactor or a pressure cell. In combination with tomographic techniques, the three-dimensional inner structure of the sample can be reconstructed. X-ray analytical techniques, such as fluorescence, absorption or scattering (SAXS, WAXS) can be used as contrast in the scanning microscope, yielding elemental, chemical, and nano-structural contrast. Currently, spatial resolutions around 50 nm are achieved. In combination with coherent x-ray diffraction imaging techniques, spatial resolutions down to 10 nm and below are reached. A variety of application examples are given.

- [1] A. Schropp, et al., J. Microscopy (2010). DOI: 10.1111/j.1365 2818.2010.03453.x.
 - [2] C. G. Schroer, et al., Phys. Rev. Lett., 101 (9), 090801 (2008).
 - [3] C. G. Schroer, et al., Appl. Phys. Lett., 88 (16), 164102 (2006).
 - [4] C. G. Schroer, et al., Appl. Phys. Lett., 82 (19), 3360 (2003).
 - [5] C. G. Schroer, Appl. Phys. Lett., 79 (12), 1912 (2001).

Tutorial TUT 4.4 Sun 18:30 HSZ 401 **2D- and 3D-microanalysis using focussed MeV-ion beams** — •Tilman Butz — Institute for Experimental Physics II, Universität Leipzig, 04103 Leipzig, Germany

This tutorial introduces the following ion beam techniques for analysis and material modification: Analysis: Rutherford Backscattering Spectrometry (RBS), mainly for thin film analysis; Particle Induced X-Ray Emission (PIXE), elemental maps, main and trace elements; Scanning Transmission Ion Microscopy (STIM), density contrast; STIM-tomography, 3D density; PIXE-tomography, 3D elemental images, main elements only. Modification: Proton Beam Writing (PBW), photo resists, semiconductors, 2D- and 3D-microstructures, creating magnetic order in graphite; Proton Beam Sculpting, 3D-microstructures with complex morphology; Targeted irradiation of living cells with counted ions, low dose radiation research.

Examples for all techniques will be given. The requirements for the application of these ion beam techniques will be discussed. Advantages compared to electron microprobes will be addressed as well as limitations, e.g. the issue of radiation damage. At present, lateral resolutions below 100 nm are achievable by STIM and about 350 nm by PIXE. Minimum detection limits depend on the element and can be as low as $0.1\mu g/g$. Microstructures with feature sizes in the order of 100 nm can be created.