B7 - Techniques and Instruments

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AUTOMATED CRYSTAL ORIENTATION MEASUREMENT BY BACKSCATTER KIKUCHI DIFFRACTION

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*A*utomated *C*rystal *O*rientation *M*easurement (*ACOM*) by *B*ackscatter *K*ikuchi *D*iffraction (*BKD*) in the SEM, also named "*Automated EBSD*", has become a standard tool in materials science during the last decade. The principal objectives of ACOM are a quantitative description of the microstructure on a grain-specific level by the determination of crystal orientations, misorientations, the character of grain boundaries, and derived entities. The grain orientations are commonly depicted in pseudo-colors on the scanning grid to form *C*rystal *O*rientation *M*aps (*COM*) of the microstructure. Stereological as well as orientation data, as sensitive indicators of the production process, are readily available from COM. ACOM thus enables a major progress in the quantitative characterization of microstructure.

The benefits are the correlation of crystal lattice orientations and phases (from ACOM) with the morphology (from the SEM micrograph) and element composition (from EDS analysis in an analytical SEM) on a submicron scale. Examples of application are given such as competitive columnar grain growth in damascene copper metallization lines, phase discrimination on metal samples, and local texture in hot extruded as well as reverse and cross-rolled magnesium based alloys.

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HIGH RESOLUTION MICROTOMOGRAPHY AND FAST RADIOGRAPHY FOR REAL-TIME AND IN-SITU CHARACTERIZATION OF POROUS MICROSTRUCTURES

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Results of high-resolution x-ray imaging allowing non-destructive investigation of the material structure are presented. The micro-structure and its evolution can be resolved down to sub-micron scale by the application of synchrotron radiation.

In-situ and real-time radiography and micro-tomography are employed for understanding structure formation processes, the optimization of fabrication technologies and the investigation of structure-functionality relations in porous materials.

Exploiting coherence properties of the radiation, holo-tomography is used for phase contrast reconstruction of organic light materials and composites with low absorption contrast. For the first time, synchrotron laminography has been established allowing e.g. the 3-d inspection of pores and cracks in flip-chip solder bumps down to sub-micron scale.

We demonstrate the potential of synchrotron-based non-destructive imaging and report results for various porous materials.

The *hardening and hydration of cement* could be observed by real-time m-resolved computed tomography. The effect of autogeneous shrinkage involved in the hydration process leading to the formation of microporosity and microcracks has been quantified on micrometer scale.

The *foaming process of metals*, fabricated by the powder-metallurgical route, was studied by in-situ radiography and ex-situ computerized tomography. Visualizing the entire foaming process inside a furnace, the formation of the foam structure, bubble growth, film thinning, drainage, film ruptures and subsequent topological rearrangements are studied and summarized in an extended model. The physics of foaming could be investigated to arrive at conclusions e.g. on the stabilizing and destabilizing mechanisms of thin metal films. We conclude on pore nucleation and early pore formation for different precursor fabrication methods.

Imaging of *non-woven materials* made of light organic fibers can be performed by methods which are sensitive to phase modulation induced by the sample in the x-ray wavefield. A propagation technique is used to obtain Fresnel diffraction patterns behind the sample. A phase contrast image of the three-dimensional sample structure can be reconstructed and serves as a basis for studying fluid flow by computer simulations.

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RECENT DEVELOPMENTS OF MULTILAYER OPTICS FOR X-RAY DIFFRACTOMETRY

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We present our recent developments of one-dimensional parabolic X-ray optics for parallel beam X-ray Diffractometry (XRD), especially for high resolution applications and reflectometry, and two-dimensional focusing X-ray optics for Single Crystal Diffractometry (SCD). You will get information on what kind of multilayer optics you could use or not for your application.

For XRD the so-called Göbel-Mirrors convert the X-ray beam coming from an X-ray tube into a monochromatic parallel beam. The mirrors consist of multilayers with laterally graded thickness, deposited extremely accurately onto a parabolic surface. Significant intensity gain was achieved using these X-ray mirrors in High Resolution Diffractometry (HRXRD) and X-ray Reflectometry (XRR). This was made possible by the development of the so-called 3rd generation GM’s. These 3rd generation GM’s consist of an ultra-precise prepolished parabolical form (with slope errors down to 1 arcsec rms) coated with an ultra-precise multilayer mirror. The talk addresses fabrication, characterization and optimization of different types of Göbel Mirrors with respect to special applications and XRD set-ups.

Our optics for SCD consists of elliptical multilayer mirrors with laterally graded thickness of the layers. They are now available for different wavelengths such as Cr, Fe, Cu and Mo.

The talk will show design, simulations and properties of the 2-D optics illustrating their advantage in the field of many different crystallography applications. We obtained flux densities above 10^{10} photons/s/mm^2 at beam diameters below 0.3 mm. Small crystals with sizes below 50 microns can now be analyzed by SCD in lab-instruments.

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THE FOCUSING MIRROR: NEW POSSIBILITIES FOR TRANSMISSION EXPERIMENTS IN THE POWDER DIFFRACTOMETER

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The capillary transmission geometry is generally accepted as the best way for minimizing preferred orientation effects in powder X-ray diffraction experiments, especially when working with organic materials, such as pharmaceutical samples. Normally, focusing incident beam monochromators are used to enhance resolution, or graded multilayer parabolic mirrors to enhance intensity.

Focusing incident beam monochromators are significantly reducing intensities. Multilayer graded parabolic mirrors are converting divergent beam to parallel beam, therefore the instrumental resolution is defined by the diameter of the capillary.

In order to combine both high resolution of focusing beam, and high intensity of graded multilayer optics, a graded multilayer elliptical mirror has been developed. In this presentation, the first results obtained with this geometry are discussed.

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GOOD PRACTICE AND SIMPLE TRICKS FOR X-RAY POWDER DIFFRACTION MEASUREMENTS: PROCEDURES FOR PREPARATION AND MOUNTING OF THE SPECIMEN AND FOR ALIGNMENT AND CALIBRATION OF THE DIFFRACTOMETER

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In the past twenty years the increases in sophistication and precision of instrumentation have greatly improved the data recorded by X-Ray diffraction. Also, the data evaluation appears to have reached almost the level of an "expert system". In comparison with these improvements the central role of how the user handles the specimens and the instrument seems to be neglected.

Careful preparation and mounting of the specimen is still a crucial first step to optimize the quality of the measurements. A step-by-step procedure will be presented that takes into account the conditions of the specimen surface, its exact position, dimensions and crystal statistics and thereby leads to a correct choice of the preparation method. Examples from practice will be given.

A sedimention method exists that produces a very thin and uniform layer of powder material, which results in sharp diffraction profiles and thus facilitates phase identification, size-strain determination and Rietveld analysis. Methods that prevent the development of preferred orientation during the preparation of powder specimens greatly improve their quantitative analysis. The protection of air sensitive materials by plastic films and capillaries will be discussed.

A versatile specimen holder with a "zero-background" substrate and built-in vertical adjustment will be demonstrated. The reduction of the background due to air scatter from the primary beam by a simple device when using a Position Sensitive Detector (PSD) is illustrated by the phase identification in tiny fragments from oil paintings.

For stress and texture determination attention will be paid to the correct orientation of the specimen with respect to the coordinate system as defined in the measuring and evaluation programs used.

The alignment of the diffractometer contains critical steps, in particular concerning beam collimation and monochromatization. Calibration measurements at regular time intervals using suitable reference materials is of great importance to keep track of diffractometer performance. A well aligned "standard" diffractometer in Bragg-Brentano geometry can even be used for reflectometry (grazing incidence) measurements of thin films.

By applying the mentioned good practice and simple tricks, the accuracy of the instrument can be used to its limits and this improves the trueness and precision of the final results.