

**MS01.02.02 KOSSEL-TECHNIQUE BY MEANS OF SYNCHROTRON BEAM EXCITATION - A NEW METHOD FOR MATERIAL CHARACTERIZATION.** Hans-Jürgen Ullrich, Jürgen Bauch, Ralph Röder, TU Dresden, Institut für Werkstoffwissenschaft, Mommsenstr. 13, D-01062 Dresden

The first KOSSEL experiments using synchrotron radiation were performed in 1992. The exposure time was drastically reduced due to the high intensity of polychromatic synchrotron radiation at HASYLAB. When using imaging plates, several seconds of exposure times are sufficient. When using X-ray films, the required time increases to the few minutes. KOSSEL patterns of crisp contrast could be obtained from elements, alloys, intermetallics, semiconductor compounds and minerals. The following problems received special attention:

- Proof of minor tetragonal distortions ( $c/a \geq 1,02$ ) in FeAl cylindrical specimen, as a function of their position in the material, determination of the value of the distortions.
- Lattice defect and texture analysis of transformer sheets. The advantage of the new method is, that the excitation depths of the X-rays for the KOSSEL patterns are extended through the entire sample thickness. Therefore, KOSSEL lines are produced at the front and rear surface of the sheet. In the case of synchrotron radiation, the pattern from the rear side of the sample are easier to compare with those of the front side in a single measurement process.

Synchrotron excited KOSSEL techniques provide a wide range of new, advantageous applications, as compared with other X-ray diffraction technique:

- Precision determination of the lattice constants in microvolumina with high accuracy ( $\Delta a/a = 10^{-4} \dots 10^{-5}$ )
- Precision determination of lattice spacings  $d_{hkl}$  of each individual net plane (hkl), with high accuracy in the analysis of residual stresses (especially the character and orientation) of the tensor of stress in a single grain
- Determination of the orientation of neighbored, crystalline grains visual in the microscope and subgrains with high precision (tilt angle  $< 0,1^\circ$ )
- Determination of tetragonal distortions of cubic lattices
- Phase identification in microvolumina by means of the topology of KOSSEL lines (qualitative phase analysis)
- In situ observation of phase transformations in environmental devices
- Measurement of the phase differences of scattered X-ray waves of polar net planes

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**MS01.02.03 SIMULTANEOUS MICRO XRF/XRD ANALYSIS OF HIGHLY INHOMOGENEOUS SAMPLES.**

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With the opening of the first really "third generation" synchrotron source in Grenoble, at fall 1994, x-ray sources of unprecedented brilliance's and qualities became available to the scientific community. Different x-ray analytical technique could now be applied on a level that was "out of imagination" only a decade ago. Here we present some preliminary results from a micro-diffraction/fluorescent experiment applied at a  $\mu\text{m}$  level carried out at one of the most powerful synchrotron microbeam available in the world right now, the BI 1 at ESRF. This beamline can now provide a  $2 \mu\text{m}$  beam with a flux density of  $10^{10}$  photons/ $\mu\text{m}^2$  at an energy of 13 keV and with a bandwidth of  $10^{-4}$ .

In this experiment x-ray diffraction and x-ray fluorescence has been combined in order to perform a kind of "micro-chemical" reconstruction of heavily inhomogeneous fly-ash particles which normally are inaccessible to conventional micro analysis. The experiment was done in a scanning mode and 2D images of different analytical information were reconstructed from the data recorded during the scan.

From the diffraction data achieved by this combined experiment, the main minerals in  $\mu$ -sized fly-ash particles have been identified and the size distributions of these minerals have also been determined. The distribution of heavy elements, recorded from their fluorescent intensity, is presented and the impact of sample surface topology and sample attenuation is discussed. Estimates of the concentration of these heavy elements are given and correlation analysis has been performed indicating that most of these elements seem to appear at the surface of the fly-ash particles.

The major features and limitations of this micro-analytical technique will be outlined and different examples on how the analytical information can be used for generating 2D images of the sample will be demonstrated and discussed.

**MS01.02.04 PROTEIN CRYSTALLOGRAPHY USING**

**CAPILLARY-FOCUSED X-RAYS** Z. Barnea<sup>a</sup>, D. X. Balaic<sup>a,b</sup>, K. A. Nugent<sup>a</sup>, R. F. Garrett<sup>c</sup>, J. N. Varghese<sup>d</sup> & S. W. Wilkins<sup>b</sup>, *a* School of Physics, University of Melbourne, Parkville VIC 3052 Australia; *b* CSIRO Division of Materials Science and Technology, Locked Bag 33, Rosebank MDS, Clayton, VIC 3169, Australia; *c* Australian Nuclear and Scientific Technology Organisation, Private Bag 1, Menai, NSW 2234, Australia; *d* Biomolecular Research Institute, 343 Royal Parade, Parkville, VIC 3052, Australia.

X-ray concentration using tapered glass capillaries is achieved by exploiting the total external reflection property of glass surfaces for glancing angles of incidence. Our group has recently demonstrated a paraboloidally-tapered glass capillary optic which produced a focused X-ray beam using a monochromatized synchrotron source. The optic produces a focal region for singly-reflected X-rays at a point 40 mm from the end of the capillary. This focus has a FWHM diameter of 40 microns and an intensity gain of two orders of magnitude over the incident X-ray intensity from the channel-cut monochromator for X-ray energies from 5 to 20 keV. We subsequently used a similar optic to obtain X-ray diffraction patterns from a crystal of hen egg-white lysozyme protein on image plates. The use of the capillary-focused beam yielded diffraction patterns 70 to 100 times faster than using an unfocused beam from the channel-cut monochromator alone. Placement of the crystal at different positions in the capillary-focused beam demonstrated the focusing of Bragg reflections and diffraction from a small volume of crystal.

The use of capillary optics with laboratory sources also holds great promise. Currently, the lower X-ray intensity available from these sources dictates long exposure times to obtain sufficient data to perform successful macromolecular crystallography. We expect that tapered capillaries can be used to reduce the exposure times required for macromolecular crystallography by a factor of more than 10, while still maintaining high resolution. The use of focused beams may also permit the study of very small crystals with laboratory X-ray sources.