

**PS01.02.17 AN INCIDENT X-RAY BEAM MONITOR FOR USE WITH PROTEIN CRYSTALLOGRAPHY AT A SYNCHROTRON SOURCE.** F. J. Rotella and R. W. Alkire, Center for Mechanistic Biology and Biotechnology, Argonne National Laboratory, Argonne, IL 60439 USA.

A compact incident beam monitor for use with protein crystallography has been developed at Argonne's Structural Biology Center at the National Synchrotron Light Source (beamline X8C). Monitoring the incident x-ray beam at a synchrotron source is critical if accurate normalization factors are to be recorded, and a beam monitor must be capable of operating through a wide dynamic range. In the present design, incident beam intensity is monitored by measuring radiation scattered from a thin polymer film into a PIN diode. The response of the diode is linear over a range of 10 orders of magnitude. For improved statistical accuracy, the scattering film can be replaced with a thin metal foil. Results from measurements with Cr, Mn, Fe and Co foils indicate that fluorescent radiation emitted by a metal foil can increase the beam monitoring signal up to 50 times that produced from scattering alone. With this detector design, the direction of the forward scattered radiation is restricted to an area not larger than the beam stop, minimizing excessive background radiation in diffraction measurements. Support materials have been optimized so that no unwanted absorption edge effects are present between 6-19.5 keV. This design makes the detector useful for monitoring incident beam intensities over a wide range of absorption edges often associated with multiwavelength anomalous diffraction (MAD) experiments. Accurate incident beam monitoring also simplifies optimization of the x-ray beam through the diffractometer collimation after each new electron orbit of the synchrotron is established. This device would be particularly well-suited for operations on third-generation synchrotron source beamlines.

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**PS01.02.18 NEW HIGH- AND LOW-TEMPERATURE FACILITIES FOR POWDER DIFFRACTION AT DARESBUURY LABORATORY.** C.C. Tang, G. Bushnell-Wye, R. J. Cernik, S. M. Clark, CLRC Daresbury Laboratory, Warrington, Cheshire WA4 4AD, United Kingdom

An induction furnace has been constructed to provide a temperature range of 290-2500 K and a cryogenic chamber using a closed-cycled refrigerator has been assembled to cool powder samples from room temperature down to 20 K. The furnace and cryostat are designed as parts of an integrated program to enhance the powder diffraction facilities at the SRS, Daresbury Laboratory. In this work we describe the commissioning of these devices for high resolution powder diffraction. Powder diffraction results from standard tungsten and silver samples are presented to demonstrate the operation of the apparatus and some of the early experimental results are included.

**PS01.02.19 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 visible 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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**PS01.02.20 A SAGITTAL FOCUSING AND SCANNING MONOCHROMATOR FOR 8-80 KeV SYNCHROTRON RADIATION AT DORIS III.** R.G. van Silfhout and S. van Dijken, EMBL, c/o DESY, Notkestrasse 85, D-22603 Hamburg, Germany

A fixed exit, double crystal Si(111) X-ray monochromator for fast energy scans over a large energy range has been designed and commissioned. Heart of the instrument is a large rotation stage operated in air that drives a platform placed in a high vacuum environment supporting both crystal assemblies. The rotation stage is linked to the monochromator crystals by a differentially pumped feedthrough hence providing a fast and accurate setting of the Bragg angle. In order to withstand the full power of a typical DORIS bypass wiggler without severe distortion the first flat crystal is cooled by water flowing through microchannels cut very close to the diffracting surface. The second crystal is mounted on an elastically deformable structure driven by piezoelectric motors. Horizontal focusing of the 60 mm wide beam is accomplished by sagittally bending the second crystal with piezoelectric based actuators equipped with precise vacuum compatible encoders. These actuators circumvent a major drawback for crystal focusing, i.e. the necessity to change the radius of curvature while scanning, and provide important corrections of crystal twist and parallelism of X-ray beam path and cylinder axis made by the bent crystal. The design was improved in terms of throughput for a wide energy range through exhaustive finite element analyses.