

refinement, or to obtain diffraction data from submicron crystal grain with high signal to noise ratio.

The microbeam has been applied to olivine ((Mg,Fe)₂SiO₄) and two unknown crystals included in a thin section of ureilite (meteorite) with thickness of 30 μm, in order to (1) obtain site occupancies of Mg and Fe in cation sites (M1 and M2) which indicate thermal history of the sample, (2) identify two unknown crystals in the rim of the olivine covered with carbonaceous material. The results are summarized below.

(1) Structure refinement was successfully carried out in an usual manner based on more than 300 Laue intensities, and site occupancy of Mg in M1 site was determined as 0.908(4) under the constraint of Mg and Fe abundant which was analyzed by EPMA.

(2) One of the unknown crystal is determined as diamond, and the thermal parameter(B) was refined to be 0.19(3)Å² by the least-squares method based on 12 Laue diffraction intensities. The other was revealed to be a face-centered cubic phase of iron (Fe) which was crystallized, toptaxial to the olivine, of Fe atoms from M1 and M2 sites under reduction by carbon. The thermal parameter (B) was also refined to be 0.06(1)Å² based on 8 Laue diffraction intensities.

[1] K. Ohsumi et al.: *Rev. Sci. Instrum.*, 66(2), 1448-1450, 1995.

MS01.05.06 STRUCTURAL INVESTIGATIONS ON MICROCRYSTALS WITH A HIGHLY COLLIMATED SYNCHROTRON BEAM. A. Bram, S. Fiedler, C. Riekel, European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France

An endstation for single crystal microcrystallography has been installed at the ESRF microfocus beamline [1]. A monochromatic beam from an undulator is demagnified by an ellipsoidal mirror. Background reduction is effected by a post collimator. A liquid N₂ cooled CCD detector with converter screen and optical demagnification is used for data collection on a K-goniometer. In order to minimize the influence of air scattering the whole goniometer can be placed in a vacuum vessel. First experiments have been performed on inorganic crystals, proteins and small organic fibres. The present setup will be shown together with the results of selected experiments.

[1] P. Engstroem, S. Fiedler, C. Riekel, *Rev. Sci. Instrum.*, 66(2), 1348 - 1350 (1995)

MS01.05.07 MICRODIFFRACTION ON Be SINGLE CRYSTALS WITH SYNCHROTRON RADIATION. S. Fiedler, A. Bram, C. Riekel¹ and M. Burghammer². ¹European Synchrotron Radiation Facility, BP 220, 38043 Grenoble Cedex, France, ²Institut f. Kristallographie, Theresienstr. 41, 80333 Muenchen, Germany.

The microdiffractometer installed on the Microfocus beamline at the European Synchrotron Radiation Facility has been used to study single crystals of Be as a function of crystal volume in order to derive accurate structure factors. Crystals of conical shape have been produced by chemical etching techniques. The smallest tips obtained had diameters between 1-2 micrometer. Crystal volumes down to a few cubic micrometer have been examined by monochromatic radiation ($\lambda=0.0687\text{nm}$) which corresponds to a minimum scattering power of $S=8 \cdot 10^{11}$ (defined as in [1]). Datasets with a resolution down to 0.04nm have been collected with an online area detector. Background scattering had to be reduced as far as possible by using microcollimators in combination with a focused beam. Results of the refinement of the extinction and the temperature factor will be reported.

W. Rieck, H. Euler, H. Schulz; *Acta Cryst.*, A44, 1099, (1988)

MS01.05.08 DIFFRACTION ANOMALOUS FINE STRUCTURE AT THE ESRF BY USING DISPERSIVE & MONOCHROMATIC DIFFRACTION. J. L. Hodeau^{1,2}, J. Vacinova¹, H. Renevier¹, P. Wolfers¹, J. F. Berar¹, M. Hagelstein², A. San Miguel², ¹Lab. Cristallographie CNRS, BP 166, F-38042 Grenoble, France, ²ESRF, BP 220, F-38043 Grenoble, France

We present several Diffraction Anomalous Fine Structure (DAFS) studies performed at ESRF and at LURE on platinum or iron oxides and on multilayers, using two different experimental modes: the **Dispersive Diffraction** mode (DD) which uses the combination of X-ray energy dispersive optics, sample goniometer and a two dimensional position sensitive detector and the **Multi Monochromatic Diffraction** mode (MMD) performed with classical monochromatic optics. We discuss our last results obtained at ESRF by using these two experimental modes for DAFS data collection. The DD technique would be precious for time dependent investigations and in-situ experiments and it could be used not only for DAFS experiments but also for other anomalous scattering experiments.

The DAFS spectroscopy provides in the same experiment information regarding the local atomic environment through X-ray absorption processes and long-range order information through diffraction processes. It can provide **site selective** and **chemical selective** structural information. Up to now, most results obtained with this method concern materials where structural information can be extracted from pure, site-selective Bragg reflections. Our studies apply the technique to highly absorbing "real" materials and to multilayers containing several anomalous atoms which do not respect the latter restriction. An accurate empirical absorption correction procedure for small highly absorbing single crystals necessary for the DAFS analysis of this kind of samples is developed. We present a **simultaneous multi-wavelength refinement** procedure of several Bragg reflections with the anomalous terms f' of different crystallographic sites as unknowns.

MS01.05.09 HELICITY SWITCHABLE HIGH ENERGY X-RAY PHASE RETARDER. J. C. Lang, G. Srajer, S. Shastri, D. Haeffner, D. Mills, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

We have developed a high-energy ($E > 50 \text{ keV}$) phase retarder capable of producing both left- and right-handed circularly polarized photons simultaneously. This phase retarder was constructed from a single Ge crystal in a monolithic Bragg-Laue design, similar to that studied previously (D. M. Mills, *Nucl. Instr. and Meth.* A266, 531 (1988)). This previous phase retarder while able to produce photons with a relatively high degree of circular polarization ($P_c > 90\%$), had a major drawback in its inability to conveniently reverse the photon helicity. The phase retarder in this study overcomes this limitation by simultaneously exciting the (220) and (2-20) crystallographically equivalent and perpendicular reflections. Each of these reflections induces an equal and opposite phase retardation between the σ and π wave fields in the Laue portion of the crystal, thus producing two x-ray beams with opposite senses of helicity. These beams are spatially separated by approximately 2 mm therefore helicity reversal is easily achieved by chopping between the two beams. A major advantage of this technique is that it retains the non dispersive geometry between the preliminary monochromator (Bragg portion) and the Laue phase retarding crystal therefore preserving the maximum possible throughput of the optic. Details of the construction and characterization of this optic will be presented.