Angle-dispersive X-ray diffraction (ADXRD) set-up for high-pressure studies using diamond anvil cells at station F3

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High pressure powder diffraction experiments in the diamond anvil cell (DAC) have special requirements for the X-ray beam. Because of the small sample size (< 100 \(\mu\)m) a narrow beam of high intensity is needed. On the other hand high-energy X-rays are necessary in order to penetrate the diamond anvils and to obtain a sufficient number of diffraction lines because the opening of the DAC is limited (usually 2\(\theta< 15^\circ\)).

These requirements could be readily fulfilled with white synchrotron radiation when high purity germanium detectors of sufficient energy resolution became available and the method of energy dispersive X-ray diffraction EDXRD was established [1,2]. However this method has some drawbacks in comparison to the conventional angle dispersive method (ADXRD). The spectra resolution is limited due to the low energy resolution of the germanium detectors (\(\Delta E/E \approx 1\%\)). Because of the small size of the detector only a very small portion (\(< 1\%\)) of the Debye-Scherrer ring is registered. In combination with the microscopic sample volume (\(< 10^4\) mm\(^3\)) this gives a poor statistics for intensity evaluation. Even so all diffraction and absorption effects are energy dependent and X-ray fluorescence lines may disturb the spectrum. This makes a Rietveld refinement from EDXRD spectra almost impossible. Finally the high intensity of the white beam may decompose delicate samples.

With third generation synchrotron facilities ADXRD experiments within a DAC become possible due to the high brilliance of these sources, enhanced by undulators and focusing optics. However, station F3 gets its beam from a bending magnet of DORIS-III.

In order to achieve a reasonable flux at F3 a compromise between intensity and wavelength resolution has been chosen by using a Mo-B\(_4\)C multi-layer double monochromator with a nominal bandwidth of \(\Delta E/E = 1.2\%\) at 8 keV. Because of the rather large layer spacing of the monochromator (\(d = 2600\) pm) the diffraction angle 2\(\theta\) for X-ray energies > 30 keV is smaller than 1\(^\circ\). The monochromator ‘crystals’ are placed in a monochromator box with independent drives for translation and rotation for each ‘crystal’. The wavelength of the monochromatic X-rays is determined by a Ge-detector which is normally used for the energy dispersive X-ray diffraction experiments at station F3. The bandwidths \(\Delta E\) (FWHM) of the monochromatic beam determined at different energies \(E\) are given in table 1.

<table>
<thead>
<tr>
<th>(E) / keV</th>
<th>(\lambda) / pm</th>
<th>(\Delta E) / keV</th>
<th>(\Delta E/E) / %</th>
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<td>41.7</td>
<td>29.7</td>
<td>0.90</td>
<td>2.1</td>
</tr>
</tbody>
</table>

Table 1: Energy resolution \(\Delta E/E\) of the multi-layer double monochromator at various energies \(E\).

A demanding task is the alignment of the tiny sample, because already small misalignments leads to strong diffraction lines from the gasket material of the DAC. For this purpose a video microscope, a fluorescence screen with a video microscope, and a photodiode with luminescence converter are available to monitor the primary beam behind the sample. The X-ray diffraction spectra can be collected with an photographic plate or an image plate. The exposure time is, depending on the size and material of the sample, from a few minutes up to 1 h. For the test experiments the MAR CCD camera from station F2.1 was available. The CCD system is especially useful for the alignment procedure because the diffraction pattern of the CCD camera can be read out in 5 seconds while the image plate requires an external scanner with a read-out time of several minutes.
The comparison between EDXRD and ADXRD is demonstrated with diffraction patterns of SmCo$_2$ and TbCo$_2$ Laves phases. In high-pressure EDXRD experiments it was found that the relative intensities of different diffraction peaks scatter quite strongly in spectra taken at different pressures (Fig. 1, left). The ADXRD diffraction pattern from SmCo$_2$ in a diamond anvil cell showed that the sample is relative coarse grained, evidenced by the spotty Debye-Scherrer rings (Fig. 1, right). By integrating over the whole diffraction rings the peak intensities become more reliable and might be used for structure refinements under pressure in the future. The peak width (FWHM) is somewhat larger in the ADXRD spectrum (2.3 %) than in the EDXRD spectrum (1.5 %).

![Figure 1: Spectra of TbCo$_2$ and SmCo$_2$ Laves phases. Left: EDXRD spectra of TbCo$_2$ with gold as pressure marker. Right: ADXRD spectra of SmCo$_2$, (inc = gasket material inconel) with CCD image (top) and an excerpt of the integrated spectrum (bottom).](image)

Some first applications of this new set-up are given in the Condensed Matter section of this HASYLAB Annual Report.

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**References**
