2θ-resolution obtainable during μ-XRPD experiments at Beamline L

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The HASYLAB microprobe beamline allows to perform μ-XRPD (X-ray Powder Diffraction) experiments on a variety of materials in transmission geometry [1]. Two important figures-of-merit of an XRPD-setup are its detection limits (i.e., the lowest abundance of a phase that is still detectable above the background noise) and its resolving power (i.e. the ability to distinguish reflections with a small scattering angle difference). Detection limits are determined by the intensity of the primary X-ray beam and the sensitivity of the detection system. The resolving power on the other hand is determined by the Full-Width-at-Half-Maximum (FWHM) of diffraction peaks as a function of scattering angle 2θ. Two types of contribution to the peak FWHM can be distinguished: (a) material-related contributions such as the finite crystallite size, strain and defects within the investigated sample; (b) instrument-related contributions such as the energy resolution ∆E/E and divergence of the primary X-ray beam. Also the point spread function (PSF) and the number of pixels of the CCD area detector used for collecting two-dimensional diffraction patterns within a solid angle behind the sample contribute to the instrumental part of the diffraction peak width.

At HASYLAB Beamline L either a double multilayer monochromator (DMM) or a double crystal monochromator (DCM) allow to select an energy band from the white beam produced by the bending magnet source. In the DMM, one of two multilayer systems can be used: Mo/Si and Ni/C. The DCM uses a pair of Si(111) crystals. The DMM’s throughput is roughly a factor 20 (Mo/Si) to 30 (Ni/C) higher than for that of the Si(111) monochromator [2]. On the other hand, the energy bandwidth is 50 (∆E/E = 1% for Mo/Si) or 100 (∆E/E = 2% for Ni/C) times wider than that of the DCM, which has a bandwidth of 0.02% at its maximum throughput [2].

The X-ray microbeam is focused down to a diameter of 10-15 µm by means of a single-bounce elliptical capillary (SBC). The divergence of the SBC focused beam is estimated between 2 and 2.5 mrad for energies in the 20-30 keV range [3], which is an appropriate range for most XRPD experiments.

For collection of diffractograms, two CCD area detectors are used: a 1K×1K Bruker SMART1000 camera with 60 µm pixel to pixel resolution and a 2K×2K MarCCD camera with a pixel size of 80 µm. Although the MarCCD camera has a larger pixel size, for capturing the same 2θ-range, it is placed further away from the sample so that twice the number of pixels are used to sample the same solid angle as for the SMART1000 camera. At high scattering angles, the same solid angle is sampled by a larger number of pixels than at lower 2θ-values. Note also that the peak distortion and widening due to projection on a flat detector is corrected for during the azimuthal integration process that calculates one-dimensional diffractograms on the basis of 2D diffraction patterns.

<table>
<thead>
<tr>
<th>Configuration</th>
<th>Monochromator</th>
<th>Energy (keV)</th>
<th>XRD Camera</th>
<th>Camera-Sample Distance (cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ni/C DMM</td>
<td>24.0</td>
<td>SMART1000</td>
<td>12.7</td>
</tr>
<tr>
<td>2</td>
<td>Mo/Si DMM</td>
<td>22.0</td>
<td>SMART1000</td>
<td>6.5</td>
</tr>
<tr>
<td>3</td>
<td>Mo/Si DMM</td>
<td>20.1</td>
<td>MarCCD</td>
<td>19.6</td>
</tr>
<tr>
<td>4</td>
<td>Si(111)</td>
<td>25.1</td>
<td>SMART1000</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Table 1. Different experimental setups at Beamline L
By means of a number of different monochromator/energy/camera distance and camera type combinations, XRPD diffractograms from a fine-grained LaB$_6$ powder standard were recorded. The details of each configuration are listed in Table 1. Each measurement was repeated 10 to 40 times, allowing to calculate the average peak FWHM and its standard deviation. In Fig. 1 the obtained average FWHM values and their 1s error bars are plotted against the scattering angle $2\theta$.

While the combination of the SMART1000 CCD camera and the Ni/C multilayer monochromator only allows to obtain a FWHM of 0.42°, slightly narrower diffraction peaks can be obtained if the Mo/Si MLM is used instead (average FWHM = 0.37°) or when the Si(111) DCM is employed (average FWHM = 0.32°). It can be concluded that the beam energy band width contributes to some extent to the final peak FWHM but that this contribution is not a major one; the peak widths can be reduced by ca 25% by switching from a primary beam with $\Delta E/E = 2\%$ (Setup 1) to an exciting beam with $\Delta E/E = 0.2\%$ (Setup 4).

The major factor determining the XRPD peak widths clearly is the total area in pixels and the quality of the CCD camera employed for diffractogram recording, as is evidenced by the FWHM values obtained by means of Setup 3, situated between 0.15-0.25°.

Figure 1. (a) The peak width versus the position is shown for the reflections of LaB$_6$, measured under different conditions: ◊: Configuration 1; △: Configuration 2; ×: Configuration 3; □: Configuration 4.

(b) Two-dimensional diffraction pattern, obtained by means of the Smart1000 detector, showing incomplete Debye ring registration in the corners of the camera area.

Note that in Configuration 3, the internal collimator of the MarCCD masks off the corners of the diffraction pattern so that only full Debye rings are recorded when the primary beam is positioned at the center of the camera. On the other hand, the square geometry of the SMART1000 camera images allows to record partial Debye ring in the corners of the camera, corresponding to large $2\theta$-values. In Configuration 2 this was done in order to cover the same $2\theta$-range as in the other configurations. This results in average peak FWHM-values with a larger spread. Since in Configuration 1 and 4, only full Debye rings were taken into account, the uncertainty on the peak FWHM does not increase with scattering angle for these configurations.

References