Progress Report on the Commissioning on the In-Vacuum, High-Resolution Powder Diffractometer

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The purpose of the experiment was to obtain high resolution powder diffraction data out to \( \sin \theta/\lambda > 1.5 \text{Å}^{-1} \) using a newly developed diffractometer, where the background is minimized by having vacuum from well upstream of the sample all the way to the image plate detector. The diffractometer was installed at beamline P02.1, Petra III. Defining all non-Bragg scattering as background, the only background source is the scattering from the capillary itself plus the Compton scattering from the sample powder¹. The first can be measured with an empty capillary, the latter can be accurately measured by a scintillation detector at 90° in the vertical plane, since at 60 keV \( \sin \theta/\lambda \) is as large as 3.4 at this scattering angle, and all scattering is incoherent Compton scattering. Using the incoherent scattering fraction from Int. Tables of Cryst. one can then determine the Compton scattering at all scattering angles. We have chosen a well characterized NIST sample of Si powder for part of this study. The diffraction patterns from the Si sample and from an empty capillary are shown in Figure 1, upper panel. By subtracting the empty capillary from the Si data, the Bragg and the Compton scattering from the Si sample should be the only to remain (Figure 1, lower panel). Despite problems with inadequate shielding, which gives rise to a higher background level than anticipated, Bragg peaks are observable at least out to \( \sin \theta/\lambda = 1.6 \text{Å}^{-1} \) and maybe out to \( \sin \theta/\lambda = 1.8 \text{Å}^{-1} \) (Figures 2 and 3). Remarkably, the widths of the Bragg peaks are essentially independent of the scattering angle and amounts to 6-7 pixels on the image plate. Each pixel is 42.5 \( \mu \) (compared to the capillary diameter of 200 \( \mu \)) and the distance from sample to IP is 300 mm, so

Figure 1: Upper panel: The powder patterns of the Si sample and of an empty capillary. The first three reflections are over-saturated and their intensities are thus not correct. Lower panel: OBS: logarithmic scale. The Si data with the capillary data subtracted. The background reaches a constant level, which originates from the Compton scattering from the Si sample.

¹In particular at 60 keV there may well be additional background due to inadequate shielding, but this can be checked by measuring the image plate response with no capillary in the beam.
this width amounts to about 1 mrad in $2\theta$ or 0.06°. An advantage of using 60 keV radiation is that the width due to the finite grain size $D$, varying as $(\lambda/D)/\cos \theta$, is so small. With a high symmetry unit cell as that of Si there is thus essentially no overlap of Bragg peaks within the entire $\sin \theta/\lambda$ range and the intensity analysis should thus be correspondingly reliable. We believe therefore that one can state, even prior to such an analysis, that this data set is at least as accurate as that reported for diamond[1], which on the other hand formed the basis for the state of the art of charge density analysis[2].

![Figure 2: The Si powder pattern at intermediate $\sin \theta/\lambda$ values after background subtraction.](image1)

![Figure 3: The Si powder pattern at high $\sin \theta/\lambda$ values after background subtraction. Peaks out to $\sin \theta/\lambda = 1.6 \AA^{-1}$ are easily detected.](image2)

References
