ESRF	Experiment title: Single crystal diffraction experiments on submicrometer crystals of CaF_2	Experiment number: HC417
Beamline: ID13	Date of experiment: from: Aug 24, 1996 to: Aug 30, 1996	Date of report: Aug. 26, 1996
Shifts:18	Local contact(s):A. Brain	*Received at ESRF:* 25SEP 1996

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Report:

Calcium fluoride CaF_2 has served as standard for microcrystal experiments [1,2] to test the feasibility of microcrystal diffraction. In the study [2] a crystal of approximately $2.2\mu m^3$ were used. The structure refinement yielded atomic displacement parameters that were higher than in powder diffraction experiments and single crystal diffraction experiments on larger samples.

This experiment was performed to reinvestigate the apparent anomaly of the atomic displacement parameters and to demonstrate the capability of submicrometer diffraction at the microfocus beamline ID 13. At the same time this experiment was used to test the vacuum option of the beamline.

During the experimental time the final components of the vacuum setup as described in the beamline handbook were installed and tested. The diffractometer itself needed to be installed and aligned as well. The installation of the vacuum components and the collimator alignment were required as well.

Tests or the instrument alignment were perfomed with a 15 μm sized single crystal of diopside ($CaMgSi_2O_6$). Reflections could be observed up 2Θ = 90°. Under vacuum conditions the experiments could be carried out without primary beam collimator. Comparisons were performed under ambient pressure and under vacuum. Under ambient pressure only the strongest reflections could be observed above the background. Under vacuum no significant background above the intrinsic detector background could' be observed. The vacuum option effectively eliminates any background due to air scattering. Despite the large primary beam cross section no background scattering from the sample holder is observed.

The observed intensities were weaker than expected from experiment HC416. This is only in part due to the 16-bunch mode.

By using the vacuum option the detector has to be positioned at approximately 260mm behind the sample compared to 50 mm in the standard setup. This limits the opening angle of the detector to 22°.

Only one dataset could be collected using a 0.2 μm^3 CaF_2 crystal at λ=0.6883Å. The scattering power of the sample corresponds to approximately 6*1010. This value is slightly less than the scattering power of the smallest kaolinite sample of experiment HC416 and represents the smallest scattering power of an individually mounted single crystal in a monochromatic diffraction experiment. The data collection was performed in the oscillation technique at nominal 2Θ = 8, 25 and 42°. Since the detector is mounted off-center, the actual 2Θ angles are approximately 12° higher. No reflections could be observed at the 42° measurement. A detailed analysis of the intenisties and strucure refinement are being carried out at present.

[1] Bachmann, R., Kohler, H., Schulz, H. & Weber, H. Acts Cryst. A41, 30-40 (1985)
[2] Rieck, W., Euler, H. & Schulz, H. Acts Cryst A44, 1099-1101 (1988)