

Experiment title: Broad band-path monochromator for perturbation crystallography

Experiment number:

MI165

Beamline:

ID11

Date of Experiment:

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Shifts:

15

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Report:

The induced changes in a sample when it is perturbed by an electric field, or irradiated by a laser beam etc. are very small. Hence studying these small changes on a conventional X-ray source is rather time consuming and several weeks for a full data set are needed. However with the now available high flux of the third generation synchrotron sources, the measuring time is reduced to a few days. To decrease the measuring time even more, a new technique, was developed.

The method is based on a broad energy-band pass[1], so that a thick Ewald shell is created and the whole mosaic spread of a particular reflection lies within this shell. Instead of taking an omega scan through the Ewald sphere, as in the conventional method, only one single setting in omega is needed to obtain the integrated intensity. The combination of the broad-band pass with a digital lock-in amplifier and a Ge-detector with a large dynamic range[2], decreases the measuring time by an other factor of 100.

After initial alignment and optimization of the beam in order to get a flat energy spectrum, a data collection on AgGa_2 was taken with this new method. Ten reflections could be measured per hour and in total 500 reflections were measured. From these 75 reflections show a significant change in integrated intensity between 0.05%-.5%. Some other reflections show no significant change in integrated intensity. And some reflections were not intense enough to get good counting statistics.

This results in 150 to 200 reflections that can be used in a structure refinement to determine the induced changes in atomic distances. Currently, a program is being developed in order to directly refine the atomic reorientation against the observed intensity changes.

It is to be noted that this new method allowed for the first time enough reflections to be measured do to a full structure refinement.

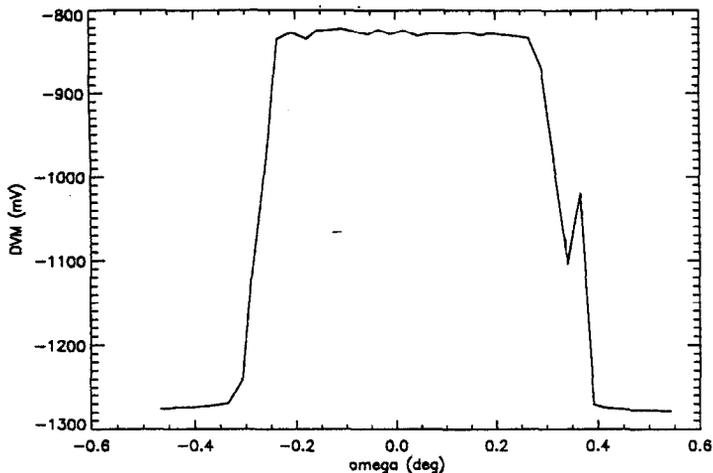


Figure 1 shows the analyser (=Si(660)) spectrum of the monochromator Si(111). The glitch on the hand right sight is from the bent laue Si crystal. The top part of the spectrum is not perfect flat due to phase contrast caused by the non-polished Be-windows. The $\Delta E/E$ is about 2%.

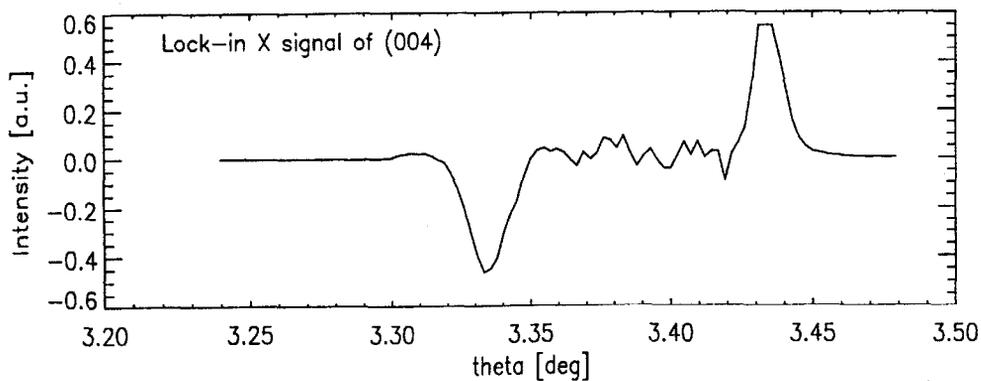


Figure 2 shows a typical rocking curve of a particular reflection. NOTE that the rocking curve was measured to be sure of the flatness of the broad-energy band. Theoretically only one single point at the plateau needs to be measured for obtaining the AI. The AI for this reflection is 0.07

- (1) Graafsma et al. Submitted to J. Appl. Cryst. (1997)
- (2) Graafsma et al. J. Synchrotron Rad. (1996). 3, 160