Experiment report – Confocal diffraction – Feb.2010 @BM26A

Introducion:

This is a simplified experiment that aims for a proof of principle. The objective is to be able to determine if a certain mineral is present in a 2D sample at a defined depth of its thin surface, in combination with an energy scan like would be performed in a fluorescence experiment.

The idea is to use diffraction in back reflection from the sample. Fixing the energy of the X-ray, the angle of diffraction of any peak is known for a certain mineral. A collimator can be used to confocally align the incoming beam with the path of the diffracted photons to the detector. This is confocal diffraction and will allow to detect the presence of a certain mineral at a defined depth. Simultaneous diffraction for different minerals is possible just by considering more diffraction angles in the collimator for the respective minerals, for the same energy. The setup configuration is illustrated in Figure 1.

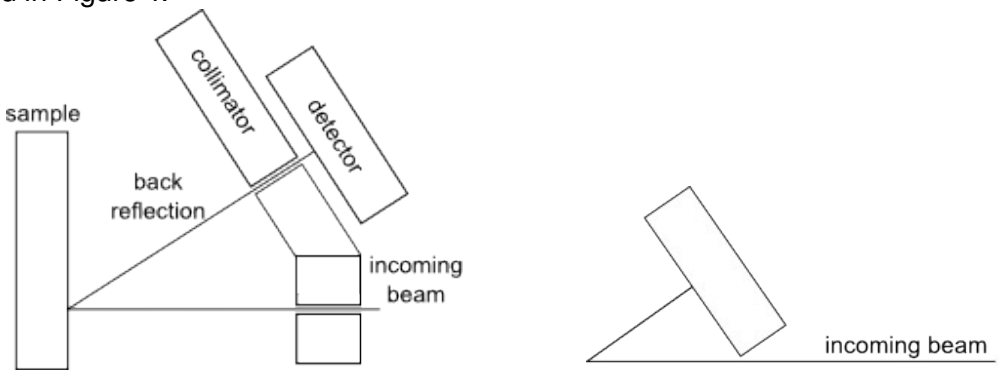

Figure 1 - Scheme of the setup: side view and top view

The angle calculated for this prototype setup, corresponds to one diffraction peak for Hematite at 8KeV. The detector, used for the first time, is a Mythen from Dectris, a linear, position sensitive detector.

Experiment:

The first thing to do is, without the collimator in place, to acquire a full diffraction pattern within the limits of the detector. The first sample to be used, was a layer of iron ore oil paint that should contain hematite based pigments. No peaks were detected with this sample. A second sample used, was standard Si powder in a capillary, and this showed to be diffracting. Third sample was hematite powder obtained by grinding a portion of hematite rock. Like the first sample, it gave no diffraction pattern. So far, the setup was like illustrated in Figure 1. The top view shows how the detector was placed sideways looking at the sample.

At this point, the detector position was adjusted and it was placed perpendicular to the beam. In this configuration, the back reflection diffraction pattern of the Si powder became sharper, like illustrated in Figure 2, on the left hand side. Trying the Hematite powder again and still no pattern was obtained. The doubt now relies on the sample. Why is this hematite powder showing no peaks just like in the artificial pigments of the oil paint?

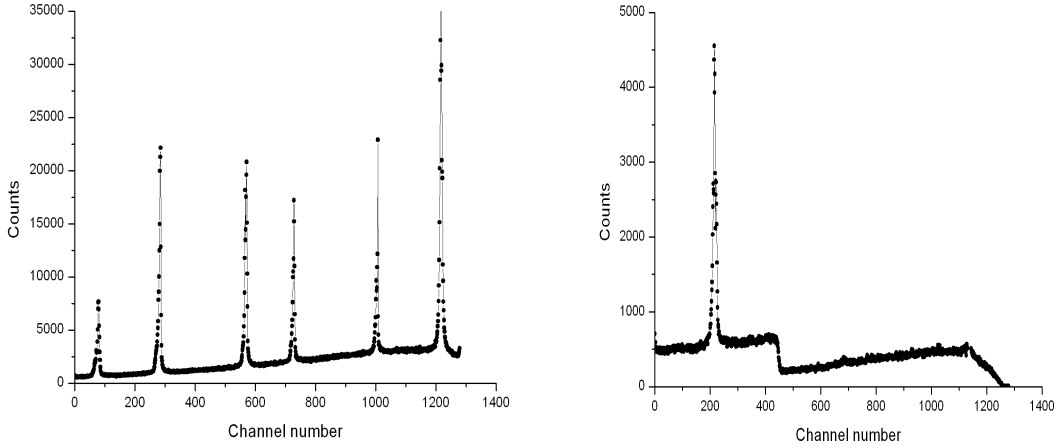

Figure 2 - Si diffraction pattern and single peak collimated

The following step was to put the collimator in place, but just the lower half of it to start. The purpose was to filter a part of the diffraction pattern. This was successful and is illustrated in figure 2, on the right hand side.

Both the sample and detector were mounted on independent motorized towers so that respective aligning would be easy. This is also useful and important when trying to make a Si peak pass through the collimator. The minimum collimator gap for the back reflected photons is of 10µm. This would give a calculated depth resolution of a few tens of microns.

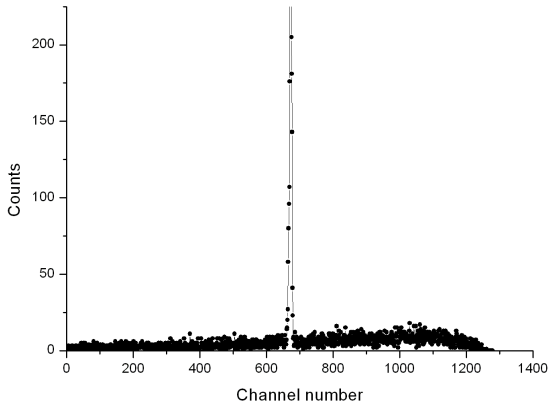

Figure 3- Si peak passing through collimator gap

To start with the full collimator mounted, the gap was increased to some comfortable 900µm. The way to displace the peaks along the detector channel number was by moving the beamline monochromator manually by small steps. A peak passed through the full collimator like illustrated in Figure 3.

The last step was to try the same but with a small gap, of about 50µm. The search for the peak passing through the gap was difficult and more time and more preparation are required. Furthermore the Mythen detector is not synchronized with the monochromator steps and it makes the procedure less controllable.

Conclusion:

For a first experiment some advances were achieved, like detecting a back reflected diffraction peak through the collimator. Next steps are to use a well chosen hematite sample and see a diffraction pattern; to define a procedure for aligning the collimator with beam and sample; try to synchronize detector with monochromator; and finally try to succeed with a small collimator gap.