Experimental report of MI-1262

The goals of our experiment were:

1. Measurement of Kossel line patterns produced by fluorescent atoms (electronic relaxation) within a short period (in the second range). Demonstrate the efficient work of our Kossel camera.
2. Determine the fine structure of many Kossel lines parallel for known, simple inorganic compounds. Obtain Kossel patterns with good enough quality for the determination of the phase and amplitude of the structure factors (first time).
3. Measurement Kossel patterns of biologically interesting samples (first time).

In our previous experiment (MI 1209) we have built a setup for Kossel line measurements. We have measure the Kossel patterns of various samples and we could determine the phases of the structure factors of these lines. However, the amplitudes of the diffraction lines could not be precisely determined and we had to measure longer than the expectation based on the incident beam intensity. According to our analyses the main reason of these problems was the detector pixel inhomogeneity. In this experiment we intended to solve this by using two different detecting elements and an improved version of the experimental setup.
Therefore we modified our Kossel camera. The main modification was the introduction of a shielded tube between the sample and detector. We also changed the mechanical setup by replacing the vertical linear detector stage with a rotational motion of the sample about a horizontal ax. We tried two detectors: a. Dectris Eiger 1M ; b. imaging plate. As beamline optics we used a similar setup as in our earlier measurements except the KB mirror, which was replaced by diffractive lennses giving a larger beam spot (approximately 80 micron) than the KB mirror. However, this was not very much disturbing, since the pixel size of the detector was also in this range (75*75 micron2).
This setup gave a spectacular improvement of data quality. We could see the most intense Kossel lines even without any background correction (fig.1.).

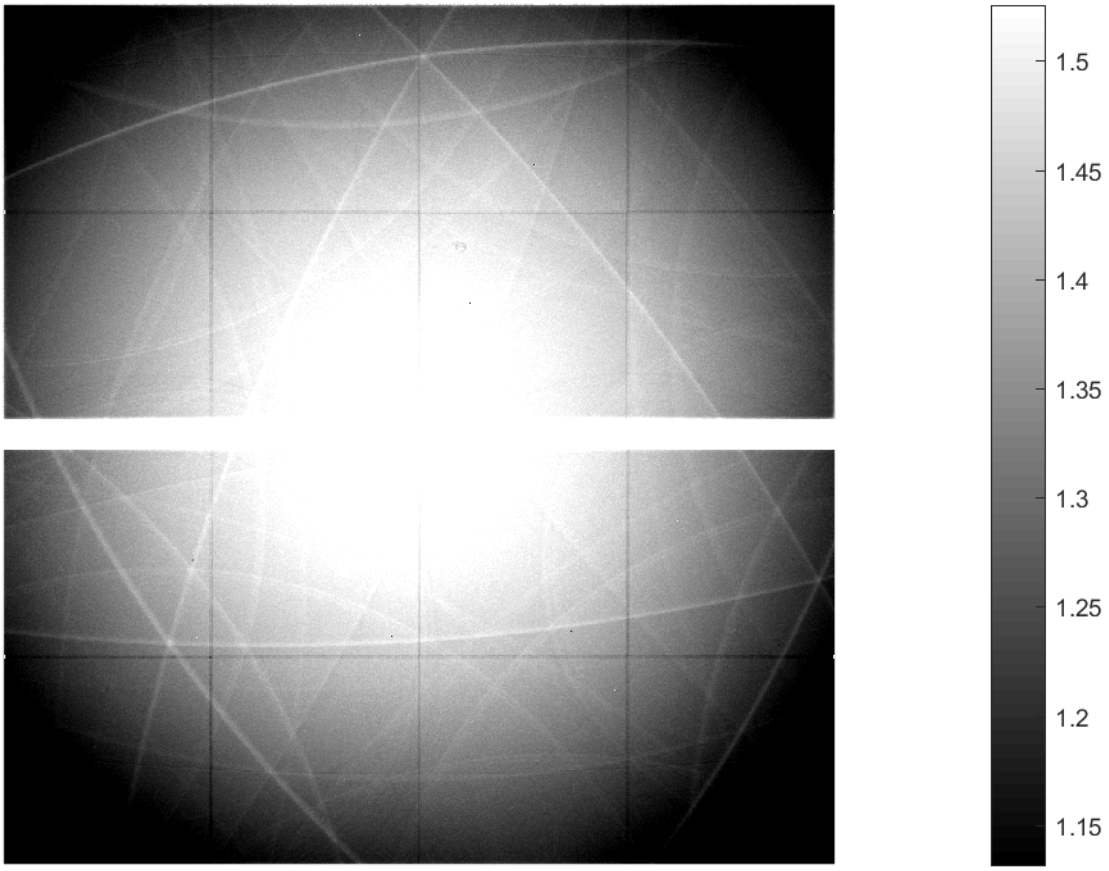

Fig.1. Kossel pattern of NiO taken at 90 mm from the sample. No correction was applied. The intensity scale is 10^8

We could take patterns of simple inorganic compounds within a second (though usually we used longer times for better statistics). So point 1 was successfully satisfied.
To determine the phase and amplitude of the structure factors (point 2) we took high quality patterns of various samples (see for example the Ge Kossel pattern fig. 2.).

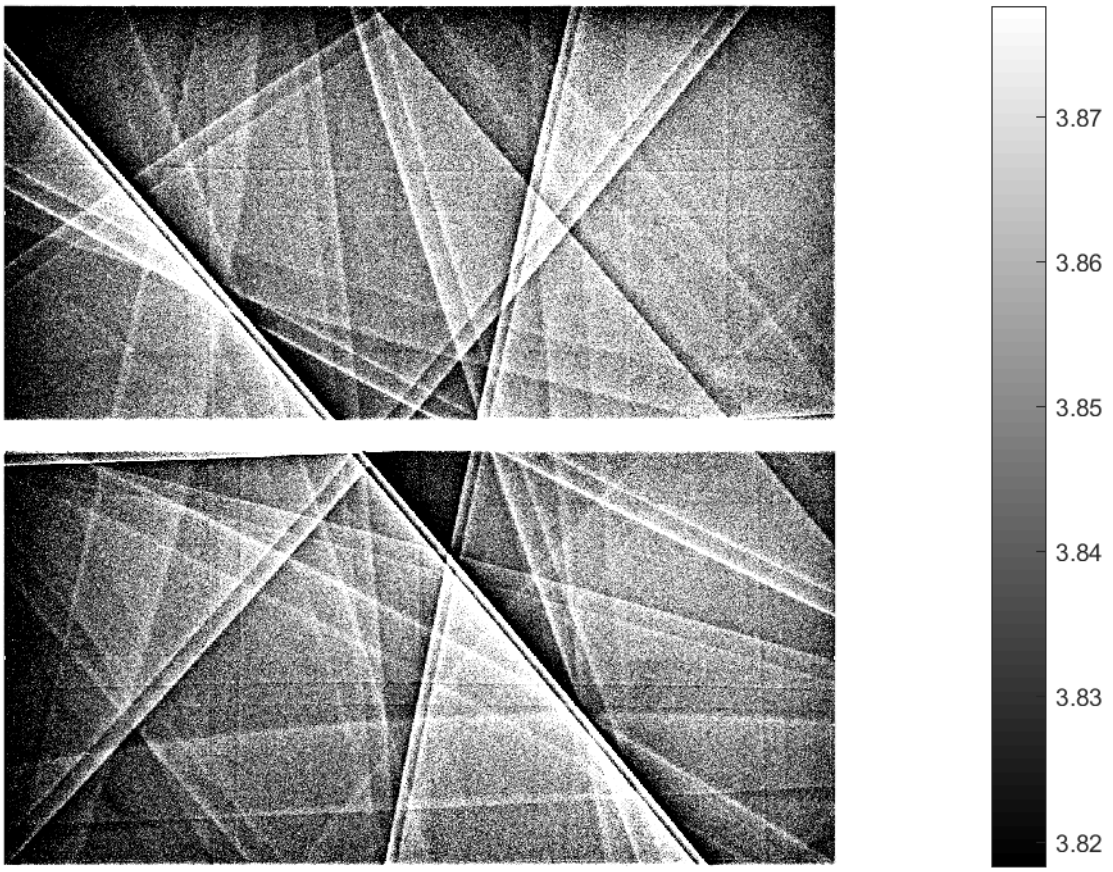

Fig.2. Kossel pattern of Ge, taken at 850 mm from the sample. The fine structure of lines are can be precisely determined, because of the good signal to noise ratio and good detector pixel homogeneity.

Compared to our earlier experiments these patters look much better. We are working on the detailed evaluation of these data. Preliminary results are encouraging; we expect to obtain the phases and amplitudes of two-three samples.

We tried the same experiments (as was described above) with image plates as detecting element. One could see Kossel lines however the image quality is very bad (see fig.3.). The reason for this is the bad image plate quality and the non-homogeneous reader. The image plate measurements are important from the point of view of future XFEL experiments. This type of detecting seems to be the best choice for single pulse Kossel pattern experiments.

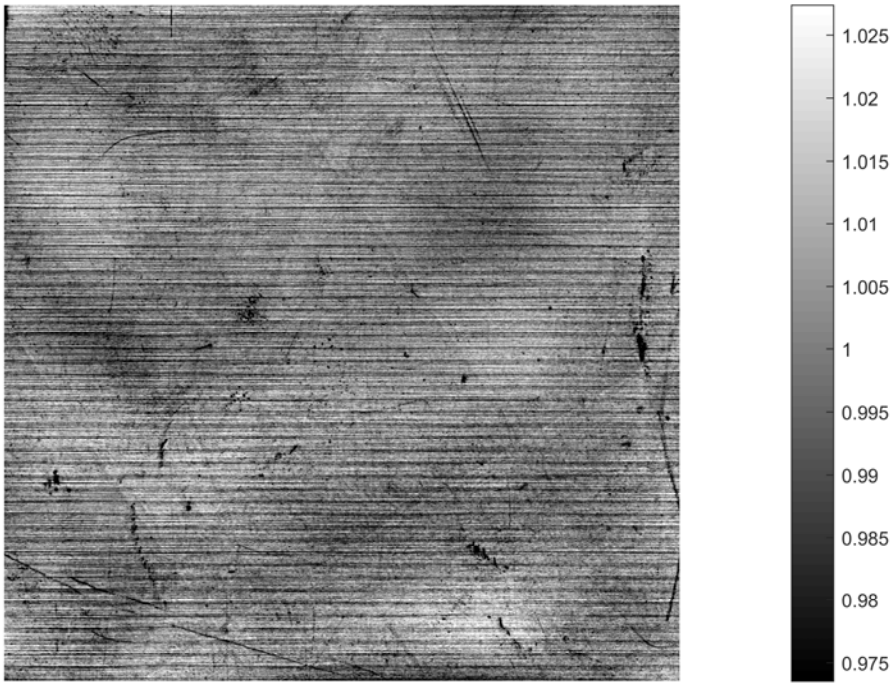

Fig.3. Kossel pattern of NiO using an image plate as detecting element. One can see both the very bad image plate quality (various inhomogeneities of the plate) and the reader problem (horizontal lines).

As far as the 3rd point is concerned (measurement of Kossel pattern of biologically interesting samples), we tried to collect data on myoglobin and hemoglobin samples. These crystals were grown by professor Naoya Shibayama and Ayana Sato-Tomita. The crystal quality were checked at our home laboratory by traditional single crystal x-ray diffraction. From the 18 protein samples we chose 3 (2 myoglobin and 1 hemoglobin) for checking. We found that the crystal quality was good. We made Kossel measurements at several samples both at liquid nitrogen and at room temperature. We measured time series to see (and avoid if possible) radiation damage. Usually, 1000 patterns were measured 1 ms each, resulting in 1s total measuring time. In a second step we measured 10 more seconds to have pictures with better statistics. However, we expected radiation damage for these longer measurements. Unfortunately, we could not see any Kossel lines. We think that the problem is at least two fold: first, the low Fe concentration of the samples results in very low fluorescent signal and secondly there is significant radiation damage for longer collection times. We believe that with further efforts one could see Kossel patterns on biological samples.

We work on the detailed evaluation of the measurements and expect one or two publications in the next year.