ESRF	**Experiment title:** Experimental development: Stress determination at the grain scale using the Kossel diffraction technique	**Experiment number:** **MA-168**
Beamline: ID13	**Date of experiment:** from: 06 December 2006 to:10 December 2006	**Date of report:** 20/02/2007 *Received at ESRF:*
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Report:

Main goals :

The knowledge of local stresses additionally to other features of the real structure is of great interest. In order to obtain **micronic or submicronic spatial resolution**, the strain measurements must be performed without sample rotation. Among the techniques under investigations which allow stress determination at the grain scale, the most developed is the white beam x-ray microdiffraction (ALS Berkeley or ESRF BM32) with a sub-micronic resolution [1]. This method needs specific optics (KB / 4 crystals monochromator), and under white beam condition, only the deviatoric stress tensor can be measured (the complete stress tensor analysis needs a second measurement with a monochromatic beam). It has the great advantage that it can be applied to any kind of crystalline material. Our purpose with this proposal is to study and develop at ESRF an alternative method, called the **Kossel diffraction**, which could allow the **same lateral resolution, a complete stress tensor analysis in one step, with a very accurate determination of the lattice parameters ($10^{-4}/10^{-5}$)**. It has also the great advantage to be very simple in terms of optics (micro focus capillary for instance and CCD camera). It is well adapted to metallic materials and can be extended to any material (pseudo-Kossel method). When a fine focused electron beam (in a SEM) or X-ray microbeam irradiates a crystal, it causes fluorescent radiation (characteristic X-rays) which may be diffracted in the target crystal itself. When using synchrotron radiation for the excitation of Kossel diffraction lines, as shown by H.-J. Ullrich and co-workers in 1992 for the first time [2], a further scaling down of the analyzed specimen area (in the range of μm^2) is not in conflict with an acceptable exposure time (a few seconds or minutes for bulk materials). The principle of the technique is schemed in Fig. 1.

Experiments and results

Nevertheless, because of the very weak signal, applications on thin films have never been proved until we performed, on Dec 2006, first experiments on ID13. We measured the Kossel lines of a copper thin film (750 nm thick) deposited on a Si(001) substrate (700 µm thick). The average grain size is about 1 to 3 µm (Fig 2a). After alignment, the beam size was 1x1.5 µm². We choose an incident beam energy (9.2 keV) just above the copper absorption edge (8.9 keV). The camera was a Mar CCD 165. The selected grains have been detected and positionned through the imaging of one Bragg peak on the Debye ring (Fig 2b). During these first tests, we showed the great influence of the background measurement and background subtraction. We especially determined a background (equivalent to a flatfield) by averaging the images obtained on several grains (here 8 grains). By this way, we succeeded in obtaining Kossel lines from the copper grains. We also observed pseudo-Kossel lines from the Si substrate (diffraction of the Si due to the Cu kα emission) (Fig 2c). This could be of great importance in the strain accuracy determination, as we can use these lines for the CCD calibration. We are now working on the use of these pseudo-Kossel lines. The measurement time is nevertheless quite long (about 12 h for 8 grains + background) and the final signal is weak and noisy. As a consequence the accuracy on the strain determination is still poor. We believe that we can increase the quality of these measurements by the following improvements:

- CCD position: during the last experiments, the sample was tilted at 45° from the incident beam and the normal to the CCD was also at 45° from the sample surface. As a first consequence, only half of the CCD was illuminated (see figure 1b). Then, the sample to CCD distance was not optimized (around 10cm). To increase the signal/background ratio, we need to be closer (4- 5 cm) with a CCD in front of the sample.
- Use of a multilayer mirror instead of a Si crystal in order to increase the energy band-width of the incident beam (=> increase of the incident flux)
- Use of a more sensitive detector (new Frelon camera)

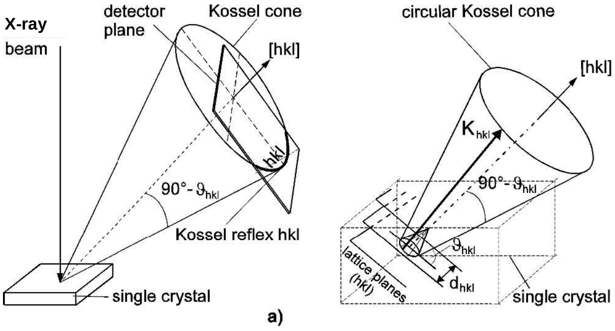

Fig. 1 : scheme of the Kossel diffraction method;

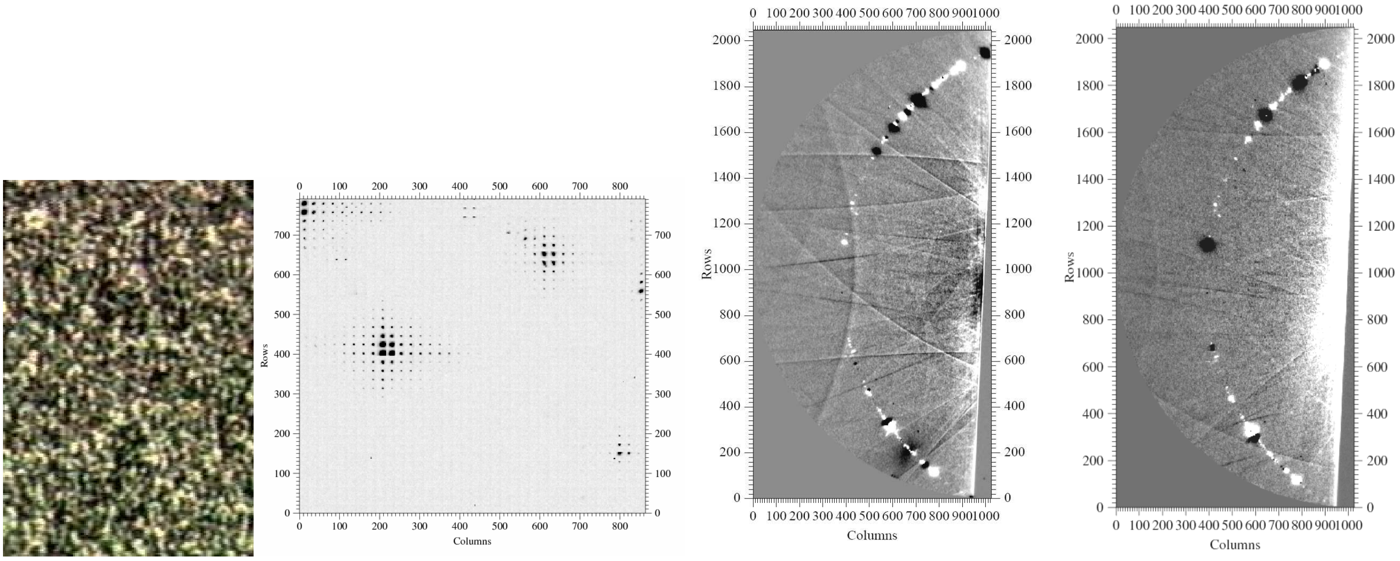

Fig. 2: a) optical image of the copper film surface b) Mapping of the grains obtained by measuring a Bragg peak on the debye ring; c) Example of images obtained on the copper film showing Kossel lines but also pseudo-kossel lines from the Si substrates (these lines are on both images).

Conclusion

We succeeded in obtaining Kossel lines from a sub-micronic copper layer. We believe that the quality of these first measurments can be greatly enhanced. We also believe that this kind of measurements is possible on thinner films. Finally, the next step of this project is 1 / to enhance the quality of these first images 2 / to determine the strain from Kossel lines. An ANR project (French funding) has been deposited this year.

1 N. Tamura et al, Appl. Phys. Lett. 80 (2002) 3724-3727.
2 H. -J. Ullrich et al, Nuclear Instruments & Methods in Physics Research A 349 (1994) 269-273