ESRF	**Experiment title:** SCANNING MICRODIFFRACTION FOR NUCLEATION OF RECRYSTALLIZATION	**Experiment number:** HS-262
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Report:

Recrystallization occurs each time a polycrystalline material is plastically deformed at elevated temperature ($T > 0.5T_f$) or when the priorly deformed material is significantly heated, as it is usual in geomaterials (eg. ice in polar ice sheets, olivine in the Earth upper mantle, ...) but also in the elaboration procedure of industrial materials. Recrystallization mechanisms are supposed to depend (i) on the relative orientation of grain boundaries and crystallographic lattices and (ii) on the stored energy of dislocations. The first can be measured either by standard X-ray texture techniques, or by Back Scattered Electron Diffraction (EBSD) in a Scanning Electron Microscope. The second property is available either by calorimetric methods or by X-ray diffraction profile analysis [l]. The calorimetric methods can not be made local at present, and do not have the resolution in order to measure elastic stored energies with good enough precision. X-ray diffraction profile analysis has been proved to bc precise enough to follow the variation of the elastic stored energy in plastically deformed metals and alloys [l]. There is good hope that this method can be made local down to the micron or even submicron scale by applying a fine X-ray beam. The first experiments of Biermann and coworkers [2] to measure the local variation of diffraction profiles on a monocrystalline Ni-base superalloy have shown that the method can be applied successfully with a Bragg Fresnel Lense (BFL) at the optic beamline BM5. For these experiments, the monocrystalline specimens were cut with different (100) planes and the profiles of the (400) reflections were scanned by the z-y movements of one of the towers at BM5. The reflections were adjusted by small tilts also available on this tower. Another important feature for the success of these measurements was the access to a high quality linear position sensitive detector borrowed by the company Braun, Munich [2].

The aim of the present experiment was to measure the dislocation density in individual grains of a polycrystalline material (low carbon steel) with usual grain size (20 - $30\mu m$). The technique consists in measuring the shape of the diffraction peak for at least 3 or 4 reflecting planes of a same grain. Each

reflection provides the value of a formal dislocation density. The real local dislocation density is given by a combination, via a geometrical correction, of all formal densities. The experimental setup requires :
1/ a spot size on the sample smaller than the grain size, and a beam with a very high photon density.
2/ to recognize a particular grain (which orientation was previously measured by EBSD) of the surface of the sample, to put the grain into the beam, and to be able to rotate the sample around that grain so that reflections on several crystallographic planes can be obtained.
3/ to measure on a linear position sensitive detector the shape of the diffraction profile with a sufficiently small background $(I_{min}/I_{max} \leq 10^{-3})$ for a proper treatment.

A linear Bragg Fresnel Lense (BFL), with a focal distance of 76 cm, was used as monochromator (24 keV) and to concentrate the beam on the sample. The linear detector (spatial resolution : $100\mu m$) was 60 cm away from the sample to have a sufficiently large angular resolution. A thin gold grid (step : $10\mu m$) was deposited on the sample to act as coordinate system. An optical microscope was installed in front of the sample, to see the grid, so that the grains to be measured can be easily recognized.

During this first experiment of our project, a large amount of experiences regarding the experimental requirements and conditions have been collected, which are absolutely necessary for the successful1 continuation of our measurements at the next beamtime.

- The background measured on the detector was extremely large (I_{min}/I_{max} could not be reduced below 10^{-1}) even by covering the detector with thick lead sheets. Furthermore, there were some technical problems with the linear position sensitive detector, what made measurements very difficult. It was thus not possible to record diffraction profiles properly. To avoid this next time we must install the mirror and the primary monochromator before the BFL, and thus use the BFL only as an X-ray collimator. We are trying to arrange for our own linear position sensitive detector which we shall bring for the next beamtime.

- The lack of a proper 4-circles goniometer was a great problem. The sample was mounted on several superimposed single mouvement stages, which were very difficult to align all together at the same time. A proper goniometer is unavoidable for our experiment. We plan to bring our own 4-circles goniometer with an x-y stage for the next beamtime.

- The thin gold micro-grid we prepared to recognize individual grains was only hardly visible with the optical microscope. In the next ESRF experience, the grid will be made thicker.

- We tryed first to align the beam with the center of rotation of the 'goniometer' and with the grain to be measured by using one of our polycrystalline sample, with the help of the optical microscope. It turned out that this method was not efficient. Reasons are that the microscope is not sensitive to X-rays, and that the polycrystalline material contains too many lattice orientations. We have tested another method which is more powerfull. We used an X-ray camera and a silicium monocrystal on which a cross of thin tungstene wire was glued. Since both BFL and tungstene wire can be seen at the same time on the X-ray camera, the alignment of beam, grain, and goniometer was possible.

This first experiment at ESRF was necessary for understanding the experimental difficulties, and for improving the setup of this experiment which will provide for the first time, once the setup will be efficient, the local dislocation density in polycrystalline materials at the scale of a few microns, a data of major importance in material science.

[l] H. Biermann, T. Ungar, T. Pfannenmiiller, G. Hoffmann, A. Borbely, H. Mughrabi, Acta metall. mater., 41, 9, 2743, 1993.
[2] H. Biermann, B. v. Grossmann, S. Mechsner, H. Mughrabi, T. Ungar, A. Snigirev, I. Snigireva, A. Souvorov, M. Kocsis, C. Raven, Scripta Mat., in press.