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Report:

Recrystallization occurs each time a polycrystalline material is plastically deformed at elevated temperature $(T > 0.5T_f)$ or when a deformed material is heated above a threshold temperature, as it is usual in geomaterials (e.g. ice in polar ice sheets, olivine in the Earth upper mantle, . ..) and in the elaboration procedure of industrial materials. Recrystallization mechanisms depend (i) on the relative local orientation of grain boundaries and crystallographic lattices and (ii) on the local stored energy associated to the lattice distortion created by the dislocation array. 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 Line Profile Analysis (LPA) [l]. The calorimetric methods is not adequate for our purpose because it can not be made local. LPA has been proved to be precise enough to follow the variation of the elastic stored energy in plastically deformed metals and alloys [2]. With conventional X-ray source, this method is however limited either to materials exhibiting a grain size larger than about 500 μm, or, for smaller grain size, to the simultaneous measurement of all grains that have an $\{hkl\}$ plane normal to the diffraction vector. Local dislocation density can be measured accurately only if several reflections are recorded for the same grain. Therefore, conventional methods cannot be used for industrial metals, which exhibit typical grain size between 1 μm and 100 μm. In a first experiment, Biermann and co-workers [3] have shown that LPA can be applied successfully with a Bragg Fresnel Lens at the optic beamline BM5 to measure local variations of the lattice parameter of monocrystalline turbine blades, at a scale of a few tenth of microns. For this experiment, symmetrical (400) reflections were used and the position of the bulk monocrystalline specimen was adjusted by small s-y translations.

The aim of the present experiment is to measure the local dislocation density in individual grains of several samples of polycrystalline low carbon steel exhibiting a grain size of 40μm, deformed 25 % in cold rolling, and annealed at several temperatures. Results expected are the dependence of the stored

energy on the lattice orientation and on the annealing treatment.

A similar experiment was attempted one year ago at BM 5 (see report HS-262) and a large amount of experiences regarding the experimental requirements was collected. During the present experiment, the setup was developed further and successful measurement could be performed. The interpretation of results is actually under progress. The main experimental difficulties to be overcome are the following :

1. profiles acquisition requires a linear (or planar) position sensitive gas filled detector, a small background $(I_{min}/I_{max} \leq 10^{-3})$, and a small spectral width $(\delta\lambda/\lambda \leq 10^{-4})$ to avoid significant instrumental broadening.

2. the size of beam needs to be smaller than that of grains in order to be sure that reflections come from a single grain, and a high brilliance is necessary to avoid excessively long acquisition time;

3. a goniometre with a very small sphere of confusion $(\approx 10\ \mu\mathrm{m})$ is required; to be able to measure several reflections from the same grain, the beam, the grain to be scanned, and the centre of the rotation of the goniometre needs to be aligned very precisely;

*To **point** 1* : mirror and monochromator from the optic hutch were installed and adjusted for a 16 keV monochromatic beam. A 2-D gas filled detector with energy discrimination, developed at ESRF, was positioned at 65 cm from the sample, giving a resolution $\delta q/q \approx 10^{-4}$.

*To **point** 2* : the beam was focussed both horizontally and vertically with a compound refractive lens (focal distance: 1.18 m, beam divergence: 9×10^{-5}) , leading to a spot size on the sample of 13 x $3.3\mu\mathrm{m}^2$ (H xV) and a gain of flux of about 55 as compared to the unfocussed beam.

*To **point** 3* : at home, a thin gold grid (step: 50 μm) was deposited on the sample surface to act as a coordinate system, and the lattice orientation of grains lying in this grid was measured by the EBSD technique, so that goniometre angles can be calculated for each reflection and each grain. The centre of rotation of the 6-circle Hubert goniometre of ID22 could be determined precisely by putting a small calibrated polystyrene ball (ϕ = 80 μm) in the beam, and visualising the phase shift produced by this ball with a high-resolution camera (resolution: 0.67 μm) placed in the direct beam. The alignment of the beam with the centre of the goniometre was easily performed by translating slightly the lens. The next crucial point was to align a particular grain of the sample with the centre of the goniometre. To do this, 2 small polystyrene balls ($\phi \approx 50\mu\mathrm{m}$) were glued on the sample surface. Each sphere was successively aligned with the centre of the goniometre with the same high-resolution camera as above; then, knowing the position of a particular grain with respect to the 2 spheres, reflections on individual grains could be obtained.

This experiment was the very first microdiffraction experiment performed in reflection for which several reflections could be reached from the same grain of size as small as 40 μm. The 15 shifts of beamtime were largely used to develop the setup and the method of alignment, but still 120 reflections coming from 20 different grains of the unannealed specimen could be measured, i.e. 6 reflections per grain and 30 min per reflection in average. The relationship between sample orientations for the different reflections insures that diffraction profiles really come from the desired grain. The accuracy of sample position was found to be as small as 50 μm, and it can be further reduced by taking more rigid translation stages for the sample holder.

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