ESRF	**Experiment title:** Experimental Determination of the Intragranular Work Hardening State in an Aluminum Multicristal	**Experiment number**: HS-391
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Report:

1. Investigated Samples

Because of some technical difficulties concerning the preparation of the aluminum multicrystal, the experience has been carried out on three copper samples (see Fig. 1): (a) undeformed single crystal, (b) and (c) cold rolled bicrystal and multicrystal (35 % reduction).

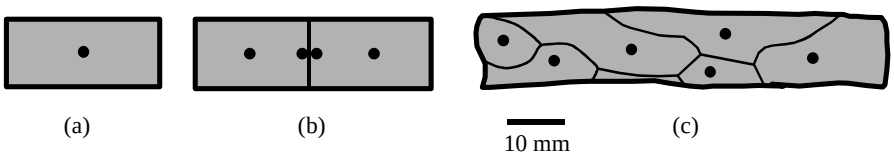

Fig. 1: investigated copper samples: (a) single crystal, (b) and (c) the deformed bi- and multicrystal. Black circles refer to X-ray spots.

These samples have been previously investigated in the laboratory by diffraction of classical X-rays ($CoK_{\alpha 1}$ of Cobalt tube), which had enabled the characterization of the diffracting surface layer. It has been shown that the dislocation density varies substantially from grain to grain indicating a specific work hardening state within each individual grain. But cold rolling is suspected to lead to a heterogeneous deformation state in the depth of the samples which is 1 to 2 mm. Such heterogeneity can not be characterized by reflection. Therefore, a volume investigation seems to be necessary for the complete description of the work hardening state.

2. Experimental and analyses of X-ray profiles

The high energy X-rays and a Triple Crystal Diffractometer (TCD), needed for the volume investigation by transmission diffraction, are available in the beam line ID15A. The wavelength was set to 0.124 nm and the size of the x-ray spot was fixed to 1×1 mm^2. Several peak profiles related to different diffraction vectors {<200>, <220>, <222> and <111>} were measured on every investigated region (see Fig. 1). After the background treatment, every profile was analyzed on the basis of the Fourier transform. The model of Groma-Ungar-Wilkens relates the Fourier coefficients to the dislocation density by the formula:

$$\ln|A(n)| = -\eta\rho^{*} n^{2} \ln(R_e/n) + Kn^{4}\ln(R_2/n)\ln(R_3/n),$$

where $\eta = \pi/2\ g^2 b^2$, **g** the diffraction vector, **b** the Burgers vector, ρ^* the formal dislocation density, n the Fourier parameter, R_e the outer cut-off radius of the dislocation structure, K a parameter related to the fluctuation of the dislocation density, R_2 and R_3 are mathematical constants. The real dislocation density is related to the "formal" one by the relation $\rho^* = C\rho$, where C is the factor of the geometric contrast of the dislocations.

3. Results

The analyses of the profiles measured on the undeformed sample (see Fig. 1) reveal very small dislocation density ($\approx 10^{12}$ m^{-2}) which confirms the high resolution of the TCD (see Fig. 2). However, the other measurements show a high level of a diffused background of almost 2 counts/s. Compared to the maximum of intensity varying between 40 to 100 counts/s for deformed samples, this background level does not allow precise analyses of the profile broadening. Therefore, special software has been written in order to treat the profile tails before the Fourier analyses (see Fig. 2). A general comparison between the dislocation densities obtained by the transmission experiment and those obtained by the previous investigations carried out in the laboratory with X-ray tube is shown in Fig. 3. According to this Figure, the dislocation densities obtained in transmission (ESRF) are almost twice those obtained by reflection (laboratory).

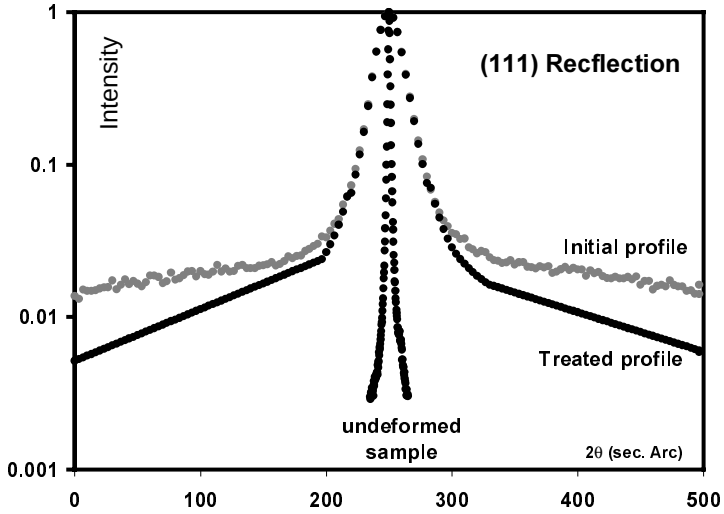

Fig. 2: elimination of the background and comparison with the undeformed sample

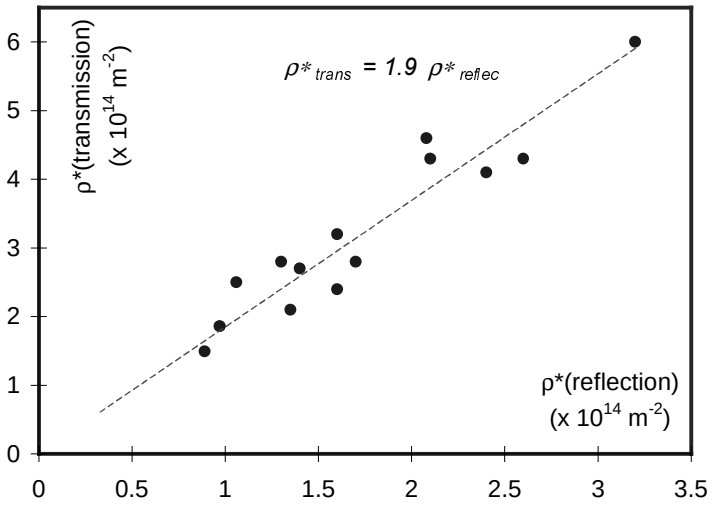

Fig. 3: relation between the reflection and the transmission experiment.

Measurements carried out on the bicrystal (see Fig. 1) show that the intergranular dislocation density on both sides of the grain boundary is the same and is the average of the two intragranular dislocation densities. This new result confirms that grain boundaries do not lead to a systematic excess of intergranular work hardening.

4. Conclusions

Results obtained from this work were very helpful for the characterization of the deformation heterogeneity in the depth of cold rolled samples. It has been shown that the relationship relating dislocation densities measured on the surface layer and in the volume is simple: ρ (volume) $\approx 2\ \rho$ (surface). A physical interpretation of such relation is not available, because of the complex strain and stress state generated in the surface layer during deformation by cold rolling.

5. Remarks :

The high level of the diffused background in the hutch does not allow an easy analyses of the x-ray peak profile broadening. The measurement of the diffraction profiles by means of the rotation of the analyzer (third crystal) implies the recording point by point, which leads to long counting time (2 – 3 hours per profile for deformed samples). In our case we may suggest the use of a linear detector which allows direct recording of the whole profile.