IMPURITIES EFFECTS ON THE MECHANISMS OF RECRYSTALLIZATION OF WIRE-DRAWN COPPER

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Within the framework of project OPEFIC partly subsidized by the French ministry of research, the mechanisms of recrystallization in cold wire-drawn copper were finely studied in order to understand how the presence of very low content of impurities (sulphur in particular) can delay the recrystallization. After plastic deformation, the quantity of stored energy, mainly in dislocations form, is a parameter determining because it is the driving force of the static recrystallization process [1-4]. This stored energy is related to many parameters such as the material type, the deformation mode, temperature and level, the crystallographic orientation, the previous deformation crystallographic texture, etc.
However, very few direct investigations have been done to clarify the influence of impurities content on the stored energy in copper [5,6]. These works allowed to get some quantitative values of stored elastic energy in global scale through differential scanning calorimetry measurements. In order to obtain a relationship between crystallographic orientation and stored energy, neutron diffraction measurements have been done on the four-circles diffractometer 6T1. The global crystallographic texture has been characterized from experimental {111}, {200}, {220} and {311} pole figures; the Orientation Distribution Functions (ODF) were calculated by the discrete ADC method [11]. The stored energy values were deduced from the line broadening measurement achieved on different poles corresponding to the main preferential orientations.

Two kinds of commercial (including 99.99 mass %) copper, having different noticeable mechanical properties and chemical compositions were studied. The first selected copper, named A material, is very pure and consequently was regarded as reference (S amount about 3.2 ppm). The second one, copper B, has a higher S amount (about 8.2 ppm). Both of them have approximately the same oxygen content (about 170 ppm). The materials resulting from continuous casting, has undergone a hot-rolling to obtain a diameter of 8 mm. This treatment was followed by a cold wire-drawing to 6.3 mm (reduction of area Δ=38 %).

After cold wire-drawing, the ODFs (figure 1) of materials A and B clearly show major <111>//DN and minor <001>//DN fiber textures at this level of reduction(38% reduction area). These two components are less pronounced in the material A than in the material B. The volume fractions of texture components including the reinforcements (determined by the Helming decomposition method [7]) are about V_f =14% and V_f =43% for the material A, and about V_f =22% and V_f =52% for the material B, respectively for the <001>//DN and <111>//DN fibers. Furthermore, it was noted that for both materials A and B the volume fraction of the fiber <111>//DN is almost twice larger than the <001>//DN, taking into account that the dispersion around the fiber components is equal to about 15°.

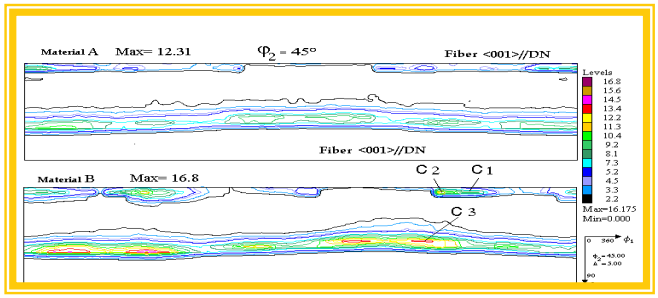

Figure 1: φ_2 sections (45°) of the ODFs after wire-drawing process for A material a) and B material b). The main reinforcements noted on the figure, are C1:{001}<110>; C2:{001}<120> and C3: {111}<112>.

Concerning the stored energy values, some line broadening measurements were carried out for the A and B materials. The figure 2 shows an example for a given point of the pole figure {200} of a set of analysed diffracted peaks corresponding to the A material after a hot-rolling and a cold wire-drawing. The full width at half maximum (FWHM) is greater after wire-drawing of about 15% for copper A and by about 23% for copper B than after hot-rolling. This broadening contains two contributions, which have been separated, i.e. the contribution due to the size (D) of diffracting

domain and the contribution due to the microstrains related to plastic deformation, from which the stored elastic energy (SE) is estimated.

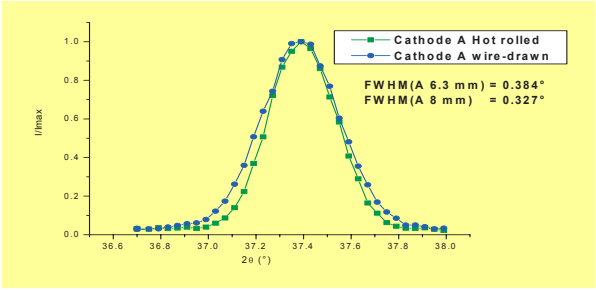

Figure 2 : θ-2θ scan of {200} Bragg peak corresponding to A material after hot-rolling and wire-drawing.

The measurements of the broadening peaks in the cold drawn copper have shown that :the stored elastic energy values are lying in a range between 1.8 - 4.6 J/mol (Table 1) and the stored elastic energy increases with the rate of residual impurities. On the other hand, the stored elastic energy ratio <111> /<001> decreases when the rate of residual impurities increases.

	<111> fiber	<001> fiber	$E^{<111>}/E^{<001>}$
A material	3,6	1,8	2,0
B material	4,6	3,8	1,2

Table 1 : Stored energy values in the grains belonging to the two fiber components [8].

The deformation substructure has been studied by Transmission Electron Microscopy. The observations are in agreement with the measurements of the stored elastic energy. Indeed the grains associated to <111>//DN fiber are more deformed than the grains associated to <001>//DN fiber. Moreover, the dislocation cells associated to the grains belonging to the fiber <001>//DN are less defined and present a weaker mean size in the presence of impurities (Figure 3).

During recrystallization annealing, the grains related to the <001>//DN fiber nucleate first by cell coalescence near in the highly deformed and misoriented regions taking into account the presence or not of the impurities.

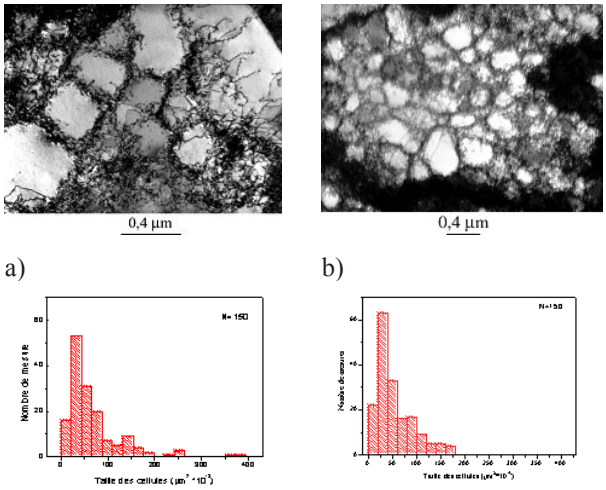

Figure 3. TEM observations and distribution of the cell size (on 150 measurements) on Material A a), and Material B b).

The stored elastic energy ratio would be a driving force during nucleation and explain why the pure copper recrystallizes first during annealing. These impurities seem to delay the process of recovery necessary to the formation of the nucleus as well as the mobility of the grain boundaries during the recrystallization by the means of an inter-granular segregation.

References

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