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Annealing twin formation and recrystallization study of cold-drawn copper wires from EBSD measurements

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Abstract

The crystallographic texture and microstructure of an electrolytic tough pitch copper have been investigated by Electron Back Scattered Diffraction (EBSD) after cold wire drawing (reduction in area between 52% and 94%) and after primary recrystallization.

The material presents a deformation texture composed of major ⟨111⟩ and minor ⟨100⟩ fibers. The evolution of the quality index of the Kikuchi patterns shows that the stored energy is lower in the ⟨100⟩ fiber than in the ⟨111⟩ fiber. Then, after recrystallization, the volume fraction of the ⟨100⟩ fiber increases at the expense of the other texture components.

The study of the grain boundary nature shows that the recrystallization twin fraction decreases with increasing strain. It is shown that this evolution is the consequence of the grain size reduction.

Keywords: Copper; Wire drawing; Recrystallization; Twin; EBSD

1. Introduction

The recrystallization development of copper (or alloys with similar recrystallization behavior such as Ni alloys) is now quite well known. Indeed recent studies [1–3] have examined and explained different steps of the recovery and recrystallization processes and their influence on the microstructure and the texture development.

So, it is now well known that to grow, a nucleus needs to have a sufficient size that can be attained by dislocation cell recovery, see Ref. [4]. For copper, when the strain amount is low, all grains, i.e. all orientations, have the same probability to grow, and the recrystallization texture becomes isotropic because of the twinning mechanism which involves the formation of new orientations. For a high amount of strain produced by cold rolling, the {100} ⟨001⟩ cube orientation dynamically recovers during deformation. Then, this component quickly develops at the expense of the deformed matrix. Consequently the recrystallization texture is essentially composed of the cube component plus its twin orientation. In all cases, when the nucleus has reached a “critical” size, it grows at the expense of the deformed matrix by bulging. This is possibly due to the large stored energy difference between the nucleus and the matrix [5]. Let us finally note that a nucleus can twin during the first stages of its growth [6] and several twin generations may be observed before complete recrystallization [7,8]. This short-term recrystallization behavior clearly shows the great importance of the twinning mechanism, which is still not well understood.

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Table 1
Chemical composition of copper given in ppm (in weight)

Element												
C	O	Si	S	Cl	Fe	Co	Ni	Zn	As	Se	Sn	Pb
2.3	170	0.03	3.2	0.02	2.8	0.03	0.09	0.17	0.01	0.03	0.03	0.26

In the present study, EBSD measurements have been performed on wires after different cold drawn reductions and also after recrystallization. This study focuses on the twin boundary volume fraction in order to understand the formation of the recrystallized microstructure.

2. Experimental details

The material classified as Electrolytic Tough Pitch (ETP) contains a minimum copper concentration of 99.99%, i.e. the impurities level is less than 100 ppm (the impurity content of this material is given in Table 1).

The copper wires were industrially hot rolled (8 mm diameter) and then cold drawn. The identity, the section reduction, the final diameter after deformation and the true strain of each wire are given in Table 2. After working, the copper wires were annealed at 260 °C for 10 min in an oil bath.

The EBSD measurements on the deformed and recrystallized samples were performed on an SEM (Scanning Electron Microscope) equipped with a W-filament. The OIM™ software was used to analyze the orientation maps.

3. Results and discussion

3.1. Deformed specimens

The wire-drawn texture in copper and some alloys can be described as a combination of the ⟨111⟩ and ⟨100⟩ fibers [9]. By neutron diffraction experiments, Gerber et al. [9] have shown from orientation distribution function calculations that the intensity of the ⟨111⟩ fiber increases with increasing strain. The ⟨100⟩ fiber is less intense than the ⟨111⟩ fiber, but the intensity related to the ⟨100⟩ ideal position tends to increase with increasing strain.

Table 2
Diameter, section reduction and true strain of the A, B, C and D cold-drawn wires

Sample name	Diameter after reduction (mm)	Section reduction (%)	True strain
A	5.54	52	0.73
B	4.24	72	1.27
C	2.57	90	2.27
D	1.93	94	2.84

These results are consistent with those described in the literature. Indeed, it has been shown that the ⟨111⟩ fiber is a stable orientation in the deformed state [10], while the ⟨100⟩ component is obtained as the result of a combined dynamic recovery and recrystallization process [11]. Gerber et al. [9] have then assumed that the volume fraction of recovered and/or recrystallized ⟨100⟩ grains in the deformed state increases for the highest strain level. This hypothesis is verified from the volume fraction calculated from EBSD measurements (Fig. 1a). This figure shows the increase of the ⟨100⟩ fiber volume fraction when the strain amount increases and Fig. 1b shows that the white points (good Quality Index—QI) correspond to the recovered or recrystallized grains of the ⟨100⟩ fiber. Let us note that the quality of the Kikuchi patterns is too bad to estimate the ⟨111⟩ fiber volume fraction.

The QI distribution can be used to estimate relative values of the stored energy, which is the driving force for recrystallization [4], in the two different fibers. Fig. 2 shows that the QI factor is greater for the ⟨100⟩ fiber than for the ⟨111⟩ fiber (with a 15° spread). Then, the stored energy is lower in the first fiber than in the second. This result has already been observed from neutron diffraction measurements [12]. Finally, it is interesting to note that the stored energy difference (Fig. 2) between these two fibers increases with the amount of strain. This gives to the ⟨100⟩ grains the ability to grow at the expense of the matrix during recrystallization, especially after high strain.

3.2. Recrystallized specimens

Fig. 3 shows the microstructural evolution along the radius of the different wires. The grain size is calculated excluding twins and it appears that the grain size decreases when the strain amount increases (Fig. 4a). Moreover, no significant change (small increase for the A sample) of the grain size is observed along the sample radius (Fig. 4b) except for the last point of each curve which corresponds to points at the periphery of the wire. In this particular area, a shear strain can modify the recrystallization mechanisms and thus the grain size.