Materials Science Forum Vols. 654-656 (2010) pp 1570-1573
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doi:10.4028/www.scientific.net/MSF.654-656.1570

Grain Fragmentation in Equal Channel Angular Pressed Copper

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Keyword: UFG, ultrafine-grained, grain size, EBSD, grain refinement

Abstract. A comparative experimental and simulation study of oxygen-free high conductivity copper produced by equal channel angular pressing (ECAP) one-pass has been carried out by using electron backscatter diffraction (EBSD) and a recently proposed grain refinement model. The grain size and misorientation distributions were extracted from the EBSD measurements. It was found that the microstructure in the ECAP deformed copper was much more refined on the TD plane. The grain size observed experimentally can be fairly well predicted by the grain fragmentation model.

Introduction

There is currently considerable interest in the development of ultrafine-grained (UFG) metals by employing severe plastic deformation (SPD) processes [1-4]. However, the grain refinement process is not yet fully understood. Existing attempts to understand grain refinement have focussed on the effect of the initial orientation of a grain and its neighbours and the deformation field itself [5-7]. However, the gradual change of the grain population due to grain subdivision has largely been ignored [6].

Very recently, a new simulation model [7] was developed in which the grain fragmentation is based on lattice distortion of crystals due to the constraining effects of their neighbors in the polycrystal. The model is applied in this study to explain the differences in the degree of grain refinement during ECAP.

Experimental Procedures

Oxygen-free high conductivity (OFHC) copper was heat-treated at 650˚C for 2h. The billet of the annealed copper was machined to 20 mm × 20 mm × 120 mm size and processed by ECAP to one pass and with die angle of 90˚. The back pressure was 25 MPa and a forward speed was 2 mm/s. Colloidal graphite was used as lubricant. Specimens were cut on three different sections from the center of the deformed sample; ED, TD and ND (extrusion, transverse and normal directions, respectively) and were examined by EBSD. The specimen were mechanically polished to 4000 grit using SiC paper and then electro-polished for 20 seconds in an electrolyte of 25 pct orthophosphoric acid, 25 pct ethanol and 50 pct distilled water at 10V, 20°C with a current of ~150 mA. The EBSD measurements were performed using a JEOL 7001F FEG SEM fitted with a HKL detector, with a step size of 0.2 µm. Grain boundaries were identified using a minimum misorientation angle of 5° between adjacent pixels. In order to have representative data, at least three maps of 80 µm × 80 µm in size were measured on each plane. Analysis was performed by using the new software of Beausir [8]. The misorientation of each pixel with his four (north, south, east and west) neighbours is examined. When the misorientation exceeds the "grain tolerance angle" (here 5°), a boundary is defined. Once all the pixels boundaries are defined, a flood-fill procedure is applied to search for sub-surfaces delimitated by a close boundary. The latter are defined as grains having an orientation corresponding to the mean orientation of all the pixels constituting the grain.

The measured orientation maps are in form of inverse pole figure maps. Misorientation distributions of pairs of first neighbors were also generated from the so-defined grain distributions.

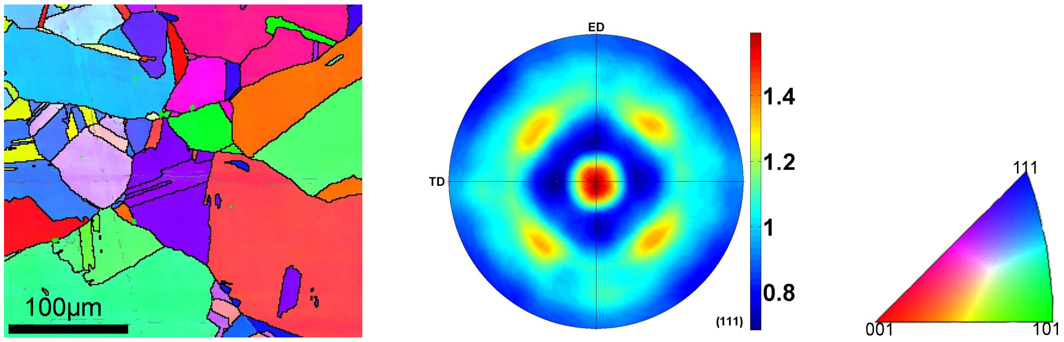
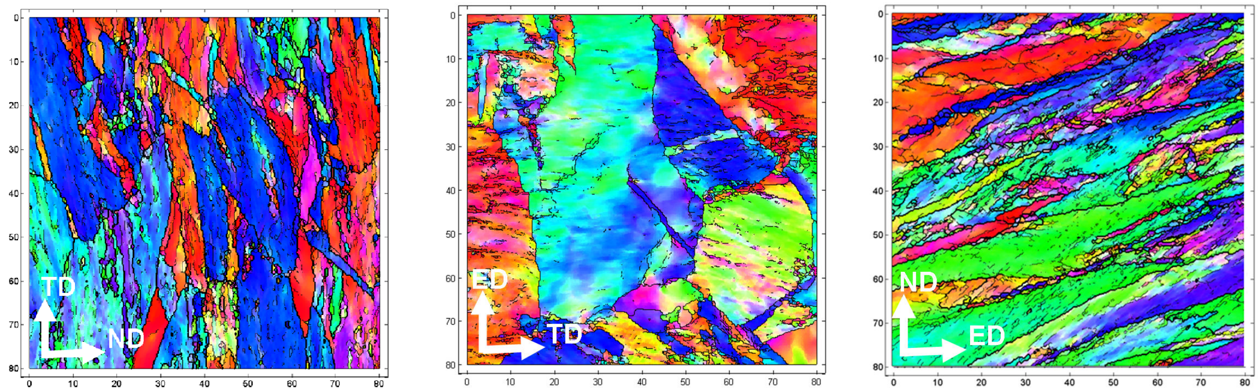


Figure 1 Representative orientation map and {111} pole figure of the as-received copper. The colour code for the orientations is also shown.

Results

The average grain size was 24 µm in the as-received copper, Fig. 1. After ECAP, the microstructure of the coarse grained copper became elongated on the ED and TD planes. On the ED plane, the original grains were flattened perpendicular to the ND direction, Fig. 2a. On the TD plane, the grain axes were inclined at about 25° with respect to ED as expected from the simple shear model of ECAP (Fig 2c). The grain morphology was similar to the initial one on the ND plane (Fig. 2b). The subgrain sizes after ECAP were in the range of 1.04-1.86 µm obtained as $d = 2 \times sqrt(S/pi)$ where S is the surface of the grain for all three planes. With the low-angle boundary fraction of about ~13% on the ED plane and ~5% on the ND plane which were measured from 15° misorientation criteria. However, ~86% high-angle boundaries were detected on the TD plane of the ECAPed 1 pass sample. The microstructure was more refined on the ED and TD planes compared to the ND plane, Fig. 2.



(a) (b) (c)

Figure 2 Representative orientation maps after one ECAP pass in pure copper: (a) ED plane, (b) ND plane and (c) TD plane. Boundaries with at least 5° misorientation are marked with grey lines.

For the misorientation calculation, the correlated distribution was computed, which is defined as the density function of misorientations between neighboring grains [8]. The measured neighbor-to-neighbor misorientation distributions of the one pass ECAP-ed specimen on the ED, ND and TD planes are displayed in Fig. 3.

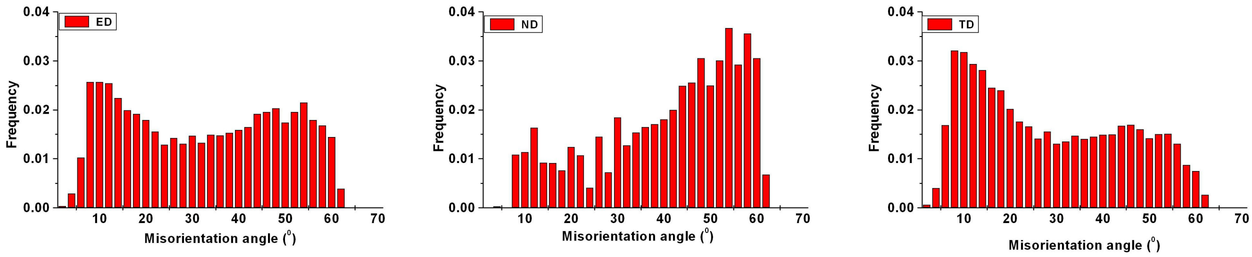


Figure 3 Representative experimental neighbouring grain misorientation distributions after one ECAP pass for copper obtained on the: (a) ED, (b) ND and (c) TD planes.

Discussion

During deformation, it is reasonable to make the assumption that the concomitant rotation of the crystallographic planes of the crystal is impeded in regions near the grain boundary so that the lattice rotation of the grain boundary region is smaller than that in the middle part of the grain. Based on experimental observations made by EBSD in plastically deformed grains [9-11], our hypothesis is that the grain interior can be divided into two zones: a shell near the grain boundary where lattice rotation is smaller and a core that is situated in the middle part of the grain, which is not affected by the grain boundary. A crystallographic plane is curved within the near-boundary shell zone due to the difference in the degree of lattice rotation.

In the simple cases that we presented in this model, the geometrical position of a grain boundary is parallel or perpendicular to each other, but during deformation the rotation-impeded distortion of a plane can be coniderably more complex. During the fragmentation, the assumption is that the core zone and the shell zone are of equal size and that each grain boundary slows down the lattice rotation with equal efficiency (being represented by a coefficient of grain boundary friction, μ, which ranges from 0 to 1). We also assume that the plane is curved as a circular arc between two adjacent points and that its local crystallographic orientation is dependent on the position along the arc and varies continuously.

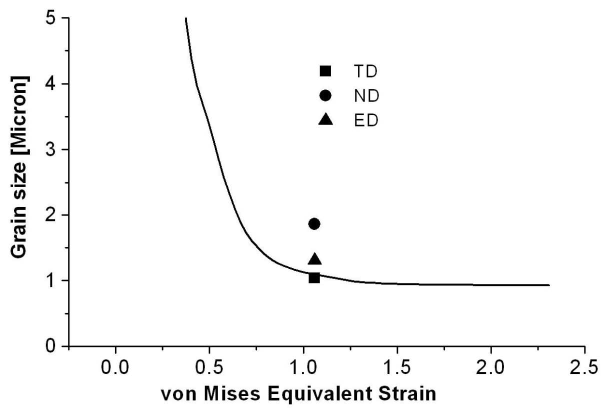
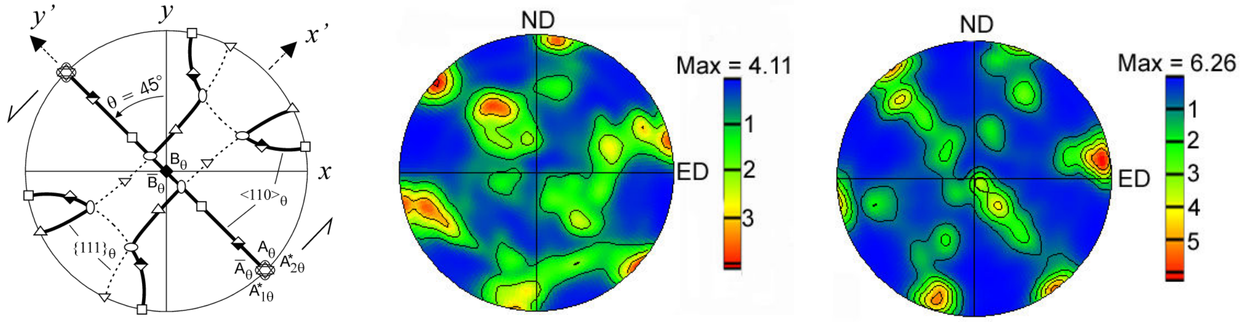


Figure 4 Simulated (continuous lines) and experimental (symbols) development of average grain size obtained in ECAP-ed 1 pass

In the simulations, a polycrystal was created from 500 grain orientations, which were selected from the initial texture, Fig. 1. Grain sizes were also allocated to individual grains according to a lognormal distribution, with the smallest grain size of 14μm and the largest of 35μm. In this case-study, the initial texture was weak and not distorted by the volume fraction of the grains in any significant way. For numerical implementation, each of the initial 500 grains having a particular orientation was subdivided into embedded cubic elements. After each strain increment, the misorientation between neighbouring subgrains was calculated, and a special algorithm was used to identify a contiguous region that was misoriented by less than 5°. The simulation procedure led to a grain size of 1.2 μm (with the coefficient of grain boundary friction of 0.5) which is fairly close to the experimental observation (Fig. 4).

To test whether the measured misorientation distributions on different planes are fully representative of the microstructure, pole figures were plotted from the orientation maps. If the local texture is very close to the known macroscopic texture of the process, then the corresponding local microstructure should be representative as well. For easy comparison, the textures obtained from the local EBSD measurements were examined and were expressed in the same reference system for both the ED and TD planes, Fig 5 (presented on the TD plane). As can be seen, both textures are near to the expected textures after one ECAP pass of pure copper [6]; the developed texture component are all near to the ideal orientations, see Fig. 5a, Therefore, the local measurements were representative of the whole sample.



(a) TD ideal orientation (b) from TD plane (c) from ED plane

Figure 5 Ideal orientation (a), textures obtained by EBSD maps from the TD plane (b) and the ED plane (c). Both textures are shown in the TD projection.

Conclusion

A comparative experimental and simulation study of ECAP-ed one-pass copper has been performed. It was found that the microstructure in the ECAP deformed copper was much more refined on the TD plane than on the other orthogonal planes. The grain size observed experimentally can be fairly well predicted by the grain fragmentation model.

Acknowledgement

C.F. Gu acknowledges Monash Access Equity Research Travel Grant (2010) for her stay in Metz, France. L.S. Tóth acknowledges an ARC International Fellowship Award through an ARC Linkage International Grant (LX0989895) from Australia, and B. Beausir is grateful to the Alexander von Humboldt Foundation for his research fellowship.

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