

Effect of strain path on grain refinement in severely plastically deformed copper

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Grain subdivision was studied experimentally with orientation imaging for oxygen-free high-conductivity copper deformed to the same equivalent strains in equal-channel angular pressing (ECAP), route A up to two passes, and in cold rolling. Grain refinement was modeled using a recently proposed grain refinement model. The refined grain size was significantly smaller in ECAP than in rolling in both experiments and modeling. The result shows that it is the lattice rotation rate that is responsible for the differences. © 2010 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

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The development of ultrafine-grained bulk materials by severe plastic deformation (SPD) processes is of interest because of the increase in yield strength resulting from ultrafine grain sizes [1–3], but this benefit is tempered by the increased energy required for SPD manufacture. Consequently we must rationalize the supposed benefit by comparing SPD processes with conventional forming techniques. Among the SPD techniques used to induce a very fine microstructure, equal-channel angular pressing (ECAP) has considerable potential compared to rolling (Figure 1).

Recently a quantitative polycrystal grain refinement model was proposed [12] which incorporates a damping effect of grain boundaries (GB) on the lattice rotation adjacent to the boundary. This damping effect produces lattice distortion and consequent subdivision of the grain. The Taylor polycrystal model is used in the model with subdivision into $3 \times 3 \times 3$ elements of an idealized grain which is initially cube shaped. These elements can then be further subdivided in the same manner down to two further levels. Starting with 500 grains, nearly 10 million can be produced. The model has previously been validated successfully for the ECAP of oxygen-free high-conductivity (OFHC) copper in route Bc, two passes [12]. There is only one parameter in the grain refinement model that is related to the lattice curvature (μ). The

same value ($\mu = 0.5$) was used in the present work as in the first application of the model [12], where successive simulations confirmed this choice of the parameter.

The present research work examines the differences in the grain refinement process when different processing routes are applied on the same material. ECAP and cold rolling were selected and the grain refinement state was characterized by orientation imaging using the technique of electron back-scattering in scanning electron microscopy. Simulation results confirm the basic hypothesis of the grain refinement model, namely, a larger lattice rotation rate leads to a smaller grain size, in agreement with the experimental observations.

The material studied was OFHC copper in the form of a rod which was heat-treated at 650 °C for 2 h, resulting in an average grain size of about 24 μm (Fig. 2a). The texture of the rod was very weak, nearly random (see the $\{111\}$ pole figure in Fig. 2b). The billet of the annealed copper was machined to dimensions of 20 mm $\times$ 20 mm $\times$ 120 mm and processed by ECAP to one and two passes, route A, in a die with 90° angle. The long axis of the original rod was the extrusion direction (ED). A back pressure of 25 MPa was applied to the billets during ECAP. The extrusion speed was 2 mm s^{−1} and colloidal graphite was used as a lubricant. The shear strain rate during ECAP can be estimated to be about 0.83 s^{−1} using Eq. (14) in Ref. [13]. Samples annealed under the same conditions were rolled at room temperature to thickness reductions of 60% and 84% in steps of 10% reduction per pass. These reductions

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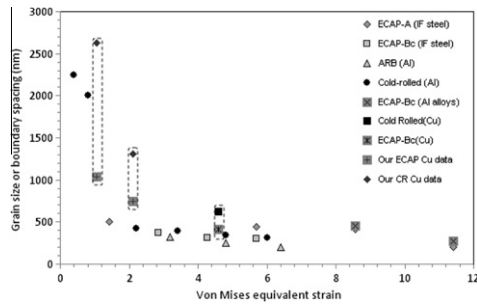


Figure 1. Comparison of the average grain size/boundary spacing as a function of the equivalent strain obtained from ECAP and rolling (Al [4,5], Al alloy [6,7], Cu [8,9] and IF steel [10,11], as well as for ARB in Al [4]). The dotted boxes highlight the comparison of grain sizes in rolling and ECAP of Cu at three equivalent strains.

correspond to equivalent strains of ~ 1.06 and 2.12 , which are the same strains as in the one- and two-pass ECAP samples. The average strain rate during rolling was about 0.4 s^{-1} . Samples with the same geometry as for ECAP were rolled so that the initial rod axis direction coincided with the rolling direction. It was not necessary to specify the orientations of the transverse direction (TD) and normal direction (ND) as the initial texture was very weak and had a rotational symmetry around the longitudinal axis anyway. The deformation state of a material element during ECAP is such that there is elongation in the ED direction and compression in the ND direction [13], exactly like in rolling. The difference between the two deformation modes is only the presence of a large rigid body rotation during ECAP (for more information about the deformation mode in ECAP, see Refs. [13,14]). However, the geometries of the two deformation processes with respect to a bar specimen are such that the initial textures in the ECAP and rolling processes are different with respect to the

deformation. The fact that the ECAP sample has to be pressed along the axis of the rod means that the texture is 90° rotated with respect to the rolling test, where it is compressed along a radial direction. Nevertheless, this difference should be only important for strong initial textures. In the present case, the texture intensity varied between 0.8 and 1.2 (Fig. 2b), which can be considered as a nearly random texture. Therefore, it is expected that the effect of the initial texture is negligible and the differences in the obtained microstructures mainly come from the differences in the applied strain paths.

Specimens cut from the center of the ECAP billet along the ND–ED plane were used for microstructural characterization. Specimens were cut from the mid-section of the rolled samples in the ND–RD plane, where RD is the direction of rolling. The samples were mechanically polished to 4000 grit by using SiC paper and then electropolished for 20 s in an electrolyte of 25% orthophosphoric acid, 25% ethanol and 50% distilled water at 10 V, 20°C , with a current of $\sim 150 \text{ mA}$. The EBSD measurements were performed using a JEOL 7001F FEG SEM fitted with an HKL detector with a step size of $0.2 \mu\text{m}$. Boundaries were identified using a minimum misorientation angle of 5° between adjacent pixels. In order to obtain representative data, at least three maps $40 \mu\text{m} \times 80 \mu\text{m}$ in size were measured for each condition of ECAP-ed and rolled copper. Post-analysis of the orientation maps was performed using functions written in Matlab.

The microstructures of the ECAP deformed copper were elongated (Fig. 2b and d), whereas the rolled samples contained a band-like lamellar structure with the long axis parallel to the rolling direction (Fig. 2c and e). The average grain size was $1.04 \pm 0.17 \mu\text{m}$ for the one-pass ECAP-ed and $2.63 \pm 0.29 \mu\text{m}$ for the 60% rolled copper. The average values further decreased to

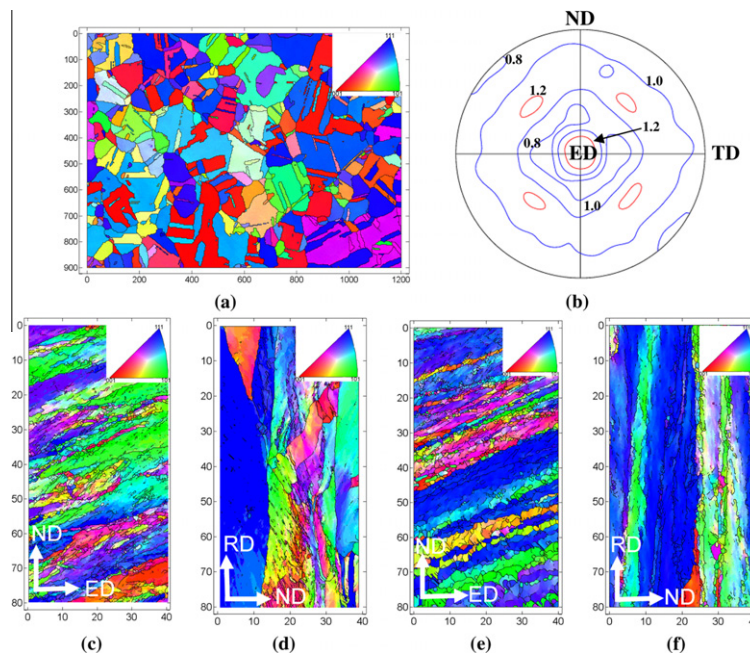


Figure 2. Representative inverse pole figures of the microstructure of (a) as-received OFHC copper; (b) $\{1\ 1\ 1\}$ pole figure of the as-received OFHC copper (isovalues: $0.8\times$, $1.0\times$ and $1.2\times$ random); microstructure of (c) ECAP one pass; (d) rolling to a thickness reduction of 60%; (e) ECAP two passes; and (f) rolling to 84%. Grain boundaries with misorientation larger than 5° are depicted by black lines. The scale is in micrometers.

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