



# Grain size dependent texture evolution in severely rolled pure copper



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## ABSTRACT

Large strain rolling – up to 97% thickness reduction – was carried out on pure copper with two different initial grain sizes: coarse-grained (CG, 24  $\mu\text{m}$ ) and ultrafine-grained (UFG, 360 nm). After rolling, classical copper-type rolling texture was obtained for the CG copper while a brass-type rolling texture was observed in the starting UFG copper, obtained by eight-pass equal channel angular pressing at room temperature prior to rolling. Using the Viscoplastic Self Consistent polycrystal model, it is found that the deformation mechanism at large rolling strain is grain size dependent and a change in the major slip mode has a large impact on the type of deformation texture. The brass-type texture in the UFG material can be simulated using  $\{111\} \langle 11\bar{2} \rangle$  partial slip together with  $\{111\} \langle 1\bar{1}0 \rangle$  slip and a small amount of twinning; for the CG material, only  $\{111\} \langle 1\bar{1}0 \rangle$  slip is needed to reproduce the copper-type rolling texture. Further, it is found that the polycrystal deformation condition approaches Taylor behavior in the UFG material whereas much more strain heterogeneity is present in rolling of the CG copper. By orientation imaging electron microscopy the geometrically necessary dislocation (GND) density is found to be decreasing in the UFG rolled samples while increasing in the CG case. This tendency corroborates the texture simulation parameters concerning strain heterogeneity.

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## 1. Introduction

Crystallographic texture is an important microstructure property which can influence the physical/mechanical and chemical behavior of a material [1]. It can affect mechanical properties such as yield stress [2], strain hardening [3], ductility [4], fatigue [5] and be responsible for deviations from isotropic behavior (anisotropy) [6] while physical properties such as electrical conductivity [7], magnetism [8] and shape memory effects [9] are also affected. Furthermore, texture plays an important role in chemical properties including phase transformations [10] and corrosion [11]. Another important microstructure parameter is grain size which can also influence the material properties and consequently a large number of studies have examined the simultaneous effect of grain size and texture on material properties [12–19]. There were, however, few attempts focused on the relation between grain size and texture [20–24]. This is important, as when two microstructure parameters affect a property, they will have an interdependent influence on the resultant material behavior.

Firstly, the texture variations as a function of grain size can be significant in hexagonal materials due to the ease of deformation twinning for

large grain sizes [20]. For cubic crystal structures, the situation is different: The rolling textures in low carbon steel were studied in [21] in the grain size range of 13–94  $\mu\text{m}$  and little effect of grain size was found. Iron was examined by Inagaki in 70% rolling [22] and a more fiber-like texture was found in the lower grain size samples; the grain size ranged from 40 to 400  $\mu\text{m}$ . Two initial grain sizes (115 and 460  $\mu\text{m}$ ) were achieved by different hot-band annealing in of a Fe–2%Si steel in [23] and significantly different textures were observed in subsequent rolling. The texture differences, however, could be attributed to the different initial hot-band textures, not to the grain size difference. Bhattacharjee et al. [24] deformed Ni by 90% direct- as well as cross-rolling for two initial grain sizes: 36 and 800  $\mu\text{m}$ . Copper-type textures were found for both grain sizes in direct rolling, however, a nearly brass-type texture was obtained in cross-rolling of the initially finer grain size material. The differences were a result of the different fragmentation processes that were assumed for the fine and large grain sizes and by the applied deformation routes.

To obtain ultrafine grain (UFG) sizes, the severe plastic deformation (SPD) technique [25] is a suitable process where continuous dynamic recrystallization takes place even at room temperature [26]. A large reduction in the grain size increases the grain boundary surface area significantly and, thus, deformation processes related to the grain boundary become relevant. One such process is the creation of stacking faults at the grain boundary, a process which was found recently by atomistic-phase field dislocation dynamic simulations [27,28]. Such

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stacking faults can rapidly cross the grain when the grain size is sufficiently small and, thus, become an efficient slip deformation mechanism. Indeed, we recently found [29] that  $1/6 \langle 11\bar{2} \rangle$  type partial dislocations are responsible for the formation of a brass-type texture in pure copper if the starting material is in UFG state.

As the effect of grain size on texture evolution was studied for rolling deformation in these cited studies, it is relevant to describe briefly the typical rolling textures. In the FCC case there are two basic textures; copper-type and brass-type [30]. Copper-type textures are characterized by Copper  $\{112\}\langle 11\bar{1} \rangle$  and S  $\{124\}\langle 21\bar{1} \rangle$  components while in brass-type textures the main ideal orientations are Brass  $\{011\}\langle 21\bar{1} \rangle$  and Goss  $\{011\}\langle 100 \rangle$  (Fig. 1). Often, low stacking fault energy (SFE) materials present brass-type textures due to their ability for deformation twinning whereas the copper-type textures are seen in rolling of high SFE metals. However, the exception is when the initial grain size is already in the UFG regime so that a strong brass-type texture was observed in pure copper [29].

The possibility of partial-dislocation mediated slip in the formation of deformation textures exists in UFG and nanocrystalline materials. Skrotzki et al. [31] considered partial slip as a major mechanism and explained successfully the texture evolution in a nano-polycrystalline Pd–10%Au alloy during high pressure torsion. They also pointed out that the deformation conditions in nano-polycrystalline materials are near to the Taylor model. The simulation work in [29] on rolling of UFG copper has shown that the brass-type texture can be the result of strong activity of the  $1/6 \langle 11\bar{2} \rangle$  type partial dislocations. Further, and similar to the report from Skrotzki et al. [31], a Taylor-type of polycrystal deformation condition was validated. Here, we aim to make a clear experimental/modeling comparison between the UFG and CG state in large strain rolling of pure copper focusing on the texture development. Texture measurements on the UFG and CG states were complemented with detailed electron back scatter diffraction (EBSD) studies for detecting variations in the geometrically necessary dislocation (GND) densities. The GNDs indicate the extent of plastic strain heterogeneity and the experimental observations corroborate the simulation results, elucidating the reasons for the grain size dependence on the evolution of texture in rolling of pure copper.

## 2. Experimental

Oxygen-free high conductivity (OFHC) copper was homogenized at 650 °C for 2 h under vacuum, resulting in an average grain size of  $\sim 24 \mu\text{m}$  (CG state). The billet of the CG copper was machined to samples with 20 mm  $\times$  20 mm cross section and 120 mm in length. At room

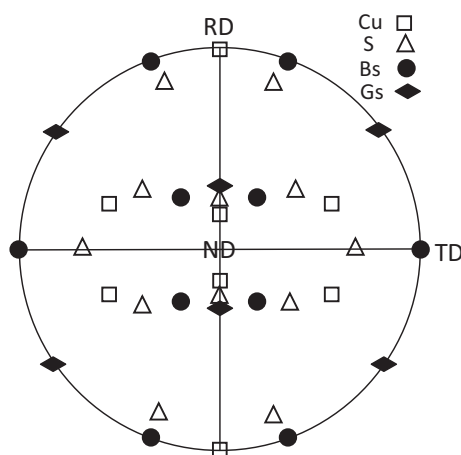


Fig. 1. Key  $\{111\}$  pole figure for the main ideal orientations of the copper texture (open symbols) and for the brass texture in rolling (filled symbols) [29].

temperature, the billet was processed by equal channel angular pressing (ECAP) using route B<sub>c</sub> and a 90° die for eight passes, resulting in an average grain size of  $\sim 360 \text{ nm}$  (UFG state). A back pressure of 25 MPa, a forward speed of 2 mm/s and colloidal graphite as lubricant were used during ECAP. The obtained UFG copper was cold rolled up to a true strain of 3.9 (thickness reduction of 97%) in a 120 mm diameter rolling mill. The rolling direction (RD) was parallel to the extrusion direction (ED) and the rolling plane was TD–ED. The CG copper was also rolled to the same strain. Samples were stored in a conventional deep freezer for later testing.

The textures of the rolled UFG and CG copper were characterized by X-ray diffraction. For bulk texture determination, the normal rolling surface of the samples (RD–TD section) was ground and mechanically polished. Partial pole figures were obtained on the  $\{111\}$ ,  $\{200\}$  and  $\{220\}$  planes up to a maximum tilt of 80° with 5° steps in a mini-materials analyzer (MMA) goniometer equipped with Cu anode and a polycapillary beam enhancer. The orientation distribution functions (ODFs) were calculated using commercial analytical software developed by ResMat Corp. The volume fractions of texture components (Copper, S, Goss and Brass) were calculated with an angle tolerance of 15° with respect to the Euler angles of the ideal texture component. The textures are presented in complete pole figures in this work which were plotted using the JTEX software [32].

The microstructures of the UFG and CG rolled copper were examined by electron backscatter diffraction (EBSD). Prior to EBSD, the longitudinal sections (RD–ND) of the samples were mechanically ground and polished to 4000 grit using SiC paper, and then electro-polished in a chemical solution of 25% orthophosphoric acid, 25% ethanol and 50% distilled water at 10 V for 30 s at room temperature. The EBSD characterization was undertaken in a Leo-1530 field emission gun scanning electron microscope with an operating voltage of 20 kV and a probe current of about 5 mA and a working distance of 20 mm. Due to the fine structure of copper before and after rolling, a step size of 40 nm was chosen and the scanning area was  $\sim 10 \mu\text{m}$  by  $\sim 20 \mu\text{m}$ . The grain boundaries were identified using a minimum disorientation angle of 5° between adjacent pixels. Prior to data analysis, a clean-up procedure was employed in the deformed samples in which the orientation of a mis-indexed point was replaced with one of its neighbors. Transmission electron microscopy (TEM) observations were carried out using a Philips CM 200 TEM.

## 3. Results

The initial CG texture had a weak cube texture component and another weak component parallel to the  $\{111\}$  axis. After eight-pass ECAP in route B<sub>c</sub> the texture changed significantly (Fig. 2) and can be characterized by a simple shear texture, if expressed parallel to the shear plane [33]. The texture of the UFG rolled copper differs from the CG rolled one (Fig. 3a) by a strong Bs component (Fig. 3b) whereas the texture during CG rolling developed Cu, S, Bs and a weak Goss (Fig. 3a) components. After a strain of 3.9 the volume fractions of the major texture components of UFG rolled copper were: 11.2% Cu, 39% S and 34% Bs, and for CG rolled copper 17% Cu, 41% S and 17% Bs.

The initial grain structure consisted of close to randomly oriented grains with practically random disorientations – apart from a peak at a disorientation angle of 60° originating from annealing twins (Fig. 4a). All microstructure features were examined in the TD plane. Before rolling, the ECAP deformed sample showed a band-like lamellar structure, with a long axis parallel to the macro-shear direction imposed by the ECAP process (Fig. 4c). After high strain rolling the deformation structure of both the UFG and CG rolled samples was dominated by a lamellar structure with elongated subgrains (Fig. 4b and c). Some relatively large elongated or equi-axed grains can be found in the CG rolled deformation structure.

Quantitative examination of the grain-to-grain disorientation distribution showed that the ECAP deformed copper skewed both low angle

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