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# Unexpected brass-type texture in rolling of ultrafine-grained copper

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Performing large-strain rolling of initially ultrafine-grained copper obtained by eight passes in equal-channel angular pressing, a brass-type –rather than a copper-type – texture was achieved. Clearly, the deformation mechanism is different in ultrafine-grained materials for subsequent roll-ing. Using the viscoplastic self-consistent polycrystal model, we find that the deformation mechanism is associated with the activation of partial  $\{111\}$   $\langle 11-2 \rangle$  slip and with a reduced quantity of geometrically necessary dislocations that lead to near Taylor polycrystal deformation conditions in ultrafine-grained copper.

**Keywords:** Brass-type texture; Ultrafine-grained; Copper; Equal-channel angular pressing/equal-channel angular extrusion; Rolling

It is well known that a copper-type texture develops during rolling of medium to high stacking fault energy (SFE) materials while a brass-type texture is obtained for low SFE materials. In a recent review [1], Leffers and Ray examine the differences between copper- and brass-type textures, and conclude: (i) the copper-type texture can be explained by a homogeneous deformation of the polycrystal, that is, by the Taylor model; and (ii) the brass-type texture originates from deformation twinning with a low volume fraction of twins but with a lamellae twin structure which forces the grain to deform by coplanar slip as an effect of latent hardening. Deformation banding is frequently observed in rolling at large strains when the material develops a brass-type texture; the deformation can localize in microshear bands that include several grains. El-Danaf et al. [2] reported that the brass-type texture develops from the moment such shear bands appear (usually oriented at  $\sim 25^\circ$  with respect to the rolling plane), so this type of texture is not related to deformation twins.

In seeking to elucidate the origin of the brass-type texture, the effect of grain subdivision on the evolution of the texture has not yet been considered [3]. Large plastic strain during severe plastic deformation induces dynamic

recrystallization by subdividing the grains via a continuous dynamic recrystallization process, even at low temperatures [4]. Consequently, the large reduction in the grain size increases the grain boundary surface area tremendously, and deformation processes that are related to the grain boundary become relevant. One example is the experimental observation of brass type texture in Cu–Ag 200 nm layered nanocomposite [5,6]; the same kind of brass-type texture was obtained in the copper phase as in the silver component. A recent study by Skrotzki et al. [7] examined the effect of  $\langle 112 \rangle$  slip on  $\{111\}$  planes on the texture evolution in a nanocrystalline Pd–Au alloy during high-pressure torsion. Their work showed by simulation that the deformation conditions are close to the Taylor model [7]. The present work aims to investigate the origin of the brass-type texture found in rolling of pure copper in which the initial grain size was already in the ultrafine grain (UFG) regime.

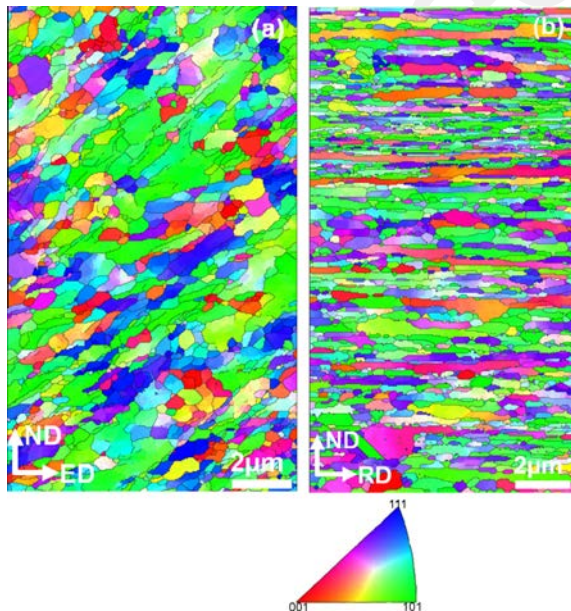
Oxygen-free high-conductivity copper was heat treated at  $650^\circ\text{C}$  for 2 h in a vacuum, resulting in an average grain size of approximately  $24\text{ }\mu\text{m}$ . Bar samples with dimensions of  $20 \times 20 \times 120\text{ mm}^3$  were machined from the annealed billet and processed by equal-channel angular extrusion (ECAE) using route  $B_C$  with a  $90^\circ$  die for eight passes, a back pressure of 25 MPa and a forward speed of  $2\text{ mm s}^{-1}$ , and using colloidal graphite as lubricant.

The obtained UFG material pieces with different starting thicknesses were cold rolled up to a true strain of 3.9 (thickness reduction of 97%) in a rolling mill with roll

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diameter of 120 mm, resulting in a final thickness of  $\sim 0.3$  mm. The rolling direction (RD) was parallel to the extrusion direction (ED) and the rolling plane was TD (thickness direction)–ED. The samples were stored in a conventional deep freezer until further testing. The microstructure of the UFG rolled copper was characterized by electron backscatter diffraction (EBSD). Prior to EBSD, longitudinal sections (RD–ND) of the samples were mechanically ground and polished to 4000 grit using SiC paper, then electropolished in a chemical solution of 25% orthophosphoric acid, 25% ethanol and 50% distilled water at 10 V for 30 s at room temperature. EBSD 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, with a working distance of 20 mm. A step size of 30 nm was chosen and the scanned area was  $\sim 10 \times 20 \mu\text{m}^2$ . The grain boundaries were identified using a minimum disorientation angle of  $5^\circ$  between adjacent pixels. For bulk texture determination of the rolled copper by X-ray diffraction, the normal rolling surface (RD–TD) of the samples was ground and mechanically polished. Partial pole figures were obtained from the  $\{111\}$ ,  $\{200\}$  and  $\{220\}$  planes up to a maximum tilt of  $80^\circ$  at  $5^\circ$  intervals in an MMA goniometer equipped with a Cu anode and a polycapillary beam enhancer. The volume fraction of texture components (copper, S and brass) were calculated with an angle tolerance of  $15^\circ$  with respect to the Euler angles of the ideal texture component. For transmission electron microscopy (TEM), a Philips CM 200 TEM was used to visualize twins in the deformed microstructure. Diffraction patterns were collected directly from the matrix and the twinned crystals, using convergent beam (microdiffraction) to verify the twin relationships.

The microstructure was examined in the TD plane. Before rolling, the ECAE deformed sample showed a



**Figure 1.** Inverse pole figure maps of (a) UFG copper obtained by 8-pass ECAE (left); (b) subsequent rolling to a true strain of 3.9 (right). Grain boundaries with disorientations larger than  $5^\circ$  are depicted by black lines. The unit triangle of the stereographic projection displays the color code for the direction of the plane normal of the map in the crystal axes.

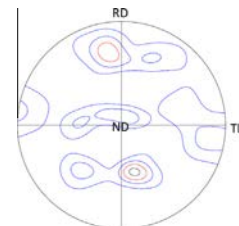
band-like lamellar structure, with long axis parallel to the macroshear direction imposed by the ECAE process (Fig. 1a). After high-strain rolling, the deformation structure was dominated by a lamellar structure with elongated subgrains (Fig. 1b). The average number-fraction grain size of the ECAE-deformed copper was  $0.36 \pm 0.2 \mu\text{m}$  which decreased slightly to  $0.32 \pm 0.2 \mu\text{m}$  after rolling. The grain size distribution varied very little with subsequent rolling strain, which implied no significant further grain subdivision of the UFG copper during rolling. There was, however, significant change in the disorientation angle distribution, where the proportion of the initially 65% high grain boundary angles (defined by a minimum angle of  $15^\circ$ ) increased up to 87%.

The initial texture of the coarse-grained copper had a weak cube texture component and another weak component  $\{111\}$  parallel to the ND axis (see the texture in Ref. [8]). After eight passes of ECAE the texture had changed significantly, and could be characterized by a simple shear texture (Fig. 2). Figure 3b and c shows the texture of the initially UFG copper rolled to a true strain of 3.9 obtained in the experiment and in the simulation, respectively. The volume fractions of the major texture components in the experiment were: 11.2% Cu, 39% S and 34% Bs, which is a typical proportion for a brass-type texture [1]. Figure 3a shows the key  $\{111\}$  pole figure for the main ideal orientations of the copper and brass-type textures.

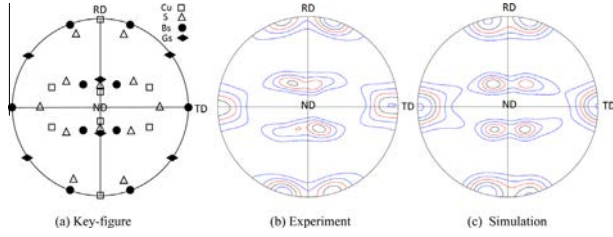
Turning now to the simulation, the viscoplastic self-consistent (VPSC) model of Molinari et al. [9] was employed in its version described in Ref. [10], which can account for grain shape effects. However, due to the grain subdivision process, the grain shape does not follow the geometrically expected elongated shape at large strain. This is why the shape evolution was not considered in the modeling. It was demonstrated by recent polycrystal grain refinement modeling that the effect of grain subdivision on the evolution of the crystallographic texture is relatively small [11], so it is reasonable to assume that the VPSC model can give acceptable texture predictions without considering grain refinement. Deformation twinning was also considered using a recently established volume transfer scheme [12].

The initial texture was represented by 200 grains in the simulations, each complemented with 12 possible twin grains initially with zero volumes. In total, 2600 grains represented the polycrystal. Hardening was not modeled because of the large strains where strain hardening is exhausted. The value of the strain rate sensitivity of slip was taken to be 0.125. The simulation was undertaken in small incremental strain steps (0.025) up to the final strain of 3.9, corresponding to 97% rolling reduction.

In line with previous simulations [7,13], the 12  $\{11-1\}$   $\langle 112 \rangle$  twinning systems were also considered as possible



**Figure 2.**  $\{111\}$  pole figure of UFG copper after 8-pass ECAE in route Bc measured on the ND (pressing) plane. Isolines: 1.0, 1.9, 2.8, 3.7.

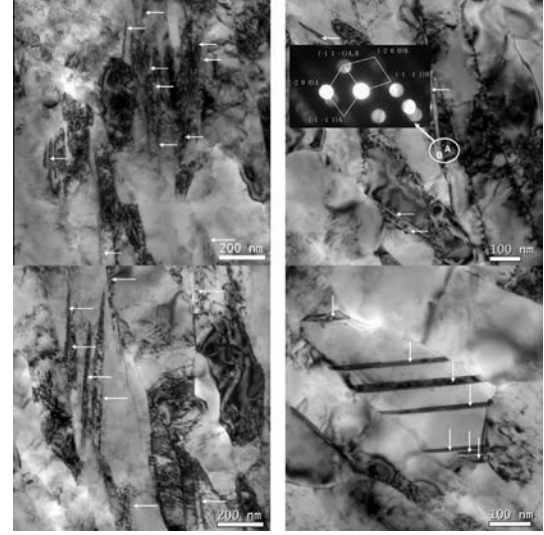


**Figure 3.** (a) Key  $\{111\}$  pole figure for the main ideal orientations of the copper texture (open symbols) and for the brass-type texture in rolling (filled symbols). Experimental (b) and simulated (c) pole figures after a true strain of 3.9 rolling of UFG Cu. Isolevels: 1.0, 1.9, 2.8, 3.7, 4.6.

slip systems which were added to the 12  $\{-111\} \langle 110 \rangle$  slip systems of the face-centered cubic crystal structure. Slip on the  $\{11-1\} \langle 112 \rangle$  systems takes place by stacking faults; the slip of a  $\langle 112 \rangle$  partial dislocation achieves a small strain on the grain with a displacement which is  $1/6$  of a full  $\langle 112 \rangle$  dislocation. As shown in Refs. [14,15], partial dislocations can cross a UFG-sized copper grain completely, thus producing plastic deformation. Consequently, they can be considered as a possible slip mechanism. Of course, when at least two such partial dislocations move on adjacent planes, a twin can be formed. The twinned part of the grain is a new orientation and can be handled with our twinning volume transfer scheme. However, it is assumed in this work that only a fraction of the partial dislocations contribute to the formation of a twin. Therefore, we introduced a parameter in the simulation – the twin forming fraction of partial dislocations ( $\xi$ ) – to account for this. This process was programmed so that only a  $\xi$  fraction of  $\langle 112 \rangle$  slip was converted into twins.  $\xi$  was varied in the simulations between 0 and 1 to ascertain the effect of twin fraction on the evolution of the texture.

From the result obtained after simulating ECAE plus rolling to a true strain of 3.9 (Fig. 3c), the simulated texture is in close agreement with the measured one (Fig. 3b). The brass-type texture is reproduced well, with strong Bs and S components. Even the asymmetry originating from the initial ECAE texture is reproduced. Regarding the simulation parameters, the most important one is the scalar  $\alpha$  coefficient in the interaction equation between a given grain and the homogeneous equivalent medium composed by the whole polycrystal (see Ref. [10] for the modeling approach). The texture closest to the experiment was obtained when the  $\alpha$  parameter was set to 10, which is a near-Taylor condition. The same reference strength was set to both the  $\langle 110 \rangle$  and  $\langle 112 \rangle$  slips as well as to twinning. The relative activity of the  $\langle 110 \rangle$  slip with respect to  $\langle 112 \rangle$  was 0.55/0.45. Concerning the fraction of  $\langle 112 \rangle$  slip that could build twins,  $\xi$  was set to 0.1, that is, 10% of  $\langle 112 \rangle$  slip was assumed to make twins. This set of parameters led to a 4.75% volume fraction of twins, which is more than the fraction of twins visible in the TEM micrographs (Fig. 4). Note, however, that Figure 4 shows the twins at a true strain of 0.8 in rolling after ECAE; at this stage significantly fewer twins are present, at about 2% fraction only.

In the simulation work, the choice of the value of the adjustable parameters is a critical issue. In this work, they were: the  $\alpha$  value in the interaction equation of the self-consistent model; the relative strength value between  $\langle 110 \rangle$  and  $\langle 112 \rangle$  slip; and the value of the  $\xi$  parameter for twin



**Figure 4.** Deformation twins (identified by arrows) in copper following rolling of initially UFG copper to a true strain of 0.8 at room temperature (after eight ECAE passes) viewed in a TEM foil from a longitudinal section. The inset in the top-right micrograph shows the diffraction pattern taken from the region indicated by A–B, where A is matrix and B is twin.

volume formation. These parameters were varied in the simulation until a good reproduction of the experimental texture was obtained. The  $\alpha$  parameter is discussed below. Concerning the critical strength of the two slip system families, the only way was to adjust its value to the texture. The experimental volume fraction of twins was very helpful in finding the appropriate value of  $\xi$ . Nevertheless, it was found that the texture evolution was essentially insensitive to the value of  $\xi$  up to an unreasonably high value, which led to 40% of the twinned volume fraction.

In addition to the good agreement of the simulated and experimental textures, we can further support the modelling conditions as follows. Recent observations in the steady-state UFG regime of copper show that the geometrically necessary dislocation density (GND) moves to five orders of magnitudes lower than the statistical dislocation density [4], while it is about the same order at an early stage of the grain fragmentation process [16]. GNDs are developed to account for deformation heterogeneities between adjacent grains, i.e. near to the grain boundaries. Their absence is clear evidence for a strong decrease in deformation heterogeneity; consequently, the deformation is more homogeneous, approaching the Taylor deformation mode. This mode requires a high value in the interaction equation of the VPSC polycrystal model, justifying the value of  $\alpha = 10$ . This is one striking novelty of the present study; while it was commonly believed that the brass-type texture originates from a Sachs polycrystal deformation mode, the present results suggest that it is the uniform Taylor deformation mode which corresponds to the brass-type texture (in UFG materials). The Sachs model concerns a highly heterogeneous deformation mode which requires the existence of a large amount of GNDs, which are not observed experimentally at very high strains [4].

It is also suggested in the present study that  $\{11-1\} \langle 112 \rangle$  slip is active in the very small grain size regime, which is the other decisive element of the formation of a

brass-type texture in UFG Cu. The activity of partial slip in nano- and UFG crystals is documented by experiments in 2004 [15]. However, it has only recently been considered in crystal plasticity simulations [7,12,17–19].  $\langle 112 \rangle$  slip takes place by partial dislocations, some of which produce deformation twins. It is difficult to estimate the exact proportion of partial slip that forms twins because nearly the same brass-type texture is obtained by simulation with low (4.75%) or high (36%) twin volume fractions. Nevertheless, TEM shows that the twin fraction is no more than 2% at a strain of 0.8 after ECAP (Fig. 4). Other simulations carried out for simple shear in nanocrystalline Pd-Cu also used the  $\{11-1\}$   $\langle 112 \rangle$  slip and successfully reproduced the experimental brass-type shear texture in high-pressure torsion [6]. A brass-type texture was also obtained in a Cu-Ag nanocomposite material showing small deformation twins in the Cu phase [5]; simulation was also carried out using  $\{11-1\}$   $\langle 112 \rangle$  slip, and a reasonably good brass-type texture was obtained when  $\langle 110 \rangle$  and  $\langle 112 \rangle$  slip were present in the same proportion.

In summary, we find a strong brass-type texture after a rolling strain of 3.9 in UFG Cu which is quite different from the usually observed copper-type texture that is known to develop in coarse-grained copper after similar rolling reductions. Our TEM observations revealed that there are deformation twins (volume not more than 2%) in UFG copper rolled to a true strain of 0.8. Simulations reveal that brass-type texture development in pure copper at very large strains is likely to result from activation of  $\{11-1\}$   $\langle 112 \rangle$  slip, which is in addition to the usual  $\{1-11\}$   $\langle 110 \rangle$  slip. The  $\langle 112 \rangle$  slip is only significant in the UFG regime and only a small part (about 10%) of the partial dislocations form deformation twins. Further, the simulation results show that brass-type rolling textures can be obtained by nearly homogeneous deformation conditions (near-Taylor model), indicating that geometrically necessary dislocations are relatively small in proportion when the grain size becomes extremely small.

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