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2015 IOP Conf. Ser.: Mater. Sci. Eng. 82 012016
(http://iopscience.iop.org/1757-899X/82/1/012016)

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Texture comparison between cold rolled and cryogenically rolled pure copper

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Abstract. Nowadays, there is a considerable scientific interest in bulk ultrafine grained materials, due to their potential for superior mechanical properties. One of the possible formation methods of nano-grained materials is cryogenic rolling. The influence of rolling at cryogenic temperatures has been investigated. Significant differences in the textures and the microstructures can be observed between the cryogenically rolled copper and conventionally cold rolled copper, reduced to the same thickness.

1. Introduction
Nowadays, there is a considerable interest in the deformation of metals at very low absolute temperatures, which is known as cryogenic deformation [1]. It is assumed that rolling at cryogenic temperature can result in enhanced grain refinement and associated strengthening when compared to rolling at room temperature (RT) [2]. It is believed that very low temperatures suppress the processes of dynamic recovery, promote an increase in the dislocation density, activate mechanical twinning [3, 4] and thereby promote the formation of extremely fine-grained structures. Another potential of cryogenic rolling is the formation of a very weak, almost random texture after annealing in Cu and Al alloys [5, 6]. There is evidence that this behavior can be linked with the occurrence of shear bands in the deformation texture.

In the present work the aim is to get a better understanding of the texture development in the cryogenic rolling process of pure copper. Therefore, the texture and microstructures of cold rolled and cryogenic rolled material, starting from an identical batch of pure copper, is studied in detail.

2. Experimental
The investigated material in this study is high purity cast copper. A hot rolling reduction (400°C) of 50% allows destroying the cast microstructure. After an annealing step of 30 min at 400°C a completely recrystallized material is obtained, called the initial material. Part of the initial material is cryogenic rolled to 50%, 75% and 90% overall thickness reduction. The other part is rolled at RT to the same thickness reductions.

Electron backscatter diffraction (EBSD) observations on both the cryogenically rolled and the cold rolled copper are made on the longitudinal plane containing the rolling and the normal directions. The textural observations are made on the mid-thickness section of the rolled samples. For the deformed materials, textures are calculated without imposed orthorhombic symmetry.

3. Results
3.1 Initial material
As the texture of the initial material can influence the texture of the deformed material [7], a detailed texture analysis of the material prior to rolling is done at the mid-thickness section. The texture (Fig 1) has an almost random distribution with the strongest peak being 2.4 x random.
3.2 Deformed state after 50% deformation
After 50% cold deformation, the texture in the mid-thickness (Fig 2a) remains weak (no peaks higher than 4 x random) and the observed texture is obviously different from the conventional β-fibre rolling texture. The ND-rotated cube component is present. After 50% cryogenic reduction (Fig 3b), also a weak texture is observed in the mid-thickness of the sample, with the same intensity as in the cold rolled copper. The same texture components are observed as in the 50% cold rolled material, namely the ND-rotated cube component. Both textures have a high degree of symmetry.

3.3 Deformed state after 75% deformation
In the material cold rolled to 75% reduction, the overall texture intensity is strengthened and both α- and β-fibers become evident in the orientation distribution function (ODF) (Fig 3a). Within the α-fiber, Goss and Brass components are noticed, whereas the copper and S components are observed within the β-fiber. The brass component is prevalent over the other components, with a maximum intensity of 13.8 x random. A high degree of symmetry is observed. After 75% cryogenic rolling (Fig 3b), the texture looks similar to the cold rolled material rolled to the same thickness reduction. However, some differences are observed. After cryogenic rolling the texture looks less symmetric. Also a weaker texture is observed, compared to the cold rolled copper, with a maximum intensity of 10.6 x random.
Figure 4 shows the image quality (IQ) maps of the cold rolled (a) and cryogenically rolled (b) copper after 75% deformation. Although the deformation texture of these two materials is similar, a large difference is observed in their microstructures. In the cryogenic rolled material, in contrast to the cold rolled material, numerous shear bands are observed. These bands form thin planar sheets that are parallel to the transverse direction and inclined at an angle of ~35° to the rolling plane. A secondary minor set of shear bands are observed inclined at an angle of ~20° to the rolling plane.

**Figure 4: Image Quality (IQ) map of the mid-thickness after 75% deformation (a) cold rolled (b) cryogenic rolled.**

### 3.3 Deformed state after 90% deformation

After 90% cold reduction the texture becomes much sharper (Fig 5a), with the strongest peak being 19 x random. The homogeneity along the α-fiber further degenerates and develops into the single brass component, although the β-fiber remains present. Another remarkable observation is the strengthening of the cube component compared to the 75% cold rolled sample.

**Figure 5: ODF sections (with φ_2 = 0°, 45° and 65°) of the mid-thickness section after 90% deformation (a) cold rolled, (b) cryogenic rolled.**

As almost half of the area of the 90% cryogenic rolled copper was already recrystallized, probably due to RT annealing [8], partitioning based on the GOS was needed to determine the deformation texture. The texture of the deformed grains after 90% cryogenic rolling is represented in figure 5b. The same texture component are observed as in the cold rolled copper, however, the texture evolves comparatively slowly. No weakening of the α-fiber is observed.

### 4. Discussion

Since the initial material is for both thermo-mechanical treatments identical and the texture of this initial copper is completely random (Fig. 1), a thorough comparison can be conducted.
After 50% cold and cryogenic reduction (Fig 2a and 2b), no large difference can be observed between the two textures. Both textures are fairly weak, nearly symmetric and none of the typical fcc rolling fibers are already present. The observed ND-rotated cube orientation is a typical shear texture component which may be the result of the roll gap geometry in this experiment.

Comparing the 75% cold and cryogenically rolled (Fig. 3) and the 90% cold and cryogenically rolled copper (Fig. 5), some difference can be perceived. The most notable difference is the slower texture evolution of the cryogenically rolled copper compared to the cold rolled one. It appears that after the cryogenic deformation the texture resembles an alloy type of texture, in contrast to the pure metal type of texture formed after cold rolling of the pure copper. A possible explanation for the alloy type of texture is the occurrence of deformation twinning [9], which is observed experimentally. Another explanation is that the development of a weaker texture after cryogenic rolling is due to the presence of shear bands. It is reasonable to assume that the shear bands retard the development of the typical rolling textures in the copper matrix.

![Figure 6: Cube volume fraction of the cold rolled and cryogenically rolled pure copper as a function of the applied reductions.](image)

Another remarkable observation is done in the texture of the cold rolled pure copper, and to lesser extent in the cryogenically deformed. Although the initial material has an almost fully random texture, after higher deformations, the cube component is appearing, albeit in minute strength. This indicates that the cube component is part of the deformation texture. Figure 6 shows the percentage of cube volume fraction of both cold rolled and cryogenic rolled pure copper as a function of the different reductions. This figure shows that cube is formed during the deformation process, where higher deformations stimulate a higher intensity of the cube component. The reason why this cube deformation component forms at higher reductions in the cryogenically rolled material than in the cold rolled material can be explained by the following suppositions. Since cube is very sensitive to shear and the higher friction in the cryogenically rolled material leads to more shear, no cube is formed at lower reductions. At higher reductions, the rolled material becomes thinner, so more heating up of the samples between every rolling pass is possible. In this way, the deformation texture will be more closely related to the deformation textures formed at RT.

5. Conclusion
The texture development in cryogenically rolled and cold rolled pure copper is compared after different rolling reductions, starting from the same randomly textured material. Whereas the cold rolled copper gives rise to a pure metal texture, the texture after cryogenic rolling resembles an alloy type of texture. The cube component is observed in both cold and cryogenic rolled copper as part of the deformation texture.

Acknowledgments
The authors wish to thank the Agency for Innovation by Science and Technology in Flanders (IWT) and the fund for Scientific Research in Flanders (FWO) for financial support (FWO10/PRJ/336). Aurubis (Belgium) is thanked for the high purity cast Cu.
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