

## Crystallographic texture and microtexture of copper drawn at 295 K and 77 K

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### ABSTRACT

The pure copper wires were wiredrawn at cryogenic and room temperatures. After deformation they presented important and significant differences in their crystallographic textures and microstructures. Therefore, this study aimed to investigate the evolution of crystallographic texture of pure copper drawn at temperatures of 295 K and 77 K and correlated them to the deformation micromechanisms. For this, the X ray diffraction (XRD) and electron backscattered diffraction (EBSD) techniques were used. The results showed that, after the deformation, the samples drawn at both temperatures showed Copper {112}<111>, X {110}<111> and Brass {110}<112> components as the most intense. In samples drawn at cryogenic temperature there was also observed the appearance of a R{124}<211> component, typical of recrystallization and the presence of the  $\gamma$  fiber {111}<uvw>, typical of deformation twin.

**Keywords:** copper drawn, 77K drawing, 295K drawing, microtexture, crystallographic texture.

### 1. INTRODUCTION

Copper is one of the most widely used CFC metals in the world, mainly due to its high electrical conductivity. However, for use in some advanced applications, such as wire for high power magnet coils, the copper has poor mechanical properties. This needs to be improved preserving its high electrical conductivity, which is a challenge. Almost all the mechanism that increases the mechanical strength decreases the electrical conductivity. In the light of this, the cryogenic deformation seems to be feasible alternative for this challenge, i.e., the strength increasing with the minimum damage in conductivity electrical [1,2]. It was demonstrated that the strength increase through work hardening has a low limit, since after some deformation the material presents a dynamic recovery and to stop increase its strength [3].

The idea of strengthening through cryogenic deformation is based in delaying that dynamic recovery [4]. Moreover, the deformation in low temperature promotes the twinning occurrence [5]. These mechanism changes can be accompanied by an interesting crystallographic texture modification.

When the crystallographic orientation distribution of the grains of a polycrystalline material is not random, there is a preferred orientation, which is also called crystallographic texture [6]. This preferred orientation can be introduced in the material by plastic deformation, recrystallization or phase transformation or a combination of them. The typical preferred orientation observed in wires is often referred as fiber textures. The texture of wire consist of orientations with a particular crystallographic direction, <hkl>, is parallel to the wire axis and other directions equally distributed around this axis, providing rotational or cylindrical symmetry to the polycrystalline structure [7].

Face-centered cubic metals and their alloys have fiber textures that are, particularly, sensitive to many variables. Textures usually observed are duplex fiber <111>||ND + <100>||ND, with the volume fraction of grains assigned to each of these directions varying greatly according to the material composition, as well as its previous deformation and heat treatment [8,9].

The stacking fault energy seems to be other important variable that controls the volume fractions of each the components (111)[uvm] and (100)[uvm], during the wire drawing of face-centered cubic (FCC) metals, after high reductions [10]. At low temperatures the deformation micro mechanisms exhibit differences in relation of the room temperature ones, for example, at cryogenic temperature the cross slip is restricted favoring the occurrence of twinning.

Whenever a FCC metal is subject to deformation by twinning, it is important to consider whether the twinning is significant in small reductions or only for high deformations, which is the actual case. Twinning can cause strong crystalline reorientations and leads to sharp changes in texture with deformation progress [11].

The purpose of this research was to investigate the evolution of crystallographic texture of pure copper wire drawn at room (295 K) and cryogenic temperatures (77 K). It, analyzed by electron backscattered diffraction (EBSD) and X-ray diffraction (XRD). The materials drawn at both temperatures showed a higher intensity the Copper {112}<111>, X {110}<111> and Brass {110}<112> texture components. Furthermore, the microtexture revealed the presence of the  $\gamma$  fiber {111}<uvw> and presence of recrystallization typical texture component, R {124}<211>.

## 2. MATERIALS AND METHODS

The material used in this study was pure copper - OFHC (C101, 99.99% Cu), processed by wire drawing in the National High Magnetic Field Laboratory [3] (NHMFL) facilities at 295 K and 77 K.

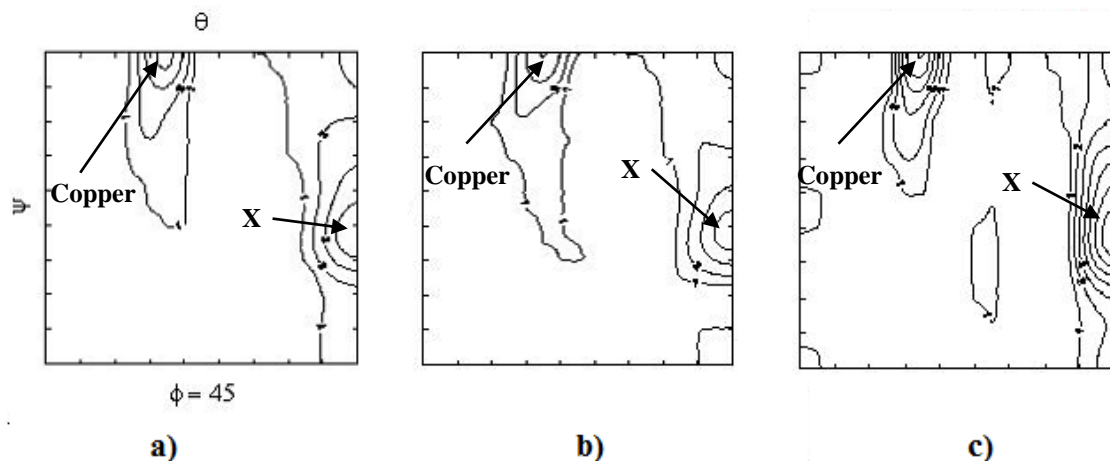
The material was received with initial diameter of 9.6 mm and was drawn to the final diameter of 2.02 mm, at 295 K and 77 K. The wires produced at 295K were drawn in a drawing bench, FENN model D51710 with electric drive, by the standard method. While those at 77 K were drawn using a cryostat attached to a servo-hydraulic tensile machine. The wire drawing process was conducted in 30 passes; the wire diameter was reduced after each pass for the die diameter. The samples were characterized for both temperatures after being reduced to 4.14 mm, 3.0 mm and 2.02 mm diameters.

For the analysis by electron backscattered diffraction (EBSD), the samples of the wire cross section were mounted in bakelite, ground, mechanically polished and then subjected to a vibratory polishing with colloidal silica (0.02  $\mu\text{m}$ ) using VIBROMET machine (Buehler). The measurements were performed by EBSD integrated with scanning electron microscope (SEM) JEOL, model 5800LV, operated at 25 KV.

The crystallographic texture analysis by X-ray diffraction (XRD) was performed in the NHMFL laboratory using the Philips X'Pert MRD diffractometer. The analyses were performed using the orientation distribution functions (ODF) generated by the popLA software and obtained from harmonic coefficients about the pole figures of the (111), (200) and (220) diffracted planes.

## 3. RESULTS AND DISCUSSION

Crystallographic texture analysis was performed on wires drawn at room and cryogenic temperatures using ODF (Roe notation), specially the  $\Phi=45^\circ$  section which contains the important crystallographic informations. The results for wire with diameters of 4.14 mm, 3.0 mm and 2.02 mm at room and cryogenic temperatures are shown in Figures 1 and 2, respectively.



**Figure 1:** ODFs of the drawn wires a) 4.14 mm, b) 3.0 mm and c) 2.02 mm at room temperature.

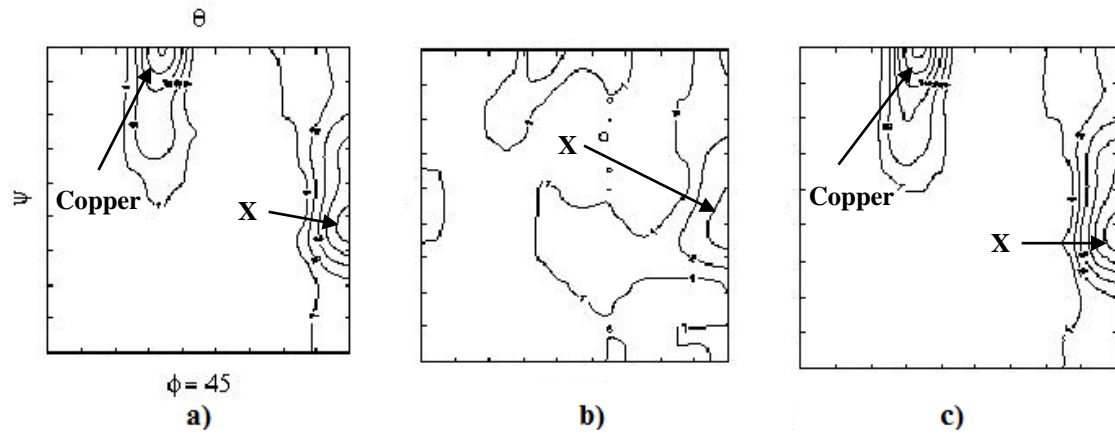


Figure 2: ODFs of the drawn wires a) 4.14 mm, b) 3.0 mm and c) 2.02 mm at cryogenic temperature.

One can observed in Figures 1 and 2 five mainly deformation texture components in the samples at both temperatures: Brass  $\{110\}\langle 112\rangle$ , Copper  $\{112\}\langle 111\rangle$ , S  $\{123\}\langle 634\rangle$  and X  $\{110\}\langle 111\rangle$  [11] and Goss component  $\{110\}\langle 001\rangle$ , which is defined by Humpreys and Hatherly [12] not only as deformation component but also as recrystallization component. In addition, the presence of some typical components of recrystallization are observed: P  $\{110\}\langle 122\rangle$  and R  $\{124\}\langle 211\rangle$ .

Texture results obtained for the samples drawn at both temperatures presented the X component  $\{110\}\langle 111\rangle$  as the most intense, as also reported by Brandão et al [5]. This component is typically found in FCC metal after large uniaxial deformation [14].

The graphs of Figures 3 and 4, which correspond the samples drawn at ambient and cryogenic temperatures, respectively, describe the evolution of the main texture components and their intensities in the wires with 4.14 mm, 3.0 mm and 2.02 mm diameters.

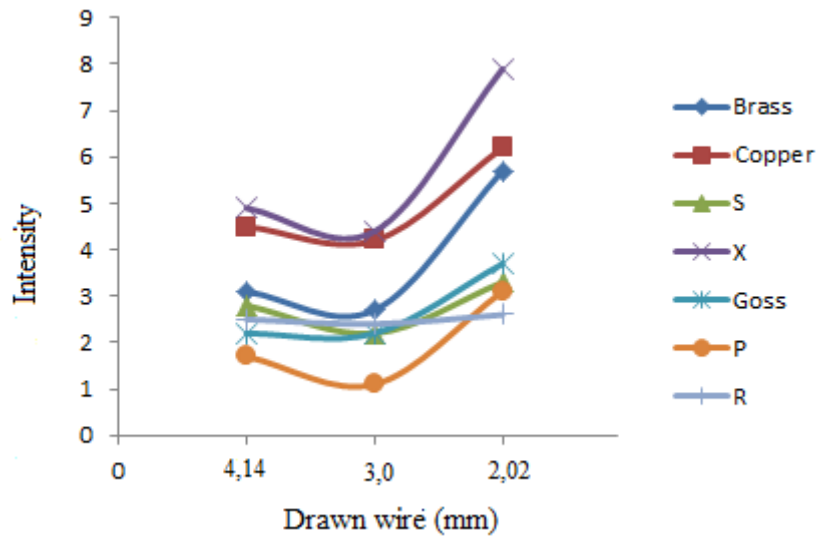
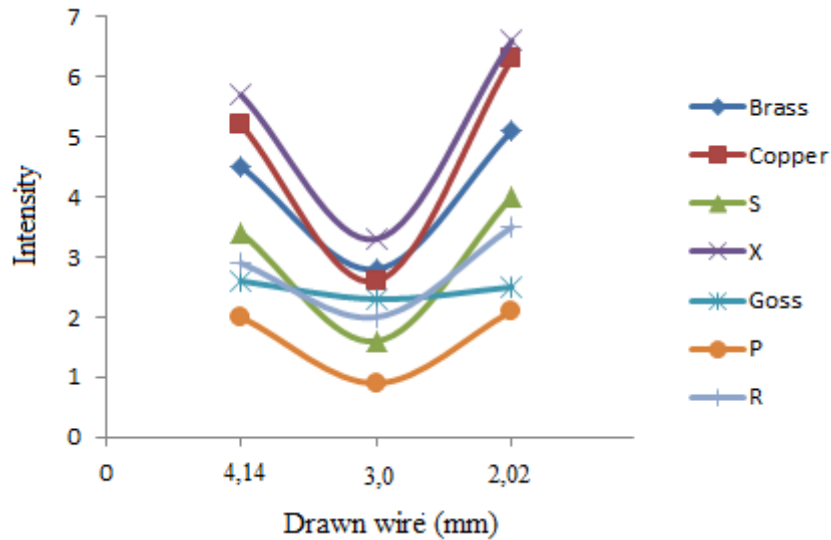


Figure 3: Evolution of the texture components of drawn wires at room temperature.

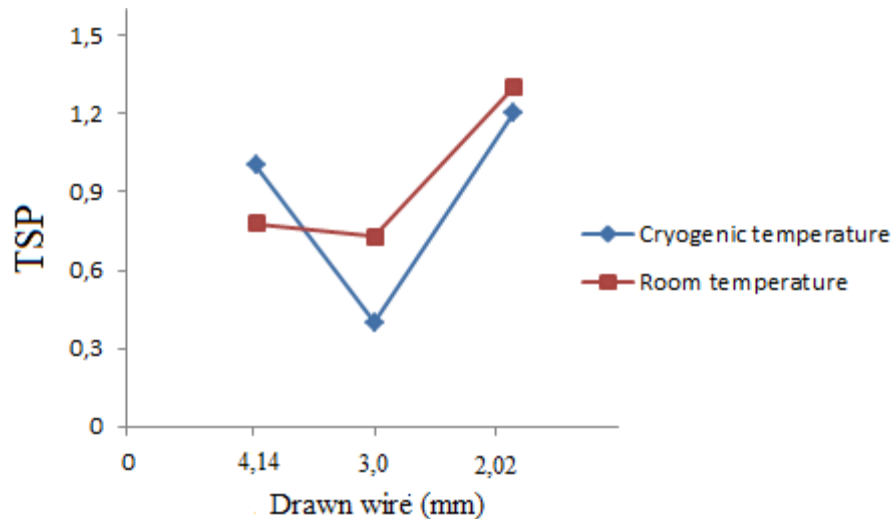


**Figure 4:** Evolution of the texture components of drawn wires at cryogenic temperature.

For the samples drawn at room temperature, it was observed that the intensities of the texture component Copper  $\{112\}\langle 111\rangle$ , S  $\{123\}\langle 634\rangle$ , X  $\{110\}\langle 111\rangle$  and Brass  $\{110\}\langle 112\rangle$  remain approximately constant during several deformation steps and increase considerably in the last stage of the deformation, and the Goss  $\{110\}\langle 001\rangle$  component has similar behavior. On the other hand, the recrystallization typical texture component, P  $\{110\}\langle 122\rangle$ , remained constant all over the deformation except in the wire with 2.02 mm drawn at room temperatures, which has its intensity increased. The intensity of the component R  $\{124\}\langle 211\rangle$  remained almost constant throughout the deformation process.

The texture evolution of the cryogenic drawn samples presented a different behavior when compared to the samples drawn at room temperature. One can observe in Figure 4 that the intensities of the Copper  $\{112\}\langle 111\rangle$ , X  $\{110\}\langle 111\rangle$ , S  $\{123\}\langle 634\rangle$  and Brass  $\{110\}\langle 112\rangle$  components started with an initial intensity and it dropped, during the deformation process, to approximately half of their original values, after increased their intensity values slightly above the initial ones. Goss component  $\{110\}\langle 001\rangle$  appears with a virtually constant intensity throughout the cryogenic drawing process. In relation to the typical recrystallization texture components, R  $\{124\}\langle 211\rangle$  and P  $\{110\}\langle 122\rangle$ , there was observed a decrease followed by another increase in their intensities during the deformation process.

The texture severity parameter (TSP), which represents the overall texture intensity considering all components, for the drawn samples are presented in Figure 5. It is observed for the room temperature samples that the TSP values remain approximately constant during almost whole deformation and they increase only at the last step, i.e., for the 2.02 mm diameter sample. On the other hand, the TSP for the cryogenic drawn samples presented unstable behavior, decreasing for the 3.0 mm sample and increasing again for the 2.02 mm diameter one.

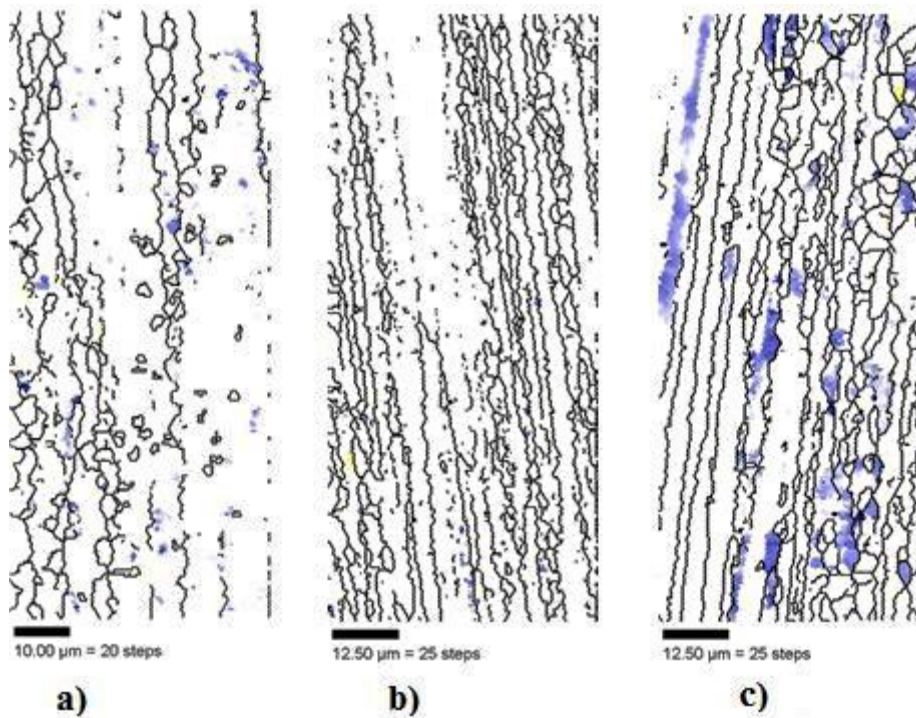


**Figure 5:** Evolution of the texture severity parameter (TSP) of samples drawn at room and cryogenic temperature.

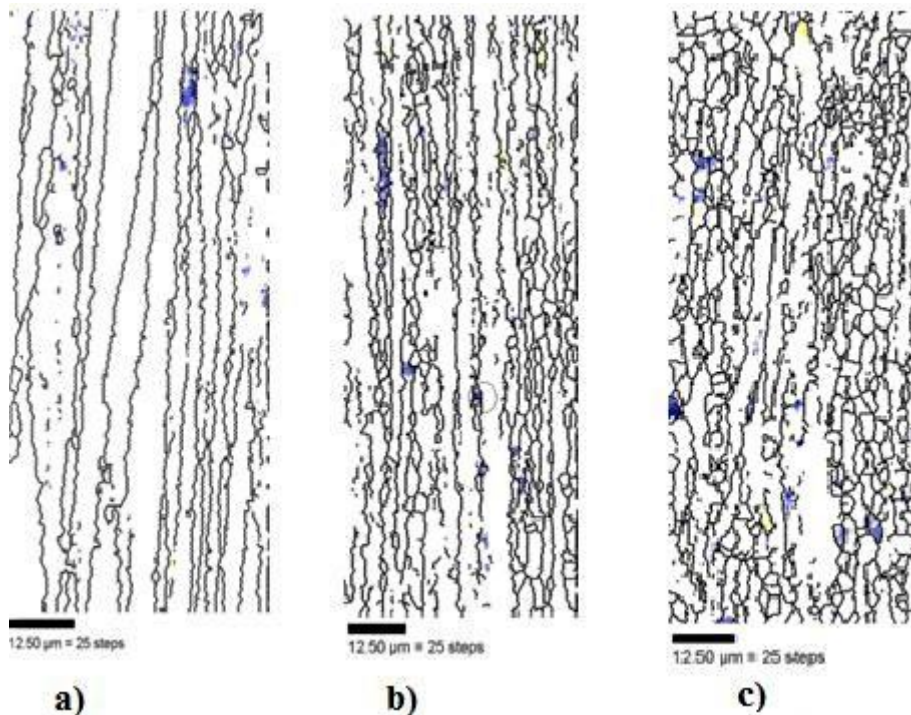
Microtexture was also analyzed using the EBSD technique for the samples drawn at both temperatures. The main objective of this analysis was to investigate the presence of dynamic recovery concomitant with deformation at both temperatures. How the recrystallization texture components were detected through macrotexture analyses, the microtexture was used to observe where and how these components appear in the local microstructures.

In Figures 6 and 7 show orientation image maps (OIM) of the samples deformed at cryogenic and room temperatures, respectively. In these maps, P component  $\{110\}\langle 122\rangle$  is in yellow and R component  $\{124\}\langle 211\rangle$  is in blue. A tolerance of only  $2^\circ$  was adopted for these maps in order to perceive the existence of some recrystallization evidence.





**Figure 6:** Recrystallization typical orientations in cryogenically drawn wire a) 4.14 mm, b) 3.0 mm and c) 2.02 mm, where in blue corresponds to orientation  $\{124\}\langle 211\rangle$  and yellow to  $\{110\}\langle 122\rangle$ .



**Figure 7:** Recrystallization typical orientations in drawn wire at room temperature a) 4.14 mm, b) 3.0 mm and c) 2.02 mm, where in blue corresponds to orientation  $\{124\}\langle 211\rangle$  and yellow to  $\{110\}\langle 122\rangle$ .

Figure 6 presents the OIM of all wires drawn at the cryogenic temperature. There, it could be noted some areas with blue color that represent  $\{124\}\langle 211\rangle$  orientation, but only in cryogenic samples with 2.02 mm diameters was observed some equiaxed grains associated with this color. On other hand, it should be noted that this orientation also appears in elongated grains, which probably are not recrystallized. The

orientation  $(110)\langle 122 \rangle$  (yellow color) appears only in some grains of these samples. In the light of this, analyzing these maps, it was possible to infer that some restoration process is taking place in the cryogenic deformed wire with 2.02 mm, fact which was already reported by Leis [15] who investigated the cross section of these same wires by transmission electron microscope (TEM).

Figure 7 shows the OIM of the samples drawn at room temperature, which shows also the recrystallization typical orientation,  $\{124\}\langle 211 \rangle$ , in blue color. These maps were plotted using the same reference colors and tolerance applied in the cryogenic ones. It could be noted that this orientation was detected associated with equiaxed grains on samples drawn at room temperature with 3.0 mm. In addition, in samples drawn at room temperature with 2.02 mm, also it was inferred these same observation.

On comparison of the OIMs of the samples drawn at room and cryogenic temperature, it was observed that the recrystallization typical orientation,  $\{124\}\langle 211 \rangle$ , is more present in cryogenic samples with 2.02 mm diameters than in samples drawn at room temperature ones, i.e., the maps of this samples is little more blue. At first, this can seem paradoxical but Leis [15] already detected restoration mechanisms in copper wire that was drawing at room temperature. Probably, this recovery was intense enough to prevent recrystallization at room temperature during deformation. Again, the opposite seems to occur during wire drawing at cryogenic temperature, where there was found little restoration but some recrystallization evidence [15], which is consistent with the observation of higher recrystallization texture components which can be associated with the possible presence of some recrystallized grains.

Through the microtexture analysis, one can highlight the presence of the  $\gamma$  fiber  $\{111\}\langle uvw \rangle$  in cryogenic samples with 4.14 mm and 2.02 mm diameters which, according to Humphreys and Hatherly [12], correspond to deformation twin. Consequently, the possible presence of twin mechanical in the cryogenic samples is consistent with less dynamic recovery observed in comparison with wire deformed at room temperature and the recrystallization texture components observed and already discussed above.

#### 4. CONCLUSION

- The texture obtained at both temperature, room and 77K, showed the most intense Copper  $\{112\}\langle 111 \rangle$ , X  $\{110\}\langle 111 \rangle$  and Brass  $\{110\}\langle 112 \rangle$  components, typical components of the deformations texture;

- Through macro and microtexture, it was possible to observe the presence of recrystallization typical texture components, R  $\{124\}\langle 211 \rangle$  at both temperatures. However, it was observed, via OIM produced by EBSD, that the  $\{124\}\langle 211 \rangle$  texture component appeared in some equiaxial grains, apparently recrystallized. Moreover, it observed that this recrystallization typical orientation was more present in cryogenic drawn samples with 2.02 mm diameters, where the recovery was little enough to allow some recrystallization;

- Microtexture revealed also the presence of the  $\gamma$  fiber  $\{111\}\langle uvw \rangle$  in samples drawn at cryogenic temperature, indicating possible presence of deformation twin.

#### 5. ACKNOWLEDGMENTS

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#### 6. BIBLIOGRAPHY

- [1] HAN, K., WALSH, R.P., ISHMAKU, A., *et al.*, "High strength and high electrical conductivity bulk Cu", *Philosophical Magazine*, v. 84, pp. 3705-3716, 2004.
- [2] EMBURY, J. D., HAN, K., "Conductor materials for high field magnets", *Current Opinion in Solid State e Materials Science*, v. 3, pp. 304-308, 1998.
- [3] BRANDÃO, L., WALSH, R.P., HAN, K., *et al.*, "New Cryogenic Processing for the Development of High Strength Copper Wire for Magnetic Application", *Advances in Cryogenic Engineering*, v. 46, pp. 89-96, 2000.
- [4] NIEWCZAS, M., BASINSKI, Z. S., EMBURY, J. D., "The deformation of copper single crystals at 4.2 °K", *Materials Science and Engineering: A*, v. 234-236, pp.1020-1032, 1997.
- [5] BRANDÃO, L., HAN, K., EMBURY, J.D., *et al.*, "Development of High Strength Pure Copper Wires by Cryogenics Deformation for Magnet Applications", *IEEE Transactions on Applied Superconductivity*, v. 10, n. 1, pp. 1284-1287, March 2000.

- [6] CULLITY, B.D., *Elements of ray-X diffraction*, 2nd ed, EUA, Addison-Wesley Publishing Company, 1978.
- [7] BARRETT, C.S., MASSALSKI, T.B., *Structure of metals*, 3rd ed, New York, McGraw-Hill, 1966.
- [8] CHOPKAR, S.T.M., “Effect of heat treatment on proportion of duplex fiber texture of aluminium magnesium silicon alloy”, *Material Science e Engineering International Journal*, v. 1, pp. 52-54, 2017.
- [9] ENGLISH, A.T., CHIN, G.Y., “On the variation of wire texture with stacking fault energy in fcc metals and alloys”, *Acta Metallurgica*, v. 13, pp. 1013-1016, 1965.
- [10] DINIZ, S.B., JÚNIOR, E.S.B., MORAES, N.R.D.C., *et al.*, “Influência da energia de falha de empilhamento na densidade de discordâncias e textura cristalográfica de metais CFC”, In: *70º Congresso Anual da ABM*, pp. 2170-2176, Rio de Janeiro, 2015.
- [11] KAUFFMANN, A., FREUDENBERGER, J., GEISSLER, D., *et al.*, “Severe deformation twinning in pure copper by cryogenic wire drawing”, *Acta Materialia*, v. 59, pp. 7816-7823, 2011.
- [12] HUMPHREYS, F.J., HATHERLY, M., *Recrystallization and Related Annealing Phenomena*, 1st ed, Oxford, Pergamon, 1995.
- [13] SOUSA, T.G., SORDI, V.L., BRANDAO, L.P., “Dislocation density and texture in copper deformed by cold rolling and ECAP”, *Materials Research*, v. 21, 2018.
- [14] HERINGHAUS, F., *Quantitative Analysis of the Influence of the Microstructure on Strength, Resistivity, and Magnetoresistance of Eutectic Silver – Copper*, PhD Thesis, RWTH Aachen University, Aachen, Germany, 1998.
- [15] LEIS, M.P.P., *Microstructura stability of copper drawn at low temperatures*, Sc.M. Thesis, IME, Rio de Janeiro, RJ, Brasil, 2003.

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