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COMPARATIVE STUDY BY TEM, EBSD AND MICROHARDNESS OF THE MICROSTRUCTURE OF COPPER WIREDRAWN AT 77 K AND 295 K

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Abstract

Copper has limited application in coils of high magnetic field electromagnets because of its low mechanical strength. In this work, the mechanical strength of copper wire was increased through severe wiredrawing in several steps at 77 K and transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and Vickers microhardness were used to study the evolution of the microstructure of the material. Comparison of the results with those obtained processing the same material at room temperature (295 K) led to a better understanding of the hardening mechanisms. The wires drawn at 295 K showed dynamic recovery structures, while the wires drawn at 77 K showed a partial recrystallization microstructure. The fact that the Vickers microhardness of the wires wiredrawn at 77 K was about 50% higher than those wiredrawn at 295 K suggests that cryogenic deformation was effective in delaying the recovery of the material.
Keywords: High strength; High conductivity; EBSD; TEM.

ESTUDO COMPARATIVO USANDO MET, EBSD E MICRODUREZA DA MICROESTRUTURA DO COBRE TREFILADO A 77 K E 295 K

Resumo

O cobre tem aplicação limitada em bobinas de eletromagnetos de alto campo magnético devido à sua baixa resistência mecânica. Neste trabalho, a resistência mecânica do cobre foi aumentada através de trefilação severa a 77 K em várias etapas. A microscópia eletrônica de transmissão (MET), a difração de elétrons retorespalhados (EBSD) e a microdureza Vickers foram usadas para o estudo da evolução microestrutural do material. A comparação dos resultados com o mesmo material processado em temperature ambiente (295 K) levou a uma melhor compreensão dos mecanismos de endurecimento. Fios trefilados em 295 K mostraram estruturas de recuperação dinâmica, enquanto que os fios trefilados a 77 K mostraram uma microestrutura de recristalização parcial. O fato da microdureza Vickers do fio trefilado a 77 K seja cerca de 50% maior do que os fios trefilados a 295 K sugere que a deformação criogênica foi efetiva em retardar a recuperação do material.
Palavras-chave: Alta dureza; Alta condutividade; EBSD; MET.

1 INTRODUCTION

The design of electromagnets capable of generating high magnetic fields is limited, among other factors, by the physical characteristics of the conducting material used in the coil. Ideally, this material should have high strength and high electrical conductivity. High strength is needed to withstand the Lorentz forces [1] and high electrical conductivity is important to avoid overheating due to the Joule effect. Therefore, studies of microstructural control have been pursued in order to obtain a material with optimal balance between mechanical strength and electrical conductivity. Recently, it was shown that a possible route for increasing the mechanical strength of copper is deformation at cryogenic temperature. This deformation can be introduced by wiredrawing at 77 K, that promotes

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an increase in the mechanical strength of approximately 50% over the same material wiredrawn at 295 K [2,3]. The idea of strengthening through cryogenic deformation is based in delaying dynamic recovery [4]. Moreover, the deformation in low temperature occurs partially through the twinning mechanism [3,5].

In this work, we studied the microstructural evolution of copper wiredrawn at 77 K and 295 K using the TEM and EBSD techniques. In addition, the samples were subjected to Vickers microhardness tests to evaluate the mechanical strength. The samples drawn at 295 K showed a negligible increase in strength with increasing deformation. On the other hand, the samples drawn at 77 K showed an increase in strength with increasing deformation and exhibited a higher microhardness than the samples drawn at 295 K. However, the work hardening of the material drawn at 77 K decreased for the highest level of deformation, probably due to partial recrystallization mechanisms.

2 MATERIALS AND METHODS

The material used in this research was pure copper - OFHC (C101, 99.99% Cu), processed by wiredrawing at 77 K and 295 K in the National High Magnetic Field Laboratory [2], located in Florida, USA.

The material was received with an initial diameter of 9.6 mm and was wiredrawn to the final diameter of 2.02 mm at the temperatures mentioned above. The wires processed at 295 K were wiredrawn in a FENN drawing bench model D51710 with electric drive. A cryostat attached to a servo-hydraulic tensile machine was used for wiredrawing at 77 K. Samples were taken with diameters of 5.0 mm, 4.14 mm, 3.0 mm and 2.02 mm for both temperatures.

TEM observations were performed using a transmission electron microscope JEOL EM-2010. The samples were ground and chemically polished by a solution of 35% nitric acid (HNO_3) in 65% distilled water down to a thickness of approximately 50 μm. The discs were subjected to ion polishing using a Precision Ion Polishing System (Model 691, Gatan) with 5 keV operating voltage, for 45 min, with a beam angle of about 4°.

For the EBSD analysis the samples were mounted in bakelite, ground, mechanically polished and subjected to vibratory polishing with colloidal silica (0.02 μm) in a VIBROMET machine (Buehler). The measurements were executed in a JEOL 5800LV scanning electron microscope operated at 25 KV and the EBSD analysis was performed using TexSEM software (TexSEM Laboratories).

Vickers microhardness tests were carried out in a microdurometer MICROMET (Buehler) with loads of 10 g (98.07 mN) and the final result was the average of six indenting marks per sample in the cross section of the wire. The areas used for the tests were ground and mechanically polished with diamond paste.

3 RESULTS AND DISCUSSION

Figures 1 and 2 show TEM micrographs of wires deformed at room temperature (295 K) and cryogenic temperature (77 K), respectively. These figures show the sample microstructural evolution taken with diameters of 5.0 mm, 4.14 mm, 3.0 mm and 2.02 mm. For the wires deformed at 295 K (Figure 1), the microstructure shows recovery cells, i.e., dislocation cells that exhibit a small decrease in wall width with increasing deformation. More details on such behavior be found in Sousa et al. [6].

A different microstructural evolution is observed for the wires deformed at 77 K (Figure 2). The wire of 5.0 mm cross section (ε = 1.30) shows evidence for twinning, usually associated with high internal energy, i.e., work hardening. Twinning also appears in the 4.14 mm (ε = 1.68) and 3.15 mm cross section (ε= 2.23) wires, but with smaller spacing between twinning boundaries. This kind of behavior was observed and discussed by several authors [4-10]. Niewczas et al. [4] studied the microstructure of copper single crystals deformed at 4.2 K and reported, in the beginning of deformation, a microstructure dominated by slip planes, followed by non-uniform deformation with twinning

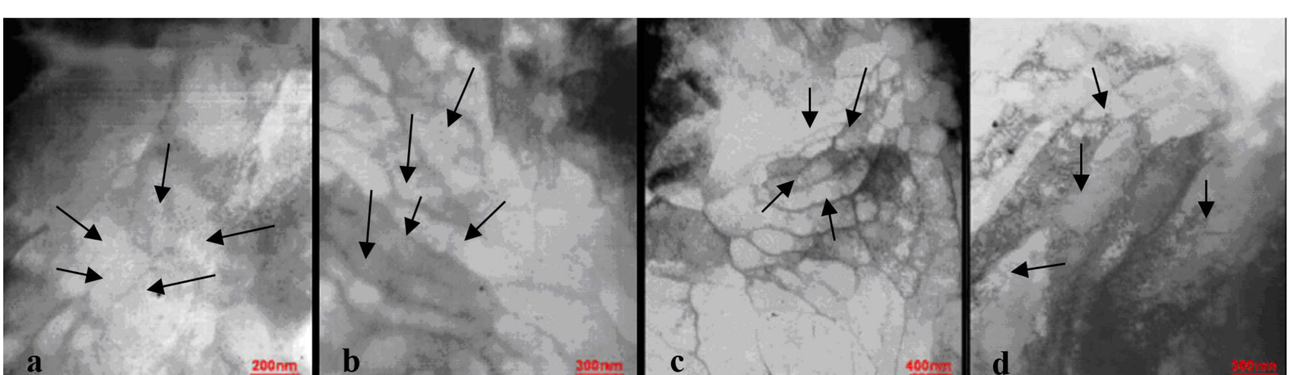

Figure 1. TEM micrographs of wires drawn at 295 K to diameters of (a) 5.00 mm (ε = 1.30), (b) 4.14 mm (ε = 1.68), (c) 3.15 (ε = 2.23) and (d) 2.02 mm (ε = 3.12).

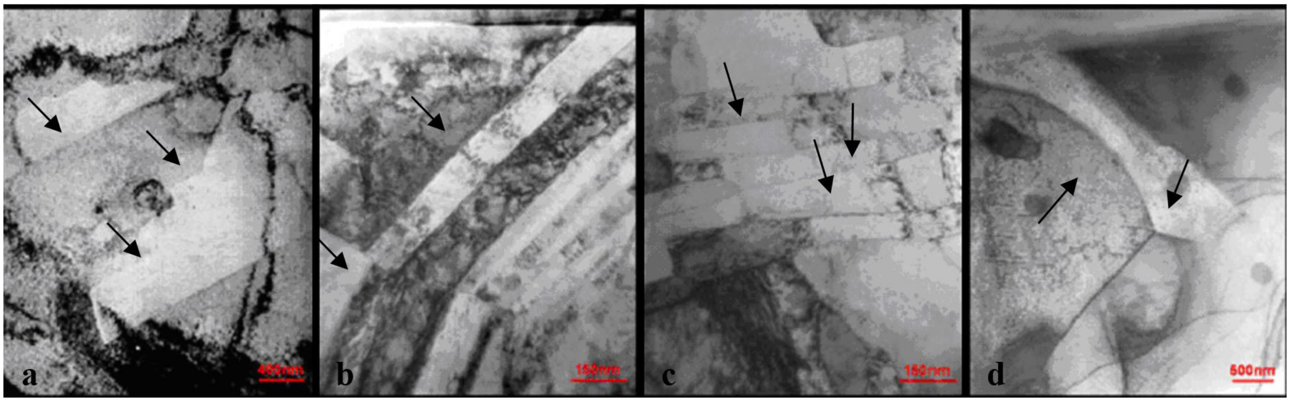

Figure 2. TEM micrographs of wiresdrawn at 77 K to diameters of (a) 5.00 mm (ε = 1.30), (b) 4.14 mm (ε = 1.68), (c) 3.15 (ε = 2.23) and (d) 2.02 mm (ε = 3.12).

which extended to 70% the crystal volume, turning finally into thin lamellas. On the other hand, the microstructure of the wire with 2.02 mm cross section (ε = 3.12) showed partial recrystallization, which is evidenced by the presence of new grains with curved boundaries.

Consequently, the possible presence of mechanical twinning in the cryogenic samples is consistent with the less dynamic recovery observed in this material on comparison to wire deformed at room temperature, that presented partially recrystallization as report by Carvalho et al. [11].

The distance between high-angle boundaries (HABs), i.e., boundaries with angles equal to or larger than 15°, evaluated by EBSD, is shown in Figure 3 as a function of deformation. For wires drawn at 295 K and 77 K, this distance decreases with increasing deformation up to ε = 2.23, but the decrease is larger for samples wiredrawn at 77 K, which show a larger distance between HABs for small deformations. This suggests that the wires drawn at 77 K exhibit more work hardening than wires drawn at 295 K. For deformations larger than ε = 2.23, the distance between HABs increases, probably due to recovery. This behavior is consistent with the microstructures observed by TEM.

The influence of deformation on low-angle boundaries (LABs), i.e., boundaries with angles smaller than 15°, may also be related to the work hardening mechanisms that operate during the deformation process. One of the most important is the subdivision of grains to accommodate the deformation, as suggested by Hugues and Hansen [12]. The cell boundaries are also low-angle boundaries and may be part of a recovery structure.

The percentage of low-angle boundaries relative to the total number of boundaries is shown in Figure 4 as a function of deformation. For wires drawn at 77 K, the percentage of LABs increased initially, up to ε = 1.77, but decreased for larger deformations. This is attributed to work hardening followed by recovery and/or recrystallization. For wires drawn at 295 K, the percentage of LABs increased initially and remained virtually constant for larger deformations, probably due to competition between work hardening and

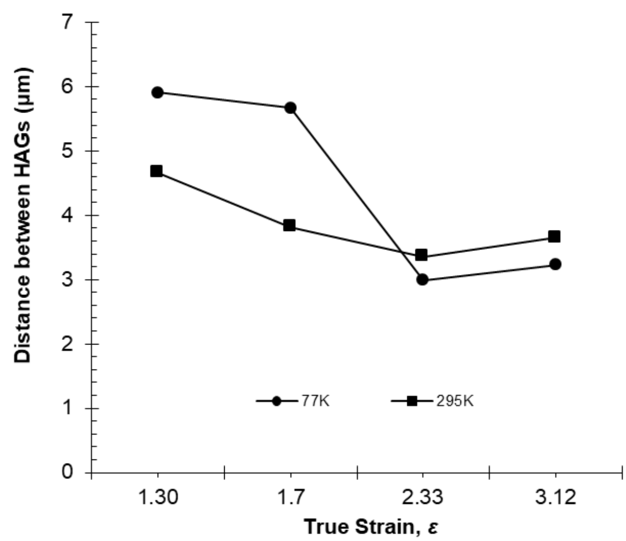

Figure 3. Distance between high-angle boundaries as a function of deformation.

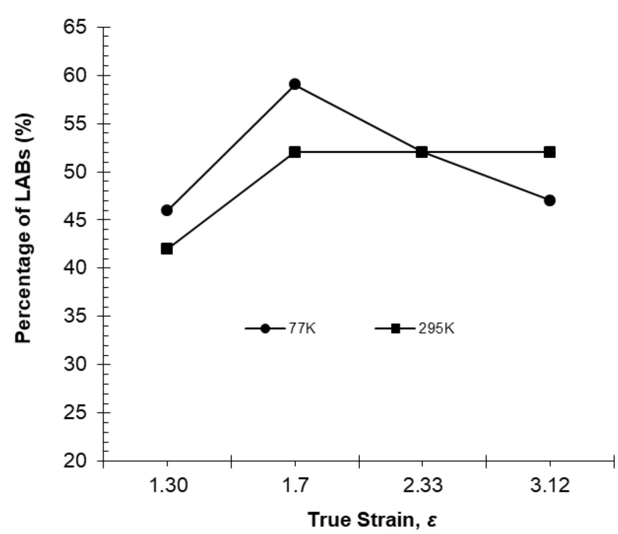

Figure 4. Percentage of low-angle boundaries as a function of deformation.

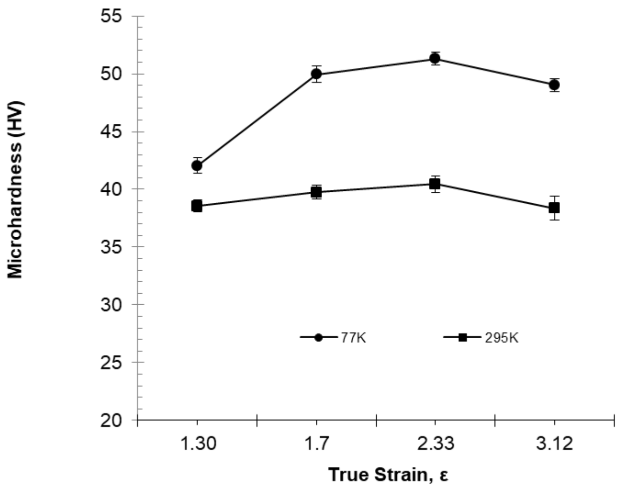

Figure 5. Vickers microhardness versus true strain for drawn wires processed at 295 K and 77 K.

recovery. This behavior is consistent with the microstructures observed by TEM.

Figure 5 shows the Vickers microhardness of the copper wires drawn at 77 K and at 295 K for different deformations. One can observe that: (a) the microhardness of wires drawn at 77 K is higher than that of the wires deformed at room temperature; (b) the work hardening for the wires drawn at 77 K is also higher than that of wires drawn at room temperature; and (c) the maximum microhardness for both temperatures is reached for $\varepsilon = 2.23$.

These results suggest that wire drawing at 77 K was mechanically effective in delaying the occurrence of the dynamic recovery. Consequently, it produced higher work hardening up to a deformation of $\varepsilon = 2.23$, thus yielding higher microhardness values. This mechanical behavior is consistent with the TEM observations. Similar observations were made by Kauffmann et al. [13]. They concluded that low temperature deformation results in increase of strength due to deformation twinning and the suppression of dynamic recovery.

For both wiredrawing temperatures, the microhardness decreases for deformations above $\varepsilon = 3.12$. This can be considered as evidence of dynamic recovery or partial recrystallization, which is consistent with microstructural observations.

The microhardness changes with deformation exhibited by the wires drawn at 295 K are much smaller than those observed for the wires drawn at 77 K. This suggests the existence of softening mechanisms that depend on deformation temperature. The microstructure observations support this assumption, since they are typical of work hardened materials. The decrease in hardness of the wire with $\varepsilon = 3.12$ can be explained by a large increase in internal energy due to additional deformation, promoting recovery or even partial recrystallization of the material. Brandão [2,3] reported that the mechanical strength of copper wires drawn at 77 K and 295 K, determined through tensile tests, show similar trends as the Vickers microhardness observed in this present work. These results are consistent with the microstructure characteristics previously described and discussed.

4 CONCLUSIONS

- The relative number of LABs, supported by microhardness tests and observations of the microstructure by TEM, suggest a competition between work hardening and recovery for the samples drawn at 295 K, which show no significant increase in strength with increasing deformation, even for the highest levels of strain;
- The relative number of LABs, supported by microhardness tests and observations of the microstructure by TEM, suggest that drawing at a cryogenic temperature (77 K) is effective in delaying recovery and therefore producing greater hardening than samples drawn at room temperature (295 K). This is reflected on the highest microhardness observed for samples drawn at 77 K;
- The Vickers microhardness of the samples wiredrawn at 77 K initially increases with deformation but decreases for the highest deformation level. This decrease in hardness is attributed to the partial recrystallization observed by microstructural analysis after the very last step of wiredrawing.

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