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ARTICLE

Microstructure and Texture Evolution of Cu-Nb Nanocomposite Wire After Heat Treatment

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Abstract: The microstructure and texture evolution of Cu-Nb nanocomposite wire after heat treatment were characterized by the scanning electron microscope and electron backscatter diffractometer (EBSD). The recrystallization, nucleation, large angle boundary, and residual internal stress of the nanocomposite were also discussed. Results demonstrate that after the annealing treatment, the grain recovery and recrystallization occurs in the Cu-Nb nanocomposite and a small number of large angle boundaries appear. The residual internal stress becomes weaker after the annealing treatment. After annealing over 600 °C, the grains in Nb filaments are recrystallized and grow significantly. The Cu-Nb nanocomposite wire has good structural stability at high temperatures.

Key words: Cu-Nb nanocomposite; texture evolution; recrystallization; grain size; spheroidization

Cu-Nb multi-filaments composites have been widely investigated in the past several decades due to their high strength and high conductivity[1]. These superior properties are beneficial to the high field pulsed magnets. The nano-filaments and Cu/Nb interface contribute to the high strength of materials[2–3]. Heat treatment has a significant impact on the morphology, texture orientation, mechanical and electrical properties of Cu-Nb composites. Sandim et al[4] studied the effect of heat treatment temperature on the microstructure of Nb filaments, and reported that the spheroidization of Nb filaments is obvious above 800 °C, and a bamboo-like structure is generated on the surface of Nb filaments due to the filament rupture caused by the spheroidization. The splitting mechanism of grain boundary and the spheroidization model of composite material are used to explain the spheroidization mechanism of Nb filaments[5–7]. Lei et al[8] studied the microstructure evolution and high thermal stability of Cu-10%Nb alloy by X-ray diffractometer (XRD) and transmission electron microscope (TEM). It is found that the diffraction peaks of Cu-Nb alloy become sharper and their intensity becomes stronger with increasing the temperature from 600 °C. This phenomenon is related to the change in lattice parameter, grain size, internal strain, and dislocation density of Cu matrix caused by the high temperature. Sandim et al[9] analyzed the microstructure evolution of Cu-Nb alloys after annealing at different temperatures, and reported that the distribution of grain boundaries is changed significantly with increasing the annealing temperature to 1050 °C, and the bamboo-like microstructure is also generated[10].

According to the abovementioned research, it is of great significance to investigate the spheroidization mechanism of Nb filaments, the crystal growth kinetics (crystal nucleation, recovery, and recrystallization), and interface diffusion mechanism through the study of the high thermal stability of Cu-Nb nanocomposites. In this research, the annealing effects on the microstructure and texture evolution of Cu-Nb nanocomposite wire fabricated by the accumulative drawing and bundling (ADB) process were investigated. The texture evolution of Cu matrix and Nb filaments after vacuum annealing process was discussed.

1 Experiment

The Cu-Nb nanocomposite wire reinforced with Nb nanotube was fabricated through ADB which contained a series of hot extrusion and cold drawing processes. The size of Nb filaments after severe plastic deformation was remarkably decreased from the millimeter-level to the nanometer-level. Finally, the Cu-Nb wires with 583^3 continuous Nb nanofibers embedded in the multiscale Cu matrix were obtained, as shown in Fig. 1. Then the speci-

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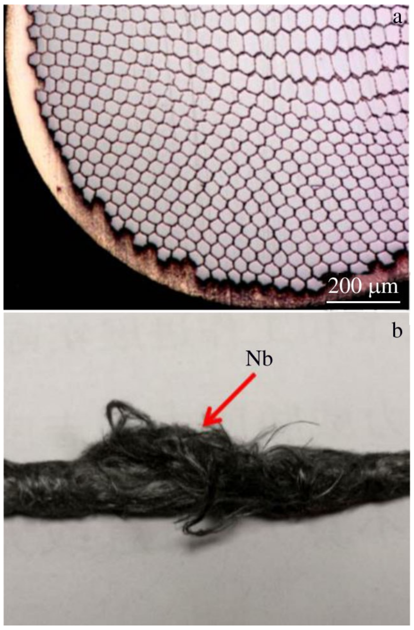

Fig.1 Appearances of Cu-Nb nanocomposite wire before (a) and after (b) corrosion

mens with diameter of 2–4 mm were annealed at 0–830 °C for 3 h in vacuum. The Cu-Nb specimens were etched by HNO_3 solution to obtain the single Nb filament for microstructure observation.

The crystal orientation evolution of Cu-Nb nanocomposite wire was observed by Germany QUANTAX electron back-scatter diffractometer (EBSD). The microstructure characterization of the deformed and annealed specimens was performed by the JSM-6700 field emission scanning electron microscopes (SEM).

2 Results and Discussion

2.1 EBSD analysis

The orientation distribution figures (ODFs), inverse pole figures (IPFs), and pole figures of the processed and annealed Cu-Nb nanocomposite specimens are shown in Fig.2. It can be seen that the Cu matrix and the Nb reinforcement phase of the Cu-Nb nanocomposite present the drawing texture of $<111>_{Cu}$ $//<110>_{Nb}$, which is parallel to the drawing direction after large plastic deformation, as indicated by the blue and red areas in Fig.2a, respectively. Furthermore, besides the $<111>_{Cu}$ $//<110>_{Nb}$ orientation, a few secondary orientations can also be observed in Fig. 2a. However, the Cu matrix and the Nb filaments are recovered and recrystallized during the annealing process at 830 °C, and the density of annealing texture, namely recrystallization texture, changes significantly due to the variation in grain size. The main orientation density of texture obviously weakens after annealing, as shown in Fig. 2d. According to the crystallographic theory[11], the slip deformation mechanism of body-centered-cubic (bcc) Nb is more complicated than that of the face-centered cubic (fcc) Cu. Therefore, the Nb phase exhibits a strong <110> silk texture parallel to the drawing direction after the large plastic deformation. However, the recrystallization texture of Nb <110> in the processed Cu-Nb nanocomposite is significantly weaker than that in the Cu-Nb nanocomposite after annealing at 830 °C. The grain orientation is changed due to the recrystallization and growth of Nb phase at high temperatures, and a typical recrystallization texture with a strong cube component appears. Thus, the strong deformation texture during plastic deformation becomes weak, which is consistent with XRD results in Ref.[12].

Fig.3 shows the grain misorientations of adjacent grains of

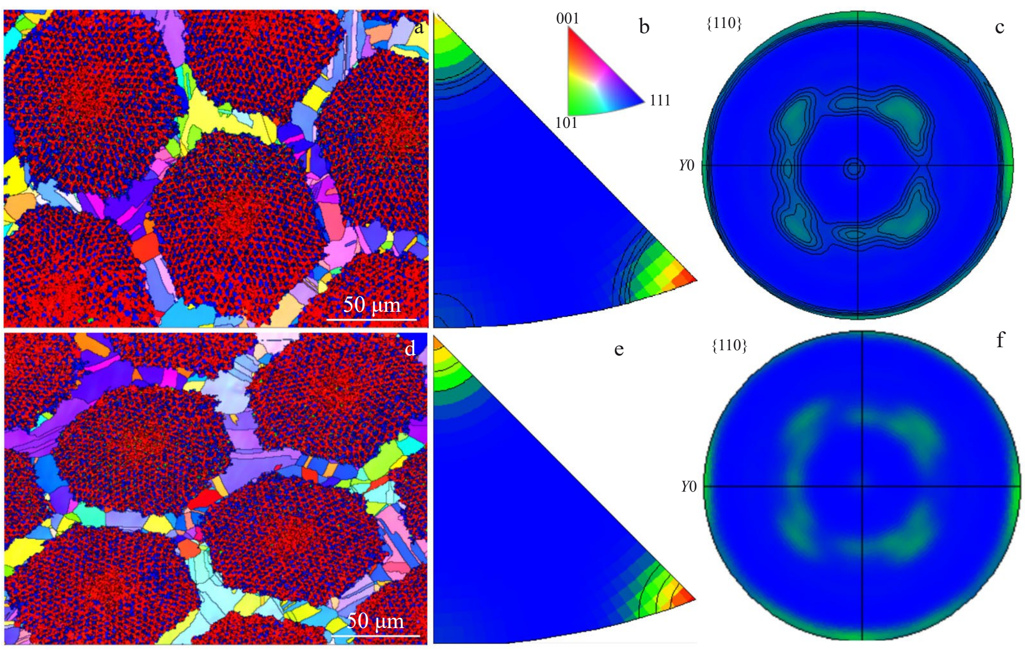

Fig.2 ODFs (a, d), IPFs (b, e), and pole figures (c, f) of processed (a–c) and annealed (d–f) Cu-Nb nanocomposites

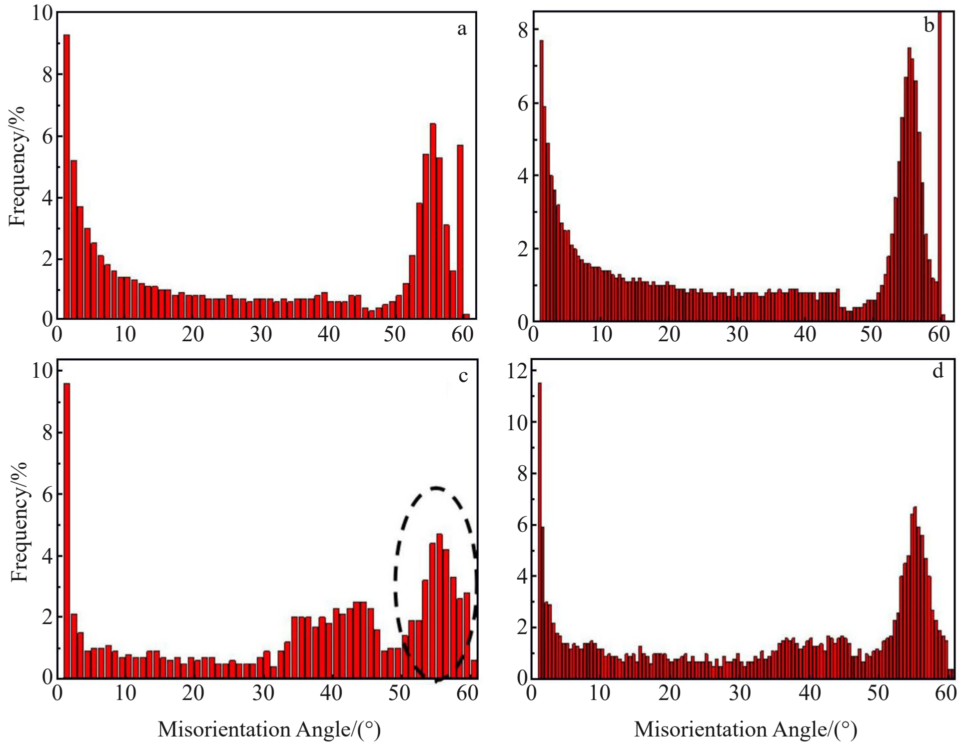

Fig.3 Grain misorientations of Cu matrix (a, b) and Nb filament (c, d) in processed (a, c) and annealed (b, d) Cu-Nb nanocomposites

Cu matrix and Nb filaments in the processed and annealed Cu-Nb nanocomposites. It can be seen that the orientation of adjacent grains in Cu-Nb nanocomposites is dominated by the small angle grain boundaries during the plastic deformation, indicating that the average orientation difference is increased with the deformation proceeding. However, after the large plastic deformation, a small number of large angle grain boundaries (50° – 60°) appear, as indicated by the marked region in Fig. 3c. According to Ref. [13], the existence of a small number of large angle grain boundaries is caused by the following reasons. Firstly, according to the crystallographic theory, the bcc Cu phase and the angle between $(111)_{Cu}$ and $(100)_{Cu}$ crystal planes of about 54° both lead to the appearance of large angle grain boundaries. Secondly, the large angle grain boundary exists with a tilted angle of about 54° inside the Cu matrix due to the multi-scale structure of the Cu matrix. Therefore, these grains result in a small number of large angle grain boundaries.

In addition, the number of large angle grain boundaries in Cu-Nb nanocomposites is increased after the high-temperature annealing. The proportion of crystal grains with an orientation difference of 54° is also increased significantly. The preferred orientations of the material is obviously weakened due to the orientation difference of the crystal grains. It is believed that the complete recovery and recrystallization of Cu and Nb phases as well as the recrystallization growth of the crystal grains are the main factors affecting the orientation difference of the crystal grains. The average orientation difference between the adjacent grains of Cu matrix and Nb filaments is decreased with increasing annealing temperature to 830 °C.

According to Fig. 4, during the large plastic deformation, the dynamic recrystallized, substructured, and deformed grains can be observed in the Cu-Nb nanocomposites. Besides, the subgrains are more obvious in the Cu matrix than that in the Nb filaments. It is found that both the Cu matrix and the Nb filaments are completely recrystallized, and the number of recrystallized grains is increased significantly after annealing at 830 °C. However, after the large plastic deformation, the number of deformed grains caused by the residual stress is decreased, and that of the subgrains in Cu matrix is increased. It is clear that the recrystallized grains of Cu matrix in the center layer are relatively small, while those in the epitaxial region are relatively large. Generally, the recovery can easily generate the subgrains[14]. According to the analysis, the Cu layers with different sizes are not uniformly stressed due to the multi-scale effect of Cu matrix during the large plastic deformation, resulting in severely uneven plastic deformation. Thus, the subgrains are generated. The recovery and recrystallization occur in the Cu matrix. The subgrain size barely changes at the beginning of recovery, whereas the subgrains begin to nucleate and grow with increasing the annealing temperature. When the annealing temperature exceeds the recrystallization temperature, the subgrain size is increased significantly. It is found that the Nb grains are mainly at the nanometer scale after the large plastic deformation. Besides, the deformation is more uniform in the Nb grains. Therefore, less subgrains are generated in the Nb filaments. These phenomena may be related to the multi-scale characteristics of the Cu matrix[15].

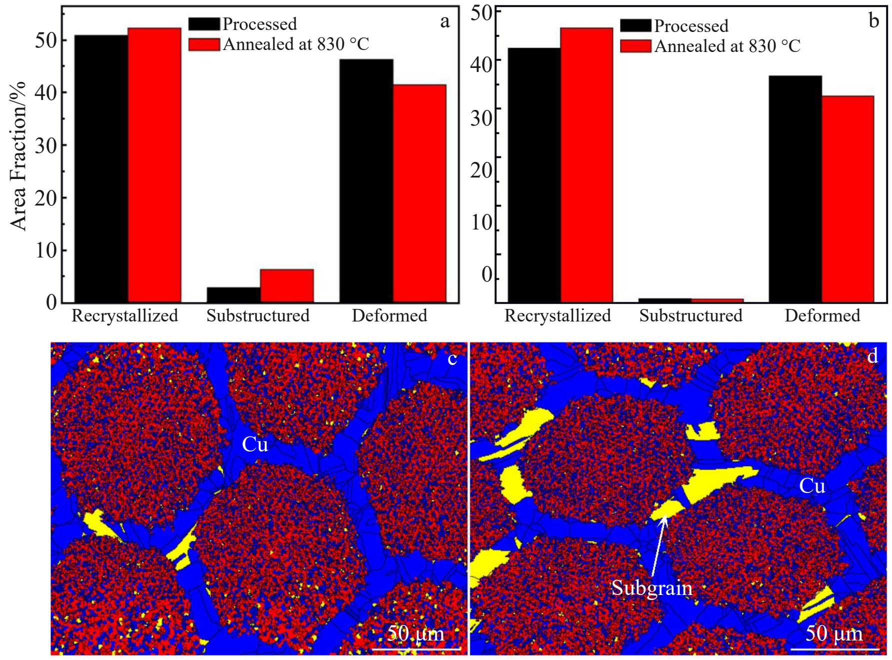

Fig.4 Grain distributions of Cu matrix (a) and Nb filament (b) in Cu-Nb nanocomposites; recrystallization distributions of processed (c) and annealed (d) Cu-Nb nanocomposites

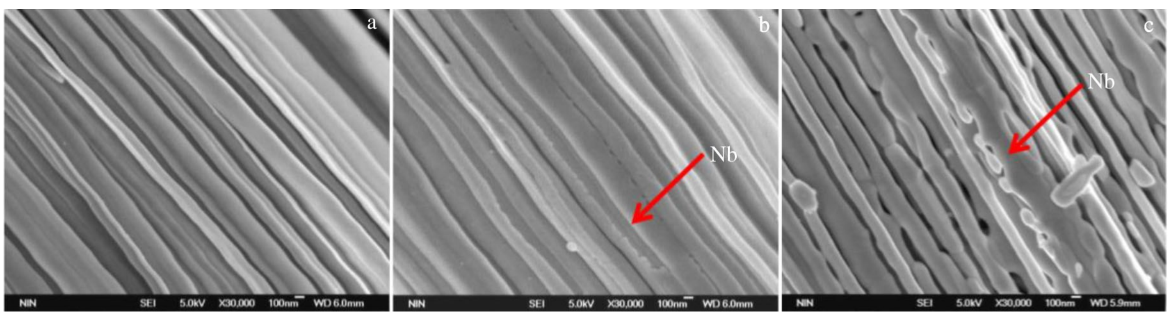

Fig.5 SEM longitudinal-section microstructures of processed (a) and annealed Cu-Nb nanocomposites at 400 °C (b) and 600 °C (c)

2.2 SEM microstructure

Fig. 5 shows SEM longitudinal-section microstructures of processed and annealed Cu-Nb nanocomposites. It can be seen that the Nb filaments slightly change after annealing at low temperature of 400 °C, whereas they change significantly after annealing at high temperature of 600 °C. Some depression forms at the edge of Nb filaments after annealing at low temperature firstly. Then, the degree of the depression is gradually increased with increasing the annealing temperature. It is found that the spheroidization of Nb filaments occurs at high temperature. In addition to spheroidization, the coarsening of the Nb filaments can also be observed. Therefore, some bamboo-shaped structures are formed on the Nb filaments after annealing at high temperature, which are caused by the coalescence at the transverse-section of grain boundaries and between the adjacent Nb filaments[16]. In addition, the structural integrity of the Nb filaments is retained to some extent, and various separation types can be clearly distinguished. This is related to the small grain size and high strain energy in the Cu-Nb nanocomposites, which are conducive to the grain recrystallization and nucleation[17–18]. Therefore, the Cu-Nb nanocomposite wire shows superior thermal stability with high strength and high conductivity.

3 Conclusions

1) The annealing treatment leads to the grain recovery and recrystallization in Cu-Nb nanocomposites. Due to the recrystallization and nucleation of grains, a small number of large angle grain boundaries appear.

2) The residual internal stress in Cu-Nb nanocomposites becomes weak after annealing treatment, resulting in the rotation of grains and grain boundaries. Thus, the Nb<111> annealed texture is significantly weakened.

3) After annealing over 600 °C, the grains in Nb filaments are recrystallized and grow significantly. Some bamboo-shaped structures appear on the annealed Nb filaments, which leads to the growth and spheroidization of the grains.

4) The Cu-Nb nanocomposite wire shows superior thermal

stability with high strength and high conductivity.

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热处理Cu-Nb纳米复合线的结构和织构演变

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摘 要：使用扫描电子显微镜和电子背散射衍射（EBSD）对热处理Cu-Nb纳米复合线的微观结构和织构演变进行了表征，同时对复合线材再结晶、晶粒形核、大角度晶界和残余内应力进行了讨论。结果表明，热处理使Cu-Nb纳米复合材料发生晶粒回复与再结晶，因此产生少量大角度晶界。热处理后，Cu-Nb复合材料的残余内应力减小。600 ℃以上热处理，Nb丝发生再结晶并且晶粒快速长大。Cu-Nb纳米复合线材具有良好的高温热稳定性。

关键词：Cu-Nb复合材料；织构演变；再结晶；晶粒尺寸；球化

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