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Annealing effects on the microstructure and texture of a multifilamentary Cu–Nb composite wire

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Abstract

We report the microstructural evolution of a Cu–15%Nb composite annealed from 100 to 1050 °C. The microstructure was characterized by scanning electron microscopy (SEM) and high-resolution electron backscatter diffraction (EBSD). Boundary splitting is the predominant mechanism to explain the spheroidization of niobium. A simple model describing this phenomenon is proposed.

Keywords: Cu–Nb composite; Microtexture; Boundary splitting; Spheroidization; EBSD

1. Introduction

Multifilamentary Cu–Nb composites are excellent candidate materials for the area of high-field magnet design owing to their high mechanical strength and good electrical conductivity [1,2].

While the room-temperature microstructures and textures of these materials have been investigated in great detail [3–7] not many corresponding studies were conducted after heat treatment. At ambient temperature the copper majority phase mostly develops a sharp ⟨1 1 1⟩-fiber texture with a secondary ⟨1 0 0⟩ component during wire drawing [7]. The body centered cubic (BCC) minority phase, typically niobium, tends to develop a pronounced ⟨1 1 0⟩-fiber texture when deformed to large wire strains at room temperature [7].

The development of microstructure and texture at high temperatures is of interest for two reasons. First, depending on the intended application, Cu-based composites must be heat treated at high temperatures, e.g., when manufacturing Cu–Nb reinforced Nb_3Sn superconducting cables [2]. Second, substantial heating of heavily drawn or rolled samples occurs when operated in steady state or pulsed magnets due to resistive heating.

The special difficulty in the investigation of the microtexture and texture development of the BCC minority phase in the annealed state lies in the fact that the niobium filaments are very fine after drawing (usually below 1 μm in diameter [7]). Such structures cannot be easily resolved by conventional EBSD techniques. High-resolution EBSD, on the other hand, is capable to resolve such fine structures easing the determination of both meso- and microtextures developed particularly in heavily deformed niobium.

Annealing at high homologous temperatures causes significant changes in the microstructure of this Cu–Nb composite, in particular those concerning the morphology and interspacing of niobium ribbons. In this regard, it is worthwhile mentioning that both the morphology and the interfilament spacing exert considerable influence on the mechanical [7,8], electrical [9–11],

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and magnetic properties [12] of multifilamentary Cu–Nb conductors. A detailed description of the microstructural evolution of the composite reported in the present investigation is given elsewhere [12].

In this paper, we report the main results concerning the microstructural evolution and texture development in Cu–15%Nb composite wires upon annealing, in particular those concerning the occurrence of partial spheroidization. A simple model is proposed to describe the microstructural evolution observed during the isothermal annealing of this composite.

2. Experimental

2.1. Material

The bundling-and-drawing process using high-purity niobium (ASTM-B-391-89 commercial grade unalloyed niobium) and OFHC-copper was used to manufacture the multifilamentary Cu–15%Nb (in vol%) composite with 418,399 niobium filaments. The Cu–Nb composite was deformed to a total areal reduction of about 10^8. Annealing treatments were performed in vacuum after every restacking step aiming the recrystallization of the copper matrix and intensive recovery of niobium filaments to allow subsequent deformation of the composite. Following the last restacking step, the composite was cold drawn up to 0.8 mm diameter with a total strain of $\eta \approx 6$, where $\eta = \ln(A_o/A_f)$ and A_o and A_f are the initial and final cross-sectional areas, respectively. Vacuum annealing of a composite with 0.8 mm diameter was performed from 100 to 1050 °C with times varying from 1 to 32 h.

2.2. Microstructural characterization

The microstructure of niobium filaments was investigated in a LEO 1450-VP scanning electron microscope operating at 10 kV in the secondary electrons mode. In order to observe the microstructure of the niobium filaments in detail, samples were deeply etched with nitric acid to remove the copper matrix. The EBSD scans were carried out in longitudinal sections of the annealed composite. In every part of this article, LD represents the longitudinal direction and TD the transverse direction. Microtexture evaluation was determined by means of automated acquisition and further indexing of Kikuchi patterns after suitable image processing in a TSL system interfaced to a JEOL JSM-6500F field emission gun scanning electron microscope (FEG-SEM) operating at 15 kV. EBSD sampling points were performed in every 0.05–0.1 μm (corresponding to the map step size). Mapping speed in the recrystallized state was about 25 patterns per second. (100)-pole figures and boundary characteristics were determined for each mapped region. Vickers microhardness tests were performed using a load of 100 g. The results presented correspond to the mean of fifteen values taken on each specimen.

3. Results and discussion

The niobium filaments develop a ribbon-like aspect with a pronounced surface grooving along their longitudinal direction after severe straining. In the cold-worked state, these filaments are about 2 μm wide and 200 nm thick. This gives a plate aspect ratio of 10. The plate aspect ratio is defined by w/t, where w is the width of the plate and t its thickness. The interspacing varies from 0.5 to about 1 μm.

Grain subdivision of high stacking fault energy metals like niobium gives rise to a microstructure subdivided by strongly misoriented lamellae bounded by lamellar boundaries (LBs), many of them with high angle character (misorientation above 15°) [12]. These LBs are nearly aligned with the longitudinal direction of the niobium ribbons and separate lamellae of different orientation constituting the elements of the microtexture [13].

The softening behavior displayed by this composite upon annealing is shown in Fig. 1. Hardness decreases monotonically with increasing annealing temperatures. For purposes of comparison, the softening behavior of the copper matrix is also shown. Metallographic inspection confirms the occurrence of full recrystallization in the copper matrix above 400 °C. Grain growth prevails at higher annealing temperatures. We would like to stress the remarkable transformations taking place in niobium filaments. A closer inspection in SEM reveals important microstructural changes in niobium ribbons in terms of morphology and further fragmentation. Annealing ranging from 100 to 600 °C did not produce any sign of recrystallization in niobium (not shown in

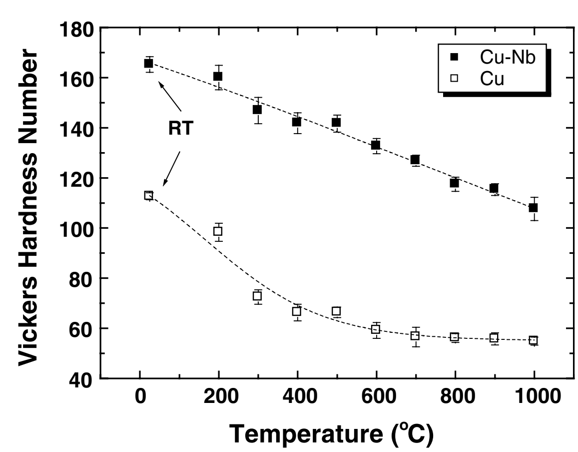

Fig. 1. Softening behaviors of the multifilamentary Cu–15%Nb composite and copper matrix. RT means room temperature.

this paper). Recrystallization becomes noticeable above 700 °C as well as the beginning of spheroidization. Both features appear to occur concurrently, however, the extent of recrystallization varies with temperature as well as from one filament to another.

We concentrate our investigation on the microstructural evolution of composites annealed above 700 °C, i.e., in the temperature range where the fragmentation of niobium ribbons becomes pronounced. The development of a single theory to explain the occurrence of shape instabilities in plate-like bodies is rather complicated. Direct cylinderization, edge spheroidization, and boundary splitting have been proposed as primary instability modes [14]. However, metallographic observation of real microstructures suggests the overlap of all these mechanisms. Based on our findings, boundary splitting seems to be the predominant mechanism during fragmentation of niobium ribbons in the present investigation. Boundary splitting is a thermal instability associated with the presence of internal boundaries lying parallel to the longitudinal axis of the plate [14]. These boundaries arise either due to severe straining (LBs) or recrystallization of the lamellar structure [15]. When the system is heated sufficiently to allow appreciable diffusion, a groove develops along the boundary. Two ridges border the groove that forms. The curvature of the ridges promotes a mass transport away from the groove. Reestablishment of the equilibrium among the surface tensions continuously deepens the grooves [16]. As the groove deepens, boundary splitting occurs. Following splitting, the ''new'' plates undergo a cylinderization process, as a consequence of their lesser aspect ratios. In such a process, there is a mass transfer from the edges towards the flat surface of the plate. After cylinderization, the disturbances of the surface of the cylinder can grow and, eventually, the cylinder can break up into an ideal row of isolated spheres [14]. These features are clearly illustrated in Fig. 2a corresponding to a composite wire annealed at 1000 °C for 1 h. Five individual niobium ribbons are seen in this micrograph. One can clearly observe the occurrence of a pronounced fragmentation, preferentially along former lamellar boundaries. In average, every individual ribbon-like filament is subdivided into four or five 0.5 μm-dia. rod-like structures. Individual spheres (as predicted by theory) are not visible throughout these fragmented structures. However, the tendency for cylinderization is very pronounced. While the fragmentation of the individual filaments proceeds, new contact points formed by diffusion-assisted processes are also observed joining the already fragmented parts with the very adjacent rod-like structures. These new contact points aid coarsening of niobium cylinders at high annealing temperatures. Similar evidence of boundary-induced ribbon splitting as we have just described was found for a Cu–14.3vol% Fe composite wire annealed for 2 h at 800 °C [17].

The recrystallization of niobium is evident and the filament morphology evolves to a bamboo-like structure above 1000 °C (Fig. 2b). This micrograph displays an enlarged view of the niobium ribbons after severe annealing at 1050 °C for 32 h. At this stage, cylinderization of niobium has occurred in large extent. Transverse grain boundaries are observed along the filaments. According to Sharma et al. [15], it is plausible admit that filament breakup is somehow accelerated at high-angle boundaries because of the higher diffusivity associated to these surfaces. After annealing at this severe condition, filament breakup occurs in many regions, however, at this stage coarsening is predominant.

At this point of the discussion, it must be stressed that boundary splitting is directly competitive with cylinderization, since the fluxes of the diffusion masses are antiparallel [16]. There are two parameters that decide which of the two processes is the dominant one: the degree of misorientation associated with LBs and the aspect ratio of the ribbon-like filaments. In general, large aspect ratios of the ribbon and highly misoriented LBs favor the boundary splitting process. Another feature has to be considered. The diameter of the niobium filaments presents a size distribution. It means that the described steps leading to breakup and further coarsening of niobium occur in part concurrently and differently

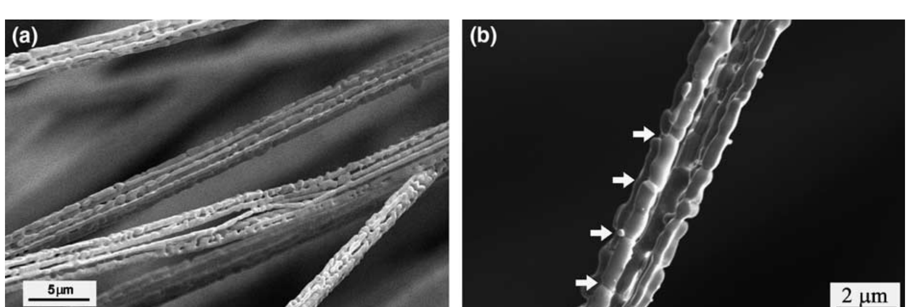

Fig. 2. Changes in the morphology of former niobium ribbons upon annealing: (a) fragmentation along longitudinal direction in specimens annealed at 1000 °C for 1 h; (b) bamboo structure and thermal grooving at the transverse grain boundaries in niobium (marked by white arrows) due to recrystallization in a Cu–Nb composite annealed at 1050 °C for 32 h (SEM, SEI).

in many parts of the same sample. Such a behavior can be attributed to the size distributions of the filaments and to the overlap of the diffusion fields in the sample [18].

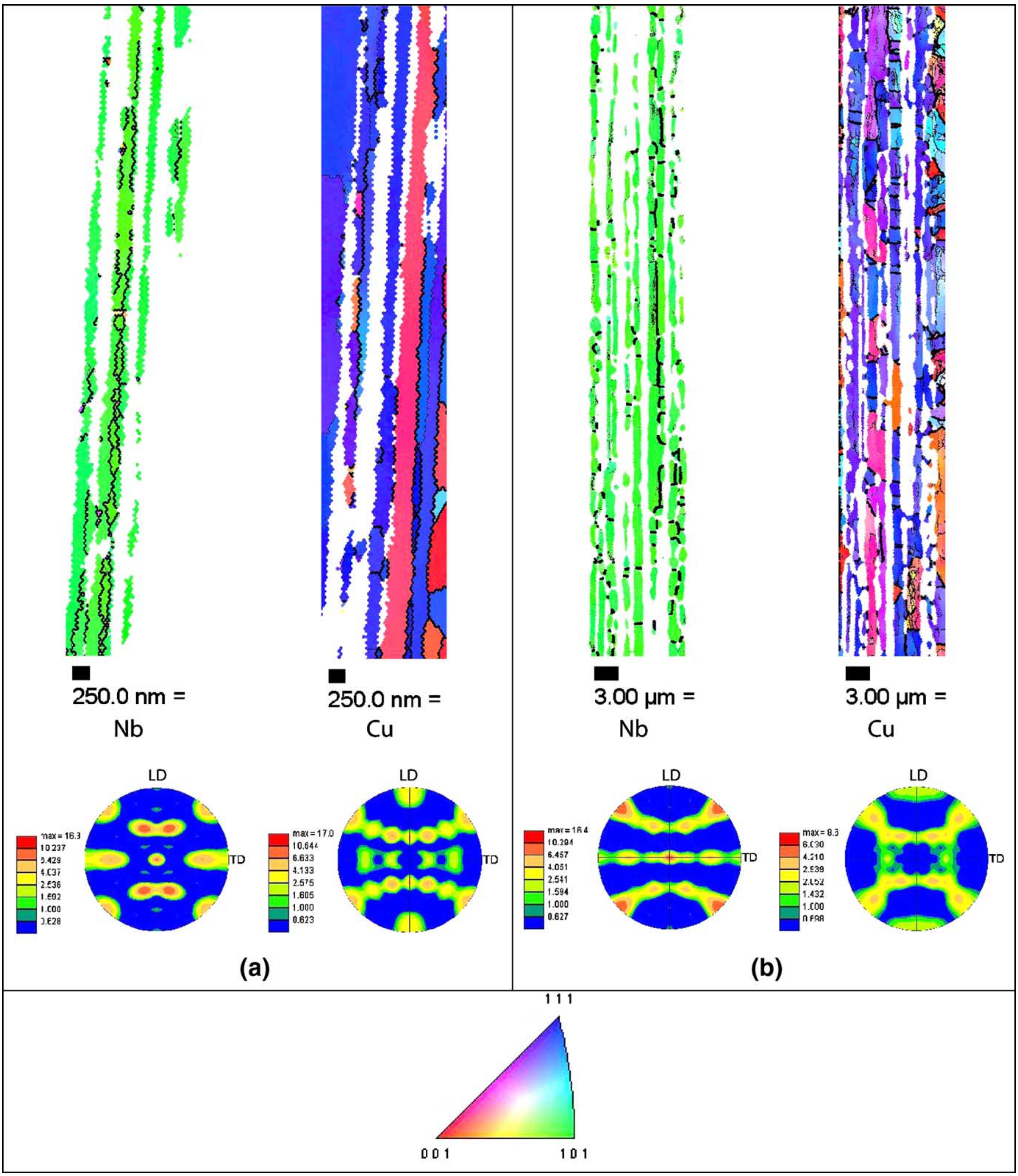

Fig. 3. OIMs and corresponding (1 0 0)-pole figures of the Cu–15%Nb composite annealed at: (a) 750 °C for 1 h; (b) 1050 °C for 8 h. OIMs refer to both Nb and Cu phases for each annealing condition. LD is parallel to the vertical in all maps. The color scheme reflects the orientation (see the inverse pole figure referred to LD).

More compelling evidences that the dominant shape instability mode for the investigated Cu–Nb system is boundary splitting are provided by high-resolution EBSD. Mappings were performed to determine the boundary characteristics in terms of misorientations (ψ) and the microtexture developed in niobium and copper. The partition of the EBSD data gives the individual orientation image maps (OIM) and textures corresponding to Nb and Cu phases. Fig. 3a depicts the OIMs of a composite annealed at 750 °C for 1 h. At this annealing temperature, a significant amount of recovery has occurred in niobium. Recovery causes a significant reduction of dislocation density easing the acquisition of Kikuchi patterns. From the OIM corresponding to Nb, it is evident that niobium ribbons are subdivided by elongated boundaries. These boundaries correspond to the former LBs found in the cold-worked state and are mostly found aligned parallel to the longitudinal direction of the filaments. Thicker black lines represent large angle boundaries and are found in majority in this map. The (1 0 0)-pole figure corresponding to the Nb phase clearly indicates a ⟨1 1 0⟩-fiber texture. The OIM of the Cu matrix shows elongated recrystallized grains. A significant amount of low-angle boundaries is present (thinner black lines). They correspond to subgrain boundaries commonly found in materials with sharp textures. Its (1 0 0)-pole figure reveals the presence of ⟨1 1 1⟩ and ⟨0 0 1⟩-texture components.

The OIMs corresponding to the composite annealed at 1050 °C for 8 h are shown in Fig. 3b. Contrarily to the features observed in Fig. 3a there is a clear predominance of transverse grain boundaries in the Nb phase. This structure is strongly ⟨1 1 0⟩-oriented as reveals its corresponding (1 0 0)-pole figure (Fig. 3b). Boundary splitting and further cylinderization have already occurred in large extent. Recrystallization is evident at this temperature as well as the development of the bamboo-like structure. This section shows individual points where cylinder breakup is clearly seen. A few isolated ‘‘spheres’’ can be distinguished in the OIM. High-angle boundaries predominate in the microstructure of the Nb phase. Two texture components are present in recrystallized copper: ⟨1 1 1⟩- and ⟨0 0 1⟩ ones, in a similar manner as shown in Fig. 3a.

4. Proposed model

Based on the results reported in the presented paper, the microstructural changes observed in a Cu–Nb multifilamentary composite can be interpreted as follows (Fig. 4). In the initial state, individual niobium ribbons are found in the copper matrix as shown schematically in Fig. 4a. An enlarged view of such a ribbon reveals the presence of elongated lamellar boundaries subdividing the microstructure (Fig. 4b). Shading schematically represents differences in terms of misorientation among the lamellae. Subgrain boundaries are formed either during plastic deformation as result of dynamic recovery or by static recovery during annealing. Transverse dashed lines represent subgrain boundaries. These morphological features remain nearly unchanged until 600 °C. At temperatures above 700 °C, fragmentation becomes noticeable firstly along LBs and at grain boundaries in regions where recrystallization has already occurred. Such fragmentation process, characterized by boundary splitting, is schematically illustrated in Fig. 4c. As the annealing proceeds, the already fragmented niobium ribbon undergoes cylinderization leading to the formation of an array of cylinders in parallel. Because of the Rayleigh instability process, each one of the individual cylinders becomes partially spheroidized. In addition to this partial breakup, new contact points join adjacent cylinders due to their coarsening, as shown in Fig. 4d. This simple model explains the changes in the morphology observed upon annealing of niobium ribbons.

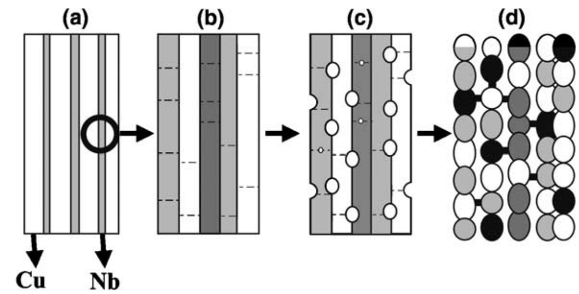

Fig. 4. Schematic model proposed to explain the fragmentation of the niobium ribbon-like structure in a Cu–Nb multifilamentary composite.

5. Conclusions

To summarize, the following conclusions can be drawn based on the microstructural changes observed upon annealing of a multifilamentary Cu–15%Nb composite:

(1) Boundary-assisted splitting becomes pronounced in temperatures above 700 °C and is mainly responsible for the fragmentation of the niobium ribbons. Further increase in temperature leads to the cylinderization of the freshly fragmented ribbons followed by spheroidization. Such a process leads to the breakup of the niobium cylinders in many regions. Concurrent coarsening hinders the occurrence of true spheroidization at higher annealing temperatures;
(2) High-resolution EBSD maps performed in specimens annealed at 750 °C show that the microstructure of niobium ribbons is predominantly

subdivided by elongated high-angle LBs ($\psi \geqslant 15°$) nearly parallel to the axial direction. With increasing annealing temperature (1050 °C) boundary distribution changes and transverse grain boundaries along the filaments become predominant, i.e., a bamboo-like structure evolves in the microstructure. These results are in good agreement with those obtained by metallographic inspection in SEM;

(3) In spite of the annealing temperature, a sharp ⟨1 1 0⟩-fiber texture is present in the fragmented niobium structure.

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