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Mechanical Behaviour of Copper 15% Volume Niobium Microcomposite Wires

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Cu-Nb microcomposites are attractive in magnet pulsed field technology applications due to their anomalous mechanism of mechanical strength and high electrical conductivity. In this sense, recently it was conceived the use of Cu 15% vol. Nb wires to operate as a high tensile strength cable for a diamond cutting tool (diamond wires) for marble and granite slabbing. The multifilamentary Cu 15% vol. Nb composite was obtained using a new processing route, starting with niobium bars bundled into copper tubes, without arc melting. Cold working techniques, such as swaging and wire drawing, combined with heat treatments such as sintering and annealing, and tube restacking were employed. The tensile property of the composite was measured as a function of the niobium filaments dimensions and morphology into the copper matrix, in the several processing steps. An ultimate tensile strength (UTS) of 960 MPa was obtained for an areal reduction (R = Ao/A, with Ao-initial cross section area, and A-final cross section area) of $4x10^8$ X, in which the niobium filaments reached thickness less than 20 nm. The anomalous mechanical strength increase is attributed to the fact that the niobium filaments acts as a barrier to copper dislocations.

Keywords: *Cu 15% vol. Nb, wire drawing, heat treatments, anomalous tensile strength*

1. Introduction

The Cu-Nb multifilamentary microcomposites are known due to their high ultimate tensile strength (UTS) combined with high electrical conductivity. Such properties justify the use of this class of material in superconductor wires for pulsed fields magnets. These composites usually contains less than 20 volume% niobium[1,2]. Due to its high UTS allied to its high ductility (true strain $\varepsilon > 2,0\%$), this composite was utilized in the present work as a high strength cable for the *in situ* diamond wires.

Diamond wires are cutting tools currently used in the slabbing of dimension stones such as marbles and granites, as well as in the cutting of concrete structures. This tool consists of a AISI 316 stainless steel cable containing spaced diamond annular segments (pearls)[3,4]. Filgueira[5] has developed a new route to process diamond wires (*In Situ* technology) in which the diamond crystal was impregnated along the overall wire surface (continuous diamond volume). As a substrate to these wires it was employed the composite Cu 15% vol. Nb. In addition, the understanding of the properties and behaviour of this class of advanced material (Cu-Nb microcomposites) is the main concern of this work.

The aim of this work is to study the mechanical behaviour of Cu 15% vol. Nb wires to operate as a high strength cable for diamond wires. In order to analyze the deformation effect of wire processing on the tensile strength, samples of several steps of wire drawing (various diameters) were prepared for the tensile tests.

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2. Literature Review

2.1. Fabrication routes

Figure 1 shows the routes used to process the Cu-Nb composites wires.

An example of the route to process Cu-Nb wires by arc melting was performed by Spitzig *et al.*[6] Cu ingots of ϕ = 6.10 mm in diameter containing 12-20% vol. Nb were prepared by arc melting. The ingots were then rod rolled to 1.3 mm and wire drawn to 0.15 mm in diameter, corresponding to an areal reduction of R = 1654 X. The Nb dendrites embedded into the copper matrix showed a diameter range of 1.0 to 10 µm after the arc melting[7]. Therefore, after mechanical conformation, the Nb dendrites deform following a preferential orientation, acquiring a "ribbon like shape" in submicrometrical dimensions, which is responsible for the high strength of the wire (for the Cu 20% vol. Nb, UTS = 2200 MPa). In this route is reached high purity of the ingots[6].

The PM route was developed by Pourrahimi[8]. Pure Cu and Nb powders were mixed at tap density and placed in Cu cans. After sealing, compaction and evacuation, the billets were processed by swaging, hydrostatic extrusion and wire drawing. Restacking of the Cu-Nb elements into the Cu tubes was used to achieve high areal reductions. The PM Cu 18% Nb composite reached the UTS = 1035MPa.

2.2. Mechanical behaviour

The majority of fabrication processes of copper-niobium wires, such as extrusion, wire drawing, swaging and rod-rolling, consists in the Nb filament thickness reduction, so they can attain dimensions in the order of 10 nm[9], where deformation reaches its saturation, because under these conditions the Nb filaments acco mmodate no further plastic deformation and rupture occurs. The areal reduction range necessary for the Nb filaments thichness to attain nanometric dimensions varies from $R = 10^3$ to 10^8 X[1,7]. The high tensile strength of the Cu-Nb composites is obtained due to the "ribbon like shape" microfilamentar structure of the niobium filaments, where the Nb microfilaments lock the interfacial dislocations originated in the copper matrix.

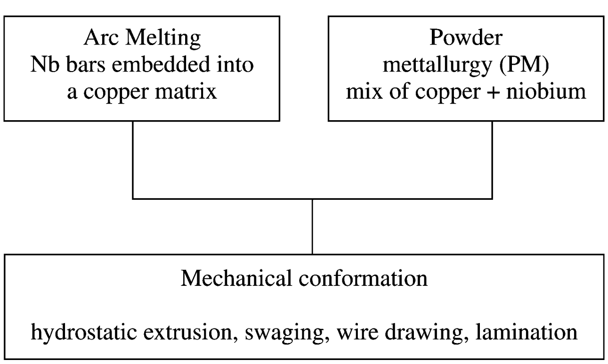

Figure 1. Fluxogram of the routes of copper-niobium wires fabrication.

Pourrahimi *et al.*[1] have found results of tensile strength of UTS = 1100 MPa for an areal reduction of $R = 5x10^6$ X, reaching UTS = 1600 MPa for $R = 5x10^7$ X . Thus, a reduction of one order of magnitude promotes a strengthening gain of 30%. This composite Cu 18 vol. % Nb was obtained by the PM route. Raabe[2] fabricated the Cu 20 vol. % Nb by the arc melting route, reaching an UTS = 2000 MPa. It confirms the Bevk *et al.*[7] results.

The amount of Nb in the composite varies from 10 to 20% vol., because this is the range in which the composite shows an anomalous tensile strength. The behaviour was theoretically explained in terms of the percolation phenomenon by Sher and Zallen[9]. The researchers cited above carried out statistical analysis on the atomic interconnectivity in several crystalline structure lattices, taking into account the universal model of hard spheres in the tridimensional space. They concluded that the critical density of percolation for CFC (Cu) and CCC(Nb) structures is approximately 15%. In other words, it means that at least 15% vol. Nb is required in the composite Cu-Nb to achieve interconnectivity between the Nb filaments in the composite. This is very desirable not only in the point of view of the composite homogeneity but also due to the CFC copper dislocations locking by the CCC niobium filaments.

3. Experimental

Figure 2 shows the Cu-Nb wires processing route.

The wires was manufactured by using Nb bars inserted into Cu tubes. Co mmercially pure Nb bars of ϕ = 6.10 mm in diameter were obtained by aluminotermy and electron-beam melting, and then hot forged. Copper tubes are co mmercially known as OFHC type. Table 1 shows the chemical analysis of both Cu and Nb. Results are in agreement with the ASTM[10] and ASTM[11] standards specifications.

In the 1^{st}, 2^{nd} and 3^{rd} restackings, the Cu ϕ_{ext} = 19,00 mm/ ϕ_{int} = 16.00 mm tube with 19 filaments was swaged (FENN 3F) to ϕ = 14.605 mm, to ensure contact between the filaments, then this billet was heat treated at 850 °C/1 h by using a vacuum (10^{-4} mmHg) furnace to promote sintering (interfilamentar cohesion) along the overall length of the bar. This heat treated bar was then swaged to ϕ = 7.925 mm and wire drawed to ϕ = 3.08 mm.

In the 4^{th} restacking 61 filaments of ϕ = 2.90 mm (from the 3^{rd} restacking) were bundled into a Cu ϕ_{ext} = 33.00 mm/ ϕ_{int} = 27.00 mm tube. Initial deformation was carried out by swaging to ϕ = 24.76 mm. The billet was heat treated at 850 °C/1 h under vacuum of 10^{-4} mmHg. This billet was swaged to ϕ = 7.925 mm, and then wire drawn until the required diameter. The stoichiometry of the composite was

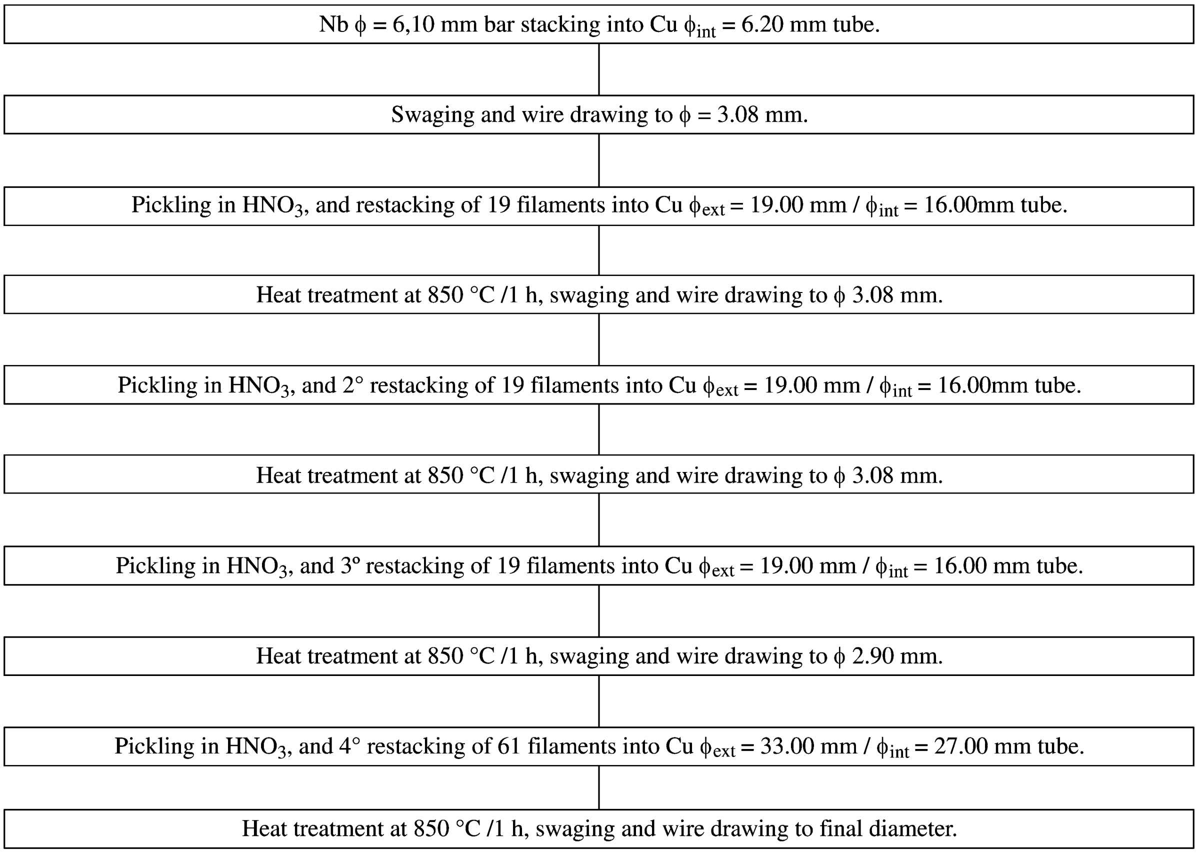

Figure 2. Processing route of Cu-Nb wires manufacture.

Table 1. Chemical composition of niobium and copper.

	Ta (%)	W (%)	Al (%)	Fe (%)	C (%)	N (%)	O (%)	S (%)
Niobium	0.11	0.02	–	0.005	0.02	0.01	0.012	–
Copper	NV	NV	NV	0.002	0.002	–	0.002	–

NV : not verified; –: absent.

determined via initial copper content control, as well as via pickling between each restacking, making use of gravimetry. The composite was manufactured in two stoichiometries: Cu 10% vol. Nb and Cu 15% vol. Nb.

The Cu-Nb composites wires were tensile tested in a Universal Testing Machine MTS-mod. 810.23M-capacity 250KN, under ASTM[12] standard specification. Samples for scanning electron microscopy (SEM ZEISS 912) were cold embedded in epoxy resin and then abraded (300-400-600-800-1200 mesh) and SiO_2 (mean particle size: 0.10 µm) polished. Samples for transmission electron microscopy (TEM ZEISS 900) were embedded in a epoxy resin, then cut in a Reichert Ultracuts ultramicroton, with a knife graduated to a degree of 10° and cut thickness of 60 nm, to ensure transparency to the electron beam. The cut pieces of the sample were collected in a 0.5% formvar-chloroform film, which was put into a 300 mesh grating.

4. Results and Discussion

The anomalous tensile strength behaviour occurs only when the Nb filaments attain a thickness of approximately t = 10 nm. Thus, its necessary a mechanical conformation that promotes areal reduction in range of R = 10^6 to 10^8 X. Figure 3 shows a transversal section of the Cu 15vol% Nb composite for an areal reduction of R = 1x27.10^7 X (after the 4^{th} restacking) and the corresponding wire diameter was of ϕ = 2.00 mm. The cells are homogeneously spaced without macro or micro defects. Each individual cell measures approximately 150 µm.

Figure 4 shows the mechanical behaviour of the composites Cu 15% vol. Nb, Cu 10% vol. Nb, and pure copper. As a matter of convenience, the areal reductions were taken in relation to the last heat treatment, which occurs after the 4^{th} restacking, for a billet diameter of ϕ = 24.76 mm. In this

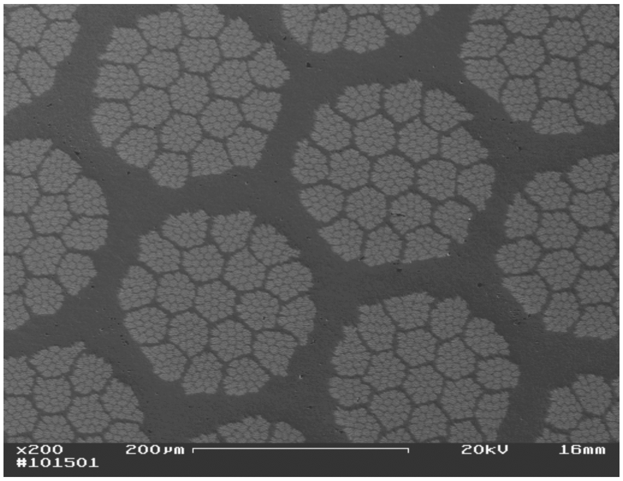

Figure 3. Cross section of the Cu15 vol.Nb. R = 1.27x10^7 X. Magnification: 200 X. MEV.

step the areal reduction is R = 8.5x10^4 X. Thus, to find the overall areal reduction, a factor of 8.5x10^4 must be multiplied by the values shown in the abscissa of the graph in Fig.4.

One can observe that the pure copper wire drawn reaches its tensile strength saturation level at the UTS = 500 MPa for the areal reduction of R = 1250 X. This result is in close approximation with the literature[1]. The composite Cu 10 vol% Nb has show a gain in UTS, and the wires broke down during wire drawing at UTS = 700 MPa for R = 2250 X, overcoming the pure copper in 200 MPa, corresponding to a strength gain of 30%. Nevertheless, this value is 30% below the UTS found by Bevk *et al.*[7] for the same stoichiometry. This is attributed to the fact that Bevk *et al.* obtained the composite Cu 10% vol. Nb by the arc melting route, ensuring Nb dendrites homogeneously spaced into the copper matrix. In this work was employed the bundling of Nb bars into Cu tubes, without further arc melting, due to the fact that it is easier to orient Nb dendrites

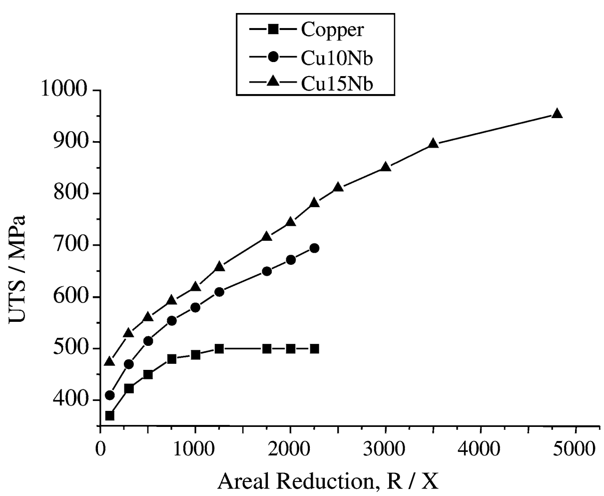

Figure 4. Ultimate tensile strength (UTS) versus areal reduction R, after the last heat treatment.

of ϕ = 10 µm in diameter than in Nb polycrystalline bars. The form is indispensable to the anomalous phenomena of the composite strength. Therefore, it should be noted that the arc melting introducts an increase in the final cost of the composite wires manufacture (equipment, energy, etc).

The Cu 15% vol. Nb wires broke down during wire drawing for a diameter of ϕ = 0,36 mm, showing UTS = 960 MPa, for an areal reduction of R = 4800 X (after the last heat treatment: $R_{overall}$ = 4,1.10^8 X). Comparing with the pure copper, it is observed that the Cu 15% vol. Nb is 2 times stronger, characterizing an anomalous strength behaviour (not predicted by the rule of mixtures). Therefore, the result of UTS = 960 MPa of the Cu 15% vol. Nb indicates that not all the Nb filaments reached the desired thickness. This is seen in Fig. 5. The arrows show the Nb filaments (darken parts) which thicknesses are in the range of t = 5-10 nm, explaining the UTS = 960 MPa. Furthermore, it can be observed that some filaments show thicknesses higher than t = 20 nm. This indicates that the strength of the Cu 15 vol.% Nb wire manufactured in this work had not reached its saturation (the graph of the Fig. 4 shows this tendency-the wires broke down due to fabrication, but the composite did not reached its saturation in deformation). Thus, higher UTS values may be reached if higher deformations are imposed to the composite, diminishing the Nb thicknesses to about t = 10 nm.

In the substructure it is observed the presence of interfacial dislocations, forming cells which are locked by the Nb filaments, characterizing (in Fig. 5) the brighter regions between the copper matrix (white areas) and the Nb filaments (dark areas).

Comparing the tensile strength of the Cu 15% vol. Nb composite with the Cu 10% vol. Nb, it is observed that an

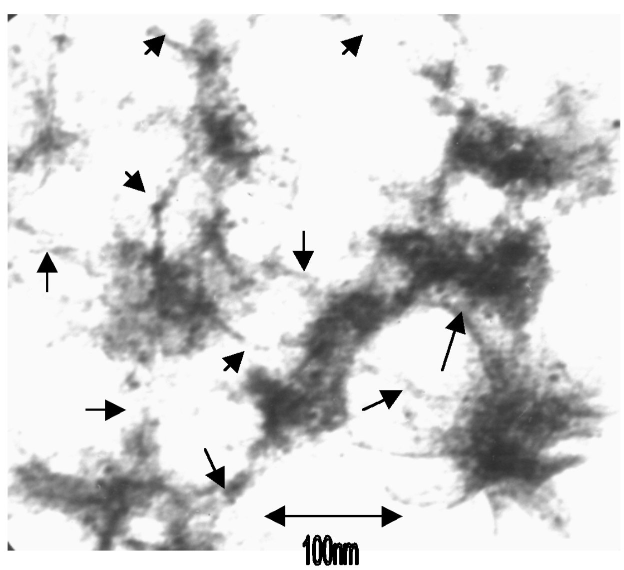

Figure 5. Nb filaments embedded into Cu matrix. R = 4.1x10^8 X. Magnification: 125 KX. MET.

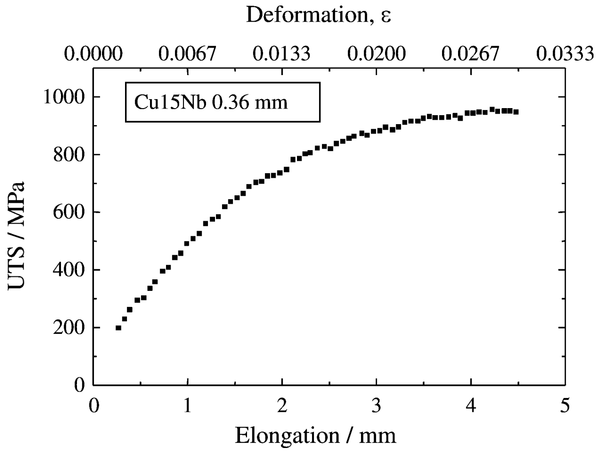

Figure 6. Stress versus strain curve for the Cu 15 vol.% Nb composite. Wire diameter of $\phi = 0.36$ mm.

increase of 5% in volume of Nb in the composite promotes a gain of 260 MPa. This result is of great relevance and it can be explained in terms of percolation critical density phenomenon; that accordingly to Sher and Zallen[9] it is necessary an amount of 15 vol.% Nb in the composite for an efficient Nb filaments interconnectivity, as to achieve a locking of the copper dislocations by the Nb filaments. It is worth to say that the result of UTS = 960 MPa obtained for the Cu 15% vol. Nb wires in this work, is in far agreement with Pourrahimi[8], who measured UTS = 1000 MPa for the PM Cu 15% vol. Nb composite laboratorial scale.

Figure 6 shows the tensile behaviour of the Cu 15% vol. Nb composite, reaching an UTS = 960 MPa. The yield stress measured at $\varepsilon = 0.002$ was $\sigma_y = 805$ MPa. It is worth to observe that the composite shows a deformation higher than $\varepsilon = 0.02$, indicating that its rigidity is in the same level of the AISI 316 stainless steel (used as the high strength cable for the co mmercial diamond wires).

4. Conclusions

Fabrication techniques employed in this work, such as swaging, wire drawing, heat treatments and tube restackings were successful to manufacture the Cu-Nb composites.

In the point of view of tensile strength, the Cu 15% vol. Nb (UTS = 960 MPa) composite wires overcome the AISI 316 stainless steel cable (UTS = 850 MPa[3]). Thus, this composite is technologically attractive to operate as a high strength cable for diamond wires. The same analysis may be done by comparing the strain before rupture. Both materials are in the same level: $\varepsilon > 0.02$.

Wire breakage occurred in the diameter of $\phi = 0,36$ mm due to the wire drawing system be inadequate to draw small diameters. As a result, deformation (strength) saturation did not occurred. Saturation would occur only when the majority of the Nb filaments attained a thickness of approximately t = 10 mm. TEM showed that only a part of Nb filaments reached this desired thickness value.

The results are in fair agreement with literature (1) and (8). Although the manufacture route are quite different, as discussed before.

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