Development and characterization of Nb$_3$Sn superconductor wire with nanometric-scale pinning centers

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Abstract. The optimization of flux line pinning in superconductors is one of the most efficient ways to improve the transport properties of these materials. The generation of pinning centers in a controlled way and with a projected distribution can contribute to the estimation of the pinning acting on the flux lines and to the improvement of the critical current densities $J_c$. The present work shows the development and characterization of a Nb$_3$Sn superconducting wire with nanometric-scale Cu(Sn) artificial pinning centers (APC) introduced in a controlled manner into the superconducting phase. These nanometric APC regions change the properties of the superconducting phase, mainly due to the proximity effect and to the inelastic scattering of the electrons on the interfaces. The final wire, with 1.064.514 Nb filaments, was heat treated to form the Nb$_3$Sn phase and the Cu(Sn) APC regions at different external diameters, enabling the determination of the flux pinning behavior with the variation of the APC dimensions, in comparison to the superconducting coherence length. The results were analyzed under the microscopic point of view determining the influence of the APC presence on the superconducting properties.

1. Introduction
The generation of projected microstructures to work as pinning centers in superconductors is a very efficient way to optimize the transport properties of these materials and to estimate the pinning forces and mechanisms acting on the flux line lattice aiming the determination of procedures to improve the critical current densities $J_c$. These projected microstructures, called Artificial Pinning Centers (APC), are introduced into the superconducting phase through successive steps of bundling and mechanical deformation. This APC technique tries to enhance the flux pinning (and the critical currents) through the introduction of normal phases inside the already existing superconducting phase [1]. The introduction of APCs enables the generation of structures comparable to the superconductor coherence length $\xi$ of the material. When the thickness of the APCs is comparable to $\xi$, the proximity effect induces superconductivity in these APCs and they are transformed into internal defects of the superconducting phases [2,3]. This produces highly efficient magnetic flux pinning and increases $J_c$.

The multifilamentary Cu-Nb$_3$Sn with APCs composites have been investigated in the last two decades due to their interesting pinning properties and superconducting characteristics. Several works described results on the improvement of the composite transport properties [4,5]. This composite can be obtained introducing APC Cu in a Nb matrix through successive bundlings, followed by mechanical deformation [6]. After the mechanical deformation of each bundling and reaction heat treatment in the presence of Sn, the regions of Cu(Sn) and Nb$_3$Sn form composites with different
superconducting properties, depending on the dimensions of these phases. By reducing the dimensions of the Cu(Sn) normal phase, the proximity effect starts to impose, and the superconducting phase of Nb₃Sn induces superconductivity on these Cu(Sn) regions.

The present work shows the development and characterization of a Nb₃Sn superconducting wire with nanometric-scale Cu(Sn) artificial pinning centers (APC) introduced in a controlled manner into the superconducting phase to enable the analysis of the flux pinning in this material. These nanometric APC regions change the properties of the superconducting phase, mainly due to the proximity effect and to the inelastic scattering of the electrons on the interfaces. These effects will act on the pinning characteristics of the new composite. The results can be analyzed under the microscopic point of view, determining the influence of the Cu(Sn) presence on the superconducting properties. This analysis leads to several important results useful to the optimization of $J_c$ in superconductors.

2. Experimental Procedure

The Cu APCs were introduced in a Nb matrix using 3 successive bundlings, followed by wire drawing. The initial material was prepared introducing one Nb rod with 15.8 mm of diameter inside a Cu OFHC tube with 19.05 mm of outer diameter, forming the monofilament that was deformed until 1.11 mm. After this step, 127 units of this monofilament were bundled in a Cu OFHC tube of 19.05 mm of outer diameter and the set was deformed down to 1.11 mm, to form the first bundling. This procedure was repeated once more and the last bundle was formed by 66 sets of the previous bundling together with 36 rods of Sn, used for the formation of the Nb₃Sn superconducting phase during heat treatment. The cryogenic and electrodynamics stabilization was obtained introducing a central pure Cu region surrounded by a Ta diffusion barrier. The third bundling was deformed until 0.7 mm in diameter, where the 1.064.514 Nb filaments and the Cu APCs had dimensions compared to the Nb₃Sn superconducting coherence length $\xi \approx 3.5$ nm.

Samples of each bundling were prepared for microstructural and low temperature characterization, aiming at the determination of the Cu(Sn) APCs influence on the superconducting and transport properties due to the proximity effect. The samples at the final diameters were heat treated to form the Nb₃Sn superconducting regions together with the Cu(Sn) APCs.

An equation was used to estimate the Nb filament dimension and the Cu APC thickness supposing uniform deformation of all materials. These numbers must be confirmed by Transmission Electron Microscopy (TEM) but the conductors will have dimensions close to those shown in Table 1, describing the expected average dimensions of the Nb filaments and of the Cu(Sn) APC regions for several final diameters of the superconductor. The wire drawing to different stages will lead to samples with different APC dimensions. It can be seen that the average thickness of the Cu(Sn) APCs are in the range between 20 and 40 nm, comparable to the superconductor coherence length.

Table 1. Approximated dimensions of the Nb filaments and of the Cu(Sn) APC regions depending on the final diameter of the superconductor wire.

| Final diameter of the superconductor (mm) | Average diameter of the Nb filaments (nm) | Average thickness of the Cu(Sn) APC regions (nm) |
|-----------------------------------------|------------------------------------------|-----------------------------------------------|
| 2.00                                    | 520                                      | 40                                            |
| 1.50                                    | 389                                      | 30                                            |
| 1.06                                    | 275                                      | 21                                            |

3. Results and Discussion

Figure 1 shows the microstructure generated after each step of mechanical deformation. One can see the very regular deformation for the first, second and third bundlings, showing the correct deformation procedure used in the work. The tape-like structures showed in Figure 1f are the Nb filaments after an areal deformation around $10^8$, while the dark regions around these filaments are the holes formed by the removal of the Cu APCs using nitric acid (for visualization).

Table 2 shows the values obtained for the critical temperature $T_c$, $\Delta T_c$, and the residual electrical resistivity ($\rho_{20K}$) for the multifilamentary samples at the final diameter of 1.06 mm, after the indicated
heat treatments. The measurements were performed using the four-probe method. The values of $T_c$ were obtained as the middle of the transition, calculated between the 10% and 90% of the transition height. The half-width of the superconductor-normal transition defined $\Delta T_c$. One can see that the transitions are very narrow for all samples. The samples presented very homogeneous Nb$_3$Sn superconducting phases, confirmed by the high $T_c$ values and the low $\Delta T_c$ values of the transitions.

The measurements of critical currents $I_c$ as a function of the applied magnetic field were performed at 4.2K, in liquid helium bath, using the four-probe method up to the maximum magnetic field of 17T. Figure 2 shows the values of the critical current densities $J_c$ measured at 4.2K for samples with final diameter of 1.06, 1.50 and 2.00 mm after a heat treatment of 220°C/100h+575°C/100h+670°C/36h. The values of $J_c$ were obtained dividing $I_c$ by the Nb filaments area present in each sample, resulting values close to the $J_c$ on the superconducting layer. This area was obtained using FEG-SEM analysis.

\begin{table}[h]
\centering
\begin{tabular}{|c|c|c|c|}
\hline
Heat treatment & $T_c$ (K) & $\Delta T_c$ (K) & $\rho_{20K}$ ($\mu\Omega \cdot \text{cm}$) \\
\hline
220°C/100h+575°C/100h+700°C/50h & 17.2 & 0.2 & 33 \\
220°C/100h+575°C/100h+700°C/100h & 17.0 & 0.3 & 60 \\
220°C/100h+575°C/100h+700°C/150h & 16.9 & 0.2 & 22 \\
220°C/100h+575°C/100h+700°C/200h & 17.2 & 0.4 & 19 \\
\hline
\end{tabular}
\caption{Critical temperature $T_c$, $\Delta T_c$, and residual electrical resistivity ($\rho_{20K}$) for each sample.}
\end{table}

It can be noted the influence of the Nb filaments and Cu(Sn) APCs dimensions on the $J_c$ behavior. The values of $J_c$ increase up to 12T when the wire diameter decreases, or decrease the Nb and APCs dimensions, as a clear indication of the pinning size influence on the transport properties. Once the wire diameter decreases, the APCs dimensions (pinning size) decrease well below the efficient size needed to optimize the pinning for higher magnetic field. The superconducting phase actually acts as a dirty Nb$_3$Sn+APC phase by the proximity effect, where the Cu(Sn) is playing more important role as the wire diameter decreases. At higher magnetic field, the induced superconductivity by the proximity effect is destroyed and the samples have almost the same behavior. Figure 2 suggests that $B_{c2}$ is depressed for these APC samples. The estimated $B_{c2}$ value around 18T is well below the values found
for regular Nb,Sn samples (above 24T). This is another clear indication that the proximity effect is playing an important role in this new kind of composite superconductor [2].

In addition, when the wire diameter decreases, the composite Nb+APC decreases in area, but probably the APCs are using more and more relative area because their higher hardness compared to the Nb filaments at the final high areal deformation (~10¹⁰). This explanation must be confirmed by TEM analysis in the future. It must be noted that not just the Cu APC thickness is decreasing with the wire diameter, but also the homogeneity and diffusion distances (Fig. 1).

![Graph](image)

**Figure 2.** Critical current density (based on the original Nb cross-section) versus applied magnetic field, measured at 4.2K after heat treatment of 220°C/100h+575°C/100h+670°C/36h.

4. Conclusion

The fabrication method used in this study efficiently generated the Cu(Sn) artificial pinning centers with nanometric dimensions. The measurements of \( T_c \) and electrical resistivities demonstrated the quality of the superconducting samples. It could be seen that the progressive reduction of the Cu(Sn) regions in the samples changed the superconducting phase. It is also important to notice the clear influence of the Cu(Sn) regions on the transport properties, due to the influence of the proximity effect on the nanometric structures. The transport measurements enabled the analysis of the pinning behavior of the samples. The presence of Cu(Sn) modified substantially their superconducting properties, changed their \( J_c \) values, and probably their upper critical field \( B_{c2} \) [7].

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