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MANUFACTURE AND EVALUATION OF Nb_3Sn CONDUCTORS FABRICATED BY THE MJR METHOD

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Abstract

The bronze matrix/niobium filament process has become established as a commercially viable method for producing multifilamentary Nb_3Sn superconductors. This paper describes a new method, the Modified Jelly-Roll (MJR) approach, which can produce a structure similar to that in a conventionally fabricated multifilamentary Nb_3Sn conductor. This approach utilizes alternate sheets of niobium expanded metal and bronze, which are rolled into a "jelly-roll" configuration and then extruded. During extrusion and subsequent drawing, the junctures in the niobium are elongated and the material develops a filamentary structure. This method may offer significant advantages in terms of reduced fabrication time and cost over the conventional approach. Results of a manufacturing development program will be presented in which two lengths of conductor were made to High-Field Test Facility conductor specifications. In addition, critical current and transition temperature measurements of the sub-elements used to construct the HFTF-type lengths will be reported.

Introduction

The MJR approach[1] for fabrication of bronze matrix Nb_3Sn superconductors is extremely versatile with respect to changes in the configuration and properties of the composite. The bronze to Nb ratio can be varied simply by changing the thickness or number of wraps of bronze sheet used in the jelly roll. The final filament size and number can be controlled by the thickness and the number of wraps of expanded Nb metal sheets. The effects of impurities or intentional ternary additions on the mechanical properties of the Nb or bronze can be assessed as the material is being reduced to sheet. In the event of processing problems, this material is easily recycled, since it has not yet been combined in a composite. To date, the MJR process has been utilized to produce over 70 experimental billets, in which the parameters such as bronze to Nb ratio, filament size and spacing, and the effects of ternary additions have been studied.

This paper describes a two-part program aimed at understanding and optimizing the MJR process. In the first stage, a number of small billets were extruded and drawn to 0.9-mm wires, which were evaluated. In the next stage, a number of first-stage billets (with fixed parameters) were used to construct a second-stage extrusion billet. The parameters of this second stage extrusion billet were chosen to meet the HFTF conductor specifications,[2] since we have a substantial data base for conventional bronze-process material made to that specification.

Experimental Procedure

The bronze used in these experiments was nominal Cu 13.5 wt. % Sn material, vacuum melted and cast in the form of rectangular strips. The actual composition of the bronze varied from 10.4 wt. % Sn to 14.4 wt. % Sn; the composition was varied intentionally in order to investigate its effect on processing. These strips were homogenized and then hot rolled to a thickness of approximately 0.4 mm. The filament material consisted of two compositions--pure Nb and Nb-0.8 wt. % Ti. This material was rolled into sheets approximately 0.3 mm thick and expanded in order to produce the metal mesh. The first-stage billets were prepared by wrapping a sheet of expanded Nb or Nb alloy and a sheet of bronze around a core rod in a spiral, or "jelly-roll," configuration. A diffusion barrier was incorporated by interleaving a Ta sheet between the final two wraps of Nb expanded metal. This package was loaded into a copper can, after which the can was evacuated and sealed in preparation for extrusion. The billets, 80 mm in diameter and 200 mm long, were extruded in a conventional hot extrusion operation to 25-mm diameter rods. Subsequently, the material was cold drawn to 0.9-mm-diameter wire with intermediate anneals of the bronze. Continuous annealing, i.e. moving the rod through the furnace on a conveyor belt, was used for straight rods between 25 mm and 10 mm in diameter. This operation resulted in a minimum of time-at-temperature accumulated in the anneals, and subsequent analyses showed no appreciable formation of Nb_3Sn. High sensitivity (inductive) T_c measurements yielded T_c values between 10.1 and 10.8 K for the samples analyzed. These results and the technique are more fully discussed in Ref. 3. The cross section for a typical length of material is shown in Fig. 1.

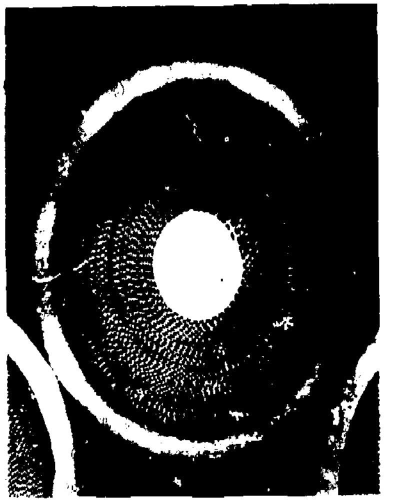

Fig. 1a. Micrograph of a typical MJR conductor cross section. Proceeding out from center, the components are bronze core, bronze and Nb expanded metal spiral wrap, Nb and Ta diffusion barrier, and Cu.

Samples from these wires were wrapped around a mandrel and heat treated for various times at 700 °C. Long sample, critical current measurements were performed to fields as high as 18.5 T at the Francis Bitter National Magnet Laboratory, with a sensitivity of 10^{-12} Ω · cm. It should be noted that high-sensitivity J_c measurements such as these are typically 10-20% lower than J_c values determined by hairpin-type samples of Nb_3Sn. Metallography and analyses by Scanning Auger Microprobe (SAM) were performed on the bronze matrix and the Nb_3Sn reaction layers.

In the second phase of this project, three first-stage billets (M74, M78, and M79) were prepared with the same nominal composition (see Table I). These billets were processed following the procedure already described, and part of the material was drawn into 0.9-mm wire. The remaining material was formed into hexagonal rods (19-mm flat to flat) which were cut into lengths and assembled into a second extrusion billet, 170 mm in diameter. The cross section consisted of one pure copper rod at the center with 36

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Fig. 1b. Enlarged micrograph showing outer filaments and diffusion barrier. Dark layer in diffusion barrier is Ta. Filaments maintain the rectangular cross section of the starting material throughout processing.

TABLE I - First Stage Exclusion Billet Parameters

Designation	Filament Composition	Bronze:Nb Ratio	Overall Bronze Composition (wt. % Sn)
M57	Nb-0.8 wt. % Ti	3.45:1	13.0
M61	Nb-0.8 wt. % Ti	2.5:1	13.2
M62	Nb-0.8 wt. % Ti	2.3:1	13.0
M66	Nb-0.8 wt. % Ti	2.96:1	13.4
M68L	Nb	2.96:1	13.5
M74	Nb	2.79:1	13.2
M78	Nb	2.83:1	14.1
M79	Nb	2.81:1	13.8

composite rods arranged around this central rod. This bundle was loaded into a copper can, evacuated, and sealed in preparation for extrusion. The billet was heated for 30 minutes at 450 °C and then 8 minutes at 650 °C, followed by extrusion to 38-mm diameter. This rod was processed to a final cross section of 5.4 mm x 11.0 mm and a length of 100 m, i.e. to the HFTF specifications. A cross section at an intermediate stage of processing is shown in Fig. 2. This final composite is 64% Cu, again to HFTF specification.

Results and Discussion

Critical Current

Critical current as a function of field to 18.5 T has been determined for 0.9-mm diameter wires of the first five samples listed in Table I. The wires were wound as coil samples on a mandrel and heat treated for 48, 96, and 168 hours at 700 °C. The changes in J_c (bronze and Nb_3Sn, excluding diffusion barriers) showed no consistent trend as a function of heat-treatment time. There is a tendency for J_c at high fields to increase with annealing time, and the

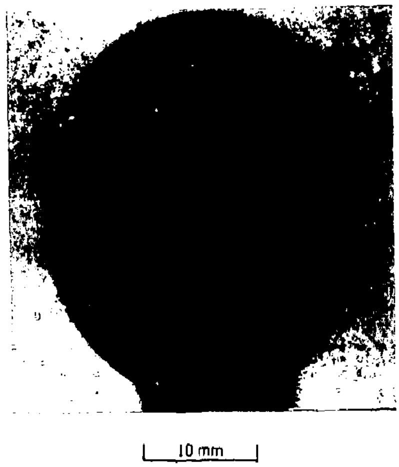

Fig. 2. Micrograph of HFTF-type conductor cross section after extrusion and drawing.

tendency is consistent with the reduction of the Sn gradient across the Nb_3Sn layers (discussed in analysis section). However, the largest change in J_c at high fields is produced by the Ti additions, as shown in Fig. 3. These samples were chosen for comparison because the other parameters, e.g. bronze to Nb ratio and bronze composition, were nearly identical (see Table I). At low fields, the sample with pure Nb produced higher J_c values. However, at higher fields, the samples with Nb-0.8 wt. % Ti produced much higher J_c values, e.g. a factor of 4 improvement at 18 T. A significant factor in this improvement is the increased upper critical

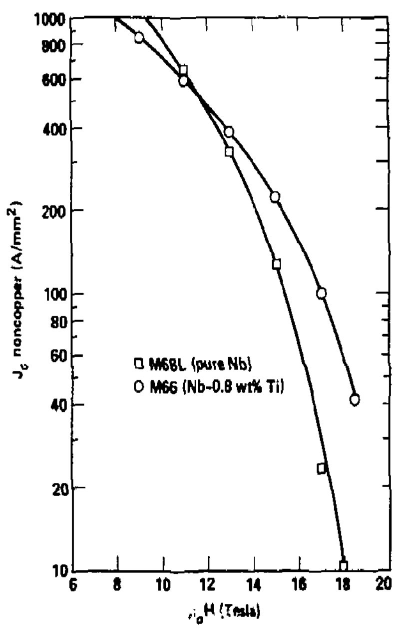

Fig. 3. Critical current density (in non-copper area) for a typical sample containing Ti additions compared with a typical sample with pure Nb cores. The enhancement in J_c at higher fields with Ti additions is quite significant.

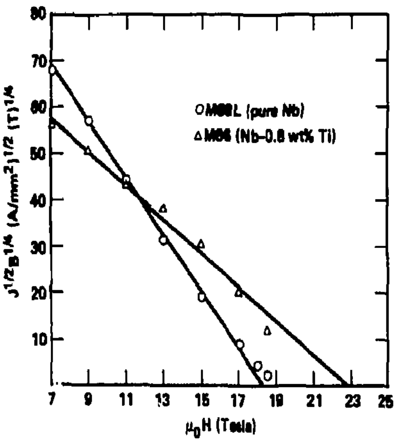

Fig. 4. A plot of $J^{1/2}$ $H^{1/4}$ vs. μH, which provides an estimate of H_{c2} by extrapolation to $J^{1/2}$ $H^{1/4}$ = 0, after Kramer.[5]

field of the sample with Ti additions. Figure 4 is a plot of $J_c^{1/2}$ $B^{1/4}$ vs. $\mu_0 H$, which allows an extrapolation to H_{c2}. The value of H_{c2} for the Ti-addition material is 23 T, whereas the value for the pure Nb sample is 19 T. Similar enhancements in H_{c2} have been observed previously for additions of transition elements to Nb_3Sn (see the review by Suenaga[4] for a discussion of alloy additions). The increase in H_{c2} is believed to be caused by the increase in the normal-state resistivity, ρ_n. However, the decrease in J_c at lower fields may be due to the same effect, i.e. the increase in ρ_n. The increase in ρ_n may decrease the effectiveness of flux pinning in the regime where $\Delta\kappa$ pinning is important. A more comprehensive study is necessary to complete our understanding of these pinning effects.

The values of J_c obtained for this MJR-processed material are comparable to the best values obtained for material processed by the conventional bronze route. In Fig. 5, we compare the J_c for a sample without Ti additions with J_c for conventional bronze samples.[6,7] The critical

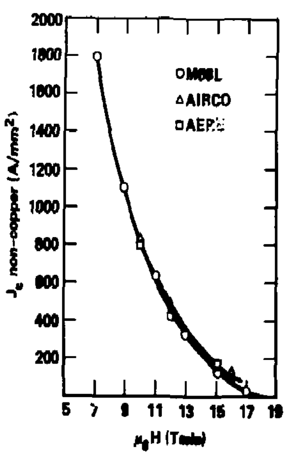

Fig. 5. Comparison of J_c values for MJR-process material with several results for optimized, conventional, bronze-process material. Filament cores in all cases are pure Nb.

currents for all three samples are comparable; if the MJR-process sample with Ti additions had been included, it would be clearly superior at fields above 13 T. The results for the best MJR sample (M62) have not been plotted, because it was made with a low bronze to Nb ratio that made a direct comparison difficult. This sample produced critical current values of 260 A/mm^2 at 15 T and 130 A/mm^2 at 17 T, which are comparable to the best values reported for conventional bronze-process material with various alloy additions.[8]

Analytical Results

Analyses were obtained for both the bronze and Nb-Nb_3Sn components as a function of heat treatment. In addition, sensitive T_c measurements were utilized in order to check for pre-reaction formation of Nb_3Sn. No appreciable formation of Nb_3Sn was detected for these samples, and these results confirm that bronze and Nb can be co-processed successfully through the extrusion and intermediate annealing steps if care is taken to minimize the time at temperature.

The SAM profiles of the bronze matrix for a sample of M68L after various heat treatments are shown in Fig. 6. These profiles are typical for the MJR process material and indicate that a substantial matrix Sn concentration gradient exists between the matrix surrounding the inner filaments and that surrounding the outer filaments.

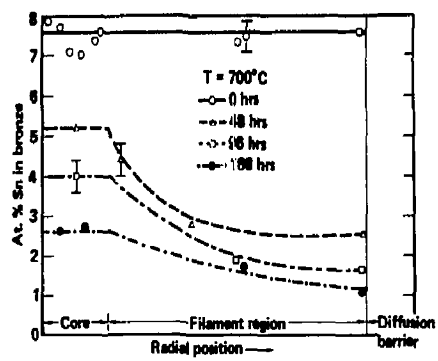

Fig. 6. Scanning Auger Microprobe results showing the evolution of Sn profiles in the bronze matrix following various heat treatments.

SAM profiles also were obtained for Nb_3Sn filaments located at various positions in the composites. Gradients in Sn concentration across the Nb_3Sn layers were observed, and the steepness of the gradient depended on the position of the filament. Filaments located near the bronze core showed rather uniform Sn profiles, especially for the long reaction times which resulted in completely reacted filaments. However, most filaments near the diffusion barrier showed steep Sn profiles, especially if the filaments were only partially reacted. If the maximum Sn concentration in the filaments is assumed to be 25 at. %, the value near the center (for filaments located near the barrier) is typically about 20 at. %. The significance of these results and comparison with results on conventional bronze-process material is discussed in Ref. 9.

These analytical results suggest that the critical current can be increased (beyond the comparatively good value already obtained) by changing the composite configuration. These changes are easily accomplished for the MJR process, i.e. the bronze core can be made smaller and additional bronze sheets can be added near the outer diameter of the spiral wrap. Experiments are planned to evaluate these changes in configuration.

HFTF Size Conductor

The HFTF-type conductor fabricated with pure Nb core is nearing completion, and assembly of an additional second stage billet incorporating Nb-0.8 wt. % Ti expanded metal is in progress. After fabrication is complete, J_c and J_c as a function of strain will be measured for these samples and the results reported at a later date.

Summary

The MJR process for fabricating Nb_3Sn has been evaluated with regard to superconducting properties and adaptability to large scale processing. Critical current values comparable to those obtained for conventional bronze process material were observed for pure Nb cores. Excellent critical current values at high fields were obtained with Nb-0.8 wt. % Ti cores. Capability for producing complex, large cross-section conductors was demonstrated by fabricating a 100-m length to HFTF specifications.

Acknowledgments

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