Development of High Performance Nb-Ti(Fe) Multifilamentary Superconductor for the LHC Insertion Quadrupoles

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Abstract—**A development program was initiated in order to develop strand with improved current density at 10.5 T and 1.9 K over existing SSCL designs. The two successful strand designs reported on here both utilized high Fe content Nb-47 wt%Ti alloys to improve the critical current density at high field by 7 %. At 10.5 T and 1.9 K, critical current densities exceeding 1450 A/mm² were obtained. In this paper we report detailed quantification of the macro- and micro-structures of these strands and correlate these with critical current density measurements at 1.9 K and 4.2 K. The high Fe content significantly reduced the α-Ti precipitate size. The linear relationship between critical current density and precipitate volume found is in agreement with earlier studies. High resolution FESEM electron backscatter contrast suggests a thin layer of high atomic number at grain boundaries.**

I. Introduction

To get sufficiently high field gradients for the interaction region quadrupoles of LHC requires a very compact magnet design operating at the highest current densities possible. This translates in practice into the choice of a Nb-Ti-(Ta) conductor operating at 1.8-1.9 K. Such conductor is strong and ductile and the fabrication techniques are well established.[1] A key issue for the present designs of low beta quads is how to make a conductor with the very best properties at 8-12 T and 1.9K. At high field and low temperature there is relatively little data and no systematic studies have been made to optimize the critical current density, J_c. A program was thus initiated by Fermi National Accelerator Laboratory in order to develop an understanding of the behavior and potential of Nb-Ti based strand at high field and low temperature.

Proposals were invited to develop Nb-Ti based strand with improved critical current, I_c, at 10.5 T and 1.9 K over existing SSCL designs using full-scale billets. Two routes were pursued, one using Ta alloying to increase the upper critical field, H_{c2}, and the other using Fe doping to improve the pinning efficiency by modifying the microstructure. The Ta alloying work is reported on elsewhere [2],[3]. Two manufacturers pursued the Fe doping approach and the results of this part of the program are reported here.

The SSC alloy specification for Nb-Ti strand limited the Fe content to a maximum of 200 ppm Fe [4]. However, many sources of Ti have inherently higher Fe contents. In fact, a small increase in the Fe content to 600 ppm was shown to improve the J_c of Nb-Ti without significantly increasing the hardness of the Nb-Ti [5]. The α-Ti precipitates produced after multiple precipitation heat treatments were smaller in size and significantly more homogeneous in size and distribution than in low-Fe Nb-Ti strand processed in the same way [5].

II. Experimental Procedure

The strand manufacturers were required to supply samples to the University of Wisconsin-Madison Applied Superconductivity Center throughout processing following the successful procedure of the SSC Phase II Research and Development program [6]. The samples supplied were fully analyzed by the UW by light microscopy, high resolution field emission scanning electron microscopy (FESEM), and energy dispersive x-ray analysis (EDS). Heat treatment optimization was carried out at the UW and by the strand manufacturers with feedback provided to the manufacturers by the UW. Testing was performed at 1.9 K and 4.2 K by the UW.

A. Strand Design

Although the strand diameter was based on the SSC inner specifications, 0.808 ± 0.0025 mm, the manufacturers were given more flexibility on filament diameter with filament diameters up to 10 μm allowed. The Cu: Supercon-

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*Also the Department of Materials Science and Engineering.

TABLE 1
ALLOY COMPOSITIONS

Alloy Identification	Alloy Composition		
	Ti, wt.%	Fe, ppm	O, ppm
596468 (OI-ST)	46	490-570	460-510
596867 (IGC-AS)	47-48	520-600	480-500

ductor ratio was 1.30 ± 0.1:1. Both manufacturers chose to use large filament diameters and Nb diffusion barriers around each filament that represented approximately 4% of the non-Cu cross-section.

B. Alloy Composition

Each manufacturer used Nb-Ti from a different alloy billet, both supplied by (Teledyne) Oremet-Wah Chang. The two alloy compositions used are listed in Table 1.

C. Strand Optimization

An extensive range of heat treatments was applied to multifilamentary composite material (which was supplied to the UW at 25 mm diameter) and some monofilamentary material was also re-stacked into 61 filament composites at the UW. The effective true strain in the OI-ST and IGC-AS composites at 25 mm diameter was estimated, by hardness measurement, at 5.8-6 compared to the measured increase in hardness with cold work of an annealed comparison strand. This strain should be sufficient to produce homogeneous precipitation [7] and so the initial heat treatments were applied at this size. The heat treatment schedules and naming conventions are shown in Fig. 1.

D. Microstructural Evaluation

The initial alloy micro-homogeneity was assessed by the application of a composition-sensitive etch [8] to cross-sections prepared by standard metallographic techniques. The use of a composition-sensitive etch allows for a qualitative assessment of homogeneity of the full ingot cross-section and the selection of representative regions for quantitative analysis by EDS. Hardness testing was performed throughout the processing of the strand in order to monitor the strain condition [9]. The microstructures of the strands were imaged using a LEO 982 high resolution FESEM in electron backscatter mode using a K. E. Developments Ltd. solid state detector. This system is capable of sub-4nm resolution and is well suited to viewing and quantifying the microstructure before it is reduced in size by final wire drawing. Images from the FESEM were quantified using Sigmascan Pro software by Jandel.

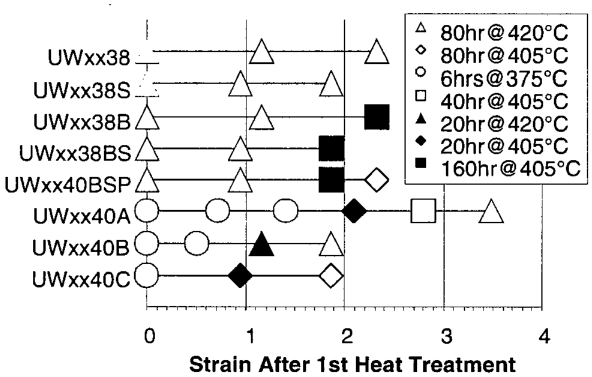

Fig. 1. Schematic diagram illustrating the UW heat treatment schedules.

E. Critical Current Measurement

Strands for critical current testing were mounted on 25.4 mm diameter stainless steel mandrels, with the magnetic field perpendicular to the wire axis. Samples were tested in superfluid helium at 1.9 K ± 30 mK. Temperature control was achieved using a manometer calibrated with a cernox temperature sensor. The voltage tap spacing was 22.5 cm and a 10^{-14} Ω-m criterion was applied across the entire wire cross-section to determine I_c. J_c was calculated using the superconducting cross-sectional area. The error in magnetic field homogeneity was less than 1 %. 4.2 K testing on the same strands was performed at atmospheric pressure with a temperature variation of less than 10 mK. Additional samples tested during initial optimization experiments at 4.2 K were mounted on standard 35 mm diameter stainless steel mandrels. The critical current densities for the Nb-Ti(Fe) strands were compared with three representative SSC production inner strands.

III. RESULTS

A. Alloys

The chemical homogeneity of the alloys was very good. No freckling was observed in the as-received cross-sections and there were uniform grain and coring substructures. The measured local Ti composition in the Nb-46 wt.%Ti alloy varied by ± 1wt.%Ti.

B. Critical Current Density

Initial studies indicated that the increase in field, at constant J_c, obtainable by reducing the temperature from 4.2 K to 1.9 K, was 2.8-3 T. This consistent shift allowed for an optimization survey at 4.2 K, which is faster and lowers the cost of testing considerably. The results of this survey for the Nb-47 wt.% Ti(Fe) strand processed at the UW from the 25 mm diameter production billet are shown in Fig. 2. The best performance in the range 5-8 T was for a strand which had been given three aggressive heat treatments (2 × 80 hr at 420 °C + 1 × 160 hr at 405 °C) with a strain space of 4 standard die spacings ($\Delta\varepsilon_t$ = 0.95) between heat treatments. The J_c at 5 T (4.2 K) was 3300 A/mm², which is the highest

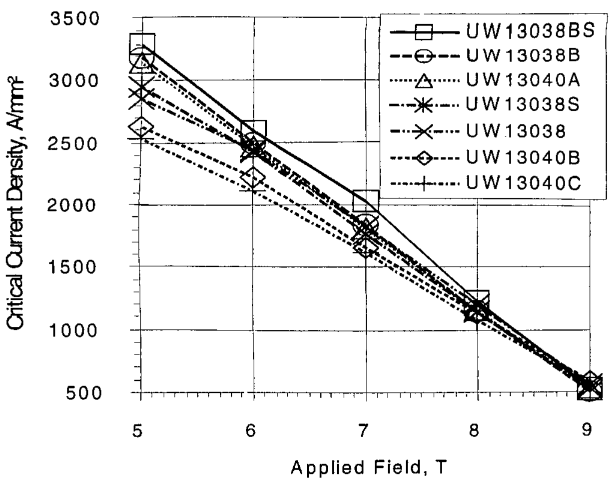

Fig. 2. Optimization of Nb-47wt.%Ti strand at 4.2 K (peak J_c). The best performance from 5-8 T was for a schedule of 2 × 80 hr at 420 °C + 1 × 160 hr at 405 °C with a reduced strain space ($\Delta\varepsilon_t$ = 0.95) between heat treatments.

we have seen for a full scale production composite that has been through a warm extrusion. The high Fe material did not benefit from the restricted heat treatments designed to reduce precipitate size (e.g. series UW xxx40A-C).

Assuming a 3 T shift, with cooling from 4.2 K to 1.9 K, the 10.5 T, 1.9 K performance can be estimated from the 7.5 T (4.2 K) measurements. Fig. 3 shows the extrapolated 7.5 T (4.2 K) J_c as a function of final drawing strain. At 7.5 T (4.2 K) the aggressive 2 × 80 hr at 420 °C + 1 × 160 hr at 405 °C heat treatment performs the best, but requires a high final drawing strain (5.1) to reach peak J_c(~1540 A/mm²). The standard 3 × 80 hr at 420 °C heat treatment series achieves an estimated value of 1510 A/mm² at 7.5 T with a final drawing strain to peak J_c of only 4.6.

Testing at 1.9 K revealed some differences from the expected 4.2 K extrapolations. The aggressive heat treatment of Nb-47wt.%Ti(Fe) did not produce the highest performance

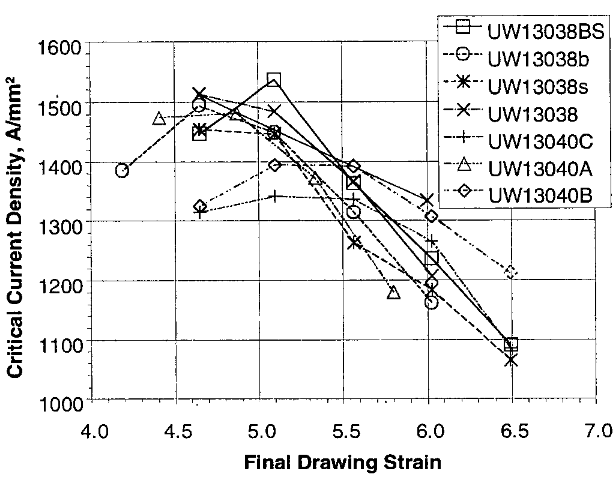

Fig. 3 The 7.5 T (4.2K) J_c (extrapolated from 7 and 8 T values) as a function of final drawing strain. The 7.5 T (4.2 K) J_c is an estimate of the 10.5 T (1.8 K) J_c, assuming a 3 T shift.

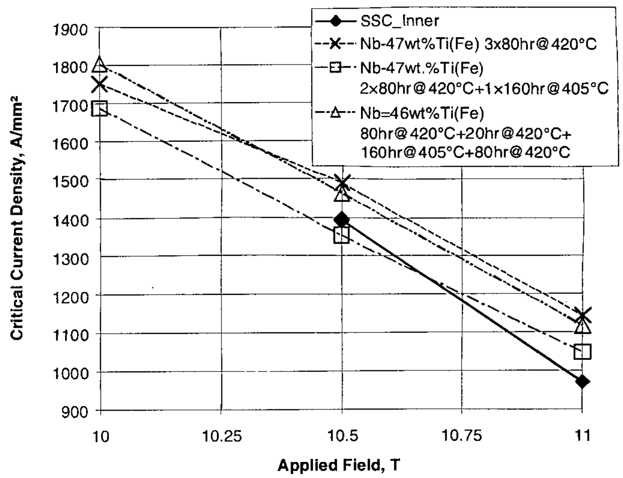

Fig. 4. A comparison of 1.9 K critical current densities for 10-11 T, for a production SSC Inner strand and three UW processed Nb-Ti(Fe) strands.

(Fig. 4). The best J_c at 1.9 K, 10.5 T was achieved by 3 × 80 hr at 420 °C heat treatment of the Nb-47wt.%Ti(Fe) strand.

Both manufacturers produced excellent strand that exceeded the non-Cu J_c of the SSC-Inner comparison strands by 7 % (1.9 K, 10.5 T), for which the J_c obtained at 1.9 K and 4.2 K was 1420 A/mm². In Fig. 5 the best values from this study are compared with the highest known values for Nb-Ti based strand. The increased Fe in the Nb-Ti binary alloy results in production scale strand that approaches the performance of the best laboratory scale strand at 1.9 K.

C. Microstructures

Precipitation heat treatment of the high Fe Nb-Ti strands produced log-normal distributions of precipitates (log mean diameter 59-97 nm and log +1std. deviation of 105-147 nm). The measured precipitate diameters, d*, are obtained from transverse cross-sectional areas, assuming a circular cross-section (the precipitates are elongated in the longitudinal direction with ~10-20:1 aspect ratio). Comparing the d* values

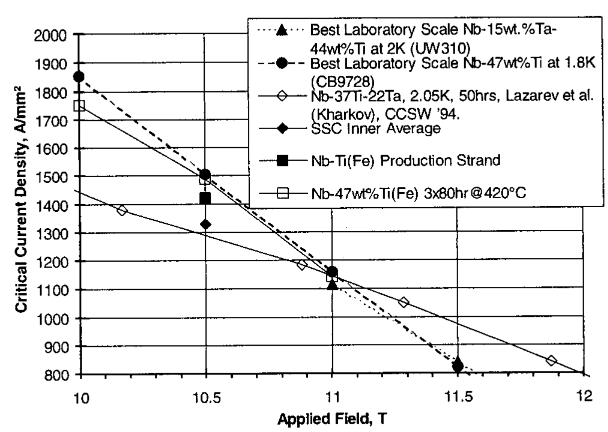

Fig. 5 A comparison of the results of this study with other high J_c Nb-Ti based strands at 1.9-2 K.

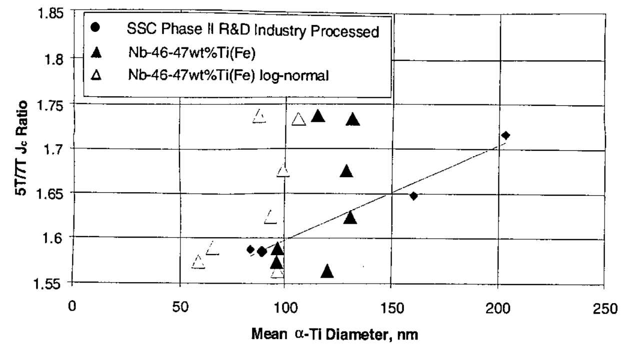

Fig. 6 5 T/7 T (4.2 K) J_c compared with measured precipitate diameters for Nb-Ti(Fe) strands in this study and a previously reported trend for SSC R&D strand [6].

for the Nb-Ti(Fe) and a Nb-47 wt.%Ti alloy both given three heat treatments of 80 hr at 420 °C the log-normal mean d* for the Nb-Ti(Fe) was 90 nm (+1 stds =140 nm) compared with 120 nm (+1 stds = 150 nm) for the Nb-47 wt.%Ti. In Fig. 6 the precipitate diameters from this study are compared with a previously reported trend in increasing 5 T/7 T (4.2 K) J_c. The Nb-Ti(Fe) strands in this study have uniformly small precipitates but a large scatter in the 5 T/7 T(4.2 K) J_c ratio.

The relationship between the quantity of α-Ti and J_c is similar to that previously reported for both Nb-47wt.%Ti and Nb-50wt.%Ti as shown in Fig. 7.

The high resolution FESEM backscattered electron images indicated regions of higher atomic number at the grain boundaries after heat treatment. The regions ranged in thickness from approximately 15-60 nm.

IV. Summary

1. A significant (7 %) improvement in critical current density at 10.5 T, 1.9 K has been achieved in production scale strand by increasing the Fe content to 500 ppm.

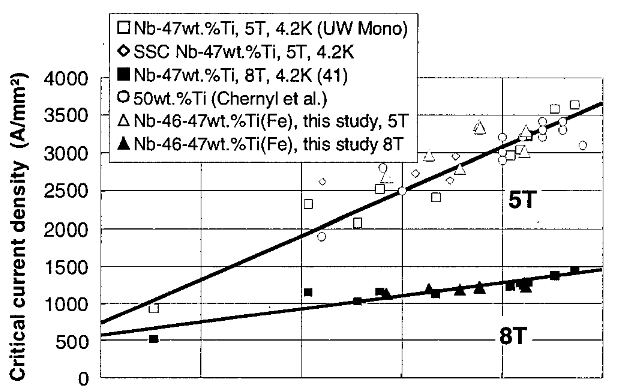

Fig. 7 The relationship between 5 T and 8 T (4.2 K) J_c and precipitate volume is similar to that previously reported for Nb-47wt.%To monofilament [10], SSC R&D strand [6], and Nb-50wt.%Ti [11]. The linear trendlines have been fitted to the conventional Nb-47wt.%Ti data.

2. The best Nb-Ti(Fe) production scale strands approached the performance of the best laboratory scale strands.
3. The increased Fe content reduced the dimensions of the α-Ti precipitates produced by heat treatment.
4. The high atomic number contrast at the grain boundaries may be regions depleted in Ti because of the relatively fast transport of Ti along the grain boundaries compared with bulk diffusion through the grains. The contrast is also not inconsistent with Nb-rich "precipitation" regions observed recently by field ion microprobe [12]. We have also observed this contrast in Nb-Ti strands and Nb-Ti-Ta [2].

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