

EFFECT OF  $Cu_4Ti$  COMPOUND FORMATION ON THE CHARACTERISTICS OF NbTi ACCELERATOR MAGNET WIRE\*

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Abstract

High critical current density,  $J_c > 2500 \text{ A/mm}^2$ , and small filament diameter,  $d \sim 3 \mu\text{m}$ , are required in multifilamentary NbTi wire used for superconducting accelerator magnets. Wires obtained from various commercial sources had  $J_c$ 's in the range 1000 to  $2800 \text{ A/mm}^2$  and  $d$ 's in the range 1 to  $23 \mu\text{m}$ . The filaments were examined by means of scanning electron microscopy in order to determine the reason for the variation in  $J_c$ . It was found that the filaments in high  $J_c$  wires had clean smooth surfaces and uniform cross section along their lengths. Filaments in low  $J_c$  wires show formation of  $Cu_4Ti$  compound particles on their surfaces and large variations in cross section. The lower critical current measured in these wires is believed to be largely due to this effect. The superconducting-normal state transition is relatively wide in these wires.

Introduction

Multifilamentary NbTi wire used in the Fermilab Tevatron and in the former CBA project has a mean critical current density,  $J_c$ , of about  $1800 \text{ A/mm}^2$  in the NbTi at  $B = 5\text{T}$ ,  $T = 4.2\text{K}$ , and  $\rho = 10^{-12} \text{ ohm cm}$ . In finished cables  $J_c$  is 10 to 14% lower. For SSC, The Superconducting Super Collider project, it is desired to achieve critical current densities 40-50% greater. Figure 1 shows results of some 750 wire and 200 cable short sample tests on CBA material. The dotted curves indicate schematically the desired increases for SSC. The NbTi filaments have mean diameter,  $d$ , of  $9 \mu\text{m}$  in the FNAL-CBA wire. At this size or larger, superconducting magnetization currents produce field errors which require internal magnet correction coils. For SSC the aim is to reduce  $d$  to 2-3  $\mu\text{m}$  so that separate, external correction coils may be used.

Large increases in  $J_c$  have been achieved as a result of research at the University of Wisconsin, Lawrence Berkeley Lab, Wah Chang, and Intermagnetics General Corp. Until recently this work has been concerned with the intrinsic  $J_c$ , as measured for wires having large sized filaments, typically  $20 \mu\text{m}$ . In wires with fine filaments variations in filament cross-section are relatively large and lead to lower observed critical current and, hence,  $J_c$ . As we show in the following sections, in order to produce fine filament wire of high  $J_c$ , the surface condition of the filaments is of paramount importance. This is due to the fact that surface imperfections, such as intermetallic compound particles of size 1 or  $2 \mu\text{m}$ , can produce large variations in individual filament diameters when  $d$  is reduced below about  $10 \mu\text{m}$ . The effective or measured  $J_c$  is thereby reduced, even though the intrinsic  $J_c$ , i.e., the value observed in wires having uniform filaments, may not be. In wires with filaments of large diameter,  $d \sim 20 \mu\text{m}$ , this effect is usually unimportant.

Observation of Copper Compound Formation

For the wires of Fig. 1 the measured  $J_c$ 's varied from 1400 to  $2200 \text{ A/mm}^2$  even though the wires were processed in the same manner in principle. (Wires of 3 manufacturers are included in this graph, but the distributions for the individual manufacturers are approximately the same). Metallographic examinations were made in an attempt to find the reason for the variation in  $J_c$ .

Figures 2 and 3 show observations of wires in which  $J_c = 2200 \text{ A/mm}^2$  and  $1400 \text{ A/mm}^2$ , respectively. In each Figure we show an optical photomicrograph of the entire cross section, a scanning electron microscope (SEM) micrograph of a group of filaments, and an SEM micrograph of a more or less typical individ-

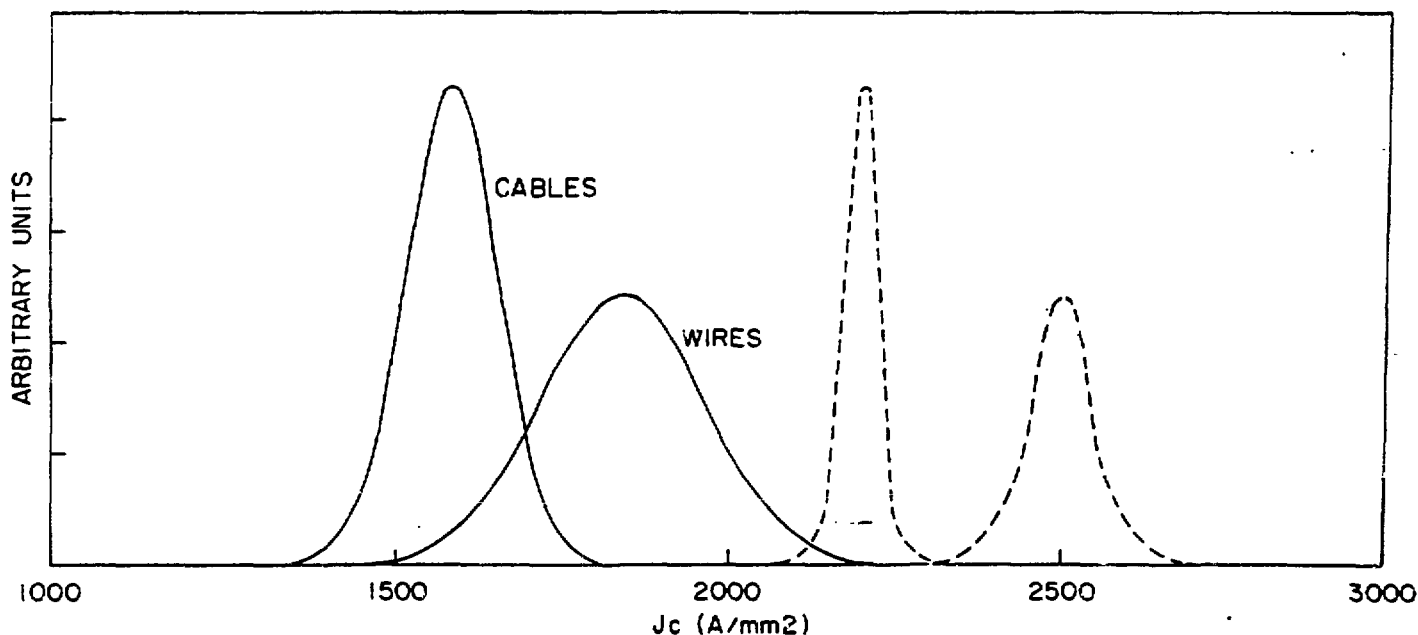


Figure 1. Normal distribution of critical current densities in wires and cables. Solid curves: CBA project results. Dashed curves: schematic of proposed characteristics for SSC project.

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ual filament. The middle photo is obtained by polishing the end of a wire and etching away the copper to a depth of roughly 50  $\mu\text{m}$ . The surface appearance of the outer ring of filaments or, as in Figs. 2-3, of the filaments surrounding the inner copper core may then be examined. The third photo is obtained by completely etching away the copper and then examining individual filaments longitudinally.

In high  $J_c$  wire the filament surfaces are relatively clean and smooth, whereas in the low  $J_c$  wire one sees considerable necking or sausageing of individual filaments and the occurrence of numerous particles or nodules. These are typically 1 or 2  $\mu\text{m}$  in size. Chemical analysis of these particles shows the presence of Cu and Ti in the ratio 4:1 with a small amount of Nb, whereas at other points on the filament surface there is no detectable copper. The particles are, therefore, intermetallic compounds, probably  $\text{Cu}_4\text{Ti}$ .<sup>2</sup> The formation of these is most likely thermally activated. This can occur during extrusion or as a result of the major heat treatments used to optimize the intrinsic  $J_c$ .

The third photo of Fig. 3 shows the presence of the compound particles at necked regions of filaments. This indicates that these hard particles lead to variations in filament diameter of the type shown in the middle photo. The measured value of  $J_c$

is an average over all filaments; it is, therefore, degraded from the intrinsic or uniform filament value.

#### Variation of $J_c$ with Filament Diameter

The presence of compound particles makes it difficult or impossible to draw wire down to filament sizes much below  $\sqrt{10}$   $\mu\text{m}$ . In Fig. 4 we show the variation of  $J_c$  with  $d$  for various experimental wires. The cross-hatched band indicates a region for which considerable data exists; these points are for wires in which the SEM photos show large numbers of compound particles, and much sausageing of the filaments. Some of the wires are optimized for maximum critical current while some are simply drawn down from optimized 20  $\mu\text{m}$  filament wire. In the latter case there is some decrease in the intrinsic critical current density, but the largest part of the fall off of  $J_c$  is caused by variations of  $d$ , which become proportionally more serious as  $d$  is reduced.

At the top of Fig. 4 are results for some very recently obtained commercial samples of fine filament, high  $J_c$  wire. Photomicrographs of the filaments in these wires are very similar to those of Fig. 2; the NbTi surfaces are smooth and clean in each case. The variation of  $J_c$  with  $d$  is relatively small and it is possible to optimize  $J_c$  even for

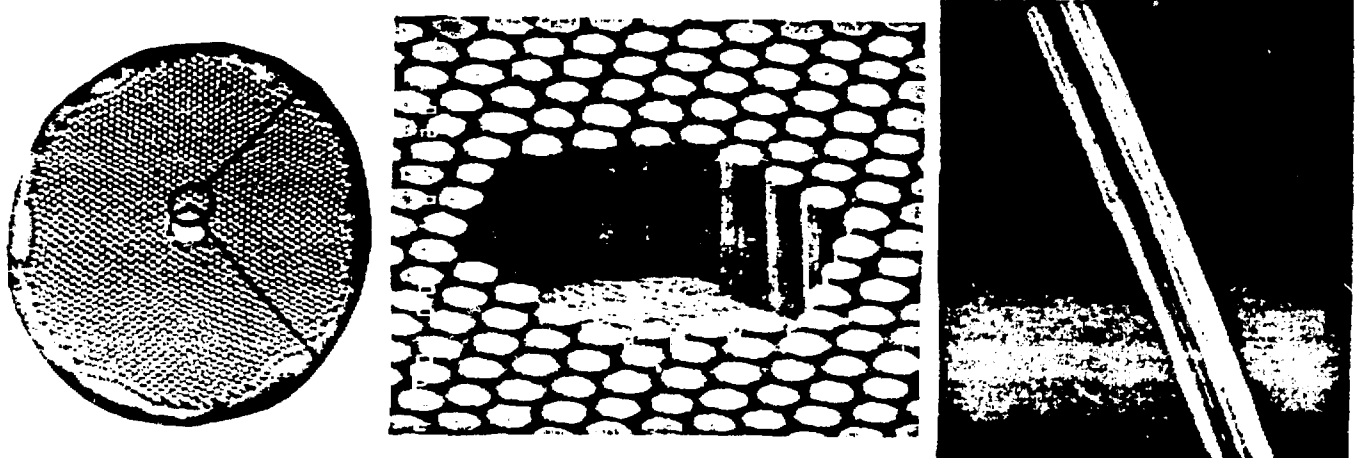


Figure 2. Photomicrographs of high  $J_c$  ( $2200 \text{ A/mm}^2$ ) CBA wire.  $d$  (wire) = 0.68 mm,  $d$  (filament) = 9  $\mu\text{m}$ , Cu/SC ratio = 1.7.

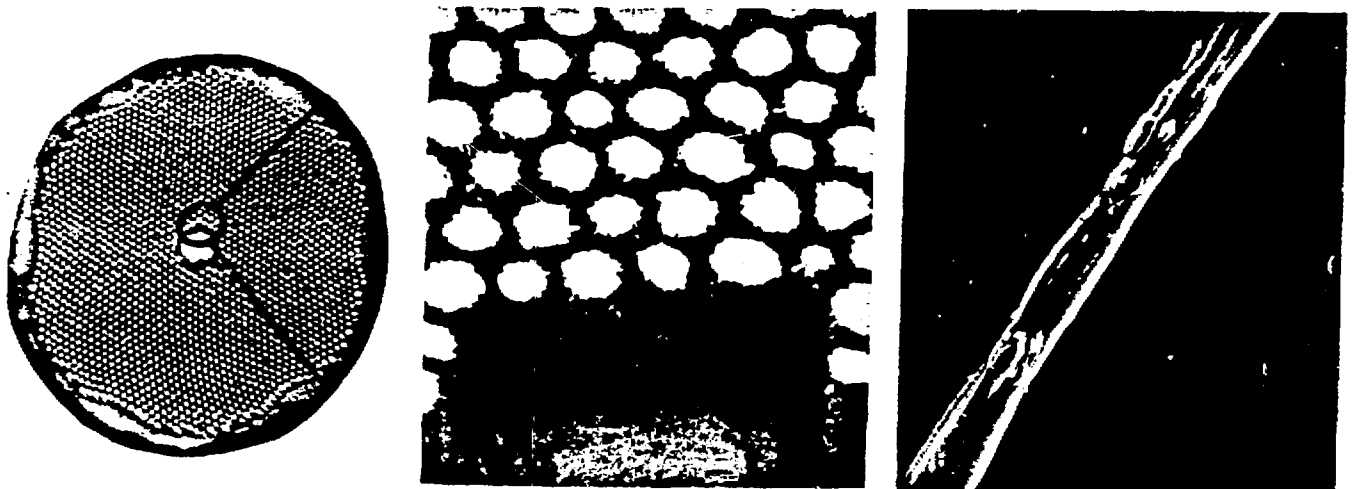


Figure 3. Photomicrographs of low  $J_c$  ( $1400 \text{ A/mm}^2$ ) CBA wire showing filament necking and compound particle formation.

the smallest diameters,  $\approx 3 \mu\text{m}$ . Methods of suppressing the  $\text{Cu}_2\text{Ti}$  formation include careful control of extrusion and major heat treat temperatures, and cladding of the NbTi billet rods with metals like Nb, Ta, or CuNi. The methods used to produce the samples of Fig. 4 are proprietary and not known by us.

#### Variation of $n$ with Filament Diameter

The resistance transition between the normal and superconducting state is of the form  $R = \text{const} \cdot I^n$ , where  $R$  is the wire resistance and  $I$  the current.<sup>3</sup> The quantity  $n$  is a measure of the sharpness of the transition. It is thought, although there is as yet no quantitative theory, to be a measure of the geometric quality of the wire, that is, of filament uniformity. The  $n$ -values corresponding to the data of Fig. 4 are given in Fig. 5. For the high quality wires, the  $n$ -values are slowly varying as a function of  $d$  and in the range 30-40. For the poor quality wires  $n$  falls rapidly as  $d$  decreases; that is, it is correlated with the fall off in  $J_c$ , and the increasing variation in filament cross section. A similar correlation exists for the data of Fig. 1;  $n$  varies from  $\approx 10$  to  $\approx 40$  as  $J_c$  varies from the lowest to the highest values. Values of  $n$  in excess of  $\approx 30$  are usually indicative of good quality wire in the sense discussed.

#### Conclusions

In summary,  $J_c$  is determined by a combination of two factors: the intrinsic critical current density determined by strain-anneal treatment, and the uniformity of filament cross section, which depends on keeping the filament-matrix interface clean and free of compound particles. It appears that production of high  $J_c$ , fine filament NbTi wire is feasible. Intrinsic  $J_c$ 's approaching  $3000 \text{ A/mm}^2$  have been achieved commercially. Several manufacturers have produced quantities of wire in which  $d \approx 2.5 \mu\text{m}$  and  $J_c > 2500 \text{ A/mm}^2$ .

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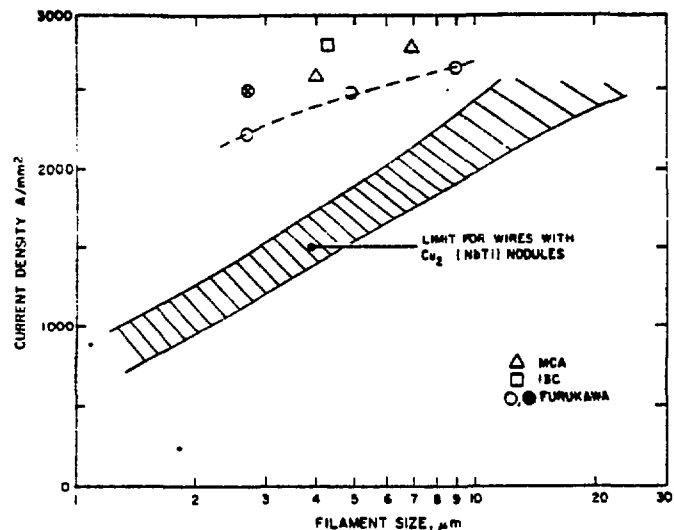


Figure 4. Critical current density vs filament diameter for experimental commercial samples.

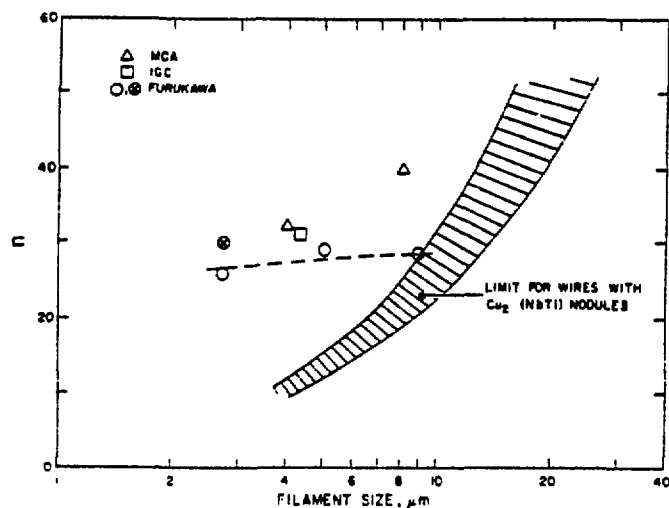


Figure 5.  $n$ -value vs filament diameter for experimental commercial samples.