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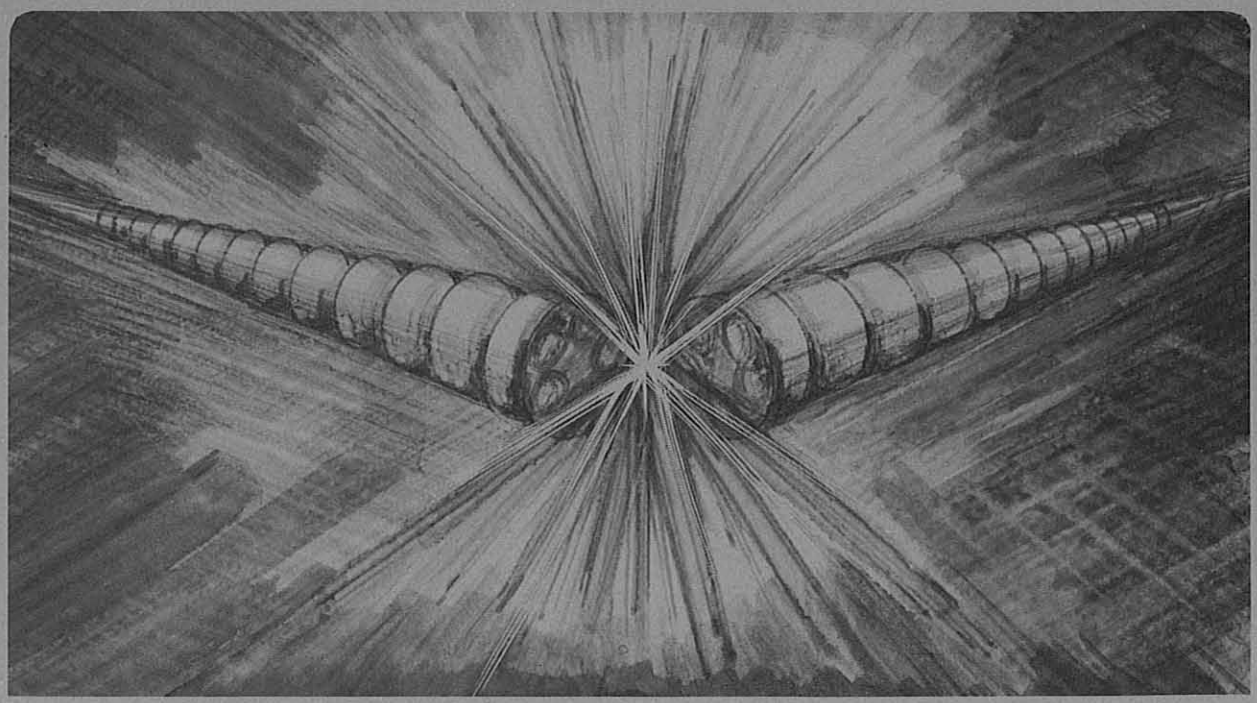

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CHARACTERIZATION OF Nb-Ti SUPERCONDUCTORS WITH ARTIFICAL PINNING STRUCTURES*

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ABSTRACT

A series of multifilamentary APC Nb-Ti superconductors have been made with Nb added as a normal second phase to provide flux pinning centers. Two compositions, 12.5vol% and 25vol%Nb in Nb-Ti, have been fabricated into multifilamentary composites using two different fabrication methods. One method used hot isostatic compaction and hot extrusion thoughout the processing. The other method (bundle-and-draw process) discontinued all hot processing at an intermediate level. While the J_c values of the bundle-and-draw wires are quite promising, the critical current of the extruded wires appears to be limited by poor uniformity of the filament cross-sectional area along the conductor length. The large values of the index of the resistive transition and small filament standard-deviation-to-average area ratios observed in the wires produced by the bundle-and-draw process suggest extrinsic factors have little effect on J_c. The variation in J_c as the wire diameter is reduced appears to be most strongly affected by intrinsic factors: Nb distribution and pinning strength. The final filament microstructure and Nb spacing are shown to be difficult to calculate, e.g., the mean Nb spacing near the final wire size may be 1/2 to 1/3 that of the calculated value.

INTRODUCTION

There are four dominant factors affecting the critical current (I_c) of a multifilamentary superconducting wire. Two of these factors are intrinsic in nature, while the other two are of an extrinsic character. This is true regardless of whether the superconducting material is an oxide or metallic alloy. Filament composition and microstructure are the two important intrinsic factors. The composition determines both the critical temperature and the upper critical field, whereas the microstructure determines the material's ability to pin fluxoids. The two important extrinsic factors are the amount of superconductor in the wire cross-section and the uniformity of the filament cross-sectional area along the conductor length. These extrinsic factors are sensitive to conductor design and are manufacturing process dependent.

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The artificial pinning center (APC)[1] materials investigated in this work are studied through effects of these four factors. The advantage of using APC materials, which introduce a normal phase in the superconducting matrix in order to pin fluxoids, is that the final microstructure can be predetermined to some extent by the initial conductor design.[2] Since the α-Ti particle distribution in conventional Nb-Ti superconductors depends on composition, heat treatment and deformation processing, it is difficult to obtain a uniform pinning structure throughout the conventional materials. However, while current calculations for predetermining the pinning site distribution and Nb thickness in APC materials can guide the wire fabrication process, they can be misleading when predicting the final microstructures in real conductors.

EXPERIMENTAL PROCEDURE

The wires used in this investigation were produced by Intermagnetics General Corporation (IGC). Four APC wires were produced with two Nb volume fractions and two fabrication methods. All four conductors used Nb-Ti alloy rods with a nominal composition of 46.5wt%Ti and elemental Nb as the pinning material. Initially two composites, A and B with 25vol%Nb and 12.5vol%Nb, respectively, are produced by hot isostatic compaction (HIP) and hot extrusion. These two composites are used as bundling sub-elements for the fabrication of the four wires. One process utilizes an additional HIP and extrusion process producing the "extruded" wire, while the other employs two bundle and cold draw steps producing the "bundle-and-draw" wire.

To quantify microstructural features, such as the variation in filament area and Nb and Nb-Ti spacing, an image processing system was assembled. The hardware consists of an IBM PC-AT, a Cohu model 4815 CCD monochrome solid state camera which can be mounted on either a Nikon metallograph or a copy stand, and a Scanman 500 for photographs. The IBM system was modified by the installation of a frame grabber, a PC-Vision Plus board.

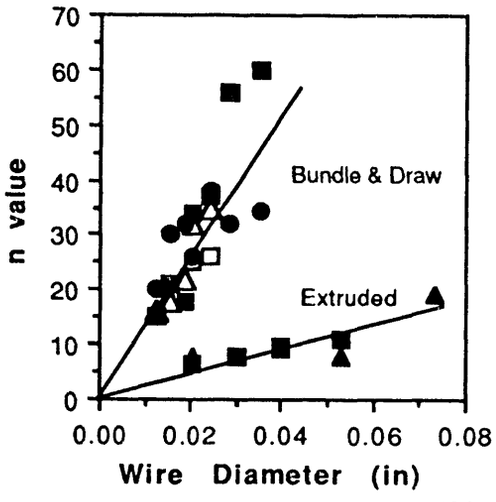

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Fig. 1. The resistive transition index n at various fields versus wire diameter for extruded and bundle-and-draw wires of composite A.

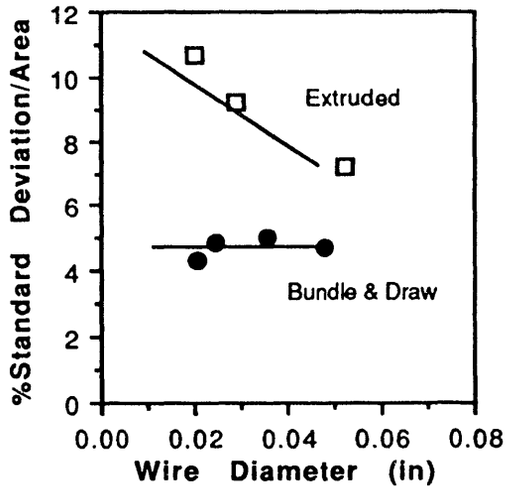

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Fig. 2 Ratio of the standard deviation of the filament area to the average area versus the wire diameter for composite A.

Current-voltage measurements to determine the critical current and n, the index of the resistive transition, were performed by H. Kanithi, et al., of IGC and are published in these proceedings.[3]

RESULTS AND DISCUSSION

Critical current measurements performed by H. Kanithi, et al.[3], on the four types of wires showed that the bundle-and-draw conductor with 25vol% Nb had the highest J_c and the larger values of n. As mentioned previously, the J_c of the conductors depends on both extrinsic and intrinsic factors. The extrinsic factors are associated with filament uniformity and integrity, while the intrinsic factors are associated with the composition and microstructure within filaments. It has been shown that n values are a qualitative measure of filament uniformity.[4,5] The n values of the extruded wire are smaller than that of the bundle-and-draw wires for all wire diameters. (fig. 1) As the diameter of the wire decreases, the n value also decreases; however, the line slopes are different for the two processes. This trend is examined from a different viewpoint in fig. 2. If one measures the filament area in a wire cross section and plots the ratio of the standard deviation of filament areas to the average filament area versus the wire diameter, then one observes a difference in this ratio between the extruded and bundle-and-draw wires. While the ratio increases as the wire diameter decreases for the extruded conductor, it is constant for the bundle-and-draw wire. The constant standard deviation of the filament area to the average filament area results for the bundle-and-draw wire suggest that the filaments are very uniform and not changing with wire diameter, while the n values suggest that the filaments are becoming less uniform with smaller diameter. These results suggest that the filaments of the extruded wire are not very uniform; this is seen in fig. 3(a). Figure 3(b) shows the filaments of the 0.020in (0.508mm) bundle-and-draw wire of composite A. The uniform filaments observed in fig. 3(b) along with the result in fig. 2 suggest that the n value may also be sensitive to intrinsic factors in the bundle-and-draw wires.

The non-uniform filaments in the extruded wires shown in fig. 3a are due to the formation of Cu Ti intermetallics during the final HIP and extrusion. Intermetallic formation occurs because an extra Nb diffusion barrier between the Cu jacket and the re-bundled monofilaments was not added during the second stage extrusion. The Cu-Ti formation was more severe in the B composite since it had a thinner Nb layer. The incorporation of a barrier would improve the wires by producing more uniform filaments. However, the Nb-Ti interdiffusion that occurred during the hot processing may also have adverse effects on the intrinsic properties of the filaments because the pinning strength of the Nb should be reduced by the Ti interdiffusion.

The J_c and n values show that processing, i.e. extrinsic factors, had a larger effect than intrinsic factors in determining the properties of the A and B composites because larger J_c values were obtained by the bundle-and-draw process of both composites. The highest J_c at 5T (3367A/mm^2) was obtained with bundle-and-draw A composite. This J_c is much larger than its extruded counter-part which only had a J_c at 5T of 1816A/mm^2. The J_c(5T) of the bundle-and-draw B composite was also larger than that of both extruded wires with 2563A/mm^2 for the 0.044in (1.118mm) diameter wire.

The Nb and Nb-Ti structure at the end of second stage processing of the A and B composites can be seen in fig. 4. Even though the initial hexagonal configuration has been significantly distorted, the measured average Nb-Ti sub-filament diameter (24.2μm) is nearly that of the calculated diameter of the Nb-Ti region (25μm). However, a line length measurement across the Nb-Ti regions gives the average distance between the Nb as 15μm. A significant difference in the Nb spacing is obtained depending on the measurement technique. Since the calculated diameter assumes one has a cylindrical Nb-Ti rod, a line length or line intercept technique should provide a better measure of the Nb spacing. Thus, the average line length across a circle is about 3/4 of the diameter. The calculated Nb spacing by a line length technique would be about 19μm. This value is about 20% larger than the measured value.

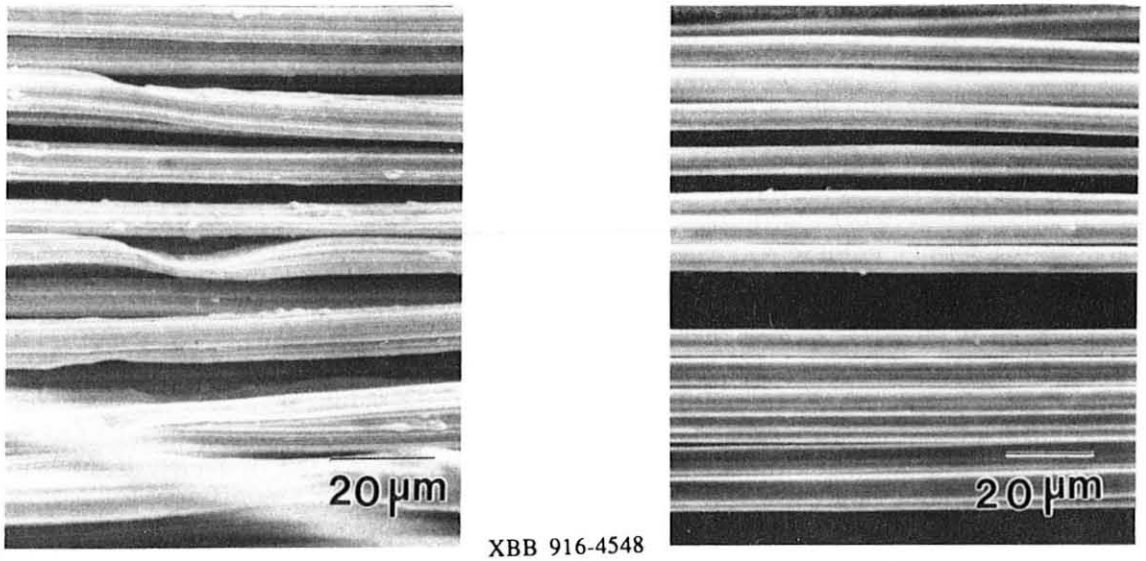

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Fig. 3 SEM micrograph of the filaments of composite A after being processed into wire and removing the Cu matrix, (a) extrusion process and (b) bundle-and-draw process.

When the hexagonal rods of the second stage, with the microstructure of fig. 4, are subsequently deformed in the third stage extrusion or bundle-and-draw process, the uniform Nb/Nb-Ti distribution is lost (figs. 5 and figs. 6). Figures 5a and 5b are SEM photographs of composites A and B, respectively, after the third stage extrusion. The average separation between the Nb layers is now 1.5μm. The average spacing of this structure is much less than that of the calculated values ideal for a cylindrical Nb-Ti rod. The diameter of such a rod would be 4μm while its average value by a line length method would be about 3μm. Both calculated values are two to three times larger than the measured spacing. These observations suggest that it will be difficult to make reliable calculations of the optimum APC pinning structure.

Composite A with 25vol% Nb produced the highest Jc in both processes. Since the Nb volume fraction is altered only by the initial Nb sheet thickness, the number of pinning centers and their distribution should be about the same in both composites. However, the Nb pinning centers in the B composite will have about half the thickness of the A composite. Since the Nb spacing in the two extruded wires is comparable (1.51μm and 1.53μm for the A and B composites, respectively) it appears that the elemental pinning force differs in the composites. The J_c peak of the bundle-and-draw B composite occurs at a larger wire diameter than the A composite. This suggests that the Nb layer thickness plays a more significant role than the Nb spacing.

The TEM micrograph of the 0.024" (0.610mm) bundle-and-draw wire of composite A with the highest J_c of all the wires is seen in fig. 6. The slight contrast variation between the Nb and Nb-Ti regions makes it difficult but not impossible to distinguish between the regions. From measurements of some of the larger Nb-Ti regions one can place and upper limit on the Nb pinning site spacing. This upper limit is about 50nm. As with the larger extruded rods this value is less than the calculated value of 89nm. If one extrapolates the result for the 1.032in (26.21mm) rod to that of a 0.024in (0.610mm) wire, then the spacing would be about 35nm. This is larger than the 22nm flux line lattice spacing expected at 5T. The J_c(5T) of the B composite (12.5vol%Nb) peaks at a larger wire diameter (0.044in, 1.117mm) than the A composite, even though the Nb pinning site spacing is similar. This suggests the Nb pinning strength may be reduced if its thickness becomes too small. Hence, two pinning aspects may have to be optimized in APC materials: pin spacing and pin thickness.

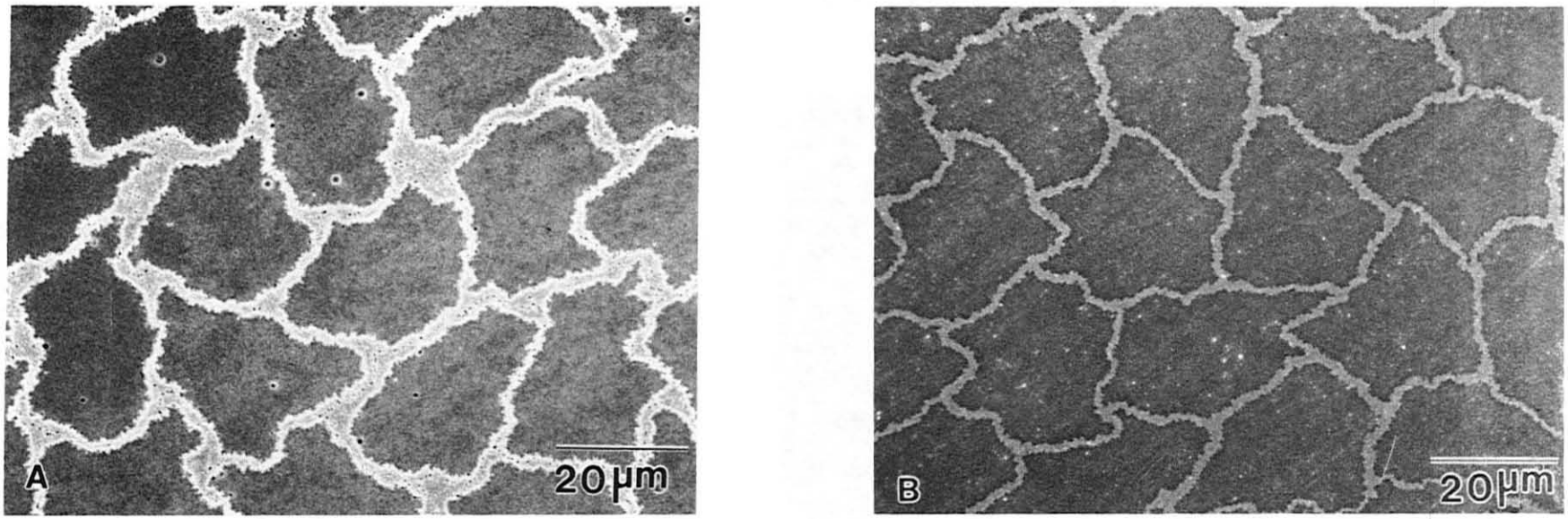

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Fig. 4 SEM micrographs of the second stage cross section (a) composite A (25vol%Nb) and (b) composite B (12.5vol%Nb). They have both received the same processing up to this point.

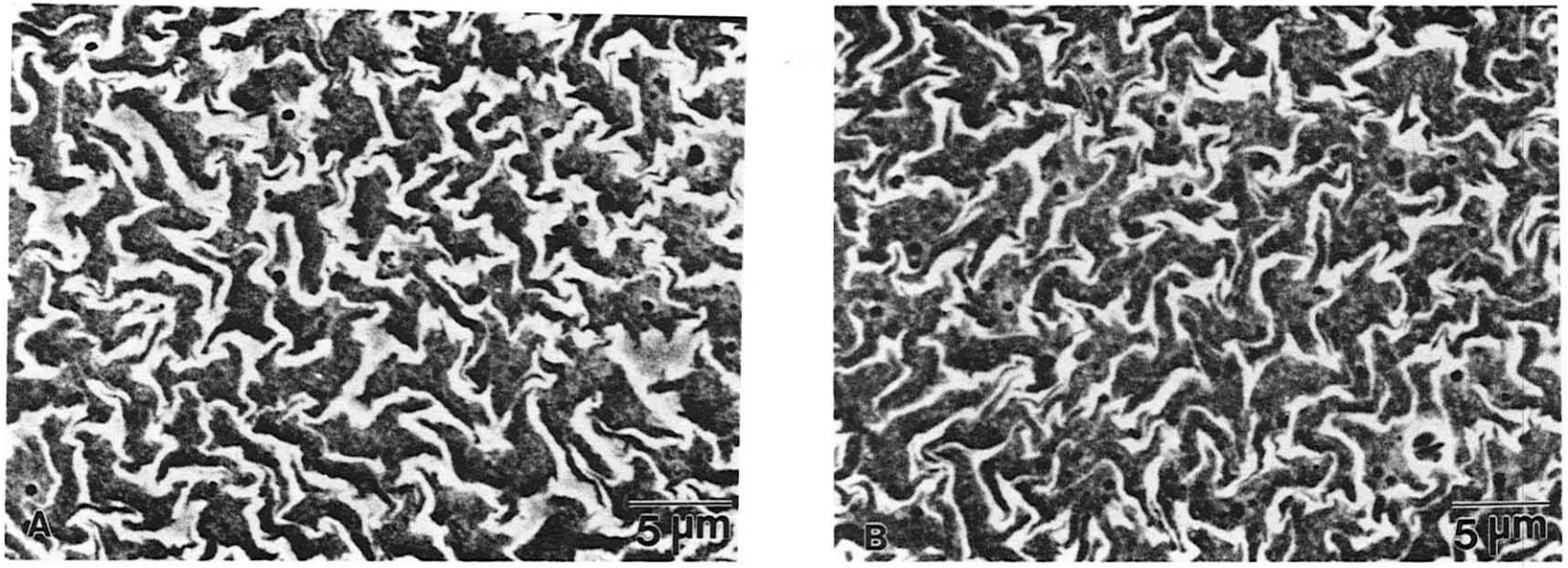

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Fig. 5 SEM micrographs showing the cross section of the third stage at 1.032in (26.21 mm). The light regions are Nb and the dark are Nb-Ti, (a) 25vol% Nb and (b) 12.5vol%Nb.

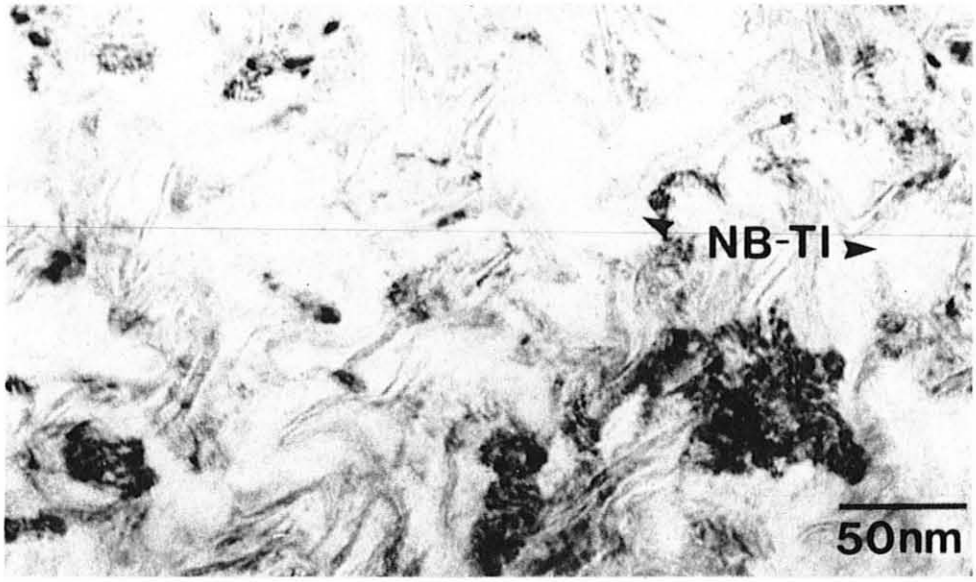

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Fig. 6 Cross section TEM micrograph of the 0.024in (0.0610mm) bundle-and-draw wire of composite A (25vol%Nb).

CONCLUSIONS

The critical current of the extruded wires appears to be limited by the poor uniformity of the filament cross-sectional area along the conductor length. It is difficult to draw any conclusions about the intrinsic factors of these wires. However, for the bundle-and-draw conductors, the large n values and small standard-deviation-to-average-area ratios suggest that extrinsic factors are not limiting J_c. The variation in J_c as the wire diameter is reduced appears to be most strongly affected by intrinsic factors: Nb distribution and pinning strength. The initial Nb and Nb-Ti configuration is lost during deformation processing, thus making reliable calculations of the final APC pinning structure difficult.

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