

## $\alpha$ -Ti PRECIPITATES IN HIGH CURRENT DENSITY MULTIFILAMENTARY NIOBIUM TITANIUM COMPOSITES

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### Summary

Transmission electron microscopy has been used to characterize the microstructure of a Fermilab composite at various sizes, as well as a variety of commercial composites in their as-delivered, final size state. A much more diverse range of precipitate morphologies than had been previously envisaged was seen. High  $J_c$  composites were, however, seen to have a similar morphology of walls of plate like  $\alpha$ -Ti in a fine sub-band structure. The precipitate size ranged from about 0.5 to 200 times the coherence length.

### Introduction

It is now well-established that the optimum microstructure of all high  $J_c$  Nb-Ti composites, whether of Nb 50 wt% Ti<sup>1-3</sup> or of the more Nb-rich alloys<sup>4-6</sup> is composed of  $\alpha$ -Ti precipitates in a fine scale sub-band structure of the order of 30-50 nm in diameter. Although the sub-band diameter has been measured in a number of cases,<sup>6</sup> there is still no good quantitative description of the combined precipitate and sub-band microstructure which would permit a quantitative explanation of the critical current density. The major difficulty in preparing such a microstructural description has been the considerable difficulty of obtaining good images of the  $\alpha$ -Ti precipitate distribution. Recent advances in the preparation of foils from Nb-Ti composites for transmission electron microscopy have given us a much better view of these precipitates. This paper reports details of the precipitate microstructure of a 2000 filament (Fermilab design) composite which we have drawn and heat-treated in a variety of ways. Also reported are the final size microstructures of several of optimized high  $J_c$  conductors, obtained from a variety of commercial sources.

The precipitate morphologies seen in these wires are considerably more diverse than those previously reported.<sup>1-5</sup>

### I. Fermilab Composite

#### Experimental Procedure

The Fermilab Nb 46.5 wt% Ti composite, containing approximately 2000 filaments, was received at 3.66 mm diameter after an area reduction of 296:1 by cold-drawing following extrusion.<sup>4</sup>

A length of the composite was heat-treated under vacuum for 160 hours at 375°C. This sample was then drawn down to a final wire diameter of 0.67 mm with a 20% reduction in area per pass. Samples for TEM examination were taken after each pass.

Thin foils from longitudinal sections of the wires had been examined previously to determine the average sub-band diameter in the filaments at each stage.<sup>5</sup> Transverse sections could be cut directly from wires with diameters of 1.5 mm or greater, but wires smaller than this had to be plated with copper to increase the total diameter to approximately 2 mm. Details of the thin foil preparation have been given previously.<sup>4,5</sup>

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### Results and Discussion

The microstructure of the composite immediately after heat-treatment at 3.66 mm diameter is shown in Fig. 1. The  $\alpha$ -Ti precipitates can be seen as regions of lighter contrast due to the atomic number effect. The precipitates have a lower average atomic number than the surrounding matrix as they are Ti-rich. Therefore, provided that the precipitates are not diffracting strongly, they transmit more of the incident electron beam and so appear lighter in the bright-field TEM image.

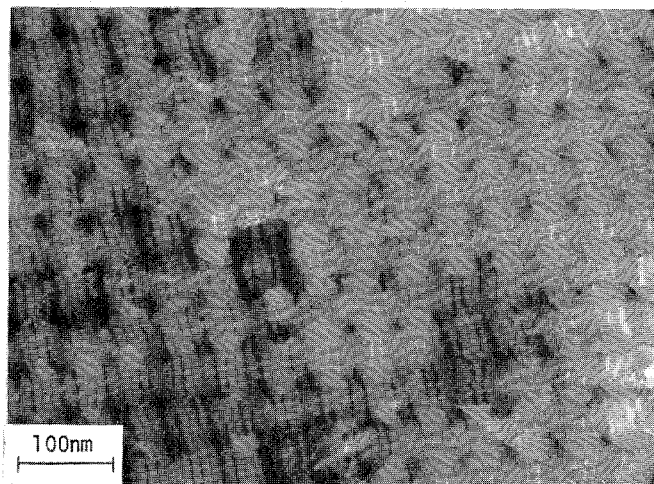


Fig. 1. Fermilab composite, heat-treated for 160 hours at 375°C at 3.66 mm diameter. Section taken at 3.66 mm dia.

Three distinct precipitate morphologies are visible in Fig. 1. There are large, equi-axed particles with sizes up to 150 nm located at the junctions between sub-bands as well as plate-like precipitates inside the sub-bands. These precipitates are smaller, approximately 40 nm in length and 3 nm in width. There is also a light-contrast second phase at the sub-band boundaries themselves. This precipitate appears to be a continuous film, approximately 2 nm thick. Electron diffraction patterns obtained from this sample only showed the presence of  $\alpha$ -Ti as the second phase, but absolute identification of all the precipitates as  $\alpha$ -Ti was not possible owing to the very fine scale of the microstructure. The sub-band structure itself was relatively regular at this stage with an average sub-band diameter of 123 nm.

On drawing down to 2.34 mm, the microstructure became more irregular, both with respect to the sub-bands and to the precipitate morphologies, Fig. 2. The  $\alpha$ -Ti precipitates appeared to have deformed with the matrix, following the same flow pattern.

The sub-band structure and the precipitate morphologies increased in irregularity on further drawing to 0.67 mm diameter, Fig. 3. In some areas of the micrograph, the precipitate structure appeared to be independent of the sub-band structure. It may be assumed that the  $\alpha$ -Ti precipitates extend along the wire axis as continuous walls although this is difficult

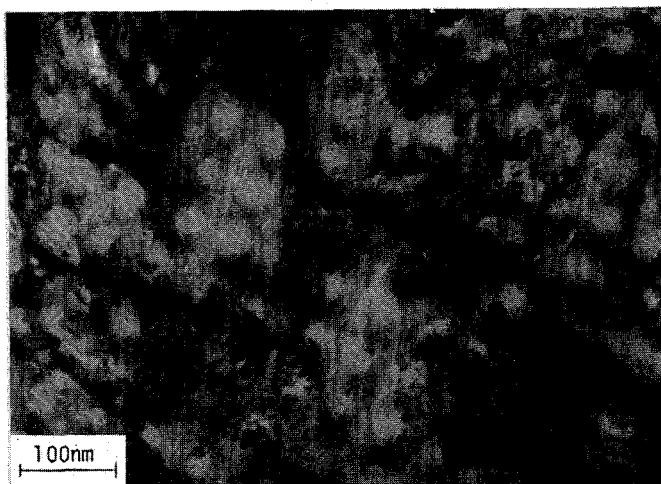


Fig. 2 Fermilab composite treated as in Fig. 1. Transverse section at 2.34 mm dia.

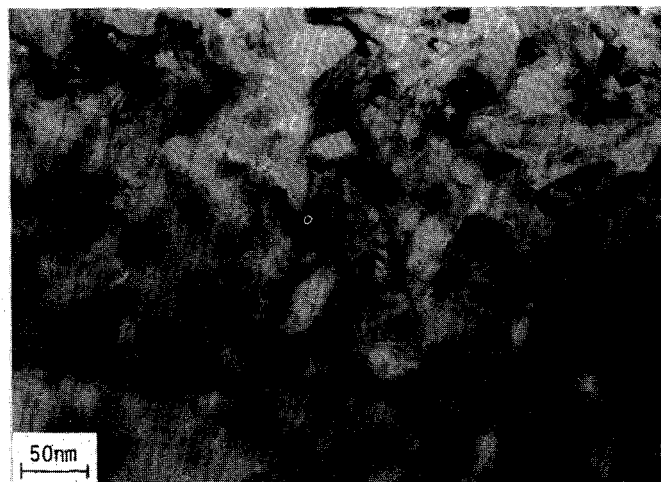


Fig. 3 Fermilab composite treated as in Fig. 1. Transverse section at 0.67 mm dia.

to determine conclusively from the examination of thin foils from longitudinal sections.

The sub-band diameter of the wire at final size was 44 nm. The dominant precipitate morphology was one of plate-like  $\alpha$ -Ti with transverse section widths of  $\sim 2 \times 100$  nm. The  $J_C$  value for this wire was only moderate, being 1540 A/mm<sup>2</sup> at 5T, 4.2K.

## II. Commercial Composites at Final Size

### Experimental Procedure

Details of the commercial composites examined in transverse section in this work are given in Table 1. The 52.7 wt% Ti and the 50.4 wt% Ti composites had been examined in earlier work.<sup>4</sup> Full details of their current density values have been given previously.<sup>7,8</sup> The remaining three Nb 46.5 wt% composites had not previously been examined and are noteworthy for having  $J_C$  (5T, 4.2K) values exceeding 2000 A/mm<sup>2</sup>.

Samples of these composites were copper-plated to a diameter of approximately 2 mm before discs were cut for thin foil preparation.

Several representative TEM micrographs were taken of each sample. These were used to measure the sub-band diameter and the  $\alpha$ -Ti precipitate sizes, inter-

Table 1: Composite Fabrication Details

Wire	A	B	C	D	E
Manufacturer	Airco	VAC	Airco	IMI	IMI
Designation	B248	BA98345	B879	F245	F275
Ti Content(wt%)	52.7	50.4	46.5	46.5	46.6
Wire dia(mm)	0.83	0.6	0.7	0.5	0.7
Filament No.	180	60	55	61	61
Cu:NbTi Ratio particle spacing and volume fraction .	1.8:1	1.2:1	1.4:1	1.3:1	1.24:1

Sub-band diameters and  $\alpha$ -Ti interparticle spacings were measured using a linear intercept method where random lines were drawn on the micrograph and the number of sub-band boundary or precipitate intersections per unit length counted. An approximate estimate of the volume fraction of  $\alpha$ -Ti present in the composites was made by measuring the area fraction of  $\alpha$ -Ti on the micrographs and assuming that all precipitates extended through the total foil thickness so that the volume fraction was the same as the area fraction. An attempt was made to use only those areas of a micrograph where the foil was thinnest in order to minimize the uncertainties of this procedure.

Several samples of a compacted monolith conductor made for the Elmo Bumpy Torus project were also examined in longitudinal section.

### Results and Discussion

Representative micrographs of transverse sections from wires A to E are shown in Figs. 4 to 8 and details of the microstructure and  $J_C$  are given in Table 2. A very striking observation to be made from these microstructures was the similarity of the 4 wires B, C, D, E, all of which had a high  $J_C$ , exceeding 2000 A/mm<sup>2</sup> (5T, 4.2K). All these wires contained arrays of elongated  $\alpha$ -Ti precipitates whose average spacing varied from about 20 to 30 nm. The morphology of the precipitates in these latter composites can perhaps best be described as plate- or wall-like. It can be clearly distinguished from the larger, more-equiaxed morphology found for the lower current density composite A.

The four composites B through E have  $J_C$  (5T, 4.2K) values which vary, in descending order, from 2610 to 2170 A/mm<sup>2</sup> and it can be seen in figures 5-8 that there is a tendency for the lower  $J_C$  composites to contain larger precipitates. This general tendency is also exemplified by composite A which had the much lower  $J_C$  value of 1660 A/mm<sup>2</sup> where the parallel wall morphology was not found and there was a high density of larger ( $\sim 100$  nm), rather equi-axed precipitates. It can be inferred that the heat treatment of composite A occurred much closer to final size than composites B-E, in view of the lack of elongated precipitates in its microstructure. All the wires contained significant volume fractions of  $\alpha$ , the estimates varying from 14 to 22 vol. %. However, these values are likely to be upper limits for reasons already noted. No apparent correlation exists between the volume fraction of  $\alpha$ -Ti and the  $J_C$  but there is an approximate inverse correlation between the particle spacing and  $J_C$  for wires B through E. It is interesting that  $J_C$  (5T, 4.2K) values of  $\sim 2200$  A/mm<sup>2</sup> could be obtained with sub-band diameters greater than 50 nm. This provides additional confirmation of our earlier observation that the inverse sub-band size dependence of  $J_C$  does not hold in 2 phase composites.<sup>4</sup>

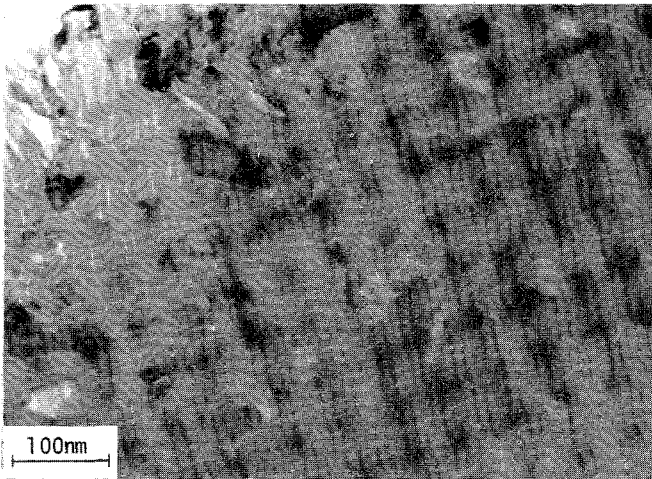


Fig 4. Composite A, transverse section

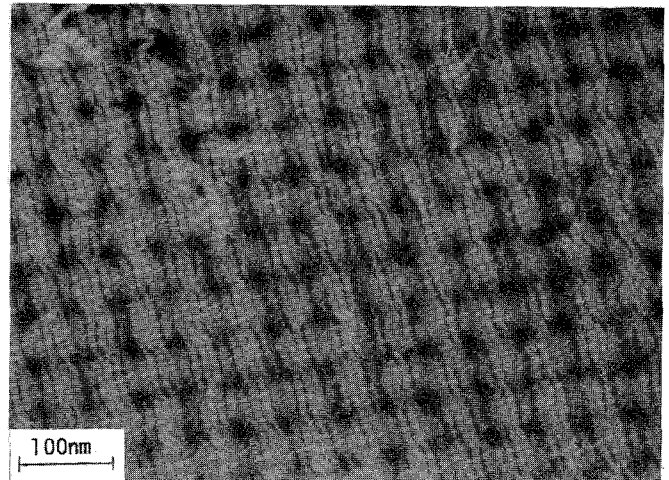


Fig 7. Composite D, transverse section

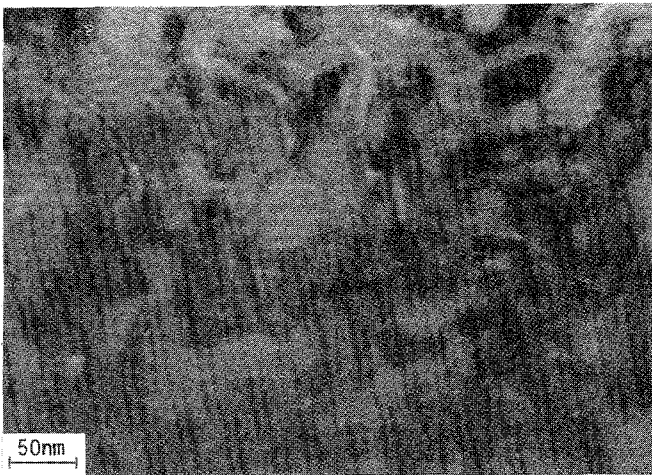


Fig 5. Composite B, transverse section

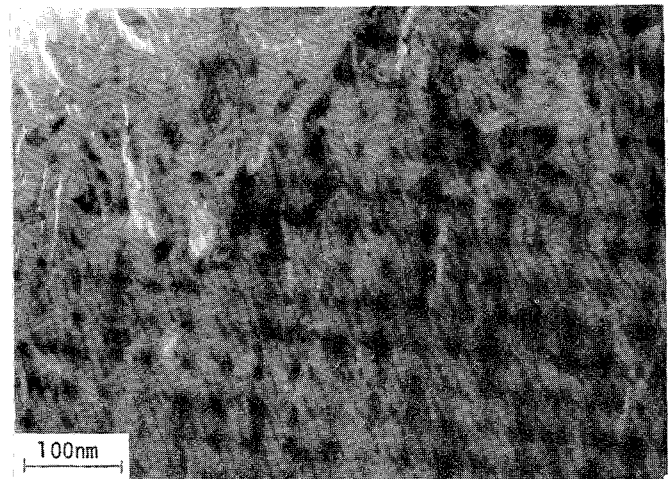


Fig 8. Composite E, transverse section

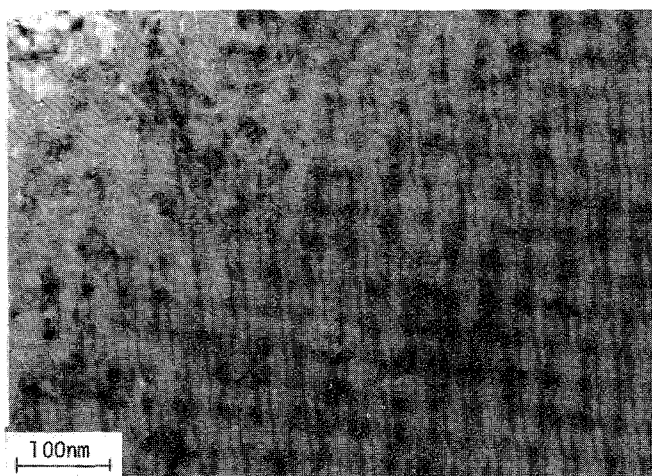


Fig 6. Composite C, transverse section

Table 2: Composite Microstructural and  $J_c$  Details

Wire	A	B	C	D	E
Sub-band dia (nm)	43	40	39	52	57
Vol % $\alpha$ -Ti	22	18	14	20	14
Particle spacing (nm)	39	21	24	31	31
$J_c$ (5T, 4.2K) A/nm <sup>2</sup>	1660	2610	2460	2340	2170

A typical micrograph from a longitudinal section of an EBT conductor is shown in Fig. 9. All the samples examined showed the presence of exceptionally large  $\alpha$ -Ti particles, ranging up to  $1.0 \times 0.5 \mu\text{m}$  in size. The sub-band diameters for these materials were in the range 52 to 70 nm.

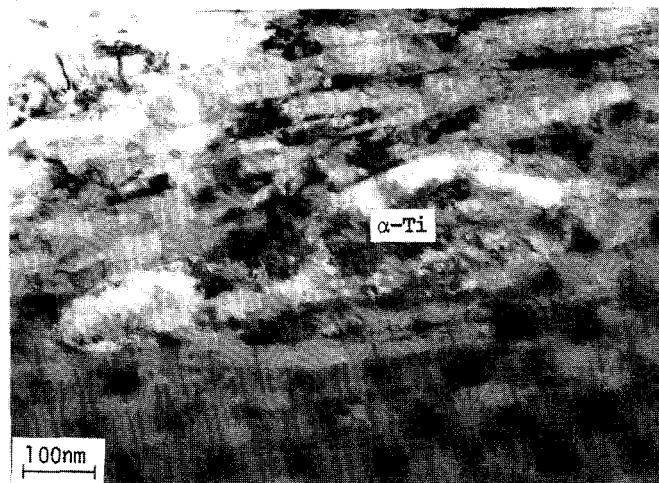


Fig. 9. EBT conductor, longitudinal section.

### III. General Discussion

The morphologies and distribution of the  $\alpha$ -Ti precipitates in all the commercial composites examined were very different from the previously reported descriptions of the precipitates as small, equi-axed particles located at the sub-band boundaries.<sup>2,3,10</sup> The four high  $J_C$  composites all contained narrow, irregular walls of precipitates of high aspect ratio, with the long dimension of the wall running parallel to the wire axis. From the study of the Fermilab composite, it would appear that this precipitate morphology is produced by the deformation of the different precipitate morphologies present in the composite after heat-treatment.

An interesting feature of the precipitate morphologies observed here is their variation with respect to the coherence length  $\xi$ , which is about 5 nm in these alloys. The thickness of the boundary films was generally less than  $\xi$ , (2-3 nm), while the thin dimensions of the walls were generally 1-2 coherence lengths. These precipitates are thus rather effective for flux pinning. The larger precipitates of size  $10-20 \xi$  are rather ineffective flux pinners and the  $J_C$  of composites containing this morphology was correspondingly low. Even larger precipitates have been seen in some cases. Figure 9 shows extremely large  $\alpha$ -Ti precipitates ( $\sim 0.5-1 \mu\text{m}$ ) found in the EBT conductor. Extended heat treatments had been given to this conductor in order to raise its  $J_C$ . Although the  $J_C$  was raised by these treatments, the fabricability was greatly reduced; this is not surprising when such large second phase particles are present.

Although the results presented here cover alloys with Ti contents varying from 46.5 to 53 wt%, we do not believe that the Ti content plays a large role in determining the precipitate morphology. Rather it is the combination of thermal and mechanical treatments which governs the microstructural state.<sup>6</sup> The present results confirm earlier findings that the  $J_C$  is no longer inversely proportional to sub-band diameter when the microstructure is 2 phase.<sup>3,4</sup> Although the microstructures presented here are complex, the similarity of the precipitate morphologies observed

in the high  $J_C$  materials gives some hope that an explicit description of the  $J_C$  in terms of the microstructure is possible. Further investigations of high  $J_C$  composites to this end are currently in progress.

### Acknowledgements

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