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Flux-pinning in multifilament niobium by dislocation cell boundaries

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Magnetization studies of multifilament niobium superconductors had previously shown that the critical current density increased with decreasing filament size (greater amount of cold-work). A transmission electron microscopic study was made on the same conductors. It was observed that the predominant microstructural features in all the samples were dislocation cell boundaries. The average cell size decreased with increasing amount of cold-work and could be correlated with the critical current density, J_c and the maximum flux-pinning force density, $F_{p_{max}}$.

1. Introduction

When a transport current is passed through type-II superconductors, flux jumps may sometimes occur which could drive the superconductor into normal resistive state. One of the ways of achieving stabilization against flux jumps is to make the conductor diameter as small as possible [1]. Commercial superconductors are, therefore, fabricated in the form of fine multifilament wires embedded in a matrix of copper or aluminium. The high conductivity matrix material also aids in removing heat from the superconductor and prevents flux jumps in one filament from transferring to a neighbouring filament.

In a recent magnetization study of multifilament niobium conductors Mathur *et al.* [2] established that the conductors with finer filaments (greater amount of cold-work) exhibited greater flux-pinning than those with larger filaments. The goal of the present study was to study the microstructure of the same filamentary conductors and determine whether a correlation exists between the measured superconducting properties (critical current density, J_c, and maximum flux-pinning force density, $F_{p_{max}}$) and the microstructure. The results of this study are reported here.

2. Experimental

The samples used for microstructural characterization were obtained from the same batch of multifilamentary niobium as used by Mathur *et al.* [2] for magnetization studies. The material was supplied in the cold-drawn condition by Supercon, Inc. The conductors had 400 niobium filaments embedded in a matrix of copper. The filament and conductor diameters of the samples are given in Table I.

TABLE I Multifilament niobium conductor description

Sample no.	Filament diameter (μm)	Conductor diameter (cm)
H-25	7.4	2.08×10^{-2}
H-29	10.0	2.85×10^{-2}
H-33	13.5	3.81×10^{-2}
H-37	18.0	5.08×10^{-2}

Fig. 1 is a scanning electron micrograph of a typical cross-section of the multifilament conductor after deep etching of the matrix. Structural examination of these heavily drawn wires requires transmission electron microscopy (TEM) since the scale of the microstructure is extremely fine.

The technique developed for preparation of thin foils for TEM from transverse sections of multifilament wires has been described in detail elsewhere [3]. Briefly, the method involves increasing the diameter of the superconducting wire by copper plating, slicing into thin wafers and finally thinning electrochemically in a solution of 85% sulphuric acid and 15% hydrofluoric acid at 20° C using a platinum cathode and applying a potential of 8 V. Electropolishing is carried out

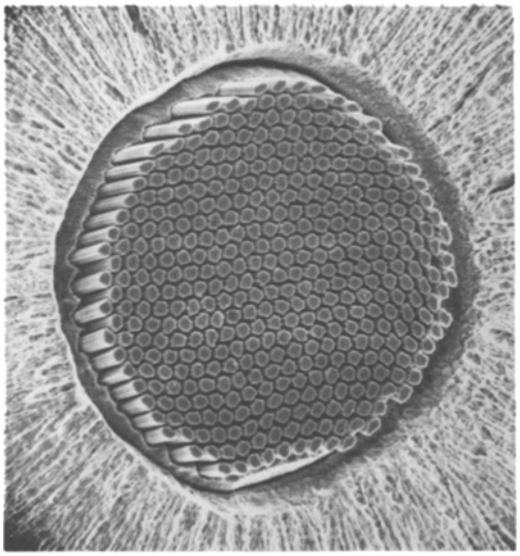

Figure 1 Scanning electron micrograph of the cross-section of multifilament niobium wire. The copper matrix has been deep etched to expose the filaments. Filament diameter 7 μm, X 250.

until about one third of the niobium filaments are completely removed from the surrounding copper matrix. At this stage of electropolishing, large areas thin enough for electron transmission are obtained in several filaments.

The foils were examined in a Philips 300 transmission electron microscope operating at 100 kV.

3. Results and discussion

The representative electron micrographs from the four multifilament niobium specimens are shown in Fig. 2. The predominant feature in all the micrographs is a well-defined dislocation cell structure. The cells are not completely equi-axed but show a tendency to be elongated due to heavy cold reduction. With increasing filament diameter (decreasing amount of cold-work) the cells appear to become larger. An attempt was made to measure the average cell size by the linear intercept method; however, the slightly elongated cell structure resulted in a large scatter in the cell size measurement depending on the orientation of the test line.

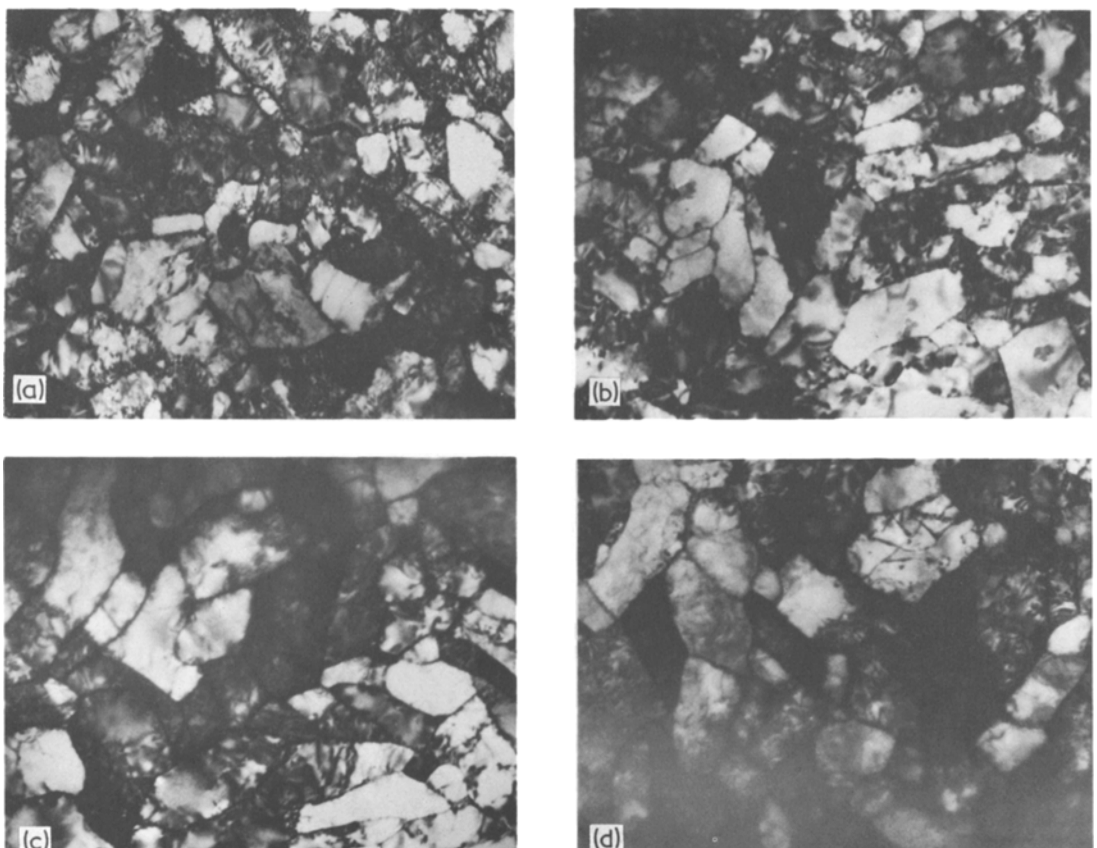

Figure 2 Transmission electron micrographs of transverse sections of multifilament niobium wires. Filament diameters: (a) 7.4 μm, (b) 10 μm, (c) 13.5 μm, and (d) 18 μm, X 75 000.

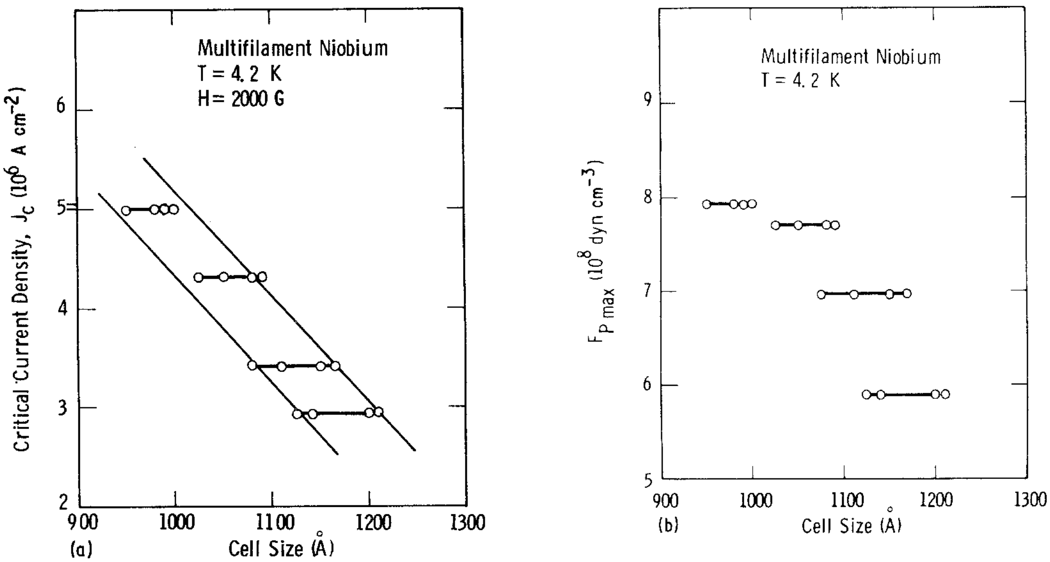

Figure 3(a) Correlation between the critical current density, J_c, and the average dislocation cell size in multifilament niobium. (b) Correlation between the maximum flux-pinning force density, $F_{p_{max}}$, and the average dislocation cell size in multifilament niobium.

To overcome this problem, the number of cells per unit area was counted and from this measurement an equivalent cell size was calculated. Four measurements were taken for each sample.

The next step is to relate the structure to the superconducting properties. Two superconducting parameters were selected. These were the critical current density, J_c, measured at a given field, and the maximum flux-pinning force density, $F_{p_{max}}$ (= $\mathbf{J_c} \times \mathbf{H}$ where $\mathbf{H}$ is the applied field).

In Fig. 3, J_c at 2000 G and $F_{p_{max}}$ are plotted against the average cell size. Note that a good correlation is obtained between the superconducting parameters and the average dislocation cell size, the smaller cells being associated with improved properties. Since finer cells are associated

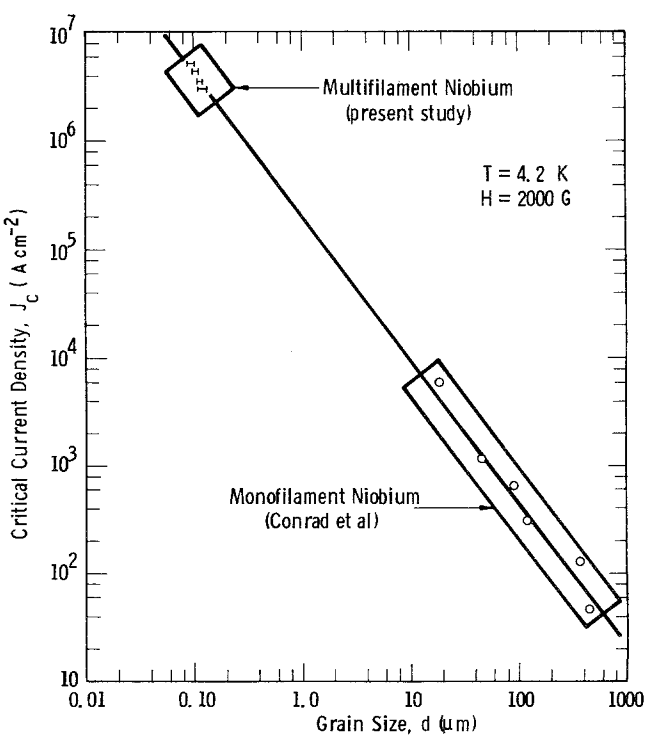

Figure 4 Correlation between the critical current density, J_c, and grain size, d, in mono- and multifilament niobium.

with greater boundary area per unit volume, the present results suggest that there is a strong interaction between dislocation cell boundaries and flux lines in multifilamentary niobium. That two-dimensional surfaces can act as effective flux-pinning centres in type-II superconductors has been well documented before [4–7]. Conrad *et al.* [4] studied the grain size dependence of J_c in commercial purity niobium monifilament superconductors (0.050 in. diameter) at 4.2 K and observed that a finer grain size resulted in higher J_c. Their J_c data at an applied field of 2000 G are plotted against grain size in Fig. 4. Also included in this figure are the results of the present study on multifilamentary niobium. The J_c values were again obtained at a field of 2000 G. It is interesting to note that the multifilamentary niobium data lie on the line extrapolated from the monofilament niobium data of Conrad *et al.* [4]. A single corre-between J_c and grain/cell size holds good over four to five orders of magnitude difference in both the parameters.

In other flux-pinning studies, Hanak and Enstrom [5] report for Nb_3Sn tapes a direct relationship between J_c and $1/d$ where d is the grain size. Similar relationship has been observed by Neal *et al.* [6] on Nb–44 wt% Ti conductors. On the other hand, Nembach and Tachikaws [7] reported a much stronger grain size dependence ($J_c \propto \exp d^{-1/2}$) in V_3Ga tapes. The results on niobium suggest that $J_c \propto d^{-1.36}$. Theoretical calculations by Anderson and Kim [8] predict a linear dependence between J_c and $1/d$. The reasons for discrepancies between the theoretical and experimental results on the grain-size dependence of J_c are not understood at this time and should be explored.

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