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The preparation of thin foils from fine multifilament superconducting wires for transmission electron microscopy

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Cross-sectional examination of superconducting wires yields valuable information in studies that correlate the microstructure with the superconducting properties. This report describes techniques for preparation of thin foils from longitudinal and transverse sections of extremely fine multifilamentary wires for transmission electron microscopy. Applications of these techniques to multifilamentary niobium wires are illustrated.

1. Introduction

The current carrying capacity of type II superconductors is believed to be controlled by the interaction of flux lines with structural imperfections. This interaction is known as "flux-pinning." A study of the relation between the microstructure and superconducting properties is, therefore, vital in any development work in superconductor technology. Commerical superconductors are available as monofilament wires (diameter < 0.010 in.) or in the form of multifilamentary wires with filament diameter < 10 μm (0.0004 in.). The scale of microstructure in these heavily drawn wires is extremely fine and demands the use of transmission electron microscopy (TEM) for structural examination.

There are several reports in the literature on the preparation of longitudinal and transverse sections of fine wires for TEM [1–6]. Most of these techniques involve increasing the diameter of the wire by dipping in a low melting alloy or by electro-deposition, slicing into discs and finally thinning electrochemically or in an ion-beam thinning unit. The above techniques have so far been applied only to monofilament wires up to 0.005 in. diameter. This report describes the methods developed for preparing longitudinal and transverse sections from cold-drawn multifilamentary niobium wires with filament diameter of only 7 μm (0.00028 in.). Fig. 1 shows a cross-section of this multifilament wire in which 400 filaments are embedded in a copper matrix.

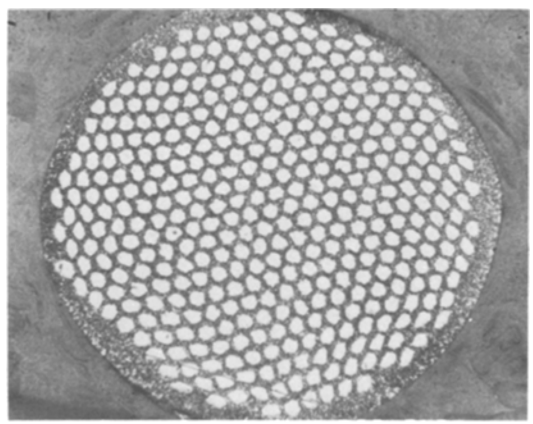

Figure 1 Cross-section of multifilament niobium wire. Filament diameter: 7 μm. Optical micrograph, × 485, reduced by 40% in reproduction.

2. Development of techniques

2.1. Longitudinal section

For preparing longitudinal sections from thin wires, Gidley and Richards [3] suggest the use of a special jig in which a length of wire is held between two jaws and directly thinned to perforation. This technique could not be successfully employed for the fine multifilament wires. Instead, a two step process has been developed for longitudinal sections and is illustrated in Fig. 2. Small lengths of wire are mounted in coldmount and are ground from opposite sides to make a thin ribbon (0.003 in.). Sections, 3 mm (0.120 in.) in length, are cut from this ribbon, mounted on a nickel folding grid and electropolished in a solution of 85% sulphuric acid and 15% hydrofluoric acid at

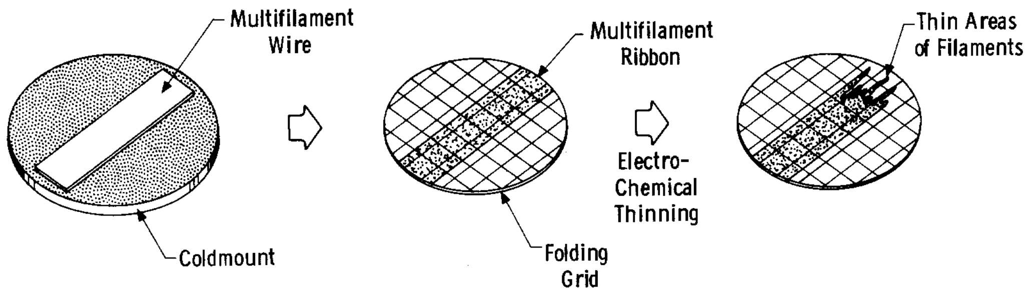

Figure 2 Foil preparation technique for transmission electron microscopy (TEM) (longitudinal section).

20° C using a platinum cathode and applying a potential of 8 V. The electrolyte attacks the copper matrix at a slightly slower rate than the niobium filament and this helps in holding the filaments together during electropolishing. Polishing is continued until thin areas suitable for electron transmission are produced in the niobium filaments.

2.2. Transverse section

Initially an attempt was made to increase the diameter of the wire by dipping it in a low melting alloy. However, this technique had to be discarded as the adherence of the solder to the wire was poor. Since the niobium filaments are embedded in copper, it was found easier to deposit copper electrolytically. Fig. 3 illustrates the sequence of operations in preparing the transverse sections. The wires are plated with copper in a standard copper sulphate electroforming solution (32 oz gal^{-1} copper sulphate and 9 oz gal^{-1} sulphuric acid) using a current density of 60 A ft^{-2}. The copper-plated wire is mounted in coldmount and thin wafers (0.025 in.) are sliced with a precision cut-off wheel. The slices are mechanically thinned to 0.003 in. by grinding on 600-grit silicon carbide paper followed by 4/0 emergy paper. Discs, 3 mm diameter, are punched from the slice and the discs are briefly dipped in hydrofluoric acid to remove the deformed surface layer. Electropolishing is carried out in the same way as for longitudinal sections. Polishing is continued until about 25% of the niobium filaments are completely removed from the surrounding copper matrix. At this stage of electropolishing, there are at least a few filaments that are thin enough for electron transmission. A unique feature in the electropolishing of the multifilament wires is that one can get additional thin areas in other filaments by giving a short additional electropolish.

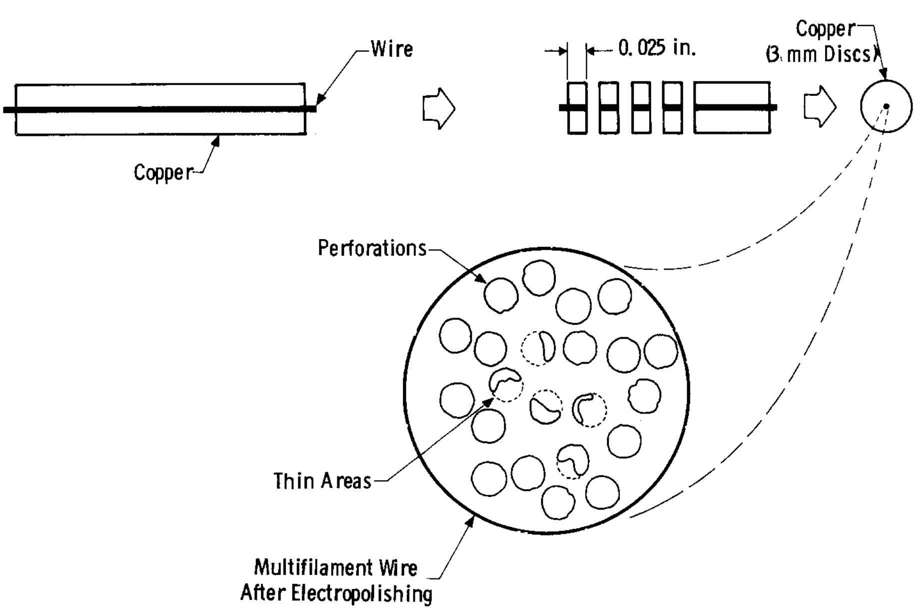

Figure 3 Foil preparation technique for TEM (transverse section).

3. Application of the technique

The foils prepared from the multifilament niobium wires were examined in a Philips 300 transmission electron microscope operating at 100 kV. Typical micrographs from the longitudinal and transverse sections are shown in Fig. 4. The longitudinal section shows a fibrous or banded structure that is characteristic of heavily cold drawn wires. The structure is also characterized by a uniform distribution of dense dislocation tangles within the bands. The boundaries of some of the bands are indistinct due to lack of contrast of dislocations. The transverse section, on the other hand, shows a well-defined cell structure and lends itself to a measurement of average cell size. The mean linear intercept cell size as measured from this micrograph is ~900 Å. The measured critical current density for this multifilament conductor at 4.2 K and at an applied magnetic field of 2000 gauss is 5×10^6 A cm^{-2} [7]. It is clear from the above that this high critical current density is associated with an extremely fine cell size. Such small cell sizes are known to be favourable for flux-pinning [8].

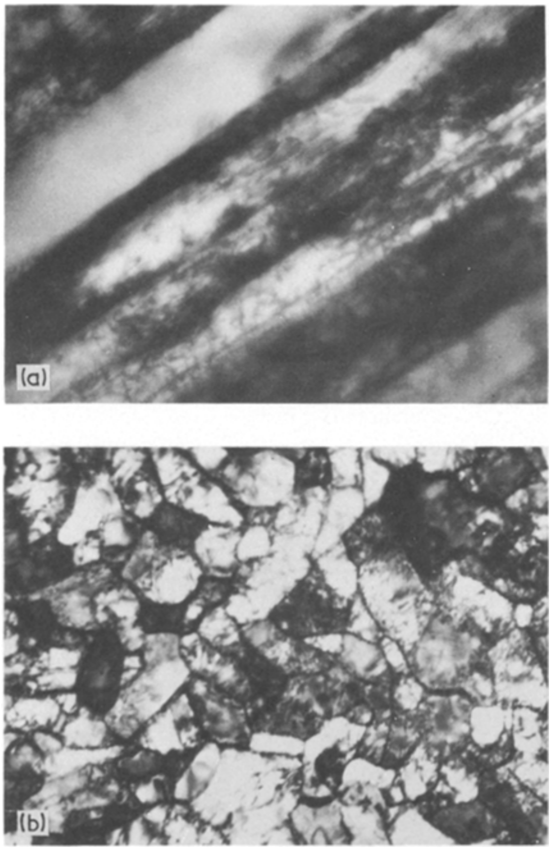

Figure 4 Electron micrographs of multifilament niobium wires (a) longitudinal section, (b) transverse section. × 100 000, reduced by $\frac{1}{3}$ in reproduction.

Finally, this technique can be applied to any other metal or alloy wires, both monofilament and multifilament, for preparing thin foils for TEM. As noted by Glenn and Duff [1], best results are obtained when the plated material polishes at a slightly slower rate than the specimen material. The technique has certain limitations that should be borne in mind. Extensive grinding during specimen preparation could introduce cold work in the material. While this is not a problem in heavily cold drawn wires, caution should be exercised in preparing thin foils from well annealed materials. Secondly, if the electro-plating is done above the ambient temperature, there is a possibility of recovery of dislocation structure in certain metals. However, with proper care, this technique can be successfully employed in preparing thin foils from almost any material.

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