

# ONE STEP INTERNAL TIN NB3SN SUPERCONDUCTOR FABRICATION

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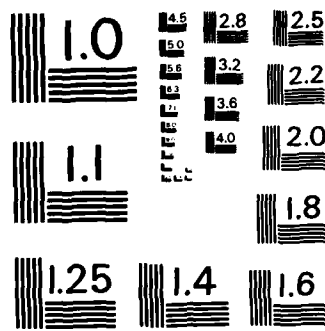
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AFWAL-TR-85-2007

ONE-STEP INTERNAL-TIN  $Nb_3Sn$  SUPERCONDUCTOR FABRICATION

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MARCH 1985

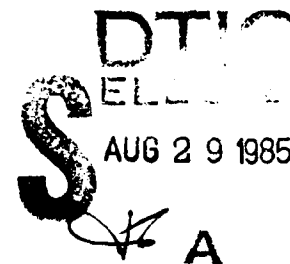
FINAL REPORT FOR PERIOD JUNE 1983 - AUGUST 1984

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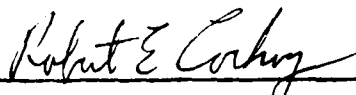


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| 14  | 04   | Internal Tin   |                                    |                      |
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## FOREWORD

This work was an effort to produce a Nb<sub>3</sub>Sn multifilament superconductor with internal tin. In addition, an attempt was made to start with very small diameter Nb to reduce the effect of work hardening. Also, the tin was distributed uniformly between filaments to permit faster homogenization of the tin prior to reaction, and also to avoid large area not available for Nb filaments. The techniques employed were a radical departure from the conventional billet packing.

I wish to thank Frank Lewicki for his excellent skill in implementing these radical techniques and the many suggestions which allowed us to accomplish as much as we did.

|   |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |     |
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### SUMMARY

This program investigated the feasibility of fabricating a  $\text{Nb}_3\text{Sn}$  superconductor with internal tin and employing only a single extrusion.

The starting materials were 0.010" dia. Nb wire and 0.010" dia. tin plated copper wire. These were co-wound into a solenoid, slit longitudinal, and packed into an extrusion can. The billet was then extruded and drawn.

The program demonstrated that the technique employed has potential to meet the above requirements. Although, more work is required to produce a useful product.

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## SECTION I

### INTRODUCTION

The object of this research is to demonstrate the feasibility of producing a  $\text{Nb}_3\text{Sn}$  superconductor in a single extrusion process with a large number of filaments with internal tin. .010 in.

The technique chosen (~~outlined in figure 1 and 2~~) uses .010" diameter Nb and tin plated Cu wires formed into a solenoid. The solenoid is covered with tin plated copper foil and isostatically compacted to a pressure of 17,000 psi. The solenoid is slit along its length. this results in a ribbon about 40 inches long by about 5 inches wide, with the Nb wires running across the 5 inch width.

The ribbon is then rolled up (Jelly Roll) around a 0.5 inch diameter Ta covered copper rod to produce a composite of about 1.5 inches in diameter by 5 inches long. The composite geometry is now a cylindrical bundle of 0.010 inch diameter Nb wire separated from each other by tin plated copper. Each Nb wire is aligned with the axis of cylinder. The cylinder is slid into a Ta lined copper extrusion can which is evacuated and sealed.

The can is extruded at a low temperature and drawn to final wire size without intermediate annealing.

The advantage of the process is that it is an internal tin process with the tin uniformly distributed through the matrix. The Nb is in a relatively soft state having been fully annealed at 0.020 inch diameter. Only one extrusion is required since the bundling technique allows a large number of wires to be precisely aligned and spaced in the matrix.

## SECTION II

### TIN PLATING LINE

The OAP plating line was set up to plate tin on the copper wires and foils which were used to assemble the extrusion billet. A schematic of the line is shown in Figure 3.

The plating solution was made up using a commercial plating additive, as shown in Figure 4.

The plating of the wire was performed at about 10 ft./min. with a deposit thickness of 0.015 mm. No problems were encountered in the continuous plating of 500 ft. of 0.25 mm wire used for the billet assembly.

### SECTION III

#### RIBBON FABRICATION

The .010" Sn plated copper wire and .010" diameter Nb wire were co-wound over a tin plated copper foil covered 1.5" diameter by 40" long mandrel. This single layer solenoid of alternate copper and niobium wires was then overlaid with two layers of tin plated .001" thick copper foil.

The assembly was wrapped in a protective layer of copper foil (to be removed later), and this, in turn, painted with a plastic to seal against leakage during isostatic compaction.

The plastic sealed assembly was then isostatically compacted to 17000 psi. The compaction is required to remove the majority of voids in the composite.

After compaction, the plastic sealant and outer layer of copper foil was removed, and the remainder of the assembly was heated at 330°C for 5 minutes to bond the assembly together.

After heating, the copper clad solenoid (Figure 5 and 6) was slit longitudinally and slid off the steel mandrel. The slit solenoid was flattened to form a ribbon about 4.5" wide, 0.018" thick, and 40" long. The ribbon contained about 2,000 wires of Nb separated by copper.

This was then wound around a 0.5" diameter Ta clad copper core rod. This was slid into a Ta line copper extrusion billet. The billet was welded, evacuated, and sealed.

### SECTION IV

#### EXTRUSION

Prior to extrusion of the multifilament billet, two solid copper billets were extruded. The first was extruded at approximately 200°C, and the second at 100°C. Based on the extrusion pressure and the temperature of the exiting rod, it was decided to extrude the multifilament billet at room temperature.

The multifilament Jelly Roll billet was extruded at room temperature. The extrusion went very well, and no external defects could be seen. The filament size distribution is large, as shown in Figure 7.

This is due to an excessive void fraction in the filament area in the billet prior to extrusion. An isostatic compaction of the billet will eliminate this defect.

## SECTION V

### WIRE DRAWING

The extruded rod was cut into approximately 15 cm long pieces for drawing experiment. The initial drawing was made on a 20% area reduction schedule. The piece was drawn as extruded with the entire outer copper stabilizer in place. Only two draw passes could be made before the outer surface of the rod showed indications of center burst.

A second piece of the extruded rod was machined to reduce the amount of copper in an attempt to get a more equitable distribution of work into the rod during the drawing process. This, too, resulted in center burst after a few die passes.

A third attempt to draw a piece of the extrusion was made by increasing the area reduction per pass to approximately 27%. This gave the same results as the previous drawing experiments.

## SECTION VI

### HEAT TREATMENT AND DIFFUSION STUDIES

Cold drawings of the Oxford Jelly Roll billet was abandoned because of non-uniform deformation in Nb-Cu warped portion of the billet. Some of the broken rods had been heat treated at below the melting point of Sn and then subsequently annealed at 220° and 750°C for several hours. The diffusion of Sn into Cu and Sn from bronze to Nb interface was studied. Three specimens have been used for S.E.M. investigation.

### SPECIMENS

- 1) Diameter of spec. 0.475" diameter. (As received).
- 2) 220°C/40 hours.
- 3) 220°C/48 hrs. + 250°C/50 hrs. + 340°C/200 hrs. + 550°C/100 hrs. + 750°C/100 hrs.

The transversal section of these specimens were polished and subsequently etched. The micrograph of the Specimen 1 (Figure 8 and 8a) shows a large number of voids are present between the Nb-Cu wire layer and Sn plated Cu layer. The voids were not removed even after isostatic pressure. These voids have been developed due to uneven distribution of Sn on Cu surface. Some of the Nb wire showed no bonding with the surrounding region (Figure 9). These defect structures, which are present from the initial stage of process, could lead to internal fracture in subsequent cold drawing.

The Speciment 2 (Figure 10 and 10a) shows microstructure after the 48 hour heat treatment at 220°C. It seems the bonding gaps around the Nb wire has become wider. No bronze layer was found between Sn and Cu interface by S.E.M. studies, but the wider gap around Nb indicated that Sn diffusion has taken place - Sn has gone into solution in Cu, leaving behind Sn vacancies, which is the cause of wider gap around Nb.

Specimen 3 (Figure 11) shows the reaction at 750°C has produced 1.5 - 2 micrometers thickness of Nb<sub>3</sub>Sn. Also, Sn has diffused into Cu. (Figure 12) shows the bonding between Cu-Cu interface.

## SECTION VII

### DISCUSSION

Center burst, an effect due to excessive strain developed along the central axis of the rod, is a result of the inhomogeneous force distribution in the die area during drawing. The effect is a function of area reduction, die angle, configuration of the rod, and relative tensile strength of the various components making up the rod.

Cross sections taken at about 1/8" internals (Figure 13) shows the effect of the non-uniform distribution of work into the rod. Voids (center burst) have developed in or around the copper core. In some areas the core is missing.

Various steps can be taken to minimize or eliminate the problem. They are as follows:

- a) Isostatically compact the billet to eliminate all voids prior to extrusion. This will result in a more uniform distribution of the filaments, and this produce better drawing characteristics in the extruded rod.
- b) Change the geometry of the packed billet by eliminating the copper core and reducing the amount of copper on the outer surface.
- c) Substitute Sn 7 w/o Cu alloy in place of the pure tin plating on the copper wires and foils used to assemble the billet. This will result in a better yield strength compatability of the various components.

In general, it appears with better compacting and electroplating (Sn + 7 Wt. % Cu) on Cu, a better result can be achieved. Hydrostatic extrusion at elevated temperature would further improve the sound workability of the billet. Probably, Nb + Cu wire of rectangular cross-section would decrease the void density. An improvement in rods and bonding gaps could be achieved if compacting is carried out at 220°C.

At new attempt should be made wit the above mentioned modification to make this Jelly Roll an alternative and economically feasible process.

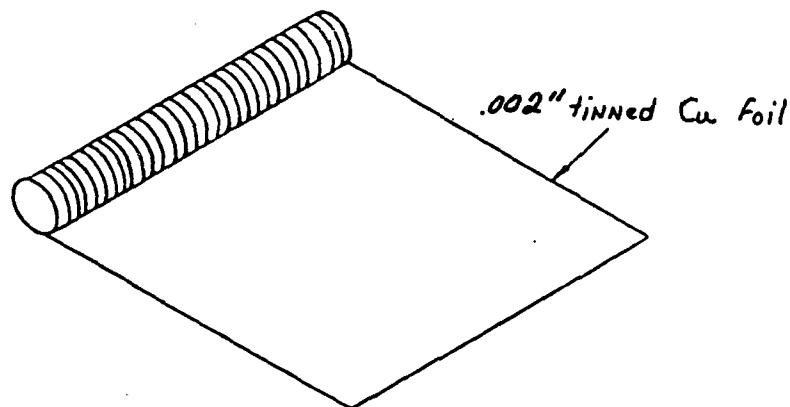
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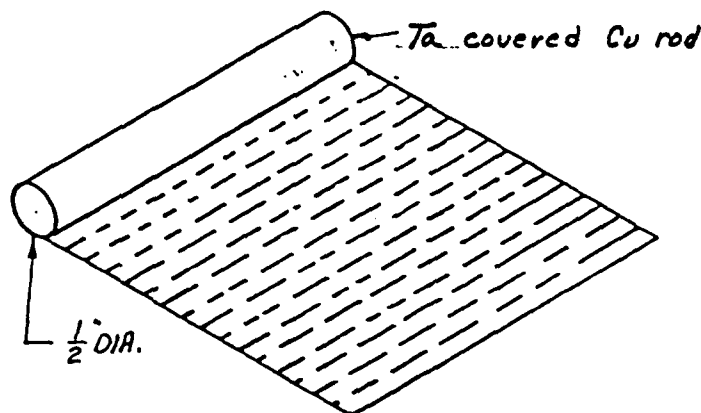
of

Cu foil covered MANDREL



.010" Nb wire  
Cu wire



Foil slit to remove  
Ribbon from mandrel

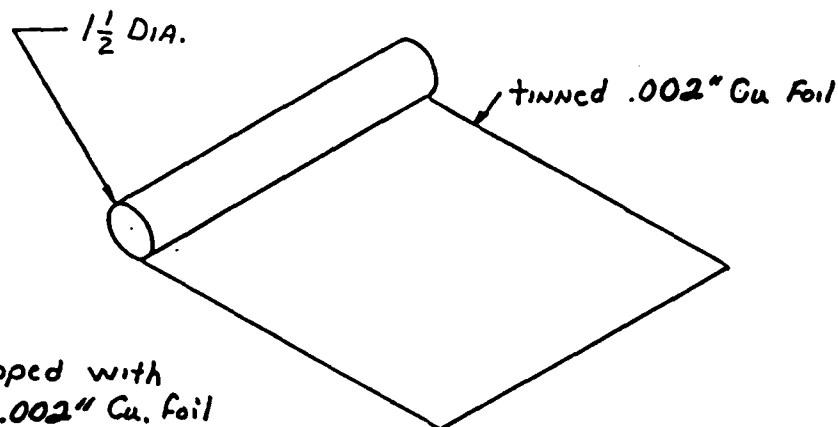


Ribbon of alternated  
Cu / Nb wire between  
Cu foil

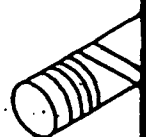
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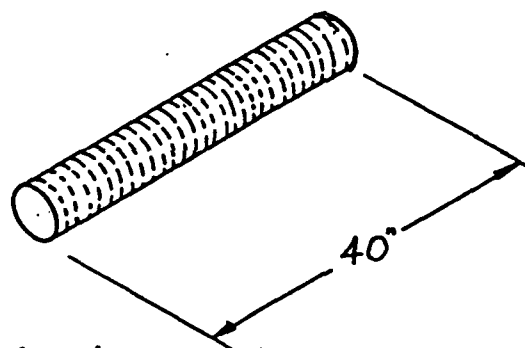
Close w  
Cu & Nb



Mandrel wrapped with  
two layers of .002" Cu. foil



.010"



INATE Nb & tin plated Cu wire  
between 2 Layers of tinned .001"  
foil (total foil thickness of .008")  
statically compacted to remove  
voids.

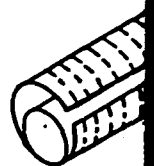
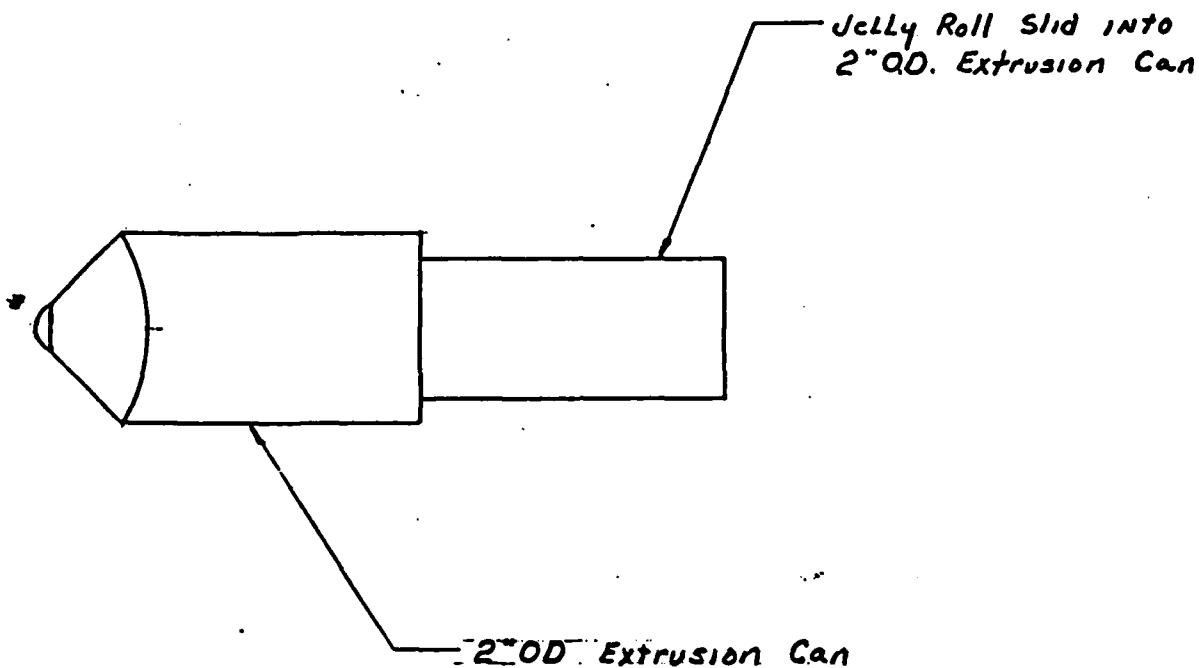




Fig 1.

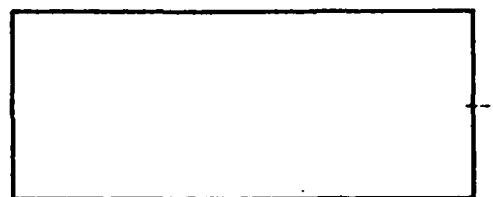
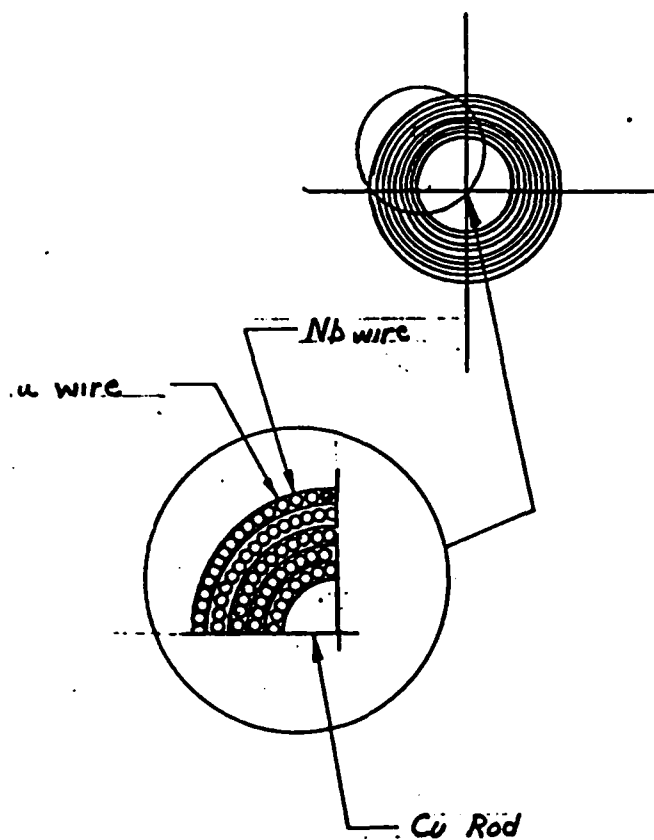
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The Can is extruded and drawn to FINAL size

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|      |       | TOLERANCES UNLESS<br>OTHERWISE SPECIFIED | TITLE: <i>Fig. 2</i><br><i>ASSEMBLY INTO EXTRUSION Can.</i>  |  |  |       |             |      |
|      |       | DEC. ±                                   | DWG.   |  |  | SIZE  | DRAWING NO. | REV. |
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Jelly Rolled Ribbon of Cu & Nb wire between Cu foil

Fig 2.

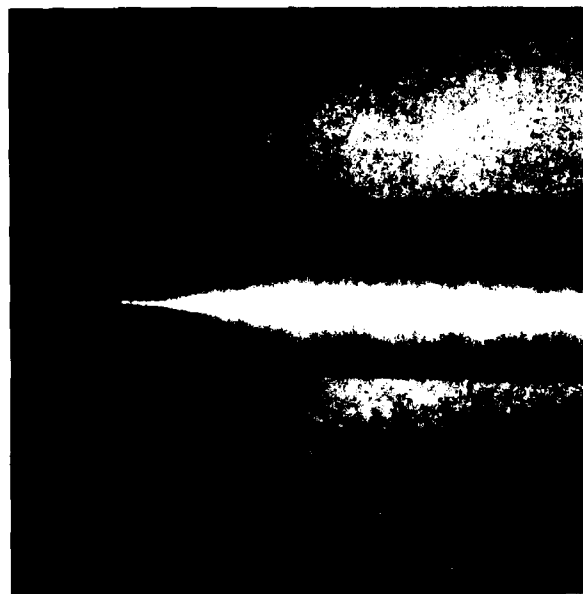
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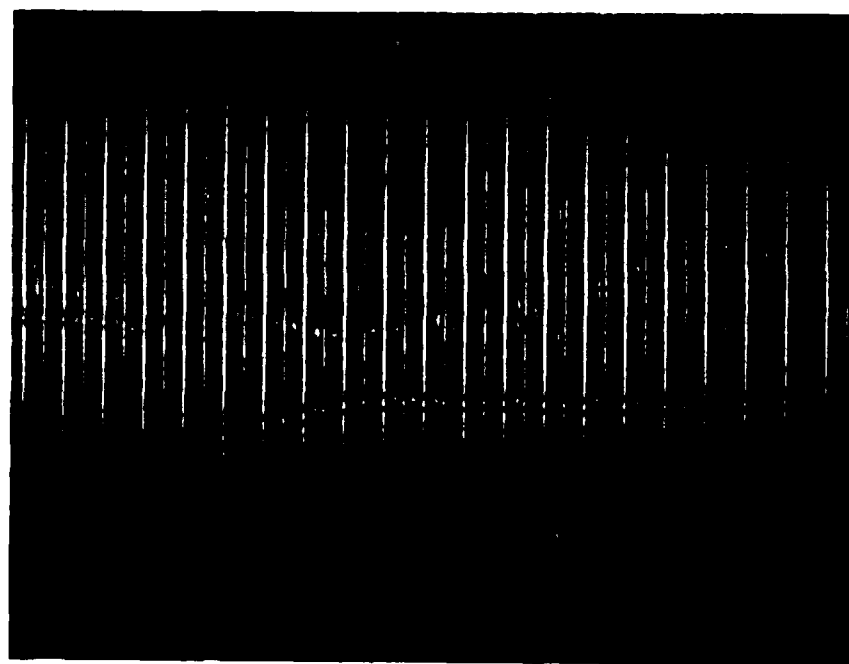
|                            |                          |
|----------------------------|--------------------------|
| Stannous Sulfate           | 30 g/L                   |
| Sulfuric Acid              | 60 g/L                   |
| *Starter F                 | 50 ml/L                  |
| Temperature                | 20°C                     |
| Agitation                  | None                     |
| Cathode Current<br>Density | 50 Amps/ft <sup>2</sup>  |
| Filtration                 | Continuous 2 micrometers |
| Anodes                     | 99.95% Tin               |

\*Product of Lea Ronal, Freeport, New York

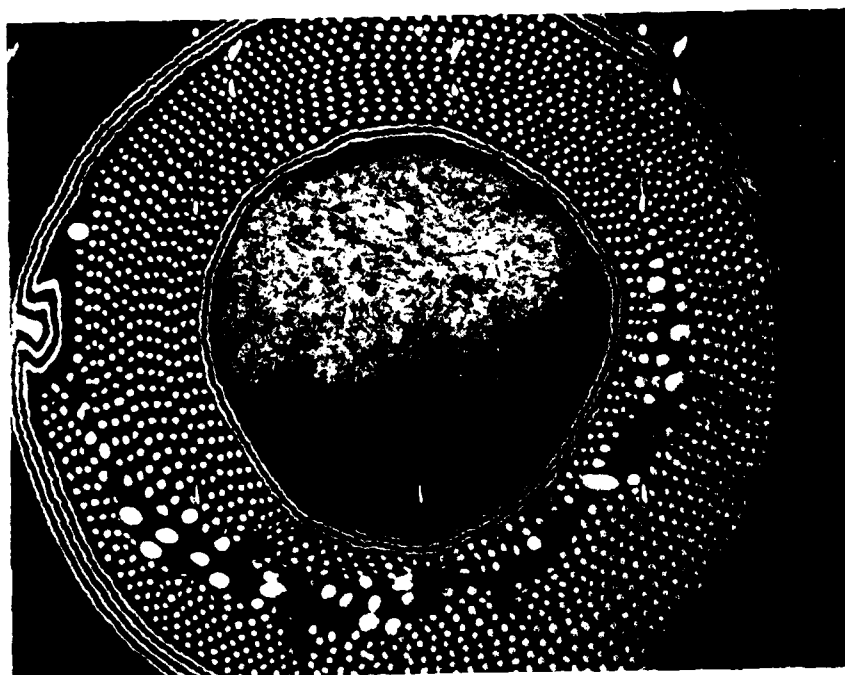
## Fig. 4 Plating Solution



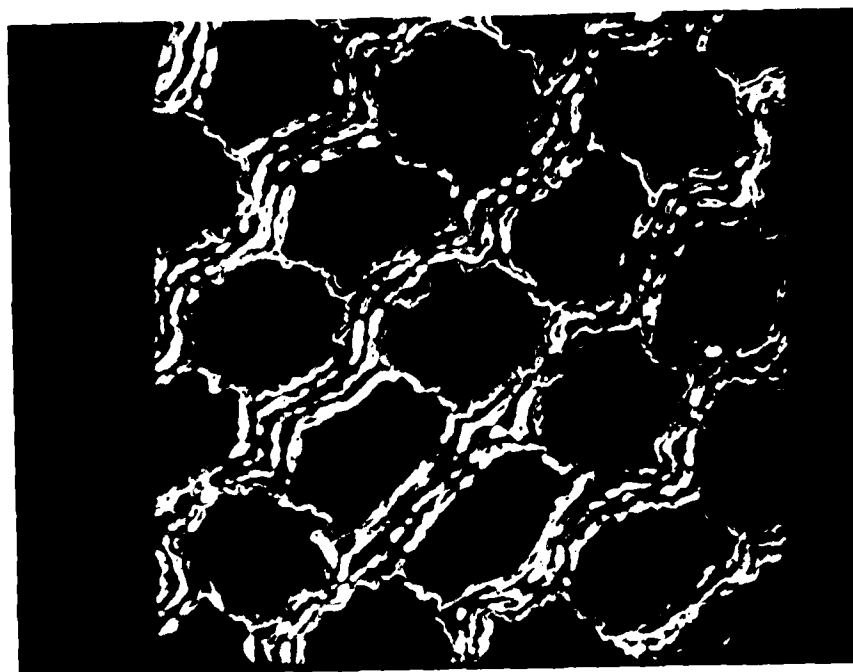
**Fig. 5 Copper Clad Solenoid After Heating**



**Fig. 6 10X Magnification Of Solenoid**



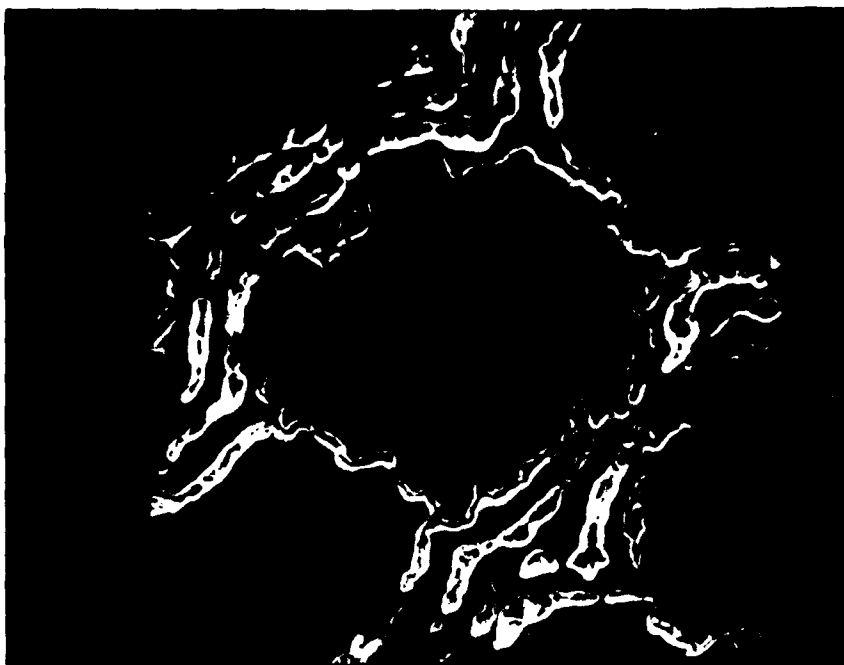
**Fig. 7 Extruded Billet Showing  
Filament Size Distribution**



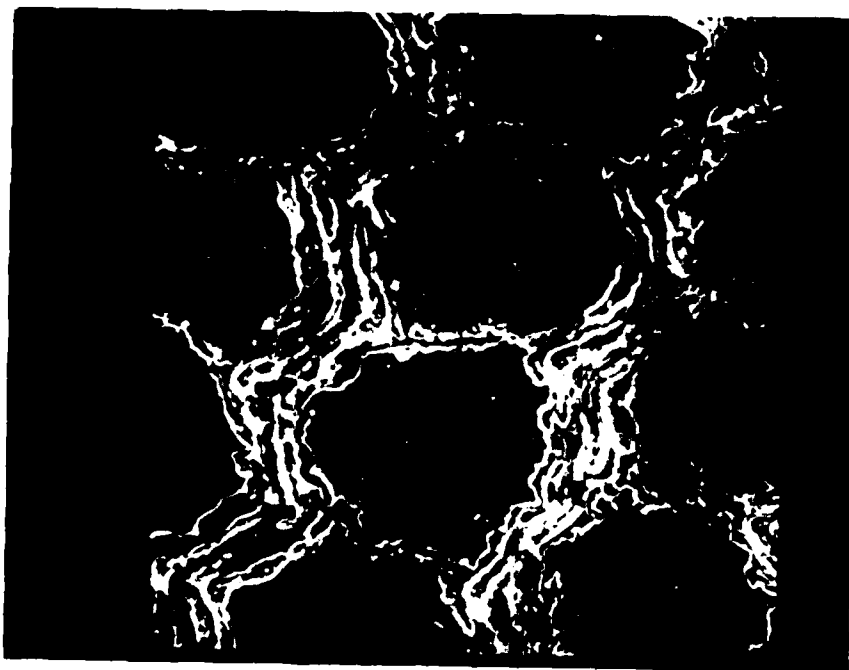
**Fig. 8 200X**



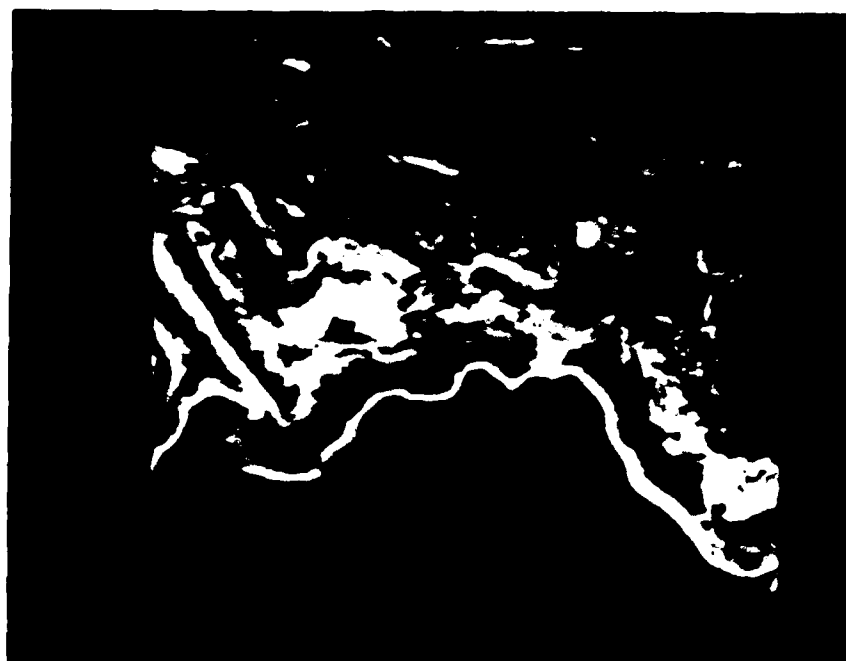
**Fig. 8a 480X**



**Fig. 9 480X**



**Fig. 10 300X**

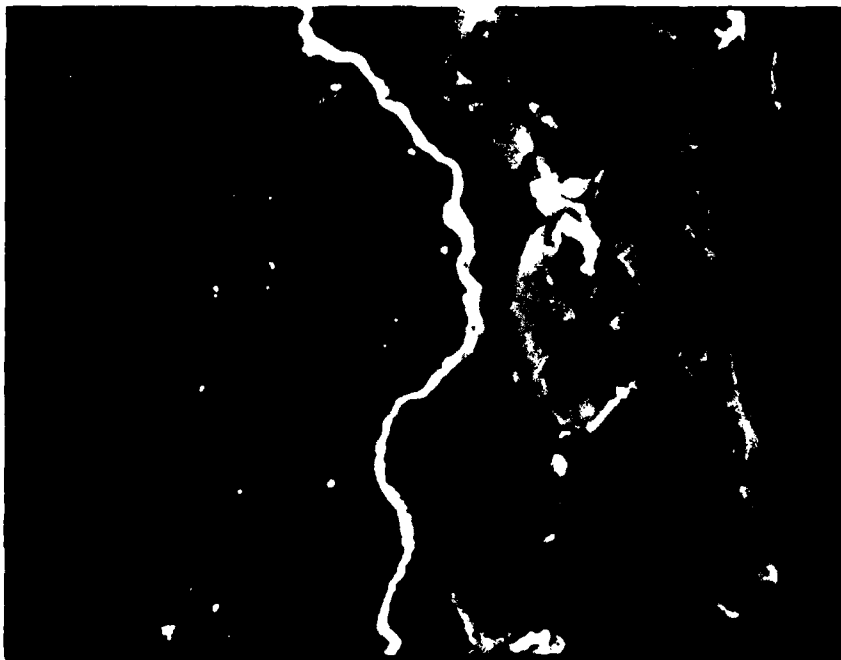


**Fig. 10a 1500X**



**Fig. 11 800X**





**Fig. 12 2000X**

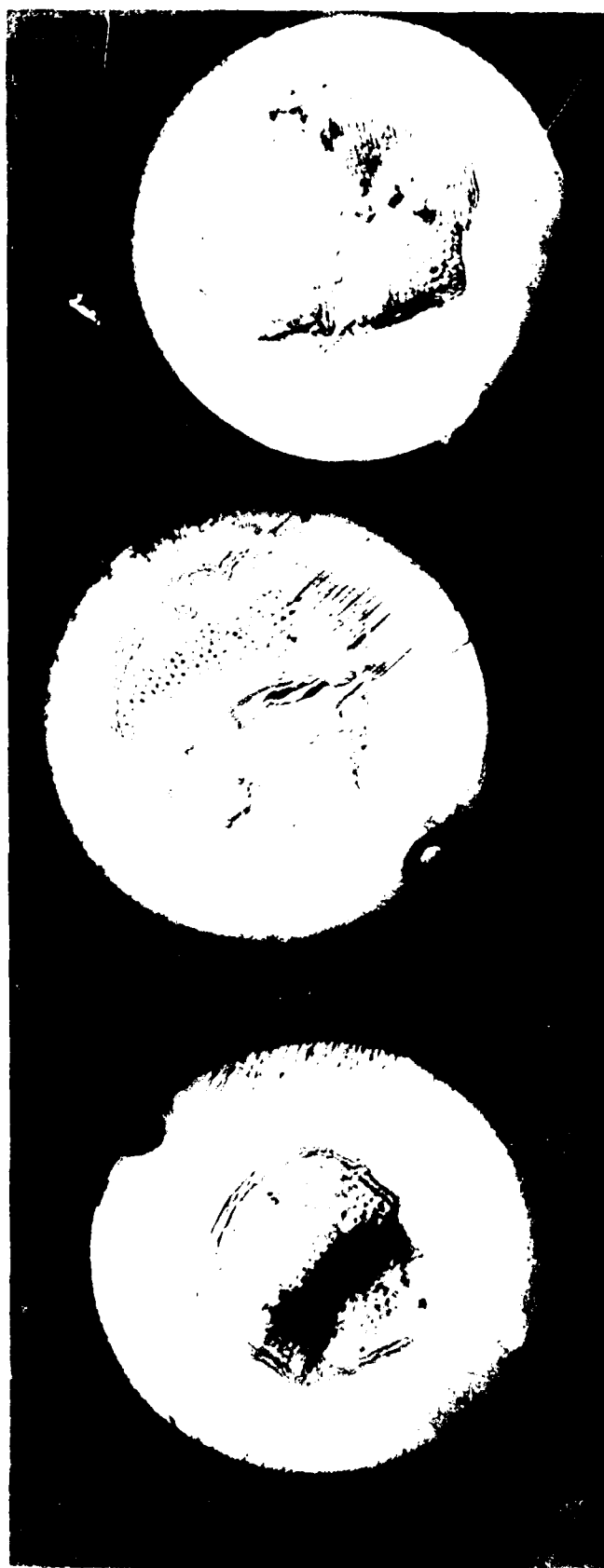


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Fig. 13  
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Fig. 13 (Cont'd)

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