AD-A158 444 ONE STEP INTERNAL TIN NB3SN SUPERCONDUCTOR FABRICATION (U) OXFORD SUPERCONDUCTING TECHNOLOGY CARTERET NJ W MARANCIK MAR 85 AFWAL-TR-85-2007 F33615-83-C-2324 1/1

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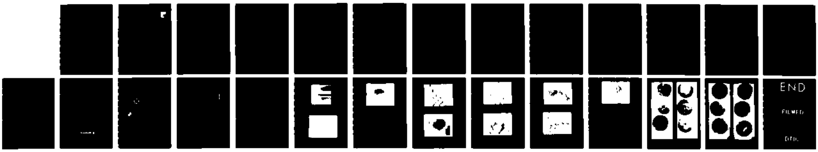

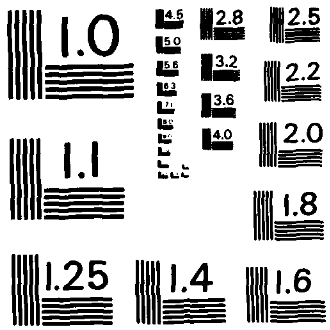

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ONE-STEP INTERNAL-TIN Nb_3Sn SUPERCONDUCTOR FABRICATION

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MARCH 1985

FINAL REPORT FOR PERIOD JUNE 1983 - AUGUST 1984

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

A two (2) inch diameter extrusion was packed with about 2,000 filaments by a novel technique.

Niobium Tin plated copper wires, each .010 inch diameter, were co-wound into a single layer along solenoid with copper foil on both the inside and outside. This was compacted and slit longitudinally to produce a ribbon with .010" Nb wires separated by 0.010" dia. Cu wire.

This configuration was Jelley rolled into a cylinder with the Nb wires parallel to the axis. The Jelley roll was placed in an extrusion can extruded at room temperature and drawn. The composite could only be drawn through several dies before center burst occurred.

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FOREWORD

This work was an effort to produce a Nb_3Sn 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.

SUMMARY

This program investigated the feasibility of fabricating a Nb_3Sn 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.

TABLE OF CONTENTS

SECTION I

INTRODUCTION

The object of this research is to demonstrate the feasibility of producing a Nb_3Sn 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 cylindrial 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 lines 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_3Sn. 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 entral axis of the rod, is a result of the inhomogeneous force istribution in the die area during drawing. The effect is a function of rea reduction, die angle, configuration of the rod, and relative tensile trength of the various components making up the rod.

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

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

1) 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.

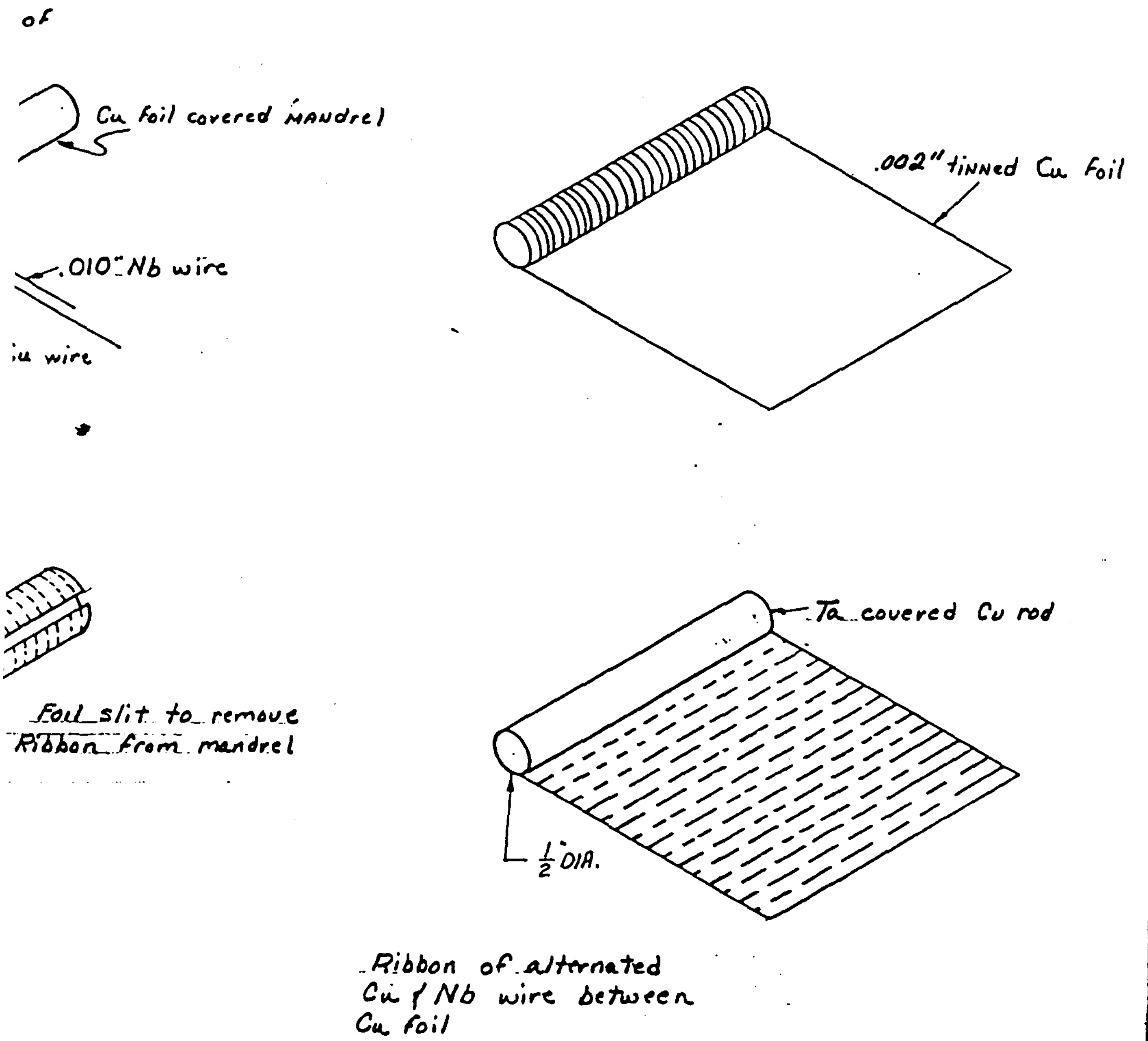
2) Change the geometery of the packed billet by eliminating the copper core and reducing the amount of copper on the outer surface.

3) 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.

LIST OF ILLUSTRATIONS



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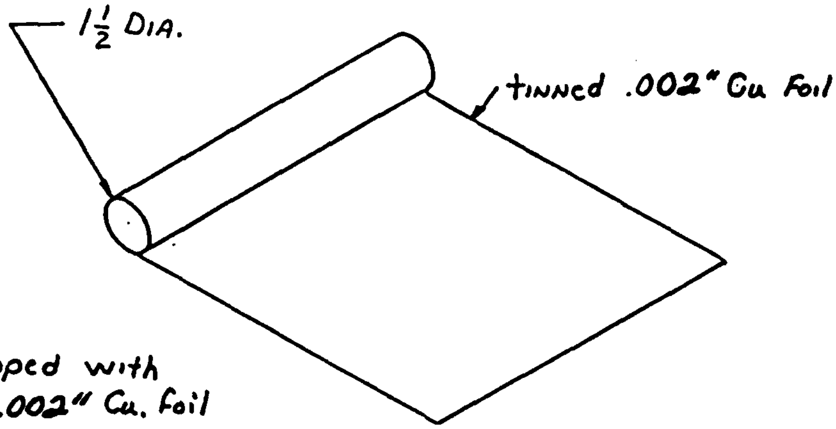
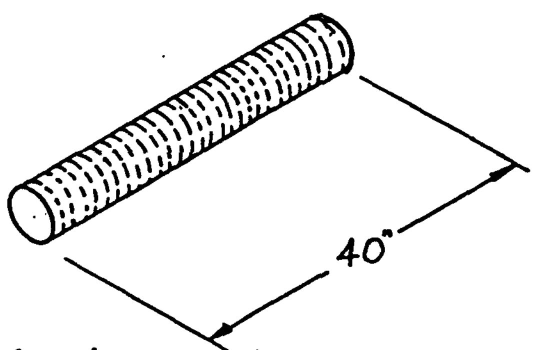
	QTY.	TITLE: Fig. 1 Single Extrusion Process for Multifilament TiN	
	TOLERANCES UNLESS OTHERWISE SPECIFIED		
	DEC ±	DWG	SIZE / DRAWING NO. / REV.
	FRACT ±	CHK	
	ANGLES ±	APPR	SCALE / SHEET OF



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



rnate Nb & tin plated Cu wire
tween 2 Layers of tinned .001"
foil (total foil thinkness of .008")
statioally comparted to remove
oids.

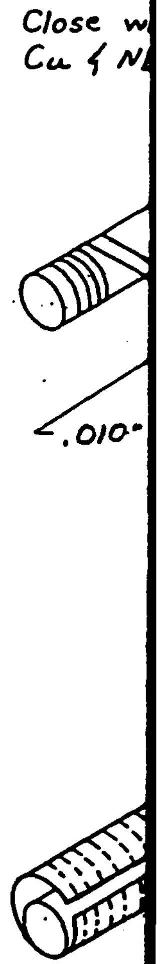
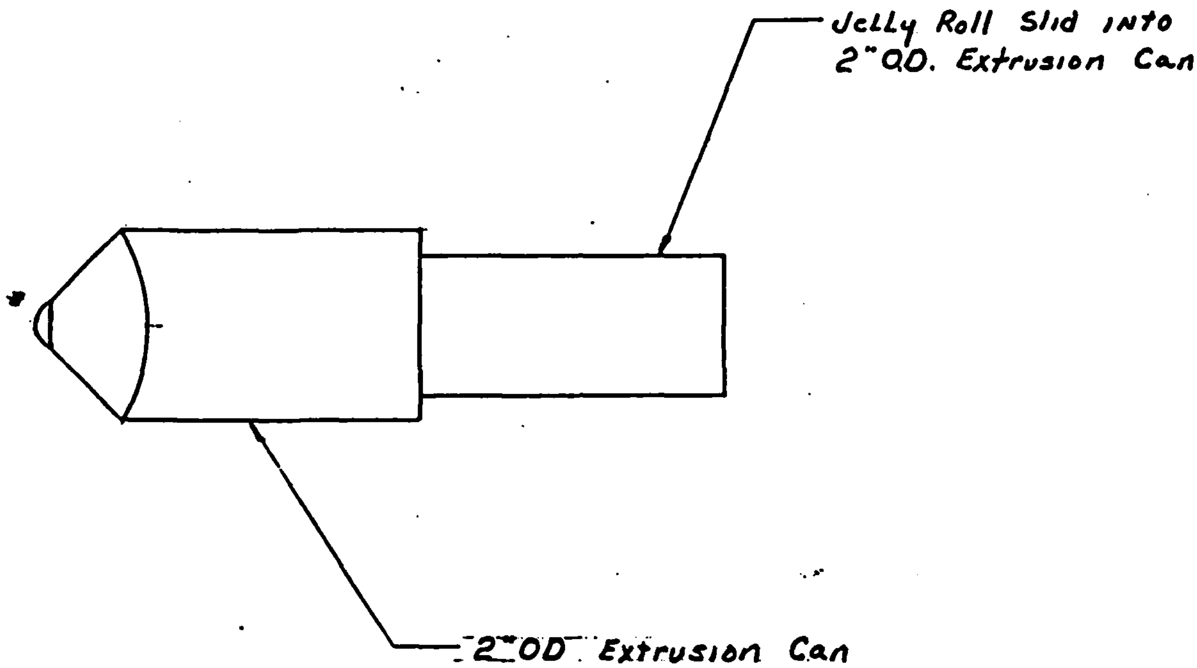


Fig 1.

ECN.	REV.	DESCRIPTION



The Can is extruded and drawn to FINAL size

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		QTY.	TITLE: Fig. 2 ASSEMBLY INTO EXTRUSION Can.				
		TOLERANCES UNLESS OTHERWISE SPECIFIED					
		DEC. ±	DWG.			SIZE	DRAWING NO. / REV.
		FRACT. ±	CHK.				
		ANGLES ±	APPR.			SCALE	SHEET OF

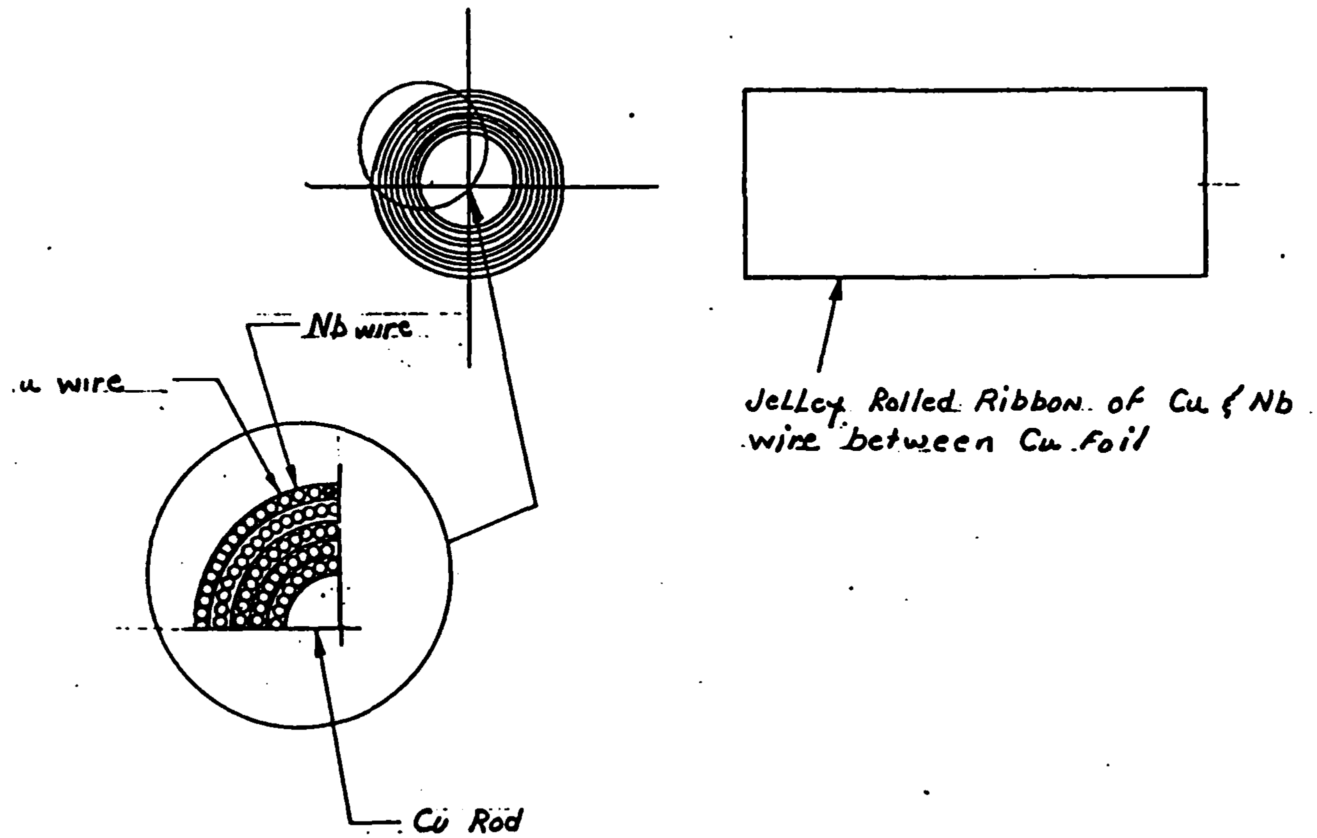


Fig 2.

ECN.	REV.	DESCRIPTION

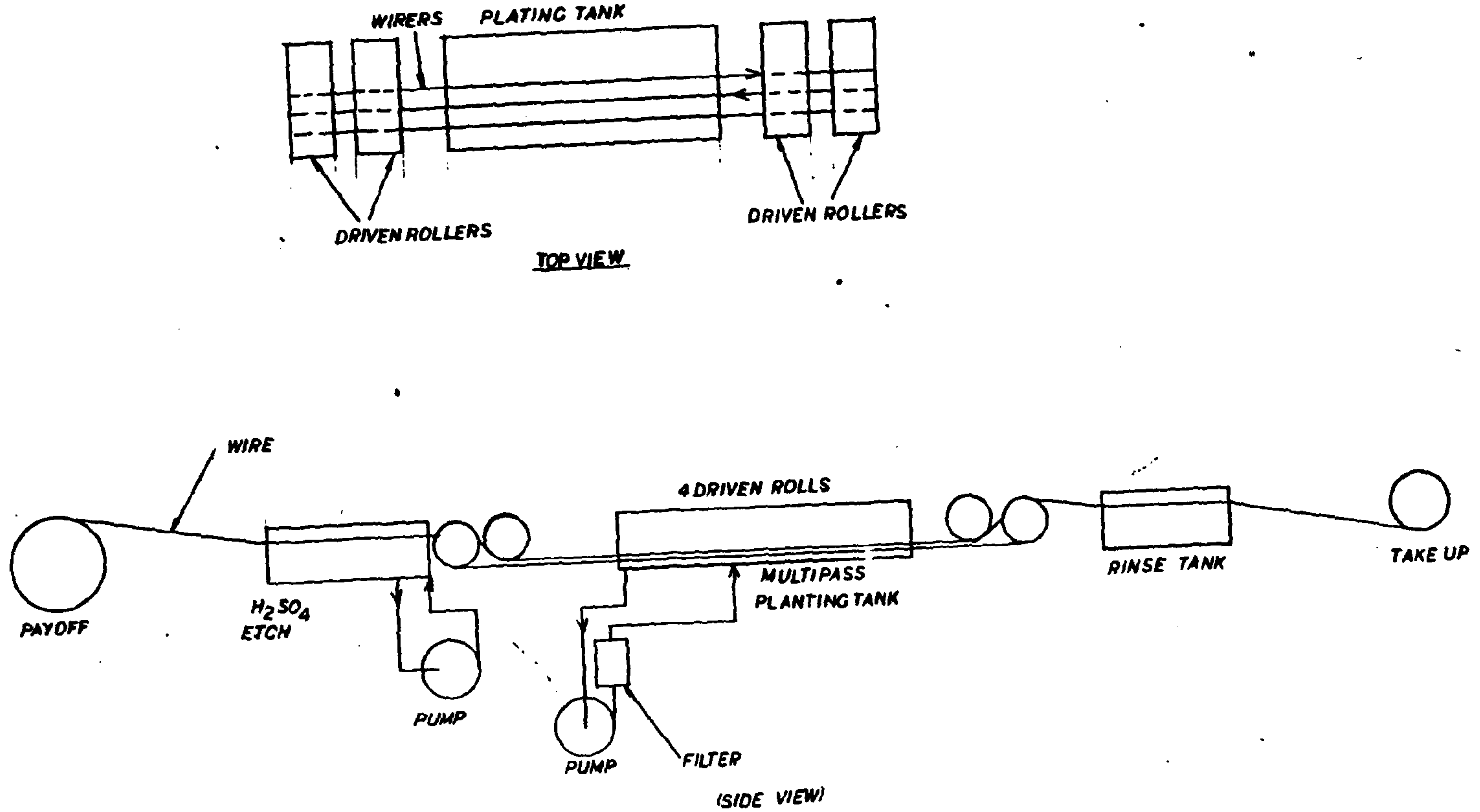


Fig 3

ECN	REV	DESCRIPTION	DATE	APPR

MATERIAL

QTY

TOLERANCES UNLESS OTHERWISE SPECIFIED:
DEC ±
FRACT ±
ANGLES ±

OXFORD AIRCO

TITLE: TIN PLATING LINE

DWG	LS	8-17-81	SIZE	DRAWING NO	REV
CHK					
APPR			SCALE	SHEET OF	

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

*Product of Lea Ronal, Freeport, New York

Fig. 4 Plating Solution

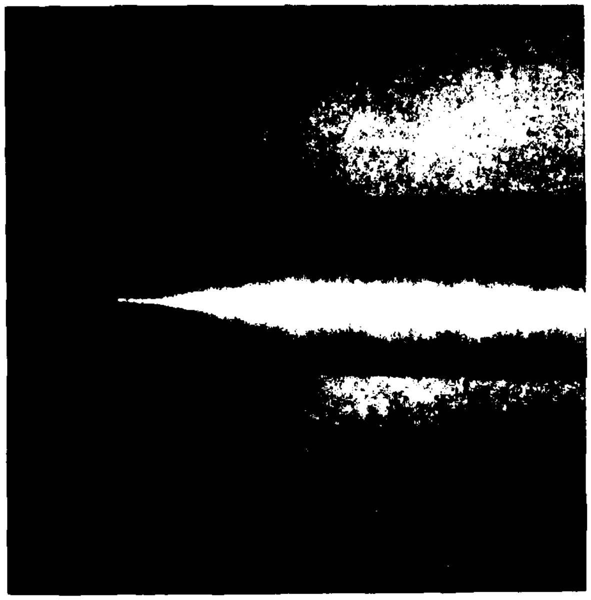


Fig. 5 Copper Clad Solenoid After Heating

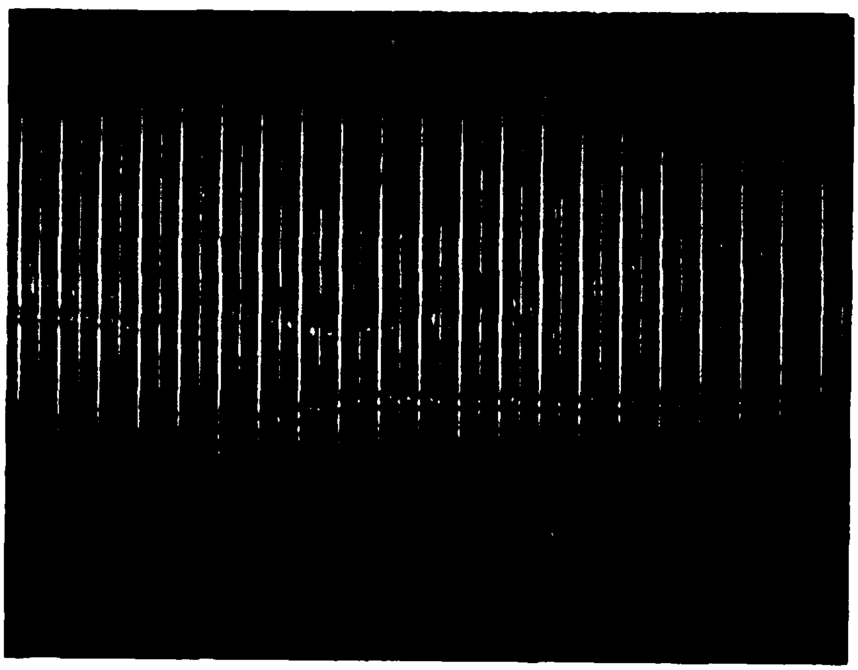


Fig. 6 10X Magnification Of Solenoid

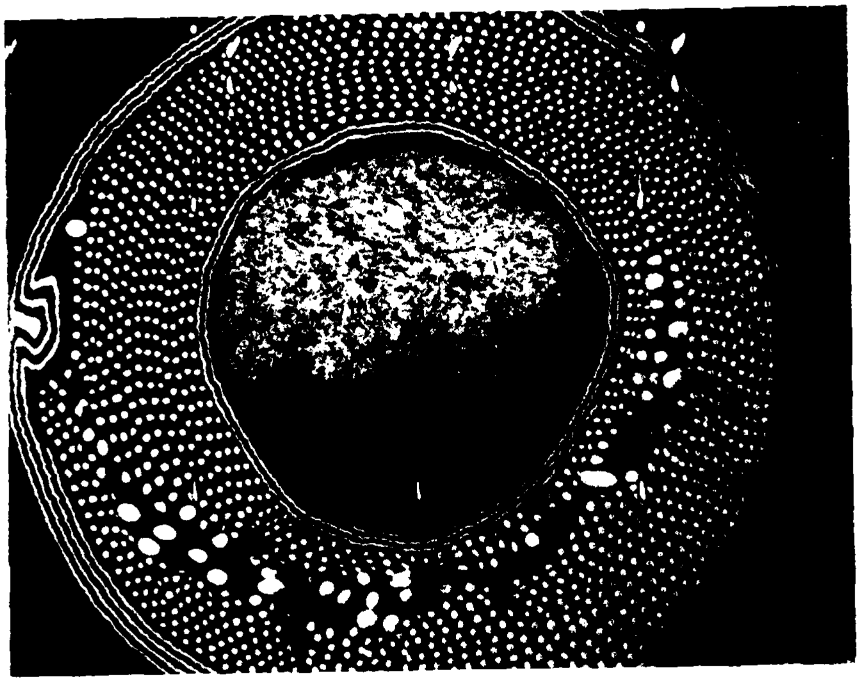


Fig. 7 Extruded Billet Showing

Filament Size Distribution

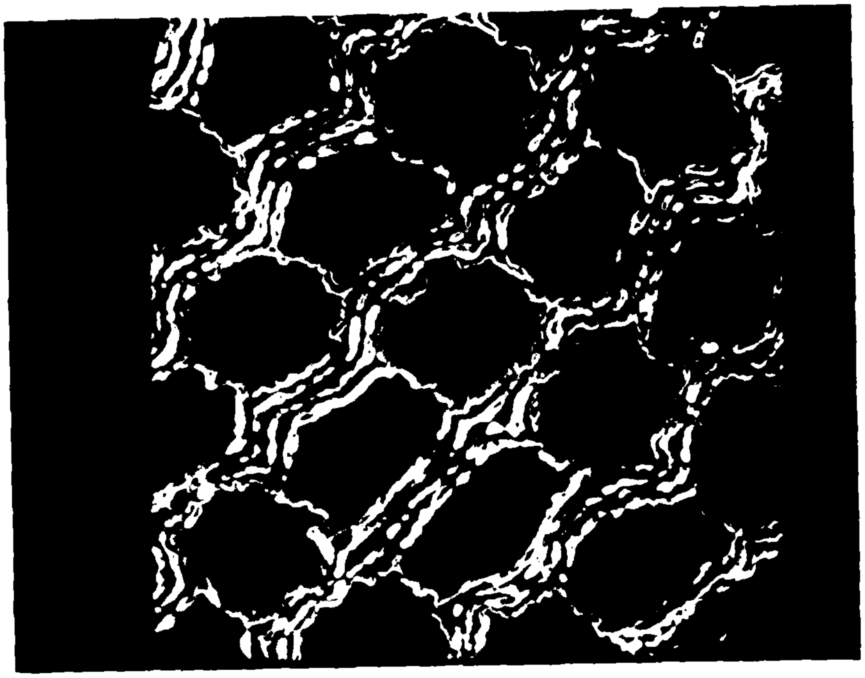


Fig. 8 200X

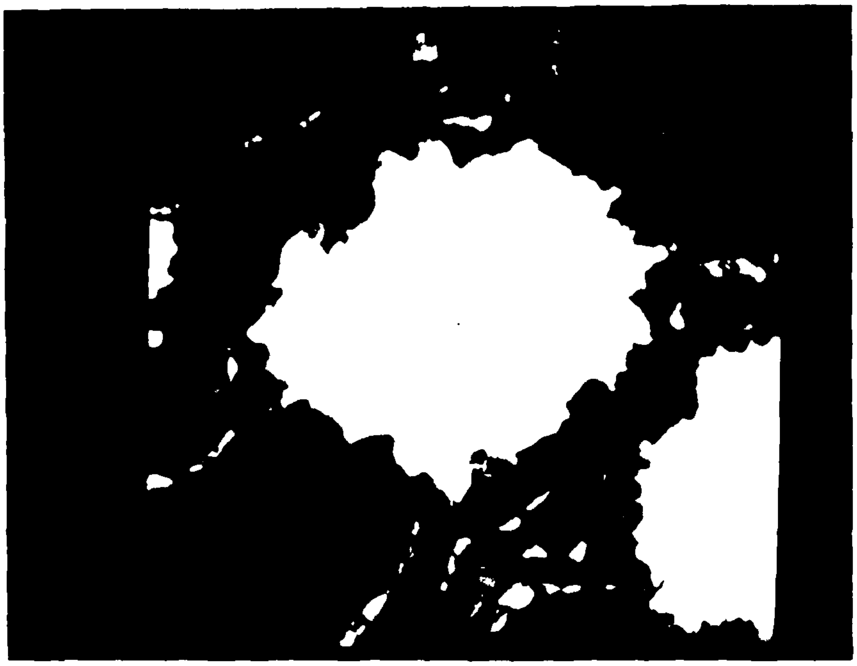


Fig. 8a 480X

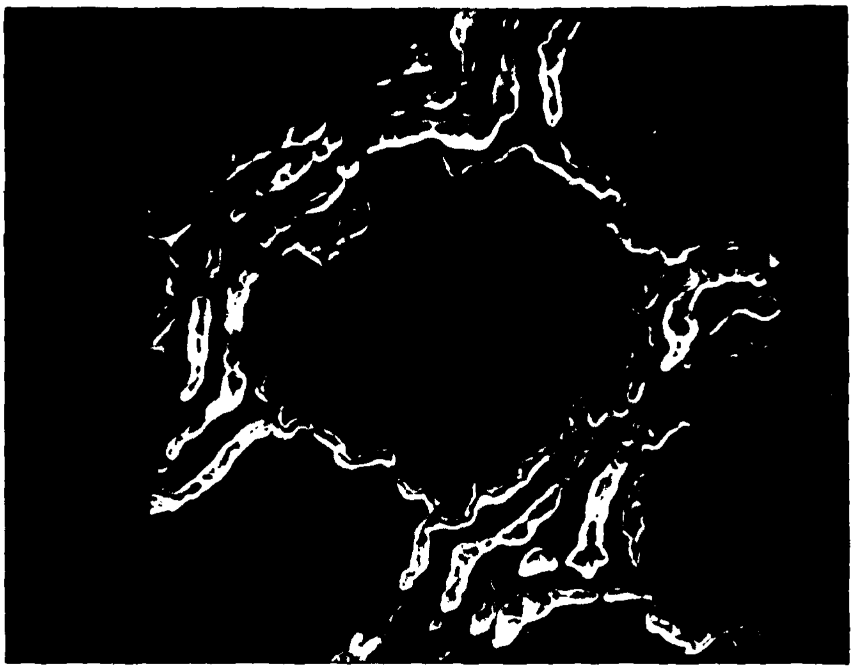


Fig. 9 480X

Fig. 10 300X

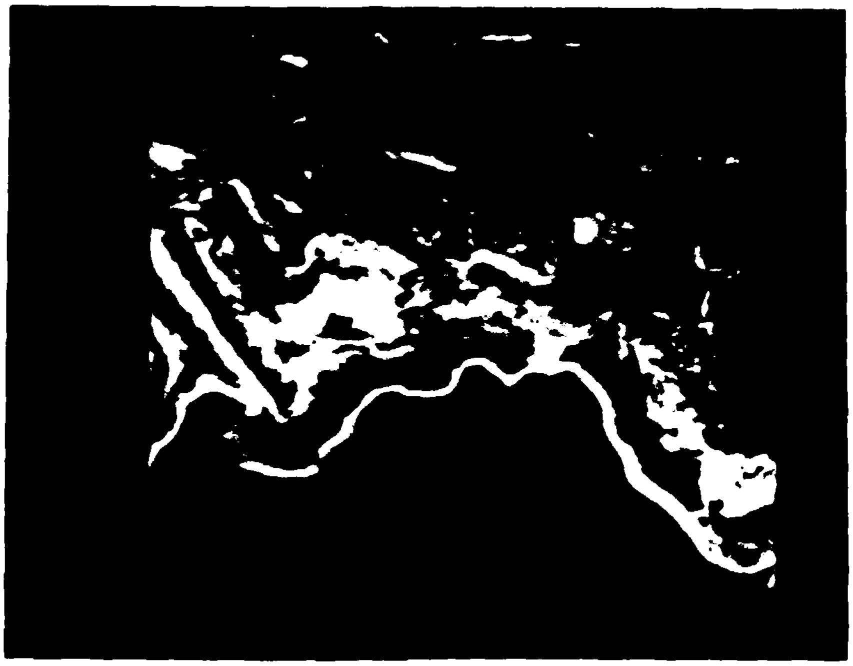


Fig. 10a 1500X

Fig. 11 800X

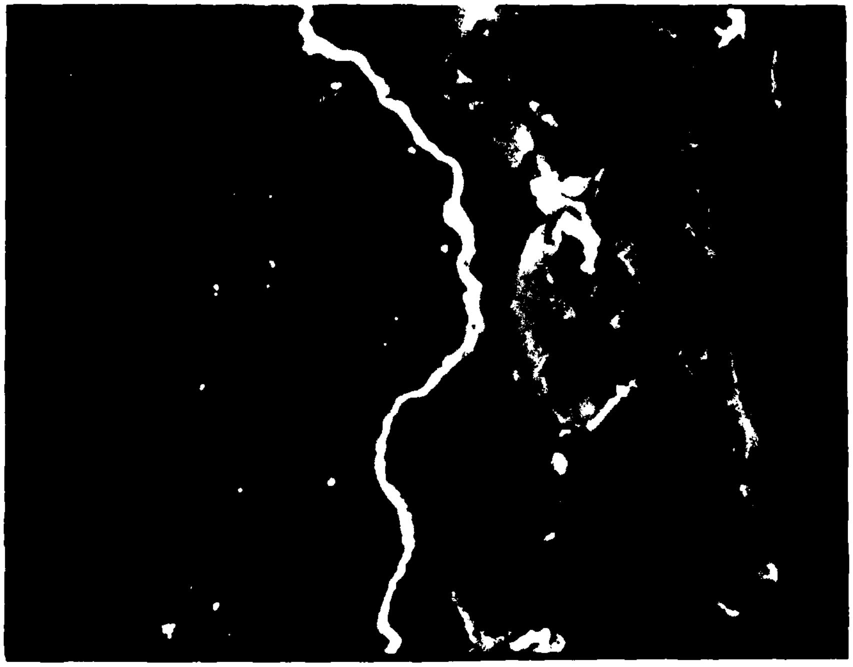


Fig. 12   2000X

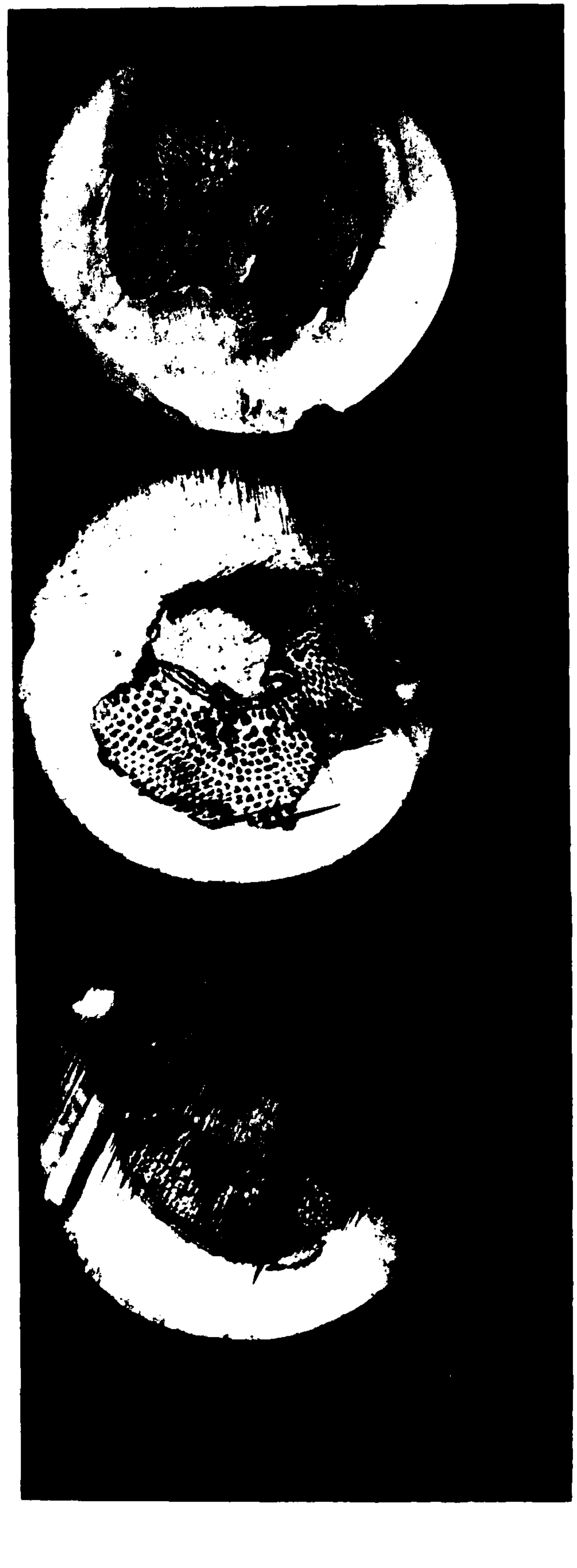
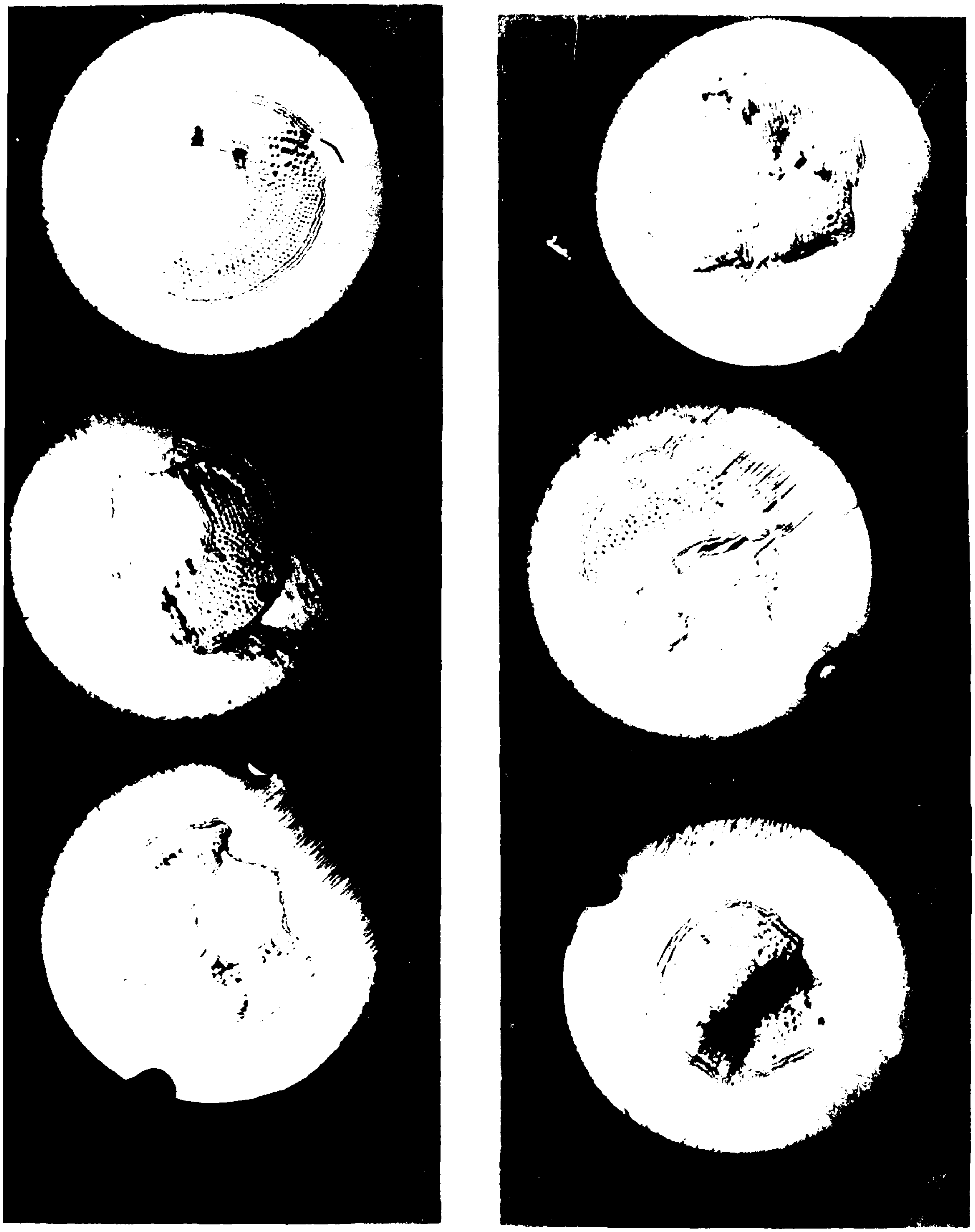


Fig. 13



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11

12

Fig. 13 (Cont'd)

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