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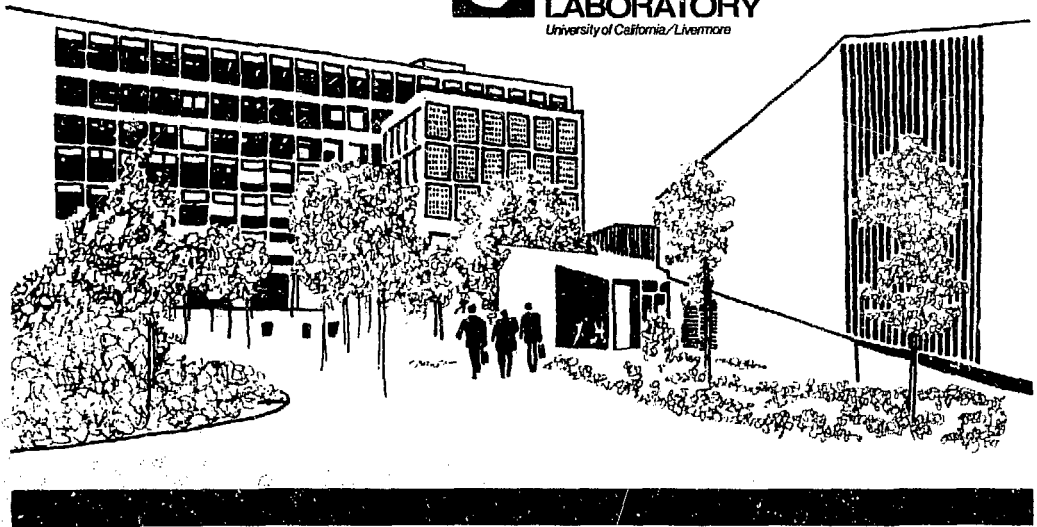
# **SUPERCONDUCTING MAGNET DEVELOPMENT PROGRAM PROGRESS REPORT:**

**July 1974 -- June 1975**

D.N. Cornish  
A.R. Harvey  
R.L. Nelson  
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October 24, 1975

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# SUPERCONDUCTING MAGNET DEVELOPMENT PROGRAM PROGRESS REPORT:

July 1974 – June 1975

## Abstract

During FY 1975, the superconducting magnet development program at the Lawrence Livermore Laboratory was primarily directed toward the development of multifilamentary  $Nb_3Sn$  conductor for large CTR machines. It was secondarily concerned with preliminary work for the MX experiment

and with the acquisition of additional testing facilities. Among the significant achievements was the construction and operation of a 27-cm-bore coil to its short-sample limit of 7-T at the windings. The coil was wound with a 100-m length of 67,507-filament  $Nb_3Sn$  conductor.

## Introduction

The main effort of the superconducting magnet development program at Lawrence Livermore Laboratory (LLL) has been directed towards the development of multifilamentary  $Nb_3Sn$  conductors for the magnets in large CTR machines. This work started in mid-FY 1974 with a supplemental appropriation of \$150 thousand that was used to initiate work by three superconductor manufacturers on three different methods for producing multifilamentary  $Nb_3Sn$ . The results were encouraging, and considerable progress was made during FY 1975 in developing practical fabrication methods.

This report describes the conductors which were produced as well

as the difficulties encountered with some of the processes. A significant achievement was the construction and operation of a 27-cm-bore coil to its short-sample limit of 7 T at the windings. The coil was wound with a 100-m length of AIRCO<sup>\*</sup> 67,507-filament conductor.<sup>†</sup> Also, it was demonstrated

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<sup>\*</sup>Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Energy Research and Development Administration to the exclusion of others that may be suitable.

<sup>†</sup>Just past the close of the report period, a second 27-cm-bore coil was wound with 300 m of this conductor and was operated to its short-sample limit of 10 T.

that the current densities required by fusion machines can be obtained at high fields, and that scaling superconducting properties to conductors with large cross sections is a straightforward process.

The report includes a brief discussion of the remainder of the program, which was largely concerned with preliminary work for the MX experiment and with the design and building of additional testing facilities.

## Nb<sub>3</sub>Sn Conductor Development

### FABRICATION

Contracts for continued multifilamentary Nb<sub>3</sub>Sn conductor development were given to three domestic superconductor manufacturers:

- Contracts to AIRCO, Inc., Murray Hill, NJ, for developing an external bronze method totaled approximately \$112,000 for FY 1975. (Reproductions of progress reports covering the period April 12, 1975 through July 1 are included as Appendices A and B to this report.)
- Intermagnetics General Corporation (IGC), Guilderland, NY, has received contracts to produce multifilamentary Nb<sub>3</sub>Sn superconductor based on an external Sn-diffusion process. In FY 1975 these contracts amounted to approximately \$83,000. (A reproduction of the IGC final report dated October 17, 1975, is included as Appendix C to this report.)

- The third superconductor manufacturer, Supercon, Inc., Natick, MA, is working on an internal bronze approach. The contracts with Supercon totaled approximately \$25,000 for FY 1975.

### AIRCO Superconductor

The metal-working steps in the external bronze approach being developed by AIRCO can be summarized as follows:

1. A composite billet consisting of 19 Nb rods embedded in a bronze matrix is prepared. Typically, the billet is about 23 vol% Nb.
2. This billet is extruded and then drawn to a hexagonal cross section. Dimensions across the flats are on the order of 0.25 in.
3. The hexagonal composite is sectioned and 187 of these lengths are assembled in a Ta-lined OFHC Cu extrusion can. Bronze fillers are used inside the Ta liner.
4. This assembly is extruded and drawn to a hexagonal cross section.

5. The second-stage extrusion product is assembled in an OFHC copper extrusion can. Strips of OFHC Cu are used inside the can to pack the assembly.

6. The billet is extruded, drawn, twisted, and rolled to the final dimensions.

The product of Step 4, a Ta liner around 3553 Nb filaments in a bronze matrix, is the basic building block for all conductors.

Table 1 lists parameters for three conductors nominally designed for 1, 3.5, and 10 kA at 12 T. In all cases, the bronze was 10 wt% Sn and the filaments are approximately 5  $\mu\text{m}$  in diameter.

After the conductor has been drawn and rolled to its final shape, it is heat treated at 650°C for up to 120 hours. During this time, Sn diffuses from the bronze to the surface of the Nb filaments, where it reacts to form a layer of  $\text{Nb}_3\text{Sn}$ . The Ta liner acts as a barrier preventing Sn from diffusing into the OFHC Cu, where even very small quantities

would seriously degrade its conductivity.

Figure 1 is a scanning electron micrograph of a conductor after the surface has been etched. The Ta liner surrounds the bronze matrix in which are embedded 3553 Nb filaments. The Sn in the bronze has reacted with the Nb to form a  $\text{Nb}_3\text{Sn}$  layer 1 to 2  $\mu\text{m}$  thick. The high-purity Cu can be seen between adjacent Ta liners.

Figure 2 shows the results of a microprobe scan on the 1-kA conductor.

Figures 3 through 5 are photomicrographs of sections of the conductors in Table 1. Further details are reported in Refs. 1 and 2.

AIRCO has also adapted an existing sheathing technique to clad a length of the 67,507-filament material with a layer of 304 stainless steel. Although done to demonstrate that it could be used to apply strengthener to the conductor, this process can also be used to add Cu stabilizer.

Thus far, AIRCO has supplied two lots of unclad superconductor containing 67,507 filaments. The first

Table 1. Parameters of AIRCO  $\text{Nb}_3\text{Sn}$  conductors.

Conductor configurations	No. of filaments	Twist pitch (mm)	Short-sample current at 12 T (A)	Cu (%)	Dimensions (mm)
19 (19 × 187)	67,507	30	1,593	34	5.0 × 1.68
73 (19 × 187)	259,369	60	4,500	50	9.3 × 3.9
187 (19 × 187)	664,411	98	11,500	39	13.75 × 6.0

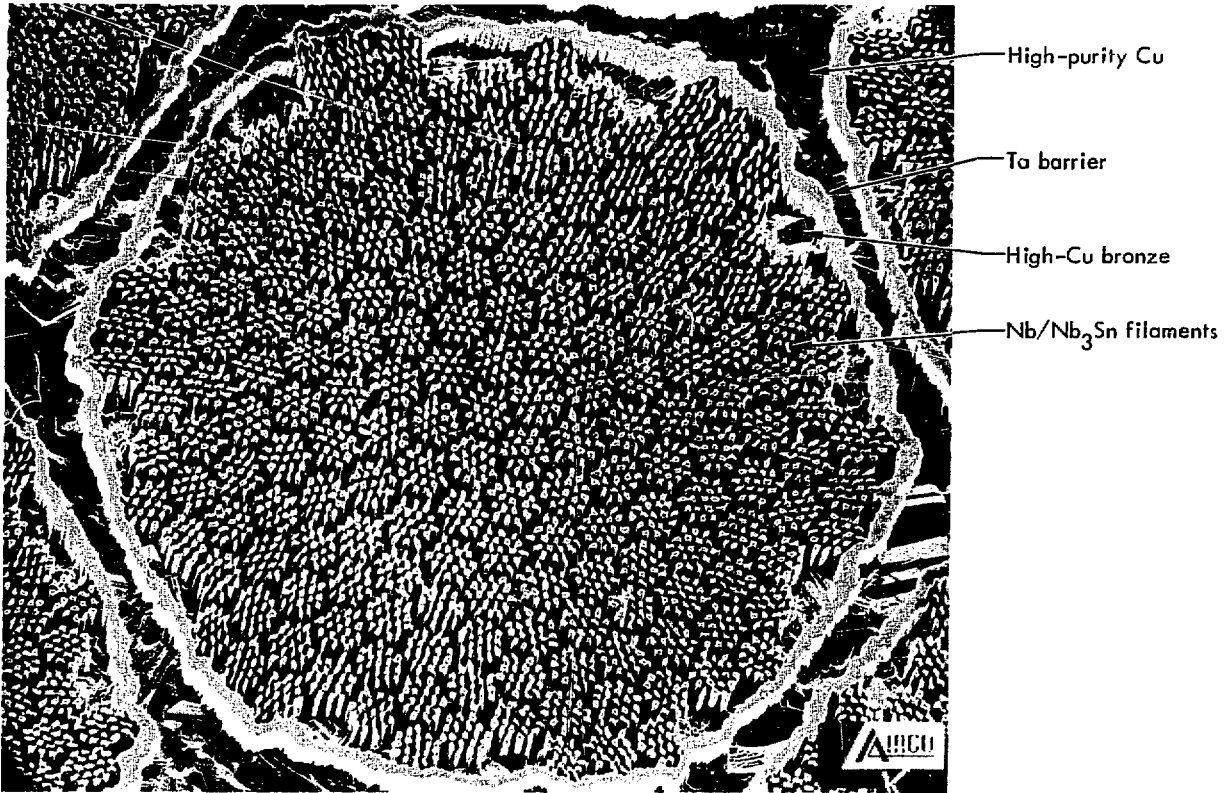


Fig. 1. Scanning electron micrograph of etched conductor section.



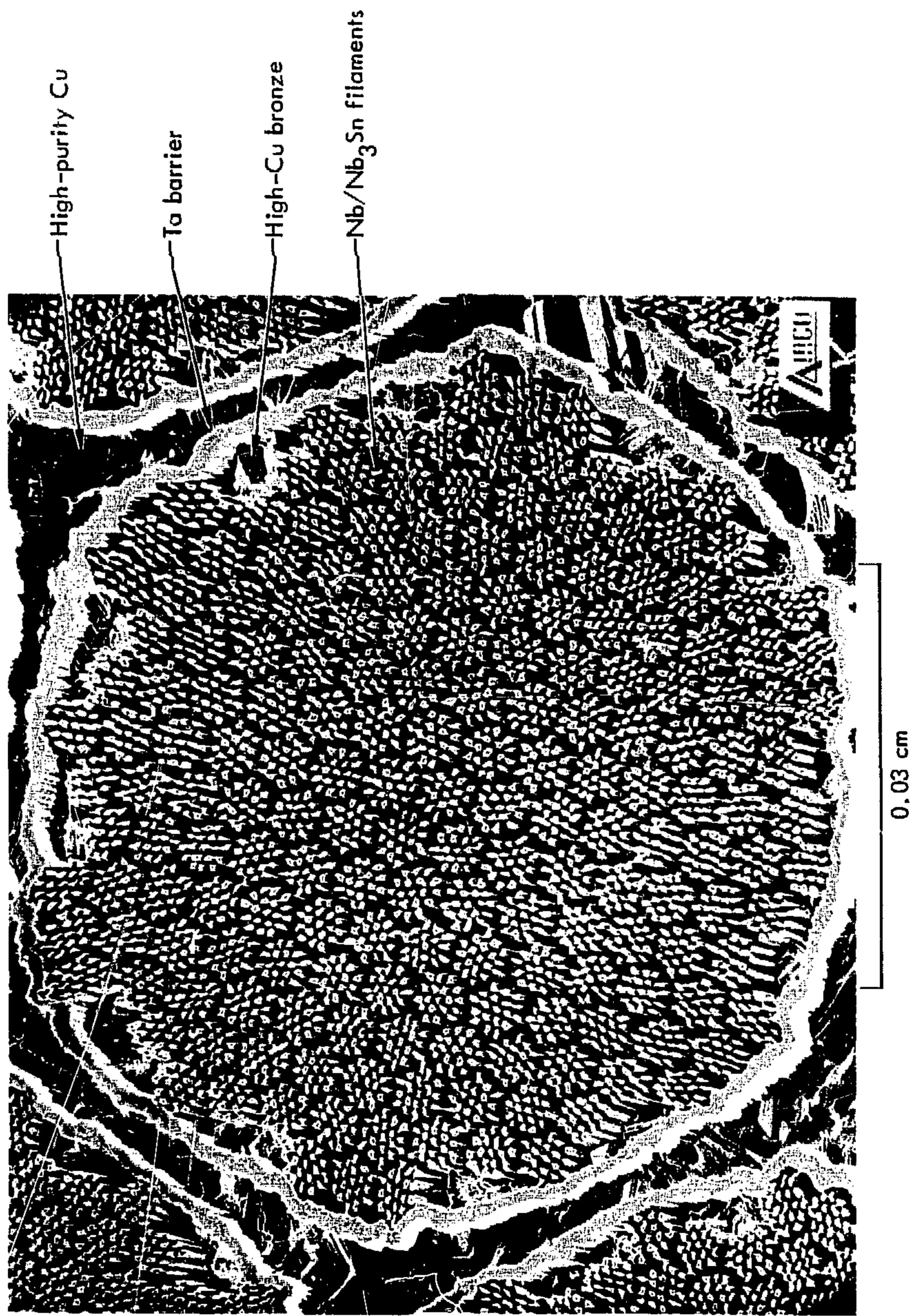


Fig. 1. Scanning electron micrograph of etched conductor section.

shipment was 325 ft long, and the second had a length of 1000 ft. An

additional 625 ft of 67,507-filament superconductor clad with stainless steel has also been received.

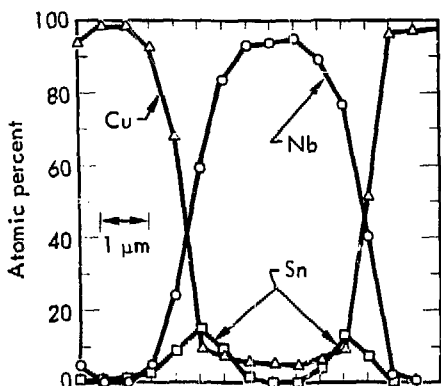
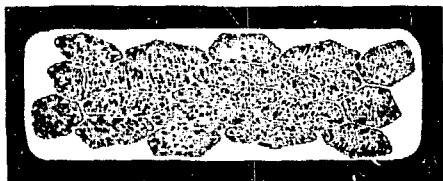


Fig. 2. Results of microprobe scan of the 67,507-filament material.

### IGC Superconductor

The process being developed by IGC involves extruding and drawing a billet that consists of Nb rods embedded in a Cu matrix. The metalworking steps in this process can proceed more rapidly than the bronze approach, for example, because the rate of work hardening in Cu is much less than in bronze. A layer of Sn is plated on the composite after it has been reduced to the final dimension. A homogenization treatment at approximately 580°C is followed by a 750°C heat treatment that produces the Nb<sub>3</sub>Sn reaction on the outer surfaces of the Nb filaments.

The resultant strand, which contains on the order of 250 filaments, is the basic unit in IGC's approach to producing high-current conductors for CTR magnets. Several hundred of



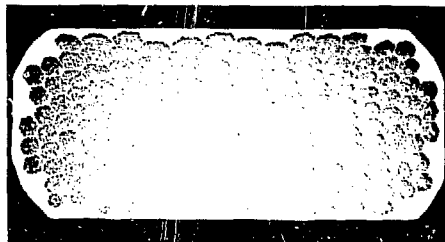
14X

Fig. 3. Photomicrograph of 67,507-filament, 1-kA conductor.



8.5X

Fig. 4. Photomicrograph of 259,369-filament, 3.5-kA conductor.



5X

Fig. 5. Photomicrograph of 664,411-filament, 10-kA conductor.

these strands will be cabled together to produce the desired high-current conductor. A strand diameter of approximately 0.25 mm is needed to make this approach economically feasible. This process was described in detail at the 1974 Applied Superconductivity Conference.<sup>3</sup>

As pointed out in Ref. 3, problems were encountered in the production of these superconducting strands. In the early stages of the program, the strand diameter was approximately 0.12 mm, and there appeared to be no production problems. However, when the process was scaled up to produce the necessary 0.25-mm-diam strands, a severe "crusting" problem was encountered: during the 580°C homogenization treatment, the outer layer of the strand physically separated from the body of the conductor. The separation did not occur at the Sn-Cu interface, but some distance in from this boundary.

IGC's effort in FY 1975 consisted mostly of metallurgical investigations to explain this phenomenon and to determine what processing steps should be changed or added to circumvent the problem. In an interim report submitted to LLL, IGC showed progress in determining the reason for the crusting problem and suggested ways of overcoming it.

In addition to this metallurgical work, a contract was given to IGC to

supply a 3-m-long cable consisting of 343 strands so that LLL could do some preliminary short-sample testing on cabled conductor. The strand diameter will be on the order of 0.18 mm; therefore, no crusting problems should be encountered with the conductor.

#### Supercon Superconductor

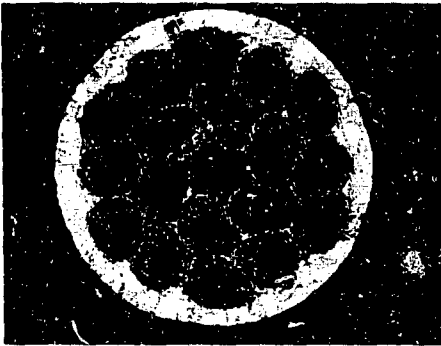
The third superconductor manufacturer, Supercon, is developing an internal bronze approach for producing multifilamentary Nb<sub>3</sub>Sn. This approach is similar to that used by AIRCO except that here, Nb tubes filled with bronze are embedded in an OFHC Cu matrix. This composite can then be subjected to a series of extrusion, drawing, rebundling, twisting, and rolling operations to produce a composite that consists of many filaments. Subsequent heat treatment causes the Sn to diffuse out of the bronze to the inner surfaces of the Nb tubes. The Nb tubes themselves act as barriers to Sn diffusion. Photomicrographs of a 7600-filament conductor processed in this way are shown in Fig. 6. The filament diameter is on the order of 20 μm and the thickness of the reaction layer is about 2 μm. The details of this process were presented at the 1974 Applied Superconductivity Conference.<sup>4</sup>

Supercon, although it is one of the world's largest superconductor manufacturers, is extremely dependent on other firms, in particular those that supply the starting materials. Because the suppliers of the bronze and Nb materials were unable to meet their delivery schedules, Supercon has not yet been able to complete the fabrication of the conductor.

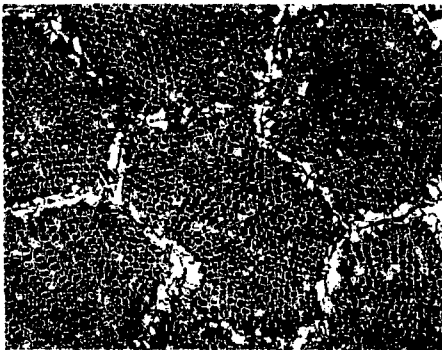
## SHORT-SAMPLE TESTS

Critical-current measurements were performed on the specimens supplied by AIRCO. For these measurements, we used a newly-assembled short-sample tester (see p. 14). The measurements were taken by first setting the magnetic field at the desired value and then slowly increasing the current through the specimen. The current was supplied by a filtered, but coarsely regulated, power supply, and was measured with a 100-mV, 10-kA shunt connected in series with the specimen.

A drawing of the sample holder is shown in Fig. 7. The sample is



20X



70X

Fig. 6. Photomicrographs of 7,600-filament conductor.

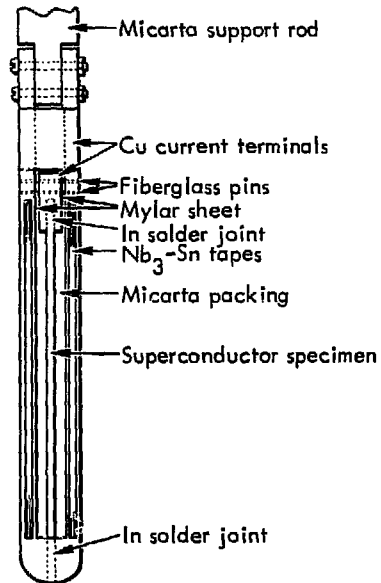


Fig. 7. Sample holder for short-sample tests.

carefully soldered into the upper current terminal with In solder. This assembly is then inserted into the body of the sample holder, and the upper terminal is pinned in position with the fiberglass pins. The lower connection is then made, also with In solder. Because the assembly is lowered into the magnetic field through a radial access port, we can change specimens without removing the magnet. The Micarta packing prevents the specimen from excessive deflection due to the Lorentz interaction between the transport current and the applied magnetic field.

Figure 8 contains unretouched tracings of several current-voltage characteristics at various field values. The noise peaks probably all result from motion of the sample in

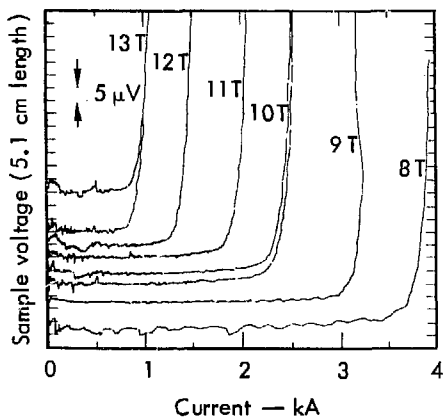


Fig. 8. Voltage vs current traces on test sample (67,507 filaments, 4.2 K,  $H_{||}$ ).

the magnetic field; however, it is still fairly easy to resolve voltage changes on the order of 0.25 to 0.5 μV. For the 67,507-filament specimens supplied by AIRCO, which have an overall area of 0.08 cm<sup>2</sup>, this voltage corresponds to a resistivity of  $1 \times 10^{-14}$  Ω-m for a 4000-A current. Resolution in resistivity measurement of this order of magnitude is needed to estimate accurately the resistance of coils of the size that will be needed for large CTR magnets.

The results of these current-voltage-field measurements are presented in the plots of critical current vs field shown in Figs. 9

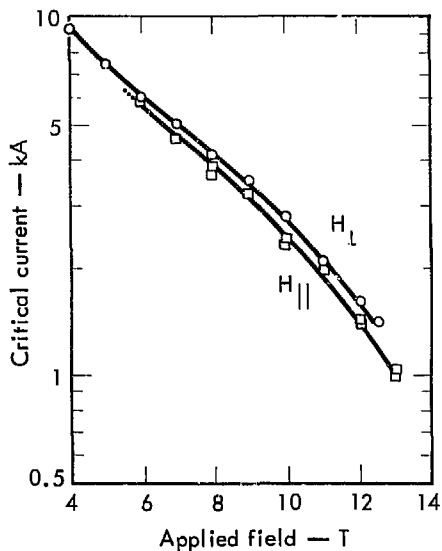


Fig. 9. Critical current of 1-kA conductor (67,507 filaments,  $5.1 \times 1.7$  mm, 34% Cu, 4.2 K).

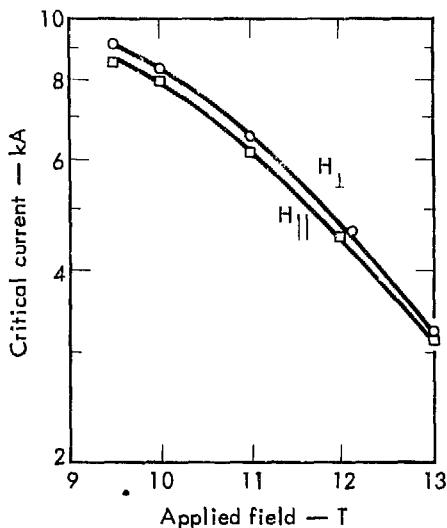


Fig. 10. Critical current of 3.5-kA conductor (259,629 filaments,  $9.3 \times 3.9$  mm, 50% Cu, 4.2 K).

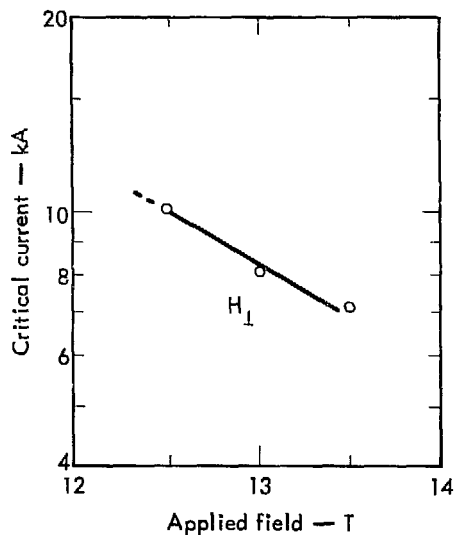


Fig. 11. Critical current of 10-kA conductor (664,411 filaments,  $13.9 \times 6$  mm, 34% Cu, 4.2 K).

through 11. In these plots, the critical current at a certain field value is that current that causes a 50- $\mu$ V drop across the specimen length of 5.1 cm. While this is a very large voltage drop, it is consistent with earlier, more insensitive measurements. More sensitive measurements should be possible when the new 10-kA regulator, described later, has been installed.

The final short-sample curve, Fig. 12, is a H-I curve normalized to the value of the critical current at 12 T for a range of sample sizes. The field dependence of the critical current is thus seen to be independent of conductor size.

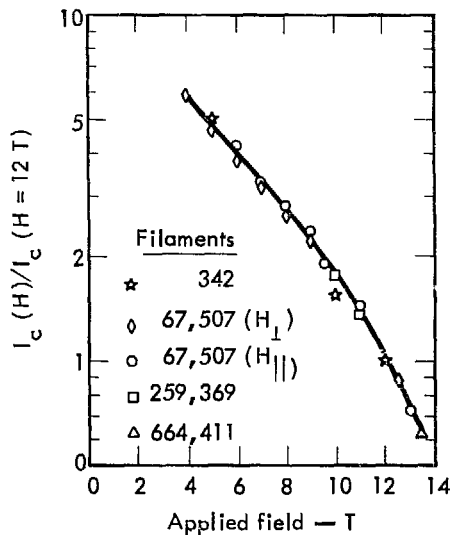


Fig. 12. Critical current normalized to that at 12 T (4.2 K).

## COIL TESTS

Thus far, we have tested a coil wound from the AIRCO-supplied 67,507-filament material. The coil parameters of this coil are:

Inner diameter	27.46 cm
Outer diameter	29.18 cm
Winding length	14.22 cm
Conductor length	100 m

The winding mandrel was fabricated from 6061-T6 Al pipe. Insulation was provided by covering 50% of the conductor surface area with a spiral wrapping of  $0.64 \times 0.06$ -mm Mylar. In addition, layer-to-layer insulation was provided by strips of epoxy fiberglass placed 0.64 cm apart. Mechanical reinforcement was provided by over-wrapping the coil with approximately 570 turns of 0.10-cm-diam 302 stainless steel with a tension of 6.9 N. The test coil, shown in Fig. 13, was inserted inside a Nb-Ti

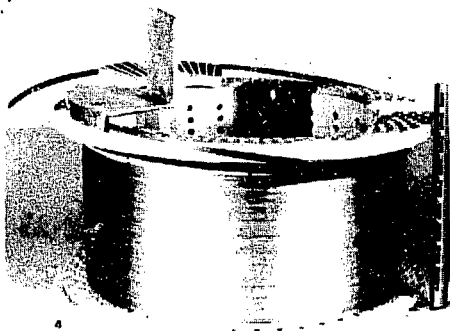


Fig. 13. Photograph of  $\text{Nb}_3\text{Sn}$  test coil.

coil capable of producing a background field of 5 T. With both coils energized, a peak field of 7.5 T could be obtained.

The resistance of the coil was monitored during cool-down. The resistance ratio  $R_{300\text{K}}/R_{20\text{K}}$  was found to be approximately 100, indicating that the Ta diffusion barriers were still intact and that they prevented Sn from contaminating the OFHU Cu. Charging-rate measurements indicated a coil inductance of 2 mH.

The results of the coil tests are summarized in Fig. 14. The cryogenic stability limit was calculated using the magnetoresistance data of Fickett<sup>5</sup> and a heat-transfer coefficient of  $0.4 \text{ W/cm}^2$  between the conductor and the liquid-He bath. The short-sample quench limit was obtained by using

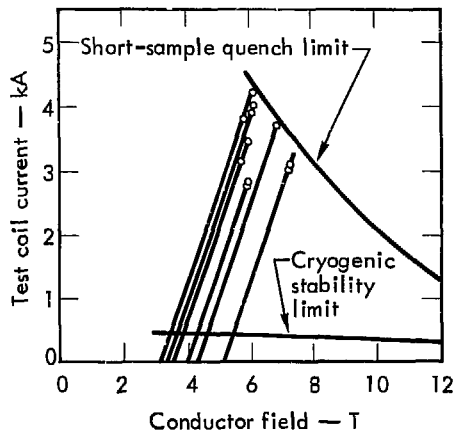


Fig. 14. Quench performance of test coil.

Fig. 10 to extrapolate the critical current measured at 6 T.

After the desired background field had been reached, the Nb<sub>3</sub>Sn test coil was charged with 20 to 50 mV. In some cases, the coil was charged incrementally; in other cases, it was

charged continually. The manner of charging appeared to have little effect on coil performance. As the data on Fig. 14 show, there was considerable "training," but the coil performed almost to the short-sample limit.

## Insulation Development

Inter-turn insulation for the Baseball II experiment was difficult to obtain, costly to fabricate, and extremely time-consuming to apply. For these reasons, we are endeavoring to develop a more proficient scheme for the upcoming magnets well in advance of their design and construction.

The anticipated requirements for this insulation present problems not readily resolved, to wit:

- The insulation must provide a network of equally spaced disks that will give approximately 50% bearing area between conductors and, in the remaining space, allow for adequate surface cooling of the conductor.
- Since the conductor must bend about two mutually perpendicular axes, the disks and their carrier must be flexible in two directions.
- The insulation materials cannot be identified without proper

testing with respect to their suitability in a cryogenic, neutron-radiation environment.

- Relatively short runs of the insulation will be required for test magnets.

Because we may not be able to obtain suppliers for this type of insulation we are designing fabrication machinery for its production. This will provide us with the flexibility of either producing short runs in-house or providing the machinery for fabrication by industry.

Our current concept of the insulation is a notched film carrier on which the disks are affixed. First impressions of appropriate machinery brought visions of a rather complex index-transfer device. Upon further investigation, however, we have developed a scheme to produce this result with a few selected punches operating in a given sequence. We have further discovered that a number of different patterns are available



using this technique. A machine incorporating a commercially available power punch could have adjustable punches to provide a wide variety of widths and patterns. A

change from one width and pattern to another could then be made quite economically. To verify the feasibility of this approach, one such strip has been hand fabricated.

## Preliminary Design of Superconducting Coils for the MX Experiment

MX is a plasma physics experiment being considered as an intermediate step between Baseball II and the fusion engineering research facility (FERF).<sup>6</sup> The main parameters of the magnetic field, which are determined by plasma conditions, are as follows:

Central (vacuum) field	2 T
Mirror field	4 T
Maximum field at conductor	7.5 T
Mirror-mirror length	3.4 m

The precise shape of the Yin-Yang magnets is also dependent on factors such as magnetic "well" depth, injector access, etc., and these studies are still being carried out. However, the main magnet parameters have been established and are not expected to change significantly:

Major radius	2.5 m
Minor radius	0.78 m
$\Delta Z$ (coil displacement)	0.78 m
Coil section	$0.28 \times 0.84$ m
Total stored energy	500 MJ
Current density	$3400 \text{ A/cm}^2$ .

Forces are a major problem. A field of 7.5 T exerts a pressure of more than 220 atm, most of which will be transmitted through the windings to a support structure outside the coil case. Figure 15 illustrates the type of structure we envisage.

Detailed design of the superconducting winding and conductors is now in hand.

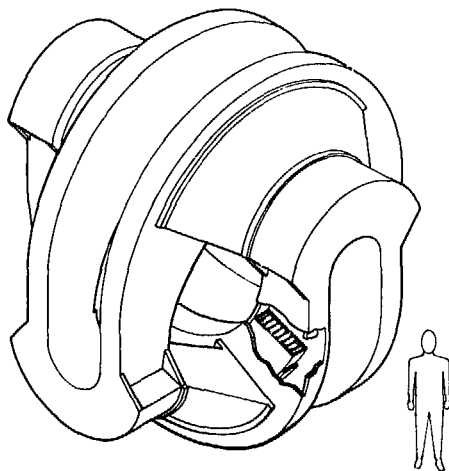


Fig. 15. MX coils and structure (tentative).

For reasons of simplicity and reliability, we propose to use a cryostatically stabilized Nb-Ti conductor in "pool" boiling He at 1 atm. Because of the high energy of the MX system, the requirement for moderately high current density, and the problems of winding a coil of Yin-Yang shape, some development work on the conductor is probably necessary.

This work is therefore directed towards the reliable and economic fabrication of a high-current conductor that has a large cooling surface, but at the same time has the capability to withstand severe crushing forces and to be bent in two planes. In one possible configuration, shown in Fig. 16, the super-

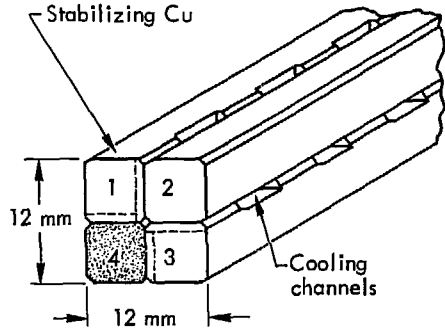


Fig. 16. Cryogenically stabilized MX conductor.

conducting filaments are all contained in one quadrant, which is then soldered to the other three OFHC stabilizing bars. Grooves, previously formed in these bars, now form internal cooling channels.

## Explosive Joints in Superconductors

The fabrication process used for Nb-Ti/Cu composites presently limits maximum length of unbroken conductor to that length that weighs about 600 lb. Because unavoidable breakages occur quite frequently, manufactured piece lengths may be shorter than this. Since joints that occupy extra space upset the arrangement of conductors in the winding, they are undesirable, and additional expense and waste is often incurred in avoiding them.

Some work has already been done in

Europe, notably by Imperial Metals Industries in Great Britain, on explosive joining of conductors, and the technique has been used in several coils. The advantage claimed is that a scarfed joint takes no extra space and appears to be superconducting, or to have an acceptably low resistance, up to a high proportion of the critical current of the conductor.

We placed a contract with Battelle, Columbus Laboratories, to investigate the explosive-joining technique and to produce sample joints for mechanical

and electrical evaluation. This preliminary study has now been completed, and the Battelle report is reproduced as Appendix D. After a few trials, joints having a tensile strength that is 80% of the tensile strength parent conductor can now be made reproducibly. It is also thought

that further minor modifications would yield joints of 100% strength.

The low-temperature properties of these joints are still to be investigated. However, even if the joint is not superconducting, its resistance is likely to be extremely low, and therefore acceptable.

## New Test Facilities

### SHORT-SAMPLE TEST FACILITY

Figure 17 shows the short-sample test facility that has been used to measure the  $H-I_c$  characteristics of short samples of superconductor. The split-pair solenoid, which was purchased from IGC generates magnetic fields up to 13.5 T. The gas flow through the leads is measured with calibrated orifice-plate flowmeters that were designed and built at LLL for this purpose and that are regulated manually by means of throttling valves. Magnetic field strength is measured by a pair of calibrated magneto-resistance probes that were built into the magnet. Two pairs of 5-kA, vapor-cooled, current leads, purchased from American Magnetics, Inc., are operated in parallel,

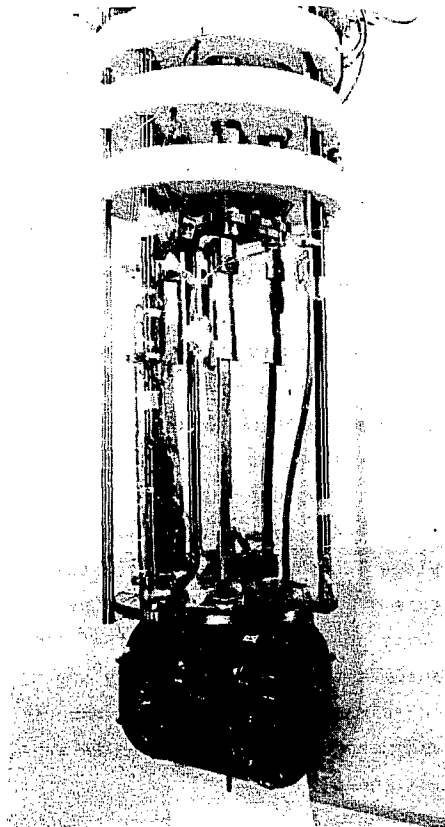


Fig. 17. Short-sample test facility.

giving us a 10-kA capability. The Dewar was purchased from CRYOFAB, Inc.

#### PRECISION 10-kA REGULATOR

During FY 1975, a precision regulator, capable of regulating the output of our existing 10-kA power supply to 1 part in  $10^5$ , has been designed and is nearing completion. A major portion of this regulator, a pre-wired transistor bank consisting of 500 transistors in parallel, was available from a previous instrument. As a result, the cost (approximately \$18,000) is about one-fourth what it would have been if the transistor bank had to be fabricated.

#### TENSILE TESTER

Detailed design work on the non-magnetic load frame of the tensile tester has been completed, and we are now negotiating contracts for fabrication of the various components. To prevent coupling with the fringing fields of the magnet used in the short-sample work, the tester will be constructed of 304 stainless steel. Because it will have a capability of applying 50,000-lb loads, it will also be used to test structural materials in later stages of the program. There is a 30-in. open space between load columns, and the head space, which is infinitely

adjustable, is about 10 ft. These dimensions will give us much versatility in testing.

The Materials Testing Group at LLL has considerable expertise in constructing such systems; therefore, the servo-hydraulic system may either be constructed "in house" or purchased from a commercial firm and interfaced to our load frame.

#### HELIUM-PURIFICATION PLANT

The present He-purification systems now serve both the Baseball II experiment and the superconductor development laboratory. When both groups are operating, there is occasional difficulty in handling and processing all of the boil-off gas. Helium inventory control is also complicated when two groups operate the same equipment. A more satisfactory method of operation would be to divide the system into two parts, each of which could process the full output from a single experimental area.

The He-purification system has a high-pressure storage capacity of about 500,000 standard cubic feet, half of which is used for dirty gas storage and the remaining half for purified gas ready to liquify. To serve two separate systems, this storage can be divided into approximately equal volumes of clean and

dirty gas. In addition, the low-pressure storage consists of two 8000-ft<sup>3</sup> gas bags that can be isolated from one another for independent use.

The total compressor capacity now consists of one 125-hp Worthington capable of 200 cfm of He gas and two 60-hp Rix compressors that can handle 100 cfm each at 1800 psi. These compressors can be connected to provide either dual or single high-capacity service.

The purification unit itself consists of a pair of identical heat-exchanger and dryer units that can process 600 cfm each, and which can be isolated to serve two systems. At present, only half of the purification unit is used; however, the changes in piping required to provide a dual purification capability are now in progress. The purifiers were obtained from the surplus equipment inventory and are considerably over-size for our He-recycling plant. However, this is an advantage because they seldom need regeneration. Under present operating conditions, this is about once a month.

Last year, the high-pressure laboratory used approximately 500,000 ft<sup>3</sup> of He gas. Since that laboratory lacked a recycling capability, all the gas was vented to the atmosphere. Our low-pressure storage area is about 750 ft from the high-pressure

laboratory. We are now installing a 4-in. gas line to recover most of this gas: this recovery not only will conserve a resource, but also will represent an annual saving of about \$25,000.

#### 1-m BORE, 8-T TEST FACILITY

As part of the development program on multifilamentary Nb<sub>3</sub>Sn conductors, a 1-m-outside-diameter Nb<sub>3</sub>Sn coil must be tested in an 8-T background field to give a combined maximum field of 12 T at the conductor.

The design of this 8-T facility, comprising a set of Nb-Ti coils complete with a new cryostat to be assembled in the pit in the existing cryogenic laboratory, has been started.

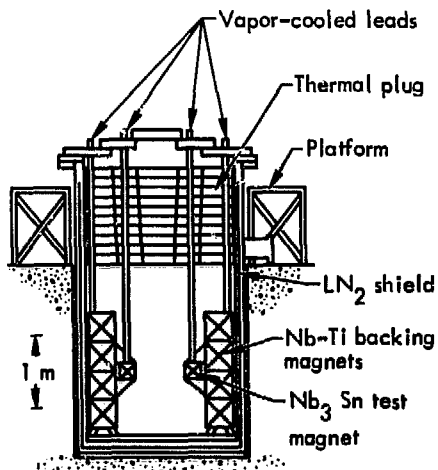


Fig. 18. Proposed 1-m bore, 8-T test facility.

The facility will also be used for proving tests on the MX conductor. A trial length of MX conductor will be wound into a solenoid sufficiently large to be representative of the MX thermal environment, but insufficient to generate the peak field. It is,

therefore, proposed to assemble it together with part of the 8-T coil system, and in this way to meet the full field requirement.

A sketch of this system, based on some preliminary concepts, is shown in Fig. 18.

## Acknowledgment

The authors would like to thank Coralyn McGregor for her invaluable

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## References

1. E. Gregory, W. G. Marancik, and F. T. Ormand, IEEE Trans. Magn. MAG-11, 295 (1975).
2. E. Gregory, W. G. Marancik, F. T. Ormand, and J. P. Zbasnik, "Multifilamentary Nb<sub>3</sub>Sn Conductors for Large Fusion Magnets," in Proc. 5th International Conference on Magnet Technology (MT-5), Rome, 1975, p. 301.
3. W. A. Fietz, C. D. Henning, and R. M. Scanlin, IEEE Trans. Magn. MAG-11, 299 (1975).
4. R. Randall, J. Wong, D. W. Deis, B. J. Shaw, and M. R. Daniel, IEEE Trans. Magn. MAG-11, 291 (1975).
5. F. R. Fickett, "Properties of Nonsuperconducting Technical Solids at Low Temperatures," in Proc. 4th International Conference on Magnet Technology, New York, 1972, p. 498.
6. T. H. Batzer, R. C. Burleigh, G. A. Carlson, W. L. Dexter, G. W. Hamilton, A. R. Harvey, R. G. Hickman, M. A. Hoffman, E. B. Hooper, R. W. Moir, R. L. Nelson, L. C. Pittenger, W. J. Silver, B. H. Smith, C. E. Taylor, R. W. Werner, and T. P. Wilcox, Conceptual Design of a Mirror Reactor for a Fusion Engineering Research Facility (FERF), Lawrence Livermore Laboratory Rept. UCRL-51617 (1974).

**Appendix A**  
**Multifilamentary Nb<sub>3</sub>Sn Superconductors**  
**for Large CTR Magnets,**  
**April 12, 1975 through June 30, 1975**  
**(AIRCO, Murray Hill, NJ)**

SUMMARY

First-stage extrusions [19 niobium filaments in 10% tin-bronze matrix] and second-stage extrusions [(187x[19Nb in Bz] Ta liner) } Cu can sufficient to fabricate 700 feet of 3 500 amp conductor have been prepared. The second-stage extrusions will be held in inventory for Lawrence Livermore Laboratory (LLL) until the final conductor configuration has been specified.

Several model configurations for providing full cryostatic stabilization of the finished conductor have been assembled in short lengths and are being evaluated. Although the early design work was based on intrinsic stabilization and protective shunting, the second-stage extrusion stock described above is equally suitable for incorporation into a conductor with cryostatic stabilization. Second-stage extrusions sufficient for making 700 feet of 3 500 amp conductor will be held in inventory for Lawrence Livermore Laboratory (LLL) until the final conductor configuration has been specified.

Three small third-stage billets configured for 1 000, 3 500, and 10 000 amps at 12 Tesla were extruded and drawn to dimensions normally used for 1 000 amp conductor to investigate the effects of filament size. Nominal filament diameters were 4.5  $\mu\text{m}$ , 2.4  $\mu\text{m}$ , and 1.4  $\mu\text{m}$ , but all contained the same percentages of Nb, Bronze, Ta and Cu (The 1 000 and 3 500 amp conductors were easily processed,



but the 10 000 amp conductor began breaking as the filament size fell below 2.9  $\mu$ . However, some usable samples of the 10 000 amp design were obtained at the 1 000 amp size. Samples from these billets were reacted for 1, 2, and 4-1/2 days at 650°C in both straight and bent configurations. These have been forwarded to LLL for testing.

Two experimental conductors containing 3 553 filaments of Nb in 13% Sn Bronze and 15% Sn Bronze were made by a two-stage extrusion process using 2 in. diameter billets. Although the first-stage extrusions were quite successful, central filament failures occurred in the second-stage extrusions and were most pronounced in the 15% Bronze matrix billet. However drawing operations were quite successful, producing wire at 20.1, 14.5, and 10 mil dia. from both billets. (Corresponding to 4, 3, and 2  $\mu$ m filament diameters). Since central filament breaking has never been observed in any of our extrusions involving Nb filaments in 10% Sn Bronze, it appears that somewhat different extrusion conditions are required for the higher-tin bronzes.

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MODEL CONFIGURATIONS FOR A CRYOSTATICALLY-STABILIZED SUPERCONDUCTOR

Seven model conductor configurations which illustrate possible methods of presenting extended cooling surfaces for liquid helium cooling were assembled. The photographs in Figures 1-3 are of short pieces of each type which are off-cuts from the samples given to Dr. Clyde Taylor of Livermore on Friday, May 16, 1975. In each case one or more conductors are enclosed in a tube which can be of copper or stainless steel and which is intended to be pierced with closely spaced small holes to give a permeable stable envelope permitting access of coolant to the major portions of all surfaces of the enclosed parts. In six of the samples the outer envelope is pierced with holes approximately 3/32" dia. on a 3/16" staggered pitch over its area. In the seventh case the holes have not been provided in the model but such would be the intent in any real application.

Type 1

This consists of 3 main conductor cylinders, each about .4" dia. They are set side-by-side with their centerlines in one plane and are encased in a close fitting stainless steel tube with pierced holes. The center cylinder represents a superconductor while the two outer cylinders provide additional copper. To allow internal channels for liquid helium, each of two outer cylinders are grooved with a single helix about .030" deep and .10" wide with a pitch 5 to the inch. Helium can thus reach most of the internal surfaces.

Type 2

This has 4 main conductor cylinders each about .40" dia. arranged with their centerlines on the corners of a square. One conductor in the sample is made of aluminum. It represents the superconductor. The other 3 provide additional copper. These 3 are grooved each with a single helix .030" deep and .10" wide with a pitch 5 to the inch. The enveloping tube of pierced copper is made to fit closely on the flanks of the enclosed cylinders but with comparatively sharp corners which provide voids to expose the outer quadrants of the cylinders into direct contact with liquid helium.

Type 3

The construction follows the pattern of Type 2. It differs in that one of the 3 copper cylinders is in the form of a tube of equal diameter. This tube is pierced with 3/32" dia. holes along its length at 3/32" pitch, each successive hole being at right angles to its predecessor. The object is to provide more access for liquid to reach the center of the construction by sacrificing some copper conductor.

Type 4

Is a single prism conductor approximately .6" square in section with a longitudinal groove provided in each face. The grooves are approximately 3/16" wide x 1/8" deep. The whole is enclosed in a pierced square copper tube. Cooling liquid helium has access to each of the 4 grooves. The prism represents a

superconductor which is deemed to be provided with sufficient copper integral in its construction.

Type 5

As in Type 4 the conductor is a square prism about .6" square. But, instead of grooves cut longitudinally into the surface, in this mode the outer surface is wrapped helically of a .2" wide x .03" thick copper strip. As the pitch of the helix is .3 this leaves a .1 wide helical void over the sides of the conductor when enclosed in the pierced outer tube. The single superconductor is deemed to be provided with sufficient copper integral in its construction.

Type 6

As in Types 4 and 5 the single conductor is square and enclosed in a pierced copper tube. In this case the sides of the conductor are incised by a continuous helical groove about 10 to the inch. The groove was formed by cutting a 10 T.P.I.-NC2 thread in a 3/4" dia. rod, filling the thread with stainless wire and squaring the rod in a turks head. The wire was removed after squaring the rod. The helical void thus provided between copper envelope and conductor provides an extended cooling surface. The single conductor represents a superconductor which is deemed to be provided with sufficient copper integral in its construction.

Type 7

This sample illustrates the enclosure of 4 conductors in a square tubular envelope of stainless steel. Although no holes are

provided in the envelope they can of course be provided in any desired pattern. In contrast to Types 2 and 3 the corners of the envelope follow the contour of the outer conductor quadrants and thus provide less exposure of the conductor's surfaces. Because none of the elements are grooved or wrapped there is no access to the center of the construction.

All the above samples were constructed by sinking the outer envelope (which in cases 1 to 6 included was predrilled) onto the core conductors. In a production operation the outer tubular sheath would be formed continuously as it and the prepared core were laid together. The sheath would be made from prepunched metal strip which would be welded continuously into a tube as it was formed into shape. The assembly would then immediately pass through form rolls to sink the sheath tightly onto the core conductors. The sheath could be stainless or copper as required. These handling and forming operations would require that the complete conductor assembly be fabricated before the final high-temperature reaction to produce  $Nb_3Sn$ .

Any of these configurations can be made using a monolithic superconductor drawn from a third-stage extrusion consisting of a number of second-stage extrusion hex rods packed in a copper can. The wall thickness of the copper can will be determined by the configuration finally chosen. We are holding in inventory enough second-stage extrusion stock to make 700 feet of 3 500 amp conductor.

FABRICATION OF STOCK FOR 700 FEET OF 3 500 AMP CONDUCTOR

Second-stage extrusion stock suitable for packing into a third-stage extrusion billet of any suitable configuration to make 700 feet of 3 500 amp conductor has been prepared.

First-Stage Extrusions:

19 filaments of niobium in a 10% Sn - 90% Cu matrix (19Nb in Bz). Two each 7 inch diameter billets were extruded and drawn to a hexagonal cross-section suitable for packing into a second-stage billet.

Second-Stage Extrusions:

187 of the 19 filament hex rods described above were packed in each tantalum-lined copper (OFHC) extrusion can, giving 3 553 filaments  $\{(187 \times [19\text{Nb in Bz}])_{\text{Ta}}\} \text{Cu}$ . Fillers cut from solid bronze hex stock were used to circularize the assembly inside the tantalum liner. Seamless Ta liners spun from sheet stock were used in these billets. Two each 4.325 inch diameter billets have been extruded to 1.125 in. dia. and 3 more are ready to be extruded early in July 1975. These five billets will provide sufficient stock for a third-stage extrusion of any suitable configuration to yield 700 feet of 3 500 amp conductor (3 500 amp at 12T and 4.2°K). These 5 extrusions will be held in inventory for LLL until the configuration of the third-stage extrusion has been specified.

FILAMENT DIAMETER EXPERIMENTS

Three 2 in. diameter third-stage extrusion billets were assembled using hexes from standard second-stage extrusion stock  $\{(187 \times (19\text{Nb in Bz})) \text{ Ta}\} \text{ Cu}$ . Hexes and billet-can walls were sized to give 34 area % OFHC copper in 1 000 amp, 3 500 amp, and 10 000 amp configurations. The billets were extruded to 1/2 inch diameter rod and drawn to 0.162 in. diameter with annealing as required. The 0.162 in. rods were twisted 1.23 Tw/in. flattened in a "Turks Head" and drawn to 0.066 x 0.197 in. rectangular cross-sections.

1 000 amp Type Configuration: 67 507 filaments.

$\{19 \times \{(187 \times (19 \text{ Nb in Bz})) \text{ Ta}\} \text{ Cu}\} \text{ Cu}$ . This material was easily reduced to the final 0.066 x 0.197 in. cross-section. At this size, the nominal filament diameter is 4.5  $\mu\text{m}$ . Microscopy showed all Ta barriers intact.

3 500 amp Type Configuration: 259 369 filaments.

$\{73 \times \{(187 \times (19\text{Nb in Bz})) \text{ Ta}\} \text{ Cu}\} \text{ Cu}$ . This material was easily reduced to the final 0.066 x 0.197 in cross-section. At the size, the nominal filament diameter is 2.4  $\mu\text{m}$ . Microscopy showed all Ta barriers in tact.

10 000 amp Type Configuration: 664 411 filaments.

$\{187 \times \{(187 \times (19\text{Nb in Bz})) \text{ Ta}\} \text{ Cu}\} \text{ Cu}$ . This material broke once as the filament diameters reached 2.9  $\mu\text{m}$ , again as filament diameters reached 2.3  $\mu\text{m}$ , and frequently as smaller filament sizes



were attained. About half of this material was finally reduced to the desired 0.066 x 0.197 inch cross-section, but in several lengths due to breakage. At this size, the nominal filament diameter is 1.4  $\mu$ m. Microscopy showed some filament breakage and some broken Ta barriers.

High Temperature Reactions: Test sets of samples of 1 000 amp type and 3 500 amp type configurations were reacted at  $(650 \pm 5)^\circ\text{C}$  for 1.0 day, 2.0 days, and 4.5 days in an Argon-purged retort. Each test set consisted of two 18 inch long straight samples and five 6 inch long samples pre-bent (in the "thin" direction) around diameters corresponding to 0.4%, 0.6%, 0.8%, 1.0%, 1.2% strain at the outer edge of the conductor. The corresponding bend diameters were 16-1/2, 11, 8-1/4, 6-5/8 and 5-1/2 inches. Both straight and curved samples were reacted in grooves in graphite forms which held them in the desired shapes during the final high temperature reactions.

Because of the limited yield of conductor from the 10 000 amp type configuration, only the 1 day reaction included a complete set of samples from this material. The 2 day reaction included two 18 inch straight samples and the 4.5 day reaction included one 18 inch straight sample of the 10 000 amp type configuration.

These samples have been shipped to LLL for testing and evaluation.

HIGHER TIN-BRONZES

Bronze ingots containing 13% and 15% tin (balance copper) were vacuum induction melted from extra-pure-grade tin and OFHC copper and cast in graphite molds. (Later analyses gave 12.85% and 14.85% Sn respectively) After solution annealing and machining, these ingots were extruded to make rod stock for fabricating 2 inch diameter billets.

First-Stage Extrusions: 19 niobium rods were loaded into 19 holes drilled in 2 in. diameter billets of each material. After loading, lids were electron beam welded on each billet in vacuum. After preheating to 1250°F, each billet was extruded to 1/2 inch diameter rod. The rods were drawn to a hexagonal cross-section 0.1245 inches flat-to-flat using a drawing and annealing schedule appropriate to 10% Sn-bronze. No unusual difficulties were encountered during these procedures. The bronze: niobium ratio was 2.41:1.

Filler-Stock Extrusions: Two 2 inch diameter solid bronze billets were also extruded to 1/2 inch diameter and drawn to 0.1245 inch flat-to-flat hex rods. These provided stock for machining fillers for circularizing the "hex-pack" inside the second-stage extrusion cans.

It was later discovered that both filler stock billets had been inadvertently made from 13% bronze. However, the fillers constitute a small enough percentage of the total bronze (about 12%) in a second-stage billet that this error was considered to be negligible.

Second-Stage Extrusions: 187 hexes containing 19 each Nb rods in 13% tin-bronze were loaded into a 2 inch OD bronze extrusion can of the same composition along with solid filler strips as described above. The bronze: Nb ratio was 3.22:1.

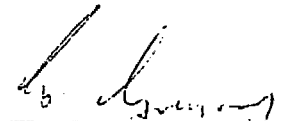
A similar billet was assembled using 15% tin-bronze in the hexes and the can with filler strips of 13% tin-bronze.

Lids were E.B. welded in place in vacuum and both billets were extruded to 1/2 inch diameter rods under the same conditions used for the first-stage extrusions. Cross-sections of the as-extruded rods showed that a number of filaments in the central regions of the billets had broken during extrusion. Central filament breakage was noticeably worse for the 15% bronze than for the 13% bronze matrix billet. Since we have never observed this effect while extruding 10% tin-bronze matrix billets, it appears that extrusion conditions must be modified for the higher tin-bronzes.

Drawing Operations: Although the second-stage extrusions were obviously faulty, drawing operations were performed to gain familiarity with the characteristics of these materials. Samples of wires from these billets were drawn to 0.0201, 0.0145, and 0.010 inches diameter using a drawing and annealing schedule appropriate to 10% tin-bronze. These sizes correspond to 4.0, 2.9, and 2.0  $\mu\text{m}$  diameter filaments respectively. Filament integrity was not appreciably worse in small diameters than in the as-extruded rod.

When satisfactory extrusion conditions have been determined, we should find it feasible to use higher tin-bronzes in fabricating superconductors.

  
F. T. Ormand

  
Approved: E. Gregory

FTO:dmo

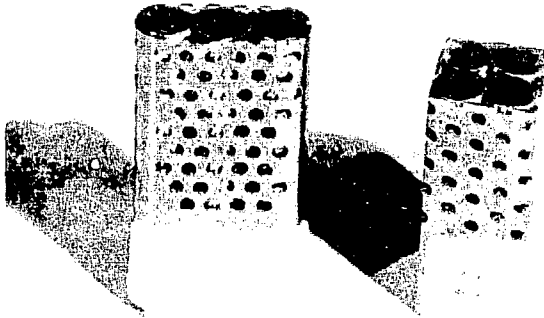


Figure 1. Model Configurations for Cryostatic Stabilization: Type 1 and 2

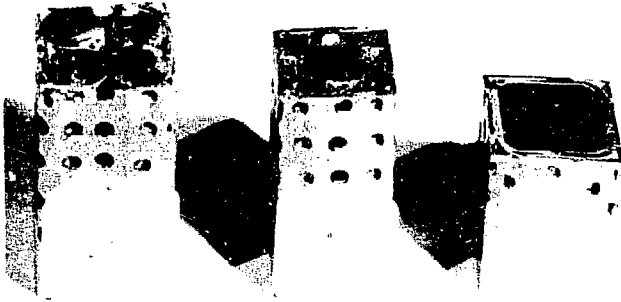


Figure 2. Model Configurations for Cryostatic Stabilization: Type 3, 4, and 5.

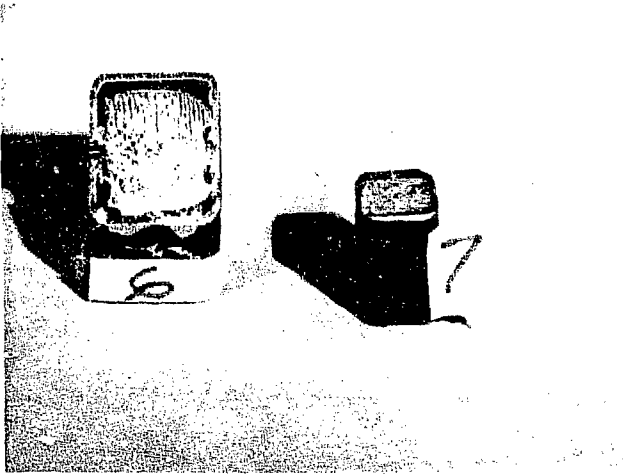


Figure 3. Model Configuration for Cryostatic Stabilization: Type 6 and 7

**Appendix B**  
**Metallurgical Investigations of the Effect**  
**of Filament Diameter and Heat Treatment**  
**on Microstructure of Nb<sub>3</sub>Sn,**  
**June 2, 1975 through July 1, 1975**  
**(AIRCO, Murray Hill, NJ)**



ABSTRACT

A multifilamentary conductor of 3553 niobium filaments in a bronze matrix is reacted under various heat treatment conditions. Determination of  $Nb_3Sn$  grain size through a new fractographic technique is described and results are compared with previous transmission electron microscopy data. Grain size is related to heat treatment time and temperature. Wire size is found to have an effect on reacted grain size.

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INTRODUCTION

Grain boundaries are thought to be the most important flux pinning centers for  $Nb_3Sn$  without second phase particles.<sup>1</sup> Thus fine grain structures are necessary for maximizing the critical current density. The grain size of the  $Nb_3Sn$  formed by the bronze diffusion method is considered to be primarily a function of heat treatment time and temperature. This is a significant advantage over commercial tape production in that small grain sizes can be produced without introduction of second phase grain growth inhibitors which may alter other properties. The multifilamentary approach offers the additional advantage of growing a significant volume of fine grained material at relatively low reaction time and temperature.

The present work examines grain size as a function of heat treatment for two multifilamentary wires. A technique which shows promise in routinely determining grain size without the difficult and time consuming specimen thinning procedures required for transmission electron microscopy is presented. This technique is based on using the scanning electron microscope to examine fracture surfaces produced by breaking the reacted wire in tension. These surfaces are expected to represent the  $Nb_3Sn$  grains due to its inherent brittleness. Very brittle materials such as  $Nb_3Sn$  characteristically fracture along grain boundaries. The data are compared with recent transmission electron microscopy results.<sup>1</sup>

EXPERIMENTAL PROCEDURE

The material used in this study is a round wire composite consisting of 3553 niobium filaments in a 10 weight percent tin bronze matrix (Fig. 1). The filaments are arranged in 187 groups of 19. This core is surrounded by a tantalum barrier layer and an outer shell of OFHC copper. The tantalum serves as a barrier layer to prevent diffusion of tin from the bronze matrix into the copper. Following two extrusion steps and many wire drawing operations with intermediate annealing, the wire diameters used are 0.02535 inch (0.06439 cm) and 0.01275 inch (0.03238 cm). The average niobium filament diameters are 4.5 and 2.25 microns respectively.

Short lengths of wires are heat treated in graphite holders inserted into flowing argon atmosphere retorts for various times at 600, 650 and 700°C. Some samples are given two stage heat treatments consisting of short times (1 to 5 hours) at 900°C either at the beginning or at the end of the normal heat treatment.

Reaction layer thickness is determined by measurements on SEM micrographs of polished and etched cross-sections. The Nb<sub>3</sub>Sn grain size is determined by a line intercept method on SEM photos of tensile fracture surfaces. A minimum of 10 lines of random orientation are used for each micrograph. The grain size is given by  $g.s. = L/nM$  where L is the length of the line used, n is the number of grain boundaries intercepted and M is the magnification. There is some error inherent in determining grain size from three dimensional fracture surfaces. Errors of  $\pm 5\%$  are expected from the combined effects of measurement errors and the magnification error of the SEM. This is felt to be a viable technique offering advantages of speed and simplicity over the difficult sample thinning required for transmission electron microscopy.

Critical temperatures are measured using a standard four point probe resistive method. Critical current measurements were unsuccessful due to the short (approximately 4 inch) sample lengths. Wire burnouts attributed to transfer length problems made meaningful results unavailable.

RESULTS AND DISCUSSION

Figures 2 through 5 illustrate the fracture surface of a single filament as seen with the SEM. The area in the center of each photograph is unreacted niobium. This is surrounded by the layer of Nb<sub>3</sub>Sn and the bronze matrix. Figure 2 is reacted at 600°C for 480 hours, Figure 3 is 650°C for 144 hours, and Figure 4 is 700°C for 24 hours. The increase in grain size with reaction temperature is best seen in Figure 5. This specimen is heat treated at 650°C for 144 hours followed by one hour at 900°C. The grain growth is readily apparent when compared to Figure 3. Figure 6 is a plot of grain size as a function of reaction temperature. The assumption has been made that the grain size of the material reacted at 650°C for 144 hours and then at 900°C for one hour is the same as if it had been reacted at 900°C. These data compare closely with data recently published<sup>1</sup> using a more difficult transmission electron microscope technique for grain size determination. The technique of Nb<sub>3</sub>Sn grain size determinations by fractography would thus seem reasonably accurate and should be very useful as a relatively fast, efficient method.

Figure 7 shows grain size as a function of reaction time for three temperatures and two wire diameters at each temperature. Grain growth is more rapid at higher temperatures as would be expected. It should be noted that the same grain size may be achieved through several different combinations of time and temperature. Unfortunately, it has not yet been possible to compare the critical current densities of the same grain size achieved through two different routes due to the problems mentioned before. Critical temperatures of all samples are between 17 and 18°K with no clear relationship to time at present sensitivities which will be improved. Temperature does appear to have a slight affect in raising T<sub>c</sub> possibly due to more complete ordering at higher reaction


temperatures. The smaller diameter wire has a smaller  $Nb_3Sn$  grain size for any given heat treatment. This effect may be attributed in part to smaller prior niobium grain size or more cold work to get to the smaller diameter. If this observation is the general case, one can infer that multifilamentary wire with extremely small filaments is beneficial due to the smaller grain size produced as well as the other well-known reasons. A study of niobium grain size and cold work effects on reacted grain size would prove interesting and is practical with the ease of fractographic grain size determination.

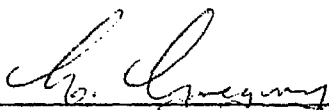
The  $Nb_3Sn$  layer thickness as a function of reaction time for various temperatures is shown in Figure 7. The data for the larger wire at 700 C is in agreement with earlier work.<sup>2</sup>

CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER WORK

Fractographic determination of  $Nb_3Sn$  grain size is shown to be feasible and relatively simple. The results obtained by this method are in close agreement with those by transmission electron microscopy. Grain size is a strong function of reaction temperature and grain growth occurs with time even at the low reaction temperatures. The more heavily cold worked composite of smaller diameter shows smaller grains for any given reaction heat treatment within the limits studied.

A more elaborate study would certainly be desirable. By using longer samples or rectangular material one could develop superconducting property data as a function of not only grain size and layer thickness, but also as a function of what processing route is used to attain them. The effects of niobium cold work or grain size prior to reaction on the  $Nb_3Sn$  grain size and properties would also be useful information in explaining the differences in behavior noted in the larger and smaller wire sizes. The grain size as a function of matrix tin content would also be an interesting study.

  
\_\_\_\_\_  
Peter Blum

Approved:   
\_\_\_\_\_  
E. Gregory

PB:msh



REFERENCES

1. R.M. Scanlan, W.A. Fietz, and E.F. Koch, Journal of Applied Physics, Vol 46, No. 5, (1975) p. 2244-2249.
2. M. Suenaga, W.B. Sampson and T.S. Luhman, Proc. Texas C.T.R. Meeting, 1972.

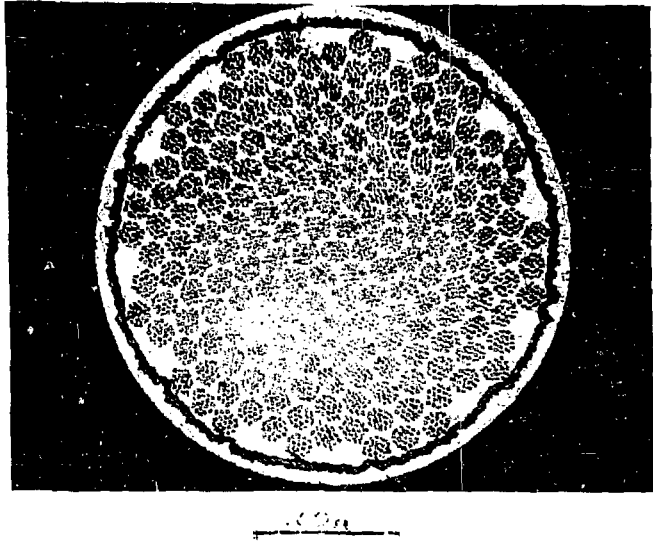


FIG. 1. 3553 filament composite used in this study.  
Diameter = 0.0324 cm

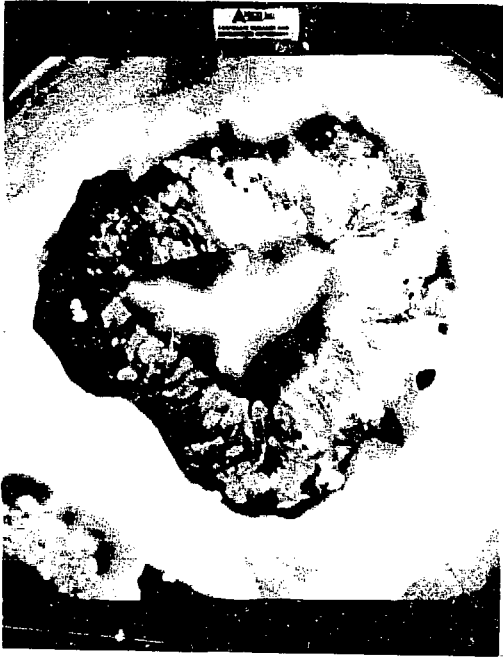


FIG. 2. Fracture surface of 0.0644 cm diameter wire. H.T.: 480 hours at 600 C Grain Size  $\approx$  862 A

1.00



FIG. 3. Fracture surface of 0.0644 cm diameter wire. H.T.: 144 hours at 650 C Grain Size  $\approx$  943 A

1.00



FIG. 4. Fracture surface of 0.0644 cm diameter wire. H.T.: 24 hours at 700 C Grain Size  $\approx$  1170 A



FIG. 5. Fracture surface of 0.0644 cm diameter wire. H.T.: 144 hours at 650 C, 1 hour at 900 C Grain Size  $\approx$  3440 A

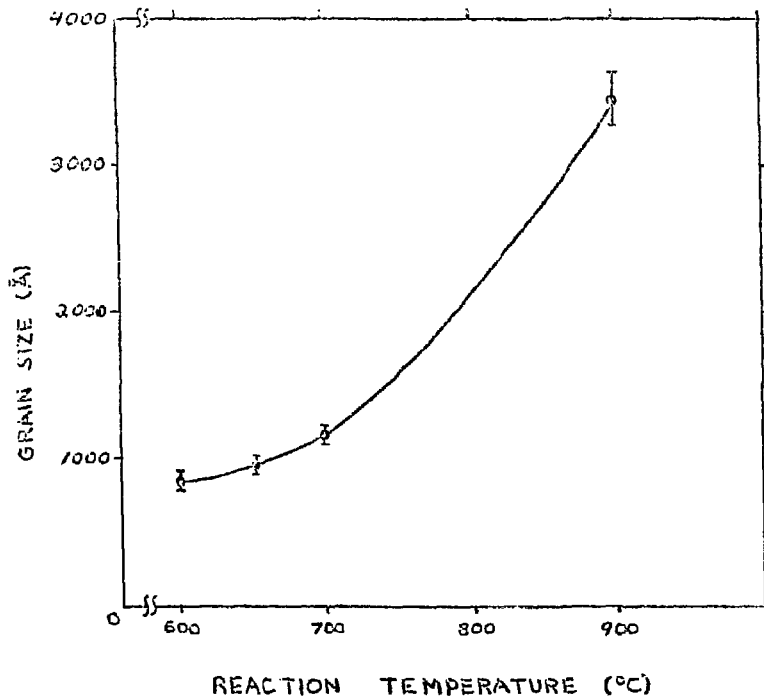
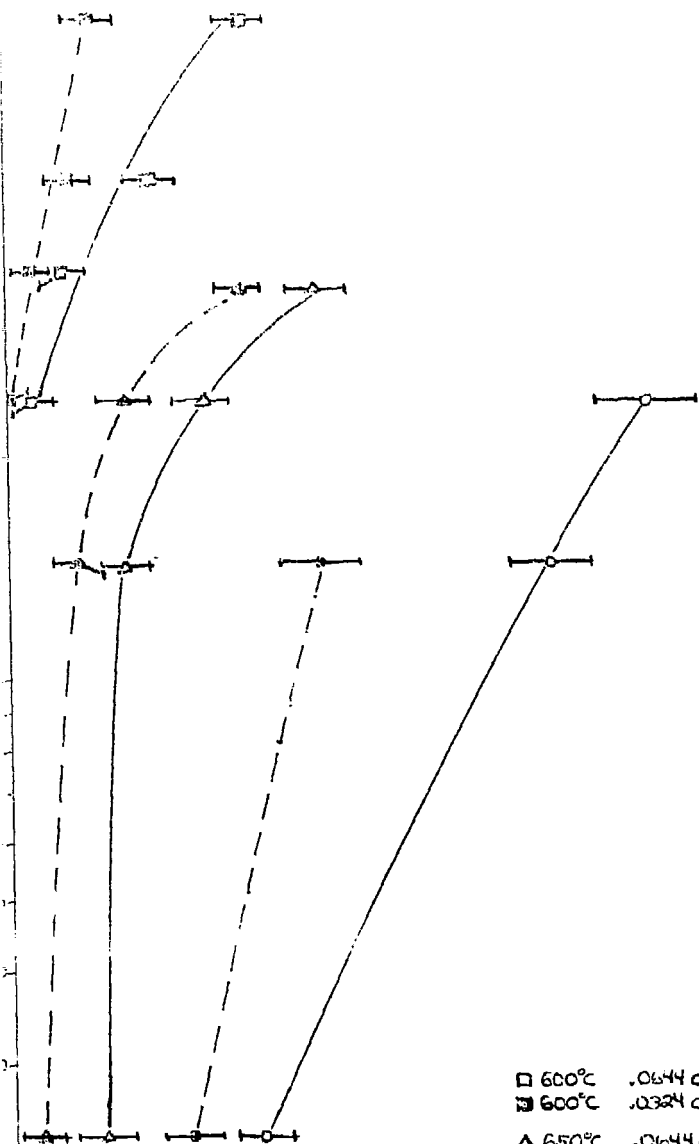


FIG. 6. Plot of grain size versus reaction temperature. Time at temperature chosen to produce a  $\sim 1\mu$  thick reaction layer.



□ 600°C .0644 cm.  
 ▣ 600°C .0324 cm.  
 △ 650°C .0644 cm.  
 ▲ 650°C .0324 cm.  
 ○ 700°C .0644 cm.  
 ● 700°C .0324 cm.

## **Appendix C**

**Cabled Multifilamentary Nb<sub>3</sub>Sn Superconductor Development  
for the LLL-CTR Program Final Report, October 17, 1974  
(Intermagnetics General Corporation, Guilderland, NY)**

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## I. INTRODUCTION

The purpose of the study described in this report is to develop the understanding and achieve resolution of problems involved in the prototype production of a multifilament  $\text{Nb}_3\text{Sn}$  conductor ultimately to be used in the LLL magnet program for Controlled Thermonuclear Reaction (CTR). The emphasis of this report will be on the steps investigated and leading to the resolution of the problems which were encountered during the attempted scale-up of the external diffusion approach; a discussion of the present alternatives for conductor design and a detailed but early-stage comparison of the economics of production for alternative conductor approaches.

The major requirements of the ultimate conductor envisioned are:

- a) a critical current carrying capacity on the order of  
10,000 amperes at 12 Tesla
- b) stabilization with high conductivity copper
- c) provisions for ventilation by liquid helium
- d) twisting and transposition to reduce losses.

Because the latter four requirements are not completely resolved at present, sufficient flexibility in conductor design and fabrication capability is of extreme importance in choosing the eventual processing alternative. In addition to these requirements the conductor in the reacted state must have sufficient mechanical flexibility to allow standard spooling and winding operations required in magnet construction. These overall considerations predicated the preference for a cabled configuration over a monolithic design.

In a cabled approach there are several alternatives. One may draw a bronze niobium composite to its final diameter and cable a group of such strands. To obtain the desired critical current of such a conductor tin may be applied to the strands by electroplating before cabling (the hybrid approach). Likewise, one may draw a copper-niobium composite to its final diameter and apply tin by electroplating. This latter approach (the external diffusion method) is potentially more attractive since a niobium-copper composite may be drawn down from the extruded rod to the final wire diameter with few, if any, intermediate anneals. Bronze, however, generally requires annealing after each 50-75% area reduction.

The simple bronze approaches are restricted to copper-tin alloys of 10-13 weight percent because of the difficulties of extruding and drawing composites with greater tin concentration. The hybrid approach and external diffusion method on the other hand, do not suffer such limitations and accordingly a lower proportion bronze remains in the reacted conductor. Minimizing the amount of this low-tin bronze is important because it has neither a high elastic modulus nor good electrical conductivity at cryogenic temperatures.

The method of externally applying tin to the copper-niobium strands of a cabled conductor has the potential of providing a conductor with improved overall current density as well as with a lower cost per ampere-meter. Intermagnetics General Corporation has successfully produced cabled multifilament  $Nb_3Sn$  superconductor by the diffusion of tin externally applied to the niobium-copper strands. Earlier production, however, involved strands of .006" diameter or less. In order to meet the initial target specifications on the conductor for the LLL-CTR program, however, a composite strand of at least .010" diameter is required. This requirement is related to economic considerations as well as to the desire for adequate packing.

A serious problem arose during the development of the .010" diameter strand cable. The amount of tin necessary for the degree of reaction with the niobium to meet our target specifications caused a separation at an interface near the outside surface of the strand. Experimental work has been conducted to more thoroughly characterize the effect and understand the mechanism underlying the separation. Furthermore, we were investigating processing approaches to overcome this problem consistent with certain economic goals. The progress made in these endeavors forms the subject of this report. Particular attention will be focused on the economic considerations of any process modification, since the problem no longer seems to be whether or not we can produce the required conductor by the external diffusion process but rather how economically we may produce it.

This report is divided into two parts. The first part deals mainly with the experiments conducted to understand the separation problem. In addition there is a discussion of the void formation common to all bronze composite conductors and its possible resolution. In a summary to this first section the economics of the steps incorporated to eliminate the separation will be assessed. The second part of the report consists of a breakdown of costs for the various processing steps which might go into the fabrication of the conductor. The total cost of a monolithic conductor, a cabled hybrid conductor and an externally diffused Cu-Nb composite conductor each having the same proportion of stabilizing copper and each capable of carrying 1000A and 10,000A at 12T are compared.

The cost estimates presented are obviously very preliminary at this stage of process development. Nevertheless, the information is sufficiently detailed to give credence to an emerging conclusion, namely that the cabled conductor configuration should be considerably less expensive under large volume manufacturing conditions.

## II. PROBLEMS ENCOUNTERED IN THE FABRICATION OF A MULTIFILAMENT $Nb_3Sn$ CONDUCTOR

Our studies under the support of the Lawrence Livermore Laboratory have concentrated on the understanding and resolution of problems involved in manufacturing of multifilament  $Nb_3Sn$  conductors which have occurred in IGC conductors<sup>1</sup> and those of other companies. These problems are summarized as follows:

1. Separation of an outer layer of a conductor strand which had been plated with tin and heat treated. (This will be referred to as the separation problem). The problem is related to the diffusion of tin into a copper substrate.
2. Void formation near the filaments. The effect has been observed primarily in conductors reacted in a niobium-bronze composite. This problem is also diffusion related.
3. Destruction of the tantalum barrier during drawing of the composite. We have observed a tendency for the diffusion barrier to tear making it ineffective as a diffusion barrier to the tin. The effect is significant enough to substantially reduce the conductivity of the pure copper at helium temperatures.

### A. The Separation Problem

The color photomicrograph in Figure 1 shows a wire strand exhibiting separation of the outer region (crusting) after plating 12 microns (90 mg/meter) of tin and heat treatment at 580°C for 24 hours. An obvious feature of this separation is that it occurs between a tin rich phase outer region and an inner region which is almost pure copper. Also, as one may

see from this photomicrograph the separation occurred about .0015" from the outside surface rather than at the original plated tin-copper substrate interface which is about .0005" from the outside surface. Therefore, the tin had diffused a considerable extent before the separation occurred.

There are indications that at any diameter of copper substrate the separation is associated with thicknesses of plated tin greater than about 5 microns. Figure 3 shows a .006" diameter composite which had been plated with 4.8 $\mu$  (17 mg/meter) and 5.5 $\mu$  (19.5 mg/meter) of tin and subsequently heat treated at 580°C for 48 hours and 750°C for 72 hours. The separation is not evident in the sample with the smaller amount of plated tin but is clearly evident in the other. The small degree of separation in the samples with the larger amount of tin did not considerably reduce its critical current carrying capacity relative to the other sample since the outer layer is sufficiently bridged to the rest of the wire to allow substantial diffusion during the homogenization anneal at 580°C. However, as the separation becomes more severe, limitation of the critical current becomes more severe. In a .010" diameter wire with the same copper to niobium ratio (2.5:1) a 9 $\mu$  tin layer is necessary for the same degree of reaction (55% of the original niobium core) as 17.5 mg/meter on the 6 mil wire. Since this thickness far exceeds the apparently critical 5 $\mu$  thickness, the separation is rather severe. Figure 4a shows a composite plated with 9 $\mu$  (60 mg/meter) of tin and annealed at 580°C for 48 hours demonstrates this high degree of separation. Evident in the separated region is a grey

tin-rich phase as well as an alpha bronze which is richer in tin than the remaining matrix. Homogenization has been hindered considerably by the separation. Reaction of the tin with niobium will likewise be hindered because of the substantially increased diffusion length from the separation region to the niobium filaments.

In addition to the adverse effect on filament reaction and critical current the outer layer separation very likely has adverse effects on current sharing and transfer to stabilizing elements which might be cabled with the multifilament strand. We do not know the degree of separation which can be tolerated in a stabilized conductor, and, therefore, feel that the only safe alternative is one which eliminates it completely.

In our analysis of the problem several hypotheses were advanced regarding its cause. In our early hypotheses, the likelihood of hydrogen inclusion was considered. This possibility, however, has been eliminated because the separation is also observed in composites which have been annealed following coating of tin by dipping in a molten tin bath.<sup>2</sup> Considering the dependence of the effect on absolute thickness, the remaining alternative is that the problem is due to certain peculiarities of the copper tin diffusion system. Our experimental evidence clearly reinforces this assumption and provides much information regarding the mechanism for the effect.

Preliminary to a discussion of the mechanism for the separation and means of avoiding it, certain aspects of diffusion in the copper-tin system will be reviewed.

When dissimilar metals are placed in contact at a temperature sufficient to carry out diffusion there is a possibility of the formation of any or all of the intermediate phases existing in the corresponding two component phase diagram at the given temperature. This feature has been demonstrated in a copper-tin diffusion system by Kawakatsu et al.<sup>3</sup> Kidson<sup>4</sup> carried out an analysis and determined that the rate of growth of the intermediate layers is determined by the diffusivity of the constituents in each of the phases and the composition gradient at the interfaces between the intermediate phases. Another feature of the diffusivity  $D$  of a given species is that its temperature dependence is roughly described by the following analytic expression.<sup>5</sup>

$$D \sim D_0 \times \exp (-kT_m/T) \quad (1)$$

where  $T$  is the temperature at which diffusion is carried out,  $T_m$  is the solidus temperature of the given composition,  $D_0$  is a constant related to the diffusion mechanism and  $k$  is an empirical constant. Applying this rule of thumb to the diffusivity of tin in copper one would conclude that a phase characterized by a lower melting temperature would exhibit a higher diffusivity of tin. Moreover, in a region of the phase diagram characterized by the existence of a homogeneous phase, there should be a composition dependence varying according to the solidus temperature. This fact has been demonstrated for the Sn-Cu system by Bastow et al.<sup>6</sup>, who showed that in the range of composition corresponding to the existence of the solid solution  $\alpha$ -bronze the diffusion coefficient of tin in copper at 736°C varies from  $2 \times 10^{-14} \text{ m}^2/\text{s}$  at 3.5 at % Sn to  $9 \times 10^{-14} \text{ m}^2/\text{s}$  at 7 at % Sn. Furthermore the article shows that the log of the diffusion coefficient of tin in copper



is approximately linearly proportional to tin concentration over all the temperatures used for the reaction of the composite (650°-850°C).

The information gathered from the previous investigations of the Cu-Sn diffusion system provides an explanation of the severe separation in the multifilament conductor produced by the external diffusion process. In the early stages of diffusion the tin moves rapidly and forms intermediate phase layers. Our process is carried out at 580°C and therefore, the possible intermediate phases are  $\alpha$ ,  $\beta$ ,  $\gamma$ , and  $\epsilon$  shown in the phase diagram of Figure 2.<sup>7</sup> The diffusion of pure tin into the intermediate phase regions is high relative to the diffusion into  $\alpha$ -bronze from the innermost interface.

The result is a shallow outer layer which dissolves most of the tin. Furthermore, the copper existing in the outer region has lower mobility than tin and therefore cannot counter balance the mass flow into this region. The consequence is a pressure developing in this region. Because of the low yield strength of copper at temperatures around 600°C, the substrate cannot restrain the separation of the expanding layer. The degree of expansion of the separated layer shown in Figure 3a is consistent with the amount of tin applied. This expansion corresponds to an equivalent thickness of copper taking up 38 mg/m of the 55 mg/m of tin applied to form an 18 w/o Sn-Cu alloy.

According to our hypothesis the separation problem involves the development of severe concentration gradients in the outer region of the conductor and the development of stresses causing expansion and separation.

In our experiments we have concentrated on the following approaches:

1. Reduction of tin concentration gradients
2. Containment of the outward expansion of the tin rich region
3. Inhibition of the growth of the intermediate phases

In the following section is a discussion of the experiments conducted by IGC to achieve a solution of the separation problem.

#### 1. Experiments involving the plating of Ni-Sn alloy

A nickel-tin alloy of approximate atomic ratio of 1:1 was applied to a 10.6 mil diameter copper-niobium composite. Our intention was to avoid the development of intermediate Sn-Cu phases by plating a stable alloy of tin instead of pure tin. By making the decomposition of the plated layer the rate controlling step, we hoped to reduce tin concentration variation in the copper region.

About 90 mg/meter of the plated alloy was applied so that 50-60 mg/meter of tin could be diffused into the composite. After annealing the plated composite at 580°C for 18 hours, a very severe separation of the plated layer was observed. The annealed composite is shown in Figure 5. In this photomicrograph one notices that there is deformation of the original copper wire caused by the distortion of the tin-nickel plated layer. We speculate that the distortion results from hydrogen trapped at the plated alloy-copper substrate interface or from residual stresses existing in the plated layer. In any event the diffusion of tin into the copper does not occur to an appreciable extent. Even if the separation problem could be overcome, the Sn-Ni alloy is too stable to permit the application of tin by this means.

Because of the problem observed and the possibility that the Sn-Ni alloy is too stable at temperatures even up to the reaction temperature (750°C), we feel that this means of avoiding separation deserves no further consideration.

## 2. Experiments involving the plating of a Cu-Sn alloy

A copper-tin alloy estimated to contain about 50-60 wt % Sn was electroplated on a Cu-Nb composite of 10.0 mil diameter using a cyanide-stannate bath. An amount of alloy plate was applied to provide about 90 mg/meter of Sn. This amount of tin is considerably more than that applied to a conductor delivered to LLL at an earlier date which showed severe separation. While samples plated in this manner showed some undesirable features such as the formation of voids near the outside filaments, the degree of reaction was close to 100% after heat treatment at 750°C for 72 hours. Figure 6 shows the strand after reaction and Figure 7 is an enlargement of the reacted filaments. As one may see from the latter photograph the only filaments affected by the voids were those immediately bordering on the voids. The remaining filaments showed almost complete reaction. We are confident that high  $J_c$  may be achieved by Cu-Sn alloy plating but are still concerned about the void formation. Hopefully the void formation can be eliminated or substantially reduced by changing the composition of the plated alloy or by reducing the amount of alloy plated.

The application of this Cu-Sn plating process to the production of multifilament  $Nb_3Sn$  would present some problems beyond those encountered with either copper, tin or nickel plating. The copper-tin plating solutions would have to be monitored and replenished more often than any

of the above mentioned plating solutions due to their low anode efficiency. The composition of the plated metal is a function of the cathode current density. Two anodes, one tin and the other copper, must be maintained at different currents due to the lower efficiency of the tin anode.

Another problem with the cyanide-stannate solution is that the composition of the plate is strongly dependent upon the cyanide and hydroxide concentrations of the bath since these are involved in the formation of the Cu-cyanide complex and stannate ion participating in plating. Therefore, in order to insure a plated alloy of a certain composition the cyanide concentration and pH would have to be carefully controlled.

An alternative to plating the copper-tin alloy may be the plating of alternate layers of copper and tin. This technique was attempted on long lengths of wire using the standard tin sulphate bath and the acid copper sulphate bath. However, the plated tin layer became irregular and crumbled off the wire substrate after the copper was plated over the tin. This effect was probably the result of a replacement reaction between the copper ions and the plated tin. Most likely this problem can be overcome by using a copper cyanide plating solution. Unfortunately, we do not presently have the facilities to deal with the large quantities of cyanide solution which would be necessary for large scale wire production.

### 3. Experiments involving different thicknesses of plated nickel

A series of experiments were conducted to determine whether the separation could be reduced or eliminated by the application of a higher melting temperature metal over the plated tin. The diffusion of tin into

nickel and vice versa is rather limited at 580°C. Nickel was, therefore, chosen as the metal to be plated over the tin. Our belief was that the nickel layer would maintain its integrity and contain the remaining composite long enough to allow the outer layer under stress from the acquisition of tin to achieve mechanical equilibrium. The application of increasing amounts of nickel did indeed have benefit as shown in Figure 3. The void formation evident in sample b was most likely due to some diffusion effect between the tin and the nickel. However, the void formation does not seem extensive enough to significantly limit the diffusion of tin.

We are not presently sure how much nickel may be applied before the niobium tin formation is adversely affected. Experiments carried out at General Electric by Dr. R. Scanlan demonstrated that nickel in plated quantities much greater than in Figure 3b is capable of severely reducing the critical current of Nb<sub>3</sub>Sn. We have to date plated smaller quantities of nickel with no apparent effect on the critical current.

With the problems of nickel plating in mind we performed the same experiment using plated copper from a cyanide plating bath instead of nickel. The results, however, were much less successful. Figure 8 shows the result of plating 33 mg/meter of copper over 61 mg meter tin plated on the copper-niobium substrate. Although a large amount of tin diffused into the composite substrate during heat treatment at 580°C, a considerable amount of tin was left in an outside region which was originally the plated copper layer.

Although the technique of plating a layer of nickel over the tin plated substrate is not a satisfactory means of containing the separation problem, it points to a likely cure for the separation problem. The

effect of the nickel layer is to apply a hydrostatic pressure counteracting the tendency for the tin rich region to expand outwardly. The same hydrostatic pressure can of course be applied by an argon atmosphere.

#### 4. Experiments involving the application of a high argon pressure

The thickness of plated nickel capable of restraining the separation of a tin rich phase from the composite substrate shown in Figure 4b gives an indication of the argon gas pressures necessary to restrain the separation. The following argument can be made. Consider the nickel plated layer as a tube subjected to uniform internal pressure due to the expanding tin rich region contained within it. The nickel layer can contain an internal pressure up to a level which results in circumferential stresses at the yield point of nickel at 580°C which we will assume to be about 50,000 psi. The equation expressing the maximum internal pressures,  $p$ , in terms of the circumferential yield strength,  $\sigma_y$ , is the following:

$$p = \frac{(b^2/a^2)-1}{(b^2/a^2)+1} \sigma_y$$

where  $a$  is the inside diameter of the plated layer and  $b$  is its outside diameter. The quantity of nickel 19 mg/meter corresponds to a layer of about  $10^{-4}$  inches. This would mean that the layer of nickel shown in Figure 4b is equivalent to an argon pressure of about 1000 psi which is quite easily applied.

There are facilities at General Electric which would allow us to apply pressures much higher than this. Samples of .010" niobium-copper composite wire plated with 70 mg/meter of tin were submitted to the Materials and Processing Labs of the General Electric Company to be annealed at 580°C under an argon pressure of 10,000 psi for 48 hours. The results were not conclusive due to severe oxidation to the plated substrate which occurred during the heat treatments. The problem could have been avoided by proper procedure and thus a repeat of the experiment has been scheduled by the Materials and Processing Lab. Unfortunately the experiments could not be scheduled by the time of writing of this report. However, the results will be reported by letter.

##### 5. Experiments involving plating over a substrate stronger than copper

In order to determine the effect of substrate yield strength on the tendency towards separation, samples of a 10.6 mil diameter bronze-niobium composite were plated with varying amounts of tin. Figure 9 shows the result of plating 30 mg/meter of tin on a bronze composite followed by an anneal at 580°C for 16 hours. No separation was evident in any cross section. Figure 10 is the composite plated with 60 mg/meter and heat treated in a similar manner. Void formation is evident near the outer filaments but is much less extensive than in a copper composite plated with the same amount of tin. A conclusion which one may draw from this experiment is that a stronger wire substrate is more capable of resisting the separation. Another implication of this experiment is that we may plate tin and homogenize in two successive steps in order to apply enough

tin to accomplish 60% reaction in our basic 10.6 mil diameter copper: niobium composite having a copper: niobium ratio of 2.4:1.

Another interesting fact discovered in our experiments with plated bronze composites is that plating of tin on a bronze niobium composite already with enough bronze to fully react the niobium can increase the degree of reaction and critical current. In one experiment we plated 20 mg/meter tin on a 10.6 mil diameter (Cu -13 w/o Sn) - niobium substrate of ratio 4.3: 1 respectively. After reaction for 72 hours a sample plated with 20 mg/meter showed a critical current 20% greater than that of the unplated composite. We must conclude that having a large reservoir of tin existing as a tin rich phase in addition to  $\alpha$ -bronze improves the kinetics of reaction.

#### B. Void Formation in the Reacted Multifilament Nb<sub>3</sub>Sn Composite

In bronze matrix composites produced by IGC and General Electric, as well as other groups, void formation precipitated at and adjacent to the filaments has been observed. However, this effect has not been observed in conductor produced by externally tinning a copper-niobium composite. We performed an experiment to determine whether the application of tin can be used to eliminate void formation in a reacted bronze-niobium composite. Figure 11a and 11b shows two samples both of which had been plated prior to heat treatment. The samples in Figure 11a, which was first homogenized at 580°C and then reacted at 750°C, shows the same degree of void formation exhibited by unplated bronze composites. The sample in 11b, however, which had been directly reacted at 750°C, shows practically no void formation.



We do not presently know how serious the void problem is but suspect that at least two problems will develop from it. Current transfer to high conductivity protective elements in the conductor composite will be affected by voids so close to the filaments. Also, the voids may result in filaments which are poorly supported by the matrix resulting in a decrease of the desirable compressive strain which exists in reacted composites. The result of this will be a conductor with a greater minimum bend diameter.

The voids forming near the filaments are very similar in origin to those observed in studies of diffusion couples. Furthermore, studies on the effect of hydrostatic pressure on such Kirkendall voids have demonstrated that they can be reduced and sometimes eliminated by hydrostatic pressures. In a paper by Clay and Greenwood<sup>7</sup> describing Kirkendall experiments in the copper-nickel system, the authors mention that void formation is completely eliminated in a Cu-Ni diffusion couple at 850°C by the application of a pressure of 3000 psi.

The void formation exhibited by the Cu-Sn plated samples is similar in nature to those appearing near the filaments. Annealing under hydrostatic pressure may be a means of eliminating void formation in this case as well.

### C. Rupture of the Tantalum Diffusion Barrier

An investigation of the tantalum diffusion barrier was conducted by scanning electron microscopy and metallography. The purpose of the study was to determine the mode by which the barrier fails in order to assess the damage which may be done to the stabilizing copper and to consider means of

avoiding the problem. Figure 12a shows the cross section of a .010" composite with a central region protected by a tantalum barrier. Figure 12b is a scanning electronmicrograph of the tantalum barrier of the same composite but at .015" diameter. One notices in this photograph the severe striation which occurs over the entire surface of the conductor. Another feature are the holes which occur along a groove. There exists a possibility of such lengthwise openings being present along much greater lengths which would lead to a significant quantity of copper being poisoned during the diffusion process.

The composite was not subjected to any anneals during the drawing steps. A high temperature anneal ( $\approx 900^{\circ}\text{C}$ ) at some point in the drawing process may have some beneficial effect.

#### Summary

Our studies of the separation problem carried out under this contract reveal that it is caused by a shallow layer of composite substrate taking up tin resulting in the expansion and separation of this layer. We have determined several means of avoiding this problem. Considering that both a copper-niobium and bronze-niobium composite can be plated with a  $5\mu$  layer of tin without separation during heat treatment, a multiple plating process with annealing following several plating steps would be a means of depositing an adequate quantity of tin. Another alternative is plating an alloy of copper and tin. However, since maintaining a copper-tin plating bath is potentially an expensive task, a more reasonable

alternative may be to plate tin and copper from a cyanide bath in alternate layers. The most economically promising solution is the annealing of the plated composite under an argon pressure of 2000 psi. Although we do not at the present time have conclusive results on the effect of hydrostatic pressure we have considerable confidence in this approach as an economic solution to the separation problem. Further experimental results on this approach will be forthcoming. A feature which must exist in order for this technique to be successful is that the separated region be entirely enclosed by material. If this is not achieved naturally with a layer of plated tin, we may have to apply an additional layer of a material such as copper or nickel.

We presently believe that the void formation near the filaments is under control at least in the cabled composites. In a monolithic composite the problem may be eliminated by the application of hydrostatic pressures. Void formation which may occur in other regions may likewise be eliminated by annealing under hydrostatic pressures.

A third problem is that of the tantalum barrier rupture. We have no concrete means of avoiding this problem at present. Further experimentation with heat treatments would be highly desirable. In a cabled composite the problem of diffusion barrier rupture may be completely avoided by including the copper as a separate strand either before reaction or after. If the strand is included before reaction a protective barrier will be necessary. However, the tantalum copper composite can be produced with less reduction of the tantalum shell.

This section of the report has dealt qualitatively with the solution of the problems observed in multifilament  $Nb_3Sn$ . The next section deals more thoroughly with the economics of the processing alternative discussed in this section.

### III. COST ESTIMATES FOR LARGE SCALE PRODUCTION OF MULTIFILAMENT $Nb_3Sn$ CONDUCTOR

Price projections have been made for the mass production of superconductor having a high current carrying capacity, by each of three processes listed below. The components of the total price are provided for each step of the process. In reviewing these figures one must keep in mind that they are based on very limited assumptions regarding the application of capital equipment costs to the price of the conductor. This is necessary because the conductor manufacturer is projecting future material requirements rather than those that are contemplating the construction of superconducting devices. However, the cost estimates for reduction schedules and other estimates have been consistently applied, so that while on an absolute basis there may be some inaccuracy, the comparative magnitude of these price projections should be meaningful.

Price estimates will be made for three processes:

1. A cabled composite produced by externally plating tin on the copper-niobium strands going into the cable.  
High conductivity copper is included prior to reaction.
2. A cabled composite produced by externally plating tin on the bronze-niobium strands going into the cable.
3. A monolithic conductor produced by codrawing the bronze-niobium section and high conductivity copper as a single monolithic conductor. The regions of bronze and niobium are surrounded by a tantalum barrier to prevent the diffusion of tin to the surrounding high conductivity copper matrix.

Cost estimates are made for a total of six conductors. The first three are all capable of carrying 1500 Amps at 12 Tesla and all have the same amount of high conductivity copper in the cross section. The difference between them is that they are manufactured by the three processes cited above. The second set of three conductors are capable of carrying 10,000 Amps at 12 Tesla.

The major determinants of the cost of the conductors are the materials and the cost of the various steps in the billet and wire reduction schedule. Prior to the cost estimates for the conductors a detailed accounting of the steps in the wire process is presented. In order to clearly explain the basis of these cost estimates the assumptions made in establishing them are listed.

Assumptions made in establishing comparative cost estimates for the production of multifilament  $Nb_3Sn$  conductors:

1. In the extrusion of a copper-niobium billet the ratio of original billet diameter to final diameter can be much greater than in a bronze-niobium billet, because the process can be carried out at a higher temperature without reaction between the niobium and bronze. For example, an 8" diameter can be reduced to 3/4" in the case of a niobium-copper billet but only extruded to 2" in the case of a niobium-bronze billet.
2. Electroplating of tin can be accomplished at a speed of 100'/minute with the application of about 75 mg/meter.

3. 75 mg/meter of tin can be applied to a .012" diameter niobium-copper composite without separation during the subsequent heat treatment. Most likely a procedure such as annealing under hydrostatic pressure, alloy plating or plating of Cu and Sn in alternate layers will be necessary to successfully apply this quantity of tin. For plating tin on a bronze composite only 32 mg/m on a .012" diameter composite. This is well within present capabilities.
4. Wire drawing can be accomplished by automatic multistage drawing equipment at 500'/min. We have assumed that constant attention is necessary which is a rather conservative assumption. Most likely a single individual can supervise several machines simultaneously.
5. Bronze requires annealing every 75% reduction in area.
6. 8" (dia.) x 24" billets are assumed. Quantities on the order of several thousand pounds of material are assumed.
7. The yield for an extrusion of a bronze-niobium as well as a copper-niobium billet is 80%.

1. Cost estimate for a monolithic conductor capable of carrying 1500 Amps at 12 Tesla (Model Conductor I)

Table II includes cost figures for steps in the processing of a monolithic conductor with a cross section as shown in Figure 13 and with characteristics described below. The design is similar to a multifilament Nb<sub>3</sub>Sn conductor which has been produced in long lengths and reported in the literature.<sup>8</sup>

Specifications for Model Conductor I:

1. # of tantalum protected regions - 19
2. Total # of reacted filaments - 67,507
3. Percentage of reaction - 64%
4. Thickness of reacted layer - 1 $\mu$
5. Composition of bronze by weight - 90% Cu, 10% Sn
6. Percentage of area of unreacted billet which is niobium - 13%
7. Percentage of area occupied by copper - 34% (.0045 in<sup>2</sup>)
8. Bronze/niobium ratio in tantalum protected region - 3.64:1
9. I<sub>c</sub> at 12T - 1590 Amps
10. Composition of bronze after reaction: ~ 4% by weight

TABLE I  
 PRICE COMPARISON OF REDUCTION SCHEDULES  
 FOR A BRONZE/NIOBIUM AND COPPER/NIOBIUM COMPOSITE

<u>Operation</u>	<u>Bronze/Niobium</u>	<u>Copper/Niobium</u>
Extrusion	\$1,000	\$1,000
Intermediate Anneal	\$2,000	--
Reduction to .010" Diameter	<u>\$6,000</u>	<u>\$5,500</u>
Total	\$9,000	\$6,500



2. Cost estimate for a cabled multifilament Nb<sub>3</sub>Sn conductor capable of carrying 1500 Amps at 12 Tesla (Model Conductor II)

Table III includes cost figures for steps in the manufacture of a cabled conductor with a cross section as shown in Figure 14 and with characteristics described below. The design provides the same critical current as well as the same amount of high conductivity copper as Model Conductor I. This design includes the protective copper in an awkward way but provides similarity to Model Conductor I. For the sake of more efficient packing twelve copper elements rather than six would be included in the second level.

Specifications for Model Conductor II:

1. Diameter of Cu-Nb strand - .012" .305 mm
2. Cu:Nb composite ratio - 2:1
3. Thickness of reaction region -  $1\mu$
4. Diameter of filament -  $5\mu$
5. # of filaments per strand - 1235
6. Critical current at 12T - 1548A
7. % of filament reacted - 64%
8. Composition of residual bronze after reaction - 4% Sn by weight
9. Total area of protective copper cabled into the composite -  $.0291 \text{ cm}^2$  ( $.004512 \text{ in}^2$ )
10. Amount of tin plated/meter - 75 mg/m
11. Diameter of smaller pure copper strands - .012"
12. Diameter of larger pure copper strands - .035"

TABLE II

COST ESTIMATE FOR 1000. LB. OF MODEL CONDUCTOR 1

<u>Description of Step</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step I - Casting of bronze for billet. Nineteen niobium elements are included. Holes in the bronze casting will be formed by drilling or provisions will be made in the casting mold.	1094 lb of unextruded material in the form of billets 8" (dia.) by 24" containing 19 Nb cores. Bronze:niobium ratio = 3.64/1 2.74 Billets	<ol style="list-style-type: none"> <li>1. Casting bronze (labor)</li> <li>2. Casting bronze (material)</li> <li>3. Drilling holes for Nb cores</li> <li>4. Niobium rod (.85" dia.)</li> <li>5. Packing billet</li> <li>6. Machining bronze ends</li> <li>7. Casting bronze ends (labor &amp; materials)</li> <li>8. Electron beam welding of billet</li> </ol>	\$22,900
Step II - Extrude niobium-bronze billet to 2" diameter, Reduce on draw bench and finally draw to hexagonal cross section. Assumed yield: 80% (2.74 Billets)	Hexagonal rods of a niobium-bronze composite. In order to produce 875 lb of niobium-bronze rod one must start with 1094 lb of billet	<ol style="list-style-type: none"> <li>1. Extrusion of the 8" (dia.) to 2" diameter rod</li> <li>2. Draw to circular cross section of about 9/16" (This process includes two anneals)</li> <li>3. Cost for annealing</li> <li>4. Handling and supervision of out of house operations</li> <li>5. Draw to hexagonal cross section</li> </ol>	\$10,400
Step III - Pack 187 of the 19 niobium filament elements into a tantalum shell with a bronze shell surrounding the elements. A copper shell surrounds the tantalum.	Billet with tantalum shell 8" dia. x 24" long (3.4 billets) 1360 pounds of billet must be produced in this step	<ol style="list-style-type: none"> <li>1. Fabricate bronze shell (labor &amp; material)</li> <li>2. Produce copper ends and outer shell (labor &amp; material)</li> <li>3. Straighten and pack 187 hexagonal elements into the billet</li> <li>4. Weld ends onto billet</li> <li>5. Produce tantalum shell (labor &amp; material) (144 lb)</li> </ol>	\$40,100

TABLE II  
COST ESTIMATE FOR 1000 LB. OF MODEL CONDUCTOR I (cont'd)

<u>Description of Shell</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step IV - Extrude billet with tantalum shell from 8" diameter to 2" diameter. Produce hexagonal elements 1.40" side to side. Extrusion yield: 80%	1360 lb of billet is required to produce 1087 pounds of finished product (3.4 billets)	<ol style="list-style-type: none"> <li>1. Extrude 8" diameter billet to 2"</li> <li>2. Draw to 1.5" diameter</li> <li>3. Draw to hexagonal cross section</li> <li>4. Handling and supervision of out of house operation</li> </ol>	\$ 8,100
Step V - Repack tantalum shelled elements into a billet along with the remainder of the protective copper	Billets containing about \$1250 lb of material 3.2 billets	<ol style="list-style-type: none"> <li>1. Pack tantalum shelled elements and additional copper into billet</li> <li>2. Machine copper ends</li> <li>3. Additional copper (170 lb.) for can</li> <li>4. Machine copper can</li> <li>5. Copper for ends (225 lb.)</li> </ol>	\$ 7,100
Step VI - Extrude billet 8" to 2" diameter. Reduce by bench drawing and bull block drawing. Final diameter .130". Flatten conductor. Extrusion yield 80%.	1000 pounds of unreacted conductor (~3.2 billets)	<ol style="list-style-type: none"> <li>1. Extrude billet from 8" diameter to 2" diameter</li> <li>2. Reduce 2" to 3/8"</li> <li>3. Reduce 3/8" → .130"</li> <li>4. Handling and supervision of out of house operation</li> </ol>	\$15,000

COST ESTIMATE FOR 1000 LB. OF MODEL CONDUCTOR I (cont'd)

<u>Description of Step</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step VII - Reacted conductors.	Reacted conductors	1. Respool conductor with separation of layers to prevent cable from welding to itself 2. Heat treatment in furnace at ~700°C under a protective atmosphere	\$ 700

TOTAL COST OF 1000 LB. OF CONDUCTOR: \$104,300

PRICE PER POUND \$104.30

PRICE PER METER: \$ 18.00 (TOTAL 5800 METERS)

TABLE III  
COST ESTIMATE FOR 5800 METERS OF MODEL CONDUCTOR II

<u>Description of Step</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step I - Pack niobium-copper billet with 1235 rods of niobium in hexagonal tubing. Weld ends and compact billet.	Billet with copper-niobium ratio of 2:1. Total billet 1.4 8" x 24" billets	<ol style="list-style-type: none"> <li>1. Machine billet ends</li> <li>2. Billet ends (material)</li> <li>3. Billet can (material)</li> <li>4. Machine billet can</li> <li>5. Copper rods and tubing (\$2.50 lb.) (240 lb.)</li> <li>6. Niobium rods (166 lb.)</li> <li>7. Weld ends</li> <li>8. Packing billet</li> </ol>	\$15,800
Step II - Pack Cu-Ta billet.	Tantalum protected copper billet. Total number of billets 2.8	<ol style="list-style-type: none"> <li>1. Billet ends (material)</li> <li>2. Machine billet ends</li> <li>3. Billet can (material)</li> <li>4. Machine billet can</li> <li>5. Copper cylinder for billet (756 lb.)</li> <li>6. Tantalum shell (96 lb.)</li> <li>7. Packing billet</li> <li>8. Weld ends</li> </ol>	\$23,600
Step III - Extrude niobium-copper billet 8" diameter to 3/4" diameter	3/4" rod of the Nb-Cu composite 1.4 billet Yield 80%	<ol style="list-style-type: none"> <li>1. Extrude billet</li> <li>2. Supervision and handling of an out of house task</li> </ol>	\$ 2,500

TABLE III

## COST ESTIMATE FOR 5800 METERS OF MODEL CONDUCTOR II (cont'd)

<u>Description of Step</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step IV - Extrude tantalum-copper billet 8" diameter to 3/4" diameter	3/4" rod of the Ta-Cu composite 2.8 billets Yield 80%	1. Extrude billet 2. Supervision and handling of an out of house task	\$4,100
Step V - Draw 3/4" niobium-copper composite rod to .012" diameter	.012" diameter composite wire	1. Draw 3/4" rod to .012" wire	\$8,800
Step VI - Draw 3/4" tantalum copper composite rod to .035" (90%) and to .012" (10%)	.035" diameter wire and .012" diameter wire to be included in final cable 2.8 billets	1. Draw 90% of wire to .035" diameter 2. Draw 10% of wire completely to .012" diameter	\$7,700
Step VII - Plating niobium-copper composite with .075 g/m of tin at 100 ft/min (984,000 ft) total	Tin plated niobium-copper composite 984,000	1. Plate 780,000 ft of wire at 500 ft/min 2. 40 lb of tin & plating solution	\$6,400
Step VIII - First cabling operation	7 strand cable consisting of one strand of high conductivity copper and 6 strands of super-conductor. Cable pitch of 9/inch yields 41,600 meters of cable	1. Cable at a pitch of 9/inch at a rate of 50 feet/min	\$1,600
Step IX - Second cabling operation	Total cable unreacted and without solder filling. A pitch of 3/inch yields 5800 meters of cable	1. Cable at a pitch of 3/inch at a rate of 50 feet/min	\$ 700

TABLE III

## COST ESTIMATE FOR 5800 METERS OF MODEL CONDUCTOR (cont'd)

<u>Description of Step</u>	<u>Final Product</u>	<u>Tasks and Materials Involved</u>	<u>Price</u>
Step X - React cable	Reacted conductor	<ol style="list-style-type: none"> <li>1. Respool conductor with separation of layers to prevent cable from welding to itself</li> <li>2. Heat treatment in furnace at <math>\sim 700^{\circ}\text{C}</math> under a protective atmosphere</li> </ol>	\$1,800
Step XI - Fill cable	5800 meters of filled cable	<ol style="list-style-type: none"> <li>1. Fill cable at 20 ft/min</li> <li>2. Total quantity of solder-50 lb.</li> </ol>	\$1,700

TOTAL COST FOR 5800 METERS: \$74,700

PRICE PER METER: \$13.00

3. Cost estimate for a cabled conductor capable of carrying 1500 Amps at 12 Tesla produced by a hybrid process

The cabling configuration of such a conductor would be same as that of Model Conductor II except that instead of using a .012" diameter copper-niobium strand plated with 75 mg/meter of tin a .012" diameter bronze-niobium strand plated with 32 mg/meter of tin would be used. The difference in processing and cost would be the following:

1. The extrusion of the original 8" diameter billet would yield a rod of 2" diameter. This would be followed by drawing of the rods from a 2" diameter to 3/4". The added price would be \$12,500 in Step III of Table III.
2. Several anneals would be necessary in Step V of Table III yielding a total increase of \$2,500 in Step V. The price per meter of the conductor would therefore be \$16.00/meter.

4. Cost estimate for a monolithic conductor capable of carrying 10,000 Amps at 12 Tesla.

Consider a conductor with the same cross sectional ratio of components as Model Conductor I. In order to achieve a critical current of 10,000 Amps the cross sectional area of the conductor would be 6.7 times that of Model Conductor I. In order to maintain the same filament size the number of tantalum protected regions would have to be multiplied by the same number. The following modifications of the processing of Model Conductor I would have to be made:

1. In Step IV of Table II the final diameter of extruded and drawn tantalum protected elements would be about  $1/6.7$  the area of that in Model Conductor I. This would add \$3,400 to the total price of the 1000 lb conductor.



2. In Step VI the final diameter would be about 6.7 times that for Model Conductor I. The decrease in the total price for 1000 lbs of conductor would be \$2,080.

Therefore the price of 1000 lbs of conductor would be \$105,600. The price per meter would be \$122.

5. Cost estimate for a cabled conductor capable of carrying 10,000 Amps at 12 Tesla produced by diffusing tin into a copper-niobium composite

The process for the production of such a conductor would be similar to that of Model Conductor II but with the following modifications:

1. The cost per meter would be multiplied by a factor of 6.7 to reflect the increase in the number of strands necessary to carry the increased current.
2. The process would involve at least one additional cabling operation at a cost of \$.12 per meter.
3. There would be a shrinkage of the conductor upon cabling resulting in a multiplication of about 1.1 of the price per meter.

The total price per meter would therefore be \$96.

6. Cost estimate for a cabled conductor capable of carrying 10,000 Amps at 12 Tesla consisting of strands produced by diffusing tin into a bronze-niobium composite

The process in the production of such a conductor would be similar to that of the 1500 Amp conductor produced by the hybrid process with the following modifications:

1. The price per meter would be multiplied by a factor of 6.7 to reflect the increase in the number of strands necessary to carry 10,000 Amps rather than 1500A at 12 Tesla.
2. The process would involve an additional cabling operation at a cost of \$.12 per meter.
3. There would be a shrinkage of the conductor upon cabling resulting in a multiplication of about 1.1 in the price per meter.

The cost of the conductor per meter would therefore be \$107.

#### IV. ADVANTAGES OF A CABLED CONDUCTOR

The cost figures of the previous section clearly show that a cabled composite involving copper-niobium strands plated with tin has an economic advantage over a monolithic conductor. Furthermore, a cabled composite produced from a tin plated bronze-niobium composite is at least competitive with a monolithic bronze composite. There are, however, several additional advantages of a cabled conductor. The cabling process introduces a degree of transposition which is impossible in a monolithic conductor. This feature reduces adverse self field effects which may lead to increased losses during the approach to an equilibrium flux distribution particularly when the field of the device is changing rapidly.

Another important benefit of a cabled approach is its design flexibility. In the development of a conductor for a particular application several conductor configurations may be considered. The building block feature inherent in the cabled approach allows one to consider several configuration alternatives without the expense of billet design and extrusions. A particular feature of this design flexibility is the ability to cable a conductor which may accommodate ventilation by the liquid helium through the conductor.

An additional feature possessed by the cabling method is the ability to include strengthening members within and throughout the conductor cross section. With the extremely high magnetic forces and resulting tensile stresses along the conductor, the ability to increase the elastic modulus of the overall conductor by cabling about tungsten or stainless steel becomes a significant advantage.

Cabling may be the only means of overcoming the problems that tantalum diffusion barriers provide both in cost and in tendency towards rupture. The tantalum protected copper elements in a cable do not have to be drawn to the degree of reduction that they are in a monolithic conductor. Furthermore, the tantalum barriers are not in as intimate contact with the bronze niobium. The consequences of these features is that less tantalum is used, there is less tendency towards barrier rupture and that if rupture occurs there is less likelihood of the high conductivity copper being contaminated.

The most important feature, however, is the increased mechanical flexibility which results from cabling. The analysis of a spring under tensile loading demonstrates how this increased flexibility occurs. The maximum shear stress ( $\tau_m$ ) in a tightly coiled spring is given in terms of the apparent tensile strain along the axis of the spring ( $\epsilon'$ ) by the following formula:

$$\tau_m = \frac{Gd\epsilon' (1 + d/4R)}{4\pi n R^2}$$

Where G is the elastic shear modular, d is the diameter of the spring wire, R is the radius of the spring and n is the pitch of the spring i.e. number of turns per unit length. The maximum shear strain in the composite ( $\gamma$ ) is given by the following formula:

$$\gamma = \tau_m/G$$

The maximum real tensile strain (  $\epsilon$  ) along the strand in terms of the axial tensile strain is therefore:

$$\epsilon = \frac{d\epsilon' (1 + d/4R)}{8\pi n R^2}$$

For the cabled composite where  $1/8d$  is the pitch and  $R=d$ , the actual strain in terms of the apparent strain is:

$$\frac{\epsilon}{\epsilon'} = \frac{5}{4\pi} = .4$$

In other words by cabling a conductor we may not only increase the modulus of the overall composite we may reduce the strain in the superconductor by a factor of 2.5.

## V. SUMMARY

During IGC's earlier development of the external diffusion process for the production of multifilament  $Nb_3Sn$  a serious problem was encountered which seemingly threatened to limit our useful strand diameter to less than .007". This problem involved the acquisition of all the plated tin by a shallow layer of the niobium-copper composite substrate. The restriction to a .007" diameter severely increased the expense of a high current conductor because of the additional drawing and cabling involved. The present study has provided insight into the mechanism of the separation as well as pointed the way to the development of economically viable process alternatives which can prevent its formation. We now realize that a multiple step plating process with annealing following each plating step is a reasonable way of avoiding the separation problem. Other alternatives available to us are Cu-Sn alloy plating and annealing under hydrostatic pressures.

In addition to the separation problem there are other problems which are common to both the external diffusion method and the monolithic conductor technique. The problem of void formation near the niobium filaments can be avoided by external tinning of the composite. The problem of rupture of the tantalum diffusion barriers can conceivably be avoided by annealing operations during the reduction of the conductor. We are, furthermore, confident that the problem of tantalum barriers can be circumvented in the cabling approach.

We have included in this report an economic analysis of the various means available to us in the production of multifilament  $Nb_3Sn$ . This analysis demonstrates the economic advantage of a cabled conductor formed by externally tinning a copper-niobium composite. These price estimates also show that a hybrid process involving cabling of a tin plated bronze-niobium composite

is at least competitive with a monolithic bronze-niobium composite.

There are several other advantages of a cabled composite:

1. Greater design flexibility.
2. Improved tensile strength by incorporating high elastic modulus material such as tungsten or stainless steel.
3. Greater mechanical flexibility resulting from cabling geometry.
4. The hope of avoiding diffusion barrier rupture problems.

## REFERENCES

1. Scanlan, R. M. and Fietz, W. A., "Multifilamentary Nb<sub>3</sub>Sn for Superconducting Generator Applications", IEEE Trans. on Magnetics Vol. Mag-11 No. 2, p. 287, March 1975 and Fietz, W. A.; Henning, C.D., and Scanlan, R. M. *ibid* p. 299
2. A. McInturff, Private Communication.
3. Kawakatsu, I., Osawa, T., Yamazuchi, H., "On the Growth of Alloy Layer Between Solid Copper and Liquid Tin", Trans. J.I.M., Vol. 13, p. 436 (1972)
4. Kidson, G. V., "Some Aspects of the Growth of Diffusion Layers in Binary Systems", Journal Nucl. Mat. Vol. 3, #1, p. 21 (1961)
5. Shewmon, P. G., Diffusion in Solids, McGraw-Hill, New York, (1963).
6. Bastow, B. D., Kirkwood, D. H., "Binary and Ternary Diffusion in the Cu Corner of the Copper-Nickel-Tin System", Journal Inst. Met., Vol. 100, p. 24, (1972).
7. Hansen, M., Constitution of Binary Alloys, McGraw-Hill, New York, 1958.
8. Clay, B. D., Greenwood, G. W., "Marker Movement and Void Formation During Interdiffusion in the Cu-Ni System and the Effect of Hydrostatic Pressure", Phil. Mag. Vol. 20, p. 1201, (1972).
9. Gregory, E., Marancik, W. G., Ormand, F. T., IEEE Trans., Mag-11, No. 2, p. 295, March 1975.
10. Timoshenki, S., Young, D. H., Elements of Strength of Materials, Van Nostrand Reinhold Company, New York (1968)
11. Foundation of Solid Mechanics, Y. C. Fung, Prentice Hall, (1965).



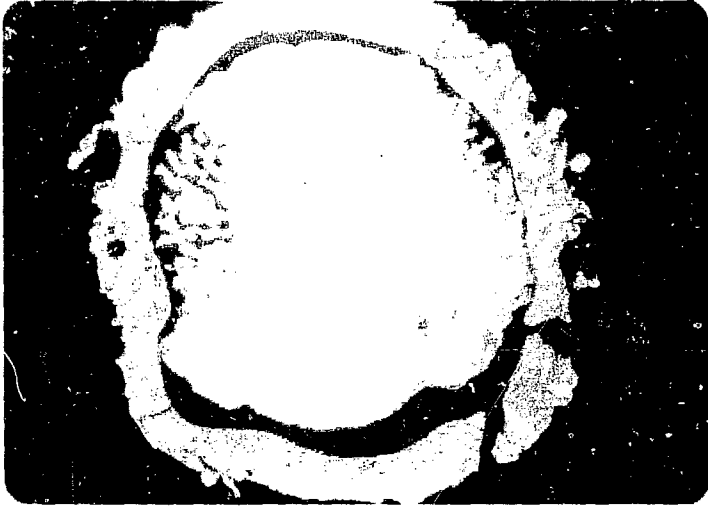


Figure 1 Color photomicrograph showing an .011" diameter composite strand exhibiting separation of the outer region having been plated with 90 mg/meter of tin and heat treated at 580°C for 24 hours. Magnification: 150X

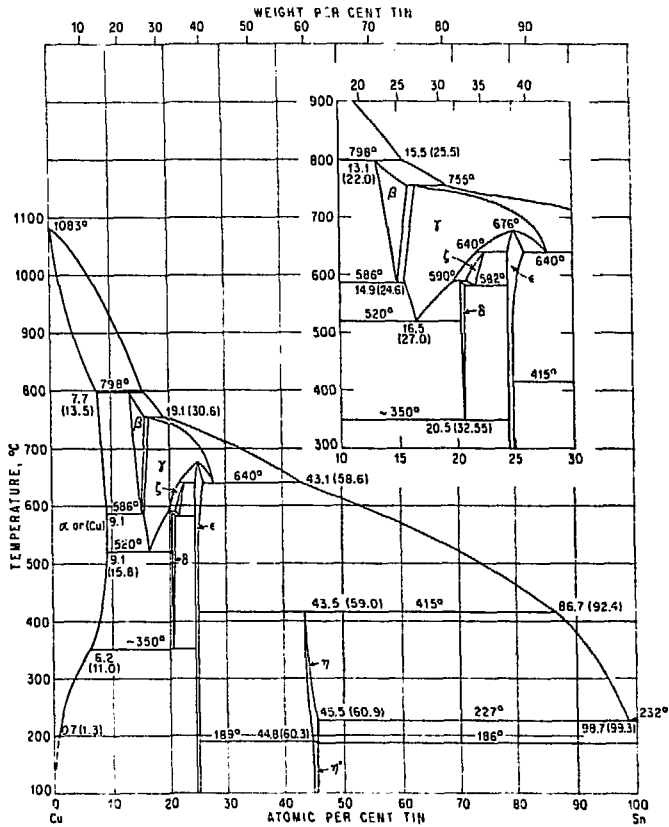


Figure 2 Phase diagram for copper-tin system.

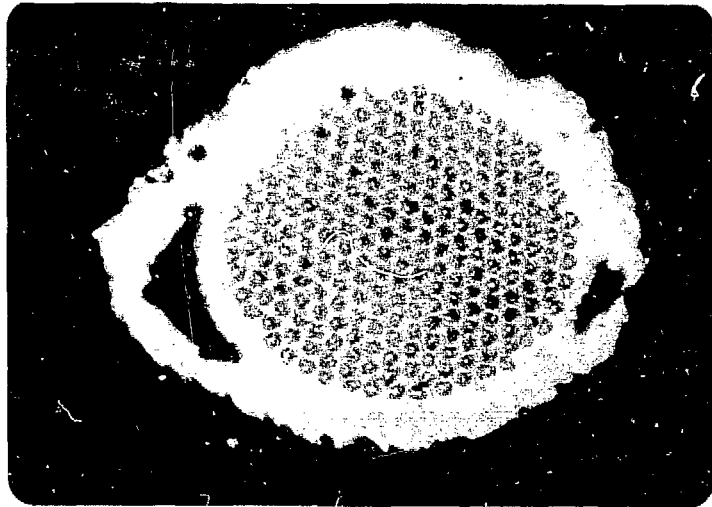
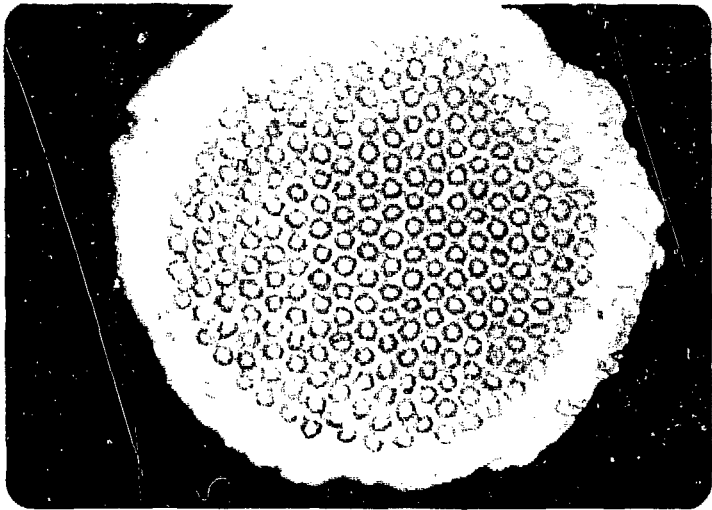


Figure 3 Copper-niobium composite (ratio 2.5:1) wire of .006" diameter which was plated with varying amounts of tin, homogenized at 580°C for 48 hrs., and reacted at 750°C for 48 hours. The amounts of plated tin were: a. 17 mg/meter b. 19.5 mg/meter. Magnification: a. 550X b. 410X

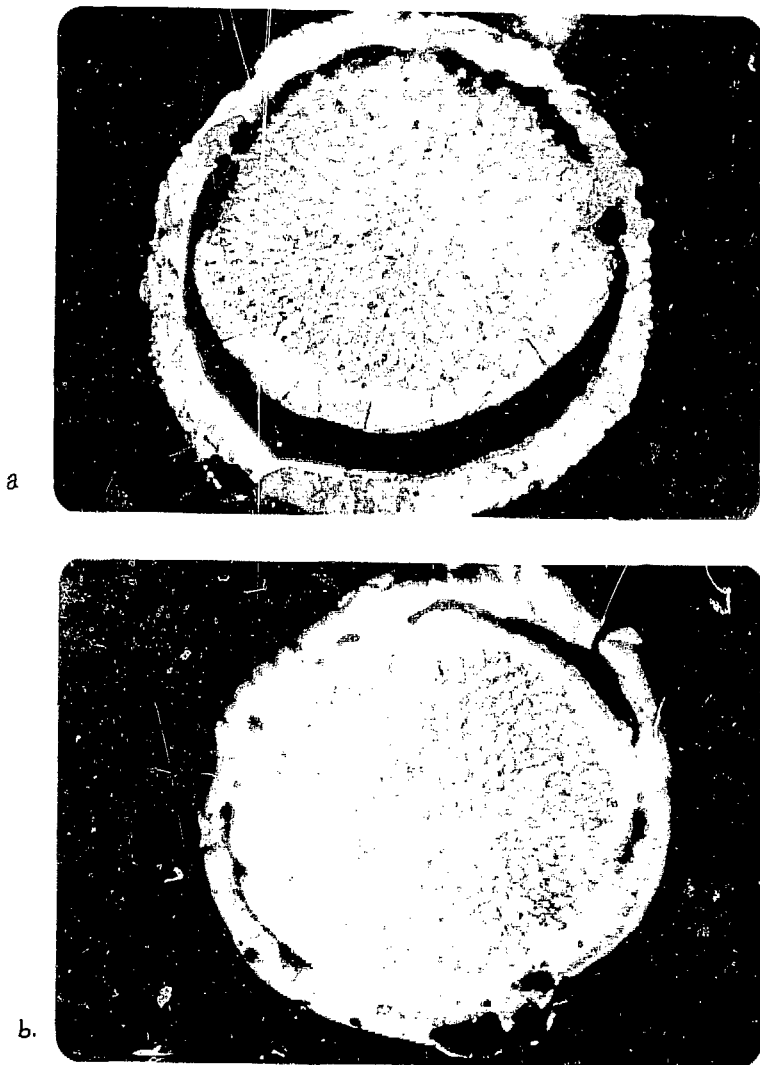


Figure 4 Composite wire (.0106" diameter) plated with 60 mg/meter or tin followed by different amounts of nickel and heat treated at 580°C for 48 hrs. The amounts of nickel plated were: a. 2 mg/meter b. 19 mg/meter. Magnification: a. 235X b. 290X

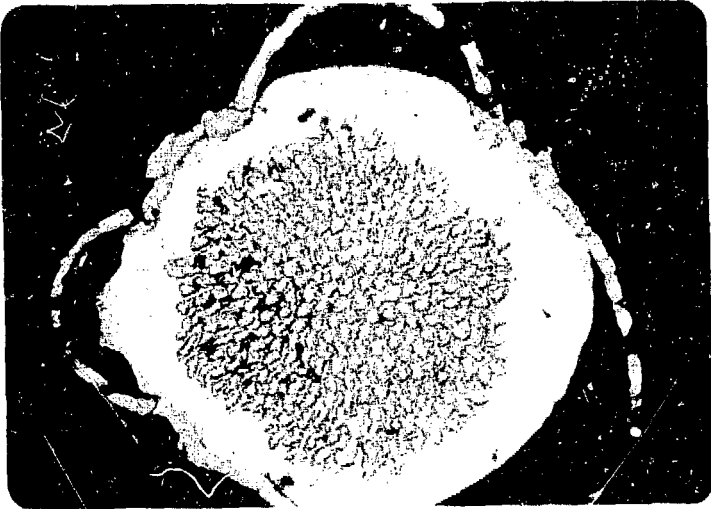


Figure 5 Photomicrograph showing the result of plating Ni-50 at/% Sn alloy on a 10.6 mil diameter copper-niobium composite. Following the plating, the sample was heat treated 18 hrs. at 580°C. The weight of the plate was 50 mg/meter. The amount of tin in this plated alloy was therefore 34 mg/meter. Magnification: 235X

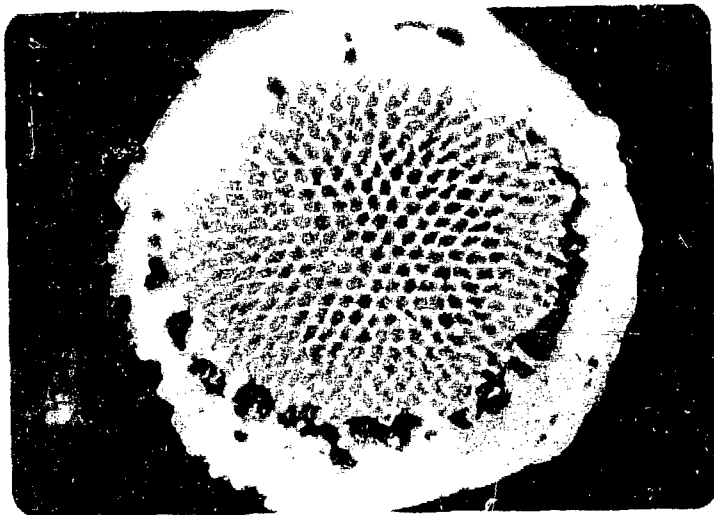


Figure 6 Photomicrograph showing the result of plating 140 mg/meter of a Cu-Sn alloy containing about 50-60 wt. % tin on a 10.6 mil diameter conductor. Following the plating the sample was homogenized at 580°C for 48 hrs. and reacted at 750°C for 72 hrs. Magnification: 295X

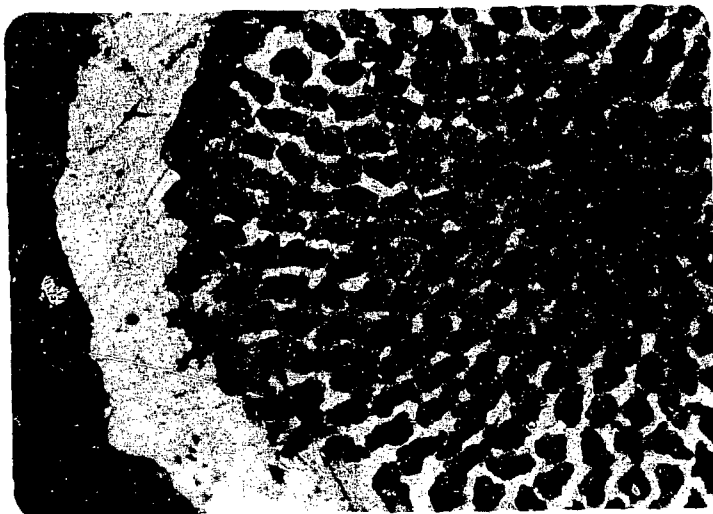


Figure 7 Photomicrograph showing the degree of reaction of the composite wire shown in Figure 6. Darker regions are those of Nb<sub>3</sub>Sn. Lighter regions of the filaments occurring as dots near the center of them or near voids are those of unreacted niobium. Magnification: 550X

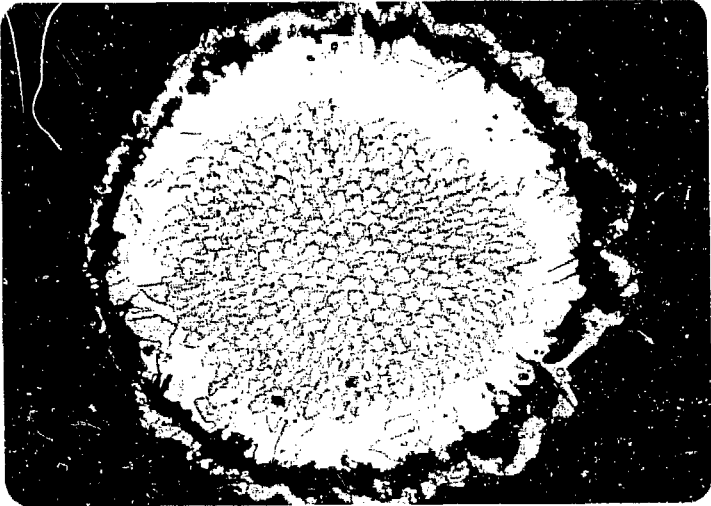


Figure 8 Photomicrograph of 10.6 mil diameter composite wire plated with 60 mg/meter of Sn, 31 mg/meter of Cu and finally heat treated at 580°C for 16 hrs. to promote homogenization. Magnification: 235X



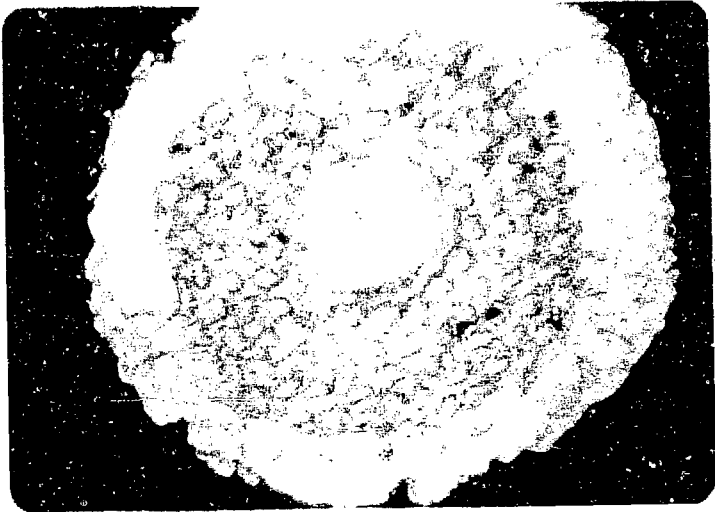


Figure 9 Photomicrograph of 10.6 mil diameter bronze-niobium composite wire plated with 30 mg/meter of tin. Following plating the sample was heat treated at 580°C for 16 hrs. No separation was evident in any cross section. Magnification: 340X

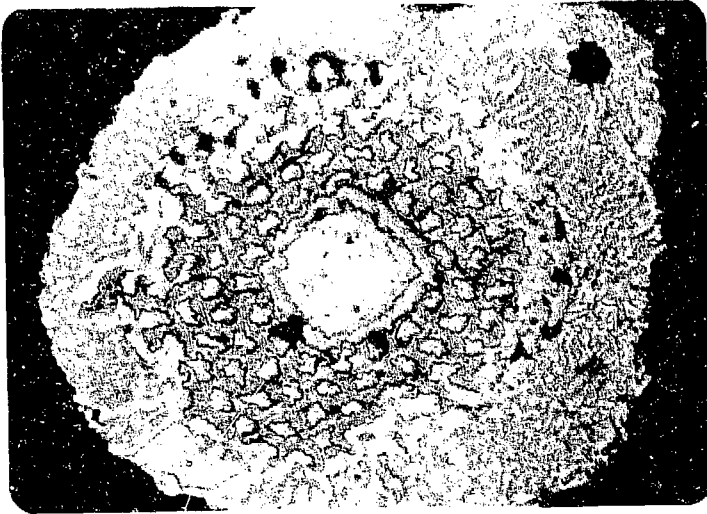


Figure 10 Photomicrograph of 10.6 mil diameter bronze-niobium composite wire plated with 60 mg/meter of tin. Following plating the sample was heat treated at 580°C for 16 hrs. The separation evident in this photograph was the worst observed in any cross section but is substantially less severe than that of the material provided earlier to Lawrence Livermore Lab. Magnification: 340X

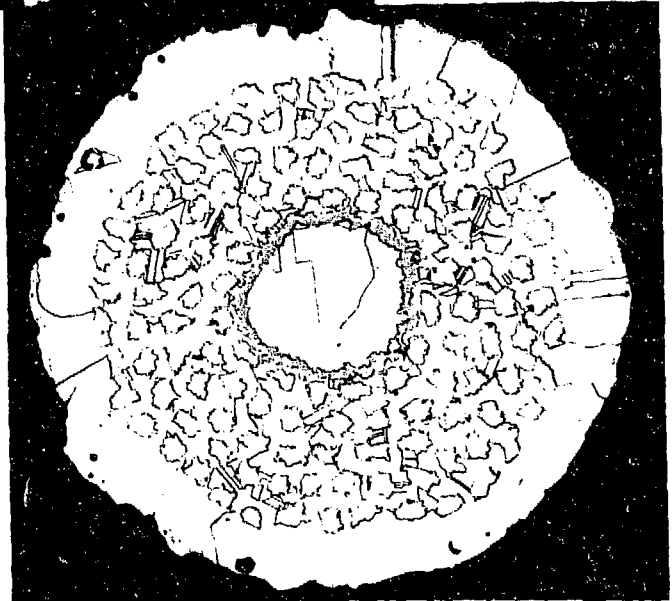
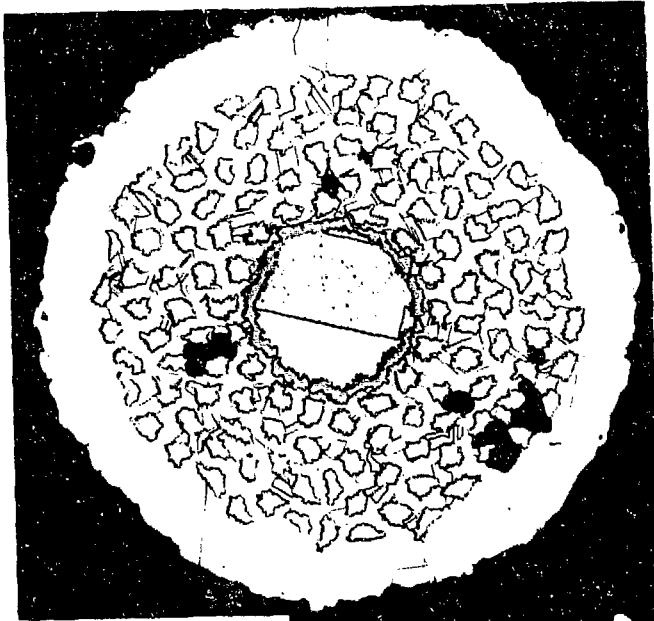


Figure 11 a. Photograph showing bronze-niobium composite which had been homogenized at 580°C for 24 hrs. prior to reaction at 750°C for 48 hrs. Magnification: 375X

b. Photomicrograph showing bronze-niobium composite which was directly reacted at 750°C for 48 hrs. (Work performed by R. Scanlan at the GE Research & Development Labs) Magnification: 375X

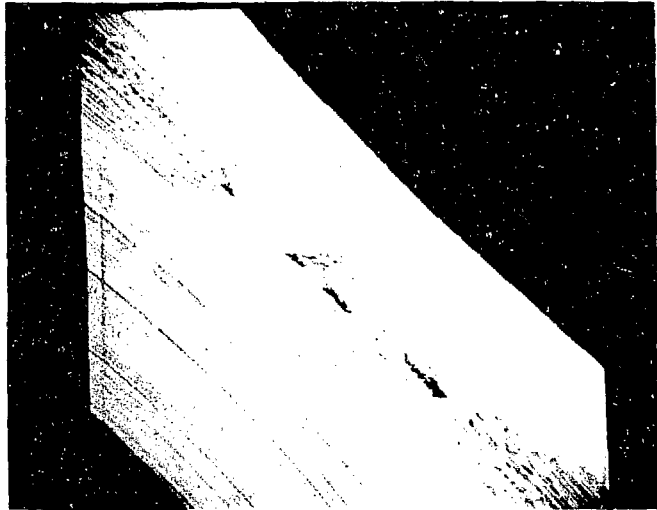
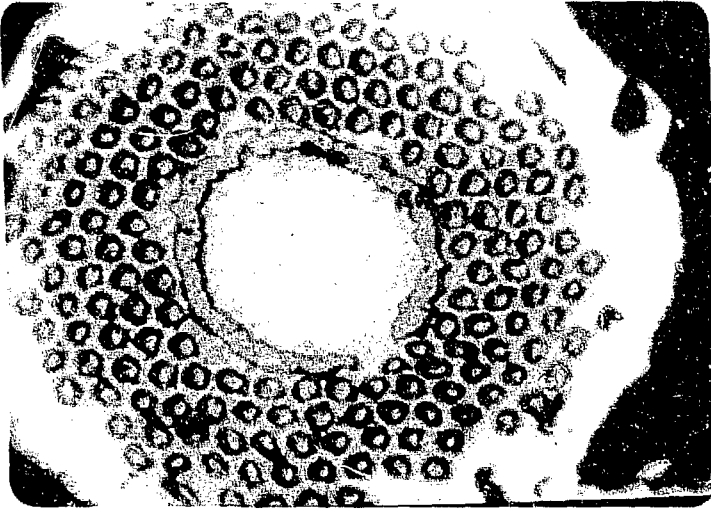


Figure 12 a. Photograph showing a .010" composite of niobium, copper, and tantalum. The ring in the center is the tantalum diffusion barrier. Magnification: 520X

b. Scanning electron photomicrograph showing lengthwise grooves and holes which occasionally develop in the tantalum barrier. The diameter of the wire composite was .015". Magnification: 500X

Figure 13  
Model Conductor I

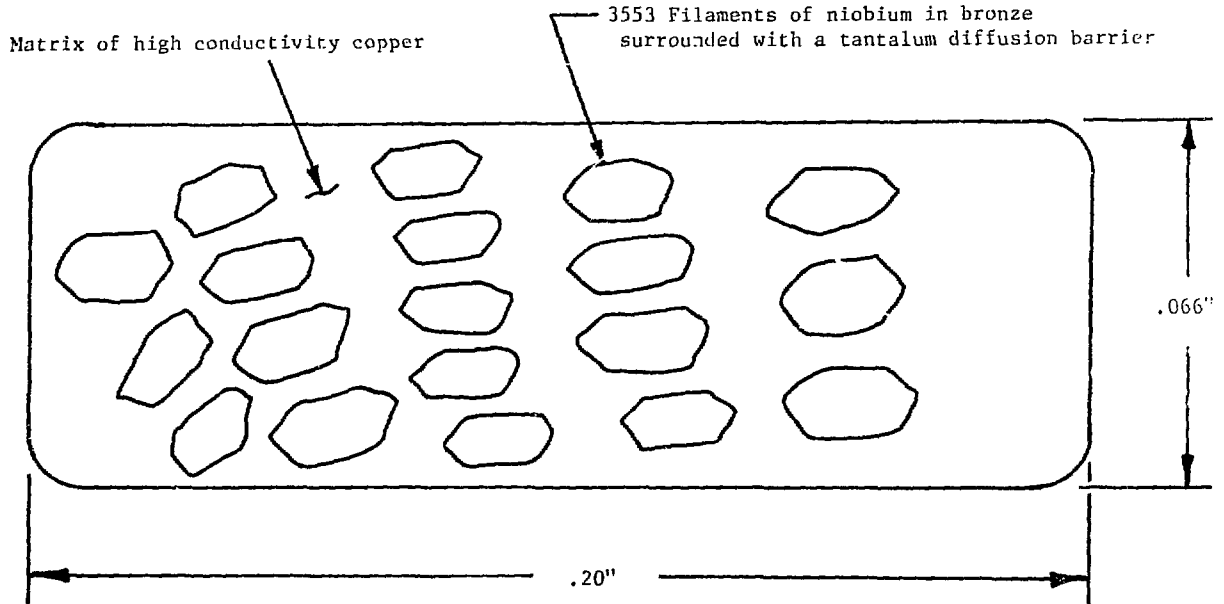
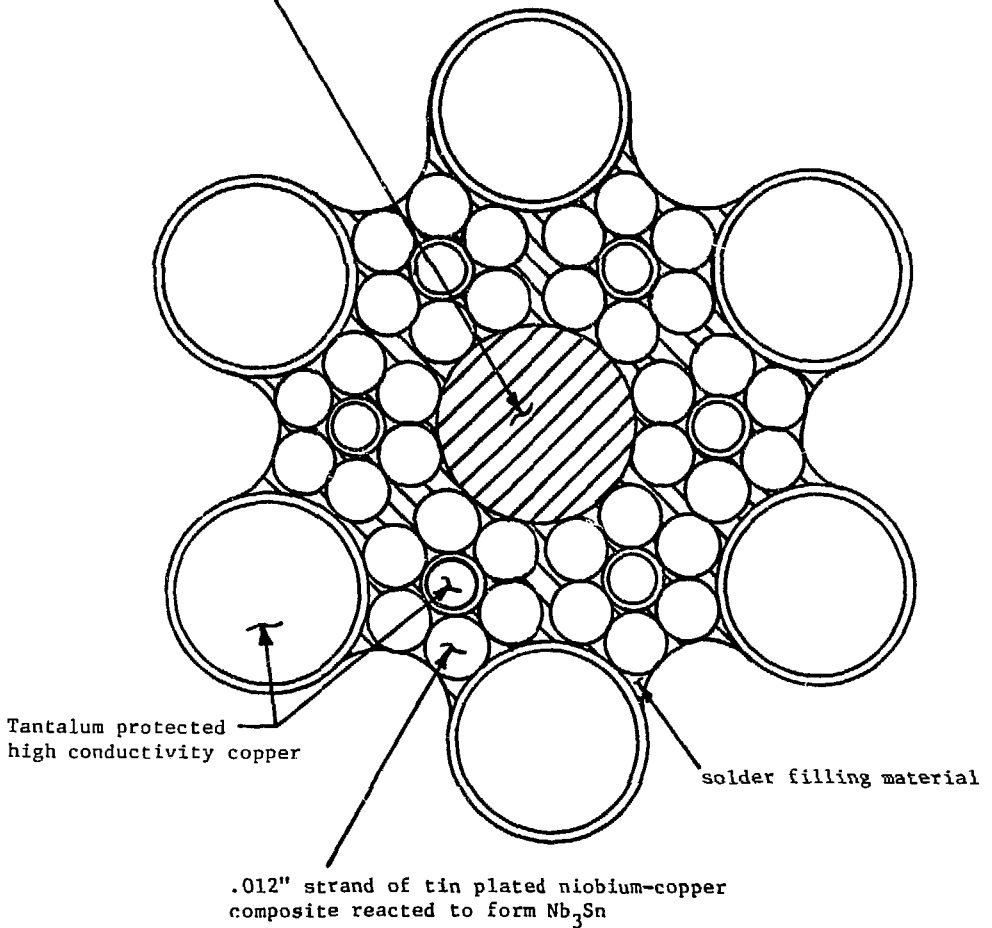


Figure 14

Model Conductor II

Tungsten or stainless steel strengthening element



**Appendix D**  
**Report on Development of Explosive Joints**  
**in Nb-Ti Superconductors, June 27, 1975**  
**(Battelle Columbus Laboratories, Columbus, OH)**

June 27, 1975

University of California  
Lawrence Livermore Laboratory  
P.O. Box 808  
Livermore, California 94550

Attention Mr. A.J. Hodges/Mr. R. Liptai

Gentlemen:

SANL 358-025

This letter constitutes Battelle's final report for the work performed under SANL 358-025. The objective of this program was to develop procedures to explosively weld CTR superconductors. This work has been completed and the following were delivered to LLL:

4-each, Welded CTR Superconductors, 18 inches (45.7 cm) long.

EXPERIMENTAL EXPLOSIVE WELDING

Research was conducted to develop the parameters for making joints by explosive welding in CTR superconductor stock, 6 mm by 6 mm cross section. This material consists of several hundred niobium alloy wires in a copper matrix. Joining was to be accomplished with a minimum loss of conductivity.

Preliminary Welding Studies

Preliminary welding parameters for the superconductor material were established with copper bar whose cross sectional area approximately corresponded to that of the superconductor (0.250 inch square or 6.35 mm square). Joints that can be explosively welded are limited to lap joints or versions thereof. A tapered or scarf joint design was selected for this application to (1) obtain a large joint surface and (2) minimize machining operations. Two tapers were investigated: 6 and 12 degrees.



The manner in which the workpieces were arranged for welding is shown in Figures 1a and 1b. The upper joint member (laminated to a copper shield plate) was positioned 0.031 inch (0.79 mm) above the lower member. The copper shield plate was formed to produce a 6 (or 9) degree angle between the upper and lower joint members.

Data obtained during these studies are summarized in Table 1. In all instances, welding was done with SWP-1 explosive, a nitrostarch sensitized ammonium nitrate explosive that detonated at 3050 m/sec.

The wave morphology and joint microstructures are shown in Figures 2a and 2b. The typical wavy interface associated with explosive welding occurred at the start of welding. As welding progressed, the wave amplitude decreased until an essentially flat interface was produced.

Based on these studies, it appeared that acceptable joints could be produced in accordance with the following parameters:

Type of explosive	:	SWP-1
Explosive loading	:	1.98 gm/cm <sup>2</sup>
Stand-off distance	:	0.031 inch
Stand-off angle	:	6°
Workpiece taper	:	12°

### Welding of Superconductor Material

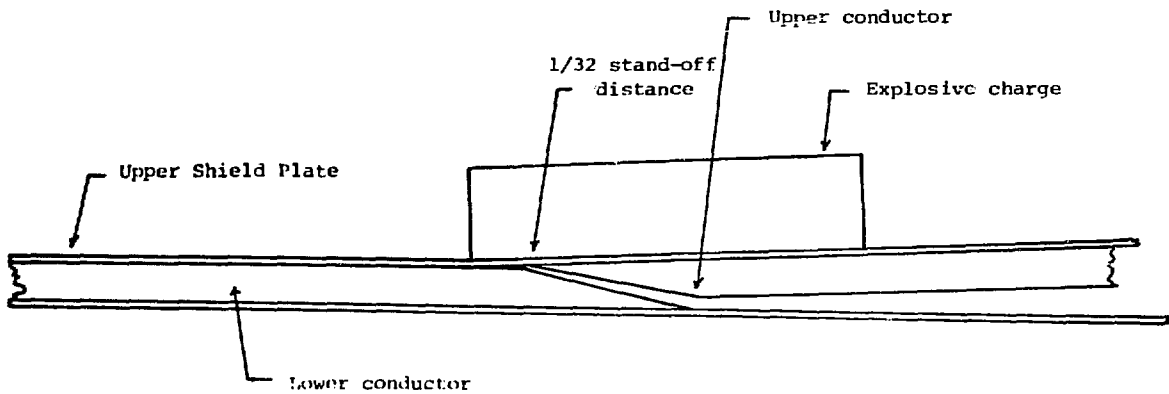
#### Initial Studies

The superconductor bar stock was machined to produce specimens with a 12 degree taper. Following machining, the component parts of the joint (workpieces, side rails, shield plates, and explosive charge) were arranged for welding as shown in Figure 1a. Since the superconductor material was stronger and stiffer than the copper bar stock, experimental welds were made with increasing explosive loading as indicated below:

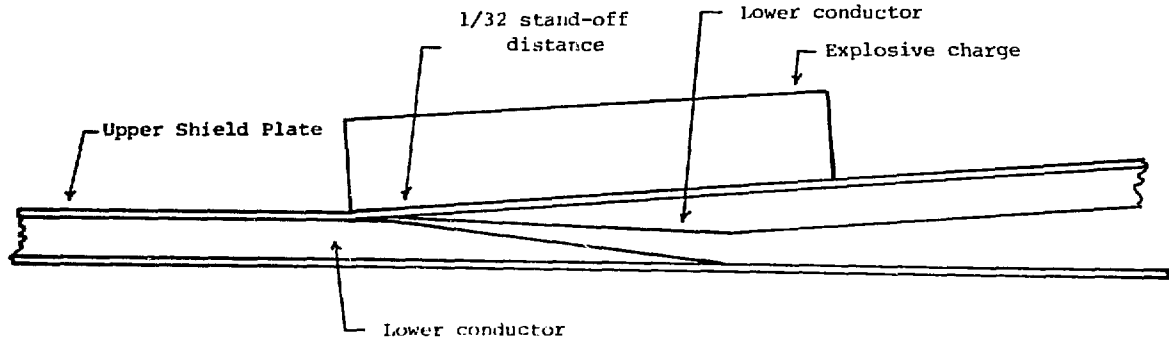
LC-1	:	1.98 g/cm <sup>2</sup>
LC-2	:	2.31 g/cm <sup>2</sup>
LC-2	:	2.64 g/cm <sup>2</sup>

Photomicrographs of these joints are shown in Figures 3a, 3b, and 3c. Some metallurgical features of these joints are illustrated in Figures 4a and 4b.

The weld made with an explosive loading of 2.31 g/cm<sup>2</sup> was most acceptable in terms of wave morphology and completeness of bonding. However, it was suspected that the difference in joint quality of Specimens LC-1, LC-2, and LC-3 was more associated with the accuracy of parts lay-up (e.g. stand-off distance, stand-off angle, etc) than the specific explosive loadings. Nonetheless, it was decided to weld specimens for joint evaluation with an explosive loading of 2.31 g/cm<sup>2</sup>.

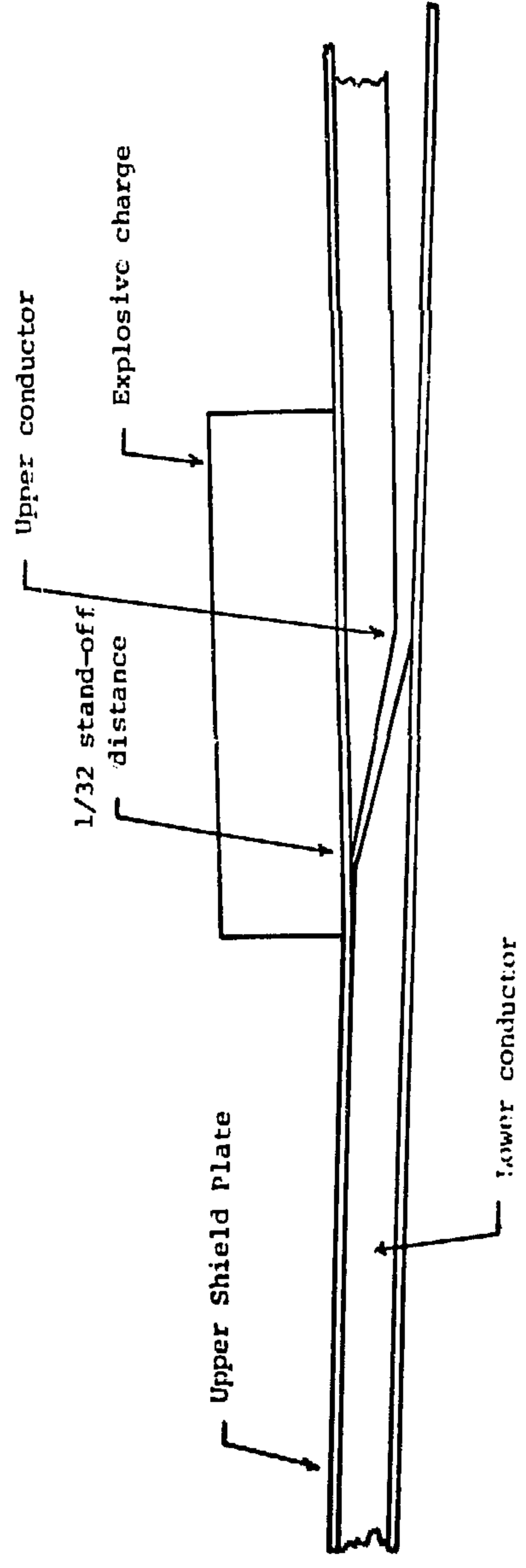


a. 120 Tapered Workpieces.

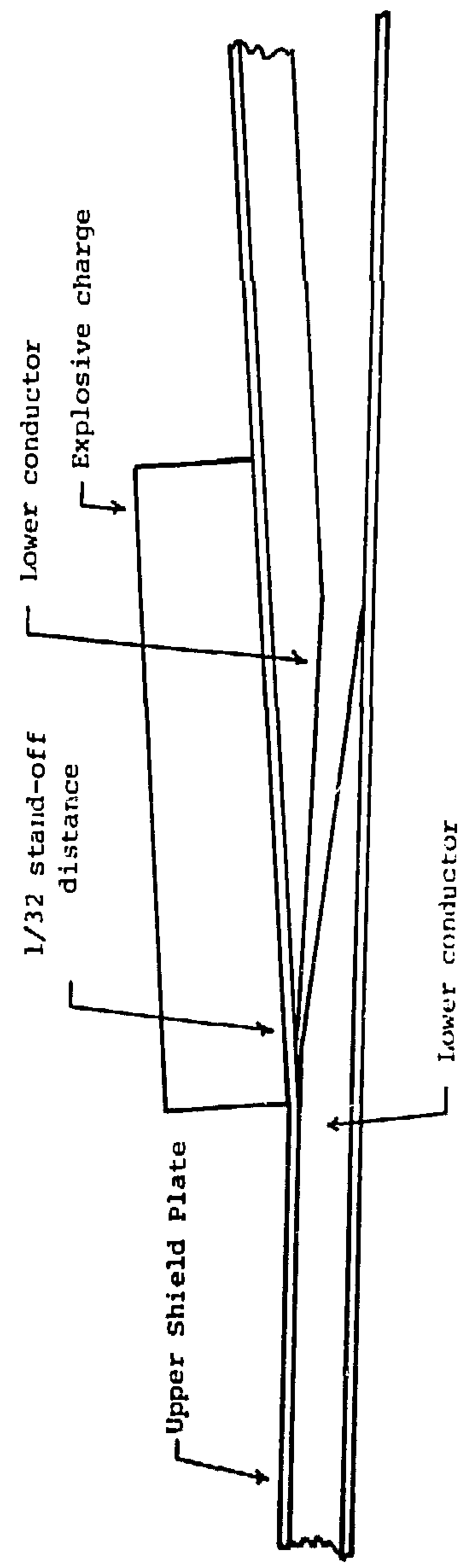


b. 60 Tapered Workpieces.

FIGURE 1. PARTS ARRANGEMENT FOR WELDING



a. 120 Tapered Workpieces.



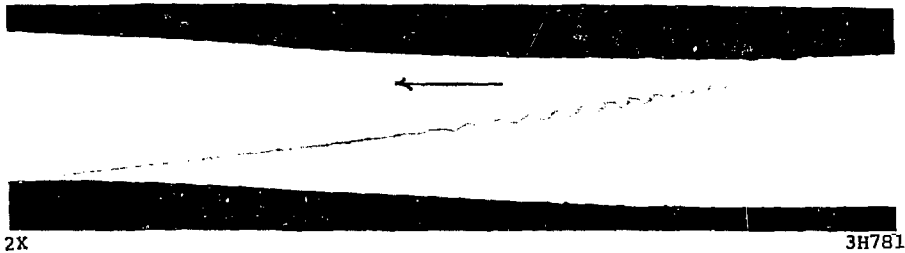
b. 60 Tapered Workpieces.

FIGURE 1. PARTS ARRANGEMENT FOR WELDING

TABLE 1. EXPLOSIVE WELDING OF COPPER BAR STOCK

Specimen Number*	Stand-Off Angle	Loading, g/cm <sup>2</sup>	Remarks
L-1	6	1.65	Welded. Short unbonded area at trailing end of joint.
L-2	6	1.65	Lengthened explosive charge. Welded as above.
L-3	9	1.65	Not welded.
L-4	6	1.98	Welded. Short unbonded area at trailing end of joint.
LL-1	6	1.65	Partially welded. Long (3/4-inch unbonded area at trailing end of joint.
LL-2	6	1.65	Lengthened explosive charge. Partially welded as above.
LL-3	9	1.65	Not welded.

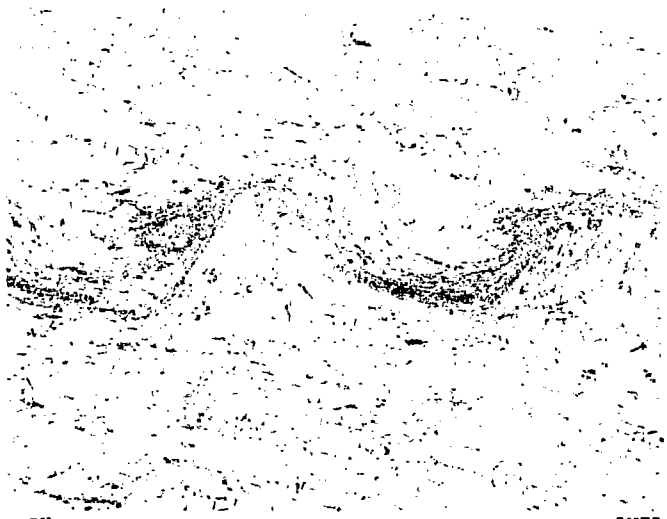
\* Specimens L-1 to L-4 were made with 12° tapered workpieces; LL-1 to LL-3 were made with 6° tapered workpieces.



2X

3H781

a. Complete Joint.



100X

3H778

b. Wave Morphology at Detonation End.

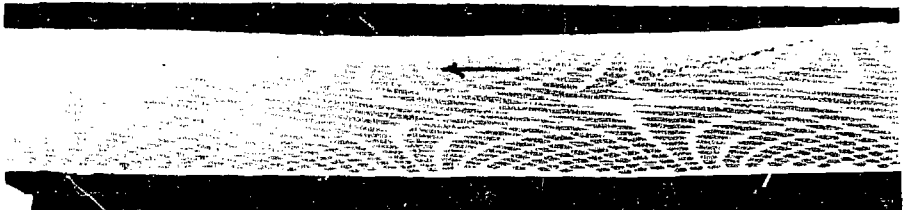
FIGURE 2. EXPLOSIVE WELDS IN COPPER BAR STOCK



5X

a. Specimen LC-1 ( $1.98 \text{ g/cm}^2$ ).

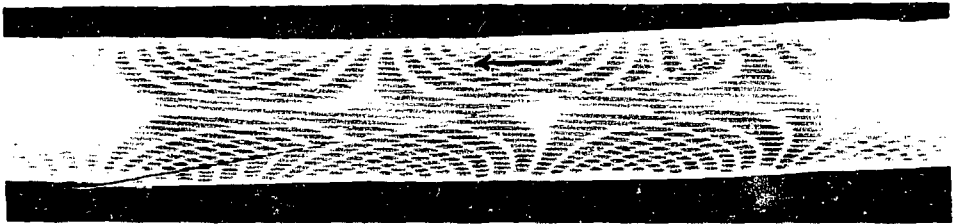
3H827



5X

b. Specimen LC-2 ( $2.31 \text{ g/cm}^2$ )

3H828



5X

c. Specimen LC-3 ( $2.64 \text{ g/cm}^2$ )

3H826

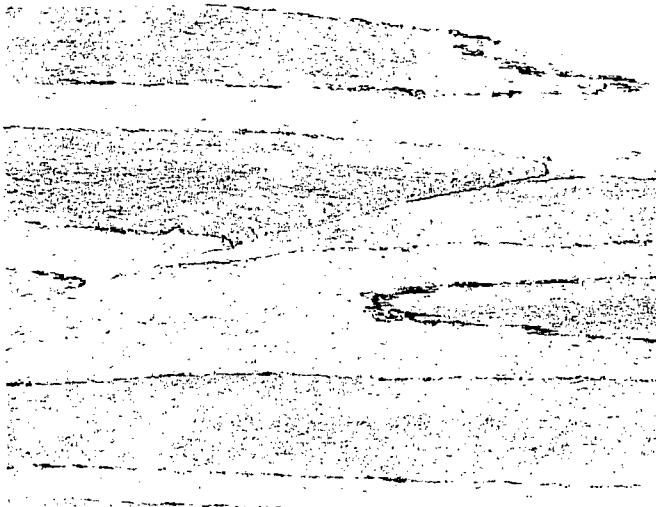
FIGURE 3. EXPLOSIVE WELDS IN CTR SUPERCONDUCTOR WITH DIFFERENT EXPLOSIVE LOADINGS



100X

3H822

- a. Wavy Interface Through Superconductor and Copper Matrix (Specimen LC-3).



100X

3H823

- b. Flat Interface Through Superconductor and Copper Matrix (Specimen LC-2)

FIGURE 4. JOINT MICROSTRUCTURES

Joint Evaluation and Procedure Modification

Several superconductor assemblies were welded in accordance with the procedures used to make Specimen LC-2. Two specimens were tested in tension with the following results:

<u>Specimen Number</u>	<u>Tensile Strength, ksi</u>	<u>% of Base Metal Strength*</u>
1	44.7	51.6
2	46.9	54.2

Upon examination of the fractured surfaces, it was determined that approximately half of the joint area was either (1) unbonded or (2) weakly bonded. In some areas, jetting had occurred during the explosive welding operation but the resulting weld had little strength. The joints were strongest in areas where a wave pattern existed and weakest where it was absent.

While joint strength was important, the probable loss in conductivity through the joint was of even more concern. Thus, efforts were initiated to improve the quality of welding. The following were investigated without noticeable effect on joining:

- (1) Lead tamping on outer surfaces of explosive
- (2) Explosive charge variation.

After considering these alternatives, it was decided that the problem was associated with the basic design of the joint. That is, as welding progressed along the scarf joint, the mass of metal to be accelerated gradually increased and the explosive loading was insufficient to provide the force required for welding.

To overcome this difficulty, an additional explosive charge was positioned on the other side of the joint assembly as shown in Figure 5. Joints welded in this manner had the following properties:

<u>Tensile Strength, ksi</u>	<u>% of Base Metal Strength</u>
68.5	79.1

These specimens exhibited a small area at the ends in which optimum welding did not occur. With further slight adjustments to the welding set-up, we believe that joints equal in strength to that of the base metal can be made.\*\*

\* The base metal strength was determined as 86.6 ksi.

\*\*Preliminary metallographic studies indicate an almost continuous wave pattern is present along the joint interface. Photomicrographs will be sent as an addendum to this report.



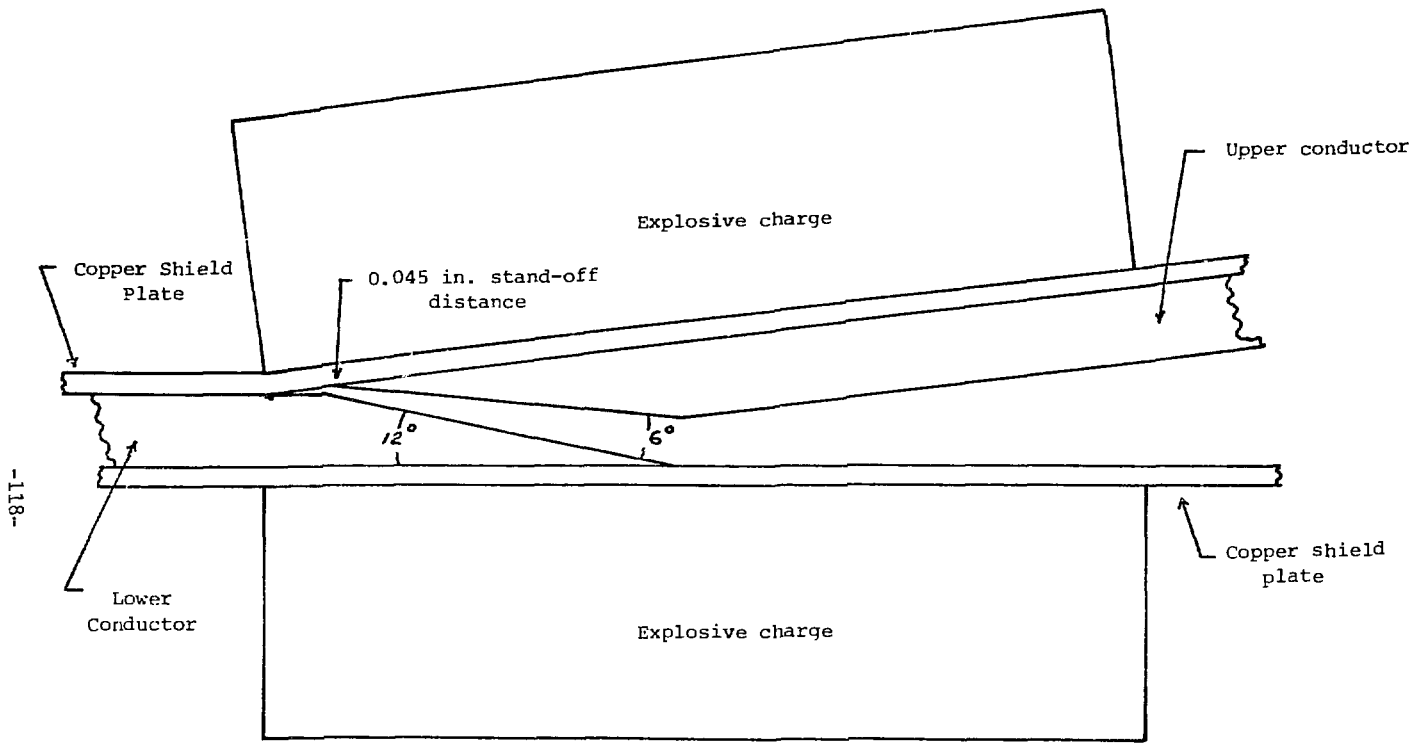


FIGURE 5. MODIFIED SET-UP FOR EXPLOSIVE WELDING OF SUPERCONDUCTOR JOINTS

June 27, 1975

Fabrication of LLL Test Specimens

Four superconductor joints, 18 inches (45.7 cm) long, were fabricated for evaluation by LLL. The component parts of the joint assembly were arranged for welding as shown in Figure 5. The final welding parameters are indicated below.

Explosive loading	:	2.31 g/cm <sup>2</sup> SWP-1
Stand-off distance	:	0.045 inch
Stand-off angle	:	6°

These specimens were examined visually and delivered to LLL.

CONCLUSIONS AND RECOMMENDATIONS

Procedures have been developed to explosively weld joints in CTR superconductor material. Joints with strengths as high as 79 percent of the parent metal strength have been produced in this manner; conductivity losses are expected to be minimal (approximately 10-15 percent or less). While additional experiments were precluded by time limitations, we believe that joints with strength and conductivity essentially equal to those of the base metal can be produced with minor adjustments to the weld parameters.

To develop a process that can be used on-site, the following will be needed.

- (1) Fixturing to assure proper positioning of the component parts of the joint assembly during welding.
- (2) Design, construction, and evaluation of an explosive containment vessel.
- (3) Refinement of welding parameters.
- (4) Detailed welding procedures.

We will be pleased to work with you in this further development. If you have any questions, please contact me.

Very truly yours,



H. E. Pattee  
Fabrication and Quality  
Assurance Section

HEP/db

cc: Mr. Donald Cornish, LLL