

# Lawrence Berkeley National Laboratory

## Recent Work

**Title**

NIOBIUM-TIN SUPERCONDUCTING WIRE BY THE INFILTRATION PROCESS

**Permalink**

<https://escholarship.org/uc/item/5126w8xr>

**Author**

Heraachalam, K.

**Publication Date**

1975-11-01

NIOBIUM-TIN SUPERCONDUCTING WIRE BY THE  
INFILTRATION PROCESS

K. Hemachalam and M. R. Pickus

November 1975

Prepared for the U. S. Energy Research and  
Development Administration under Contract W-7405-ENG-48

**For Reference**

Not to be taken from this room



LBL-4532  
c. |

## **DISCLAIMER**

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

-1-

## NIOBIUM-TIN SUPERCONDUCTING WIRE

## BY THE INFILTRATION PROCESS

K. Hemachalam and M. R. Pickus

Materials and Molecular Research Division  
Lawrence Berkeley Laboratory  
University of California  
Berkeley, CA 94720

## ABSTRACT

Superconducting wires containing filaments of the A-15 compound,  $\text{Nb}_3\text{Sn}$ , were made by powder metallurgy techniques. This article describes the various approaches employed for achieving a desired level of porosity in rods of sintered niobium that were later infiltrated with liquid tin. The influence of various mechanical deformation processes on filament morphology is reported. The critical current density of finished wires was measured under pulsed fields. The results indicate a pronounced  $I_c$  dependence on the type of deformation process used. Encouragingly high values of current-carrying capacity were observed.

## INTRODUCTION

Technologically important A-15 superconducting materials have been produced by several methods in various geometries. The latest trend in fabricating multifilamentary superconductors seems to be focused on the bronze technique. Although this technique has been proved successful for two compounds,  $\text{Nb}_3\text{Sn}$ <sup>1-3</sup> and  $\text{V}_3\text{Ga}$ ,<sup>4-6</sup> it involves a tedious repetitive bundling procedure and a long heat treatment. A simple casting method developed by Tsuei<sup>7-9</sup> yields wires with relatively poor current-carrying capacity especially at high magnetic fields. The development of a new approach which overcomes these shortcomings is described in this paper. Based on powder metallurgy techniques involving infiltration, it is applicable to superconducting compounds such as  $\text{Nb}_3\text{Sn}$ ,  $\text{V}_3\text{Ga}$  and  $\text{Nb}_3\text{Al}$ . A requirement is that the second component (Sn, Ga or Al) should wet the high melting component at a temperature below that at which the A-15 compound forms by a diffusion reaction. The infiltration process was first employed to fabricate a superconducting tape characterized by an array of  $\text{Nb}_3\text{Sn}$  filaments in a ductile niobium matrix.<sup>10</sup> The superconducting properties of the tape were reported recently.<sup>11</sup> Modifications have now been made to adapt the main features of the tape-process for making the superconductor in the form of wire. A wire provides several advantages: the filament morphology is superior from the standpoint of the adiabatic stability criterion,<sup>12</sup> and the wire can be used as the basic element of a multicore conductor or a multistrand cable for handling large currents. Figure 1 shows schematically the various steps involved in the present wire-process.

## PROCESS DEVELOPMENT

Three methods were investigated to produce porous niobium rods. In the first, -325+400 mesh niobium powder of 99.92% purity was packed in a niobium tube. The powder was pressureless sintered for 10 minutes at  $2220 \pm 20^\circ \text{C}$  in a vacuum of the order of  $10^{-4}$  mm Hg. Fig. 2(a) shows a typical cross section of the sintered rod after tin impregnation. The tin infiltration was normally performed by a 30 sec. immersion of the sintered rod in a  $700^\circ \text{C}$  tin bath. The level of porosity and the pore-size (Fig. 2(a)) were so large that the tin did not form fine filaments when the compact was reduced to wire. In addition, as a result of shrinkage, huge gaps were formed locally near the Nb tube walls. These difficulties were circumvented by a two-stage sintering of finer powder. First, a -400 mesh Nb powder was pressureless sintered in a mullite tube at  $1600\text{--}1650^\circ \text{C}$  for 5-10 minutes. The mullite tube was removed to obtain a self-supporting rod which was then resintered at  $2220 \pm 20^\circ \text{C}$  for 10 minutes. This procedure yielded an acceptable porosity, ~20%, as indicated in Fig. 2(b).

A similar pore structure was achieved more conveniently in a different manner. Niobium powder (-250+400 mesh) was isostatically compacted at  $207 \text{ N/mm}^2$  (30,000psi) into 3mm diameter rods. These rods were then sintered and infiltrated with tin. The cross section in Fig. 2(c) illustrates a typical pore structure obtained by this approach. Due to the simplicity of the method and the ease of controlling of the porosity, isostatic compaction was selected to produce wires discussed later in this report.

After tinning, the sintered rods were clad with an annealed niobium tube. Because niobium is susceptible to galling, the assemblies were re-clad in tubing of another metal (copper or monel) having a much lower galling tendency when wire drawing was to be used as a deformation mode. The intermediate sheath of niobium served as a diffusion barrier preventing any reaction between the outer clad and the infiltrated core during the final heat treatment. The assemblies were reduced to wire by one or a combination of deformation processes: swaging, form rolling and wire drawing. The diffusion heat treatment was carried out at  $960 \pm 10^\circ\text{C}$  for 5 minutes on samples that were encapsulated in argon filled quartz tubes.

## RESULTS AND DISCUSSION

A metallographic examination of wire sections indicated that swaging produced a characteristic structure, Fig. 3(a), related to the severe twisting action of the rotating hammers. As can be seen from Fig. 3(b) and 3(c), form rolling followed by wire drawing, and wire drawing alone, yielded wires with much more isotropic cross section with respect to filament morphology. Figure 4 shows a longitudinal section of a form-rolled and wire-drawn sample after the formation of  $\text{Nb}_3\text{Sn}$ . The inductively measured superconducting-to-normal transition (mid-point) temperature of the samples was  $18.0 \pm 0.1^\circ \text{K}$ .

A pulsed field technique described in Reference 11 was used to determine the critical current vs magnetic field relationship of the wires at  $4.2^\circ \text{K}$ . The current density values shown in Fig. 5 were computed on the basis of the core area consisting of  $\text{Nb}_3\text{Sn}$  phase and niobium matrix. Good agreement had been previously observed between the pulsed field and steady field data when the current densities in the latter were calculated at a resistivity of  $10^{-12}$  ohm-cm. Some typical results are shown in Fig. 5 for the current carrying capacity of wires with 0.45mm dia. cores as a function of transverse magnetic field. The three curves were obtained from wires whose cross sections are shown in Figs. 3(a), 3(b), and 3(c). The wires that had been reduced by wire drawing, and by form rolling followed by wire drawing carried more than twice the amount of current carried by the fully swaged one. Partial form rolling prior to swaging improved the  $I_c$  vs  $H$  characteristics of the wire only slightly (not plotted in Fig. 5) over the fully swaged wire. Since the volume fraction of  $\text{Nb}_3\text{Sn}$  seems to be the same for all the wires, the marked



difference in their current densities must be related to the nature of the filament disposition in the conductor itself.

In these wires the reduction ratio (cross sectional area of sintered rod  $\div$  core area of wire) was only about 45. The final Nb<sub>3</sub>Sn filaments were about 4-7  $\mu$ m thick. Owing to the limited ductility of the sintered compacts, further reductions were not possible. However, recent attempts to improve the ductility by an increased sintering temperature and a better vacuum have resulted in reduction ratios higher than 200. With the filament size reduced to the order of 1-2  $\mu$ m, considerably larger currents have been observed in preliminary tests. Further work is planned to study the influence of filament thickness on  $I_c$ , and the mechanical properties of the wires. The results will be presented in a future communication.

This research was supported by the U.S. Energy and Development Administration through the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory. The authors wish to thank Mr. John Holthuis and Mr. John Jacobsen of this Laboratory for their valuable technical assistance.

## REFERENCES

1. A. R. Kaufmann and J. J. Pickett, J. Appl. Phys. 42 (1971) 58.
2. M. Suenaga and W. B. Sampson, Appl. Phys. Lett. 18 (1971) 584.
3. M. Suenaga, W. B. Sampson, and C. J. Klamut, Applied Superconductivity Conf., Argonne (1974); IEEE Transactions on Magnetics, MAG-11 (1975) 231.
4. K. Tachikawa, Proceedings of the Third International Cryogenic Engineering Conference (Transcripta Service, Illife Science and Technical Publication, Surrey, 1970) 339.
5. K. Tachikawa, Y. Yoshida and L. Rinderer, J. Mat. Sci. 7 (1972) 1154.
6. K. Tachikawa, K. Itoh and Y. Tanaka, Applied Superconductivity Conf., Argonne (1974); IEEE Transactions on Magnetics MAG-11 (1975) 240.
7. C. C. Tsuei, Science 180 (1973) 57.
8. C. C. Tsuei, M. Suenaga and W. B. Sampson, Appl. Phys. Lett. 25 (1974) 318.
9. C. C. Tsuei, Applied Superconductivity Conf., Argonne (1974); IEEE Transactions on Magnetics MAG-11(1975) 272.
10. M. R. Pickus, V. F. Zackay, E. R. Parker and J. T. Holthuis, Int. J Powder Met. 9 (1973) 3.
11. M. R. Pickus, K. Hemachalam and B. N. P. Babu, Mat. Sci and Eng. 14 (1974) 265.
12. M. N. Wilson, C. R. Walters, J. D. Lewin and P. F. Smith, J. Phys. D Appl. Phys. 3 (1970) 1517.

FIGURE CAPTIONS

1. Schematic diagram of filamentary  $\text{Nb}_3\text{Sn}$  superconductor fabrication process.
2. Cross sections of sintered niobium rod infiltrated with tin; a) pressureless sintering of -325 + 400 mesh Nb; b) two-stage sintering of -400 mesh Nb; c) isostatic compacting and sintering of -250 + 400 mesh Nb. (White areas are tin.)
3. Cross sections of heat treated wires produced by a) swaging only; b) form rolling followed by wire drawing; c) wire drawing only.
4. Longitudinal section of a form-rolled and wire-drawn specimen after heat treatment. (Dark filaments are  $\text{Nb}_3\text{Sn}$ .)
5. Pulsed field dependence of current-carrying capacity. The three curves correspond to the photomicrographs (a), (b) and (c) of Fig. 3.

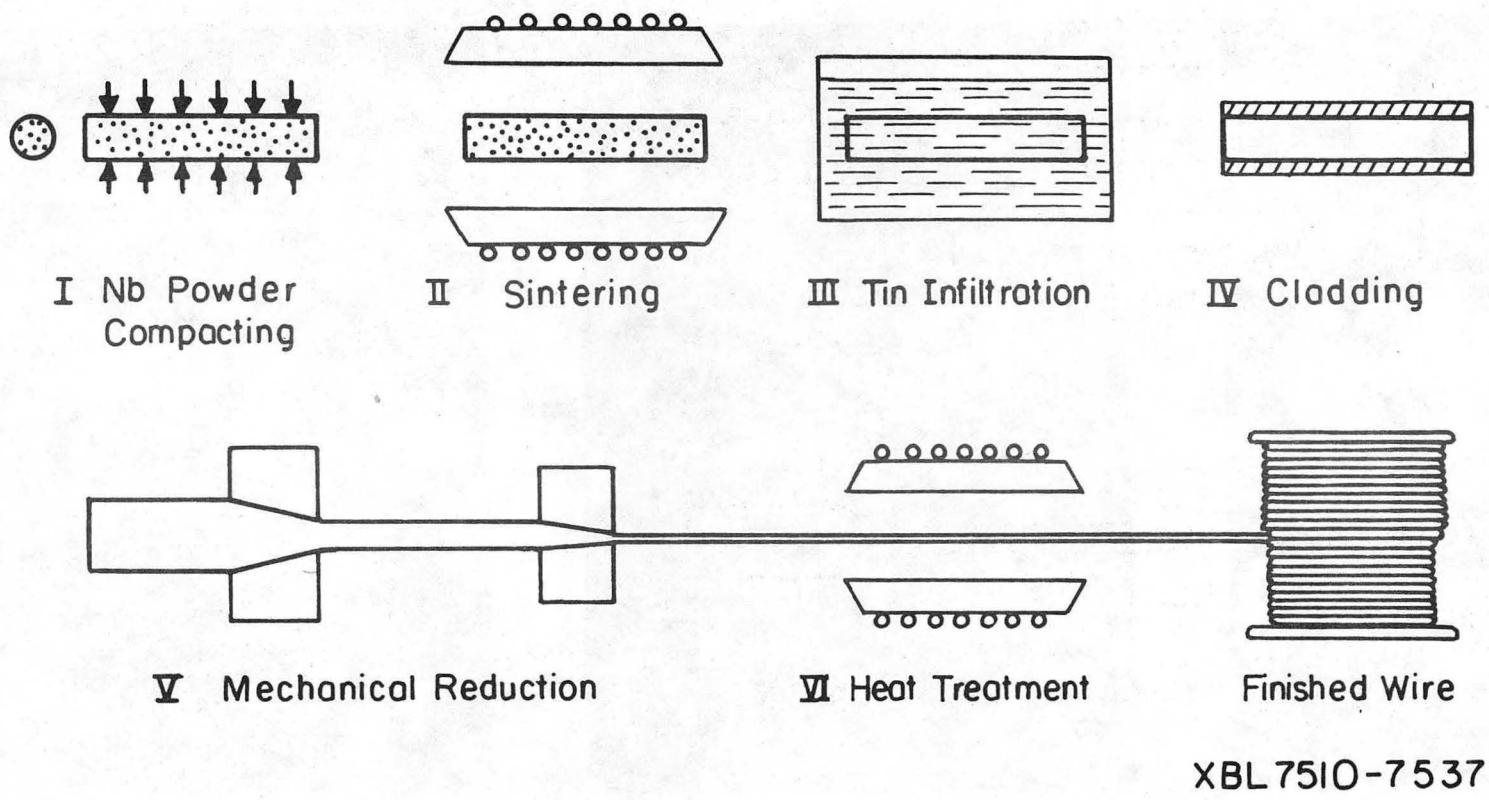
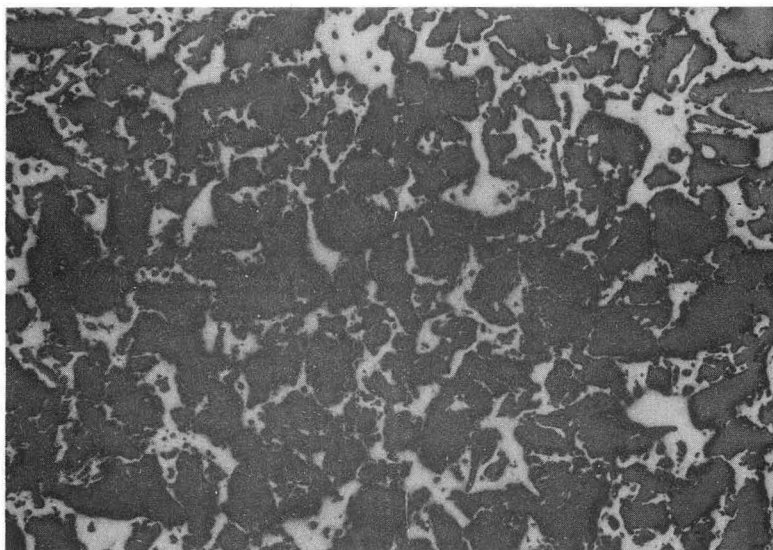
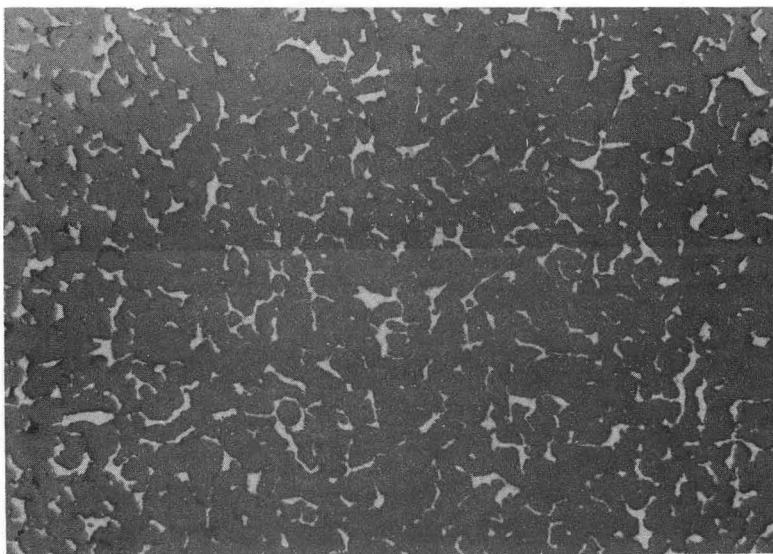


Fig. 1

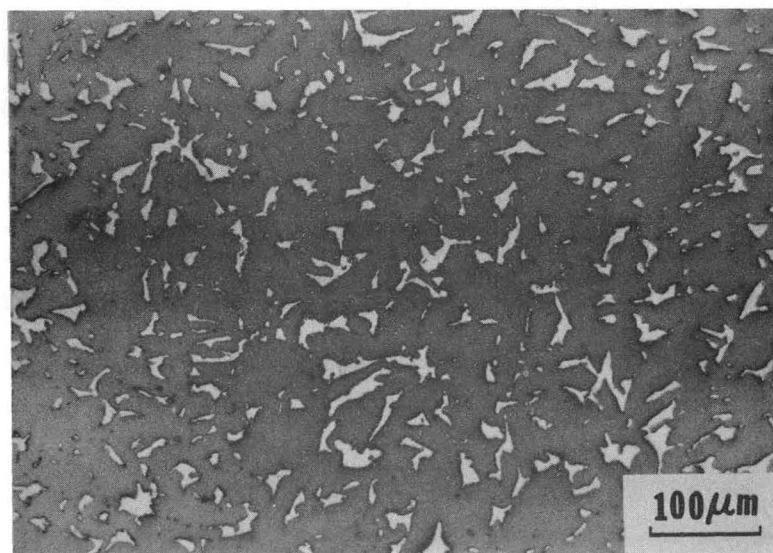
00004407183



**a**



**b**

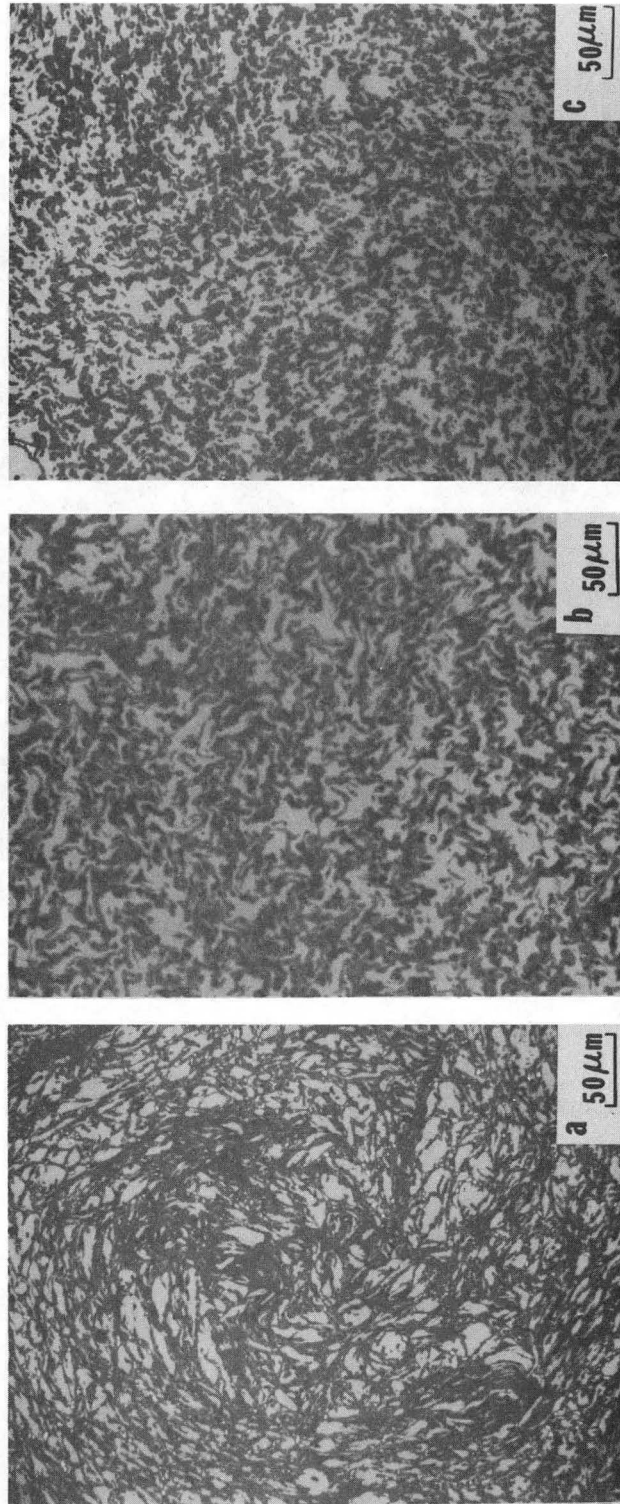


**c**

XBB 7511-8190

Fig. 2

-11-



XBB 7511-8189

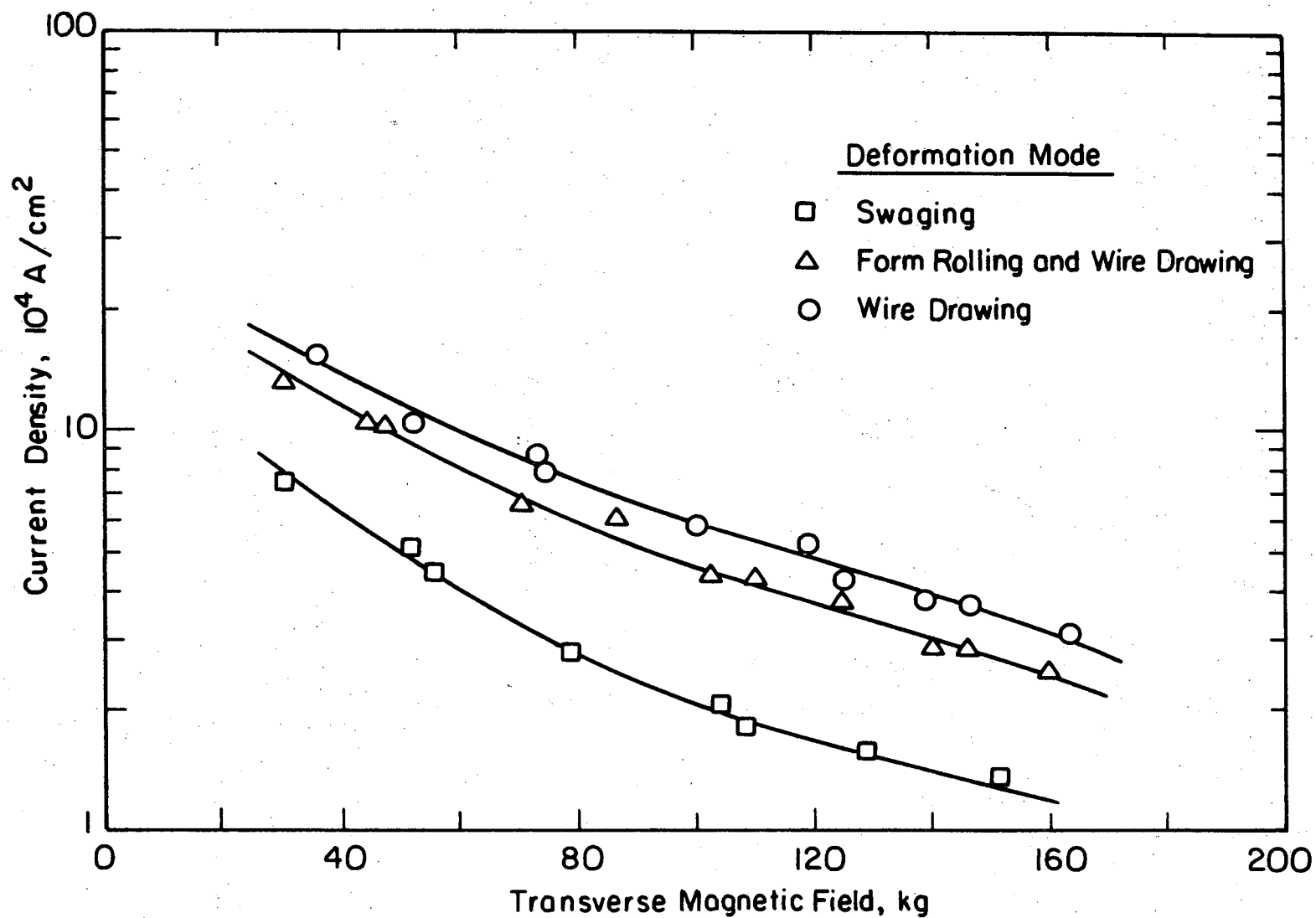
Fig. 3

-12-



XBB 7511-8188

Fig. 4



XBL 7511-7570

Fig. 5



0 0 0 0 4 4 0 7 0 0

**LEGAL NOTICE**

*This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.*

TECHNICAL INFORMATION DIVISION  
LAWRENCE BERKELEY LABORATORY  
UNIVERSITY OF CALIFORNIA  
BERKELEY, CALIFORNIA 94720