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REPORT NO. TDR-169(3250-13)TN-1"

### **Electronics Research Program**

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Low-Temperature Electronics

#### SEMIANNUAL TECHNICAL NOTE

(1 JULY - 31 DECEMBER 1962)

#### 29 APRIL 1963

Prepared by F. L. VERNON, JR. Electronics Research Laboratory

Prepared for COMMANDER SPACE SYSTEMS DIVISION

UNITED STATES AIR FORCE

Inglewood, California

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#### ABSTRACT

The initial experimental work on the high-field superconducting characteristics has started with an investigation of the effect of grain size on the critical field  $(H_c)$ -critical current  $(I_c)$  relationship. Measurements have been made which show an anomalous behavior of  $H_c$  versus  $I_c$  for a certain range of grain sizes. Future work will be directed toward obtaining a complete experimental and theoretical description of the relationship between the structural variations and the electrical characteristics.

Initial experiments on the properties of superconducting thin filaments have been performed on samples consisting of lead imbedded in a copper matrix. The results have indicated that size-dependent behavior occurs before it would be expected on a theoretical basis. It is possible that the normal copper electrons are severely affecting the superconducting properties of the lead. Additional experiments are being planned to define the phenomenon more completely.

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#### I. INTRODUCTION

The application of unique properties of materials at liquid helium temperatures in performing useful electronic functions has received considerable notice during the past few years, because of the development of workable ways of obtaining liquid helium environments. Furthermore, several outstanding scientific breakthroughs have enhanced understanding of low-temperature phenomena, particularly superconductivity, and have focused attention on previously unsuspected unique properties. Practical devices are being produced in such areas as computers (cryotron), infrared detectors (superconducting bolometers), and high-intensity magnets (high-field superconductors). The objectives of the present program are to study the properties of materials at low temperatures and to determine their potential usefulness in performing electronic functions.<sup>1</sup>

Research in low-temperature electronics during this reporting period has consisted of, first, design and assembly of specialized laboratory apparatus for carrying out experiments and, second, performance of experiments on high-field superconducting alloys. Two principal areas of study have been pursued. One of these is a cooperative project with the Physics Department of the Materials Sciences Laboratory. As this project progresses, various high-field superconductors will be prepared in a controlled manner to determine the effects of different types of defects, dislocations, and strains on superconducting characteristics. The first superconducting samples fabricated and tested were made from niobium.

The other area is the study of the interaction of thin filamentary superconductors imbedded in a metallic matrix of a normal metal. If the superconducting samples are small compared with the penetration depth, coherence effects • between electrons of the normal metal and those of the superconducting metal can be studied, and information about high-field superconductors will be gained. These two projects will be discussed in subsequent paragraphs.

#### II. DISCUSSION

#### A. SUPERCONDUCTING PROPERTIES OF NIOBIUM WIRES

For many years, certain modifying effects of strains and plastic deformations on the properties of superconductors have been known, but not understood. These effects include a change of critical temperature, critical field, or critical current. Early research in this field was usually confined to polycrystalline samples of soft superconductors, and the induced strains and plastic deformations normally were accomplished at low temperatures.  $^{2-4}$ The most outstanding effects in obtaining large critical field have occurred with alloys such as Nb<sub>3</sub>Sn and Nb<sub>3</sub>Zn formed with hard superconductors. However, the alloyed systems are very complicated metallurgically, and the mechanisms which cause these alloys to be superconducting are not understood.

A study of Nb<sub>3</sub>Sn and other alloys is planned after preliminary studies have been made of simpler systems. Pure niobium has been chosen for initial studies because it has some of the properties of high-field superconductors. Its critical field  $H_c$  can be made higher than its bulk critical field  $H_{cb}$  if dislocations and defects are introduced to the metal. In addition, a considerable amount is known about the metallurgy of niobium. Therefore, through the use of controlled defect structures, it will be possible to correlate the effects with the resulting superconducting characteristics.

There are two types of theories, not entirely independent, to explain highfield superconductivity. Both types use the fact that if a superconducting specimen is thin enough to allow field penetration through it, then the critical field  $H_c$  for the sample will be greater than the bulk critical field  $H_{cb}$ .<sup>5</sup> A brief description of the two theories is as follows.<sup>6</sup>

In the first type, inhomogeneities are assumed to exist in filaments that are thin enough to have an enhanced critical field. These filaments, in fact, may be dislocations. As the applied field is raised, the filament sizes shrink, thus increasing their critical fields even further.<sup>7</sup> This has the effect of spreading out the magnetic transition so that very large fields are required to quench superconductivity entirely.

In the second type, it is postulated that in the presence of a magnetic field a superconducting sample can lower its free energy by breaking up into normal and superconducting regions that are thin enough to allow flux penetration. The normal-superconducting interface area is effectively increased by this means, and, thus, the high-field properties can be thought of as resulting from a negative surface energy. <sup>8, 9</sup>

It is from these two general points of view that the experimental results will be interpreted when a sufficient amount of data has been taken to show a complete picture. The experimental procedure and the initial results will be described in the following paragraphs.

#### 1. Experimental Procedure

The niobium samples under test were processed from commercially obtained 0.050 in. diameter wire. Specimens were annealed at temperatures between 1100° C and 1400° C for varying lengths of time so that a variety of grain sizes would result. The maximum grain size of 7.45  $\times$  10<sup>-3</sup> cm<sup>2</sup> was obtained by a four-hour, 1400° C anneal. The smallest grain size obtained was 0.95  $\times$  10<sup>-3</sup> cm<sup>2</sup>, resulting from a one-hour, 1100° C anneal.

The magnet and dewar setup has been shown in a previous report.<sup>1</sup> Figure 1 shows the top of the sample holder with the flange that attaches to the dewar, and the twisted voltage and current leads which are attached to the samples under test. Figure 2 shows the bottom of two sample holders, one to be used in the future for flat samples and the other presently in use with the niobium wires (approximately one inch long) intact. The current leads are soldered to the ends of the niobium, and voltage leads are spot-welded about one-quarter inch from each end.



Fig. 1. Top section of sample holder.



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#### 2. Experimental Results

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The measurements consisted of determining I<sub>c</sub> versus H<sub>c</sub> at T =  $4.2^{\circ}$  K and for the field parallel and transverse to the axis of the wire. The results of these measurements are given in Figs. 3, 4, 5, and 6. A fairly consistent trend is that I<sub>c</sub> versus H<sub>c</sub> (||) (H field parallel) curve is above the I<sub>c</sub>-H<sub>c</sub> ( $\perp$ ) (H field perpendicular) curve for the higher currents. For the lower currents, the critical fields are more nearly the same. This is consistent with the picture that a transverse field opposes and adds to the field created by the current. At high values of current, the magnitude of its field is great enough to increase the field at the surface of the superconductor substantially and, thus, lower the critical field. Conversely, when small currents are being applied to the sample, the effective field at the surface is the applied field. When the field is parallel to the sample axis, it is normal to the circumferential field of the current. Thus, the resultant field is a vector addition and is not as large as a direct addition. In this case, higher applied fields are necessary to reach the critical value than in the H  $(\perp)$  case. Again, at low current values, the effect of the current generated field is negligible.

An outstanding anomaly is observed with the magnetic field normal to the axis for two separate samples in Figs. 5 and 6. The effect is quite repeatable and, as can be seen, is characterized by a sharp decrease of  $I_c$  followed by a rather slow increase of  $I_c$  as  $H_c$  is decreased. The wires in which this occurred were annealed for the longest times and resulted in the largest crystal sizes of 7.45  $\times$  10<sup>-2</sup> cm<sup>3</sup>. For the other two cases, involving small grain sizes, the effect was absent. The phenomenon just described has been noticed before.<sup>6</sup> The agreement between our results and those presented in Fig. 6 of Ref. 6 is striking.

A thorough theoretical description of the situation has not been advanced, and a discussion of the physical explanation of our results will be postponed until a better experimental picture can be developed and an applicable theory can be found. During the next report period, experimental work will proceed

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Fig. 3. Critical field-critical current curves for Niobium Sample 22.



Fig. 4. Critical field-critical current curves for Niobium Sample 17.

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Fig. 5. Critical field-critical current curves for Niobium Sample 15-1.



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Fig. 6. Critical field-critical current curves for Niobium Sample 15-2.

along the lines of obtaining a more precise correlation of physical structure with superconducting properties as a logical continuation of the abovedescribed work.

#### B. THIN FILAMENTARY SUPERCONDUCTORS

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An investigation of an unusual class of superconducting materials is in progress. These materials are fabricated by a process which results in the inclusion of a large number of continuous, parallel filaments of a superconducting metal in a wire of some other metal. Prompting this investigation is the fact that when one or more dimensions of a superconductor are reduced sufficiently, the superconducting properties are known to change (see, for example, Ref. 10). Recently, Bean and co-workers <sup>11</sup> synthesized a material by forcing soft superconducting metals into porous Vycor (glass). The resulting material has the properties of a hard superconductor.

Because of the technological and basic importance of hard superconductors, it appeared desirable to investigate the properties of a material in which the filament diameters and densities could be varied in a controlled way. To that end, a method of fabrication suggested by J. W. Henderson of this laboratory was successfully used to prepare samples. (It was learned subsequently that a nearly identical method of fabricating filamentary superconducting material was being used by Seraphim and co-workers.<sup>12</sup>) The samples are formed by a wire-drawing process: A wire of superconducting metal is encased in a sheath or tube of some other metal. This combination is drawn down to a reduced diameter. Pieces of this material are resheathed and then redrawn. This process, which reduces the diameter of the superconducting filaments by an order of magnitude, is repeated until the required diameter is reached. Suitable pairs of materials for this process should have similar rates of work hardening, low solubilities in one another, and similar annealing temperatures.

Initial work has been confined to samples of lead filaments in a copper host. The copper can be softened sufficiently at annealing temperatures just below the melting point of lead to permit the repeated drawings required by the

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fabrication process. In addition, these materials are not mutually soluble. The resulting specimens have been sectioned, polished, and microscopically inspected. The lead filaments are nearly circular in cross section, and their geometry after drawing corresponds to the geometry before, as expected. The final specimen diameters are about 0.5 mm, and specimens having 3750 lead filaments with diameters of 1300 Å have been fabricated and investigated.

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Preliminary investigation of the samples has been restricted to measurements of the electrical conductivity. The measurements are made at dc using a conventional four-terminal network. Precautions minimizing stray thermal emf permit the observation of potentials as small as 0.1  $\mu$ V. The sample holder can be rotated to orient the magnetic field with respect to the sample.

Samples having filament diameters of  $4\mu$  were observed to behave like bulk lead. The resistivity of these samples goes to zero at 4.2°K in fields less than about 500 Oe, as expected. However, samples having filaments of 1300 Å diameter no longer behave like bulk lead. Some preliminary results of our investigation are shown in Figs. 7 and 8. Note that at any temperature shown, the sample has superconducting properties in fields larger than the critical field for bulk lead at that temperature. The resistance does not disappear at low fields but decreases to some finite value as the field is reduced to zero. Figure 8 shows that the resistance at 4.2°K in zero field is current dependent, and a reasonable extrapolation indicates a zero resistance at zero measuring current. If the current is assumed to be distributed equally among the various lead filaments, the magnetic field due to these currents is very much too small to account for the finite resistance observed. It appears likely, therefore, that the current is not uniformly distributed among the filaments, a possibility which is being explored.



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#### III. SUMMARY

The initial experimental work on the high-field superconducting characteristics of niobium has started with an investigation of the effect of grain size on the critical field-critical current relationship. Measurements have been made which show an unusual variation of superconducting properties with grain size. Future work will be directed toward obtaining a better experimental picture of the relationship between the structural variations and the electrical characteristics.

Initial experiments on the properties of superconducting thin filaments have been performed on samples consisting of lead imbedded in a copper matrix. The results have indicated that size-dependent behavior occurs before it would be expected on a theoretical basis. It is possible that the normal copper electrons are severely affecting the superconducting properties of the lead. Additional experiments are being planned to define the phenomenon more completely.

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