YEAREND REPORT ON PROJECT

Fabrication of Superconducting
Wire Composites
by
Directional Solidification

by

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Project Sponsored by Magnetic Fusion
Energy Division, DOE

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Introduction

This research project was officially begun in Ames when we received notification of its funding, Jan. 17, 1977. Because of prior commitments and the need to hire a technician experimental work was not begun until early February. Therefore, this report covers work done over a 8.5 month period from February 1 to October 15, 1977. The work done in the first two weeks of October has been included because it has tied together some loose ends on the important question of the nature of the Nb-Cu phase relationships.

The purpose of this research project is to develop a new process for producing composite $\text{Nb}_3\text{Sn}/\text{Cu}$ superconducting wires, and to evaluate their critical current densities and the stress dependency of the critical currents. It is hoped that one or both of these properties will be improved by the new process over values obtained on wires produced by the tape or bronze processes.

The new process involves directional solidification of Cu-Nb or Cu-Nb-Sn alloys. The key idea is to produce continuous filaments of Nb along the length of wire by maintaining aligned growth of an array of niobium dendrites at the growth front. This will produce dendrite filaments continuous along the length of the solidified ingot as shown in fig. 1. The ingot will then be drawn into wire form and the Nb_3Sn produced by a diffusion process. Initial experiments will be done on Cu-Nb alloys and in this case the Sn will be supplied from a surface coating. Subsequent experiments will use Cu-Nb-Sn alloys and in this case the Sn will be supplied from the bronze matrix which will form upon solidification in the regions in-between the Nb dendrites.

Fig. 1. Alloy of Cu-Nb solidifying by growth of an aligned array of Nb dendrites into the liquid

With this new process it should be possible to produce continuous filaments in the size range of 200-1000⁰Å and it is hoped that these small sizes will produce J_c values as good or better than bronze processed material because of flux pinning at the Nb₃Sn/Cu interfaces. The small size should also produce improved resistance to degradation of J_c with mechanical deformation because of the smaller strains inherent to smaller filament sizes.

Background

There have been two studies^{1,2} of the binary Cu-Nb phase diagram reported in the literature. The phase diagram of Popov and Shiryaeva¹ is shown in fig. 2 and it is seen that they find a monotectic reaction. The later work of Allibert et al.² does not show the monotectic but only a nearly horizontal liquidus as seen in fig. 3. It is generally concluded^{3,4} that the diagram of Allibert et al.² is correct because of insufficient evidence by Popov and Shiryaeva to support a monotectic reaction. An unpublished thesis by Schelle⁵ also supports this conclusion.

Figure 4 presents the phase diagram of the Ag-Pb system and one sees that it is quite similar to the Cu-Nb phase diagram of fig. 3. As discussed in the proposal for this research the Ag-Pb system was used as a model system for the Cu-Nb system and directional solidification experiments were carried out. It was demonstrated that aligned dendrites of Ag could be produced in the 1 μ m range if sufficiently high temperature gradients and rates were employed. Therefore, the first critical questions to be answered in the present study were:

Fig. 2. The Cu-Nb phase diagram proposed by Popov and Shiryaeva¹

Fig. 3. The Cu-Nb phase diagram proposed by Allibert et al.²

Fig. 4. The Pb-Ag phase diagram

1) Can aligned growth of Nb dendrites be achieved in the Cu-Nb system as in the model system?

2) Can the dendrites be made as small as $1\text{ }\mu\text{m}$ as in the model system?

Once these questions have been answered affirmatively, developmental work will proceed to investigate the influence of composition, temperature gradient and growth rate upon dendrite morphology and consequent J_c values. Also, developmental work is needed to optimize the Sn plating and diffusion processes.

Experimental Work

In the experimental work on the Ag-Pb model system the alloys were contained in small diameter tubes. A six inch length of alloy was held in the molten state at a temperature of around 100°C above the liquidus and then directionally solidified by lowering through an o-ring seal into a rapidly flowing water bath. With Nb-Cu alloys such a scheme would require holding the molten alloy at $1800\text{-}1900^\circ\text{C}$ and at these high temperatures one anticipates problems of corrosive attack by the molten alloy on the container tube. To alleviate such problems a design was developed in which only a small zone is rapidly heated to $1800\text{-}1900^\circ\text{C}$ and quickly solidified directionally. With this approach the alloy is at temperature for only a matter of fractions of a minute.

a. Directional Solidification

The experimental design developed for directional solidification is shown in fig. 5. The heart of the apparatus is the concentrator which acts as a secondary to a coil energized by a 25 KW, 450 kc high

Fig. 5. Directional solidification apparatus

frequency power converter. The concentrator acts to concentrate the magnetic field into a short length along the tube containing the alloy. This tube is about 2 mm inside diameter and the Cu-Nb alloy is placed inside the tube as a wire having a diameter slightly less than 2 mm. During solidification the concentrator is driven upwards causing a small molten zone to pass up the tube. A very steep temperature gradient is produced at the solidification front by the water cooling finger which extends up below the concentrator. As the Cu-Nb wire melts it fills the entire diameter of the container tube and it is necessary to feed the wire downward through the top of the apparatus to avoid void formation in the solidified rod.

Initial experiments were carried out in thin walled Al_2O_3 tubes. These did not produce dendrite alignment and it was thought that the loss of alignment was due to severe convective mixing produced by the hf currents induced directly in the alloy to produce the heating. Therefore it was decided to use a tube which was an electrical conductor. Tantalum tubes were selected because of their availability, high melting temperature, and the fact that they could be coated on the inside with a thin layer of TaC, which is relatively inert and has the highest melting temperature of all known materials.

Two techniques were developed to carburize the Ta tubes. In the first technique the Ta tube was heated inside a platinum wound furnace as shown in fig. 6. Carburization was produced by passing methane gas through the inside of the tube. This method is limited by the melting temperature of the furnace windings to carburization temperatures of around 1600°C . The second method is carried out by

Fig. 6. Platinum wound resistance furnace scheme for carburizing the inside surface of Ta tubes

a small modification of the apparatus of fig. 5. A stainless steel tube is fastened to the top of the Ta container tube. The stainless tube extends out of the top of the apparatus. The methane gas is now passed up the Ta tube and out the stainless steel extension tube. The Ta tube is heated by passing a hot zone upward at a controlled rate. In this technique carburization may be carried out at temperatures up to the melting point of Ta, 3000°C.

b. Alloy Preparation

The directional solidification technique described above requires the preparation of Nb-Cu alloys in rod form. The rods must have a uniform composition along their lengths and, in addition, the Nb particle size within the rods must be quite small to insure that complete homogenization occurs within the molten zone during the relatively short time the zone remains in the molten state. A standard technique used at our laboratory and elsewhere to prepare alloys having high melting temperatures is nonconsumable arc melting with a tungsten electrode upon a water cooled copper hearth. However, examination of fig. 3 reveals that for an alloy of 20% Nb there exists a 600°C temperature gap between the temperature where solidification starts and finishes. When this is coupled with the fact that Nb melts at 2468°C compared to 1083°C for Cu it becomes clear that it might be difficult to prepare homogeneous alloys by nonconsumable arc melting; this difficulty was found to be the case in the work of Schelle.⁵ For this reason our initial experiments employed a consumable arc melting technique. In this technique the Cu-Nb forms one of the electrodes for the arc. This electrode,

made from rods of pure Cu and pure Nb in the desired weight ratio, was arc melted into a graphite crucible. However, it was found that homogeneous alloys could not be produced unless the graphite crucible was allowed to become sufficiently hot to maintain a molten layer across the crucible. Because of the danger of carbon contamination and the relatively large Nb dendrite size produced by this technique it was abandoned in favor of the chill casting technique described below.

The chill casting unit designed for this project is shown in fig. 7. A small hole is drilled through the center of the bottom of a crucible and a tube of the same material as the crucible is seated to fit the hole by grinding with diamond powder. The Cu and Nb metal are charged into the crucible and melted by the heat transferred from a Ta susceptor which is heated by an induction coil through a concentrator coil. A W/W-26Re thermocouple runs down the center of the tube as shown in fig. 7 and it allows the melt temperature to be monitored. After the Cu and Nb have been melted and homogenized the tube which plugs the hole in the crucible bottom is raised and the metal pours through the hole and is solidified in a water cooled Cu mold of 1/2" diameter. The chamber of fig. 7 is evacuated to a pressure between 0.2 to 2×10^{-5} Torr prior to the experiment. The vacuum is maintained until after the Cu melts and a pressure of around 100 Torr is then introduced for the remainder of the casting operation.

The chill casting unit of fig. 7 is limited to castings of around 75 gms. A new unit has been constructed and checked out

Fig. 7. The chill casting apparatus

which provides castings up to around 200 gms. The design of the new unit has been modified somewhat to facilitate ease of loading but it is essentially the same as the unit of fig. 7.

c. Tin Coating and Diffusion

In order to coat the Cu-Nb wires with a reproducible thickness of Sn the device shown in fig. 8 was developed. The wire is drawn through a molten tin bath in the manner shown. The thickness of the Sn coating is fixed by the diameter of the die located at the exit point from the bath. The die is made by swaging thick walled stainless steel tubes around a tungsten wire and then removing the wire by etching. The Sn is caused to wet the wire by employing a flux on the bath.

The apparatus used to diffuse the Sn into the wires in order to form Nb_3Sn is shown in fig. 9. The wires are contained inside evacuated quartz tubes which are placed inside a hollow copper cylinder. The temperature gradient along the copper cylinder is held at zero by use of a three zone furnace and the three thermocouples placed on the cylinder to monitor its temperature profile.

d. J_c Measurements

Facilities have been developed to measure critical currents up to 600A in magnetic fields of 9 Tesla both at DC and at 60 HZ over the temperature range from 15 to 4.2K. The diagnostic tools are standard electronics. Measurements at higher magnetic fields are to be done at the National Magnet Laboratory.

Fig. 8. The apparatus used to tin plate wire

Fig. 9. The furnace design for the diffusion experiments

Experimental Results

a. Alloy Preparation

Initial experiments were carried out in the apparatus of fig. 7 using both Al_2O_3 and ZrO_2 crucibles but without the Ta susceptor so that the hf heating current was induced directly into the Cu-Nb alloy. This method proved very difficult because the crucibles broke due to their poor thermal shock characteristics. Therefore, crucibles of boron nitride, BN, were used and this eliminated the thermal shock problem and, also, the molten alloy did not wet the BN. However, it became clear that the alloy was picking up an impurity from the BN crucibles in spite of the non-wetting, because they became mushy (2-phase) at around 1800°C after initially being all molten. Finally a series of alloys was prepared in Al_2O_3 and ZrO_2 (CaO stabilized) crucibles where the thermal shock problem was reduced by use of the Ta susceptor shown in fig. 7. The alloys were prepared by holding at 1850 - 1880°C for 5 minutes and then chill casting under argon. Initial alloys were all made up at a composition of 40 wt percent Nb in Cu. These alloys exhibited well behaved chill cast structures. The Nb distribution appeared fairly uniform and the Nb dendrite size was quite fine, on the order of 2 - $3\text{ }\mu\text{m}$, see fig. 10.

The experiments at 40% Nb were repeated several times with reproducible results. However, when alloys of 20 wt % Nb were prepared under identical conditions the structure was found to be a mixture of dendrites and small circular regions which we term spheroids, see fig. 11. A 3 dimensional picture of the spheroid structure produced by deep etching the Cu matrix and examination in

Fig. 10. Structure of Cu-40 wt % Nb alloy chill cast from a $\text{ZrO}_2(\text{CaO})$ crucible at 1850-1880°C. SEM photo at 1000X.

Fig. 11. Structure of a Cu-20 wt % Nb alloy chill cast from a $\text{ZrO}_2(\text{CaO})$ crucible at 1850-1880°C under conditions identical to Fig. 10. SEM photo at 1000X.

Fig. 12. Three dimensional structure of spheroids revealed by deep etching of Cu phase. SEM photo at 25° tilt and 3500X.

Fig. 13. Structure of a Cu-20 wt % Nb alloy chill cast from a Y_2O_3 crucible at 1850-1880°C under conditions identical to Fig. 11. SEM photo at 1000X.

the SEM is shown in fig. 12. It is seen that they are spheres rather than cylinders, and that their outer surface is a shell of Nb which encloses a 2-phase mixture of Cu and Nb in the interior. The spheroid structure suggests a monotectic reaction such as indicated by the phase diagram of fig. 2. If such a phase diagram were in fact correct it would mean that our directional solidification scheme would be limited to only those compositions outside the immiscible liquid region. However, we did not believe the monotectic diagram correct primarily because of lack of metallographic evidence in Schelle's work.⁵ Therefore, a rather extensive chemical analysis was made of the chill castings. All analyses were done at the Ames Laboratory. The techniques used for these analyses were as follows: For O, H and N, vacuum fusion, for C, combustion, and for the remaining elements, spark mass spectroscopy. Results of initial experiments are shown to the left of the vertical line of Table I. The pure Nb and Cu used to make up the alloys were also analyzed and the contribution of impurities from these starting metals, based upon an alloy of 20 wt % (14.6 at.%) Nb, is listed in the right hand column of Table I. The numbers enclosed in boxes indicate impurities which were clearly picked up from sources other than the starting metals. The vacuum fusion and combustion analyses are good to around $\pm 5\%$. However, the mass spectroscopy measurements are only semi-quantitative and differences of a factor of 2 to 3 may not be significant. It should be noted that the mass spec analysis measured the impurity level of all elements and only those elements having significant levels are shown in Tables I and II. Note also, that all concentrations except H are reported as atomic ppm rather than weight ppm.

Table I. Chemical Analysis of Experiments where Spheroids were Present. [All analyses are reported as atomic ppm except H which is weight ppm]

Crucible Element	BN	Al ₂ O ₃	ZrO ₂ ^(a)	ZrO ₂ ^(a)	Starting Nb and Cu ^(b)
O ^(c)	9,900	16,000	4600	3000	180
N ^(c)	20,000	62	19	87	89
H ^(c)	140	11	7	7	24
C ^(d)	164	67	45		63
B	20,000	8	2		4
Na	8	3	6		1
Mg	3	37	60		4
Al	3	1000	40		3
Si	86	210	100		68
Y	<.2	<.2	<.3		<.2
Zr	<.9	10	87		1
Ta	54	73	200		140
Fe	6	10	10		20
Ni	1	<1	12		3
Ca	100	30	20		1

(a) Stabilized with CaO (supplied by Ventron Corp.)

(b) Nb from Wa Chang Corp. and Cu from Asarco.

(c) Vacuum fusion analysis.

(d) Combustion analysis.

The boron nitride crucibles used in this work initially and reported in Table I all contain a significant level of B_2O_3 which might account for some of the impurity pickup from these crucibles. Because this material is such an attractive crucible candidate an additional experiment was carried out on a pyrolytic BN crucible which was virtually impurity free. However, a small amount of visual attack of the crucible by the Cu-Nb melt was evident and chemical analysis indicated a pickup of 3000 ppm B. It is concluded that BN crucibles will not be satisfactory for the present study.

It is apparent from Table I that a number of impurities such as O, Mg, Al and Ca were picked up by the alloys and could be inducing the monotectic reaction. There are two main sources of impurities possible, (1) contamination from the crucible material and (2) contamination from impurities carried in the argon. Therefore, zone refined high purity Cu and Nb was purchased from Materials Research Corp. and the argon was replaced with a special high purity grade of argon. In addition, new crucible materials were used, Y_2O_3 stabilized zirconia crucibles and Y_2O_3 crucibles. The impurity analysis for alloys chill cast after a 5 minute hold at 1850-1880°C in these materials with the zone refined metals and high purity argon are shown in Table II to the left of the vertical line. In all three experiments spheroids were not present in the chill casting. (Note: There were very small spheroids present at the chill wall but these are easily removed by machining. The source of these spheroids is discussed further in reference 6]. A typical structure for these alloys is shown in fig. 13. The alloys of figs. 13 and 11

Table II. Chemical Analysis of Experiments where Spheroids were Absent. [All analyses are atomic ppm except hydrogen which is weight ppm]

Crucible Element	ZrO ₂ ^(a)	ZrO ₂ ^(b)	Y ₂ O ₃ ^(c)	Starting Cu and Nb
O ^(d)	2300	2100	760	87
N ^(d)	29	19	34	25
H ^(d)	6	6	5	1
C ^(e)	57	96	62	37
B	3	3	<20	0.5
Na	<3	2	3	1
Mg	8.6	4	2.2	<0.1
Al	30	100	10	0.3
Si	40	20	11	48
Y	5.3	2.5	500	<0.2
Zr	230	110	1	<0.5
Ta	40	40	30	18
Fe	9.7	8.5	9.0	<3
Ni	6	0.9	1	<0.2
Ca	3	5	10	<1

(a) Stabilized with Y₂O₃ (Slip cast from powder supplied by Zircoa).

(b) Stabilized with Y₂O₃ (Supplied by Zircoa).

(c) Slip cast from high purity Y₂O₃ supplied from Ames Lab.

(d) Vacuum fusion analysis.

(e) Combustion analysis.

were made to the same composition, 20 wt % Nb, and chill cast under identical conditions. Comparison of the microstructures shows that it is critically important to this study that the spheroid structures be eliminated.*

It was apparent to us at this point that the spheroids were impurity induced and were most likely due to O impurities, but might just possibly be due to Ca or Si since these two impurities are also higher in alloys which display spheroids. An additional experiment was done to verify that the spheroids were induced by oxygen impurities. The experiment was identical to that of the second column in Table II except that 2.4 wt % of Cu_2O was placed in the crucible. The Cu_2O was produced by oxidizing high purity Cu. Large spheroids were found in the chill casting and the vacuum fusion analysis showed 10,500 ppm O and 87 ppm N impurities. Hence, this experiment confirms that the spheroids are produced by O impurities and may be removed by casting with either Y_2O_3 stabilized ZrO_2 or Y_2O_3 . At the present time the best crucible material appears to be Y_2O_3 .

*One might point out that the spheroid structures, although present in the chill cast material, might be absent from the directionally solidified material, because the spheroids are probably produced by a nucleation event and nucleation is more prevalent in a chill casting than in a steady state directional solidification experiment. However, directional solidification experiments on the alloys of Table I produced spheroids in great profusion.

b. Directional Solidification

The first priority of this phase of the work after development of the solidification apparatus shown in fig. 5 was to find a container tube which would carry the heating currents and not react with the alloy. The carburizing equipment shown in fig. 6 was not entirely satisfactory. The TaC layer on the inside of the tubes did not have a uniform layer thickness and appeared to be dangerously thin in many locations. In addition, the Ta tubes tended to warp quite a bit during the carburizing treatment. Both of these difficulties were overcome by going to the second method of carburizing described above where the carburizing was done at higher temperatures in a modified form of the apparatus of fig. 5. Figure 14 shows the carbide layer on the Ta wall. The white region at the bottom of this photo is a thin layer of remnant carbon which had been deposited upon the Ta wall. The Ta tube is at the top, the carbide layer is at the center, and it is seen that the carbide layer has separated from the carbon coating. The carbide layer actually is two adjacent layers, an inner layer of Ta_2C in contact with the Ta tube and an outer layer of TaC. The fracture surfaces within the carbide layers show the Ta_2C/TaC boundary at the right of fig. 14.

The carburized Ta tubes were not found to be entirely satisfactory for the directional solidification experiments. Although they adequately prevented attack of the Ta, loss of the Ta/carbide bond was noted in many places. For this reason Nb tubes were purchased and these tubes will be carburized to form NbC/Nb₂C layers.

Fig. 14. Carbide layer, TaC/Ta₂C as revealed on fracture surface of Ta tube. SEM photo at 1500X.

Fig. 15. Longitudinal section showing aligned Nb dendrites in a Cu matrix. Alloy of Cu-40 wt % Nb prepared by chill casting from Al₂O₃ crucible followed by directional solidification in carbide coated Ta tube at 4mm/sec growth rate. Optical photo at 238X.

We expect this carbide to be at least as stable as the $\text{TaC}/\text{Ta}_2\text{C}$, and if attack of the underlying tubes does occur it will only produce a small change in the Nb content of the alloy. Initial experiments on carburizing of the Nb tubes have been carried out and they reveal that the time-temperature cycle will have to be modified considerably from the Ta tubes to avoid preferential grain boundary carburization.

The directional solidification experiments have all employed carburized Ta tubes with a 0.25 mm wall and an I.D. of 1.75 mm. To date we have only done experiments on the alloys of high oxygen content and these alloys produced spheroid structures at the 20 wt % Nb compositions. At 40 wt % Nb, however, no spheroids were encountered and extremely encouraging results have been obtained as shown in figs. 15 and 16. Fig. 15 is a longitudinal section showing good alignment of the primary stalks of Nb dendrites. The transverse section of fig. 16 shows the dendrites as simple crosses indicating that the secondary branches are very short. These results indicate that the desired aligned dendrite growth with minimum side branching and small stalk sizes can be achieved with high temperature gradients and growth rates. The size of the primary dendrite stalks of fig. 16 ranges from 2 to 3 μm and their number in the 1.75 mm wire is around 50,000.

c. Wire Drawing

Initial experiments have been carried out on the chill cast alloys to develop the wire drawing, tin coating and J_c measurements technique. The alloy used for this work was a 40 wt % Nb composi-

Fig. 16. Transverse section of same alloy as in fig. 15. Optical photo at 792X.

Fig. 17. Longitudinal view of 10 mil Cu-40 wt % Nb wire after moderate etching of the Cu phase. Alignment of Nb dendrites produced by mechanical elongation is demonstrated. SEM photo at 5700X.

tion cast from $\text{ZrO}_2(\text{CaO})$ crucibles. It was hot swaged at 900 to 1000°C in stainless from 0.5 to 0.375 inches diameter to remove cavity and microporosity produced in the chill casting operation. It was then cold swaged to 70 mil, annealed and then drawn to 10 mil. Some of this 10 mil wire was placed upon a stub and a small amount of the Cu was etched from it to produce the result shown in fig. 17. It may be seen that the randomly oriented Nb dendrites of fig. 10 have been aligned quite significantly. The sample was then deeply etched to remove all Cu and figs. 18 and 19 taken. The bundled alignment of the drawn Nb dendrites is apparent in fig. 18, and in fig. 19 it is seen that the fiber sizes are quite small, on the order of .05 to 0.3 μm diameters. Transverse sections of the 70 mil wire have been successfully prepared after some difficulty and fig. 20 shows the irregular cross sectional shape of the Nb dendrites at this stage of drawing. Unfortunately, we have not been able to obtain any clear SEM pictures of the transverse sections of the 10 mil wires because of the very fine microstructure.

The 10 mil wire could be reduced further without fracture to a diameter of only 6 mils. However 3 mil wire was easily prepared from the 10 mil wire after it was annealed.

d. Tin Coating and Diffusion

Initial tin coating experiments were carried out with the apparatus of fig. 8 using 10 mil copper wires. The coating operation worked quite well. However, when experiments were carried out on the 10 mil wire of 40 wt % Nb-Cu alloys they were not successful because of difficulty in finding a low temperature flux which would

Fig. 18. Longitudinal view of Cu-40 wt % Nb wire lying upon an aluminum stub after Cu was completely etched away. SEM photo at 460X.

Fig. 19. Higher magnification picture of a region of fig. 20. SEM photo at 9000X.

Fig. 20. Transverse section of chill cast Cu-40 wt % Nb alloy after reduction to 70 mil wire. SEM photo in specimen current mode at 1000X.

permit the wires to be wetted by the tin. Therefore, this method was abandoned at least temporarily in favor of simply electroplating the 10 mil wires. Initial experiments have employed an alkaline stannate bath. Results with this bath have not been as reproducible as desired due to difficulty in filming the Sn cathode. It is believed that this difficulty may be reduced by using an acid bath and the required chemicals for such a bath have been ordered.

At the writing of this report the initial tin diffusion experiments are being carried out in the apparatus of fig. 9. They are being done on the 10 mil wire coated with the electroplating technique.

e. J_c Measurements

The primary goals of the superconducting measurements were to determine:

1. J_c vs H for various Nb filament diameters in the 100 to 5000 Å range
2. Length of Nb filaments (continuity)
3. Resistivity of the Cu matrix
4. T_c vs filament diameter in proximity effect regime.

Critical current measurements for the chill cast $Nb_{40}Cu_{60}$ samples are shown in Fig. 21. A sample with wire diameter of 0.010" and filament sizes ranging from 1000 to 5000 Å had a critical current of 147 A or a J_c of 2.8×10^5 A/cm² (based on the total cross section of the wire). Just before the quench, say at 140 A, the resistivity of the specimen is less than 3×10^{-15} Ω-cm. In low magnetic fields the transition is sudden with no sign of flux flow. After the

Fig. 21. Critical current versus magnetic field for Cu-40 wt % Nb alloys chill cast and drawn

0.010" wire was annealed and drawn to 0.003" diameter the filament size was reduced to the range from 300 to 1500 Å and the critical current rose by a factor of 2 to $J_c = 5.4 \times 10^3$ A/cm². At currents just below J_c , the resistivity was less than $2 \cdot 10^{-16}$ Ω-cm and the phase transition showed no flux flow region in low magnetic fields. From the upper limit on the resistivity one can put a lower limit on the filament length by the relation

$$\rho_{\text{eff}} = \rho_N \left(\frac{\ell^2}{d^2} \right)$$

where ℓ is the filament length and d is the filament diameter. The data show that the filaments must be at least 0.7 cm long. They may be much longer. A second 0.003" diameter sample was measured with the same result as the first sample within 10%.

J_c vs H curves fall off very rapidly above the critical field of bulk Nb (3000 Oe) and approach zero at about 16,000 Oe. The temperature dependence of J_c and H_{c2} both follow $(1-t^2)$ rather well, where $t = T/T_c$.

In the normal state, all samples measured show a resistivity of 4.1 μΩ cm at room temperature and 1.2 μΩ cm at 4.2K. In the normal state the Cu has much lower resistivity than Nb so this value gives a good measure of the Cu mean free path. Using $\rho \ell = 0.67 \cdot 10^{-11}$ we find a mean free path of about 600 Å in the Cu.

Critical current and resistivity measurements also have been made at 60 Hz. The samples quench when the peak current first reaches the DC value of J_c . Hence the RMS critical current is down by a factor of $\sqrt{2}$. It is more difficult to put a lower limit on the

resistivity at 60 Hz because the inductive signal is so much larger. We can say, however, that the resistivity at 60 Hz is less than $10^{-11} \Omega\text{-cm}$.

Conclusions to be drawn from these studies of Nb filaments are given in the summary and conclusions section.

Summary and Conclusions

Equipment has been designed, built, and checked out which allows (a) the chill casting of Cu-Nb alloys, (b) directional solidification of batch quantities of Cu-Nb alloys, (c) tin plating, diffusion annealing and mechanical drawing of the alloys, and (d) measurement of critical currents and their magnetic field dependency.

The following significant results have been obtained:

- (1) It was shown that a monotectic reaction does occur in Nb-Cu alloys at compositions of 15 and 20 wt % Nb, but the reaction is induced by oxygen impurities. Consequently aligned dendrite growth may not be achieved in Cu-Nb alloys near these compositions unless the oxygen content is kept below around 3000 ppm atomic.
- (2) It was demonstrated that aligned dendrite growth may be achieved in Cu-Nb alloys with dendrite stalk sizes on the order of 1 to 2 μm and with very little side branching. Wires with 50,000 Nb filaments were demonstrated in the initial studies.
- (3) Critical current measurements on chill cast Cu-40 wt % Nb alloys have yielded the following results:
 - a) J_c values are on the order of $5 \times 10^5 \text{ A/cm}^2$.
 - b) J_c increases with decreasing filament diameter

- c) For current densities just below J_c the resistivity is at least 10^{10} lower than pure Cu at room temperature.
- d) Filament lengths are at least 0.7 cm.
- e) The resistivity of the Cu matrix is comparable to commercial wire.
- f) Temperature dependence of J_c is approximately $J_c = J_0(1-t^2)$.
- g) The proximity effect is not detrimental to J_c in the filament size range of a few hundred Å's.
- h) J_c at 60 Hz is the same as J_c at DC.

Future Work

Future work will proceed roughly as follows:

- 1) Finish study of compatibility with crucible materials.
- 2) Develop our tin plating and diffusion processing to optimize J_c values of chill cast and drawn wire.
- 3) Carry out initial studies on chill cast Cu-Nb-Sn alloys to determine if the addition of Sn will cause the as-cast alloys to become too brittle for wire fabrication.
- 4) Prepare wires from directionally solidified alloys and measure their critical current properties and dependency upon the state of stress.
- 5) Optimize the critical current properties and stress dependency with respect to alloy composition, solidification rate, and diffusion processing.

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