Fermilab

TM-690
1610.000
August 17, 1976

RESULTS OF THE FERMILAB[+] WIRE PRODUCTION PROGRAM

B.P.Strauss*, R.H.Remsbottom*, P.J.Reardon*¶,
C.W.Curtis[†] and W.K.McDonald[†]

I. BACKGROUND

In the Spring of 1974 the commercial wire business had reached a low ebb and continued supply by multiple sources was not encouraging. Fermilab, then just starting on its Energy Doubler program, was faced with the problem of maintaining multiple sources of supply as well as a number of technical problems in obtaining the high critical current densities required for the dipole magnets. To alleviate these problems it was decided to embark on a multifaceted program to provide a usable high production method of fabricating multifilament composite superconductor.

Besides obtaining a usable production method we wished to explore several other technical innovations. First we wished to determine the effect on production of the prior processing of the NbTi rod. Furnace cooled and gas quenched NbTi rod was used for comparison. Secondly, in other projects we were having difficulty with Ti depletion by diffusion from the alloy matrix. Therefore a Nb diffusion barrier was used in one of the test billets. Thirdly, in an effort to ultimately reduce ac losses in our finished magnets we plated some of the copper with Ni so that diffusion layers of cupronickel would be formed during subsequent processing. Finally, in order to avoid some of the upset in the cross section pattern of the rod during extrusion, the use of bonded, preformed copper and alloy hexes was tried in the place of individual hexoid copper tubes and alloy rods. We will discuss each of these programs below.

II. PROCESSING

Design

Basic design features of the superconductor manufactured in this program are as follows:

1. 8 in. diam extrusion billet size
2. 3000 filaments (nominal) of Nb46.5Ti
3. Cu to superconductor (S.C.) ratio of 2:1 (nominal)
4. Final size wire of .0375 in. and/or .025 in. diam with twist of 2/in.

The major features of billets A, B, C and D are shown in Table I.

The nickel plating in billets A and C is intended to serve as a barrier to eddy currents which increase loss capacity in multifilamentary superconductors, particularly in ac or ramped applications. The use of pure nickel in the form of plating is unusual for this application; standard industry practice is to utilize a cupronickel alloy cladding to form a high resistance barrier with this feature is desired.

TABLE I
Features of Test Billets

Billet	Type of S.C.	Condition of .084 in.∅ S.C.	Other Features
A	Nb46.5Ti	Recrystallized & gas quenched (fast cool)	Inside surface of Cu extrusion can was plated with nickel.
B	Nb46.5Ti Clad with pure Nb	Recrystallized and furnace cooled (slow cool)	
C	Nb46.5Ti	Recrystallized and furnace cooled (slow cool)	Nickel was plated as follows: a) outside surface of .120 in. hex Cu surrounding each Nb46.5Ti filament b) inside surface of the Cu extrusion can
D	Nb46.5Ti	Recrystallized and gas quenched (fast cool)	----

The use of a thin cladding of pure niobium tightly surrounding each Nb46.5Ti filament was incorporated into billet B as one solution to the interdiffusion of Cu and Nb/Ti during extrusion preheat. In particular, the diffusion of Ti into the surrounding Cu matrix is considered undesirable for several reasons: 1) the titanium loss in the superconductor may alter its electrical properties, and 2) the goal of high resistivity ratio Cu adjacent to the superconductor filaments is compromised.

Assembly (or stacking) of a superconducting billet most often consists of building an array from three basic components: 1) straight lengths of hexagonal o.d., round i.d. Cu tubes, 2) straight lengths of solid, pure Cu rod for pattern and/or filler, and 3) straight lengths of round superconductor wire (or rod). When assembling components 1) and 3), a gap of several thousandths of an inch is required between each wire and the Cu tube i.d. to permit insertion of the wire. This results in a significant amount of void space in the stacked billet, and tight straightness requirements for both wire and tubing. The void space fraction becomes greater as the number of filaments is increased; for a billet of roughly 3000 filaments, the void fraction would normally be too great for reliable extrusion. An isostatic compaction process is sometimes used to lower the void fraction, but costs are high and results unpredictable.

In this program an alternative technique was used: round Cu tubing was sunk tightly onto Nb46.5Ti wire by drawing through a hexagonal die. This eliminates the above mentioned clearance gap and produces a hexagonal shape which can be tightly packed. Straightened lengths of hexagonal Cu/Nb46.5Ti and hexagonal pure Cu filler rod are stacked together to complete the array.

Manuscript received August 17, 1976.

*Fermi National Accelerator Laboratory, P.O.Box 500, Batavia, Illinois 60510.

[+] Operated by Universities Research Association, Inc. under contract with the United States Energy Research and Development Administration

¶ Present address: Princeton University, Plasma Physics Laboratory, Princeton, N.J. 08540.

[†] Teledyne Wah Chang Albany, P. O. Box 460
Albany, Oregon 97321

Presented at the 1976 Applied Superconductivity Conference, Palo Alto, California. To be published in the IEEE Trans.Mag. (1977).

III. INPUT MATERIALS

Copper

Alloy 101 CDA copper tubing of the following dimensions and quantity was purchased from Small Tube Products, Inc., Altoona, Pennsylvania: .170 in. o.d. × .115 in. i.d. × 10 ft lengths; 38,500 ft total.

The copper tubes were examined for cleanliness at Teledyne Wah Chang Albany (TWCA) by drawing lint-free, soft cloth pads through them. The pads emerged from the tube with dark residue of a very thin layer of copper corrosion film. Sectioned tubes, however, displayed a bright and shiny surface visually. It was decided to perform a cleanliness test per specification ASTM B280-74 which would insure i.d. cleanliness of the Cu tubing. TWCA and Small Tube Products, Inc. both performed the washing of the inside of representative tube samples with trichloroethylene or carbon tetrachloride as described in ASTM B280-74. Each sample tested had either no detectable residue or a level of residue less than half the specified maximum of 0.038 g/m^2. Small Tube Products, Inc., pointed out that additional cleaning of Cu tubing ordered in short lengths (i.e., about 2 ft) is normally required due to the final sawing operation at their plant which utilizes a spray coolant of water soluble oil. However, since our tubing was ordered in 10 ft lengths, the need for additional cleaning would not be expected.

The 8 in. diam extrusion cans and lids used in the program were provided by Fermilab. These components are made from certified CDA alloy 101 copper materials.

Nickel

As previously discussed, billet C was stacked with copper clad Nb46.5Ti filaments which were plated with nickel. Selection of the type of nickel plate was based on chemical purity, visual uniformity and adhesion, and microhardness. The following types of nickel plating baths were considered: dull Watts (warm bath), Watts (room temperature), standard sulfamate, and low stress sulfamate. The microhardness data in Table II show the primary reason why low stress nickel sulfamate plating was chosen:

TABLE II
Hardness of Various Nickel Platings

Type of Ni Plating	DPH Hardness, 50 Gram Load
Low stress sulfamate	191
Standard sulfamate	247
Watts (room temperature)	335
dull Watts (warm bath)	225

Visual examination of the roughly 3100 Cu/Nb46.5Ti filaments plated with low stress nickel sulfamate indicated good plating uniformity. Metallography performed on two random samples from the production run showed a plating thickness of .0005-.0008 in. (max).

Two Cu extrusion cans were plated on the inside with nickel. A dull Watts nickel bath was chosen because of its purity and simplicity of application for the part configuration involved. The bath was warmed to about 130°F and poured into an acid cleaned extrusion can. A nickel anode was suspended into the bath and a voltage of 6 volts, 35 amps, applied for 30 minutes. Micrometer readings showed that an average of .0015 in. Ni plating was applied per surface.

Nb46.5Ti

The basic superconductor unit in this design is .084 in. diam Nb46.5Ti wire in the vacuum annealed (recrystallized) condition. Material from TWCA heat 590531 (930 ppm oxygen) was chosen for the superconductor. Properties of this heat were monitored closely during processing at TWCA from ingot to .084 in. wire. At 6 in. diam and 1.6 in. diam, metallographic tests were performed following beta anneal and water quench to study DPH micro-hardness, grain size, and percent recrystallization. At 1/2 in. diam the Nb46.5Ti rod has fully certified to the Fermilab specification in the vacuum annealed (recrystallized) condition. Some of the test results at 1/2 in. diam are as follows:

- product hardness (avg of 8)	145 HV10
- R.T. UTS (avg of 4)	73.3 ksi
- R.T. YS (.2% offset; avg of 4)	70.3 ksi
- R.T. % El (2 in. gage; avg of 4)	28%
- R.T. % RA (avg of 4)	80%
- Percent Recrystallization	100%
- ASTM Grain Size	8.5-9

Six hundred pounds of the 1,697 pound yield of heat 590531 at 1/2 in. diam were started down to .084 in. diam wire for stacking (approximately 95 pounds of this 1/2 in. diam Nb46.5Ti rod was clad with niobium for billet B). Rods corresponding to the middle one-third of the ingot were selected.

Recrystallization of the Nb46.5Ti wire was performed at the stacking diameter of .084 in. First of all, coils of the .084 in. wire were vacuum annealed and furnace cooled as follows:

1-1/2 hr at 1475°F/furnace cool. Time to
cool from 1475°F to 575°F (300°C) - about
90 minutes

Then a portion of the wire (160 pounds) was gas quenched at Vac-Hyd Processing Corp., Torrance, California, for billets A and D. The process was:

1 hr. at 1475°F/He gas quench. Time to cool
from 1475°F to 500°F - about 4 minutes.

Tensile tests at room temperature and metallography tests were performed to compare the .084 in. diam Nb46.5Ti in the gas quenched and furnace cooled conditions. The tensile results showed that the gas quenched rods had higher elongation and reduction in area when compared to the furnace cooled rods. This agrees with other work.[1]

Hexagonal Cu/S.C. Lengths

To process round Cu tubing (.170 in. o.d. × .115 in. i.d. × 10 ft) and .084 in. diam superconductor into .120 in. across the flats hexagonal Cu/SC "building blocks", four major operations were employed: loading, drawing, sawing, and straightening.

Inserting the coiled wire into the 10 ft Cu tube lengths in the dry condition proved to be difficult. Even inserting relatively straight Nb clad Nb46.5Ti lengths, which retained a bow from being vacuum annealed in loose coils, with a carefully filed "nose" required much effort and time. As a result, a stain-free liquid solvent was used as a "lubricant" during loading.

Drawing of the tube assemblies was performed on an electric powered, chain driven drawbench at the intermediate speed, 18 ft/min. The .120 in. flat-to-flat hex die was positioned inside a steel die box filled with lubricant. About 1/16 in. thick rubber diaphrams were used on either end of the die box to allow passage of the material through the box while retaining the lubricant. As drawn, the hex composite length was about 205 in., 4 in. of that being the swaged "point". Pointing was performed on a standard two die swage to a typical diameter of .118 in. The finished hex rods were sawed to the required length, and deburred.

Straightening was performed on small, hand operated roll straighteners. Each straightener has three sets of plastic rolls machined to fit the .120 in. flat-to-flat hex shape. The straightener frame is made of steel and has adjusting screws which move a set of rolls up and down, allowing the proper offset to be determined and maintained.

Extrusion Billet Assembly

Assembly of the four extrusion billets was performed by Magnetic Corporation of America who was used later as a drawing source. Standard industry procedures for cleaning and stacking worked well using the hexagonal Cu/SC composite lengths. Experiments showed that the Cu/Nb46.5Ti wire interface on the hexagonal composites is tight. No liquid seepage occurs between the Cu and the wire during cleaning.

It was decided to give each billet a unique identity by replacing several Cu/NbTi hex elements with pure Cu hex elements to form a small pattern. The pure Cu keys are positioned near the outside of the filament array very close to the inner surface of the extrusion can, as shown in Fig. 1.

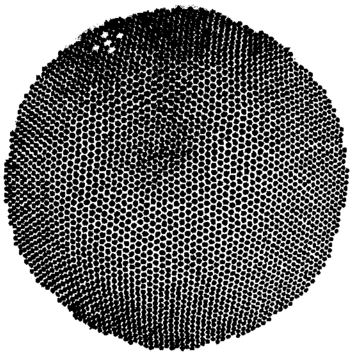

Fig. 1. Cropping of the tail end of billet A after extrusion. Note the identification symbol at the 11 o'clock position. The diameter of the cropping at this point is 2.25 in.

Cleaning experiments were conducted on Ni plated filaments and a Ni plated can cropping prior to final cleaning of these components for stacking. A proposed cleaner for Ni (H_2SO_4-H_2O) did not remove fingerprints or oily smudges; whereas, a short time (20-30 seconds) in the normal copper cleaner (H_2SO_4-HNO_3-H_2O) removed such surface markings without any apparent pitting problems. A slightly mottled surface appearance was observed on the can cropping sample, but a solvent wipe with a clean cloth showed the nickel to be clean, with no residue present. It was decided to use a short duration acid copper cleaning procedure for the nickel plated components.

With the rear lid of the packed extrusion can in place, each assembled billet was placed in an electron beam welding chamber which was then evacuated. A typical cycle includes keeping the composite billet in vacuum overnight to remove gases inside and then electron beam welding the rear lid in place.

Billet Extrusion

Extrusion of the four billets was performed at TWCA on a Lombard Hydraulic Press with a 3500 ton rated capacity. The extrusion ratio for all billets was 12.6:1, based on an 8 in. diam input billet and a 2.25 in. diam extruded rod product. Preheating time for each billet was 50 minutes or less, using an induction coil furnace. Preheat temperatures for billets A, B, C, and D were as follows: 1110°F, 1220°F, 1110°F, 990°F (respectively). Water quenching of each billet was performed in a movable 25 ft long tank placed just beyond the runout table of the extrusion press.

The product of extrusion billet C contained "bubbles" along the length. To examine these surface protusions, billet C was cut into two pieces. The C-1 piece toward the nose was 12 ft 4 in. long and the C-2 piece toward the tail was 5 ft 7.5 in. long. Analysis of the bubbles indicated they were disbonded regions between the nickel plated can and the array which was also nickel plated. Possible explanations for this condition are: 1) the lower packing density of billet C compared to the others, 2) outgassing from the Ni plating or from Ni salts formed during acid cleaning, or 3) a combination of the above.

The ends of each extruded rod were prepared to view the keying elements. In all cases, no perceivable twisting or relative movement of the keys was observed from nose to tail.

Rod Drawing (2.25 in. Diam to .335 in. Diam)

The extruded rods were shipped to Phelps-Dodge, Elizabeth, New Jersey, for further processing. A 15% reduction schedule was used from the starting diameter of 2.25 in. diam nominal to 0.335 in. diam.

Wire Drawing (.335 in. Diam to Finished Size)

At the onset of the program, it was established to draw all wire to a final size of .0375 in. diam with a twist pitch of 2 turn/in. Subsequently, Fermilab decided that the final diameter of some of the wire should be decreased to .025 in. diam with the same twist pitch of 2/in. As a result, some wire was completed with a final diameter of .0375 in. and some with .025 in., depending on contractual agreement with the three vendors involved. Starting with .335 in. diam material, the drawing vendors processed this material to final size on their normal production equipment, using procedures considered as standard within their respective facilities. Airco, Intermagnetics General Corp., and MCA participated in this part of the program as well as in the thermal processing below.

IV. THERMAL PROCESSING

Three basic schedules of wire drawing and heat treating were used. Two participants used a scheme reviewed by Critchlow et al.[2] This method features an extended anneal midway in the processing schedule from 0.335 in. and then cold work to the final size. The second method featured only cold work to the final size. The third method used was similar to that described by McInturff et al.[3] Here one hopes to form flux pinners by precipitating a finely dispersed second phase by an extended heat treatment at final size. This last method has been mainly used with high Ti alloys. All methods used quick up to 300°C and down anneals to soften the copper during the reduction schedule.

V. RESULTS

In examining the various schedules of wire drawing and heat treating, the Critchlow type of schedule provided the highest and most uniform data from billet to billet. It consists of a long anneal at 400±20°C at a cold work point giving about 99% reduction in area from the extrusion size. Several quick copper anneals at 300°C may be interspersed to aid in fabrication. A final anneal at finished size both peaks up the resistivity ratio of the copper as well as the critical current of the alloy by moving dislocations to subcell walls. Using this method critical currents of $1.7 \times 10^5 A/cm^2$ at 5T, 4.2°K could be maintained in all billets.

Measurement of ac losses was not performed in any of the samples, so the effect of the diffusion formed CuNi layer in the wire from billet C is not known. The highest J_c value at 5T, 4.2°K ($1.81 \times 10^5 A/cm^2$) was obtained for a sample from billet B (Nb clad Nb46.5Ti filaments). However, the production weight yields from both billet B and billet C were considerably lower than from the billets of more standard design.

Gas quenching (fast cooling) the Nb46.5Ti rods from the recrystallization temperature just prior to stacking (i.e., billets A and D) did not produce any product with improved current.

The copper cladding and sinking method looks promising and should save production costs. We found that in spite of this it was important to attain good packing density in the billets to assure uniform filament pattern and reduce breakage in wire drawing.

Overall a procedure was found for fabricating wire in large production lots. This production process, while not producing the optimum performance, achieved reproducible results and is suitable for constructing dipole magnets. At present, twenty tons of Cu/Nb46.5Ti superconductor for the Energy Doubler program have already been completed or are in process.

REFERENCES

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