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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 as half of the industry was then going through bankruptcy. 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 billet identity and features shown in Table I are basic to an understanding of this program.

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.Ø 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 since the titanium loss in the superconductor materially affects its electrical properties.

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 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.

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

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

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

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 Wah Chang 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, the situation did not exist in our Cu tubing order which specified 10 ft tube lengths.

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. Heat 590531 was approved for forging and next sampled at 6 in. diam, following an air anneal of 1/2 hr at 1600°F and water quench (95% recrystallization). To assure 90% recrystallization in production, the air annealing cycle at 6 in. diam has been standardized at 1 hr at 1800°F/water quench. Three 1/4 in. thick, full diameter samples were sawed and submitted for metallographic examination: one located 22 in. from the original ingot top, one located about 194 in. from the original ingot top (middle sample) and one located at the bottom of the ingot. Edge and center DPH hardness tests, as well as grain size and percent recrystallization examinations, were conducted with the following results:

	Top	Middle	Bottom
% Recrystallization	95	95	95
Average ASTM Grain Size	4	3-1/2	3
DPH, 10kG load			
Edge	142	149	162
Center	166	149	148

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). Rods corresponding to the middle one-third of the ingot were selected. Room temperature tensile and metallographic samples were taken in duplicate at random locations following annealing of the Nb46.5Ti at 1/2 in. diam and before and after annealing at .084 in. diam.

Gas quenching of 160 pounds of .084 in. diam Nb46.5Ti wire for billets A and D was performed at VacHyd Processing Corp., Torrance, California. The process was:

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

It should be noted that the above wire had been annealed for 1-1/2 hr at 1475°F and furnace cooled at Wah Chang prior to the anneal at VacHyd. The process for the furnace cooled wire was:

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

Processing of .170 in. o.d. × .115 in. i.d. × 10 ft copper tubing and coils of .084 in. diam NbTi wire (or approximately 17 ft lengths of .084 in. diam Nb clad Nb46.5Ti wire) to produce .120 in. "across the flats" hexagonal pieces of Cu/S.C. can be broken into the following operations: loading, drawing, sawing, and straightening.

Inserting the coiled wire into the 10 ft Cu tube lengths, without using solvent as an aid, 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 in the dry condition.

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. Short lengths of wire still protruding after drawing were cut off with a wire cutter. The finished hex rods were sawed to the required length.

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 one of the industrial vendors who was used later as a drawing source. Standard industry procedures for cleaning and stacking were used.

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.

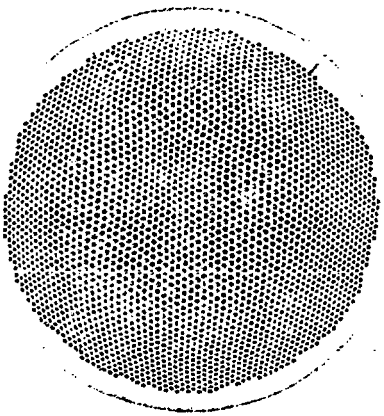

Fig. 1. Cropping of the tail end of billet A after extrusion. Note the identification symbol at the 11 o'clock position. The diameter of the cropping at this point is 2.25 in.

Cleaning experiments, using Ni plated filaments and a nickel plated can cropping, were conducted prior to stacking. The proposed nickel cleaner (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. 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. This was thought to be due to nickel salts that formed during cleaning as well as the low packing density of the billet.

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.

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 $1.7 \times 10^5 A/cm^2$ could be maintained in all billets. We did not measure the ac losses in any of the samples so the effect of the diffusion formed CuNi layer is not known.

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 that would be acceptable for constructing dipole magnets. It is felt that this method could be peaked up with time.

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