

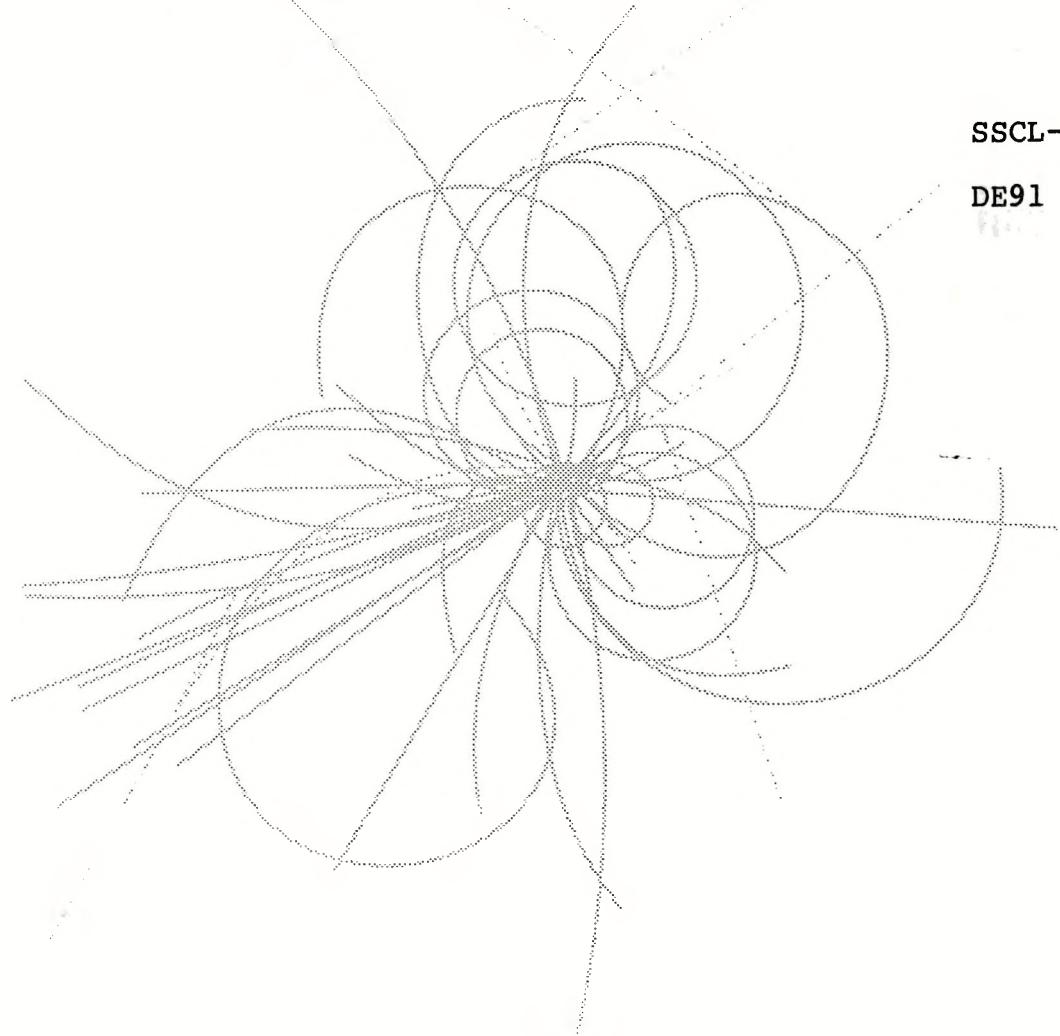
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Summary of the Performance of Strand Produced for the 1990 SSC Dipole Program

**D. Christopherson, D. Capone, J. Seuntjens,
D. Pollock, and C. Hannaford**

March 1991

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Produced for the 1990 SSC Dipole Program***

D. Christopherson, D. Capone, J. Seuntjens, D. Pollock,
and C. R. Hannaford

Magnet Division
Superconducting Super Collider Laboratory†
2550 Beckleymeade Ave.
Dallas, TX 75237

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SUMMARY OF THE PERFORMANCE OF STRAND PRODUCED FOR THE 1990 SSC DIPOLE PROGRAM

D. Christopherson, D. Capone II, J. Seuntjens, D. Pollock,
and C. R. Hannaford

Magnet Division
Superconducting Super Collider Laboratory*
2550 Beckleymeade Avenue
Dallas, TX 75237

Abstract: In 1990 and at the beginning of 1991, more than 4 million feet of wire was delivered to support the SSC Dipole Program. This wire was fabricated to meet specification SSC-MAG-M-4141, and test results and various statistics are compiled here. Certain strengths and weaknesses in the performance of the delivered strand are discussed, including analysis of strand breakage in certain billets. Test results of cable manufactured for 40 mm dipole magnets and 50 mm dipole magnets are reported, and a brief overview of the 1991-1992 Conductor Program is included.

INTRODUCTION

This work is a compilation of test data and analysis results on superconducting wire delivered during the past year. The focus is on material that has been fabricated into cable and used for the SSCL Magnet Program. The billet numbers used here are chosen strictly for the purpose of vendor neutrality and have no significance beyond this. Table 1 is a list of the billets included in this paper and the basic statistics of each. Unless otherwise stated, the reported values are derived from the raw data from the vendors' measurements. A companion paper presented by Erdmann et al.¹ addresses critical current (I_c) measurement variation between different facilities.

BILLET PERFORMANCE DATA—MECHANICAL

As we near the production scale-up period, parameters such as piece length and yield become very important issues. Historically, manufacturing performance in these areas has been inconsistent. However, progress has been made over the last year, and we have seen some promising results from the 13 batches of material covered here. Figure 1 illustrates the piece-length performance of each batch of material against the SSC-MAG-M-4141 requirement of 90% of the order delivered in lengths greater than 10,000 feet.

*Operated by the Universities Research Association, Inc., for the U.S. Department of Energy under Contract No. DE-AC02-89R40486.

Table 1. Basic statistics of billets produced during 1990. Type 1.8, 1.5, and 1.3, strand are for nominal Cu/SC ratios of 1.8:1, 1.5:1, and 1.3:1 respectively.

Billet No.	Billet Type	Total Length ft	%L>10K ft	Yield	Mean I_c	Mean Cu/SC	Alloy Source
1	1.8	333976	26	65	316	1.80	TWCA
2	1.8	382489	77	74	325	1.73	TWCA
3	1.8	371245	62	72	323	1.75	TWCA
4	1.5	236831	29	NA	370	1.52	TWCA
5	1.3	117680	95	NA	362	1.27	TWCA
6	1.3	126021	97	NA	365	1.27	TWCA
7	1.5	100773	60	NA	330	1.48	TWCA
8	1.5	166931	66	NA	333	1.46	TWCA
9	1.8	372346	87	72	311	1.80	Cabot
10	1.8	370859	45	72	313	1.79	Cabot
11	1.8	396047	85	77	306	1.79	Cabot
12	1.8	395765	93	77	311	1.79	Cabot
13	1.8	350699	93	68	309	1.79	Cabot

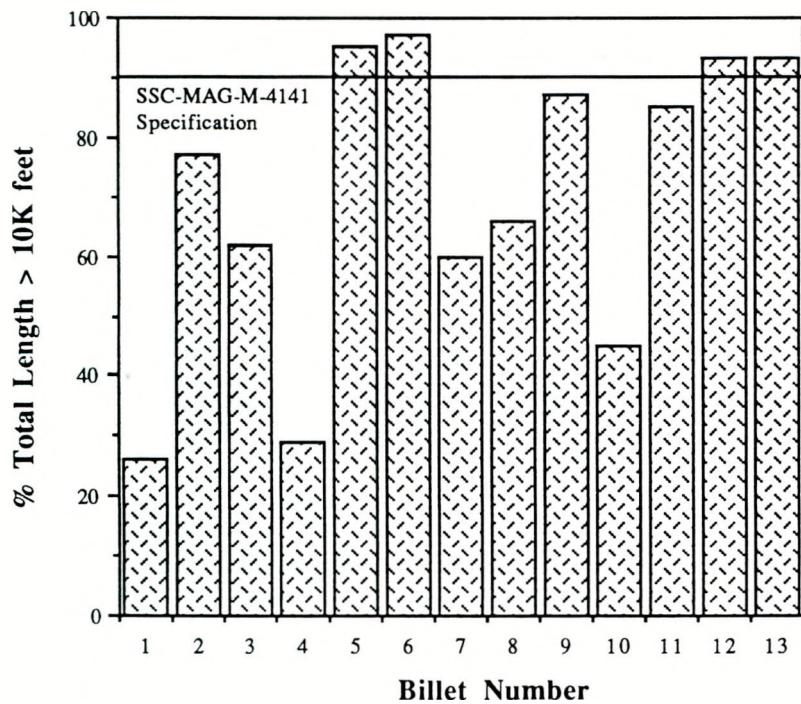


Figure 1. Piece-length performance of billets produced during 1990. SSC-MAG-M-4141 specifies that 90% of total delivery be in pieces greater than 10,000 ft.

Material with poor piece length and yield has been examined; in some cases reasons for this performance have been identified. Batches 7 and 8 were initially designed to be Type 1.3 strand, and they represent the second half of billets 5 and 6. At the request of the SSCL, this material was delivered to meet the Type 1.5 specifications by skipping an important occlusion-removing step in the vendor's normal process. The short lengths of billet 10 were

attributed in part to a machine problem during the drawing of this material. The extremely poor piece lengths of billets 1 and 4 were determined to be due to inclusion problems by wire break analysis performed at the SSCL. Billets 2 and 3 were of marginal piece length and have also been proven to contain inclusions that limited their drawability. Discussion of this break analysis work is included later in this paper.

An excessive number of wire breaks can substantially reduce the amount of final strand delivered for an order, showing that piece length is a contributing factor to the yield characteristics of a billet. In many cases, the nature of the R&D contracts governing this work called for extensive sampling and the processing of partial billets, preventing comparable yield statistics. However, SSC Outer billets 1, 2, 3, and 9–13 did not involve such contracts. Multifilament yield discussions will concentrate on these billets.

The multifilament yield was calculated by assuming a 12-in.-diameter billet with a 28 in. (2.33 ft) core length. Using the idea of conservation of volume, we obtain the equation:

$$L_f = (2.33 \text{ ft}) \left(\frac{12 \text{ in}}{0.0255 \text{ in}} \right)^2,$$

where L_f is the final length in feet of 100% yield of Outer conductor at the final diameter of 0.0255 in. This equation gives us a 100% theoretical yield of 516,000 ft for an SSC Outer billet. Dividing this number into the total length of delivered wire for a billet gives us the yield illustrated in Figure 2. This is an approximation based on an assumed multifilament billet size and does not account for any pickling or shaving steps. From these calculations, the multifilament billet yields have ranged from 65% to 77% during the last year. Increasing this yield is one of the main goals of development efforts of 1991.

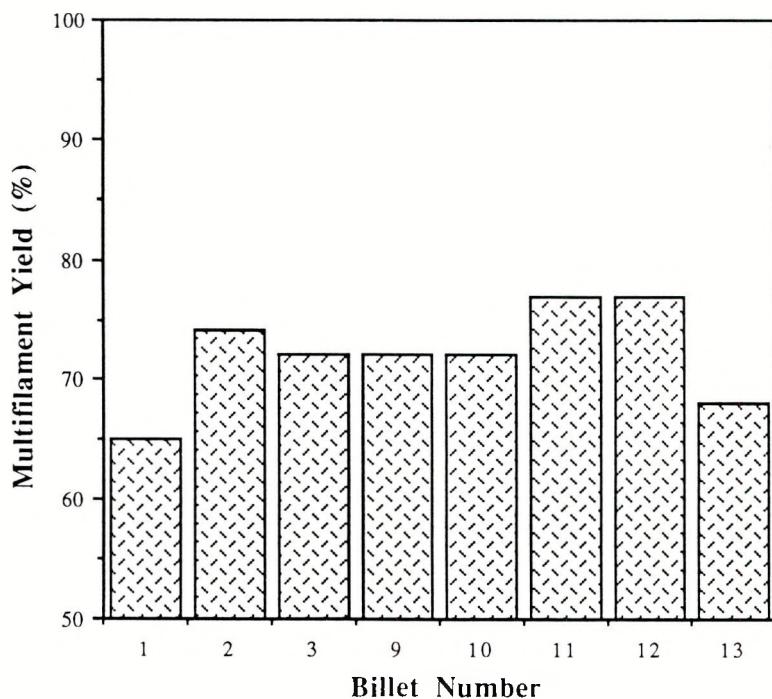


Figure 2. Multifilament yield statistics. These are approximate values derived from calculations involving an assumed initial multifilament diameter and core length.

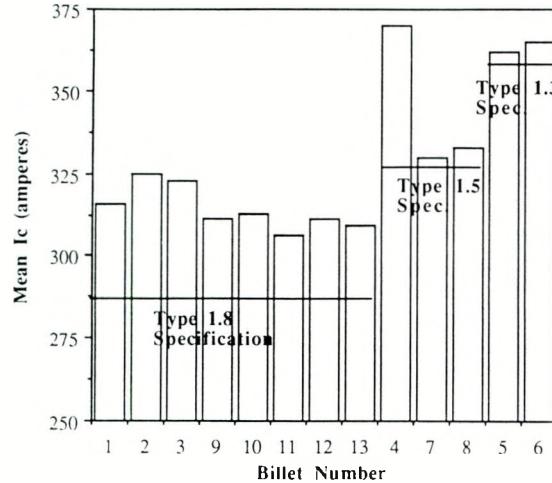


Figure 3. Mean critical current of each billet compared to its respective specification. SSC-MAG-M-4141 requires Type 1.8 material to be 285 A at 5.6 T, Type 1.5 to be 328 A at 7.0 T, and Type 1.3 to be 357 A at 7.0 T.

The slope of the critical current vs. field values reported in Table 3 are calculated from measurements made at Brookhaven National Laboratory (BNL). Ic measurements at 5 T and 6 T on samples of strand determine this slope, which is essentially linear over intermediate fields. The reduction of this slope is important in order to reduce the low field magnetization of the strand at fields near the injection field of 0.6 T. Because magnetization is proportional to Ic, a steeper slope leads to higher magnetization at low fields. For this reason, a flatter Ic vs. H slope, while maintaining acceptable Ic at high fields, is desirable.

Table 3 also lists the mean and standard deviation of the Cu/SC ratio of each billet. All billets meet their respective Cu/SC specifications, and in most cases the standard deviation is small. For reasons of material uniformity, standard deviations of 0.04 and 0.05 seen in a few of these billets are less desirable. With a well-defined and controlled process, Cu/SC ratio standard deviations of 0.02 can be attained with a negligible decrease in material yield. This should become the case as larger lots of material flow through each vendor's production system in the next year.

Table 3. Electrical measurement statistics of billets produced in 1990.

Billet	Mean Ic (A)	STD Dev Ic (A)	Slope Ic vs. H	Mean Cu/SC	STD Dev Cu/SC
1	316	3.5	1.26	1.80	0.02
2	325	6.3	1.25	1.73	0.02
3	323	7.1	1.25	1.75	0.05
4	370	5.7	1.26	1.52	0.03
5	362	7.7	1.24	1.27	0.04
6	365	5.5	1.24	1.27	0.02
7	330	2.5	1.24	1.48	0.04
8	333	2.1	1.24	1.46	0.02
9	311	3.5	1.25	1.80	0.02
10	313	3.4	1.25	1.79	0.02
11	306	4.8	1.25	1.79	0.02
12	311	4.3	1.25	1.79	0.02
13	309	7.4	1.25	1.79	0.04

Another important mechanical characteristic is the springback of the strand. This tendency of the wire to spring back toward its original shape after deformation is critical to cable fabrication. Strand with a high springback tendency will not lie flat after cabling and will "pop out" along the faces of the cable. This pop-out problem has been observed in cable recently manufactured using R&D strand with springback values greater than the specified limit. Springback is quantified by using a specialized fixture that measures in angular degrees. With a 5 lb. counterweight on one end, the other end of the wire is wound around a spindle for the count of 10 revolutions. The weight is then secured so as not to affect the reading. The spindle is released and allowed to unwind slowly as the wire springs back toward its original shape. Each counter-revolution adds 360 degrees to the value, plus the last partial revolution, for a total springback measurement that cannot exceed 980° for SSC Inner billets and 1090° for SSC Outer billets.

Springback is dependent upon wire processing, heat treatments, and strand geometry, and varies among vendors. All of the material discussed in this work met the specifications. Table 2 shows the springback statistics of the Outer billets.

Table 2. Springback statistics of SSC Outer billets. (SSC-MAG-M-414 requires the spring back to be less than 1090° for SSC Outer material)

Billet No.	No. of Tests	Mean°	Min.°	Max.°	Std Dev.°
1	89	825	736	920	31
2	32	820	766	858	23
3	55	805	746	900	28
9	3	797	782	814	16
10	9	835	811	846	11
11	32	840	794	890	25
12	22	820	780	896	26
13	25	806	752	870	28

BILLET PERFORMANCE DATA—ELECTRICAL

Figure 3 shows how the mean I_c of each billet compares with the I_c specification for each type of wire. The mean I_c data show that each billet comfortably meets its respective I_c requirement. However, we are also interested in the standard deviation of the I_c and the slope of the I_c vs. field (H) plot. Table 3 lists these results for each billet.

The standard deviation of the critical current, which is also related to the standard deviation of Cu/SC ratio, is important to cable performance. Large differences in I_c from one strand to another can lead to variations in coil performance if the strand mixing during cabling is not sufficient. For the same reasoning, the I_c standard deviation between billets is just as important as the I_c standard deviation within a billet. As we near production stage, we will produce cable made up of strand from many billets. These billets must be nearly identical in all specified variables.

Although billets 9–13 have been processed identically, our database is still too small to be able to draw any firm conclusions about billet to billet variations. However, preliminary indications are promising. The I_c standard deviation between all pieces of billets 9–13 is 5.2 A (1.7% variation from a mean of 311 A), with the I_c ranging from 290 A to 322 A at 5.6 T. Deviations of this magnitude are further narrowed simply by strand mapping the cable by piece length alone, independent of the strand I_c . A strand map of these 5 billets resulted in a cable I_c range of 70 A (0.6%) before degradation.

CABLE PERFORMANCE

Table 4 is a list of the electrical test results from BNL on cable fabricated using strand from the batches mentioned earlier. Descriptions of these test methods and accuracies can be found elsewhere.²

These cables have been manufactured for use in the 40 mm Collider Dipole Program, the 50 mm Model Dipole Program, and the 50 mm Accelerator String Test Program. Each cable meets its respective critical current specification, and in some cases greatly exceeds it. One reason for this is the low cabling degradation values, especially for the 50 mm Inner design cables. This cabling degradation as reported by BNL is calculated by dividing the measured I_c of the cable by the product of the average measured I_c of uncabled strand samples and the number of strands in the cable. The reported degradation is an estimate based on the average strand I_c from a limited number of samples. In addition, the cable measurements are corrected for self field effects while the virgin strand is not. These factors combine to make it possible for the calculated degradation to have a negative value, although realistically it cannot be less than zero. Explanations for the low degradation in the 50 mm cables are being investigated; initial indications are that the width-to-thickness ratio in these cables is improved over that of the 40 mm designs.³

Table 4. Cable fabricated for the SSC Dipole Program. Measurements were performed at Brookhaven National Laboratory. Outer cable is tested at 5.6 T, Inner cable is tested at 7.0 T. 50 mm Outer cable is denoted by SSC-4-*-*₁, 50 mm Inner cable by SSC-3-*-*₁, 40 mm Outer cable by SSC-2-*-*₁, and 40 mm Inner cable by SSC-1-*-*₁.

Cable I.D.	Length (ft)	I _c (A)	Spec. I _c (A)	Degrad. (%)	Billet
SSC-3-I-00021	1060	11024	9990	-4.30	4
SSC-3-I-00022	1120	10827	9990	-2.50	4
SSC-2-I-00023	4031	9294	7860	1.70	2 & 3
SSC-2-I-00024	2310	9470	7860	-0.10	2 & 3
SSC-2-I-00025	1969	11415	10152	-0.50	2 & 3
SSC-4-I-00026	1969	11341	10152	0.10	2 & 3
SSC-4-I-00027	5520	11615	10152	-1.50	2 & 3
SSC-4-I-00028	1320	11075	10152	2.30	2 & 3
SSC-4-I-00029	1160	11075	10152	2.30	2 & 3
SSC-4-I-00030	660	10944	10152	0.10	1 & 3
SSC-4-I-00031	645	10944	10152	0.10	1 & 3
SSC-4-I-00036	1381	10886	10152	0.20	12
SSC-4-I-00037	5568	10339	10152	4.90	9-13
SSC-2-I-00032	4305	8789	7860	4.30	1
SSC-2-I-00033	3428	8953	7860	2.80	1
SSC-2-I-00034	1015	8901	7860	3.20	1
SSC-3-S-00021	2340	10079	9990	-3.70	7 & 8
SSC-3-S-00022	1052	10079	9990	-3.70	7 & 8
SSC-3-S-00023	4200	10079	9990	-3.70	7 & 8
SSC-3-S-00024	1258	10917	NA	-2.20	6

DISCUSSIONS

All of this material has been manufactured to meet SSC specification SSC-MAG-M-4141. Material performance comparisons against this specification reveal only one major deficiency: piece length. Historically, billets meeting the piece-length requirement of 90% of

delivered pieces longer than 10,000 ft have been the exception rather than the rule.⁴ Considering the finished strand diameter and filament size, this is an aggressive requirement to meet, but the volume of conductor procurement required to supply the SSC project necessitates long piece lengths. In fact, the specifications to which future material is being ordered, SSC-MAG-M-4145 (Inner strand) and SSC-MAG-M-4146 (Outer strand), are more demanding in terms of piece length. These documents call for the acceptance of only those pieces longer than 1500 m. This can seriously reduce the yield of a billet experiencing drawing difficulties.

Wire Break Analysis

Wire break analysis has been done at the SSCL on several billets experiencing severe piece-length problems. When possible, samples of both ends of the breaks were mounted and polished for metallographic examination. Because of the possibility of losing an important feature of the break during polishing, many repetitions of delicate polishing and microscopic observations were performed on selected samples. Even when exercising great care, there is a danger of polishing away an inclusion or other artifact of the failure. Scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX) were performed on samples in which an interesting feature was seen. Approximately 25 samples from various billets were examined for the cause of failure. Only a few are singled out here and discussed at length.

SEM and EDX analysis were performed on two selected breaks of billet 1, one at 0.064 in. diameter and the other at 0.0397 in. diameter. These breaks were similar in appearance, although a cup and cone morphology was more evident in the latter sample. In both specimens, the filament array showed no signs of distortion until very near the failure region. When material was removed in the systematic manner described above, it appeared that the failure originated at a point between the copper jacket and the filament array. In the case of the 0.0397 in. break, several foreign particles were discovered at that location and were analyzed. These inclusions exhibit silicon, manganese, and tin signals. Because our equipment is unable to accurately detect elements having an atomic number less than fluorine, we can only conjecture that these particles are oxide or carbide inclusions that were imbedded on the copper can wall during can cleaning or billet packing.

Three of the wire breaks from billet 4 were chosen for similar analysis. The diameters of these breaks were approximately 0.07, 0.064 and 0.058 in. The two smaller diameter samples were very much the same as the breaks described above in billet 1. A failure near the can wall revealed foreign inclusions at the origin. These particles contained Al, Si, and Ca. The 0.07-in.-diameter sample differed; it appeared that the failure originated at the interface between the filament array and the copper core. An inclusion containing silicon was found at the bottom of the cup portion of the fracture.

Wire breaks from the order that includes billets 9–13 show stainless steel inclusions; Figure 4 shows an SEI image and spot X-ray spectrum on one of these inclusions. Although these billets had decent piece-length performance, the presence of these recurring particles suggests that a much better performance is possible. Also included in this order is a series of five Inner billets which are currently being delivered. Preliminary indications show the piece length of these billets to be extremely poor. Wire break analysis results from the vendor and SSCL agree that many stainless steel inclusions, similar to those seen in Figure 4, are present in the billets.

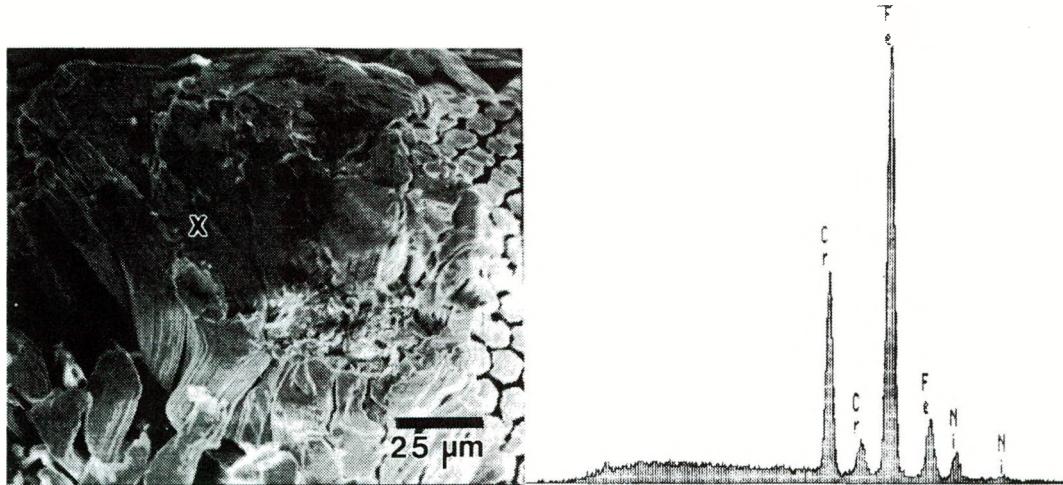


Figure 4. SEM image and EDX spectrum of a stainless steel inclusion. The point of the spot X-ray analysis is marked.

Severe piece-length problems were also experienced in a series of R&D billets separate from those discussed above. Similar analysis techniques were used to determine the cause of these drawing difficulties. Many samples were examined, both at the SSCL and at the vendor's location; all had very similar and peculiar characteristics. Figure 5 shows an SEM image of a break cross-section. The large filament and surrounding distorted array are not the puzzling aspect of this fracture; however the large Nb-rich region covering nearly half of the large filament area led to questions of alloy homogeneity, barrier integrity, and monofilament assembly procedures. EDX showed this region to be essentially pure Nb, which suggests that it may be the diffusion barrier. When analyzed by EDX, the alloy composition of the anomalous filament appeared homogeneous and similar in composition to the surrounding filaments. Deeper polishing revealed that the large filament and barrier actually reduced to normal size and geometry approximately 50 microns away from the fracture area.

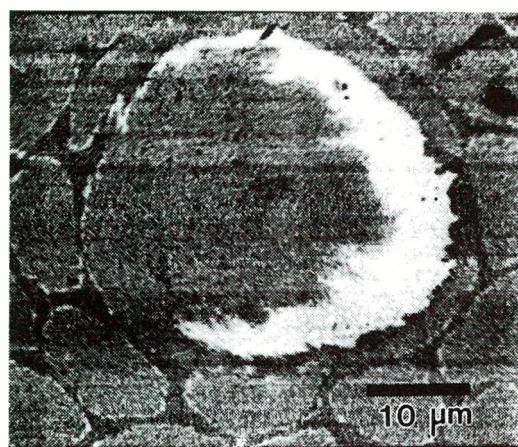


Figure 5. SEM image of a strand break cross section. The light colored region is the Nb diffusion barrier.

The vendor's retrospective investigation of the processing of this material revealed a possible set of circumstances which resulted in this material failure. The monofilament anneal was performed at a relatively small diameter in a nitrogen atmosphere. At this size, the copper jacket was thin enough, and the temperature high enough to allow grain boundary diffusion of nitrogen into the niobium barrier, forming locally hard areas. These hard regions caused a filament failure, leading to a strand failure several drawing steps later.

ALLOY SOURCE COMPARISONS

During the past year, drawing difficulties in billets, such as those previously discussed, and observable microstructural differences between alloy from different suppliers have drawn attention to the NbTi alloy stock as a possible concern. So far, we have not found any reason to believe that the alloy is the cause of these difficulties; in fact, we have shown that these piece-length problems most likely originated elsewhere.

Visible "tree rings" are present when some Cabot alloy at a filament size of 200 microns or larger is etched with hydrofluoric acid, and line scan EDX analysis on *unetched* surfaces show macro inhomogeneities of ± 1 wt% Ti. This is within the specification window of ± 1.5 wt% Ti. For comparison, line scan EDX of samples from 5.75-in.-diameter ingots of Teledyne material showed *micro* segregation of ± 1 wt% Ti. Due to our machine resolution, the reported sizes of these samples are the limit below which this segregation cannot be seen.

A review of billet performance gives no evidence that wire problems or variations can be associated with the product of one specific alloy supplier. Critical current variations between billets 1, 2, and 3, which used Teledyne alloy, and 9-13, which used Cabot alloy, resulted from Cu/SC ratio differences and diameter variations. Furthermore, the performances of billets 9-13 prove that SSC specifications can be achieved with Cabot alloy regardless of the macrosegregation present in the material.

1991-1992 CONDUCTOR DEVELOPMENT PROGRAM

The 1991-1992 Conductor Development Program consists of two phases. Phase I is separated into two parts running in parallel and designated as Phase IA and Phase IB.

Phase IA is a development effort designed by each vendor to improve the manufacturability and production efficiency of his baseline process. Included in the development plan of each vendor is a required investigation of certain parameters dictated by the SSCL. These required parameter studies include piece length, yield, and alternative alloy sources.

Phase IB is a baseline process order for a required amount of finished cable (3400 kg for SSC Inner and 3500 kg for SSC Outer). Each vendor is to use his baseline design to deliver the required amount of conductor to meet SSC Specification SSC-MAG-M-4147 (Inner cable) or SSC-MAG-M-4148 (Outer cable).

Phase II is an order for a required amount of cable (6120 kg for SSC Inner and 6300 kg for SSC Outer), and is to begin when Phases IA and IB are completed. Work on Phase II will not start until the vendor passes a production readiness review. This material is to be manufactured to meet the same specifications, using knowledge gained through Phase I.

At this time there are seven vendors negotiating contracts to participate in this two-year program. This number may decrease depending upon negotiation and performance during the qualification program. We are confident that the SSCL, along with the superconducting cable

vendors, will gain the experience and knowledge necessary to supply the Magnet Program with ample high-quality conductor through the duration of the project. In addition, the SSCL is committed to assisting other potential wire vendors who wish to qualify using another funding source.

SUMMARY

The electrical performance of strand produced for the SSC Magnet Program over the past year has been very encouraging. Critical current specifications have been satisfied using alloy from two vendors, and the variations of I_c within a single billet are promising. Likewise, the critical current measurements among 5 billets processed identically had a standard deviation of only 5.2 A at 5.6 T.

However, the piece length and yield statistics of much of this material is a major weakness. The largest factor contributing to these difficulties is cleanliness. The large frequency of inclusions found in the limited number of tested samples gives an idea of the amount of impurities that are present throughout the length of these billets. The need for the improvement of piece length and yield is obvious, and as we move toward supplying conductor for the industry prototype magnets, the 1991-1992 Conductor Development Program will emphasize this requirement.

ACKNOWLEDGEMENTS

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