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EVALUATION OF VARIOUS FABRICATION TECHNIQUES FOR FABRICATION OF FINE FILAMENT NbTi SUPERCONDUCTORS



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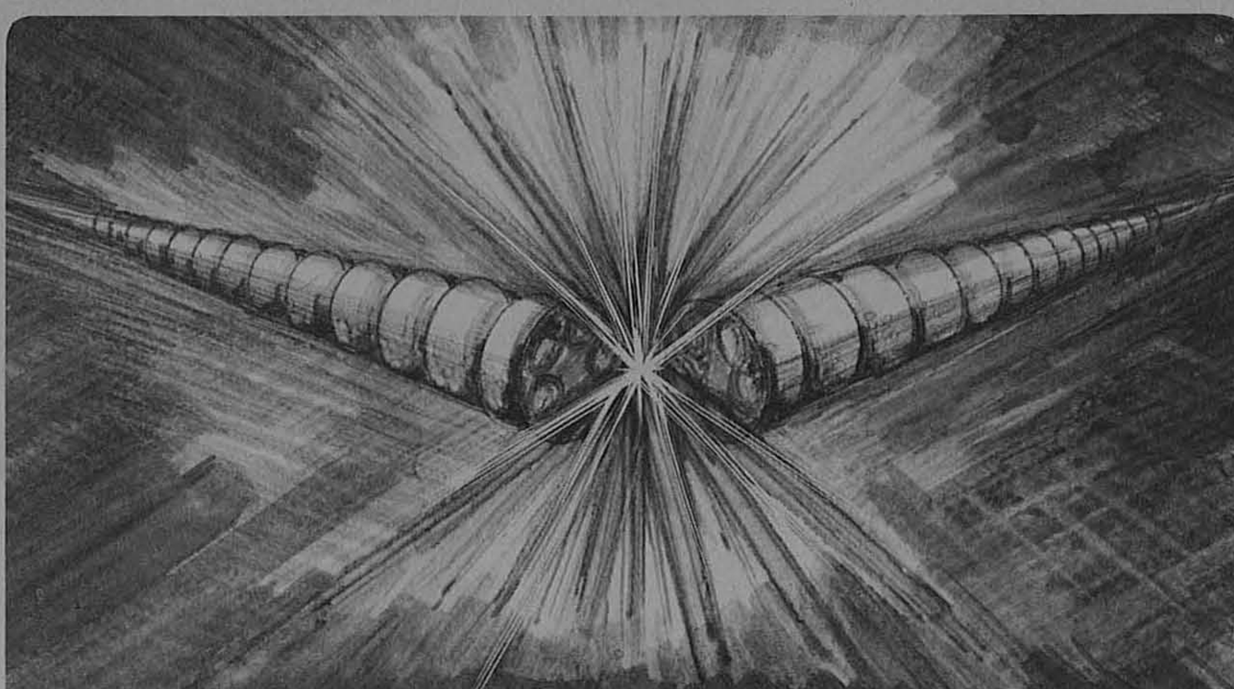

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# EVALUATION OF VARIOUS FABRICATION TECHNIQUES FOR FABRICATION OF FINE FILAMENT NbTi SUPERCONDUCTORS,*

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## Abstract

The successful fabrication of a fine filament high current density NbTi superconductor can have a significant impact on the cost of the Superconducting Supercollider. Consequently, we have been exploring various approaches for fabricating this type of superconductor, in collaboration with several superconductor wire manufacturers. The techniques investigated include double conventional hot extrusion, large single stack conventional hot extrusion, and warm hydrostatic extrusion. The important conductor properties (critical current density, piece length, yield, and cost) will be compared for the various approaches. Finally, the feasibility of manufacturing production quantities of a fine filament conductor will be assessed.

Table I. Wire and Cable Specifications for Accelerator Dipole Magnets

| Dimensional Tolerances | Tevatron/CBA | HERA | SSC |
|---|---|---|---|
| Strand | ±.0076 mm | ±.01 mm | ±.0025 mm |
| Surface | Sn/AG Coating | Sn/AG Coating | Bare |
| Fil.Diam. | 8 μm | < 20 μm | 5 μm |
| Twist Pitch | 13 mm | 25 mm | 25 mm |
| Cu:SC | 1.8/1 | 1.8 +0.2 -0.1 | ±2% |
| Keystone Angle | 2.05° | 2.2° | 1.68° |
| Cable Mid-thickness (mm) | +.000 outer edge -.025<br>+.000 inner edge -.025 | ±.02 | ±.0127 |
| Width (mm) | +.000 -.025 | ±.025 | ±.025 |
| Cable Edge Radius | – | >.25 mm | – |
| Min.Length Strand (m) | 305 | 400 | 764 |
| Min. Length Cable (m) | – | 385 outer<br>600 inner | 675 outer<br>542 inner |
| Dim. Q.C. | 10-stack | 10-stack | 10-stack |
| RRR | 45 | 60 | 70 |
| $J_c$ | 1800 A/mm$^2$ 5T | 2400 A/mm$^2$ 5T | 2750 A/mm$^2$ 5T<br>1100 A/mm$^2$ 8T |

## Introduction

In September, 1986, the Magnet Selection Advisory Panel chose a high field magnet design for the Superconducting Super Collider (SSC) dipole magnets. The conductor specified for these magnets is NbTi with a filament diameter of 5 μm and minimum critical current density ($J_c$, 4.2 K) of 2750 A/mm$^2$ at 5T and a $J_c$ (4.2 K) of 1100 A/mm$^2$ at 8 T. These values are considerably higher than those specified for existing dipoles in the Tevatron or those under construction for the HERA project (see Table I.). Consequently, a conductor development program is underway to insure that superconducting cable can be made to these specifications in a cost effective manner. The initial goal for this development program was to produce industrial-size billets of multifilamentary NbTi with a filament size of 18-20 μm and a $J_c$ (4.2 K, 5T) greater than 2400 A/mm$^2$. This goal was met in 1984[1] and the results were confirmed by the fabrication of 12 additional billets[2]. However, if strands with 20 μm filaments are used in the SSC dipoles, powerful correction coils are necessary in order to correct the field distortion produced at the accelerator injection field (0.3T). This field distortion is produced by magnetization effects which are proportional to the size of the superconductor filaments and hence can be reduced by reducing the filament diameter. Consequently, a second phase conductor development program was initiated in 1984 with the goal of producing strands with high $J_c$ (> 2750 A/mm$^2$ at 5T) and a fine filament diameter (< 5 μm).

## Experimental Details of the Strand Development Program

Initial experimental results[3,4] and a review of the published literature on fine filament NbTi conductors[6,7] indicated that conventional fabrication techniques would not produce fine filaments with acceptable properties. One problem is the formation of a brittle intermetallic compound layer between the Cu matrix and the NbTi filaments as a result of the interdiffusion which occurs during the thermomechanical processing of the composite. Another problem is the progressive deterioration of the uniform filament cross section, described as "sausaging", which becomes much more pronounced as the filament diameter is reduced. Sausaging behavior is caused by the intermetallic formation, but can also result from other causes and be present in the absence of intermetallic compound formation, as described below.

Fine filament fabrication problems have also been studied in detail for the case of multifilamentary $Nb_3Sn$, since, in this case, $Nb_3Sn$ is produced by the diffusion and reaction between Nb filaments and Sn in the bronze matrix. In order to limit reaction temperature and time, the Nb filament size must be maintained at 5 μm or less. The results of several fabrication studies[8,9,10] on $Nb_3Sn$ have been reported, and these studies provided useful direction for resolving the problems associated with the fabrication of fine filament NbTi.

Several potential processes for the fabrication of fine filament NbTi were identified, and a matrix of experiments (Table II.) were begun in order to evaluate both technical feasibility and cost. Two

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approaches for eliminating the problem of intermetallic compound formation were investigated. First, a diffusion barrier was applied around each NbTi filament in order to prevent the interdiffusion of Cu and Ti. This approach was investigated in research and development billets, processed at Supercon[11,12] and Intermagnetics General Corp. (IGC)[11,13]. Although the use of diffusion barriers had been described[5,7] or alluded to[6] previously, it was necessary to demonstrate that this approach could be used in large scale fabrication and in a cost effective manner.

Table II. Billets Fabricated in SSC Phase I Fine Filament R&D Program

| Designation (Subcontractor) | Filament Size at Final Wire Diam. | Extrusion Technique | Experimental Goals |
|---|---|---|---|
| LBL-1 (Supercon) | 8 μm | Lg. single stack | High $J_c$, diffusion barrier |
| LBL-2 (Supercon) | 6 μm | Lg. single stack | demo, economics, fil. spacing |
| 5914-12 (IGC) | 5 μm | Dbl. hot extrusion | |
| 5914-221 (IGC) | 2.7 μm | Dbl. hot extrusion | Fine fil., diffusion barrier |
| 5914-222 | 2.7 μm | Dbl. hot extrusion | demo, economics, fil. spacing |
| 5940-21 (IGC) | 5 μm | Lg. single stack | Production experience |
| 5940-22 (IGC) | 5 μm | Lg. single stack | Production experience |
| 5941-1 (IGC) | 5 μm | Lg. single stack | Alt. NbTi source eval. |
| Hydrostatic-1 (Nat'l Standard) | 2.7 μm | Warm hydrost. | Hydrostatic extrusion eval., fil. spacing |
| Hydrostatic-2 (Nat'l Standard) | 2.7 μm | Warm hydrost. | Hydrostatic extrusion eval., fil. spacing |
| Hydrostatic-3 (Nat'l Standard) | 2.7 μm | Warm hydrost. | Hydrostatic extrusion eval., fil. spacing |

In the first phase of the fine filament program, Supercon constructed two production-size billets (305 mm diameter); one was designed for SSC inner layer cable and the other for SSC outer layer cable. Likewise, IGC constructed two billets, 254 mm in diameter, one with a copper to superconductor ratio of 1.3:1, and the other with a ratio of 1.8:1 (see Table II for additional details). Finally, the feasibility of using reduced temperature processing in order to reduce intermetallic formation without the use of a diffusion barrier was investigated by use of warm hydrostatic extrusion and short time intermediate heat treatments. This option was explored with three billets, 165 mm in diameter, extruded at the National Standards production facility in Perth, Scotland.

In order to reach the requisite large number of filaments in the composite strands, two billet assembly approaches were investigated. Billets LBL-1 and LBL-2 were stacked at Supercon by assembling approximately 4,165 Cu-clad monofilament NbTi rods. The advantages of this large single stack approach include (i) a uniform environment which is provided for nearly all filaments, and (ii) higher yields which are made possible as a result of reduced loss in extrusion. The 5914 series billets and the hydrostatic extrusion billets were fabricated using a double stacking approach. The advantages of this approach include (i) handling of fewer elements in the assembly of each billet, and (ii) the ability to handle the large number of filaments required to reach ultrafine (1 μm or less) filament sizes.

## Discussion of Strand Improvment Program Results

The primary purpose of this strand evaluation program was to gain information on the following parameters: critical current density, process reliability, and ability to fabricate strand into cables. We now discuss the results of processing the billets described in the previous section into strands in order to evaluate $J_c$ and drawability (piece length). Subsequently, the strands with acceptable $J_c$ and drawability were then cabled for evaluation of cabling degradation. These results will be described in a later section.

The large single stack billets (LBL-1 and LBL-2) showed the best critical current results. Samples with optimized heat treatments yielded $J_c$ values

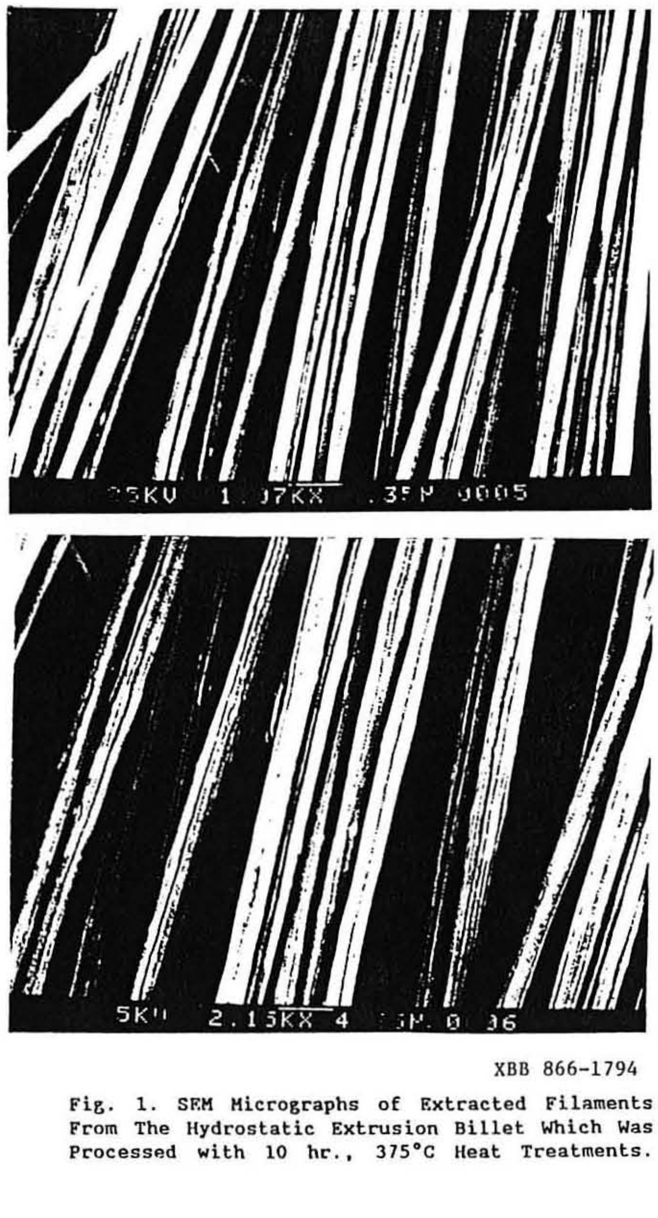

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Fig. 1. SEM Micrographs of Extracted Filaments From The Hydrostatic Extrusion Billet Which Was Processed with 10 hr., 375°C Heat Treatments.

over 3100 A/mm$^2$ at 5T and over 1170 A/mm$^2$ at 8T. The n-values (for a discussion of the relationship between n-values, $J_c$ and filament quality, see paper MF-7, these proceedings) for these strands were quite high - approximately 30 for 8T and 50-60 for 5T measurements. The double stack billets yielded $J_c$ values in the range of 2300-2500 A/mm$^2$ at 5T and n-values of about 25. The hydrostatic extrusion billets yielded low $J_c$ values and low n-values even in the absence of any appreciable intermetallic compound formation. For example, one sample was processed with minimal heat treatments (three at 375°C for 10 hrs.). SEM analysis (Fig. 1) showed little intermetallic compound formation, but considerable sausaging; this result was confirmed by critical current measurements which yielded n-values of approximately 25.

The results for the hydrostatic extrusion billets were dominated by filament sausaging behavior and can be explained by examining the interfilament spacing compared with the spacing used in the other billets (Table III.). Billets LBL-1 and LBL-2 had uniformily spaced filaments and showed no tendency toward sausaging (Fig. 2). Billets 5914 had bundles of closely spaced filaments separated by thick copper layers. The filaments within the bundles were uniform, but those at the edges exhibited sausaging (Fig. 3). The hydrostatic extrusion billets, with comparatively wide spacing within the

Table III.
Filament Spacing in Fine Filament Material

| Material | Fil.Diam. | Spacing/ Fil.Diam. | Spacing at Final Wire Size | Fil.Quality |
|---|---|---|---|---|
| LBL-1 | 8 μm | 0.11 | 0.9 μm | Excellent n = 60 |
| LBL-2 | 6 μm | 0.17 | 1.0 μm | Excellent n = 50 |
| 5914-12 | 5 μm | 0.2 | 1.0 μm | Sausaging at edge of 7X bundle n = 25 at 5 μm fil.diam. n = 13 at 2 μm fil.diam. |
| 5914-221 | 2.7 μm | 0.14 | 0.36 μm | Sausaging at edge of 19X bundle n = 25 |
| Hydrostatic | 2.7 μm | 0.4 | 1.0 μm | Sausaging at edge and interior of 19X bundle n = 10-25 |

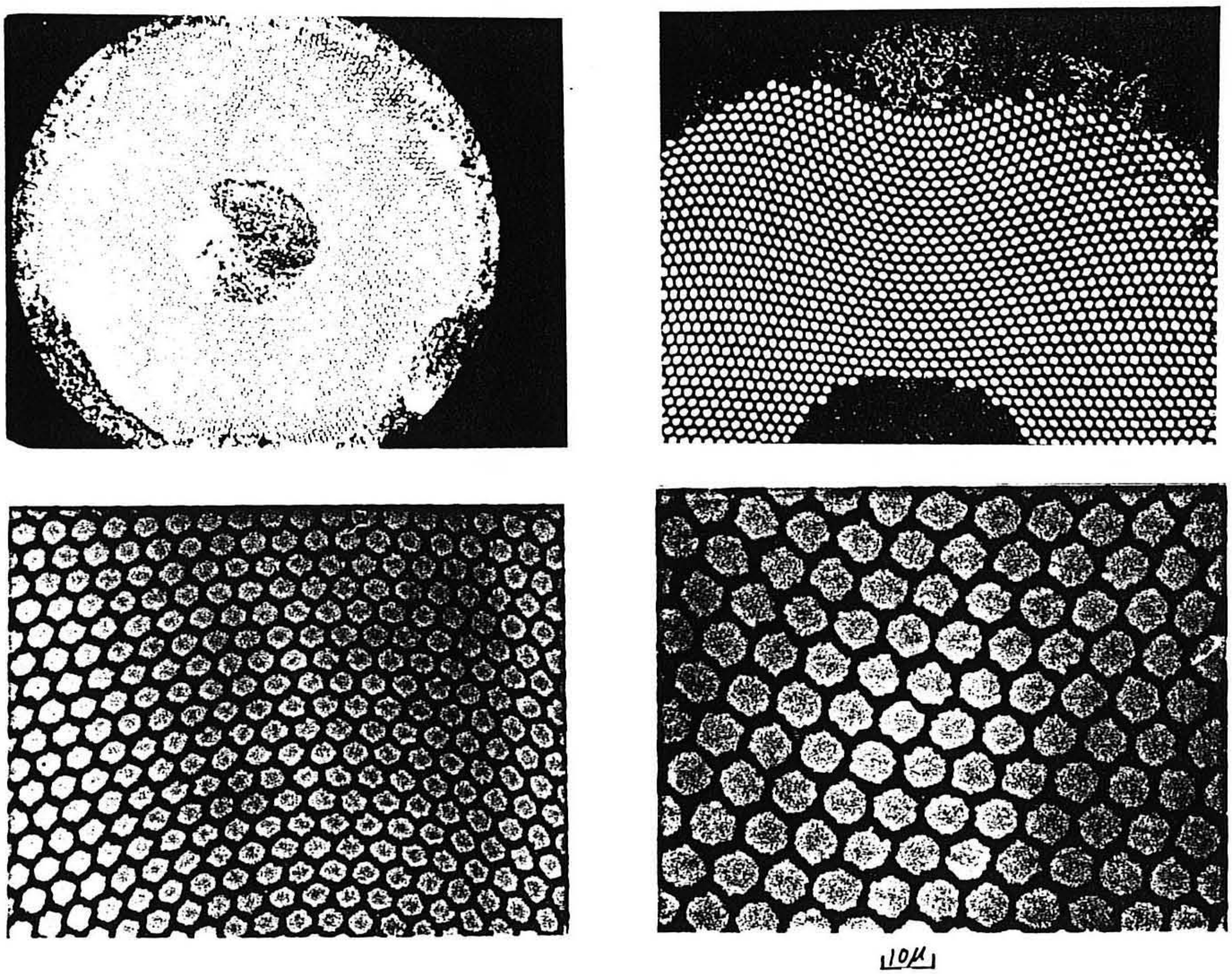


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Fig. 2. LBL-1 - Fine Filament NbTi Made by Large Single Stack Approach at Supercon, Inc.

bundles, showed sausaging, both at the edges and within the filament bundles.

From these results, we conclude that filament spacing must be maintained uniform and close together. However, for this application in SSC dipoles, a minimum spacing requirement has also been identified. Ghosh and Sampson [14] have shown that a filament spacing of about 0.4-0.5 μm is required to avoid electrical coupling of the filaments at the SSC injection field of 0.3T. This coupling is believed to originate from a proximity effect and may be enhanced in this case due to the high purity copper matrix (the high purity is maintained by the diffusion barrier). Thus, filament spacing must be chosen to satisfy both the mechanical stability and the electrical requirements; we conclude from this study that a value of spacing to filament diameter of about 0.15 is appropriate.

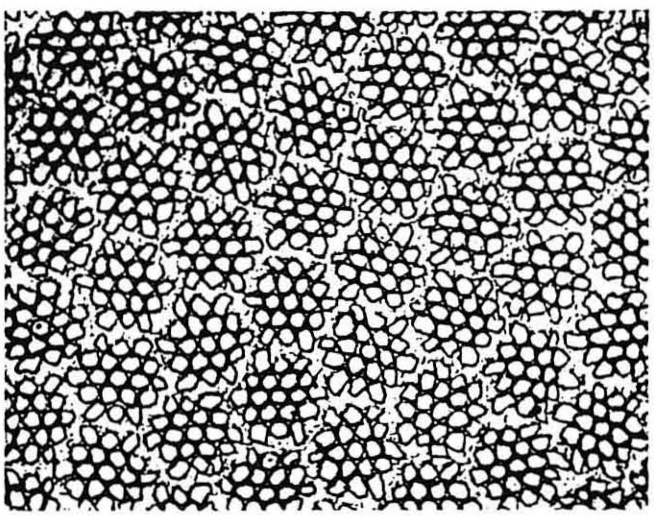

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Fig. 3. Micrograph of Billet 5914-221 Showing Filament Sausaging at Edge of 19 Filament Bundles.

With regard to the evaluation of drawability, initial samples from all billets, except the 5940-5941 series, showed some problems. Several different sources for these drawing problems have been identified and eliminated in subsequent processing. Copper-to-copper bonding problems were present in portions of billets 5914-221, 5914-222, LBL-1, and the hydrostatic extrusion billets. The problem is due to a combination of the large interfacial surface area present in these billets, and low extrusion temperatures employed. The problem was most severe for the hydrostatic billets which were heated to 200°C and extruded at a rather low ratio area reduction of 10 (exit temperature was about 450-500°C).An attempt was made to remove these defects by hot isostatic pressing the extruded rods at a 25 mm diameter; results showed a partial improvement, but some unbonded areas remained. Subsequent attempts to draw material with these defects resulted in wire breakage at the fine wire sizes. These studies confirm that a high extrusion temperature, or a pre-extrusion bonding step, is desirable for achieving high product yields.

After the portions of the billets showing bonding problems were set aside, additional drawing problems were encountered. Experiments were performed in which the drawing die parameters were changed and drawability improved dramatically. The tentative conclusion is that the high $J_c$ fine filament composites develop a high tensile strength and thus require somewhat different processing than more conventional NbTi composites. After the die changes were made, both billet LBL-2 and billet 5914-221 yielded over 80% of the material in lengths greater than 3,000 m; billet 5940-21, produced after these problems were identified and corrected, yielded one length over 12,000 m length and over 85% of the material in lengths over 3,000 m.

## Cable Produced from Fine Filament NbTi

A series of 23-strand and 30-strand cables were made to SSC specifications from the strands described above. When compared with cables made to the same specifications from strands with 18-20 μm filament size, these cables showed two primary differences. First, the amount of degradation in critical current which occurred during cabling was reduced from typically 10-15% for the large filament material to 0-8% from the fine filament material. Second, the strands were more difficult to cable and showed some tendency to protrude from the cable when the cable was bent around a small diameter under light tension. In order to present this information in a more quantitative manner, we have developed a springback test for assessing the cabling ease of various strands. Results indicate that the high $J_c$ fine filament strands have a relatively high springback value. Several experiments are underway to reduce this springback. First, we find that cold-worked strands exhibit less springback than strands in which the Cu matrix is annealed, so we are making cables with cold worked strands for evaluation. Second, we are studying a modified heat treatment sequence with less cold work in the final stage to see if such processing will produce a high $J_c$ strand with less springback.

## Conclusions

1. The use of diffusion barriers appears to be a reliable and cost effective modification to conventional fabrication for applications where fine filaments are required.

2. The large single stack approach to billet assembly yields the best product for the SSC specification of 5 μm filaments in 0.6 to 0.8 mm diameter wires. For finer filaments or other applications requiring a larger number of filaments, it may be necessary to use a double stacking approach. If so, the largest feasible number of elements should be stacked together in order to minimize the number of sausaged filaments which occur at the perimeter of the stack.

3. Fine filament strands can be produced to the SSC specification of 2750 A/mm$^2$ in a reliable and cost effective manner.

4. Cable made from high $J_c$ fine filament strands shows decreased degradation, but a greater springback tendency, than cable made from strands with 20 μm diameter filaments and $J_c$ = 2400 A/mm$^2$ at 5T.

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