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Magnetization Studies of High Jc Nb3Sn Strands

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Abstract— Magnetization measurements have been made on several high J_c Nb_3Sn strands fabricated by different internal-Sn designs. In general these conductors have high magnetization at low fields, often exhibiting flux-jumps that are characteristic of large superconductor diameter. The effective filament size d_{eff} is approximately the size of the sub-element because the filament pack within each sub-element is fully coupled. Dividing the filament pack of the sub-element by adding Ta is effective for reducing d_{eff} and magnetization instability. But, some residual coupling across the dividers seems to remain below 6 K, perhaps due to Ta_3Sn. Implications for accelerator magnets are discussed.

***Index Terms*—Effective filament diameter, magnetization measurement, niobium-tin compounds, superconducting filaments and wires**

INTRODUCTION

THE requirements of high-field magnets (12-16 T) suitable for high energy particle accelerators has pushed up the critical current density J_c of Nb_3Sn wires. At present this limit is about 3000 A/mm^2 within the non-copper region of the wire at 12 T and 4.2 K. Oxford Instruments - Superconducting Technology (OI-ST) has been at the forefront of development, achieving this record J_c in internal-Sn strand designs using the restacked-rod process (RRP) [1]. Earlier OI-ST also developed high J_c (>2000 A/mm^2) using the patented Modified Jelly Roll (MJR) method, which employs a coiled Nb mesh instead of extruded Nb rods [2]. In both cases, these high current densities were a result of designing sub-elements with high Nb-alloy and Sn area, while simultaneously reducing the Cu content to minimal levels and surrounding each sub-element with its own Nb diffusion barrier. A side-effect of this design is the unavoidable coalescence of the Nb filaments during reaction to form a continuous region of Nb_3Sn. In addition, the barrier is allowed to react, adding a second continuous current-carrying region. Figure 1 shows the reacted sub-elements of a RRP strand at 0.8 mm diameter, which resemble continuous tubes. Furthermore, depending on the duration of reaction at 650-700 °C, and in sections where the barrier has thinned during wire-drawing, the reaction can penetrate through to the copper outside the barrier. All of these features contrast with those of lower-J_c (~750 A/mm^2) low-loss wires developed for fusion magnets (*e.g.*, ITER), where the Nb and Sn content are kept low and the diffusion barrier is usually Ta.

The increase in J_c has thus come at the expense of large superconductor dimension. With the number of sub-elements N = 54, and with 50% copper stabilizer, a typical sub-element diameter d_N is ~80 μm. This leads to magnetization values that are two orders of magnitude higher than for typical Nb-Ti accelerator magnet conductors, which may produce unacceptably large error fields. In a separate paper we show that the flux-jumps lead to a "magnetic" instability at low fields in these strands carrying transport currents [3].

In this paper we describe some magnetization measurements of internal-Sn strands produced by the MJR and RRP process, and explore one option of reducing the magnetization by incorporating Ta to sub-divide the tube-like reacted superconductor.

I. SAMPLE PREPARATION AND MEASUREMENT PROCEDURES

Samples of wire ~ 30 cm long were reacted in vacuum. All samples were given the same initial steps of 48 h at 210 °C followed by 48 h at 400 °C. Final reaction temperatures were 665 °C +/- 10 °C for times ranging from 72-180 h, the temperature varying downward from the reference thermocouple with higher mass loaded into the furnace. At the same time, samples for critical current (I_c) measurements were reacted separately, as described elsewhere [4]. After reaction, a hexagonal bundle of 7 wires were potted in epoxy and cut using a diamond saw to a length of 6 mm. A typical wire twist pitch used to make these 6-around-1 bundles was 13 mm. Note that after reaction the diameters D of all the strands increased by approximately 5-6%. However, J_c is calculated using the un-reacted strand diameter and non-copper fraction.

Magnetization and susceptibility measurements were made with a commercial SQUID magnetometer with the field

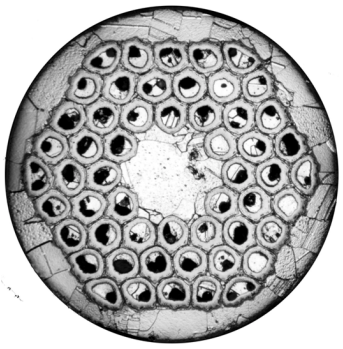

Fig. 1 A cross section of a reacted Nb_3Sn internal-Sn strand with J_c of ~3000 A/mm^2. Rings of Nb_3Sn surround the former location of solid tin. Grain boundaries in the copper matrix are also evident.

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TABLE I
STRAND PARAMETERS

Sample	Billet	Final HT	ΔM (3 T) kA/m	Non-Cu %	D mm	N	d_N μm	J_c(12 T) A/mm^2	J_e(12 T) A/mm^2	J_e(3 T) A/mm^2	d_{eff} μm
A	MJR 202	675°C / 72h	173.6	38.5	0.80	42	77	1948	729	4027	102
B	MJR 163	675°C / 180h	177.6	39.5	0.80	42	78	1812	688	3881	108
C	RRP 7054	675°C / 180h	254.9	50.0	0.70	54	68	2655	1266	5797	104
D	RRP 7054	665°C / 72h	273.1	50.0	0.70	54	68	2899	1449	7045	91
E	RRP 7260	665°C / 72h	124.7	51.2	0.90	54	88	1966	1007	5555	53
F	RRP 7260	675°C / 180h	103.6	51.2	0.90	54	88	2046	1004	5783	42
G	RRP 7261	665°C / 72h	313.4	51.7	0.90	54	88	2320	1148	6858	108
H	RRP 7261	675°C / 180h	305.1	51.7	0.90	54	88	2491	1220	72886	99

transverse to the wire bundle. The background solenoid (maximum field 5 T) was in persistent mode during the measurement and field steps are made with no overshoot. Hysteresis loops were acquired at 4.5 K using a typical field step of 0.1 T. Widths of hysteresis loops ΔM at 3 T field were calculated using the total wire volume. The susceptibility of the sample was measured while warming in a 10 mT field after having been cooled to 4.5 K in zero-field.

Critical current measurements were made at 8-11.5 T at BNL and at higher fields at OI-ST. Besides the 12 T J_c for the non-Cu region, we also determine the "engineering" current density J_e as the equivalent current density within the total wire cross-section. We extrapolate using an exponential fit to estimate J_e at the 3 T fields where ΔM was determined, and these values of J_e and ΔM were used to calculate the effective filament diameter d_{eff} [5] from the critical state model:

$$d_{eff} = (3\pi/4)\Delta M / J_e \quad (1)$$

Note that d_{eff} is only a representation of the magnetization and is used to gauge the multifilamentary nature of the composite. For internal-Sn Nb_3Sn wires, this d_{eff} is always larger than the individual filament diameter prior to reaction, which is indicative of filament bridging [5-7]. In the present case, where the filaments coalesce into a tube of superconductor, it is comparable to the sub-element diameter d_N.

II. MEASUREMENTS

Table I presents a summary of the different wires that were measured and the results that were obtained. Samples A and B have the same design features, whereas sample C and D are the newer high J_c RRP design. Samples D and E are from a billet with Ta-dividers, G and H are from a control billet without the dividers, and in these billets the tin-content is reduced to match the niobium content in the filament pack. The longer reactions times are usually used by OS-IT to optimize J_c. However, recent studies have shown that comparable J_c can be obtained with shorter times. This is discussed in subsequent sections.

A. MJR-Type wire

Figure 2 shows the susceptibility of wires from two MJR billets with different reaction times. The shoulder before the transition at 9 K shows that for longer times the Nb-barrier is more fully reacted. For the long reaction times, the barrier, which is not always uniform, can be breached where it is thin, allowing Sn to leak into the stabilizing Cu and resulting in a much lower *RRR* (the ratio of resistance at 295 K to that at 18 K). For instance, sample B has an *RRR* of 3 compared to 61 for sample A. However, the magnetization of the two wires is fairly similar, as shown in Fig. 3. A common feature of all the wires is the observation of flux jumps at low fields [8-10], which in Fig. 3 is seen below 1 T. Note that sample A is cycled from positive to negative fields, whereas sample B is only

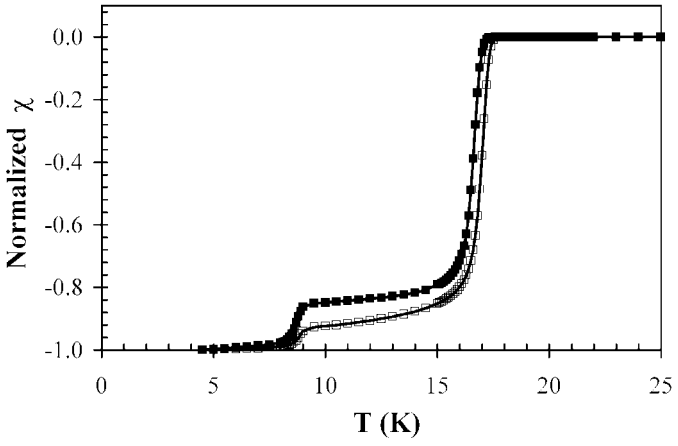

Fig. 2 Plot shows the normalized susceptibility versus temperature for samples A (triangles) and B (filled circles). The 9 K transition is that of the un-reacted Nb-barrier.

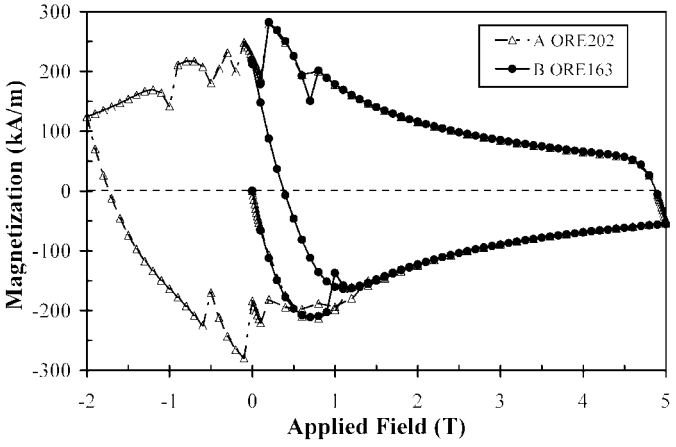

Fig. 3 Volume magnetization of sample A and B at 4.5 K. Drop in magnetization is an indication of flux-jump. Since measurements are taken at intervals, the extent of the drop is hidden by the re-magnetization after the flux-jump.

cycled through positive fields as a conductor in a magnet would be. This can produce different flux-jump behavior.

B. RRP-Type wire

The 0.7mm diameter RRP wires from billet 7054 have a higher percentage of Nb and Sn in the sub-element as compared to the MJR wires, as discussed earlier. Consequently the J_c at 12 T is also higher, and larger magnetization and flux-jump instabilities up to 1.5 T are found. In this case, reaction times of 72 h were sufficient to fully react the barrier with the wire having a J_c(12 T) of 2900 A/mm^2. For longer times, the J_c and the *RRR* of the wire drops. Fig. 4 shows the magnetization of the two wires from billet 7054.

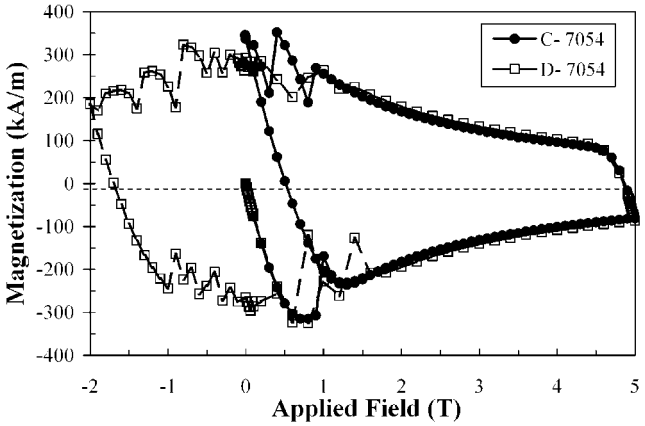

Fig. 4. Plot of magnetization at 4.5 K for samples C and D.

C. RRP-Type wires with Ta divider

One way to reduce magnetization and hence d_{eff}, in the strand is to introduce non-superconducting internal barriers to divide the filament pack in the sub-element. Zeitlin *et al*. have used Nb60wt%Ta fins to separate the Nb filament array with some success [11]. In OI-ST's innovative strand design (Billet 7260), copper clad Ta rods were used to split the filament pack into six approximately rectangular regions, and Ta sheet was placed against the Nb-barrier to prevent it from reacting and forming a continuous ring of Nb_3Sn, as shown in Fig. 5. Light microscopy analyses, as exemplified in Fig. 5, indicated that the Ta filament spokes indeed seemed to divide the reacted Nb_3Sn area, while the Ta sheet seemed to likewise be effective for preventing barrier reaction.

This scheme appears to have been quite successful at achieving the intended electromagnetic changes as well: The magnetization of samples F and H, which were reacted for 180 h at 675 °C and should have full coupling if it is to emerge, are compared in Fig. 6. This shows an obvious reduction in the overall magnetization and the field at which flux jumps appear. In Figs. 7 it is obvious that this suppression occurs throughout the superconducting state, indicating that indeed the length scale for the dimension of the superconductor has been reduced closer to the adiabatic threshold. In Table I, both samples E and F exhibit clear reduction in the value of d_{eff} over their counterparts, samples G and H respectively.

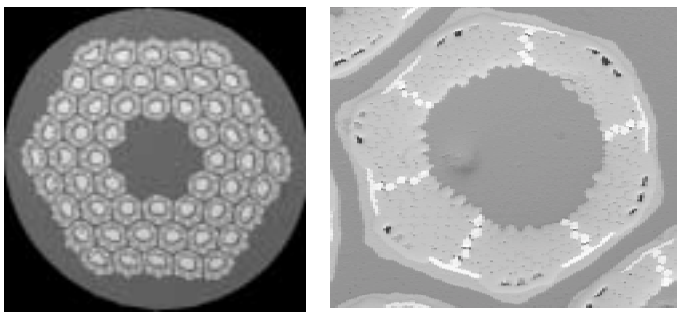

Fig. 5 Photomicrographs of un-reacted 0.8 mm wire from billet 7260. A reacted sub-element is shown at the right

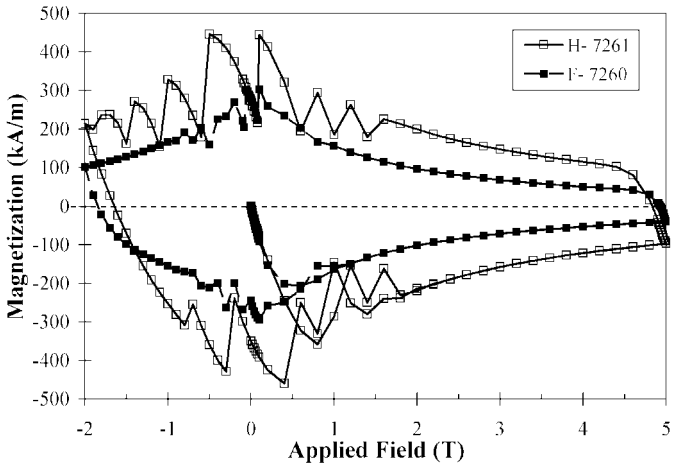

Fig. 6. Volume magnetization of samples F and H as a function of field at 4.5 K. As magnetization decreases the incidence of flux-jumping also decreases.

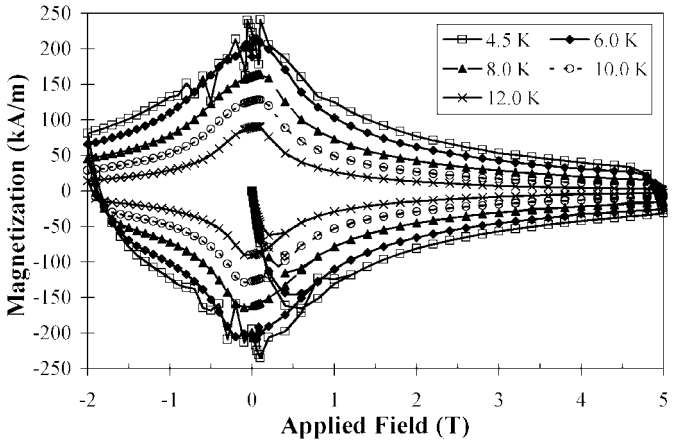

Fig. 7. A plot of magnetization vs. field for sample F at different temperatures. No flux jumps are observed above 6 K.

One consequence of introducing dividers and barriers is that they take up part of the non-copper area that could be used for Nb_3Sn, and hence the J_c of the composite will be lower. This assumes of course that the critical current density within the Nb_3Sn itself is maintained at a constant level. Compared to a control billet (7261) made without these modifications, the expected J_c reduction for billet 7260 is ~15%, as indicated in Table I for both final reactions studied.

Sumption *et al*. [12] have calculated the effect of sub-divisions on the magnetization of a superconducting cylinder. These calculations estimate that the magnetization for wires

from 7260 would be a factor of 5 lower than that of wires from billet 7261. However, while our data show a lower magnetization for billet 7260, the reduction in magnetization at 4.5 K is only a factor of 3. We speculate that the divided Nb_3Sn filament packs are still coupled by narrow regions of superconductor with a lower critical temperature T_c. Such a region should produce an extra contribution to the magnetization, but might not contribute to the transport J_c. In particular, Ta_3Sn has a T_c of ~7 K and an upper critical field of ~5 T at 4.5 K [13].

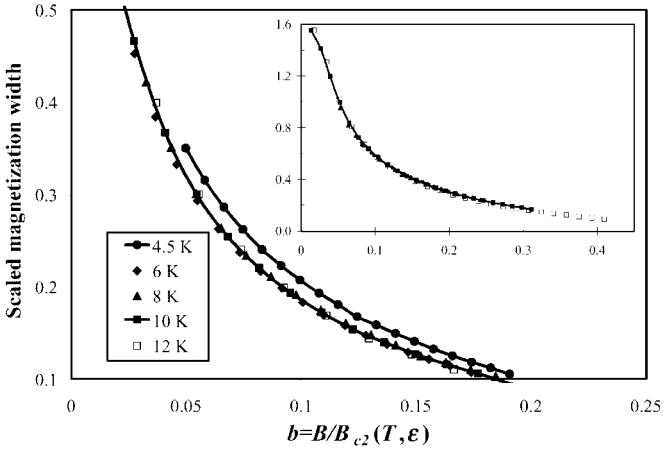

Fig. 8 Plot of the scaled magnetization width for sample F (Ta-divided) as a function of the reduced field *b*. Inset shows the same plot for sample H (from the control billet).

To explore this possibility, the magnetization was measured at 6, 8, 10, and 12 K. This is shown in Fig. 7 for sample F. From these measurements, we scale ΔM ($\propto J_c$) as a function of reduced field $b=B/B_{c2}(T,\varepsilon)$ at the different reduced temperatures $t = T/T_c$ using Summer's formulation [14]:

$$\Delta M_S = C(\Delta M)B_{C2}(T,\varepsilon)^{1/2}(1-t^2)^2 \ . \qquad (2)$$

Here B_{c2} is the upper critical field, expressed with its temperature T and strain ε dependence, B is the applied field, $t=T/T_{c0}(\varepsilon)$, $T_{c0}(\varepsilon)$ is the critical temperature in zero field, and C is a scaling coefficient independent of temperature and field. The following parameters were used: strain $\varepsilon = -0.0016$, B_{c2} = 28.5 T, and T_{c0} = 17.8 K for Sample F and 18.3 K for sample H, respectively. The result of this scaling analysis is shown in Fig. 8. Notice that the data set at 4.5 K lie off of the scaling curve, which is evidence that an additional component of magnetization contributes to those data. This could indeed be due to Ta_3Sn. By contrast, sample H from billet 7261, which received the same reaction but does not incorporate tantalum, shows good scaling with temperature for magnetization widths at 4.5, 6.0, 8.0, 10.0, and 12.0 K (inset in Fig. 8).

III. Conclusion

In present Nb_3Sn strand designs, high J_c has been achieved at the expense of large magnetization and flux-jump instability. This magnetic instability can, at low fields, give rise to unpredictable error fields in magnets, hence for accelerator high field magnets it is desirable to have d_{eff} less than ~40 μm. Moreover, the tendency to initiate flux jumps at low fields makes those sections of magnets prone to quench, even though the operating current might be well below the critical current in the high-field regions of the magnet.

The use of Ta dividers is effective for reducing the magnetization in these strands. However at 4.5 K, there appears to be some residual coupling of the divided filament sections, which could be due to Ta_3Sn. This leads to higher than expected magnetization there. Otherwise, the divided strands produce the desired reduction in superconductor dimension. It is possible that with further refinements of this design, strands could be made that meet the 40 μm d_{eff} goal. It is also significant that J_c too has been reduced due to the loss of Nb_3Sn area. Hence R&D should be made to further increase the critical current density of the Nb_3Sn layer itself.

Acknowledgment

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