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Heat Treatment Optimization of Rutherford Cables for a 15 T Nb_3Sn Dipole Demonstrator

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***Abstract*— FNAL has been developing a 15 T Nb_3Sn dipole demonstrator for a future Very High Energy pp Collider based on an optimized 60-mm aperture 4-layer "cos-theta" coil. To increase magnet efficiency, the coil was graded by using two cables with same 15 mm width and different thicknesses made of two different Restacked Rod Process (RRP®) wires. Due to the non-uniform field distribution in dipole coils the maximum field in the inner coil will reach 15-16 T, whereas the maximum field in the outer coil is 12-13 T. In preparation for the 15 T dipole coil reaction, heat treatment studies were performed on strands extracted from these cables with the goal of achieving the best coil performance in the corresponding magnetic fields. In particular, the effect of maximum temperature and time on the cable critical current was studied to take into account actual variations of these parameters during coil reaction. In parallel and in collaboration with OST, development was performed on optimizing Nb_3Sn RRP® wire design and layout.**

***Index Terms*— Accelerator magnet, critical current density, Nb_3Sn strand, Rutherford cable.**

I. INTRODUCTION

GLOBAL interest in the last few years for a Hadron Collider (HC) with energy above the LHC reach for High Energy Physics research [1]-[3] has produced a world revival in Nb_3Sn. A ~100 TeV center of mass energy machine in a ~100 km tunnel requires dipoles with a nominal operation field of 15-16 T and appropriate operation margins, and can presently be obtained only with the Nb_3Sn technology. Record fields in accelerator magnets (i.e. with beam aperture) were so far obtained at LBNL in 1997 (13.5 T, cos-theta D20) [4] and 2008 (13.8 T, block type HD2) [5]. Surpassing the 14 Tesla brick wall is a crucial goal and inherent demonstration of innovation, and a number of laboratories in the world have established this goal.

FNAL has started the development of a 15 T Nb_3Sn dipole demonstrator based on an optimized "cos-theta" coil design with 60-aperture [6], [7]. A 4-layer coil design was graded by using two cables with the same 15 mm width and different thicknesses. The cable in the two innermost layers has 28 strands 1.0 mm in diameter and the cable in the two outermost layers has 40 strands 0.7 mm in diameter [8]. These two cables were optimized using state-of-the-art Restacked Rod Process (RRP®) wires.

For cost-effective 15-16 T accelerator magnets, the critical current density J_c(15T,4.2K) of commercial Nb_3Sn composite wires has to be pushed from the present state-of-the-art for RRP® wires of ~1,650 A/mm^2 to ~2,000 A/mm^2 [9], [10]. Only so much improvement can be obtained through heat treatment (HT) optimization. Further development was therefore carried out in collaboration with Oxford Instruments - Superconducting Technology (OST), which produced three R&D billets to try and optimize design and layout parameters of their trademarked RRP® process.

To evaluate results from the HT studies for the 15 T dipole demonstrator, critical current was measured for strands extracted from the existing cables. Preliminary results from OST are also presented on the outcome of RRP® wire R&D. This paper summarizes the results obtained in these studies.

II. STRAND AND CABLE SAMPLE PARAMETERS

A. Strand and Cable Description

Table I shows parameters of the RRP® wires of 0.7 mm and 1.0 mm size produced by OST and used in the Rutherford cables for the 15 T dipole outer and inner layer respectively. The wires denoted as RRP1 and RRP2 have a 108/127 and 150/169 stack design respectively. This notation represents the number of superconducting (SC) bundles within the billet layout over the total number of SC and Cu restacks. D_S is the equivalent subelement diameter calculated in the approximation of round instead of hexagonal geometry. The final HT steps shown in Table I are those used by OST to obtain the data shown. All the wires also have extra Cu between the subelements. Pictures of the RRP® wire cross sections are shown in Fig. 1.

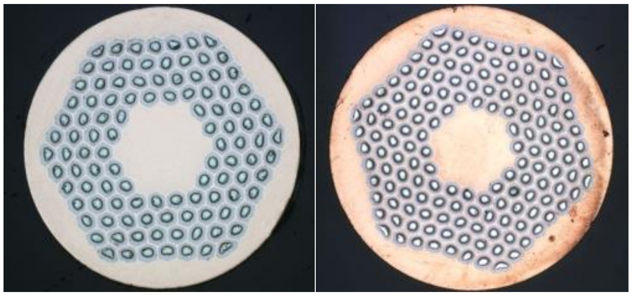

Fig. 1. Cross sections of the 0.7 mm 108/127 (LEFT) and of the 1 mm 150/169 (RIGHT) RRP® wires.

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TABLE I
STRAND PARAMETERS – OST DATA

Strand ID	RRP1	RRP2
Stack design	108/127	150/169
Ternary element	Ti	Ti
Production year	2012	2014
Diameter *d*, mm	0.7	1.0
I_c (4.2K, 12 T), A	451-490	1,052-1,111
J_c (4.2K, 12 T), A/mm^2	2,560-2,722	2,597-2,710
I_c (4.2K, 15 T), A	229-245	566-619
J_c (4.2K, 15 T), A/mm^2	1,289-1,365	1,395-1,502
D_S, µm	41	58
Twist pitch, mm	14-16	23-24
Cu fraction λ, %	53.2-54.4	47.5-48.4
RRR	101-226	343-374
Final HT step	640ºC/50h	665ºC/50h

The cables for the 15 T Dipole are described in Table II and their cross sections are shown in Fig. 2. They were fabricated in one pass using FNAL cabling machine [11] and included an 11 mm wide stainless steel (SS) core to suppress interstrand eddy currents and obtain the required field quality and ramp rate dependence in magnets.

TABLE II
CABLES FOR DEVELOPMENT OF 15 T DIPOLE

Coil	Cable N x d, mm	RRP® Strand Type	Cable length, m	Cable t_{mid} x w, mm^2	Lay angle, deg.
15 T Dipole Outer Layer	40 x 0.7	RRP1	374	1.251 x 14.71	16.8
15 T Dipole Inner Layer	28 x 1	RRP2	420	1.803 x 14.79	15.5

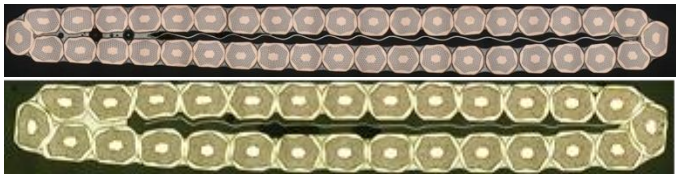

Fig. 2. Cross section of keystoned cable for the 15 T Dipole outer layer (TOP), and for the 15 T inner layer (BOTTOM). Both cables in Figure are ~15 mm wide and include a SS core.

B. Heat Treatments

A three step reaction cycle, which is typical for Internal Tin Nb_3Sn composite wires, was used. For all cables, the first two steps were 72 h at 210°C and 48 h at 400°C unless otherwise specified. Samples were heat treated in a 3-zone controlled tube furnace in argon atmosphere. Two calibrated, sheathed and ungrounded, type-K thermocouples, TC1 and TC2, were mounted in the vicinity of the samples for temperature monitoring. Table III specifies the parameters used in the last HT step for these studies. The column labelled "ST-HT" indicates standard heat treatments.

TABLE III
HEAT TREATMENT NOMINAL PARAMETERS

Strand ID	ST-HT			
RRP1	640ºC/48h	650ºC/48h	660ºC/48h	
RRP2	665ºC/50h	665ºC/100h	670ºC/100h	675ºC/100h

An experiment was also conducted in pre-heat treating the RRP2 wire at ~210°C for 3 days before using it in cables [8]. In this previous study, round wire and extracted strand samples from cables with different packing factors were then heat treated to complete the standard cycle. In order to better understand the results from [8], an accurate damage analysis was performed on cable cross sections (see next section).

C. Microscopy Damage Analysis

For each packing factor [8], data were taken on two to six cable cross sections, depending on consistency of results, in order to ensure reproducibility. Samples were mounted and prepared using standard metallurgical techniques.

The samples were then inspected on a Nikon Eclipse MA200 microscope at magnifications of up to 50X. Breakage of subelements, merging of niobium, and merging of tin were counted as damaged subelements and are included in the chart. Unclear cases of merging were counted as 0.5 damaged subelements. Fig. 3 gives an example of damage from broken and merged subelements. The total number of damaged subelments was then normalized to the number of cross sections of cable inspected.

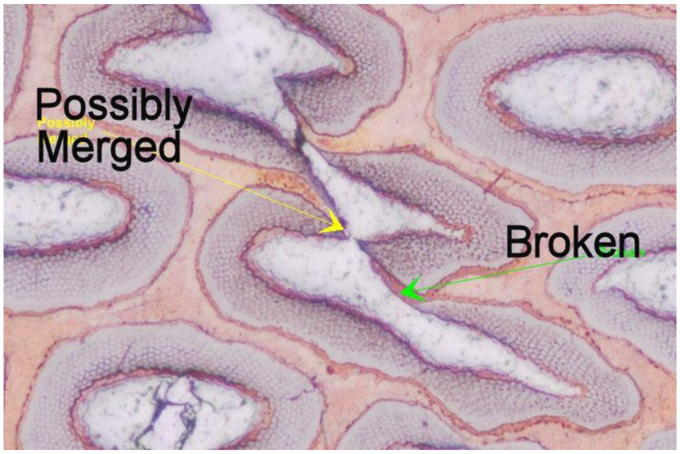

Fig. 3. Examples of damaged subelements in cross section of deformed RRP® wire.

D. Heat Treatment Optimization

Sample preparation and critical current testing techniques used at FNAL for Nb_3Sn wires are detailed in [11], [12]. The Residual Resistivity Ratio (RRR) was measured as the ratio of the wire resistivity at room temperature over its residual resistivity at 20 K. Unless otherwise specified, the results below are shown for extracted strands.

For the RRP1 extracted strand, increasing the last temperature step by 20°C to 660°C with respect to the standard HT produced a ~5% increase of both the J_c(12 T) and the upper critical field B_{c20}, as obtained from fitting the data with a parameterization. The RRR reduction was of about 50%, i.e. to ~90 in value. These results, as averaged over at least 4 billets for each temperature, are shown in Fig. 4 as function of the maximum HT temperature at 48 h. The error bars in the plots represent the error on the mean.

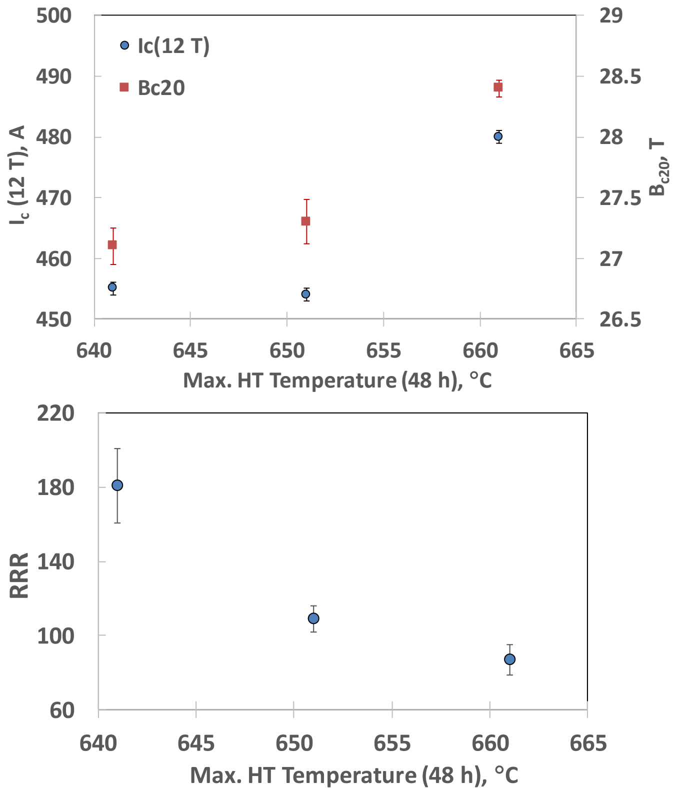

Fig. 4. I_c(12 T) and B_{c20} (TOP), and RRR (BOTTOM) for the RRP1 extracted wire as function of its maximum HT temperature at 48 h. Results are shown as average over at least 4 billets, and the error bars represent the error on the mean.

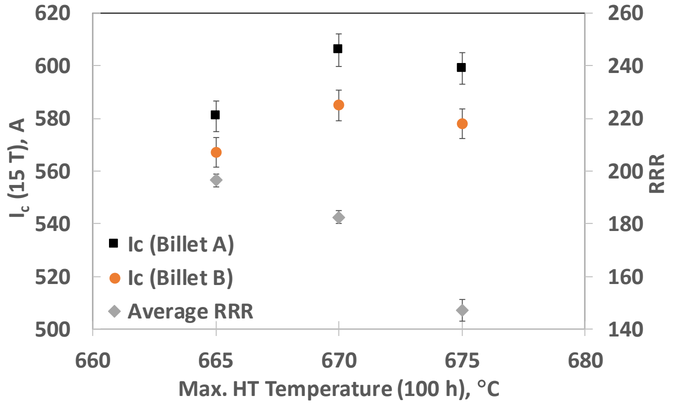

Fig. 5. I_c(15 T) and RRR for the RRP2 extracted wire from two different billets as function of their maximum HT temperature at 100 h. The error bars represent the measurement uncertainty for the I_c and the average on the mean for the RRR.

For the RRP2 extracted wire, doubling the time to 100 h at the standard HT temperature produced a ~5% increase of the J_c(15 T), with a RRR reduction of about 75% to ~200 in value. The upper critical field B_{c20}, as obtained from fitting the data with a parameterization, did not change significantly. The I_c(15 T) further improved for the RRP2 extracted strand when in addition to doubling the time, the temperature was increased. This is shown in Fig. 5, where the I_c(15 T) and the average RRR of the RRP2 extracted wire from two different billets is plotted as function of their maximum HT temperature at 100 h. The error bars in the plot represent the measurement uncertainty for the I_c and the average on the mean for the RRR. An overall improvement in J_c(15 T) of 8 to 9% is presently observed for RRP2 for 100 h at 670°C compared to the standard HT.

E. *Wire Pre-processing*

In [8] it was found that cables made with RRP2 wire that had been pre-heat treated at 210°C before cabling showed less critical current degradation at cable packing factors above 90% than in the RRP2 wire used as-is in the cables. Their RRR was somewhat reduced, but at the nominal *PF* of 87% it was still ~200. To further understand the mechanisms involved, an accurate damage analysis was performed on several cable cross sections. Fig. 6 shows the normalized number of damaged subelements in cable cross sections as function of cable packing factor for as-is and pre-treated RRP2 strands. These results confirm that pre-treating wires at 210°C before using them for cable fabrication does not increase subelement damage. By pre-treating the wire at the lowest temperature step, this method could be therefore considered in the future to reduce the actual HT time of coils, thereby reducing risk and, perhaps coil fabrication cost.

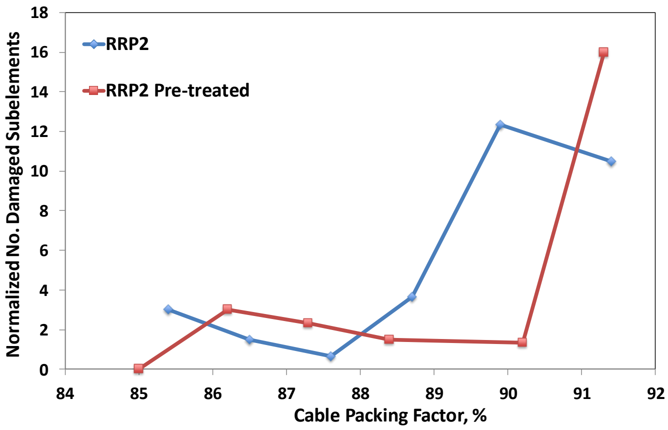

Fig. 6. Normalized number of damaged subelements in cable cross sections as function of cable packing factor [8] for as-is and pre-treated RRP2 strands.

III. Nb_3Sn WIRE R&D WITH INDUSTRY

Short of acting on the inherent flux pinning mechanisms of Nb_3Sn, a method of increasing J_c in superconductors is that of optimizing their layout. A most recent endeavor in this direction was attempted by OST in collaboration with FNAL, to try and push RRP® state of the art average J_c(15T,4.2K) from 1,650 A/mm^2 to 2,000 A/mm^2, for the 169 stack strand at 1.0 mm (D_S~58 μm). Other parameters associated to the present J_c performance in these wires include a RRR typically larger than 200 and a B_{c2}(4 K) of ~26 T.

For this R&D, OST fabricated three restacks from three subelements along two basic subelement modification ideas – chemical and Local Area Ratio (LAR) optimization. The Sn level used was the same for all three billets – “standard” tin, or a Nb to Sn ratio of 3.4:1, which is used for maximum J_c. Wire samples were produced between 0.8 mm and 1.2 mm in diameter.

A. Chemical Optimization

It has been long known that Ta doped Nb_3Sn is optimized at a higher temperature than Ti doped Nb_3Sn (~695°C vs 665°C). In addition, it has been observed that at a fixed lower temperature, e.g. 640°C, Ti doped strand reacts quicker and more completely than Ta doped strand. This property was used to create a subelement where the filaments are Nb with Nb-Ti dopant rods, but the diffusion barrier is Nb-Ta. Such design should allow using the lower temperature, which is best for the Ti doped filaments, as the barrier is expected to react more slowly, resulting in higher RRR for a given D_S or tin concentration. If this occurred, Sn concentration could be increased for higher J_c but with a still acceptable RRR.

B. Local Area Ratio (LAR) Optimization

The present high J_c subelement design has a nominal Cu:Nb monofilament local area ratio (LAR) of ~0.20. This high percentage of Nb means both a high fraction of Nb_3Sn in the non-Cu area, and a relatively high Sn% in the bronze. However, it used to be unclear if the present LAR is the optimal LAR at all wire sizes, and whether the LAR needs to be the same through the subelement. It is known from previous work [13] by the Conductor Development Program [14] that a LAR ~0.14 has too little Cu, causing Sn diffusion problems, excessive dissolution of the subelement, and very low J_c values. It is also known from previous commercial work at OST that the J_c is definitely lower for LAR>0.25. Finally, it is known that with the present LAR value of 0.2, as the subelement size is reduced the J_c is reduced, no matter how aggressive the heat treatment. By adjusting the LAR throughout the subelement, one might have been able to better control the desired properties.

TABLE IV
R&D BILLETS BY OST - OST DATA

Strand ID	RD1	RD2	RD3
Stack design	150/169	150/169	150/169
Ternary element	Ti	Ti	Ti
Production year	2016	2016	2016
Diameter d, mm	0.8 to 1.2	0.8 to 1.2	0.8 to 1.2
I_c (4.2K, 15 T) for 1 mm, A	651 max.	657 max.	665 max.
J_c (4.2K, 15 T) for 1 mm, A/mm^2	1,713 max.	1,684 max.	1,715 max.
D_S, μm	46 to 69	46 to 69	46 to 69
Cu fraction λ, %	50.9-51.6	49.9-50.5	50.5-50.8
B_{c2}(4K) at J_c^{max}, T	26.7	27.0	26.5
RRR at J_c^{max}	161	30	97
Final HT step	680ºC/50h	680ºC/50h	665ºC/100h

For the first billet used for LAR optimization, a homogenous LAR value of 0.23 was used. For the second billet, the LAR values were graded from a maximum of 0.25 in the innermost row of subelements to a minimum of 0.14 in the outermost row. Wire samples between 0.8 mm and 1.2 mm were obtained from the three billets, and underwent extensive heat treatment optimization and characterization studies at OST. Maximum heat treatment temperatures ranged from 650°C to 680°C and durations of the last step ranged from 50 h to 100 h. As preliminary data shown in Table IV indicate, the main result from this R&D endeavor is that, at least for round (i.e. non deformed) wires, there was no major J_c or RRR performance difference seen with any of the subelement redesigns when compared to the standard high J_c subelement design (aside from a RRR problem caused by bad barrier in billet RD2).

These results reinforce the concept that the J_c of state-of-the-art Nb_3Sn wires has plateaued, which is a strong argument for now investing research in improving inherent flux pinning in Nb_3Sn.

IV. CONCLUSION

A 4-layer 15 T Nb_3Sn dipole demonstrator for a future Hadron Collider is being developed at FNAL. The Rutherford cable used in the two innermost layers has 28 strands of 1.0 mm diameter and the cable used in the two outermost layers has 40 strands of 0.7 mm diameter. Heat treatment optimization studies were performed on these cables based on existing Nb_3Sn RRP® wires. The I_c(15T) for the inner cable was improved by 8 to 9 % at 670°C, and the I_c(12T) for the outer cable was improved by ~5% at 660°C, producing in both cases an acceptable RRR.

For large cable compactions, in excess of 90%, pre-treating the wire at 210°C before cabling is feasible.

OST and FNAL have also invested in R&D on layout optimization for state-of-the-art, high J_c RRP® wires. The OST 169-restack conductor within this latter study had an average J_c(4.2K, 16T) ~ 1,300 A/mm^2 and its cost was ~$1,700/kg. This was obtained with a Nb to Sn ratio of 3.4:1, which corresponds to ~53%at. Nb, which is presently the achievable upper limit for Nb content in a wire. Preliminary results indicate that the J_c of Nb_3Sn wires has plateaued. It is clear that to achieve the cost reduction required in magnets for a Hadron Collider, the target increase in J_c can only be achieved by disruptive progress, and that for this reason it is now necessary to invest in research aimed at improving the inherent flux pinning of Nb_3Sn.

ACKNOWLEDGMENT

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