

The Effect of Heat Treatment on the Stability of Nb₃Sn RRP-150/169 Strands

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Abstract— The magnetic stability of superconductor strands and cables is a key issue in the successful building and operation of high field accelerator magnets. In this work, we report the study of a state-of-the-art 0.7 mm Nb₃Sn Restacked-Rod-Process (RRP) strand manufactured by Oxford Instrument Superconductor Technology. This conductor will be used in Rutherford cable for a 15 T Nb₃Sn dipole demonstrator being built at FNAL. Particularly, this study focuses on the impact of varying heat treatment conditions on the stability of the strand. Both the stability against internal flux jumps and external thermal perturbations are studied.

Index Terms— Nb₃Sn, stability, superconducting wires.

I. INTRODUCTION

SUPERCONDUCTING MAGNETS fabricated with multi-filamentary Nb₃Sn strands and cables are the best candidates for high field accelerator magnets in the field region of up to 16 T. A key challenge to Nb₃Sn wires and cables is their magnetic instability [1]-[5], which can significantly depress their current-carrying capability to a fraction of the theoretical limit. The instability of Nb₃Sn composite wires can be attributed to the redistribution of magnetic field inside a single superconducting filament or a strand as a whole (flux jump) induced by a perturbation. It was shown theoretically and confirmed experimentally that small filament size and low RRR of copper matrix are essential to a stable, high- J_c Nb₃Sn composite strand. While there have been considerable amount of experimental and theoretical studies on this topic, they largely focused on some early Nb₃Sn strands. With the development of Nb₃Sn techniques in recent years, it is necessary to continue this work, both experimentally and theoretically, on state-of-the-art conductors.

At Fermi National Accelerator Laboratory (FNAL), the design and fabrication of a 15 T dipole demonstrator magnet is in progress [6], [7]. In this work, we present some experimental study of the stability of Nb₃Sn composite strands that will be used in this magnet. This work is also done jointly with variation of the heat treatment parameters.

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II. EXPERIMENTAL

A. Conductor Description

The Nb₃Sn composite strand used in this study is a 0.7 mm diameter Restacked-Rod-Processed (RRP) strand manufactured by Oxford Instrument Superconducting Technology. It has a 150/169 restacking design with 150 subelements distributed in Cu matrix, and an equivalent subelement diameter D_{eff} of about 38 μ m. This strand will be used in the Rutherford cable for winding the two outmost layers of the 4-layer 15 T dipole magnet [8]. The basic information and a cross section image of the strand are shown in Table I and Fig. 1.

B. Heat Treatment and Sample Preparation

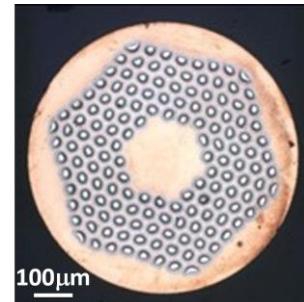


Fig. 1. Optical microscopy image of the Nb₃Sn RRP-150-169 strand cross section.

TABLE I
STRAND INFORMATION

parameter	
Stack architecture	RRP, 150/169
Outer diameter	0.7 mm
Cu fraction λ , %	52%
D_{eff}	38 μ m

To investigate the effect of varying heat treatment conditions on the stability of Nb₃Sn strands, three different heat treatments (HT1, HT2 and HT3) were used. These heat treatments all began with a 48 hours dwelling at 210 $^{\circ}$ C and then a 48 hours dwelling at 400 $^{\circ}$ C. As for the final step, HT1 dwelled 50 hours at 665 $^{\circ}$ C, HT2 50 hours at 645 $^{\circ}$ C and HT3 100 hours at 675 $^{\circ}$ C. All the heat treatments were performed in a 3-zone

furnace tube under flowing argon gas and the temperature deviation was less than 2 °C. HT1 is the standard procedure recommended by the vendor. Samples for I_c measurements were tightly wound on Ti alloy ITER barrels and the samples for residual resistivity ratio (RRR) measurements were treated as straight strands.

C. Measurement Technique

Two types of transport measurements were performed using the barrel samples. The first, $V-I$ tests were done by ramping up sample current in a fixed background magnetic field until a superconducting transition or a premature quench occurred so that the critical current I_c (defined by the 1 $\mu\text{V}/\text{cm}$ criterion) or the quench current $I_q(V-I)$ can be defined. A transition means a smooth voltage-current curve up to current slightly higher than I_c can be measured, whereas a quench means a sudden jump of voltage far beyond the 1 $\mu\text{V}/\text{cm}$ criterion and usually at a current below the expected I_c . The second type, $V-H$ tests, were done by ramping up the background magnetic field from zero on a sample carrying a current I until a quench occurred and the quench current $I_q(V-H)$ was determined as a function of field. In these tests, the sample barrels were covered with Stycast epoxy and immersed in liquid helium bath. The magnetic field was perpendicular to wire axis and the Lorentz force pointed inward toward the barrel.

The critical current density J_c is defined as the critical current I_c over the non-Cu area of a composite strand. B_{c2} was determined by fitting the $I_c(B)$ curve using Summers parameterization described in [11].

The minimum quench energy (MQE) tests were done with similar barrel samples. Quench-inducing heaters made of a carbon-filled epoxy (Ecobond-60L) were installed on strands. This technique has been adopted in similar studies, where the detailed description of heater fabrication technique can be found [9], [10]. A diagram illustrating the heater circuit is shown in Fig. 2. During the test, single 50-500 μs long voltage pulses were generated by a power supply (KEPCO-400) to deposit heat into the sample carrying a transport current I supplied by the main power supply. The heater current used the strand as a return path to the power supply. The DAQ system recorded the voltage across the heater and another resistor of known resistance in series connection with the heater, which was used to calculate the current through the heater. The heat deposited was calculated by integrating the heater voltage and current over the pulse duration. With gradually stepping up in the pulse strength, a separate quench detection/protection system monitoring the voltage across the sample was finally triggered and the main power supply was shut down. The critical heat that triggered the quench protection was defined as the MQE. The typical heater size was 5 mm long and the width of heater was trimmed to be as close to the diameter of the strand as possible to make sure that the majority of the heat was deposited directly on the strand. The resistance of the heater can be roughly adjusted by the amount of epoxy applied. For this study, a heater resistance between 20-100 Ω was found to be most suitable for the MQE level of this conductor and the output

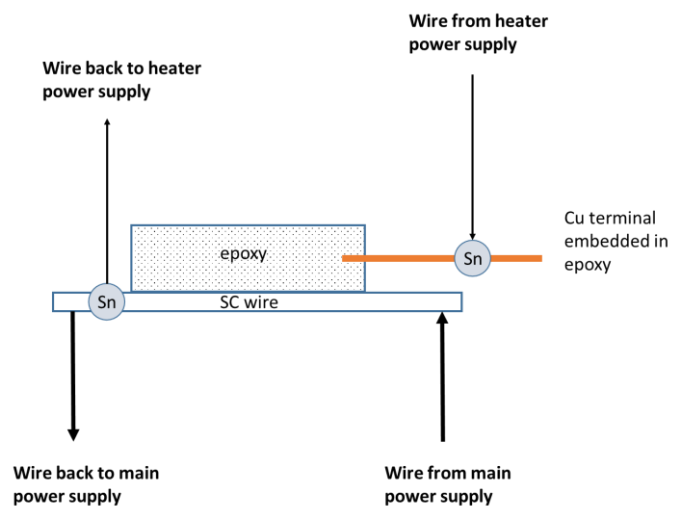


Fig. 2. Circuit of epoxy heater used for MQE study.

capability of the power supply. In experiment, the heater voltage did not exceed 10 V. Under such condition, the heater current is very small comparing with the current from the main power supply.

RRR tests were done on straight strands by measuring their resistance at multiple temperatures in magnetic field both parallel and perpendicular to sample up to 15 T. A 3 A bi-polar current source was used in the test.

III. RESULTS.

A. Effect of varying heat treatment on transport properties

The effect of heat treatment conditions is summarized in Table II. With the results of HT1 as the baseline, HT2 depressed both J_c and the upper critical field B_{c2} but resulted in samples with higher RRR. In contrast, HT3 increased J_c and B_{c2} but degraded RRR.

B. Stability

The results of transport $V-I$ and $V-H$ tests are shown in Fig. 3. Limited by the maximum current limit of 1800 A of the power supply, the cases where neither a transition nor a quench occurred till this limit are labeled as “power supply limit”. In

TABLE II
EFFECTS OF HEAT TREATMENT ON TRANSPORT PROPERTIES

HT#	J_c (4.2 K, 12 T) KA/mm ²	B_{c2} (4.2 K, fitted) T	RRR ($\rho_{300\text{K}}/\rho_{20\text{K}}$, no magnetic field)
HT1	2.64	25.3	190
HT2	2.56	23.7	234
HT3	2.85	26	90

these figures, the fitted $J_c(B)$ dependence is from the model suggested by Summers [11] and the experimental data. In $V-I$ tests, in HT1 samples, premature quenches did not happen until the field was lower than 6 T. On the other hand, premature quenches occurred in HT3 samples at fields below 8 T which is consistent with higher J_c and lower RRR in these samples (see Table II). In $V-H$ tests, the $I_q(V-H)$ was generally lower than

$I_q(V-I)$. For HT1 samples, $I_q(V-H)$ dropped fast in the low field region (0-2 T) and then slowly decreased with field in the medium field region (2-6 T). Comparing with HT1, HT3 samples showed a sharper and larger “dip” of $I_q(V-H)$ in the low field region. On the other hand, HT2 samples with lowest J_c and highest RRR did not show sharp change of slope in the trace of $I_q(V-H)$ versus field.

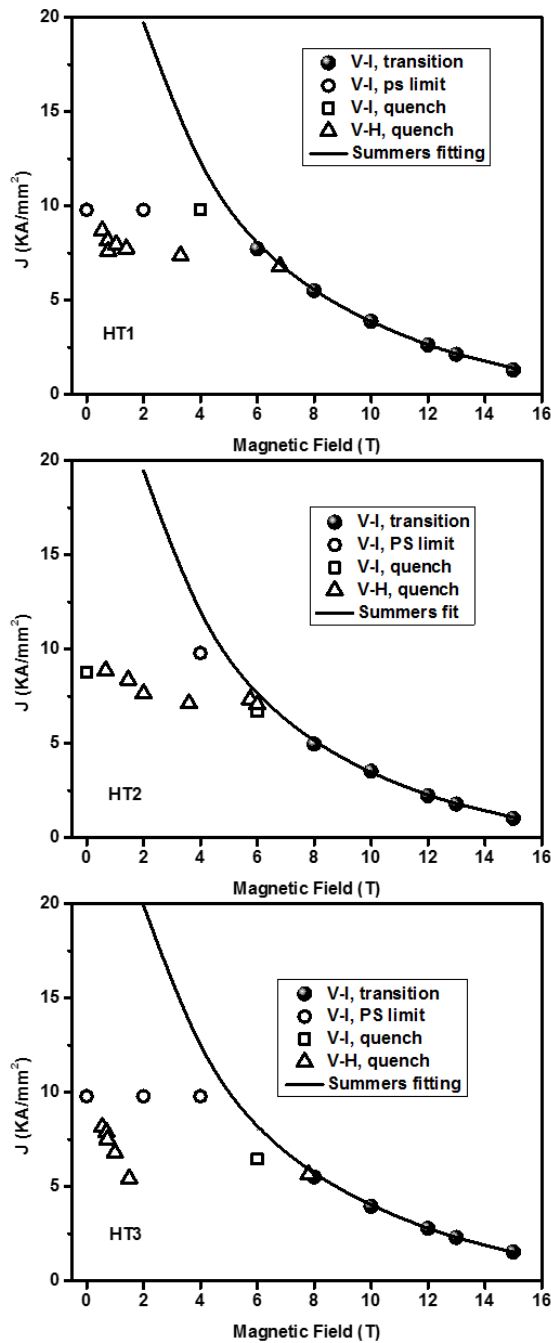


Fig. 3. Transport $V-I$ and $V-H$ results of HT1, HT2 and HT3 samples.

C. RRR

The RRR results with both magnetic field perpendicular and parallel to the wires are shown in Fig. 4. As can be seen,

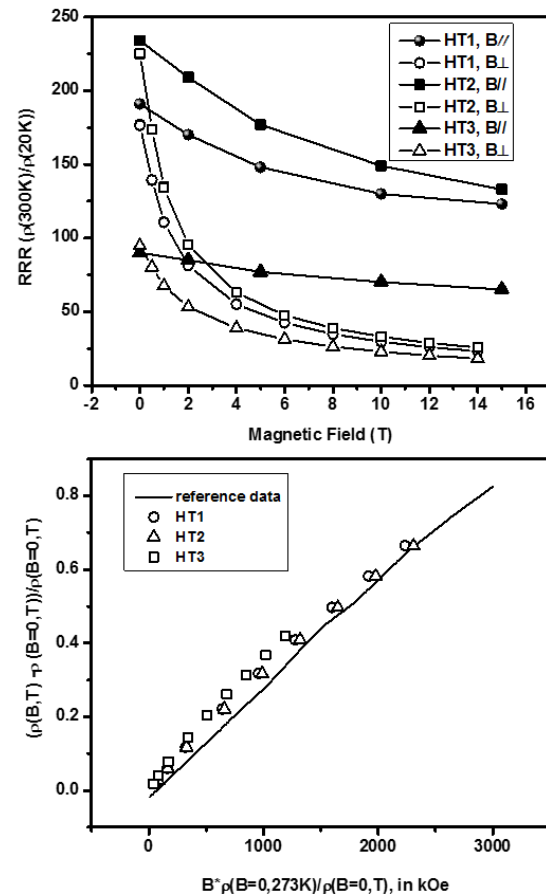


Fig. 4. RRR of HT1, HT2 and HT3 samples. Top: RRR dependence of magnetic field, both the results of field perpendicular and parallel to the wires are shown. Bottom: Kohler's plot. The reference curve is extracted from reference [13].

perpendicular fields have much stronger effect on the dependence of strand RRR on magnetic field. In HT1 and HT2 samples, the RRR data show a stronger dependence on magnetic field. In contrast, the RRR of HT3 samples is less affected by magnetic field. The reduced susceptibility of RRR to magnetic field is a result of copper matrix contamination.

The data measured in the parallel field are also presented as a Kohler's plot [12] and compared with the reported data of Cu from reference [13]. In a Kohler's plot, at temperature T , the relative change of resistivity induced by magnetic field is plotted as a function of the product of magnetic field and the ratio of zero-field resistivity at 273 K and temperature T . The data points from all the three heat treatment samples converge well with the reference curve.

D. MQE study

In the MQE test, we first investigated the effect of pulse duration on the value of MQE results. Fig. 5 shows the results from a HT1 sample at 15 T with a transport current corresponding to 60% I_c . For pulse widths between 50 μ s and

150 μs , pulse duration has very little impact on MQE. As a result, in later studies, we chose 100 μs pulse duration as the standard experimental setup.

Fig. 6 shows the comparison of MQE of HT1 and HT2

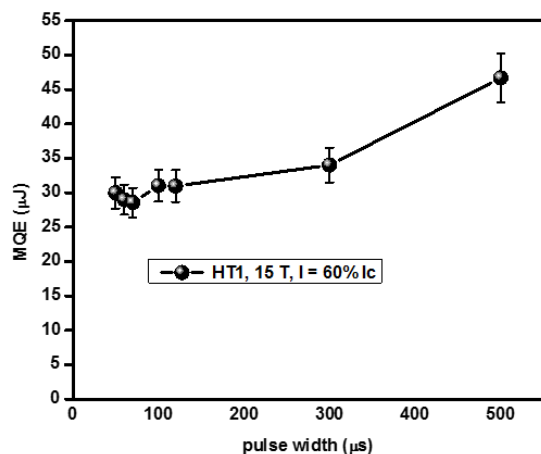


Fig. 5. Effect of pulse duration on the measured minimum quench energy (MQE). The data shown are from a HT1 sample carrying 60% I_c in 15 T. For pulse durations shorter than 150 μs , the MQE was almost independent of pulse duration.

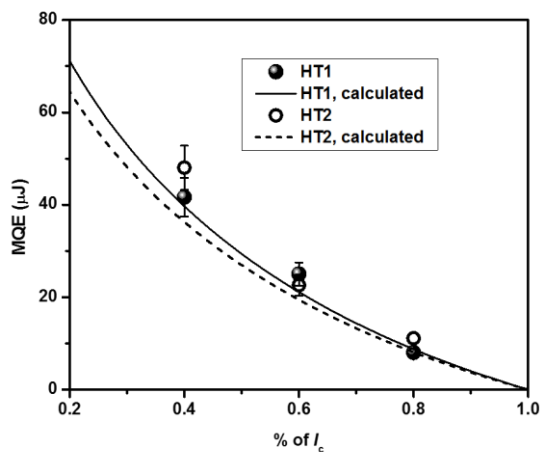


Fig. 6. Minimum quench energy (MQE) of HT1 and HT2 samples in 12 T field.

samples in 12 T field. The error bar of the data points is because of the finite step used in increasing heat pulse strength and the uncertainty of measurement of the dimension of the epoxy heater. The experimental data points are compared with a theoretical calculation. The MQEs are calculated by integrating the specific heat of the composite strand section covered by the heater over the temperature margin, which is a function of operating current and applied field. I_c and B_{c2} data are obtained from the fitting of the transport I_c measurements using [11]. The specific heat data of Nb_3Sn and copper are from reference [14]. The experimental data agree well with calculation. On the other hand, these data do not show a very strong impact on MQE from varying the heat treatment procedure.

IV. DISCUSSION

Our results showed a trade-off between the improvement of transport property by varying heat treatment procedure and the strand parameters responsible for its stability. Within the parameter space explored by this work, higher temperature and longer dwelling time in the final stage of the heat treatment generally led to higher J_c and B_{c2} but at a cost of lower RRR. From the viewpoint of understanding stability issue of strands, varying heat treatment conditions can be an effective method to systematically prepare sample sets with various levels of stability.

We also observed a clear correlation between the strand stability and RRR values. It should be pointed out that, in this study, even the least stable samples had a RRR > 50, much higher than that of the early powder-in-tube (PIT) and modified jelly roll (MJR) strands [15]. Fitting this dataset with adiabatic model (*e.g.* the one suggested in reference [3]) did not show very satisfying results. This means that the dynamic heat transfer should be included to model the instability of these strands. Simulation work that considered the effect of RRR [16] suggested that the beneficial effect of improving RRR on stability is most obvious for strands with RRR < 100 but tend to saturate with RRR > 100. This is consistent with the degraded stability observed in HT3 samples that had a RRR about 90. Another key parameter in modeling is the value of D_{eff} . Though D_{eff} based on geometric approximation has been widely adopted, it still needs verification by means of microstructure analysis and magnetization study.

V. CONCLUSION

By varying the standard heat treatment procedure, we showed a correlation between heat treatment parameters, transport properties and stability of the resulted strands. The MQE of strands did not show a clear dependence on heat treatment conditions. Similar experimental work will continue with newly-developed conductor batches, such as thicker RRP strands of 1.2 and 1.5 mm diameter.

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