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Effects of Temperature Ramp Rate during Heat Treatment on Hysteresis Loss and Critical Current Density of Internal Tin Processed Wires

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Abstract: It has been shown that temperature ramp rates utilized in heat treatment schedules for internal tin processed Nb<sub>3</sub>Sn wires substantially influence both hysteresis loss and critical current density  $J_c$  of the wires, i.e. a slow ramp rate (e.g. 6°C/h) favors a higher  $J_c$  while a fast ramp (e.g. 60°C/h) results in a low hysteresis loss of the wire.

## 1. INTRODUCTION

Multifilamentary Nb<sub>3</sub>Sn wires are one of the critical components for a successful construction of a magnetic fusion reactor -- providing the required high magnetic fields for confinement of high temperature plasma. In the current international effort for demonstration of the feasibility of the reactor (International Toroidal Engineering Reactor, ITER), very stringent requirements are specified for both the hysteresis loss and the critical current density (≤600 mJ/cc and ≥700 A/mm<sup>2</sup>, respectively) of Nb<sub>3</sub>Sn wires used for the central coil of the reactor. Although it has been frequently demonstrated that these requirements can be met in short specimens, it has also been shown that fabricating wires meeting the specification consistently in long lengths is not trivial. Thus, in order to improve consistency in the properties of the wires, a systematic investigation was carried out to study the effects of the temperature ramp rate to the final reaction temperature on the losses and the current densities. Here, we briefly report the results of this study showing that (1) a slow ramp causes an outward motion of the inner rows of the Nb filaments within a subelement resulting in enhanced bridging among the filaments and thus in increased hysteresis losses, and (2) that while a fast ramp avoids or minimizes this movement of the filaments, it results in a nonuniform Sn distribution at the initial stage of Nb<sub>3</sub>Sn formation possibly leading to lower critical current densities.

### 2. EXPERIMENTAL PROCEDURE

Two 19-subelement internal tin processes Nb<sub>3</sub>Sn wires were used for this study. These were fabricated at Advanced Superconductors, Inc. of Intermagnetic General Corp. for the U.S. ITER program. Each subelement consists of 4 rings of ~3 µm filaments (172 in total) in a Cu matrix surrounding a tin core (See Figs. 1 and 2). A detail of the wire geometry and fabrication process is presented in Ref. 1. The heat treatments of the wires for hysteresis and critical current measurements were performed on the U.S. standard heat treatment forms and were done under argon or in vacuum. For metallography of the wire cross sections, long U shaped wires, which were encapsulated in quartz tubing, were used and these were taken out of a furnace at various stages of the temperature ramps to examine the reaction kinetics of the elemental components in the wires.

The hysteresis losses were measured using a SQUID magnetometer for one half cycle of a  $\pm 3$  T cycle at 4.5 K. Critical current densities of the wires were determined for H = 12 T with an electric field criteria of  $E_c = 0.1~\mu\text{V/cm}$  at National Institute of Standards and Technology, Plasma Fusion Center, Massachusetts Institute of Technology, and Applied Superconductivity Center, University of Wisconsin.

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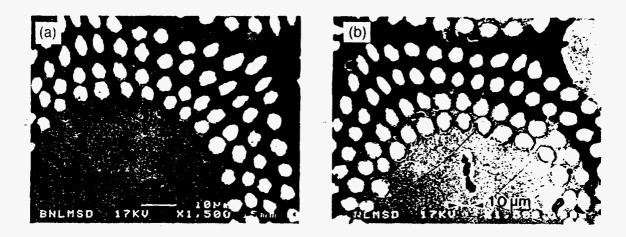


Fig. 1. Shows the difference in the inner filament motion at 475°C depending on the temperature ramp rate (a) 60°C/h and (b) 6°C/h.

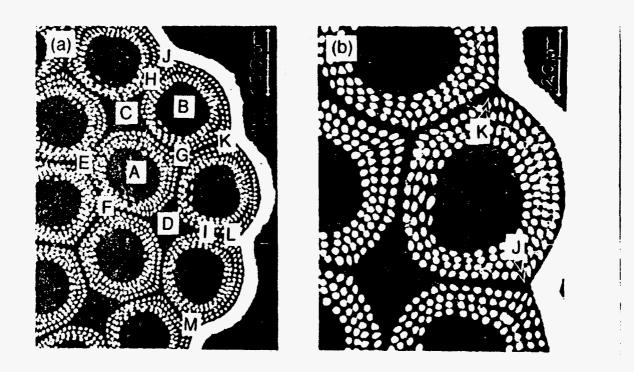


Fig. 2. (a) A cross section of the wire at 600°C indicating the locations of the areas where the compositional analyses were made. (b) A back electron scattering image of a wire with 60°C/h illustrating non-uniformity in the Sn content.

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# 3. RESULTS AND DISCUSSION

The values of the hysteresis losses and the critical current densities,  $J_c$ , for a number of heat treatment schedules (HTS) are tabulated in Table I. for the two wires. Although the number of experiments for each category of HTS is limited, a clear trend emerges from the table in the dependence of the values of  $J_c$  and particularly the hysteresis loss on the temperature ramp rate. For example, as seen in the wire samples, A:3,4,5 holding at a temperature of 375°C for 24 h is particularly deleterious for the losses unless this is followed by a very rapid ramp (~1000°C/h) as illustrated by wires A:1,2. On the other hand, the values of  $J_c$  is the highest when a very slow temperature ramp (6°C/h) is employed as illustrated in the wires A:6 and B:3. Since hysteresis loss is proportional to both the effective filament size and  $J_c$ , the ratios of the loss to  $J_c$  is also tabulated in Table I to show how each HTS influences the relative effective filament size. This also clearly shows that the effective diameter of the filaments are strongly influenced by the ramp rate.

In order to investigate the above observed ramp rate dependencies on the loss and  $J_c$ , a metallurgical examination was performed on the wires which were taken out at various temperatures during each of the temperature ramps to the final temperatures. The metallurgical observation of the wire cross sections utilizing optical and electron microscopy can be summarized as follows:

1. A slow temperature rise results in an outward movement of the inner filament rings reducing the interfilamentary spacing and enhancing the coalescence of the filaments, and thus results in a larger effective filament size. This outward motion of the filaments is associated with the movement of the  $\varepsilon$  and the  $\alpha$  phase interfacial boundary (See Fig. 1). When this interface moves slowly by a slow ramp rate (6° C/h) or by a holding time at low temperatures (e.g. 375°C), the filaments are dragged along outward with the advancing interface due to the conversion of the n to the  $\varepsilon$  phase. On the other hand, the motion of the filaments is minimal if the temperature is raised at faster rates (e.g. 60°C/h). This results in a lower hysteresis since the coalescence of the filaments are lessened due to a larger separation among the filaments than those by a slow ramp rate. (A further study is needed to understand how the filaments are moved with the advancing  $\varepsilon$  phase front.) (2) On the other hand,  $J_c$  is higher for the wires with a very slow ramp rate (6°C/h) than those with a fast rate (60°C/h). This is speculated to be related to the fact that the Sn distribution at the temperature (~600°C), at which the reaction to form Nb<sub>3</sub>Sn becomes significant, is much more uniform for a slow ramp rate than those for a fast rate. (See Fig. 2 and Table II.) For example, even the outermost wire locations (e.g. positions; K, L, M, and N) the Sn content of the matrix is nearly the maximum for the \alpha phase if the wire is heated at the rate of 6°C/h. On the other hand, the matrix is essentially pure Cu at the similar positions for 60°/h.

#### 4. ACKNOWLEDGMENTS

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#### REFERENCES

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Table I. Temperature Ramp Rate Dependence on Hysteresis Losses and Critical Current Densities.

Wire	Ramp Rate (C°/h)	Temp. Hours (C°,h)	Ramp Rate (C°/h)	Temp. Hours (C°,h)	Non-Cu <sup>(a)</sup> Loss (mJ/cc)	Non-Cu <sup>(b)</sup> J <sub>c</sub> (A/mm <sup>2</sup> )	Effect. Fil. Dia. (mJ/cc)/ (A/mm <sup>2</sup> )
A1.	15	375(24)	999	660(198)	456		
2.	15	375(24)	999	660(240)	389	736	0.528
				+ 740(6)			
3.	15	375(24)	75	660(198)	592		
4.	15	375(24)	75	650(240)	576	731	0.788
5.	15	375(24)	75	700(190)	561	751	0.747
6.	6			660(240)	499	801,796	0.623
7.	60			650(240)	411	771,736	0.546
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B1.	15	375(24)	75	660(240)	587	749	0.784
2.	15	375(24)	75	650(175)	592	705	0.840
3.	6			650(175)	518	705,740	0.715
4.	75	***	••	660(175)	350	669,631	0.538
5.	75			660(240)	405	686,678	0.595
6.	15	375(0)	75	650(175)	468	681,693	0.681

Table II. Compositions of the Cu-Sn Matrix at 600°C.

Heating Rate	6°0	C/h	60°C	60°C/h		
Position*	Cu (atm.%)	Sn (atm.%)	Cu (atm.%)	Sn (atm.%)		
A	85.23	14.77	84.22	15.78		
В			83.44	16.56		
C			83.66	16.34		
D 1	86.76	13.24	84.22	15.78		
2	82.22	17.78				
E	90.88	9.12	85.53	14.47		
F	90.88	9.12	85.38	14.62		
G			83.64	16.36		
Н			81.26	18.74		
I		***	80.98	19.02		
J	91.52	8.48	99.72	0.28		
K	91.44	8.56	99.97	0.21		
L	91.46	8.54				
M 1	92.87	7.13		****		
2	91.95	8.05				

<sup>(</sup>a) Hysteresis losses for ±3 T at 4.5 K.(b) Measured at NIST, MIT, and Univ. of Wisconsin.