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Image Analysis of Nb_3Sn Strand For ITER Application: A comparison with SSC Nb-Ti

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Abstract: We have quantified the distribution of filament sizes and filament separation in internal Sn type Nb_3Sn strand manufactured to ITER specification. In Nb-Ti strand for the SSC we had found a clear relationship between filament cross-sectional uniformity in a given cross-section and the *n*-value for an entire strand, but for the Nb_3Sn we are not able to find one. In these Nb_3Sn strands, however, there no clear relationship between *n*-value and filament uniformity, and there is considerable variation in filament size and spacing within a single cross-section.

1. INTRODUCTION

The two primary keys to the successful development of high critical current density, J_c, in production superconductor for the SSC project were the control of the nanostructure and the control of filament sausaging. Since the critical current density is approximately constant from one filament to another, a filament that is 20% lower in cross-sectional will carry ~20% less current. The smaller diameter filament cross-sections initiate the resistive transition. The sharpness of the resistive transition can be characterized by the index, *n*, of the power law, $V \propto I^n$, where V and I are the voltage and current respectively. The measurement of the *n*-value became a useful diagnostic of this type of performance limitation and was introduced as a specification for SSC strand and has been carried into the specification of ITER strand. In order to reduce sausaging, Phase I R&D SSC strands incorporated Nb diffusion barriers around the Nb-Ti filaments in order to reduce the formation of brittle Cu-Nb intermetallics at the filament-matrix interface during heat treatments. These conductors also benefited from work done by Eric Gregory and co-workers on the relationship between inter-filament spacing and filament shape stability.[1] Filament sausaging is typically measured by image analysis of cross-sectional area uniformity. High et al.[2] quantified the filament sausaging in SSC Phase I R&D strands and the coefficient of variation (the standard deviation in the filament cross-sectional areas normalized by the mean cross-sectional area) was found to range from 5-13%. They found that the J_c declined sharply with increasing sausaging and that sausaging was increased with longer and higher temperature heat treatment. The clear implication was that the diffusion barrier (1-2% of the total Nb-Ti area) was too thin in these strands to inhibit intermetallic formation under aggressive high J_c heat treatments. When the barrier was subsequently increased to 4% of the strand area for the SSC Phase II R&D strand, to the 3-5% level and became independent of heat treatment.[3]

In Nb_3Sn strand the control of filament uniformity has proved to be more difficult and a relationship between *n*-value and filament uniformity has not been established. This paper summarizes the progress of some recent studies on these issues by our group.

2. EXPERIMENTAL PROCEDURE

Internal Sn type strand was supplied by IGC in both the reacted and non-reacted forms. This strand design uses a stack of 19 sub-elements each containing 162 Nb-Ta filaments and a central Sn core. Six additional Sn cored spacers were placed between sub-elements.[4] Unreacted and reacted cross-sections are shown in figure 1a and figure 1b. The polished cross-sections were given a short etch in a solution of 50vol.% H_2O, 37%vol. HNO_3 and 13vol.% HF to remove smeared surface layers. Back-scattered Electron Images were digitally recorded on a Tracor ADEM SEM at 15kV. Quantitative image analysis was performed on a Megavision 1024XM system with image sizes of 1024x1024 pixels with a gray-scale depth of 256.

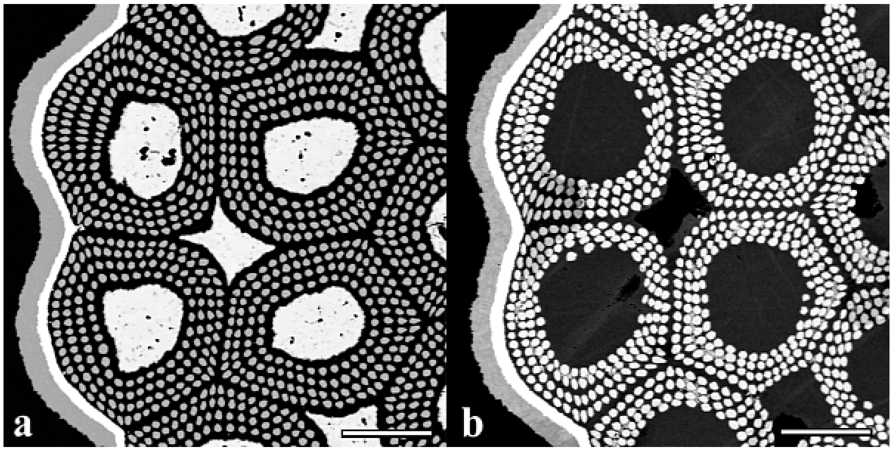

Figure 1 SEM-BEI images of IGC 15T-2 in transverse cross-section, a) before and b) after heat treatment (660C/240h). Scale bars are 50μm in length.

In addition to filament uniformity, we were also interested in filament separation because this impacts coupling of the filaments and magnetization loss. To do this we developed a simple method of measuring the minimum separations of filaments from each other. The digitized filaments were artificially expanded by 1 pixel iterations and the number of filaments touching each other recorded until all the filaments were joined.

3. RESULTS

In Table I we compare the coefficient of variation for filaments in similar composites before and after heat treatments. The filament non-uniformity is quite high, ~10-15% rather than the 3-5% of SSC Nb-Ti. The cross-sectional uniformity deteriorates by only 1-2% in the reaction process. The ITER requirement for *n*-value is 20 and one of these strands falls below that value but, despite a range of *n*-values from 19 to 31, no corresponding variation in coefficient of variation was observed. In Table II we compare the minimum filament separation for the same samples as in Table I. The separation is similar from sub-element to sub-element before reaction. After reaction the separation is greatly reduced especially when inter-filamentary voids are produced. This is a result of filament movement away from the Sn core, as can be observed in Figure 1.

Table I. Comparison of coefficient of variation in filament cross-sectional areas for three fully reacted composites of the same design in both the reacted and unreacted condition.

Composite	**M5**, *n*=31 (12T, 4.2K)		**5T**, *n*=19 (12T, 4.2K)		**15T-2**, *n*=26 (12T, 4.2K)	
Sub-element location	Unreacted	Reacted	Unreacted	Reacted	Unreacted	Reacted
Outer row	13%	15%	13%	13%	12%	14%
Outer row	13%	14%	14%	14%	12%	15%
2nd row	10%	14%	12%	14%	13%	13%
2nd row	12%	18%	12%	15%	14%	14%

Table II. Comparison of minimum filament separation in μm before and after heat treatment for three similar fully reacted composites.

Composite	M5		5T		15T-2	
Sub-element location	Unreacted	Reacted	Unreacted	Reacted	Unreacted	Reacted
Outer	1.7	0.8	1.2	0.9	1.8	0.9
Outer	1.7	0.7	1.6	1.0	1.8	0.8
2nd row	1.4	1.1*	2.0	1.0	1.8	1.0
2nd row	1.6	0.7	1.7	1.0	1.8	0.8

* = voids in center of sub-element

In Table III we compare 2 strands from the same composite that have received 2 different heat treatments. The heat treatments have resulted in partially reacted filaments. For the data in tables I and II we artificially separated filaments that were physically touching into separate filaments. The data from separated filaments is appropriate for the evaluation of filament shape stability but in terms of physical properties coupling (both by touching and by proximity) must be taken into account and in Table III we show the very different values obtained before and after separation. There is a large difference in the degree of reaction from sub-element to sub-element in the same cross-section under partial heat treatment. The degree of reaction is related to the location of the voids created during reaction: If the voids occur in the center of the sub-element the reaction occurs more slowly and the filaments are further apart (as in the 10 % unreacted case here), if the voiding is inter-filamentary the reaction is faster but the filaments are closer to each other. The effect of the void location on the degree of reaction was also observed by Dietderich et al. in an early internal Sn composite.[5]

Using the same technique we used to measure filament separation, we can also simulate the effect on apparent filament size and uniformity by incremental proximity coupling. The results of this simulation are shown in Figures 2 and 3 respectively. Surprisingly large simulated coupling distances >500nm are required to have a significant impact on apparent filament diameter. This does not agree well with effective filament diameter measurements for this type of conductor.

4. CONCLUSIONS

1. Variation in filament cross-sectional area is higher in these ITER Nb_3Sn composites than in high performance Nb-Ti SSC production strand.
2. Most of the variation in filament cross-sectional area is produced prior to reaction.
3. Inter-filamentary spacing is reduced by ~50% during heat treatment.

Table III. Comparison of filament cross-sectional area variation for >99% reacted and ~90% reacted composites showing the effect of physical coupling on the apparent filament uniformity.

Heat Treatment	% of filament unreacted	Coefficient of Variation for Filament CSA, %			
		Entire Filament		Reacted Area only	
		Joined	Separated	Joined	Separated
6C/h ramp to	<0.1%	30	14	30	14
660C/240h	0.1%	31	13	31	13
75C/h ramp to	0.1%	17	13	17	13
650C/100h	10.2%	14	13	16	14

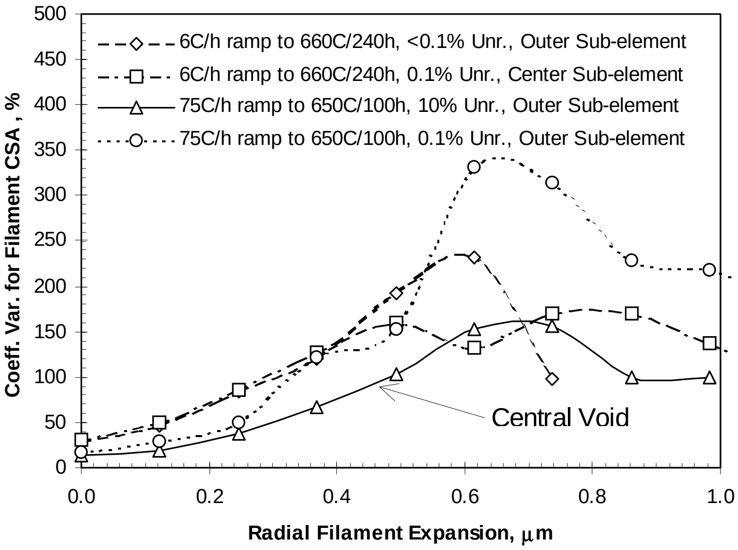

Figure 2. Effect of radial filament expansion by image analysis on apparent filament uniformity.

4. Filament separation and reaction rate can differ markedly within a single strand cross-section. This appears to be related to the location of Kirkendall voids in the strand.
5. Proximity coupling of filaments cannot be simply explained by physical proximity within one transverse cross-section of strand.

Acknowledgments

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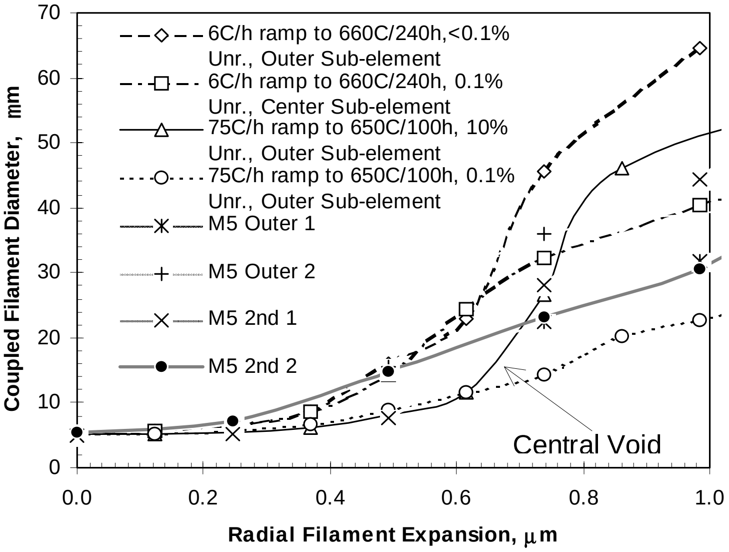

Figure 3. Effect of radial filament expansion by image analysis on apparent filament diameter.