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The vital role of a well-developed Sn-Nb-Cu membrane for high J_c RRP® Nb_3Sn wires

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Abstract* — The importance of obtaining the highest possible critical current density, J_c, in Nb_3Sn conductors at the smallest possible effective filament diameter, D_{eff}, remains strong. Recent experiments with the latest RRP® wire designs (which yield the highest J_c of any commercial Nb_3Sn wire) have revealed significant new aspects to the complex phase evolution in the temperature range below the A15 formation. Here we report metallographic studies designed to quantify and understand the phase evolution throughout the heat treatment. We show that between ~250°C and 408°C, a uniform and reproducible annulus of the so-called *Nausite* phase (a Nb-Sn-Cu ternary) is formed at the interface between the sub-element core and the Nb/Cu filament pack. By tracking the components we demonstrate that this annulus acts as a *membrane*, separating the Sn supply (in the sub-element core) from the surrounding filament pack. Surprisingly, below 408ºC when the core is all solid η and/or ε (Cu-Sn phases), there is an inward osmosis-like flow of Cu from the filament pack—*through* the Nausite membrane—into the Sn reservoir, *but* very little outward diffusion of Sn into the filament pack. This asymmetric diffusion process allows much, but not all, of the Sn-rich η phase to be converted to the higher melting point ε phase. This function is critical, because by minimizing the *η-to-liquid* transition above 408°C, the Nb filament pack is protected from an irregular and rapid Nausite growth (a consequence of the liquid attacking the filament pack). Furthermore, we observe that smaller diameter wires with degraded J_c tend to produce less effective Nausite membranes which draw less Cu into the core, resulting in larger amounts of η below 408°C and therefore more attacks on the filament pack upon liquefaction above 408ºC. In addition we show that these small diameter wires not only produce more Nausite after liquefaction, but also contain more *disconnected A15* at the filament-core interface, which we have found to be a result of the Nausite transformation into Nb_3Sn through the $NbSn_2$ – Nb_6Sn_5 pathway. We believe that in order to advance RRP® technology to smaller D_{eff} we must first understand the most effective way to promote a uniform Nausite membrane that allows an efficient consumption of the core η, therefore mitigating liquefaction above 408°C.

I. Introduction

The Restacked-Rod Process (RRP®) composite strand design is currently the Nb_3Sn wire technology that is closest to meeting the requirements for future particle accelerator applications like the hi-Lumi LHC and other particle colliders beyond the LHC [1]. Over the last decade, the RRP® technology has undergone significant advancements regarding stack count (adding more sub-elements to the wire cross section in order to reduce sub-element size, D_{eff}) while preserving yield, high critical current density, J_c, and high residual resistivity ratio, RRR, [2], [3]. Unfortunately, there seems to be a threshold D_{eff} below which the high J_c (> 2800 A/mm^2) and good RRR (>200) representative of this technology no longer holds [4]. In order for future magnets to benefit from smaller D_{eff}, the reasons for this limitation must be understood.

II. Background Research

Recent studies of the RRP® wire heat treatments have suggested that Nausite (a Sn-Nb-Cu ternary phase) can influence the diffusion of Cu and Sn during the early stages of the heat treatment, altering the strand quality [5]. It was also observed in a low Local Area Ratio (LAR) wire design, such as today's RRP® design, that a ring of Nausite forms around the Sn-rich core of each sub-element during the 400°C dwell.

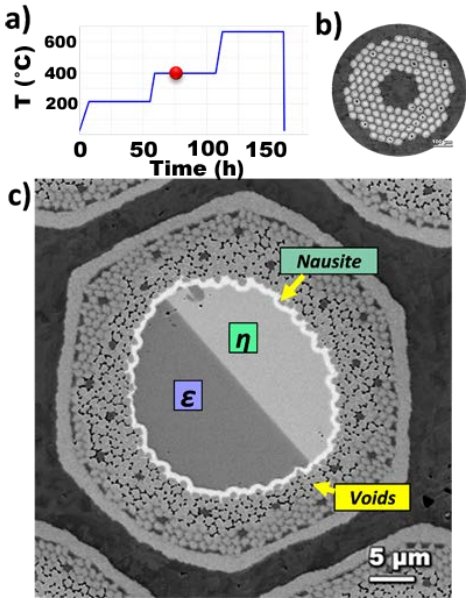

Fig. 1. (a) The standard heat treatment profile of an RRP® wire, (b) a transverse cross section of a 132/169 RRP® wire, and (c) a detail of a sub-element during the ~400°C dwell showing the Nausite membrane which allows Cu into the core while strongly restricting Sn diffusion into the filament pack. Voids left behind by the Cu migration through the Nausite are very evident in the filament pack.

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This Nausite membrane limits the Sn diffusion outwards into the filament pack while at the same time permits Cu diffusion into the core (as shown in Fig. 1). The Nausite then decomposes at higher temperatures, at which point, a very rapid Cu diffusion towards the core occurs—often producing a large degree of porosity between the Nb filaments [5]. It was noted that this entire process happens before the Sn diffuses into the filaments to form Nb_3Sn (*i.e.* before the heat treatment, HT, reaches ~ 600°C). The important fact here, is that the Nausite produces undesirable large grain and discontinuous Nb_3Sn [6], which, as in PIT strands [7] is not expected to contribute significantly to the transport critical current density and thus reduces the efficiency of the non-Cu package. This morphology originates from decomposition of Nausite into $NbSn_2$, Nb_6Sn_5 and finally Nb_3Sn [8], [9].

Our studies explore these transformations in more detail so as to understand the reasons behind the degradation of properties in RRP® wires as wire size decreases.

III. Experiment Setup

We have studied two different 132/169 RRP® billets at three different sizes each, 0.85 mm, 0.7 mm and 0.6 mm having sub-element sizes of 49 µm, 41 µm and 35 µm respectively. Small sections of wire (~13 mm) are heat treated in sealed quartz tubes using various heat treatment schedules. The samples are sequentially pulled out of the furnace and quenched at various points in the HT in order to study the microstructure by high resolution SEM images.

IV. Results

Using digital image analysis (IA) we have identified that the most critical event during the heat treatment happens below the liquefaction of η (at 408°C). During the ~400°C dwell, Cu diffuses through the Nausite membrane and increases the Cu content in the core, effectively transforming η into ε. Fig. 2 shows how the η fraction in the core evolves for three different sizes of the same billet. The behaviors are very similar but the smaller wires show a higher scatter and a less uniform decay. Table 1 shows the Nausite area % and the liquid area % in the sub-element 2 h and 45 min *after* the liquefaction of η. There is a higher content of Nausite for smaller wires.

V. Discussion and Conclusion

Through systematic study of the changes that occur before A15 formation, we have shown the important role that is played by the formation of a uniform Cu-Nb-Sn membrane in controlling Cu diffusion into the core. Exploiting this new perspective we are exploring modifying the HT and/or wire design to optimize this process. The goal is to promote a uniform Nausite membrane that will enhance inward Cu diffusion so as to prevent the liquefaction of η and thus avoiding irregular growth of Nausite after liquefaction—which, if not controlled, produces disconnected Nb_3Sn that does not contribute to J_c. As a first step we have found that elimination of the traditional first dwell at ~215°C does not affect J_c properties (all billets at multiple sizes produced the same J_c, n-value, and H_k with or without the ~215°C dwell), thus simplifying the HT scope for future HT studies.

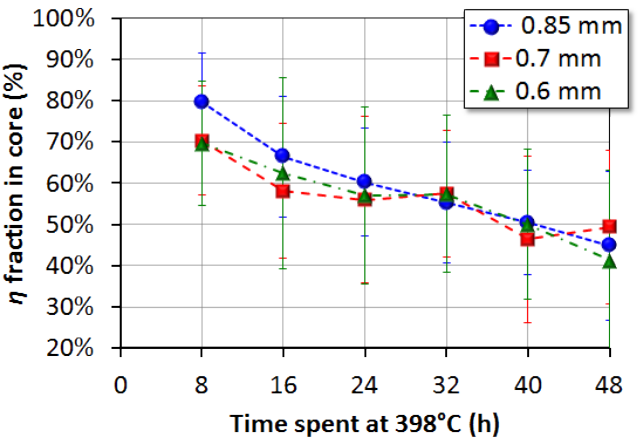

Fig. 2. Phase evolution of η (area fraction in the core) for three different wire diameters using the same composite design.

Table 1 Nausite and liquid area % (from IA of quenched samples) in the sub-elements after liquefaction of η.

Billet size	Nausite %	Liquid %
0.85 mm	7.4	0.2
0.70 mm	8.5	0.1
0.60 mm	9.5	0.1

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