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RAPID COMMUNICATION

Influence of Ti and Ta doping on the irreversible strain limit of ternary Nb_3Sn superconducting wires made by the restacked-rod process*

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Abstract
Nb_3Sn superconducting wires made by the restacked-rod process (RRP®) were found to have a dramatically improved resilience to axial tensile strain when alloyed with Ti as compared to Ta. Whereas Ta-alloyed Nb_3Sn in RRP wires showed permanent damage to its current-carrying capacity (I_c) when tensioned beyond an intrinsic strain as small as 0.04%, Ti-doped Nb_3Sn in RRP strands exhibits a remarkable reversibility up to a tensile strain of about 0.25%, conceivably making Ti-doped RRP wires more suitable for the high field magnets used in particle accelerators and nuclear magnetic resonance applications where mechanical forces are intense. A strain cycling experiment at room temperature caused a significant drop of I_c in Ta-alloyed wires, but induced an increase of I_c in the case of Ti-doped strands. Whereas either Ti or Ta doping yield a similar enhancement of the upper critical field of Nb_3Sn, the much improved mechanical behavior of Ti-alloyed wires possibly makes Ti a better choice over Ta, at least for the RRP wire processing technique.

The noteworthy improvement in the non-Cu critical-current density (J_c) of internal-tin, restacked-rod process (RRP®), Nb_3Sn superconducting wires to over 3000 A mm^{-2} at 4.2 K and 12 T enabled the construction of a record 16 T dipole magnet [1, 2]. Nonetheless, the high magnetic field dipoles and quadrupoles required for the large hadron collider (LHC) upgrade may necessitate pushing the performance of Nb_3Sn to even higher limits.

The RRP technique consists of stacking Cu-clad Nb rods around a Sn core. Each filament bundle (subelement) has its own Sn source and is surrounded by a Nb distributed barrier. The RRP design increases the fractions of Nb and Sn, and decreases that of Cu, so as to maximize the amount of Nb_3Sn formed and to promote a homogeneous delivery of Sn to Nb rods through the thin inter-filamentary Cu network during the reaction heat treatment. The good diffusion of Sn into Nb results in a near stoichiometric composition of Nb_3Sn throughout the entire A15 volume, which is essential for achieving a high current-carrying capacity in the wire [3]. However, the migration of interstitial Cu to the Sn core during the heat treatment causes Nb_3Sn filaments to coalesce into one solid tube inside each subelement. The large diameter (of several tens of micrometer) of this Nb_3Sn tube has challenging ramifications, detrimental to the stability and mechanical integrity of magnets made of such a wire, since it causes a significant increase in hysteretic losses and magnetic flux

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jumps [4, 5], and a considerable reduction in the tolerance of the wire to axial and bending strains [6–9].

In this rapid communication, we show that despite the large size of agglomerated Nb_3Sn filaments, it is possible to dramatically improve the tolerance of RRP wires to tensile strain by doping Nb_3Sn with Ti instead of Ta. The irreversible strain limit (ε_{irr}), which denotes the onset of fractures in the Nb_3Sn, is found to be much higher in the Ti-alloyed wires as compared to Ta-alloyed strands. We confirmed this finding in different RRP wire architectures comprising 54, 108 or 198 Nb_3Sn subelement counts. In this rapid communication, we will focus solely on the 54/61 design (54 Nb_3Sn + 7 Cu subelements) because it has the largest subelement size ($\sim$65 μm) among the wires studied.

The two RRP wires presented herein were fabricated by Oxford Superconducting Technology (OST), had the same 54/61-subelement architecture, a non-Cu fraction close to 54%, and a wire diameter of 0.7 mm. Alloying was in the amounts of 4 at.% Ta (billet #8781) and 2 at.% Ti (billet #9415), respectively, so as to obtain an optimum increase of the upper critical field [10]. The strands used different alloying approaches; whereas billet #8781 used Ta-alloyed Nb rods, billet #9415 had Nb-47 wt% Ti rods inserted amongst the Nb rods in each subelement pack. Both wires received identical heat treatments at 640 °C for 48 h that also included two intermediary steps at 210 °C and 400 °C for 72 h and 48 h, respectively. This heat treatment schedule does not lead to optimized J_c for either of these two wires, but was used in order to obtain a high residual resistivity ratio (RRR) for a better electrical stability of the wires. The RRR was about 251 and 369 for billet #8781 (Ta-doped) and billet #9415 (Ti-doped), respectively, indicating that the purity of the copper matrix was similar in the two strands. The peak value of the non-Cu J_c versus strain we obtained at 12 T and 4.0 K was 2668 A mm^{-2} and 2940 A mm^{-2} for the Ta- and Ti-alloyed strands, respectively.

We used a Walters' spring device for applying axial strain (ε) to the sample [11, 12]. The Cu–Be spring used had a T-section design, four active turns, and a wide elastic strain range between -1% and $+1$%. Each Nb_3Sn sample was heat treated on a stainless-steel mandrel, transferred onto the spring, and soldered to it at $\sim$200 °C by use of a Pb–Sn solder. Three pairs of voltage taps were attached to the sample and covered one full turn each ($\sim$8 cm long), allowing three separate determinations from each specimen. Data statistics were further improved by measuring at least two samples per type of wire. The relatively long separation of voltage taps allowed determining the critical current (I_c) at electric-field criteria (E_c) as low as 0.01 μV cm^{-1}. All data presented herein were extracted by use of the more common 0.1 μVcm^{-1} criterion, but conclusions remain valid even for the more sensitive criterion of 0.01 μV cm^{-1}. Measurements were carried out in liquid helium at 4.0 K, in a magnetic field of either 12 or 16 T. The estimated uncertainty due to random effects in estimating ε_{irr} was $\pm$0.02% strain.

The $I_c(\varepsilon)$ behavior of the Ta- and Ti-doped wires is compared in figure 1. The sample was loaded and (partially) unloaded several times to determine ε_{irr}. Each pair of unprimed

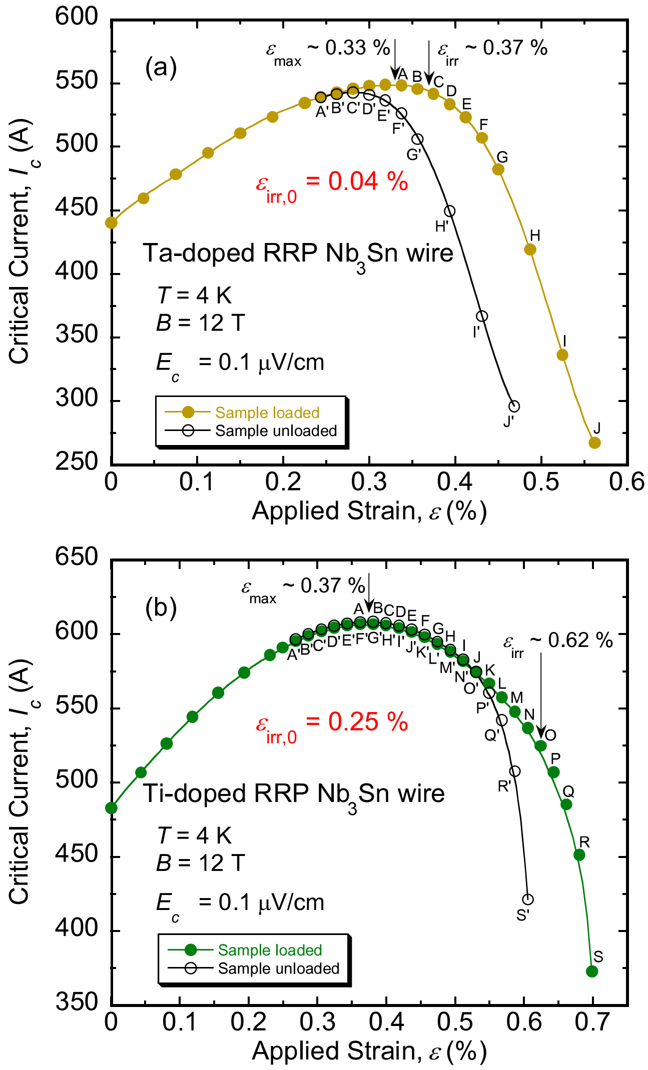

Figure 1. Comparison of I_c (ε) at 4 K and 12 T for (a) Ta-doped and (b) Ti-alloyed RRP wires. The intrinsic irreversible strain limit $\varepsilon_{irr,0}$ ($\sim$0.25%) for the Ti-doped wire is substantially higher than that of the Ta-alloyed strand ($\sim$0.04%).

and primed letters used in figures 1(a) and (b) indicates a loaded strain point and its corresponding partially unloaded strain point, respectively. For example, strain was released from point A to point A′, then increased to point B and released again to point B′ and so forth. ε_{irr} corresponds to the strain that produces the first deviation of the unloaded curve from the loaded curve due to a permanent degradation of I_c.

The $I_c(\varepsilon)$ curve goes through a peak at a strain ε_{max} that corresponds to the compressive pre-strain experienced by the Nb_3Sn filaments after cool-down from the heat treatment temperature to 4 K. However, because the sample is soldered to a Cu–Be spring, the *absolute* values of ε_{max} and ε_{irr} are artificially higher due to the thermal expansion mismatch between Cu–Be and the sample. These strains can also change slightly from sample to sample depending on the amount of specimen tightening during mounting. Whereas the values of ε_{max} and ε_{irr} quoted are not exact, those of the *intrinsic* irreversible strain limit ($\varepsilon_{irr,0} = \varepsilon_{irr} - \varepsilon_{max}$) truly represent the amount of tensile strain that can be applied to the Nb_3Sn in the wire before filaments begin to fracture.

The Ta-doped wire had $\varepsilon_{irr,0} \approx 0.04\%$ (figure 1(a)). The six segments measured of this wire (2 samples × 3 voltage pairs) have shown very little scatter of $\varepsilon_{irr,0}$. Furthermore, the value of $\varepsilon_{irr,0}$ was only 0.02%–0.07% for E_c between 0.01 and 1 μV cm^{-1}. The small value of $\varepsilon_{irr,0}$ indicates that this wire has a very low tolerance to tensile strain, in agreement with reported results on RRP wires [6–9]. The new finding is that depicted in figure 1(b), showing the remarkable enhancement of $\varepsilon_{irr,0}$ to about 0.25% for the Ti-doped RRP wire. This indicates that a relatively high tensile strain can be applied safely to this RRP wire despite its large subelement size. Again, $\varepsilon_{irr,0}$ showed very little scatter among the six segments measured of this strand, and varied only from 0.21% to 0.28% when E_c changed from 0.01 to 1 μV cm^{-1}.

The relevance of this finding is further evidenced by a room-temperature (RT) strain cycling experiment. I_c was first measured at 4 K and 16 T as a function of strain up to $\varepsilon \approx \varepsilon_{max}$ (~0.30% for both Ta- and Ti-doped samples). Strain was then released to zero and the apparatus was warmed up to RT. At RT, strain was cycled 10 times between 0% and 0.30% (~ε_{max} at 4 K). Thereafter the apparatus was inserted in the magnet, and I_c was re-measured as a function of strain. This experiment was originally designed to determine a strain limit for safe handling of the wire at RT, so as to prevent damage during magnet fabrication and assembly.

After RT strain cycling of the Ta-doped specimen, we obtained a 17% degradation of I_c (figure 2(a)), and a drastic depression of the n-value (exponent in the fit I^n of the V–I curve around I_c) from ~30 to less than 10 (figure 2(b)). The low n-value is indicative of fracture damage of the subelements. The degradation of I_c determined at 0.01 μV cm^{-1} was as high as ~40%, indicating that the sample did not tolerate the RT strain cycling.

At RT, the compressive pre-strain ε_{max} is expected to be smaller than that at 4 K, as may be the case for the Nb_3Sn fracture strain. Considering that the internal forces on wire components during thermal contraction cancel each other [13], simplified calculations suggest that the compressive pre-strain at RT should be smaller roughly by about 0.1% in comparison to its value at 4 K when the sample is not soldered to the active turns of the spring. If the sample is soldered to the spring as is the case of the results presented herein, the shift of the compressive pre-strain is dictated mostly by the spring thermal expansion. In this case, the expected shift of the compressive pre-strain would be similar to the differential thermal expansion between the spring and the wire from 4 K to RT, which is approximately about 0.05%.

An applied strain of 0.30%, which is safe at 4 K since it is below ε_{irr}, becomes higher than the fracture strain at RT and causes the degradation observed. This highlights the limitations of having such a small $\varepsilon_{irr,0}$ (~0.04%), and suggests that the safe strain margin at RT is even smaller than that at 4 K. Hence, more care is needed in handling this strand at RT during magnet assembly.

In contrast, RT strain cycling of the Ti-doped wire under the same conditions was not only safe but caused I_c to increase by 5.4%, as shown in figure 3(a). I_c determined at 0.01 μV cm^{-1} also increased by 5.3%. Even though ε_{max} of the Ti-doped wire is expected to be reduced at RT by an amount similar to that for the Ta-doped wire, its $\varepsilon_{irr,0}$ is large enough to keep the fracture strain at RT higher than the applied cycling strain (~0.3%). The choice of the cycling strain value (~ε_{max} at 4 K) was arbitrary, but judicious enough for highlighting the differences between the Ta- and Ti-alloyed wires.

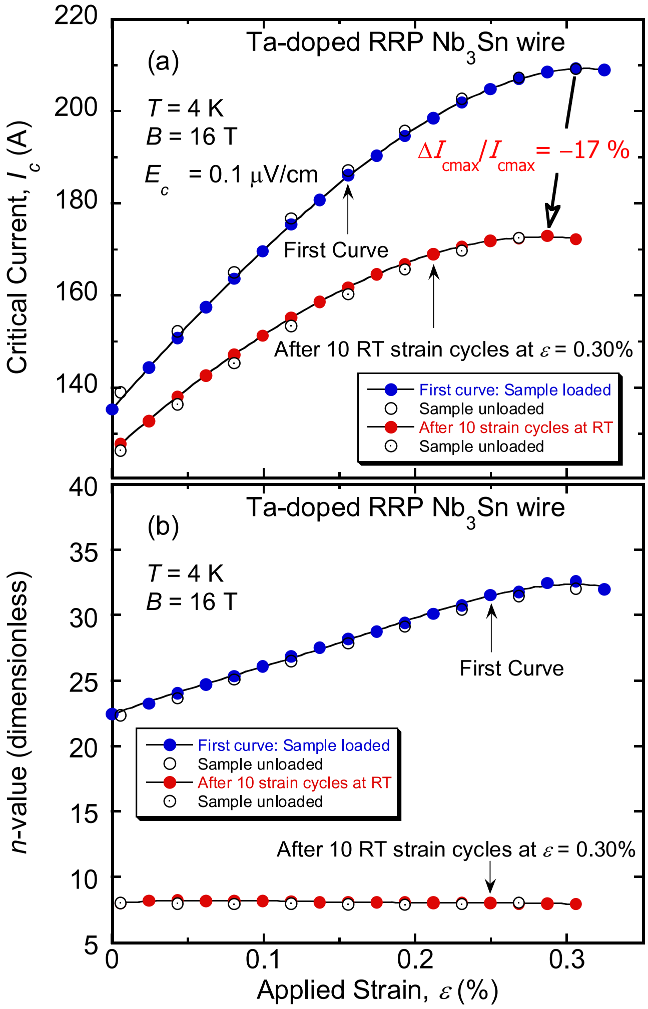

Figure 2. Room-temperature strain cycling of the Ta-doped RRP wire at a strain of 0.3% (~ε_{max} at 4 K) caused (a) a noticeable degradation of I_c (with a small shift of ε_{max}), and (b) a drastic depression of the n-value.

The increase of I_c for the Ti-alloyed wire is due, most likely, to a reduction in the three-dimensional strain of Nb_3Sn through the yielding of the wire matrix during RT strain cycling [14, 15]. This behavior is very similar to that seen after several hundreds of axial strain cycling at 4.2 K of internal-tin and bronze-route Nb_3Sn wires (candidates for use in the international thermonuclear experimental reactor (ITER)) [16], and fairly similar to that obtained through repeated wire bending at RT of a bronze-route Nb_3Sn wire [17]. A technique to effectively exploit such a phenomenon may be worth developing for RRP wires to boost magnet performances, especially since only a small number of RT strain cycles is needed.

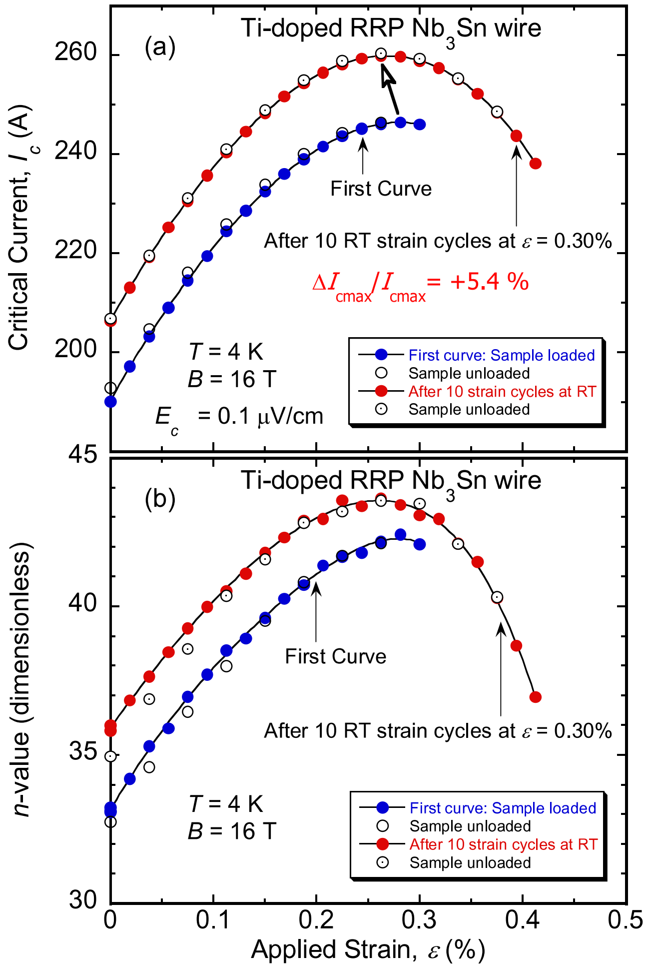

Figure 3. Room-temperature strain cycling of the Ti-doped RRP wire at a strain of 0.3% ($\sim\varepsilon_{max}$ at 4 K) caused an increase in (a) I_c (with a small shift of ε_{max}) and (b) n-value.

Non-Cu hysteretic ac losses measured in the Ta- and Ti-doped wires were very similar and large ($\sim$4800 mJ cm^{-3} for a $\pm$3 T field cycle), indicating that filaments have indeed agglomerated in both strands. Microstructural studies of these wires will be conducted to identify the mechanisms, if any, by which Ti doping improves the strain tolerance of RRP wires. It is possible that Ta actually weakens the fracture toughness of Nb_3Sn rather than Ti improving it. The irreversible strain limit of Ti-doped RRP wires reported herein seems to match or even exceed that of some internal-tin ITER wires [1], despite the fact that the filament size ($\leqslant$10 μm) in ITER strands is far smaller than that of the subelements in RRP wires. The multiple parameters that govern the fracture strain and fracture mechanisms in Nb_3Sn keep the causality between the wire design and its strain tolerance nontrivial. In future work, we will attempt to extend the correlation to ITER wires and other high J_c Nb_3Sn strands in order to shed more light on this matter.

Acknowledgments

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