Report of the activities at UNIGE in 2021

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The goal of a 100 TeV proton-proton collider set by the high-energy physics community initiated a focused R&D program coordinated by CERN to push Nb_3Sn towards its upper limit of performance. The baseline configuration for this Future Circular Collider (FCC) requires superconducting dipoles generating a magnetic field of 16 T. The prime challenge to reach this target is the development of conductors that exceeds the performance of state-of-the-art Nb_3Sn wires in terms of both critical current density (J_c) and tolerance to mechanical stresses. The required advancement of Nb_3Sn technology must build on novel processing routes scalable at the industry level and with a full control of the material both at the nanoscale dimension, tailoring the vortex pinning landscape to enhance J_c, and at the microscale dimension, linking the electromechanical behavior to the wire architecture.

Two research agreements have been signed between UNIGE and CERN under the umbrella of CHART2, with the scope of

1) investigating methods for the inhibition of the grain growth in Nb_3Sn by means of nanoparticles that form through an internal oxidation process. Grain boundaries represent the primary centers for vortex pinning in Nb_3Sn: higher current densities are thus obtained in materials that have finer grains. Our focus is on the development of fabrication routes of Nb_3Sn superconductors with enhanced critical current performance and scalable for industrial production;
2) assessing the electromechanical limits of state-of-the-art and R&D Nb_3Sn wires. It appears clear from various conceptual studies that the design of a 16 T dipole entails electromagnetic stresses in the 200 MPa range and, thus, it becomes crucial to establish precisely the inherent limitation of the conductor.

1. Advanced Niobium-Tin superconductors for next generation particle colliders

We are investigating novel procedures to enhance the critical current density of Nb_3Sn wires up to the required goal of 1’500 A/mm^2 at 4.2 K and 16 T. This target, which is a mandatory performance requirement for a compact accelerator magnet, is at the upper boundary of the state-of-the-art best wire performance and exceeds by about 50% the performance specified for the industrial production of HL-LHC Nb_3Sn. We have chosen to concentrate primarily on internal Sn rod-type wires, because they are closer to meeting the FCC expectations, but the possibility of exploring alternative routes like powder-in-tube or other tubular configurations is not disregarded. Our efforts are focusing on methods to introduce an oxygen source that could inhibit the growth of Nb_3Sn grains during the reaction heat treatment by

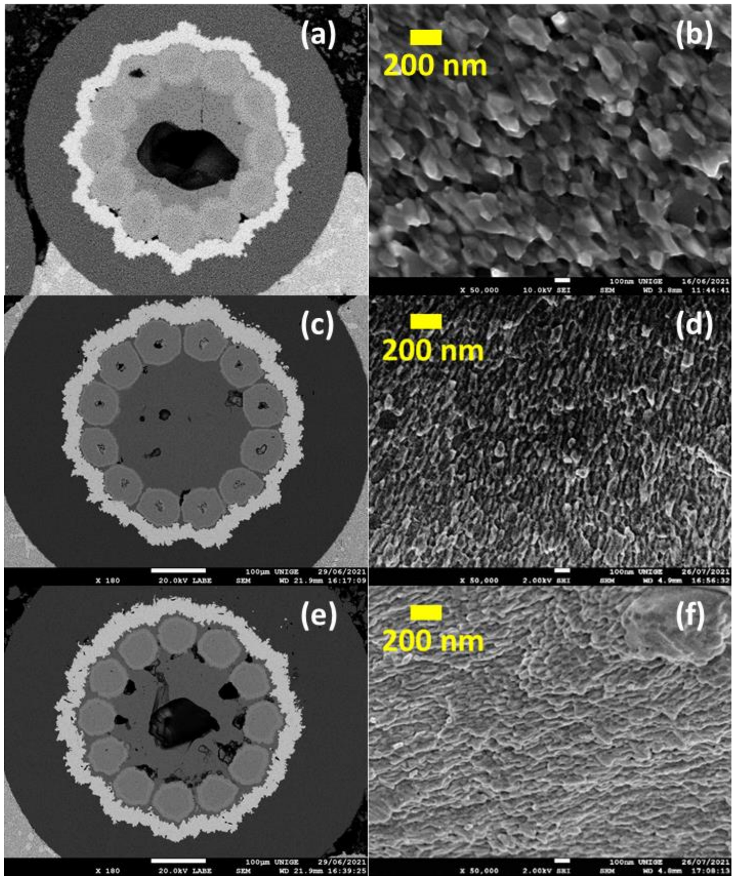

Figure 1 SEM images of the wire cross sections (left) and of the corresponding Nb_3Sn grains at fractured surfaces (right) for samples based on Nb-7.5Ta-2Hf in (a) and (b), Nb-7.5Ta-2Hf + SnO_2 in the core configuration in (c) and (d), and Nb-7.5Ta-2Hf + SnO_2 in the annular configuration in (e) and (f). The refinement of the grain size in the samples containing an oxygen source is evident.

forming oxide particles and, thus, enhance the critical current density. In the previous reporting period, we have obtained the refinement of the grain size in Nb_3Sn by producing mono-filamentary wires where an oxide powder was introduced in the central hole of Zr-doped Nb tubes (with and without Ta) deformed down to 0.2 mm in diameter [1]. The subsequent step in view of the development of internally oxidized multi-filamentary wires of the internal Sn type was the manufacture of test-bed sub-elements, with a reduced count of filaments. Internal Sn rod-type wires are made restacking a large number of sub-elements, each of them consisting of an array of Nb-alloy filaments inside a Cu matrix with a Sn core at the center. Our test-bed sub-elements are made of twelve Nb-alloy filaments in a Cu matrix around a Sn core, the filaments alloys being: Nb-1Zr, Nb-7.5Ta-1Zr and Nb-7.5Ta-2Hf (all weight percentages). Two routes were tested for the introduction of the oxygen source (SnO_2 or CuO nano-sized powders) and evaluated by their impact on the superconducting properties and on the deformability of the assembly:

1) Oxide in the Core: it is an approach similar to the one used for the mono-filamentary wires. A hole is drilled in the center of the Nb-alloy rod, it is filled with oxide nano-powders and finally the rod is deformed to the desired dimensions for the assembly of the sub-element;

2) Annular Oxide: the oxide powder is introduced between Nb-alloy and Cu. The oxide is deposited through a dip-coating process at the surface of the Nb-alloy rod which is then introduced in a Cu

tube. Dip-coating is performed using a slurry containing oxide nano-powders, typically SnO_2, and ethanol. The Nb-alloy rod is dipped repeatedly into the slurry: after each iteration we wait 10 minutes to let the ethanol evaporate and then we measure the thickness of the layer, repeating the process until we deposit enough material to oxidize completely the Zr or Hf present in the Nb-alloy.

Both configurations lead to the diffusion during the heat treatment of oxygen from the powder source into the Zr- or Hf-containing Nb-alloys and this results in the enhancement of J_c due to the formation of ZrO_2 or HfO_2 nanoparticles and the consequent refinement of the Nb_3Sn grains. High magnification SEM images of fractured Nb_3Sn wires, reported in *Figure 1*, allow us to make a direct comparison of the grain size of Nb_3Sn in wires without oxygen source in the composite, with SnO_2 in the core of the filaments, and with SnO_2 in the annular configuration, all made with the same starting Nb-alloys. The introduction of SnO_2 in both core and annular approach permits to reduce the grain size of Nb_3Sn from 100 nm in the reference wires without oxygen to approximately 50 nm. A summary including composition, oxygen source configuration and grain size of various types of wire is reported in *Table 1*.

Table I. Main parameters and properties of selected Nb_3Sn wires

Filament material, (wt%)	Metal oxide powder	Metal oxide configuration	Average grain size, (nm)
Nb-7.5Ta-2Hf	-	-	110
Nb-7.5Ta-2Hf	SnO_2	Core	45
Nb-7.5Ta-2Hf	SnO_2	Annular	45
Nb-7.5Ta-1Zr	-	-	105
Nb-7.5Ta-1Zr	SnO_2	Core	55

Normalized pinning forces of the same wires reported in *Figure 1* are shown in *Figure 2(a)*. Non-oxidized samples exhibit a peak of the pinning force at approximately 0.2 as expected when vortex pinning is dominated by the grain boundaries. The introduction of oxygen through the core approach determines a shift of the peak to approximately 0.24, suggesting an additional point-like defect type contribution to the vortex pinning due to the presence of ZrO_2 or HfO_2 precipitates. The annular configuration determines a further shift of the peak to 0.33 and this implies that point-like defects due to the oxide nanoparticles become the dominant pinning mechanism.

Transport critical current measurements have been performed on selected samples at 4.2 K and in magnetic fields up to 19 T. As expected, the oxidized wires exhibit a large enhancement in terms of layer-J_c (i.e. the critical current dived by the area of Nb_3Sn in the wire) with respect to the non-oxidized wires. In *Figure 2(b)*, we have estimated what would be the non-Cu J_c (i.e. the critical current divided by the wire cross-section area minus the Cu area) of an internal Sn rod-type wire having the same layer-J_c as the one of our samples. Very encouragingly, the resulting non-Cu J_c at 4.2 K and 16 T exceeds largely the FCC target of 1’500 A/mm^2.

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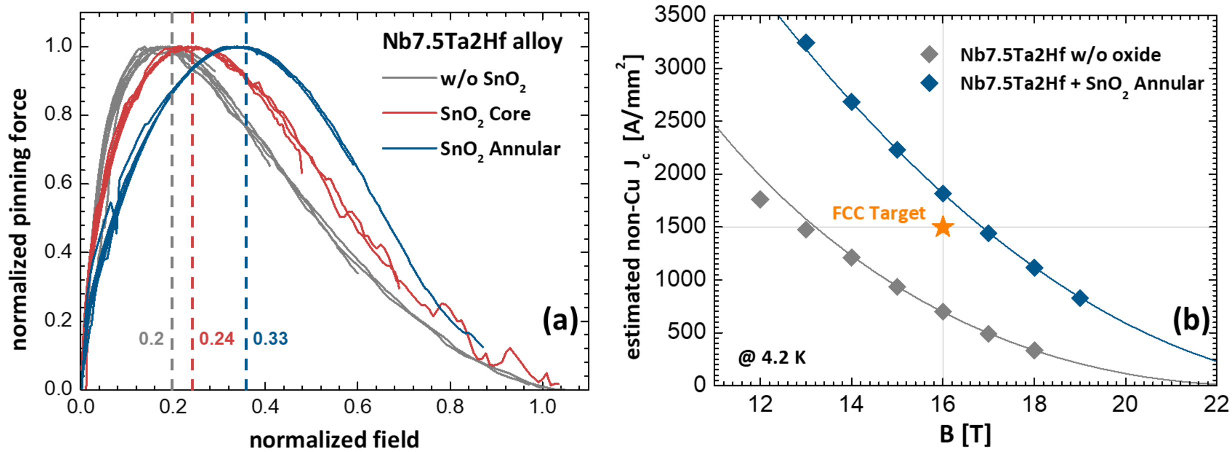

Figure 2 (a) Comparison of the normalized pinning force as a function of the reduced field between the wires made with the Nb-7.5Ta-2Hf alloy using different oxidation approaches: non-oxidized (gray), core (red) and annular (blue). The shift of the peak position towards higher reduced field values is consequence of an increasing density of oxide precipitates acting as point-like pinning centers; (b) Estimated non-Cu $J_c(B)$ at 4.2 K corresponding to the layer $J_c(B)$ measured at 4.2 K on the wires made with the Nb-7.5Ta-2Hf alloy: gray diamonds correspond to the wire without oxygen source, blue diamonds to the wire containing SnO_2 in annular configuration.

2. Multiphysical properties of advanced superconductors

The experience gained with the development of the 11 T dipoles for the High-Luminosity upgrade of the LHC (HL-LHC) shows that severe damages to the Nb_3Sn wire performance occur at a nominal stress level of ~150 MPa. On the other hand, it appears clear from various conceptual studies that the design of a 16 T dipole entails peak stresses in the 200 MPa range and, thus, it becomes crucial to establish precisely the mechanical limits at which Nb_3Sn can operate safely. The coils of an accelerator magnet are wound using insulated Rutherford cables composed of many multi-filamentary wires transposed together. Cables and ultimately wires inside the cables are subjected to different types of mechanical loads, appearing both during magnet construction and operation. The studies performed at UNIGE focus on the electromagnetic stresses arising during the operation, when the most severe loads are those acting in the cables' transverse direction. In particular, we performed during this reporting period an extensive measurement campaign focused on assessing the electromechanical limits of the Restacked-Rod-Process (RRP) Nb_3Sn wire developed for the HL-LHC 11 T dipole project, when tested in conditions aiming to reproduce its state inside accelerator coil windings.

Both round and 15% rolled wire samples have been investigated as a part of the campaign. The rolled samples seek to mimic the deformation of the superconducting wires due to cabling. One important result is that the transverse stress value leading to a permanent reduction in the critical current of 5%, assessed by convention with an applied field B = 19 T, lies in the range of 150–170 MPa. The highest values were measured on the 15% rolled wires, whose geometry favors a more homogenous redistribution of the transverse load within the conductor. The so-called irreversibility stress limit, σ_{irr}, is also controlled by the rigidity of the wire impregnation. Our tests are performed using the relatively soft epoxy-L impregnation, but previous measurements on Powder-In-Tube Nb_3Sn wires have shown an increase of σ_{irr} by ~50 MPa

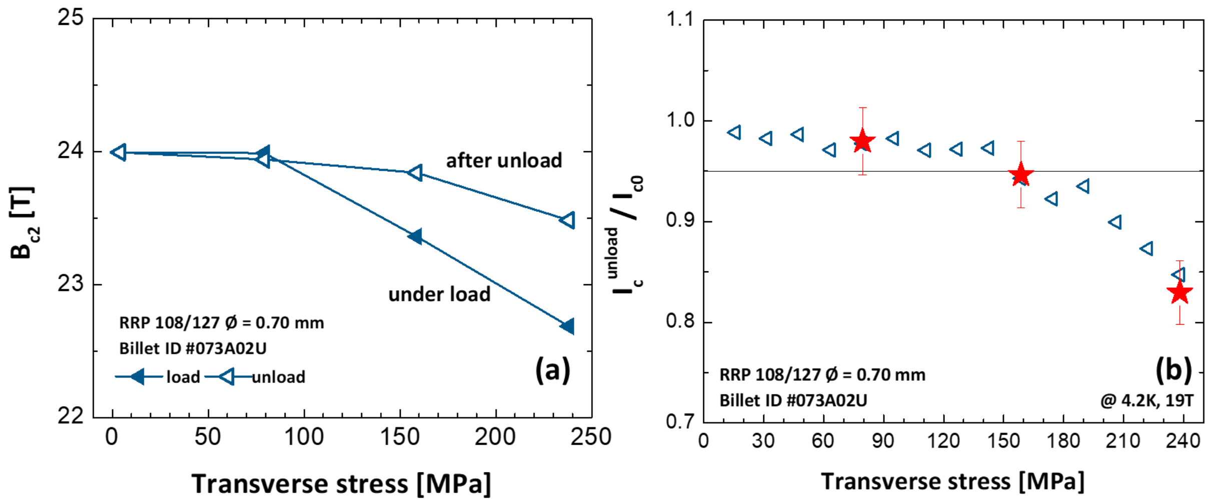

Figure 3 (a) Evolution of B_{c2} at 4.2 K under load (solid triangles) and after unload (open triangles) for increasing values of the applied stress in the transverse direction, as measured on a round RRP wire sample with a diameter of 0.7 mm; (b) Normalized critical current after unload measured at 4.2 K, 19 T (open triangles) for a round RRP wire sample with a diameter of 0.7 mm plotted together with the expected permanent reduction of I_c due to residual stresses (red stars), as calculated using the measured values of B_{c2} after unload.

when adding a glass fiber reinforcement [2], which is commonly used in accelerator magnets based on Nb_3Sn. Hence, our results suggest that the nominal stress levels experienced by the conductor during assembly and operation of the 11 T dipoles should not translate into a visible permanent reduction of the magnet performance and that there must be locations where the actual stress experienced by the conductor in the magnet exceeds largely the nominal stress.

It is now well assessed that the permanent reduction of the critical current in a wire occurs from the combination of the effects of cracks in the Nb_3Sn filaments and plastic deformation of the Cu matrix. We developed a method to identify the dominant degradation mechanism from the evolution of the upper critical field after stress unload, B_{c2}^{unload}, along the transverse loading history. Moreover, this method allows us predicting the fraction of critical current lost after unload due to the residual stresses imposed to Nb_3Sn by the plastically deformed Cu matrix. *Figure 3(a)* shows the evolution of B_{c2} under load (solid triangles) and after unload (open triangles), as measured on a round wire sample. In particular, the observed decrease of B_{c2}^{unload} when unloading from increasing stress values (the reduction of B_{c2}^{unload} after unload from 240 MPa is about 0.5 T), represents a clear signature of the effects of residual stresses. In *Figure 3(b)* we report the normalized critical current after unload measured at 19 T together with the expected permanent reduction of I_c due to residual stresses, as calculated using our model. Interestingly, the measured reduction of I_c after unload from up to 240 MPa can be fully accounted for with the reduction of B_{c2} due to residual stresses. This suggests that cracks in Nb_3Sn have a negligible role in the degradation of I_c in the explored stress range. An independent confirmation of this conclusion comes from a study combining X-Ray tomography and deep learning networks [3,4]. We performed X-Ray tomography experiments at the High Energy Scattering beam line of the European Synchrotron Radiation Facility (ESRF) on Nb_3Sn wire samples submitted to transverse loads at various pressures. The resulting tomography images were then analyzed by means of deep learning Convolutional Neural Networks to identify cracks inside the Nb_3Sn filaments. In particular, we have analyzed the exact same wire sample whose critical current vs transverse stress values are shown in *Figure 3(b)*, after its final unload from 240

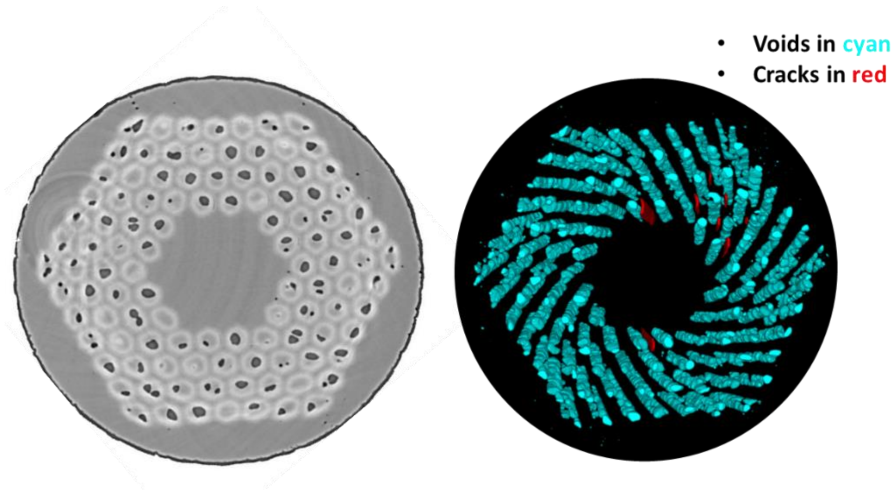

Figure 4 X-ray tomography cross section of the 0.7-mm RRP wire tested at the ESRF facility after I_c measurement under transverse load up to 240 MPa (left). 3D void reconstruction and crack detection performed by means of deep learning Convolutional Neural Networks (right): very few cracks were detected, none of them interrupting the sub-elements.

MPa. Surprisingly, only very few cracks were detected, as shown in *Figure 4*. Hence, this observation provides a further experimental evidence that residual stresses dominate the permanent reduction of I_c up to stress values of ~200 MPa. On the other, CERN reported the observation of filament cracking in the midplane of 11 T short model coils and this suggests that the conductor in this magnet was experiencing peak stresses well beyond the nominal 150 MPa.

References

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Conference presentations

- *Frontiers of Nb_3Sn and REBCO Conductor Technology for Future Applications in High Magnetic Fields*, Presenting author: Carmine SENATORE, invited oral at MRS Spring Meeting 2021, April 17 – 23, 2021;
- *Deep learning applied to X-ray tomography as a new tool to analyze the formation and propagation of cracks in RRP Nb_3Sn wires*, Presenting author: Tommaso BAGNI, contributed oral at EUCAS 2021, the 15th European Conference on Applied Superconductivity, September 5 – 9, 2021;
- *Evaluation of approaches to introduce oxygen sources for the internal oxidation of Zr and Hf in rod type Nb_3Sn wires*, Presenting author: Gianmarco BOVONE, invited oral at EUCAS 2021, the 15th European Conference on Applied Superconductivity, September 5 – 9, 2021;

- *Degradation of I_c after mechanical unload in high performance Nb_3Sn wires: the role of filament breakages and residual stress*, Presenting author: Carmine SENATORE, contributed oral at EUCAS 2021, the 15th European Conference on Applied Superconductivity, September 5 – 9, 2021;
- *Recent developments of Nb_3Sn wires for application*, Presenting author: Carmine SENATORE, invited oral at ISS2021, the 34th International Symposium on Superconductivity, November 30 – December 2, 2021;