Development of internal Sn Nb_3Sn wires with internal oxidation

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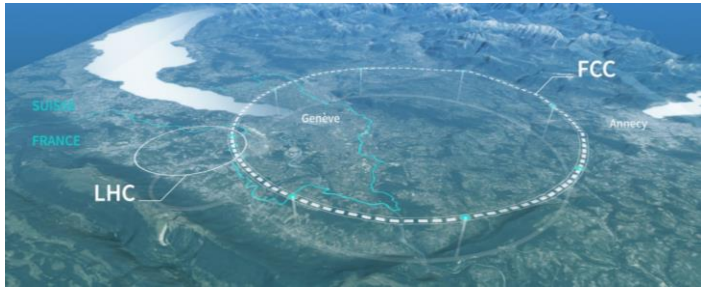
CERN, Switzerland

Outline of the presentation

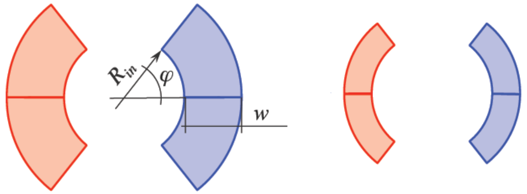
- ***State of the art on the internal oxidation process***
- ***Nb_3Sn wires are not created equal***
    - *Differences between PIT and RRP wires*
    - *Is there a better wire technology for accelerator magnets?*
- ***Wires manufacturing at UNIGE***
    - *Monofilamentary wires*
    - *Simplified subelements*
    - *Multifilamentary wires with and without internal oxidation*
- ***Conclusions***

The importance of oxidation for Nb_3Sn

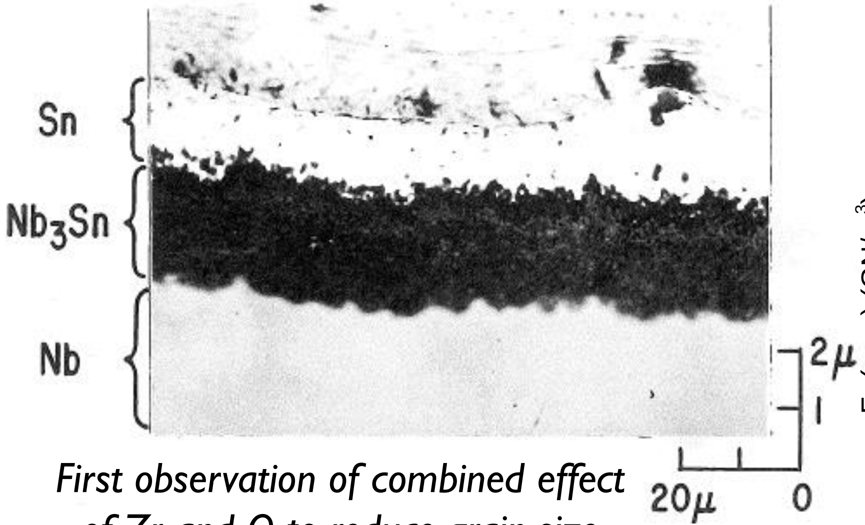


LHC	FCC-hh
27 km, 8.33 T	100 km, 16 T
14 TeV (c.o.m.)	100 TeV (c.o.m.)
1'300 tons NbTi	~10'000 tons Nb_3Sn

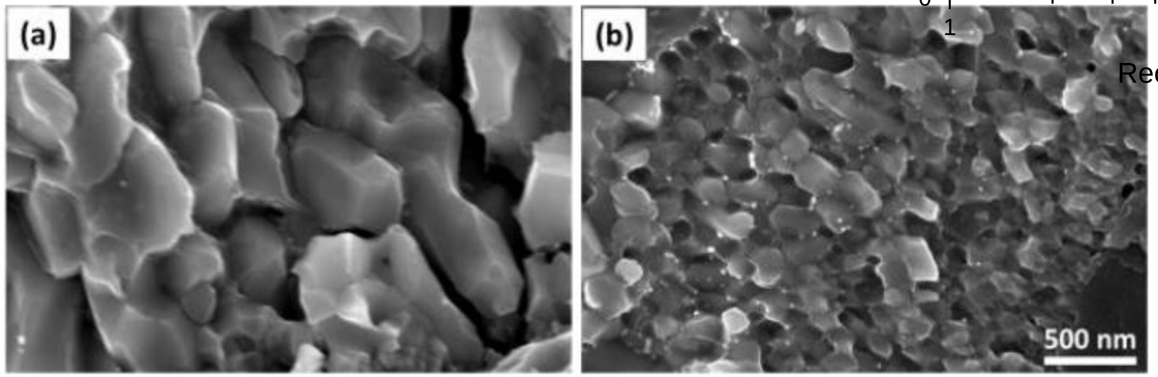
B [T]	16	16
J_{op} [A/mm^2]	300	2x → 600
w [mm]	76	38
A_{coil} [mm^2]	20'000	7'000



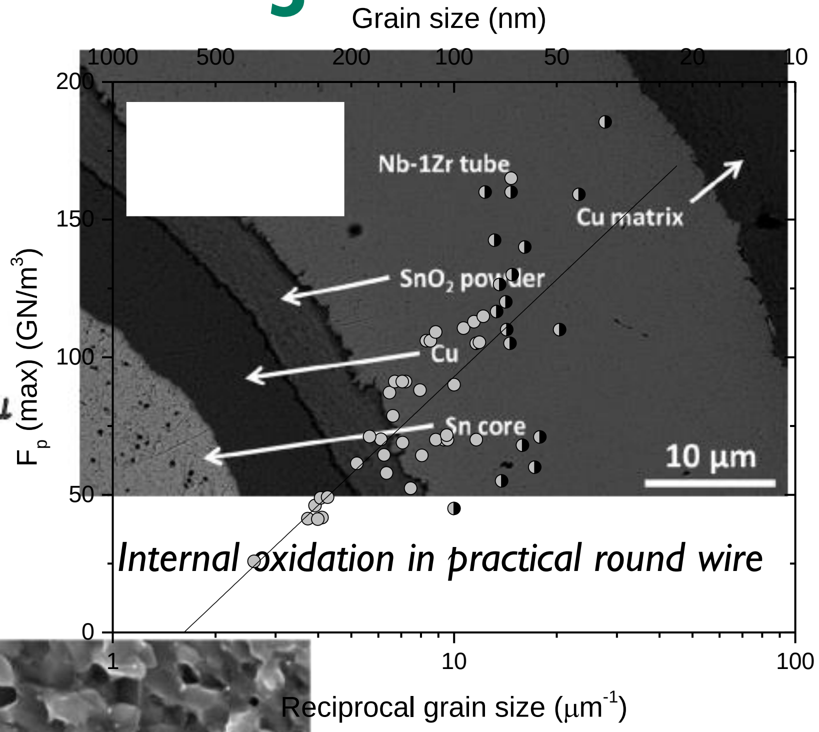
Doubling the operating current density brings a reduction of the superconductor area to one third



First observation of combined effect of Zr and O to reduce grain size



Grain refinement induced by ZrO_2 precipitation

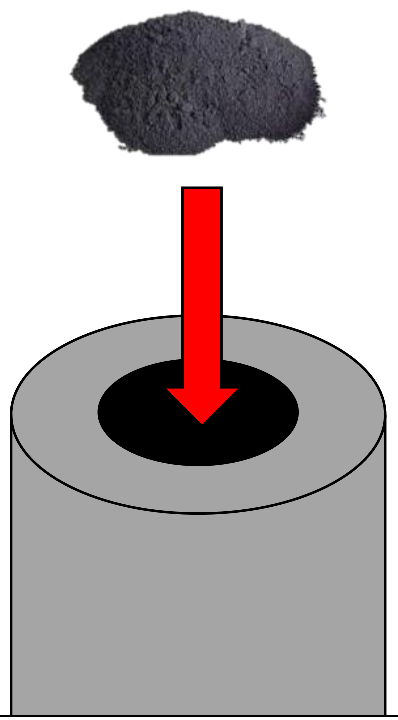


Internal oxidation in practical round wire

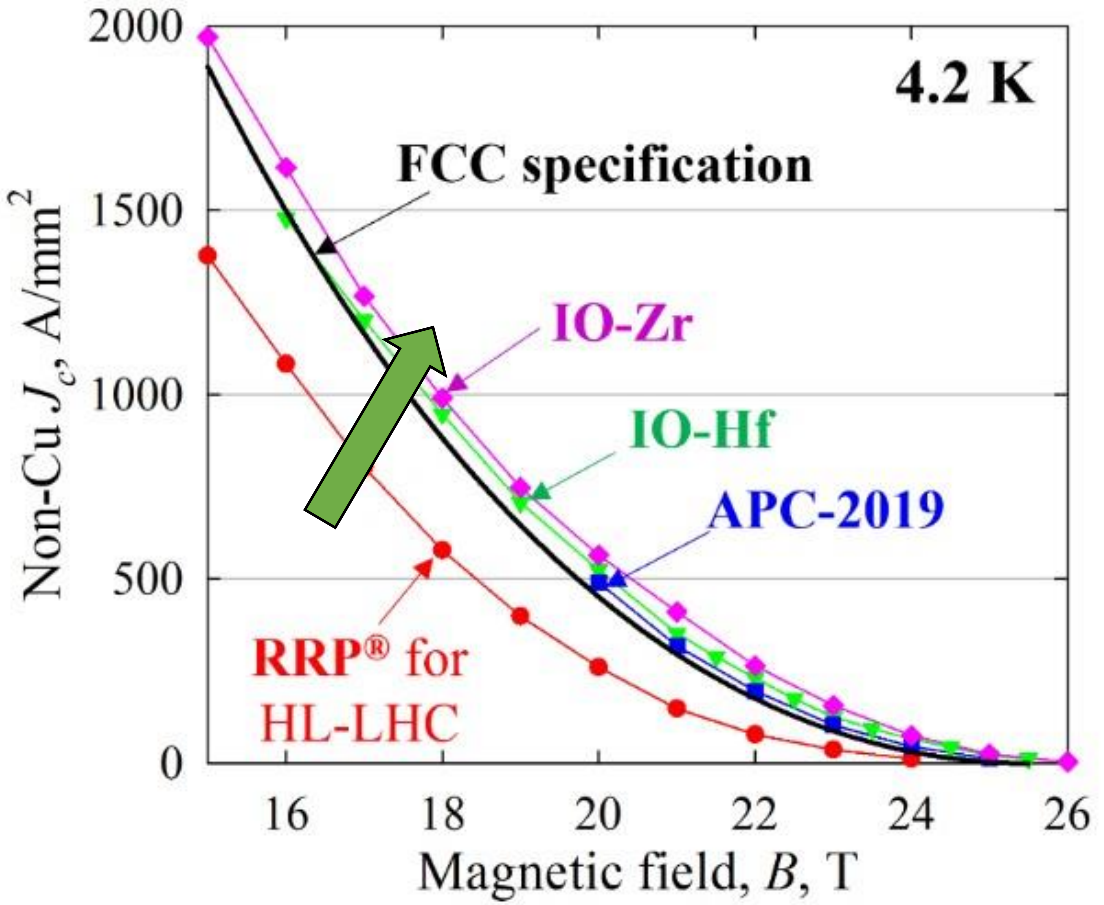
- Benz M G, *Trans. Met. Soc. AIME, 242: 1067-70* (1968).
- Rumaner L E, Benz M G, and Hall E L, *Metall Mater Trans A 25, 213–219* (1994) **DOI:** 10.1007/BF02646689.
- Xu X et al.; *Appl. Phys. Lett. 104,* 082602 (2014) **DOI:** 10.1063/1.4866865.

State of the art of internally oxidized wires

Mix of Cu, Sn and
SnO_2 powders

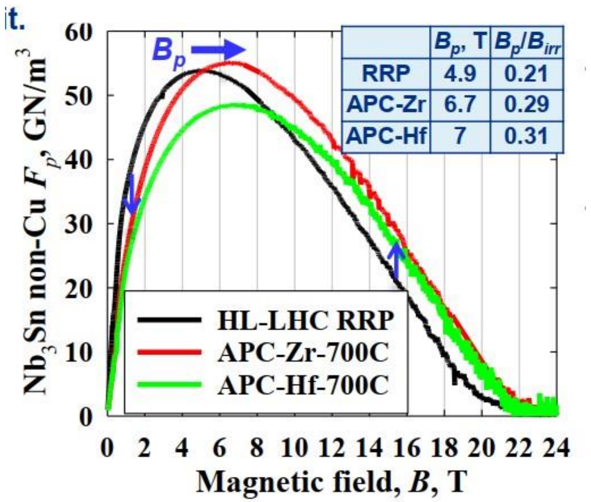


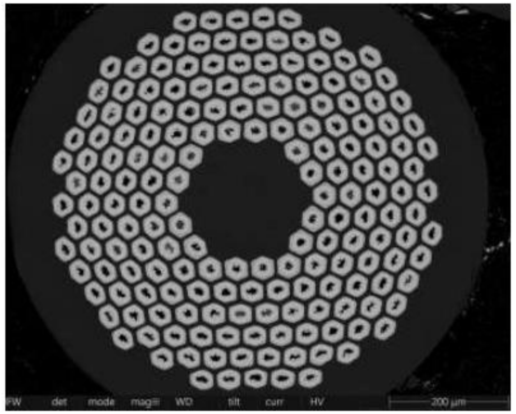
Alloys for tube:
Nb, 7.5% ^{wt} Ta, 1% ^{wt} ***Zr***
Nb, 7.5% ^{wt} Ta, 2% ^{wt} ***Hf***



Tubes with powders restacked into a 48/61 wire

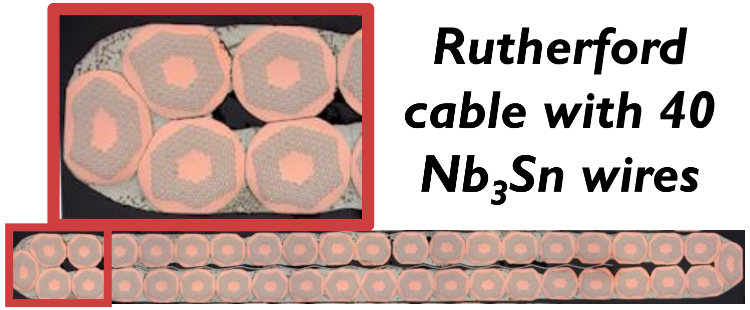
- Xu X et al. *Supercond. Sci. Technol. 36 085008 (2023)* **DOI:** 10.1088/1361-6668/acdf8c.
- Xu X et al. *Supercond. Sci. Technol. 36* 035012 *(2023)* **DOI:** *10.1088/1361-6668/acb17a*



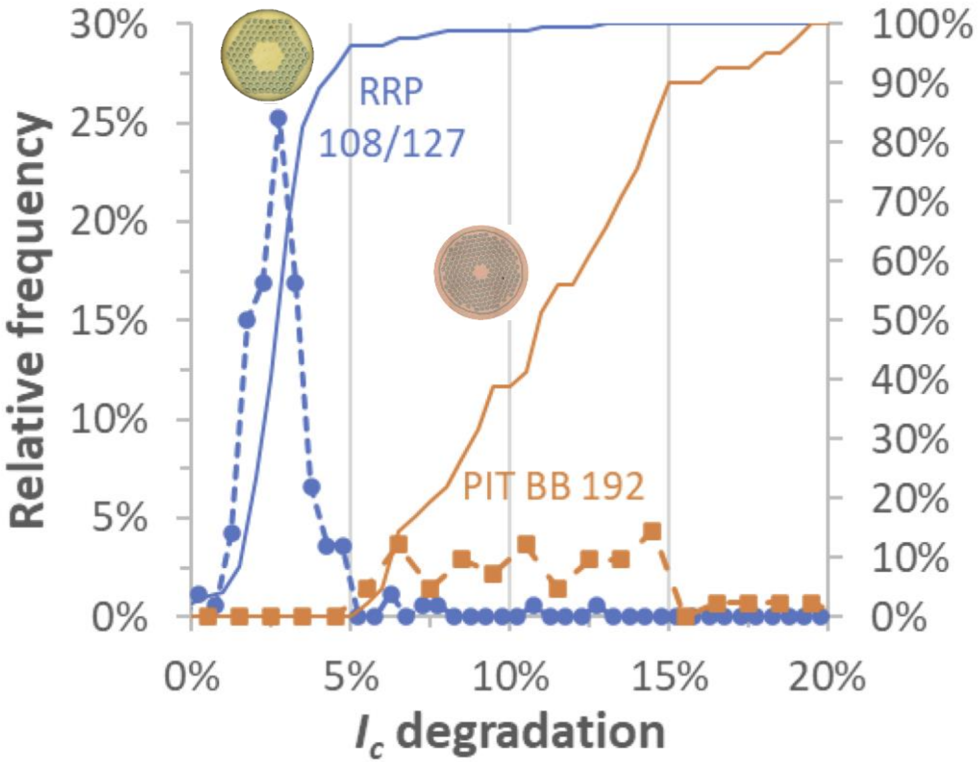


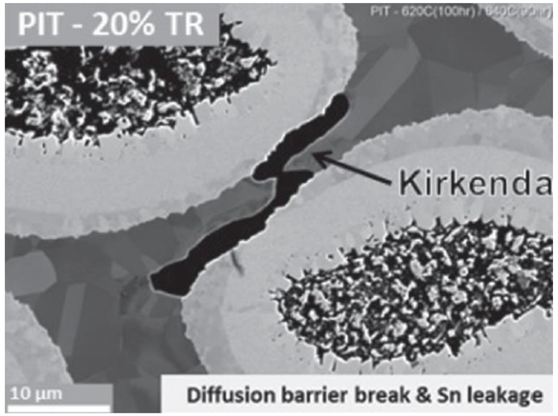
180/217 restacked wire
D_{fil} *= 38 μm*

Manufacturing process: RRP vs PIT



Rutherford cable with 40 Nb_3Sn wires





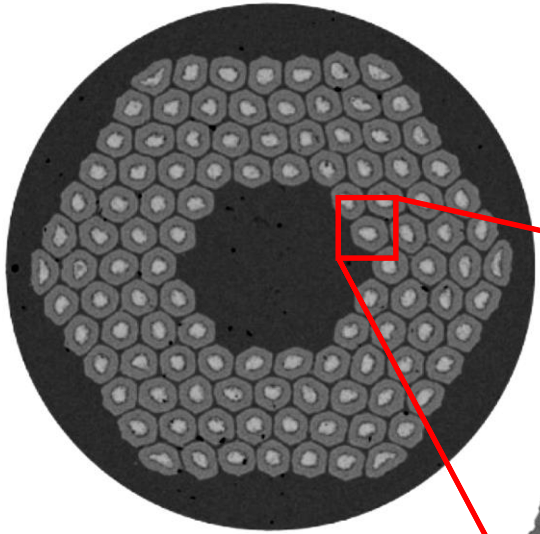
Distorted filaments produce Sn leak and thus are only partly reacted, leading to the lower I_c

Section of a Nb_3Sn dipole magnet

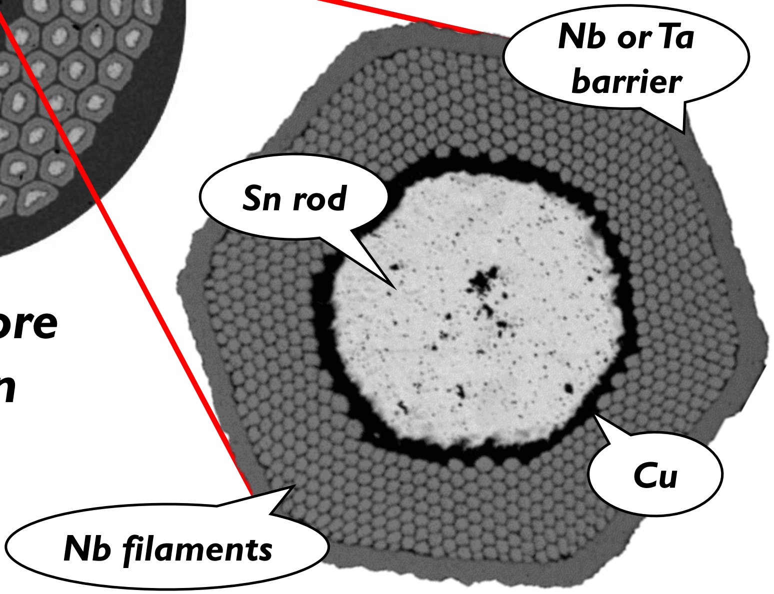
Cabling degradation is much less pronounced in Internal Sn RRP wires compared to PIT wires

- S. Hopkins et al., IEEE Trans. Appl. Supercond., 34 (2024) 6001308 **DOI:** *10.1109/TASC.2024.3375274*
- M. Brown et al., Supercond. Sci. Technol., 29 (2016) 084008 **DOI:** *10.1088/0953-2048/29/8/084008*
- C. Segal et al., Supercond. Sci. Technol., 29 (2016) 085003 **DOI:** *10.1088/0953-2048/29/8/085003*

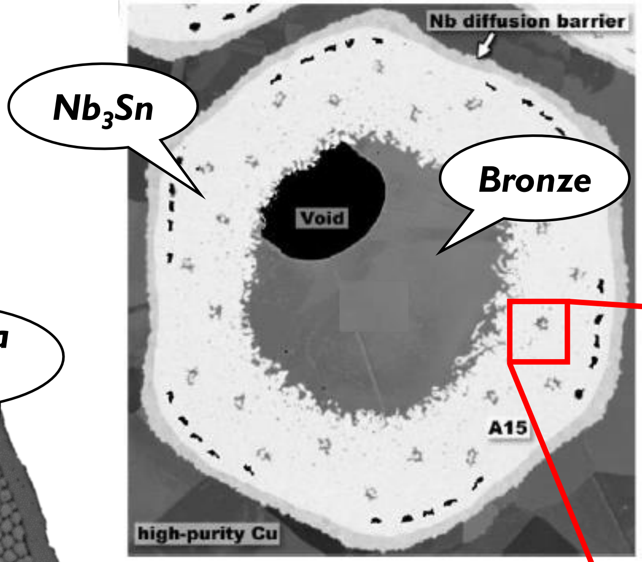
How it’s made: high-J_c internal Sn Nb_3Sn wire



Wire before reaction

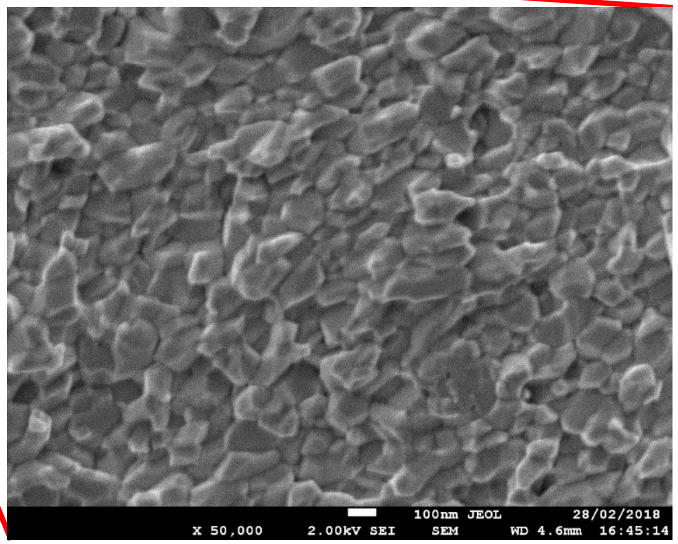


Subelement



Subelement after reaction

No powder in the wire layout! Where should we add the OS?



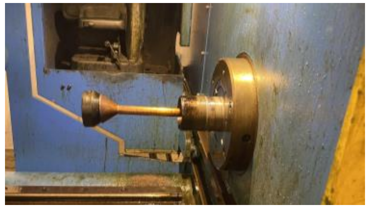
Nb_3Sn microstructure

Critical current density $\propto 1/(\text{grain size})$

How it's made: high-J_c internal Sn Nb_3Sn wire

Filament preparation

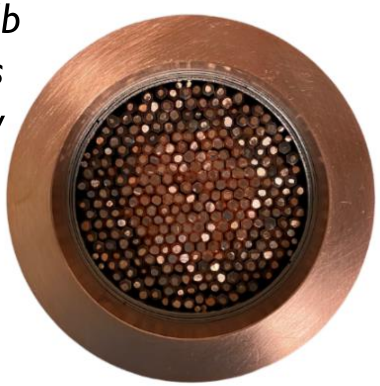
Filament extrusion



Cold deformation

Subelement preparation

Cu and Nb filaments assembly

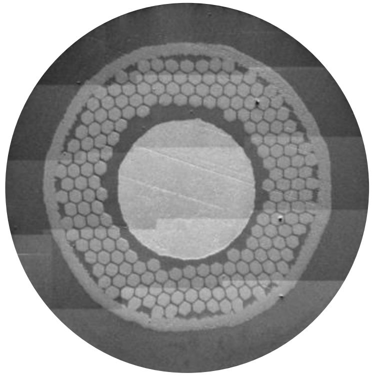


Billet preparation

Hot Isostatic Press

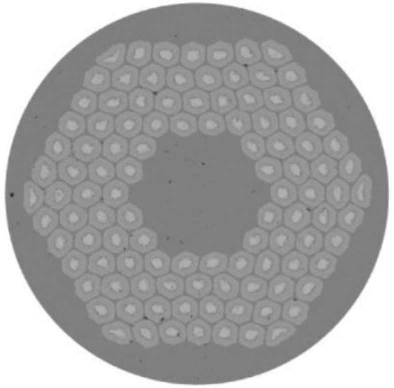
Hot Extrusion

Sn rod insert



Cold deformation to reach restack size

Wire assembly



Restack of the sub-element in a Cu tube, and cold deformation to final wire size

The goals of our project at UNIVERSITÉ DE GENÈVE

FACULTÉ DES SCIENCES

- *Develop a process to introduce oxide powder in an internal Sn rod-type wire to refine Nb_3Sn grains*

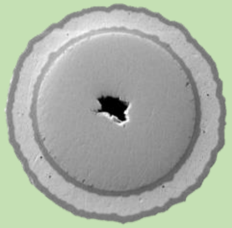
- *Produce long prototype Nb_3Sn wires (unit lengths <u>> 20 m</u>), matching the FCC targets for critical current density*

- *Optimize the process for scaling up to an industrial production*

The three steps to get there:

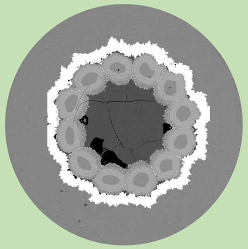
1. *Monofilamentary wires: Material study*

   Test of various alloys and oxides (and their combinations)

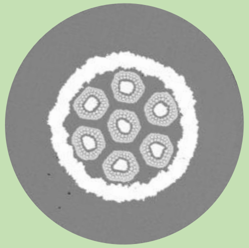


2. *Simplified subelements: Current transport*

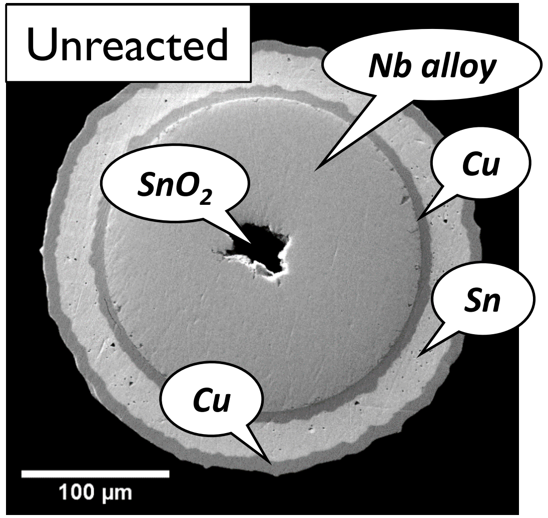
   Deformation process, filaments arrangement and oxygen source configurations

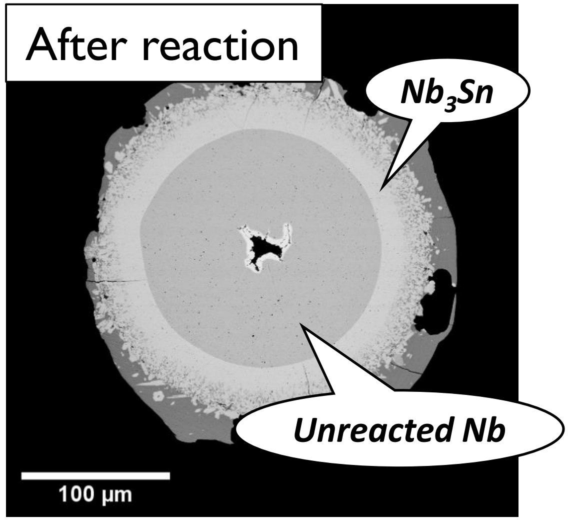


3. *Development of multifilamentary internal Sn rod-type wires with internal oxidation!*



Monofilamentary wires





Wire configuration

Nb-7.5wt%Ta (REF.)
Nb-1wt%Zr + SnO_2
Nb-7.5wt%Ta-1wt%Zr + SnO_2
Nb-7.5wt%Ta-2wt%Zr + SnO_2

Pronounced grain refinement in Zr containing samples

PAPER

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Very high upper critical fields and enhanced critical current densities in Nb_3Sn superconductors based on Nb–Ta–Zr alloys and internal oxidation

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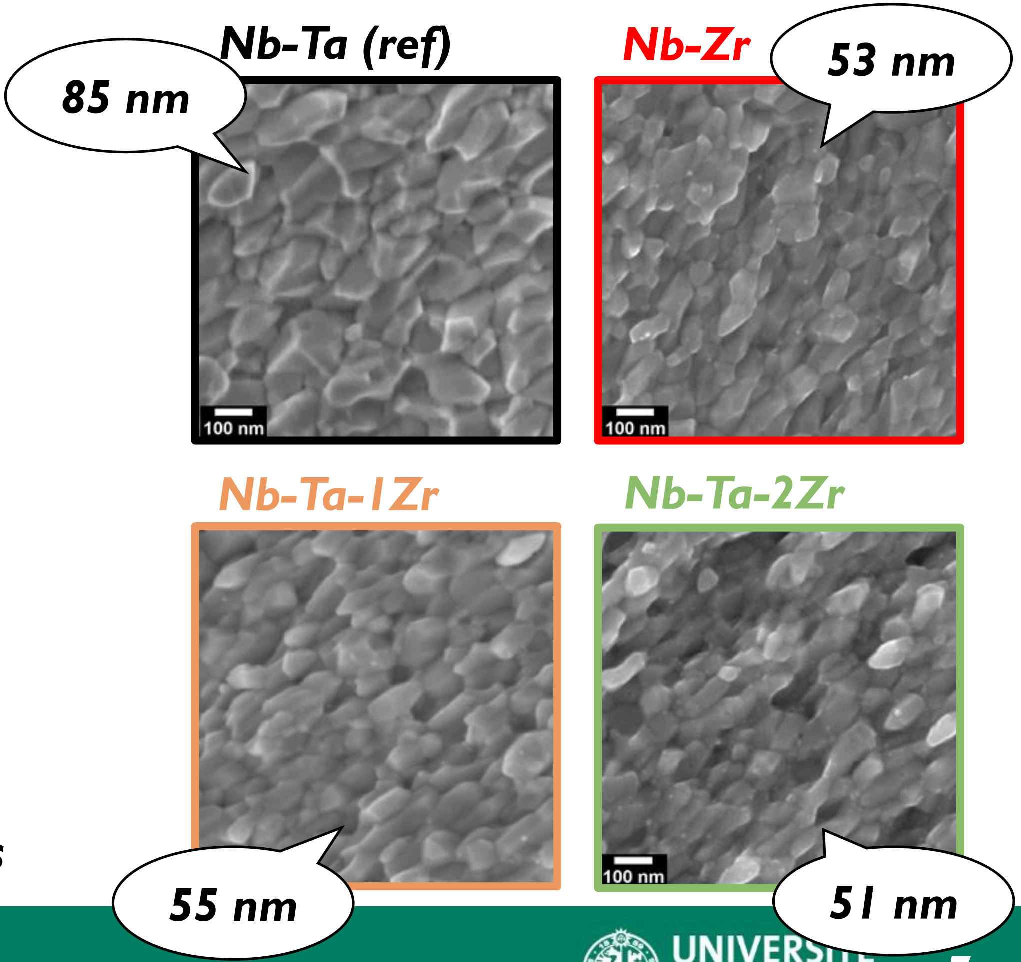
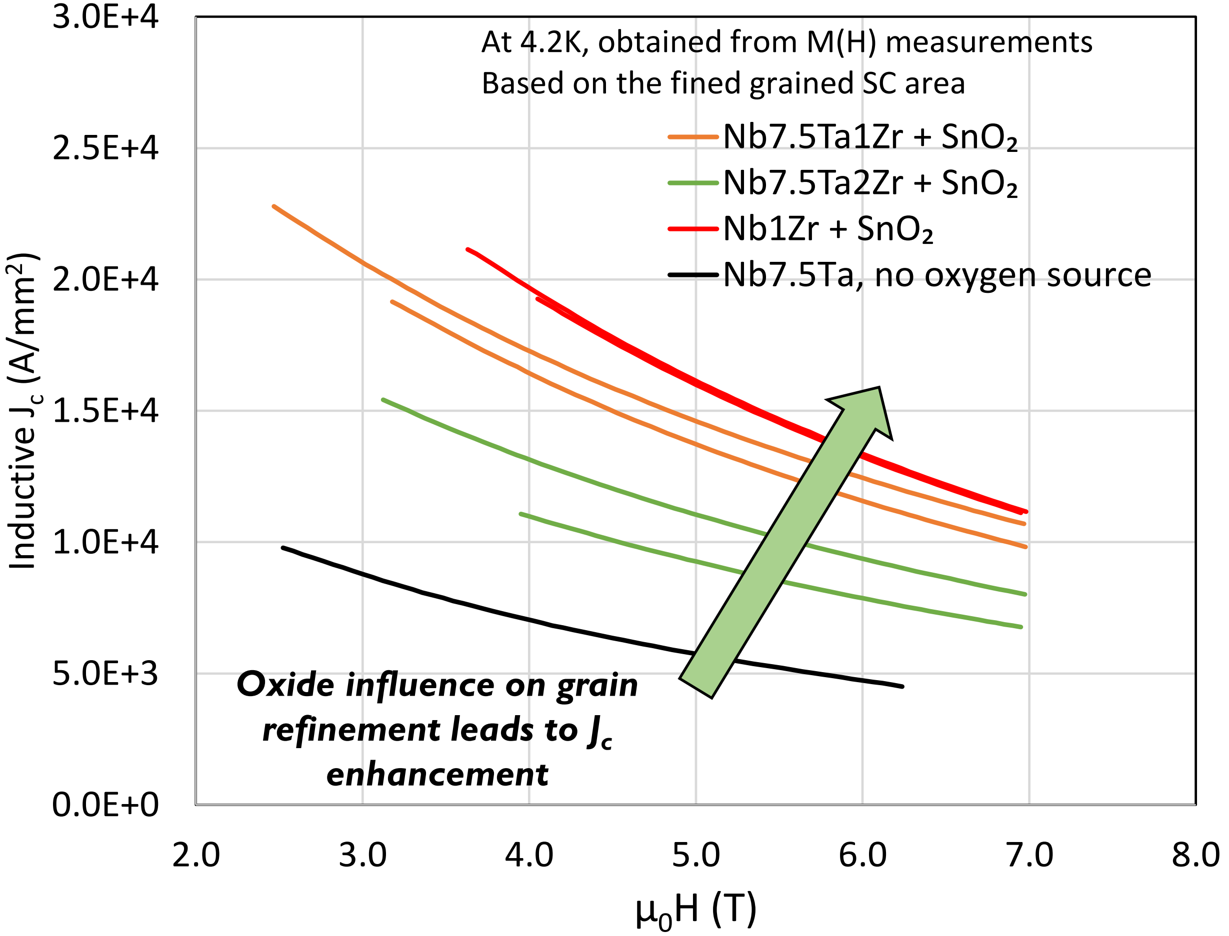
[3] French National High Magnetic Field Laboratory, Grenoble, France

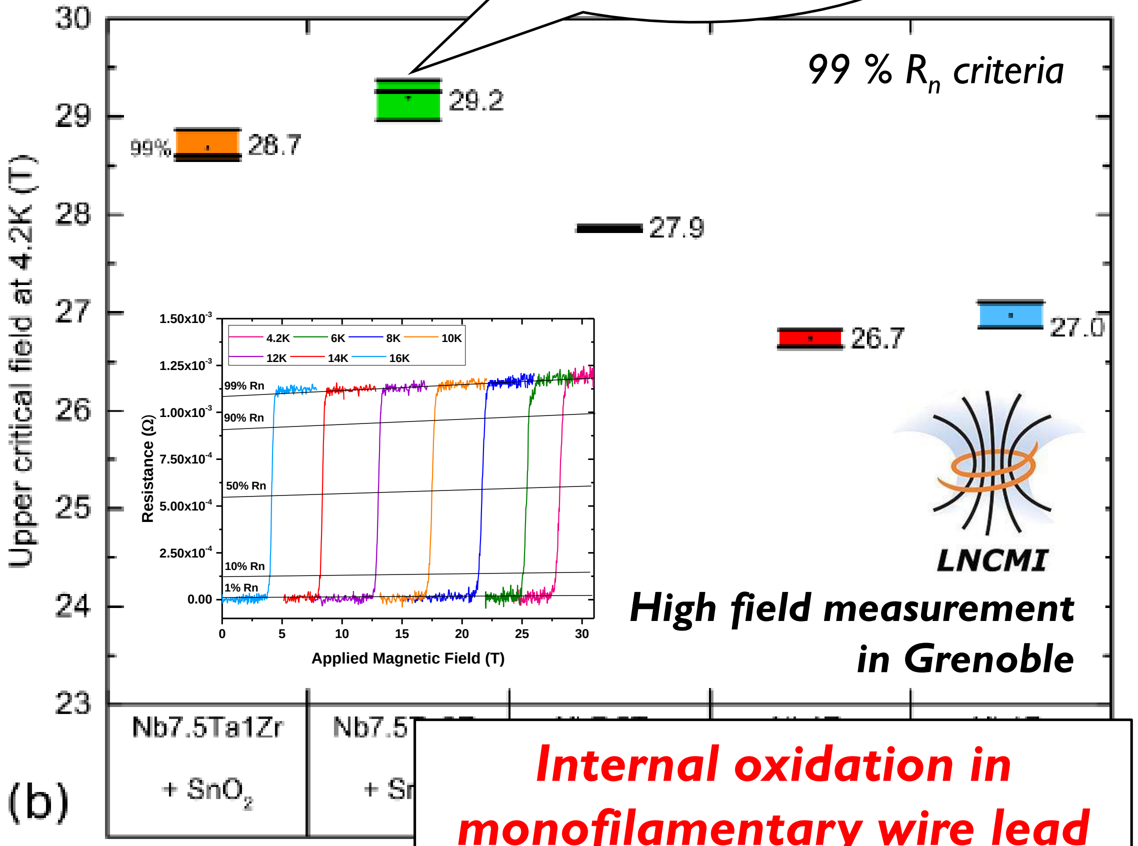
DOI: 10.1088/2515-7639/abe662



Monofilamentary wires

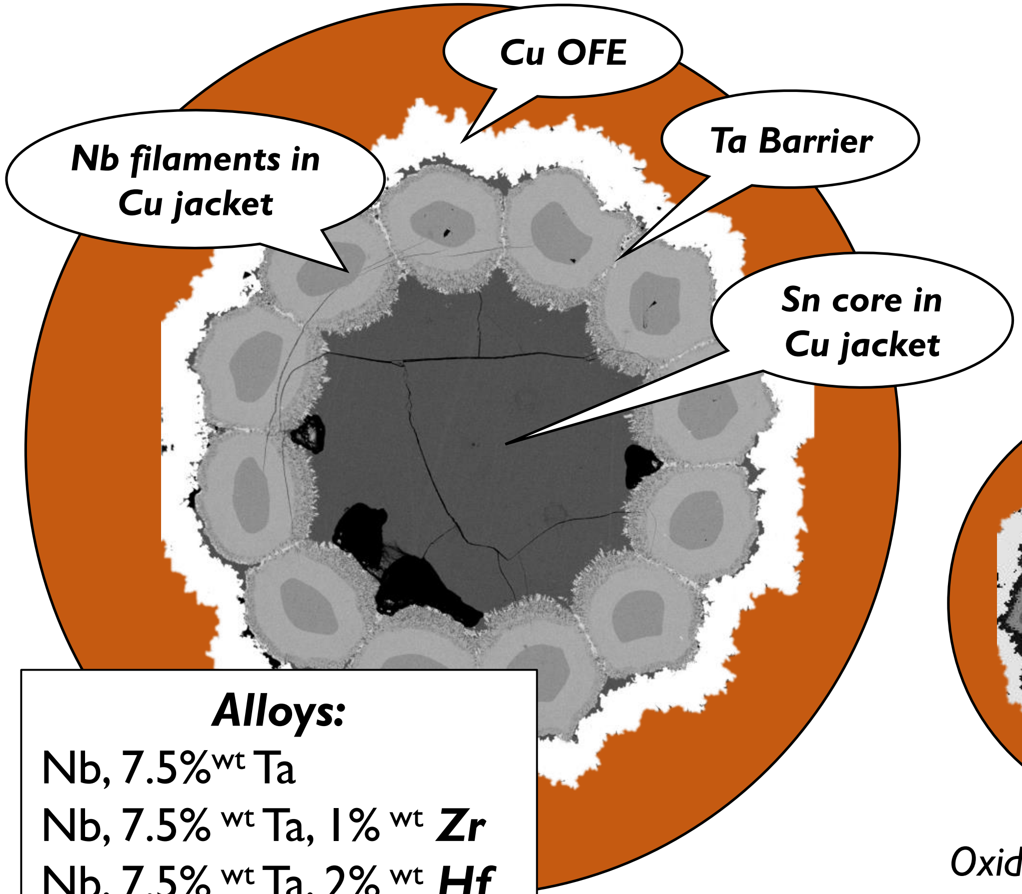


Upper Critical Field is drastically enhanced



Internal oxidation in monofilamentary wire lead to grain refinement, enhanced J_c and B_{c2}

Simplified multifilamentary wires layout and fabrication process



Alloys:

Nb, 7.5%^{wt} Ta

Nb, 7.5% ^{wt} Ta, 1% ^{wt} ***Zr***

Nb, 7.5% ^{wt} Ta, 2% ^{wt} ***Hf***

Effects of the oxygen source configuration on the superconducting properties of internally-oxidized internal-Sn Nb_3Sn wires

G Bovone[1,*], F Buta[1], F Lonardo[1], T Bagni[1], M Bonura[1], D LeBoeuf[2], S C Hopkins[3], T Boutboul[3], A Ballarino[3] and C Senatore[1]

[1] University of Geneva, DQMP, Geneva, Switzerland
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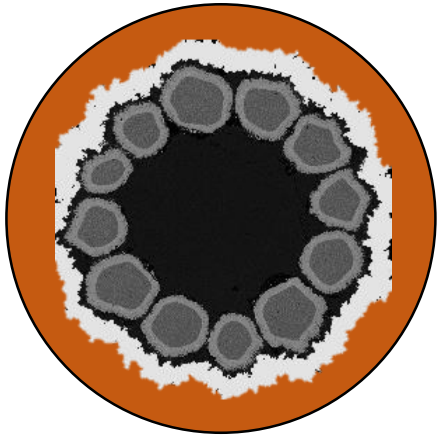
[3] European Organization for Nuclear Research CERN, Geneva, Switzerland

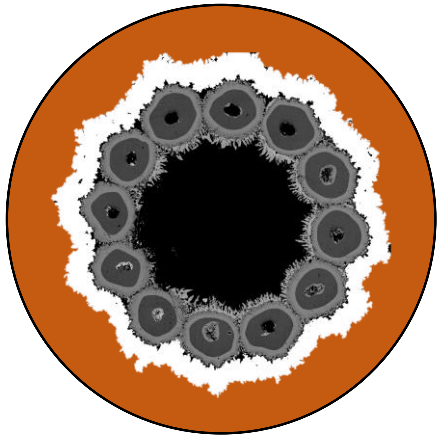
DOI: *10.1088/1361-6668/aced25*

AnnularOS



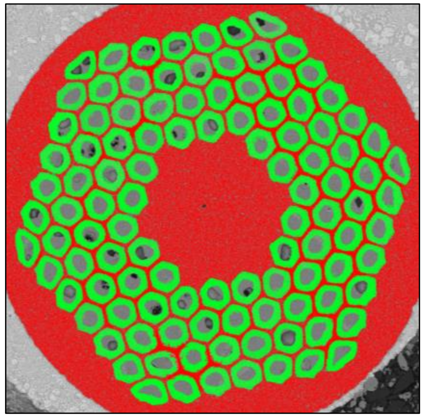
Oxide powder between the Nb-alloy filament and the copper jacket

CoreOS

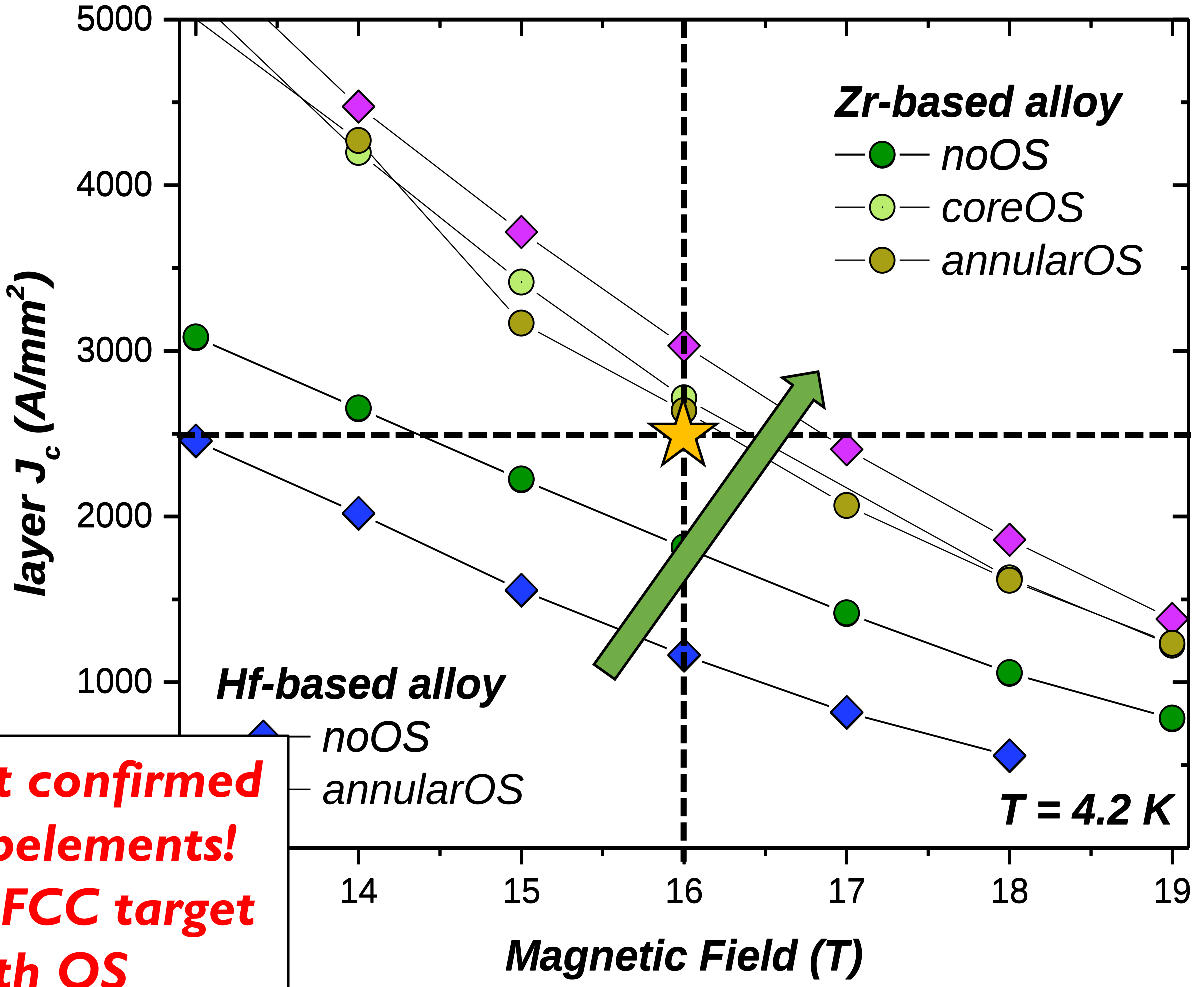


Oxide powder inside the Nb-alloy filaments

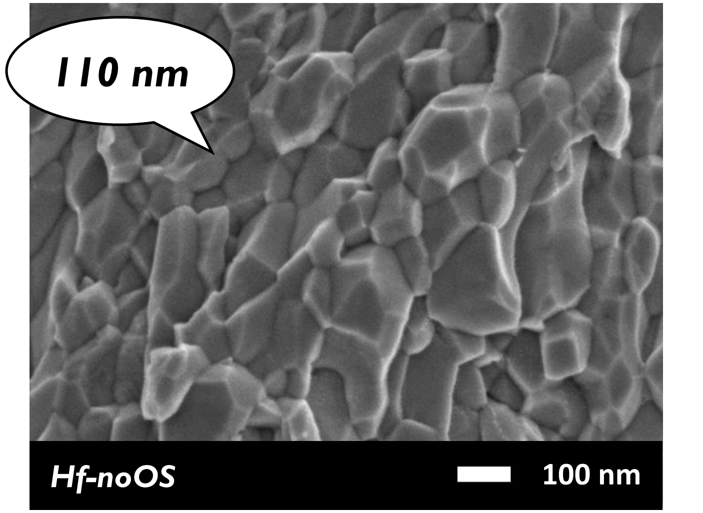
Effects of the internal oxidation on the superconducting properties

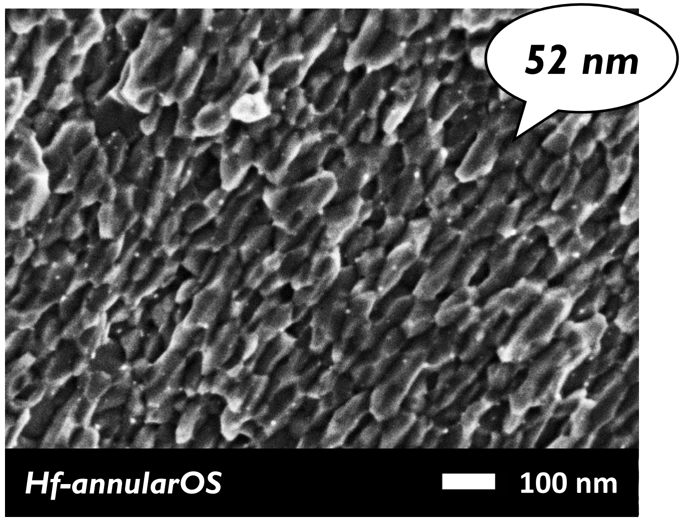


The FCC target for layer J_c is calculated based on the area fraction of reacted Nb_3Sn in RRP wire for HIlumi

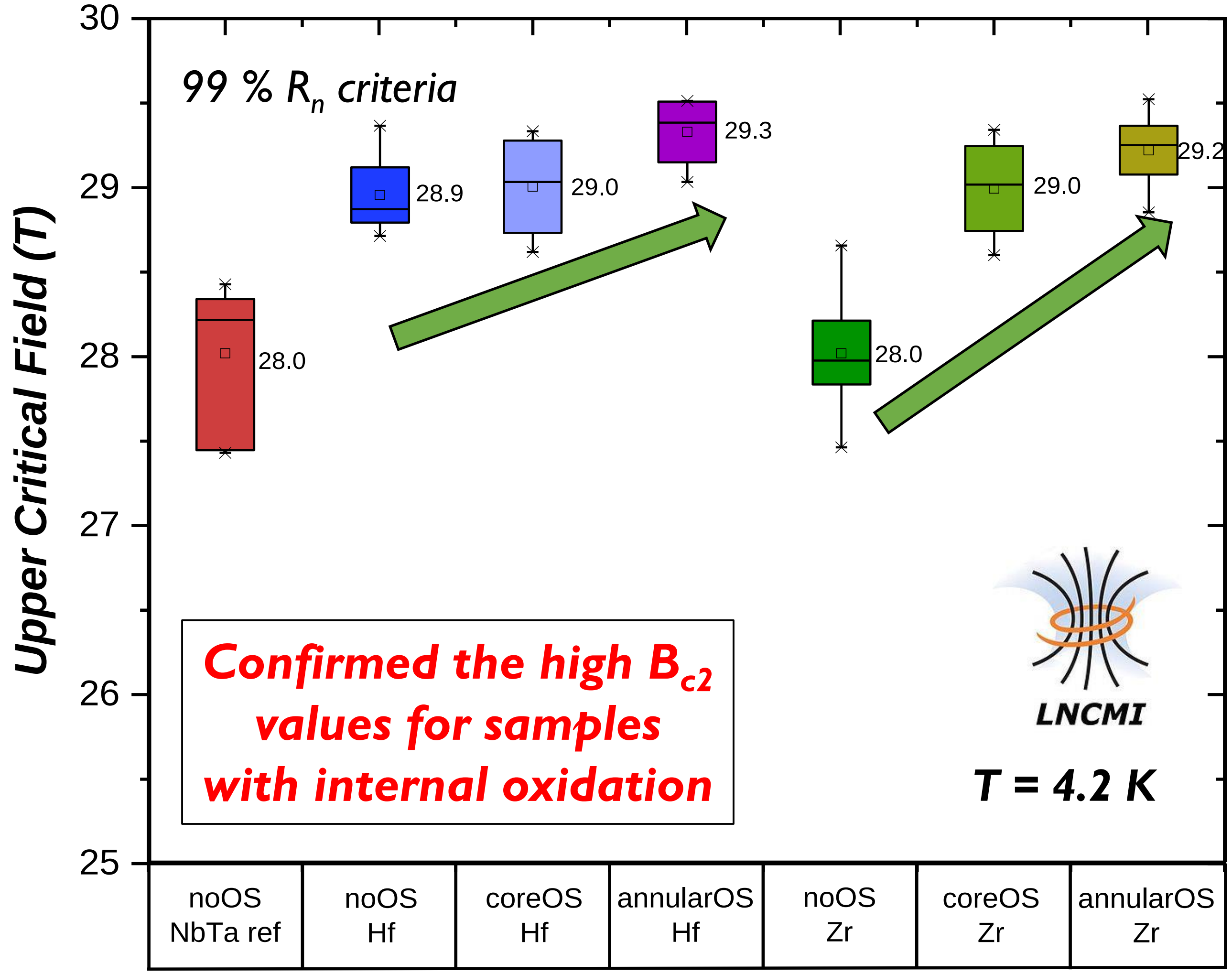


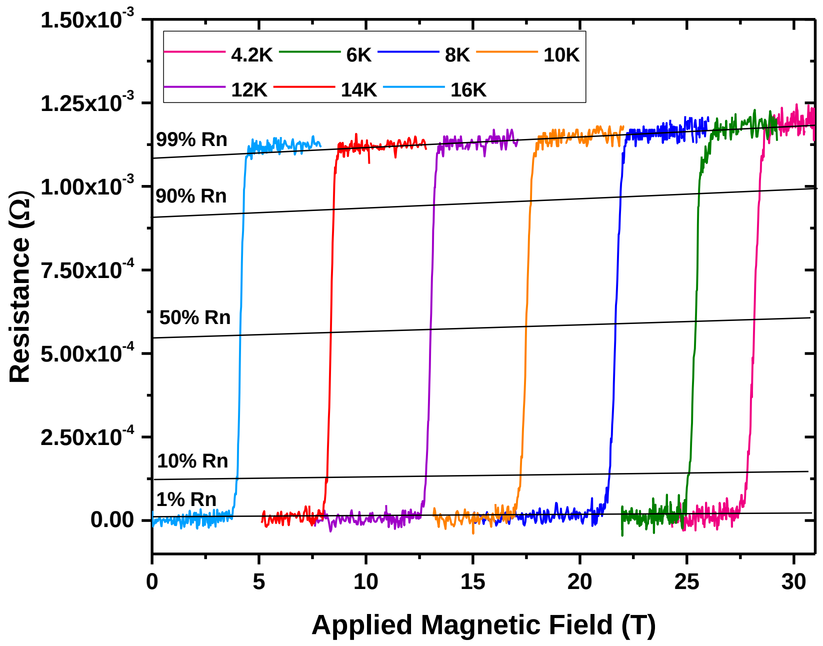
Grain refinement confirmed in simplified subelements!
Layer-J_c beyond FCC target in wires with OS





B_{c2} enhancement

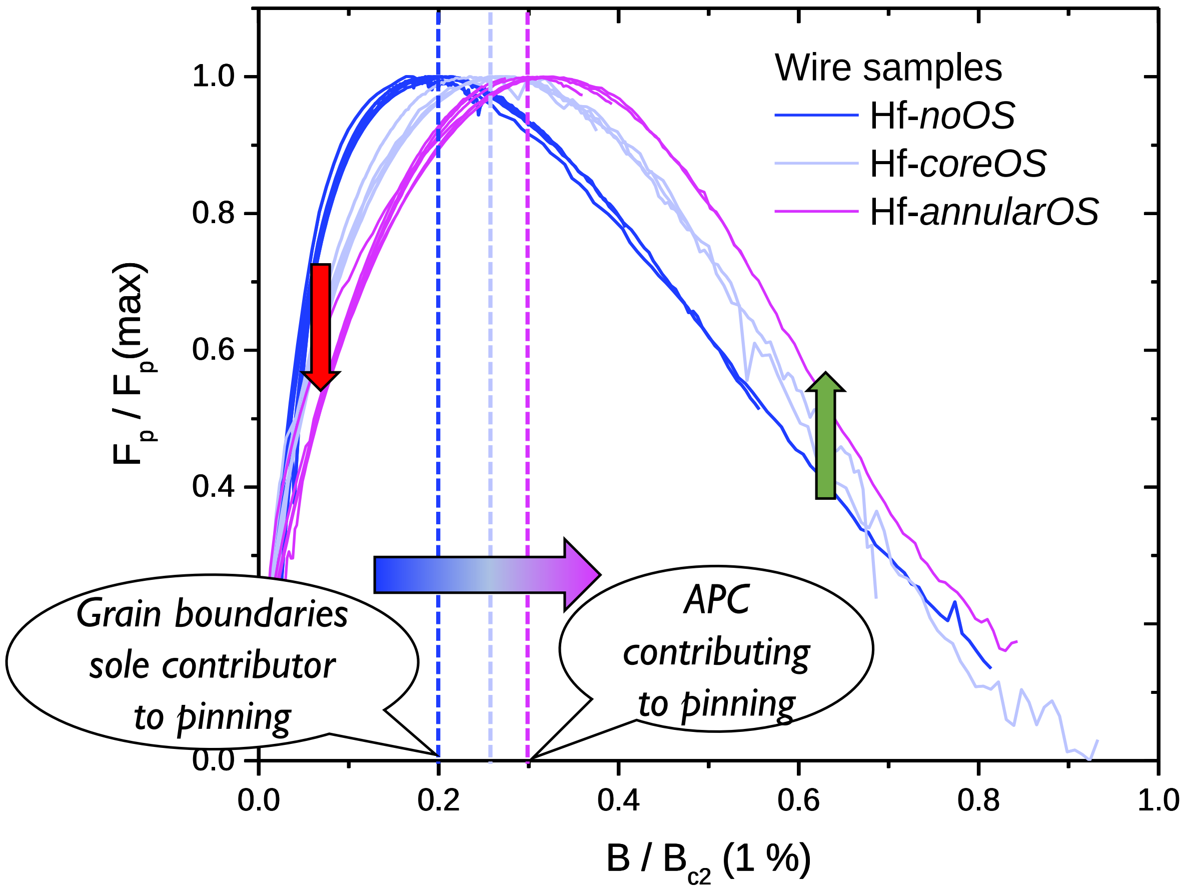




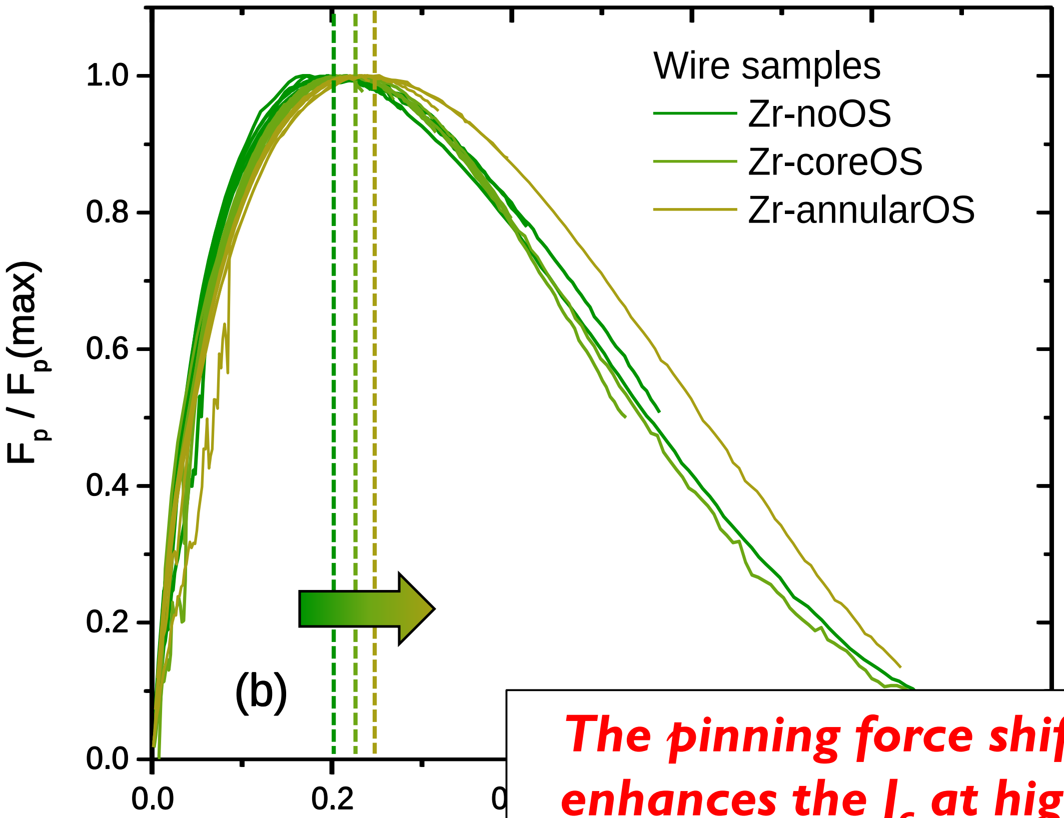
Nb alloy	*B_{c2} (T) 4.2 K, 99 % R no-OS*	*B_{c2} (T) 4.2 K, 99 % R Core-OS*	*B_{c2} (T) 4.2 K, 99 % R Annular-OS*
Nb 7.5%^{wt} Ta 1%^{wt} Zr	***28.0 ± 0.6***	***28.97 ± 0.4***	***29.2 ± 0.4***
Nb 7.5%^{wt} Ta 1%^{wt} Hf	***28.9 ± 0.6***	***29.0 ± 0.4***	***29.3 ± 0.4***
Nb 7.5%^{wt} Ta	***28.0 ± 0.8***	***N/A***	

Precipitates and pinning contribution

$F_p = J_c \times B$



Change of dominant pinning mechanism *induced by the presence of* ***oxide precipitates****, Artificial Pinning Centers (APC), Shift with NbTaHf-alloy is larger*



The pinning force shift enhances the J_c at high magnetic field, while reducing magnetization at low magnetic field

XANES investigation on precipitates

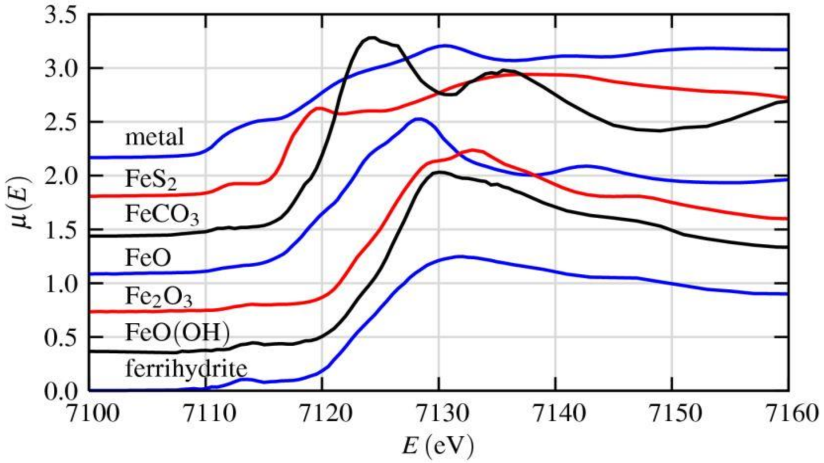
IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 34, NO. 3, MAY 2024 6000205

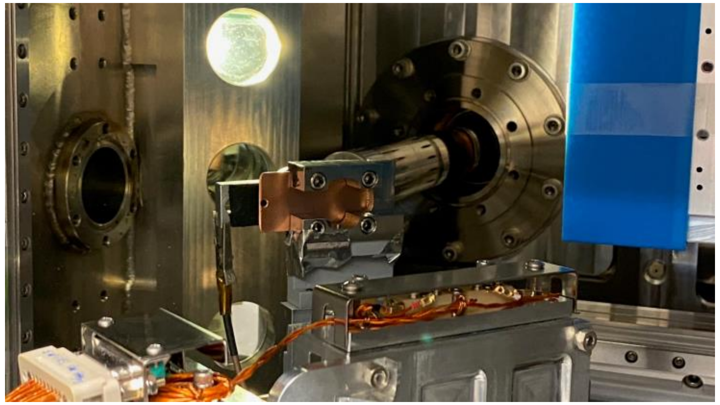
X-Ray Absorption Spectroscopy to Investigate Precipitated Oxides in Nb_3Sn Wires With an Internal Oxygen Source

G. Bovone, F. Buta, F. Lonardo, M. Bonura, C. N. Borca, T. Huthwelker, S. C. Hopkins, A. Ballarino, T. Boutboul, and C. Senatore, *Senior Member, IEEE*

DOI: 10.1109/TASC.2024.3354232

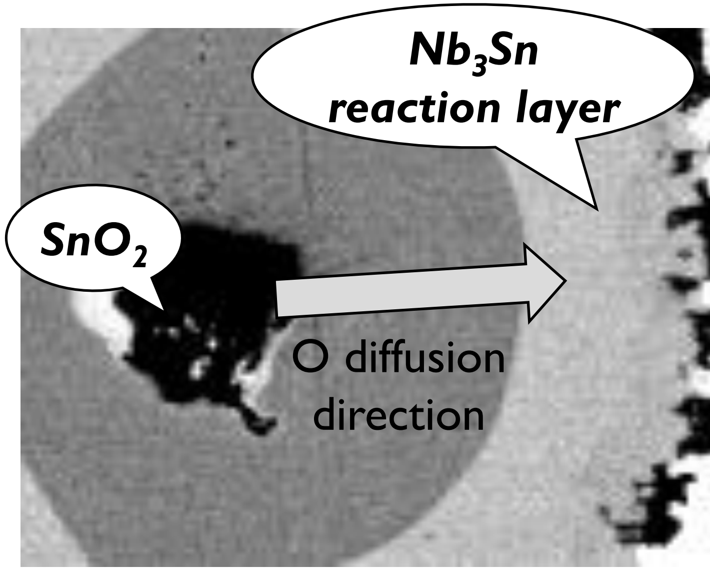
Atomic specific *technique, sensitive to the* ***chemistry*** *and to* ***crystal environment***





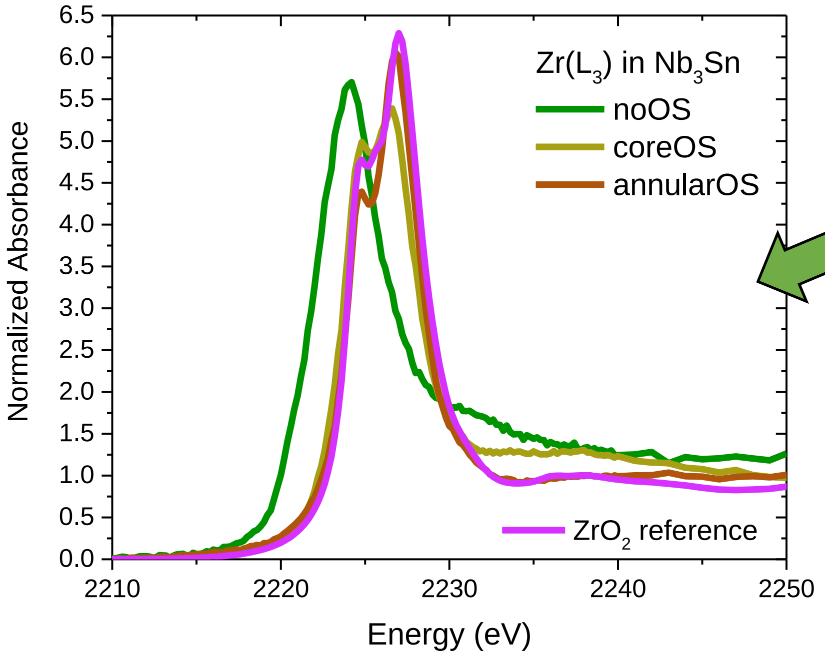
Phoenix Beamline

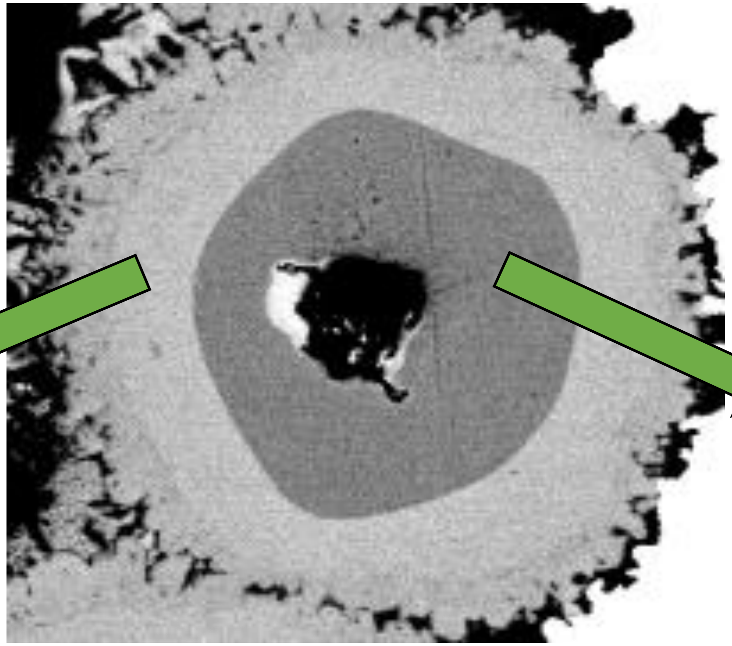
Investigation of ***Zr spectrum*** *in* ***different region*** *of the* ***reacted (and unreacted) wires***



What did we learn from XANES?

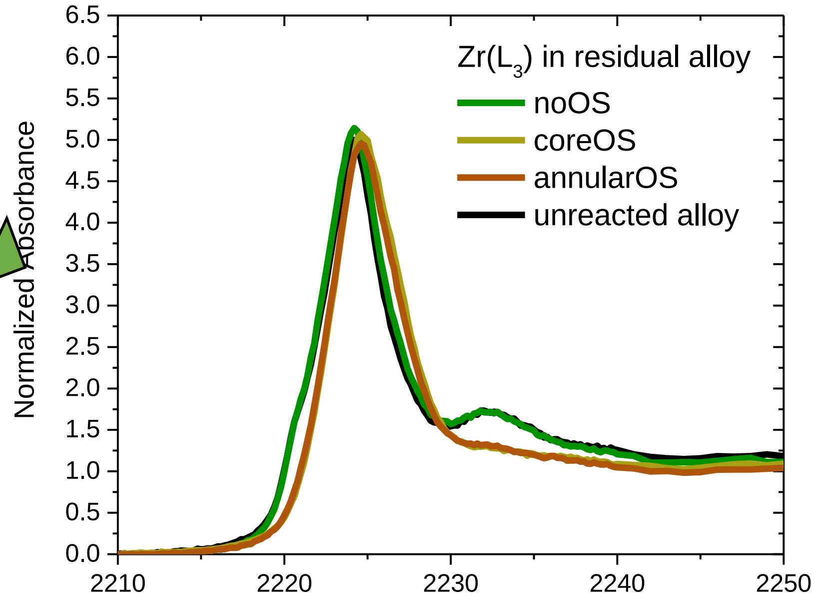
Zr in Nb_3Sn is fully oxidised to ZrO_2





ZrO_2 precipitates due to the lower solubility of Zr and O in Nb_3Sn compared to Nb

Zr in the residual alloy is not oxidized



ZrO_2–like spectrum found <u>only</u> in Nb_3Sn! Different Zr spectrum in residual alloy, despite oxygen diffusion

X-Ray Absorption Spectroscopy to Investigate Precipitated Oxides in Nb_3Sn Wires With an Internal Oxygen Source

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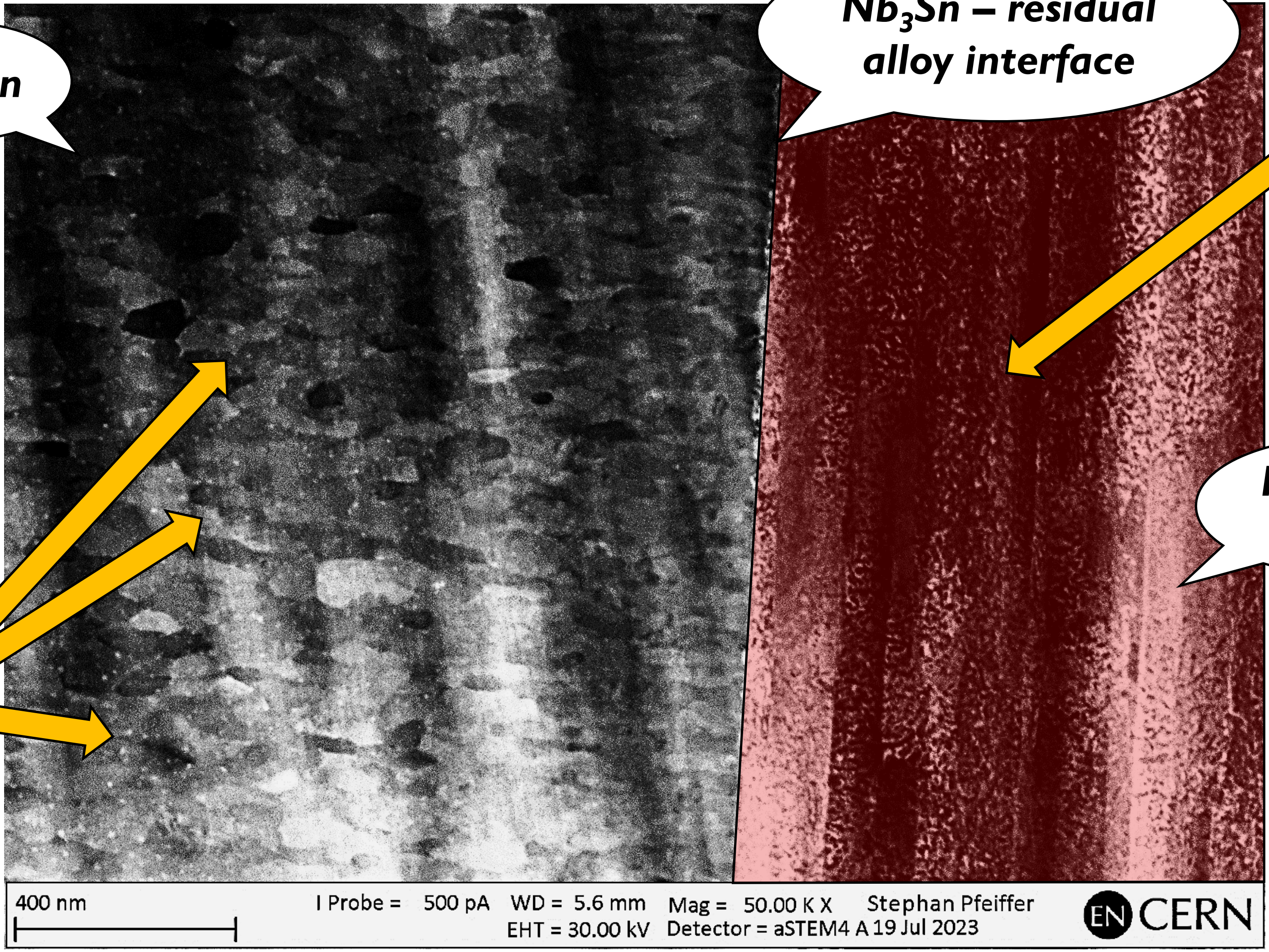
DOI: *10.1109/TASC.2024.3354232*

Precipitates at the interface



Uniform distribution of ZrO_2 precipitates

No observable precipitates and no ZrO_2 fingerprint from XANES

Courtesy of Stephan Pfeiffer, CERN

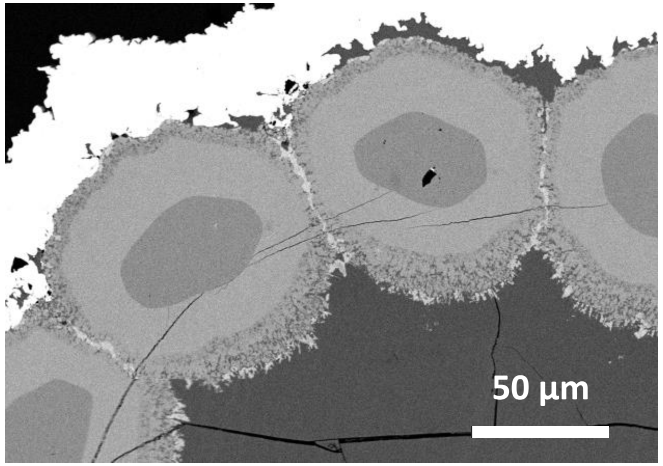
- Hall E L et al. *MRS Online Proceedings Library (OPL)*, Volume 205: *Symposium F – Kinetics of Phase Transformations*, 263 (1990) ***DOI:*** [*10.1557/PROC-205-263*](https://doi.org/10.1557/PROC-205-263)

Heat treatment optimization: reaction layer thickness

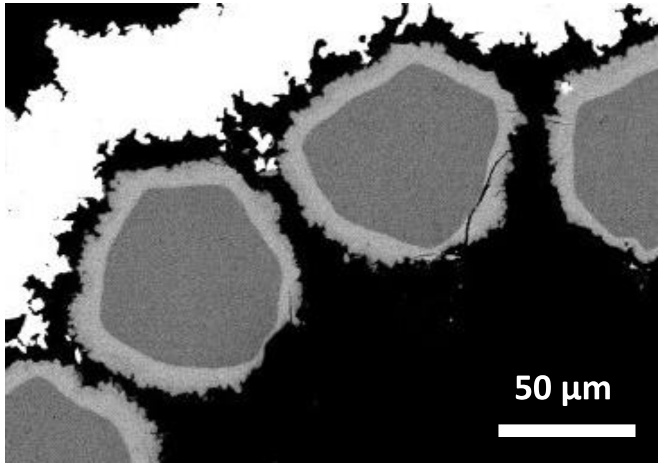
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS



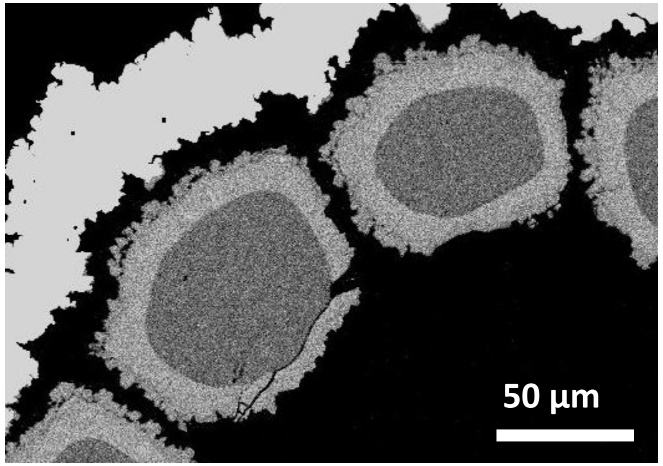
Introduction of OS reduces the Nb_3Sn reaction kinetics

Higher temperature can promote reaction, but we want small grain size!

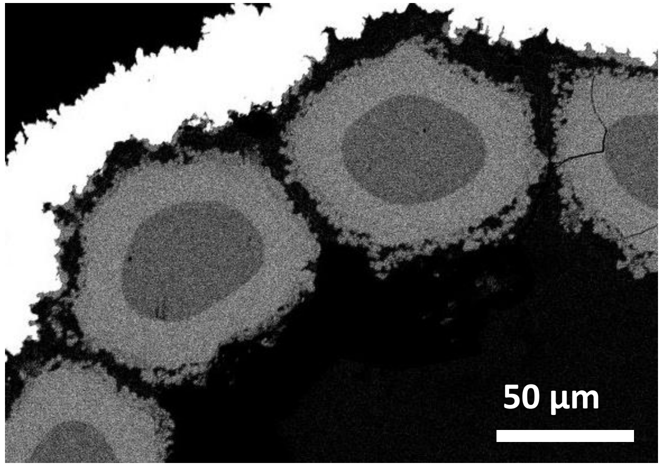
Significant increase of layer thickness at 700 °C

With OS

700 °C × 50 h



700 °C × 100 h

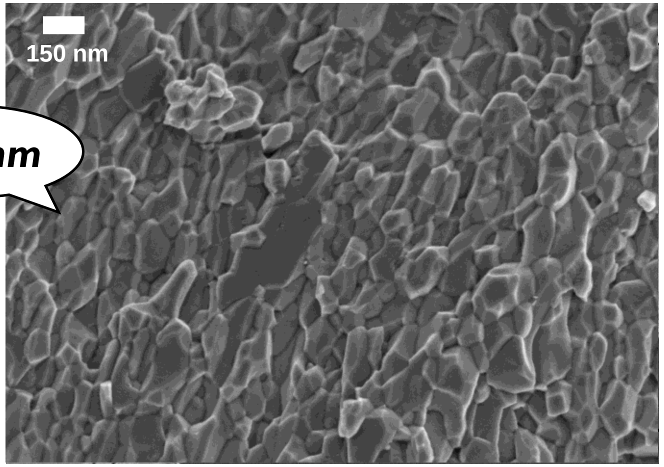


Heat treatment optimization: reaction layer thickness

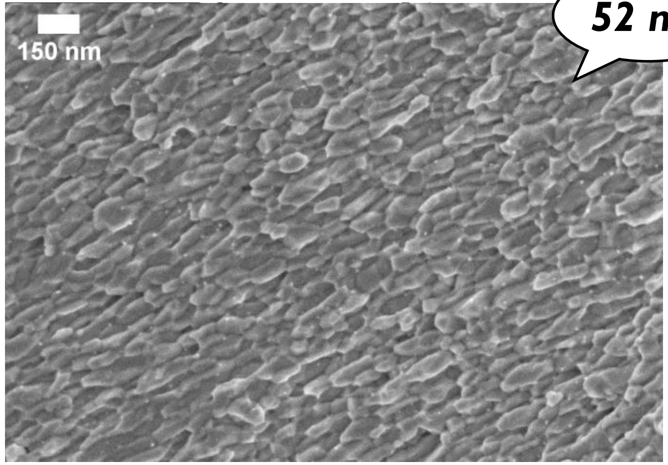
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



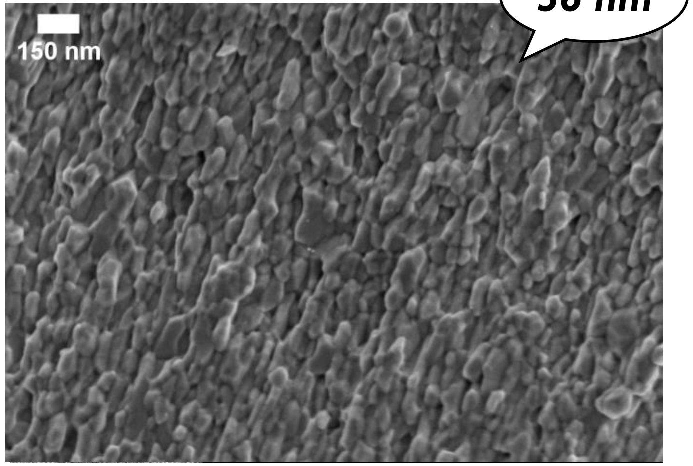
With OS



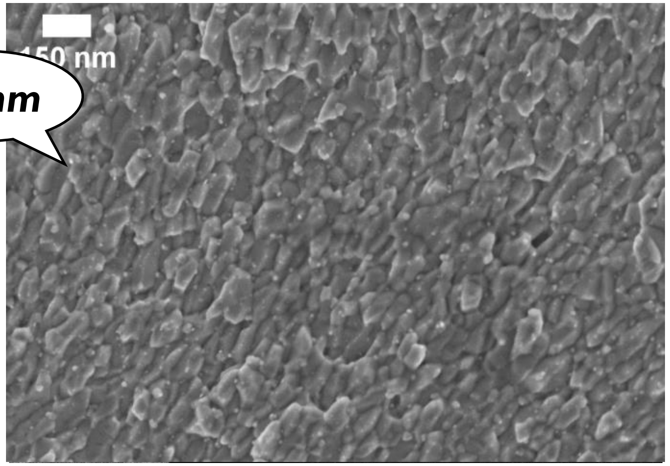
Introduction of OS reduces the Nb_3Sn reaction kinetics

With OS

700 °C × 50 h



700 °C × 100 h



Higher temperature can promote reaction, but we want small grain size!

Significant increase of layer thickness at 700 °C but modest grain growth

Heat treatment optimization: layer-J_c

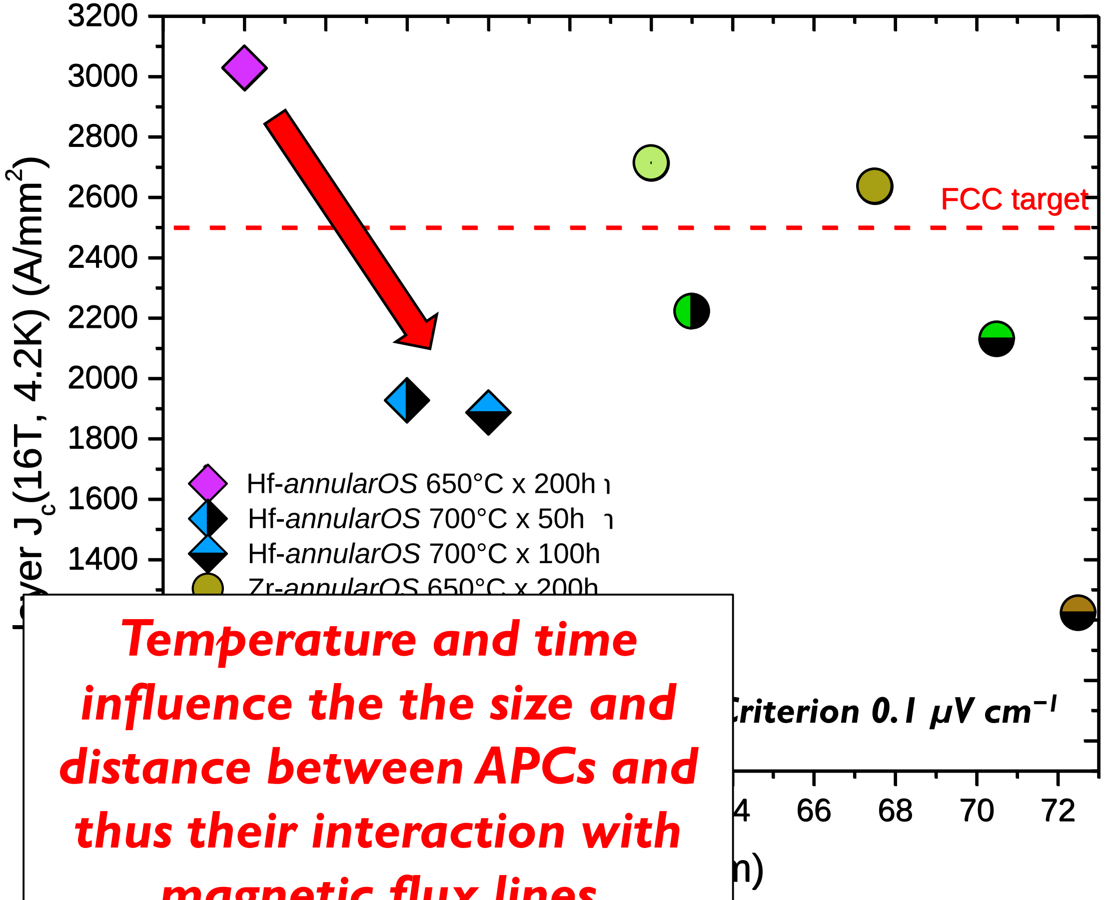
IEEE TRANSACTIONS ON APPLIED SUPERCONDUCTIVITY, VOL. 34, NO. 5, AUGUST 2024 6000305

Influence of the Heat Treatment on the Layer J_C of Internal-Sn Nb_3Sn Wires With Internally Oxidized Nanoparticles

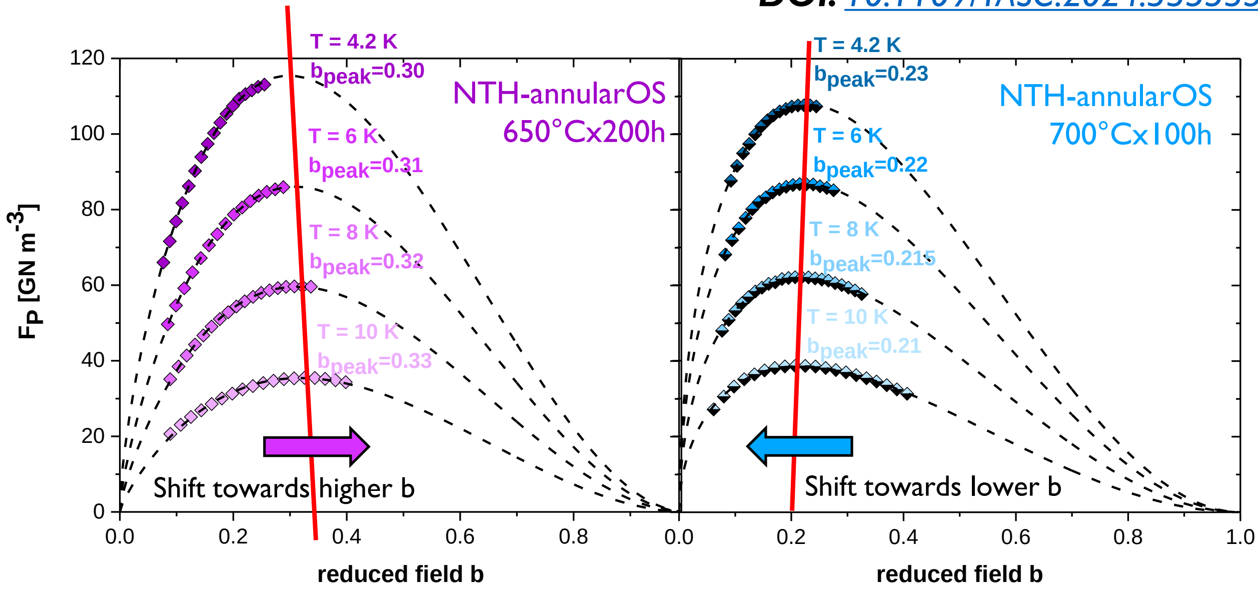
F. Lonardo, G. Bovone, F. Buta, M. Bonura, T. Bagni, B. Medina-Clavijo, A. Ballarino, S. C. Hopkins, T. Boutboul, and C. Senatore, *Senior Member, IEEE*

DOI: 10.1109/TASC.2024.3355353

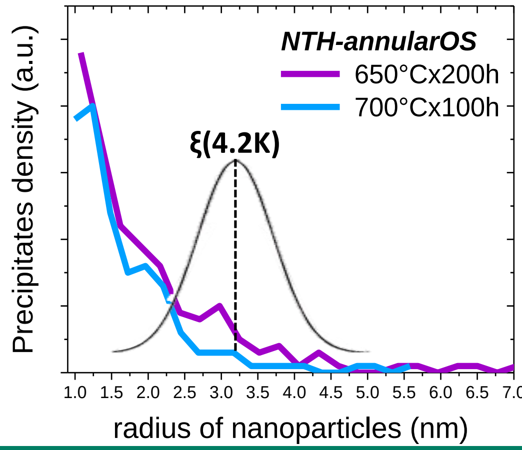
Why J_c is lower despite the small grain size?



Temperature and time influence the the size and distance between APCs and thus their interaction with magnetic flux lines

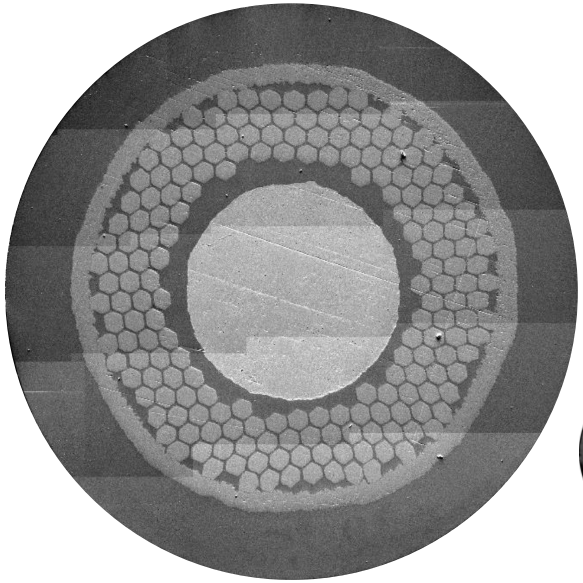


Sample reacted at higher temperature have lower precipitates density matching the size of flux lines at 4.2 K (and higher temperature)

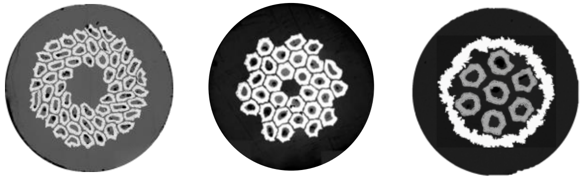


Getting back the knowledge from industry

Fabrication of RRP wires was mainly carried by industry. Few academic study on the production, we need to gather the knowledge and close the gap



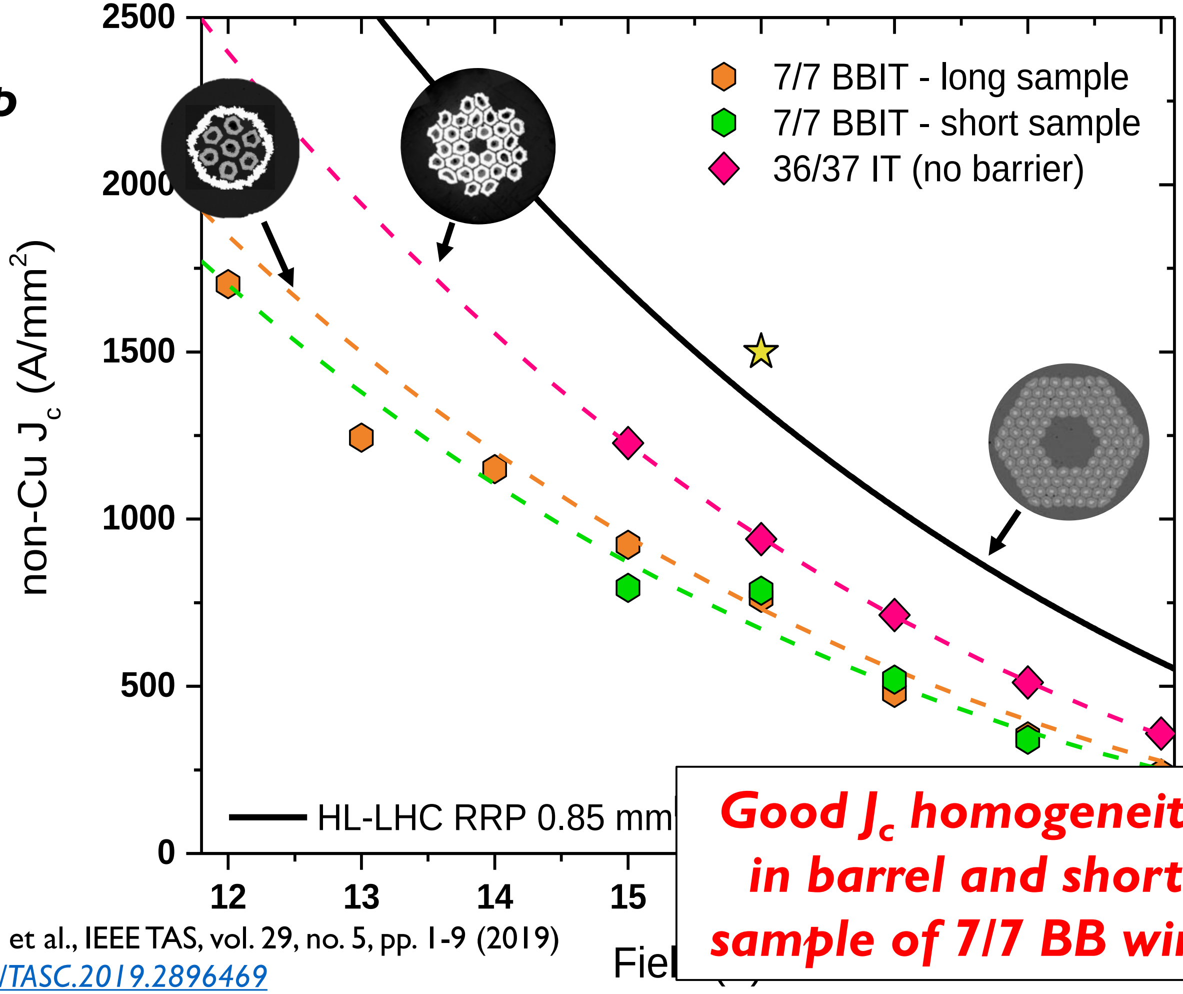
Fabrication of restacked wires without OS, with subelement containing ~ 200 filaments



54/61 *30/37* *7/7 with barrier*

Restacked wires manufactured with different layout, obtained in a max length of approx. 3 m

Wires without OS

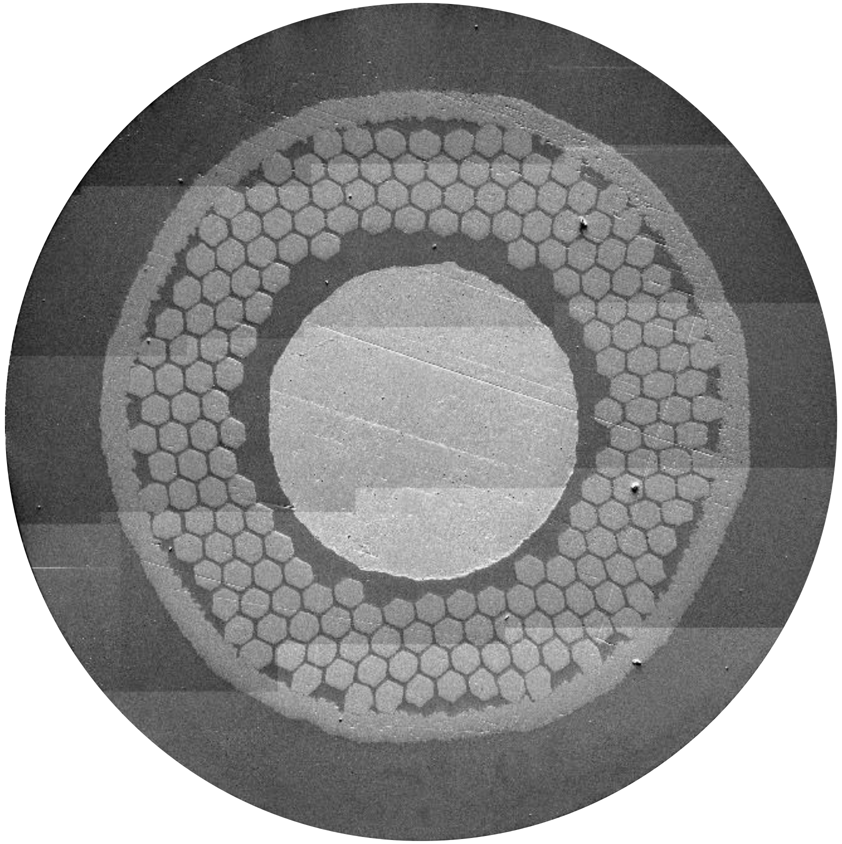


Good J_c homogeneity in barrel and short sample of 7/7 BB wire

[1] Ballarino A et al., IEEE TAS, vol. 29, no. 5, pp. 1-9 (2019)
DOI: 10.1109/TASC.2019.2896469

Approaches to implement internal oxidation

Where should we place the OS in a RRP wire?



OS at subelement level

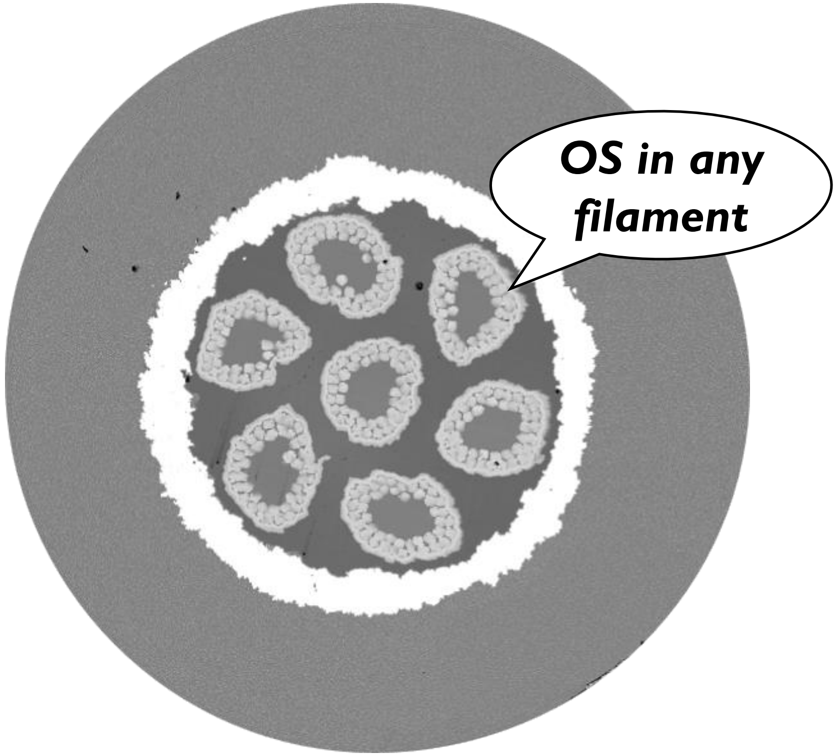
Nb alloy filaments
substituted with OS

OS at filament level

OS inside each filament
of the subelement

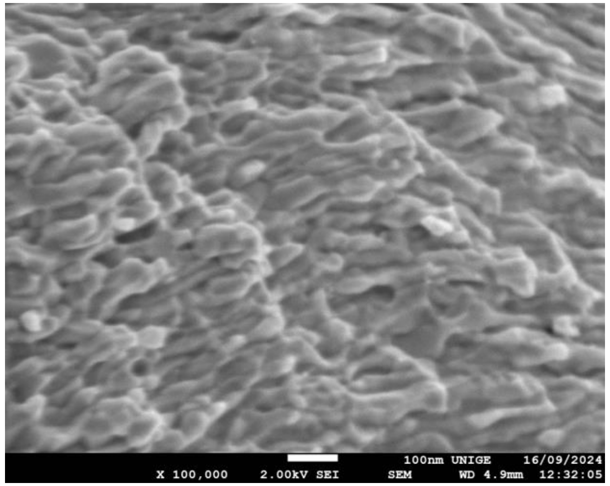
Does these approaches work?

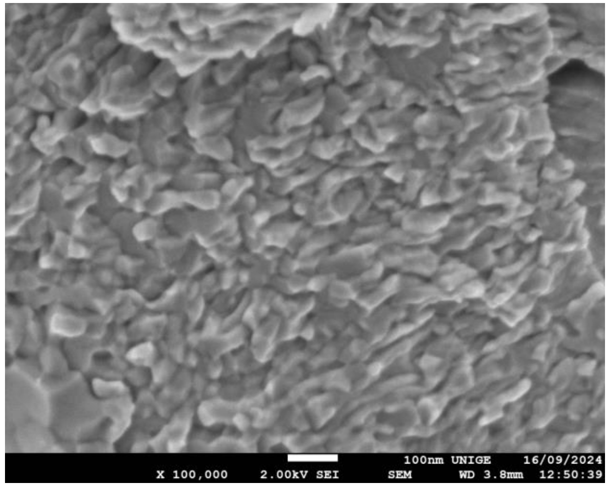
Heat treatment (HT):
550°C × 100 h + 650°C × 200 h

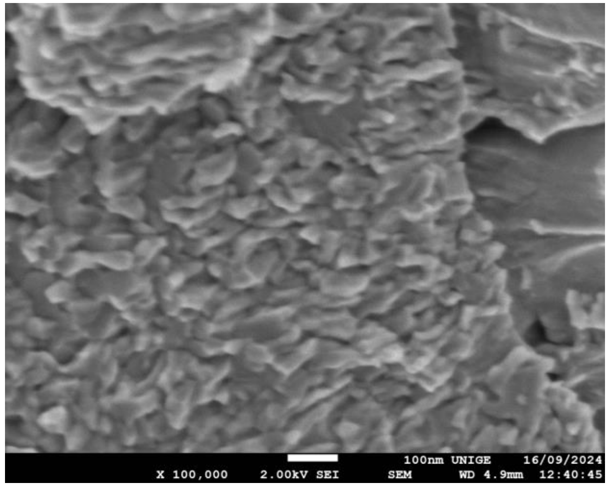


Wire fabricated with 7 subelements, made of 42 cold deformed NbTaHf alloy filaments.

Exploring how to implement the technique in extruded filaments and subelements.







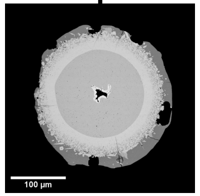
*Nb_3Sn grains refined down to **60 nm**!!*

Internal oxidation successfully implemented in a (short) Nb_3Sn rod-type multifilamentary wire

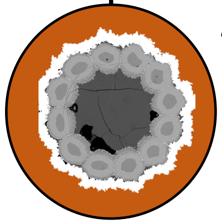
The UNIGE timeline

2029
Production of unit length sufficient for testing in Rutherford cables

Phase 1 — **2019** — **2025** — **Phase 2** — **2029**

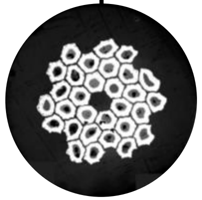


2019
Monofilamentary wire with internal oxidation

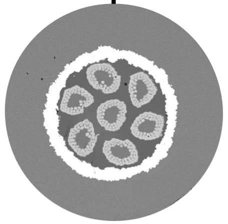


2021
Simplified subelement with internal oxidation

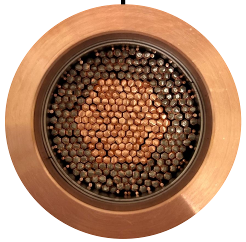
Early 2023
Extruded *subelement with NbTa alloy*



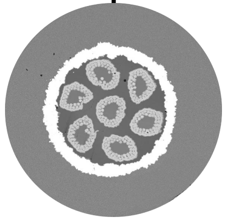
Late 2023
Restacked wire from ***extruded*** *NbTa subelement*



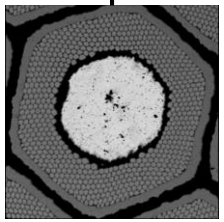
Mid 2024
Restacked wire from ***cold deformed*** *subelement with NbTaHf alloy and OS*



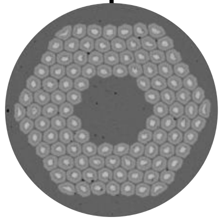
Early 2025
Extruded *subelement with NbTaHf alloy and OS*



Mid 2025
Restacked wire from ***extruded*** *subelement with NbTaHf alloy and OS*



2026 - 2027
Optimization of ***extruded*** *subelements with OS, filament count, Cu/Nb ratio, Nb/Sn ratio, OS disposition*



2027 - 2028
Development of wire restack with a high count of subelements. Wire reinforcement studies

Summary and future perspectives

- ***Internal oxidation process*** *proved by multiple sources to be an effective way to reach the* ***FCC target for non-Cu J_c***
- *Material studies on* ***internally oxidised monofilamentary*** *wires showed* ***grain refinement*** *and* ***enhanced J_c and B_{c2}***
- ***Presence of APCs*** *in prototype* ***rod-type subelement enhance layer J_c*** *above the* ***FCC specifications*** *in addition to* ***record-high B_{c2}*** *and change of pinning mechanism (****point defect****)*
- ***X-Rays Absorption Near Edge Structure (XANES)*** *on Nb_3Sn wires shows the* ***formation of the APC*** *precipitates is* ***concomitant to the formation of the Nb_3Sn layer***
- ***Preliminary steps*** *moved in the fabrication of* ***extruded subelements and restacked wires***
    - ***Restacked wires*** *fabricated from* ***extruded subelement*** *with* ***NbTa alloy, without OS***
    - ***Grain refinement (60 nm) in a wire from cold-deformed subelements with NbTaHf and OS***

Thank you for your attention

...and make Nb_3Sn great again!