UNIVERSITÉ DE GENÈVE
FACULTÉ DES SCIENCES

CERN

Swiss National Science Foundation

CHART
Swiss Accelerator Research and Technology

HFM
High Field Magnets

WP1.3:

Nb_3Sn wire development by Internal Oxidation

***Gianmarco BOVONE*, Florin BUTA, Francesco LONARDO, and Carmine SENATORE**
Department of Quantum Matter Physics, University of Geneva, Switzerland
Department of Nuclear and Particle Physics, University of Geneva, Switzerland

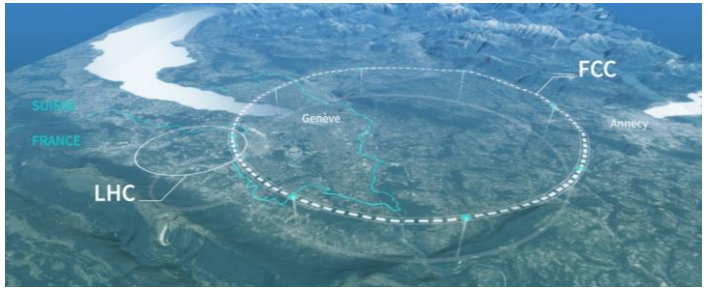
Simon C. HOPKINS, Amalia BALLARINO, and Thierry BOUTBOUL
CERN, Switzerland

HFM annual meeting 2023

Outline

- *Motivation: the importantance of internal oxidation for Nb_3Sn*
- *Implementation of the internal oxidation in simplified multifilamentary wires*
  - *Effect of the internal oxidation on the superconducting properties*
    - *Critical current density above FCC target*
    - *High critical field measured at LNCMI*
  - *What drives the enhancement?*
    - *Material studies by XAS at PSI*
- *Optimisation of heat treatmen:*
  - *Nb_3Sn layer thickness vs Layer J_c*
- *Development of advanced multifilamentary wires with internal oxidation*

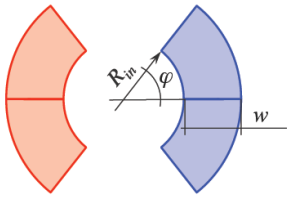
The importance of oxidation for Nb_3Sn



LHC
27 km, 8.33 T
14 TeV (c.o.m.)
1'300 tons NbTi

FCC-hh
100 km, 16 T
100 TeV (c.o.m.)
~10'000 tons Nb_3Sn

B [T]	***16***
J_{op} [A/mm^2]	***300***
w [mm]	***76***
A_{coil} [mm^2]	***20'000***



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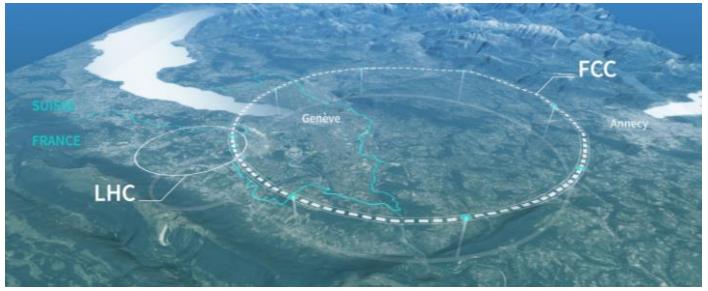

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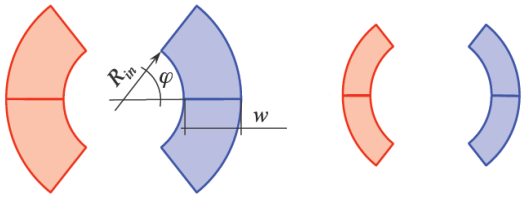
B [T]	***16***		***16***
J_{op} [A/mm²]	***300***	***2x***	***600***
w [mm]	***76***		***38***
A_{coil} [mm²]	***20'000***		***7'000***



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

$A_{coil} \propto$ SC mass $\propto$ $$

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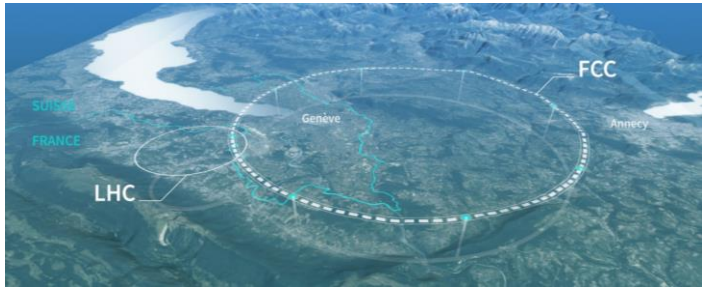

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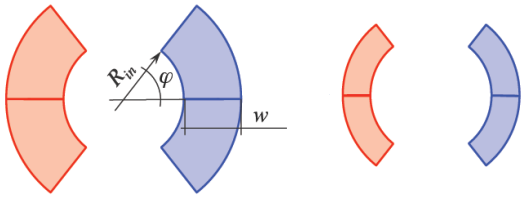

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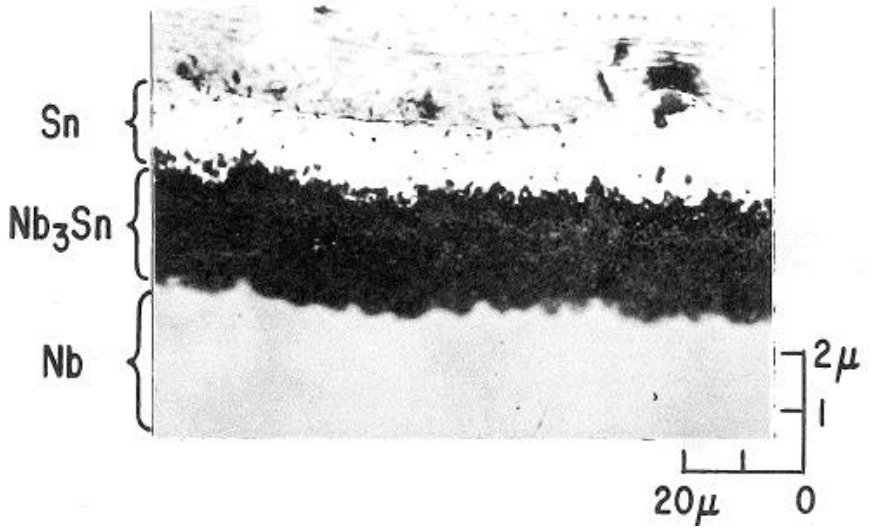
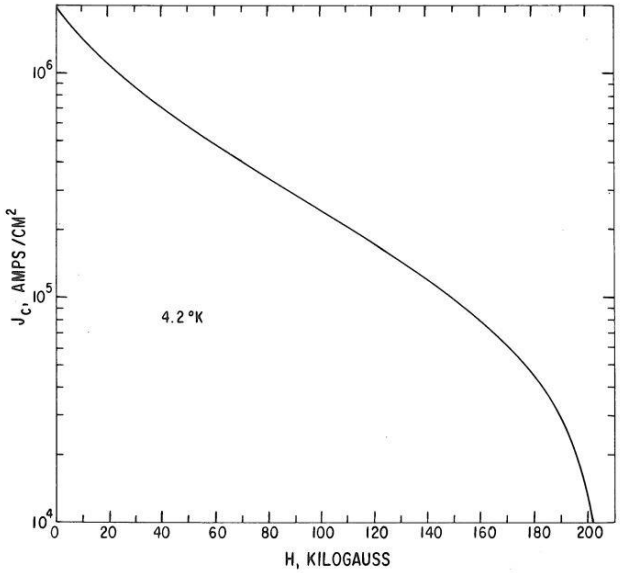


First observation of combined effect of Zr and O to reduce grain size



- Benz M. G. *Trans Met Soc AIME* 242.6 (1968).
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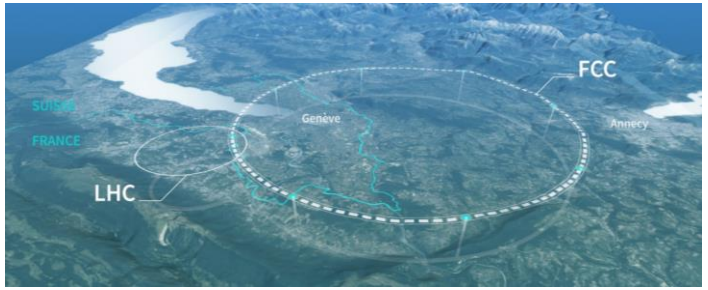

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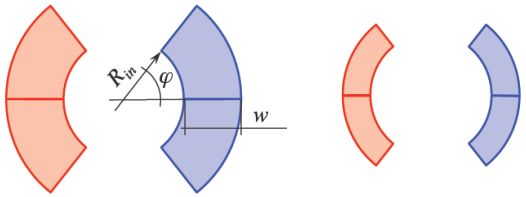

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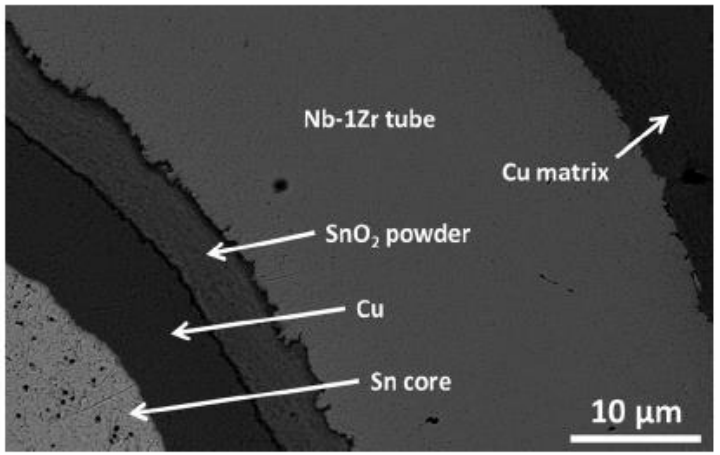
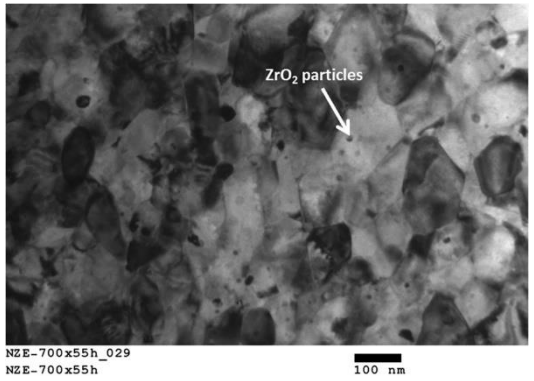


Internal oxidation in practical round wire



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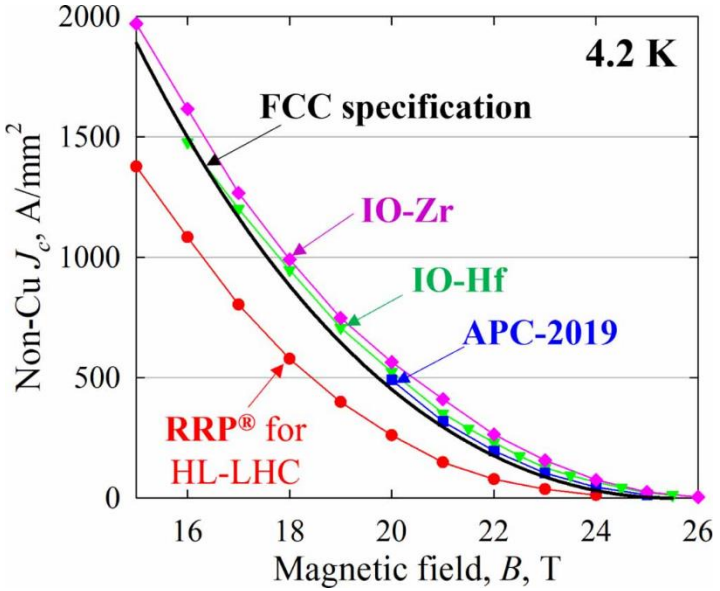

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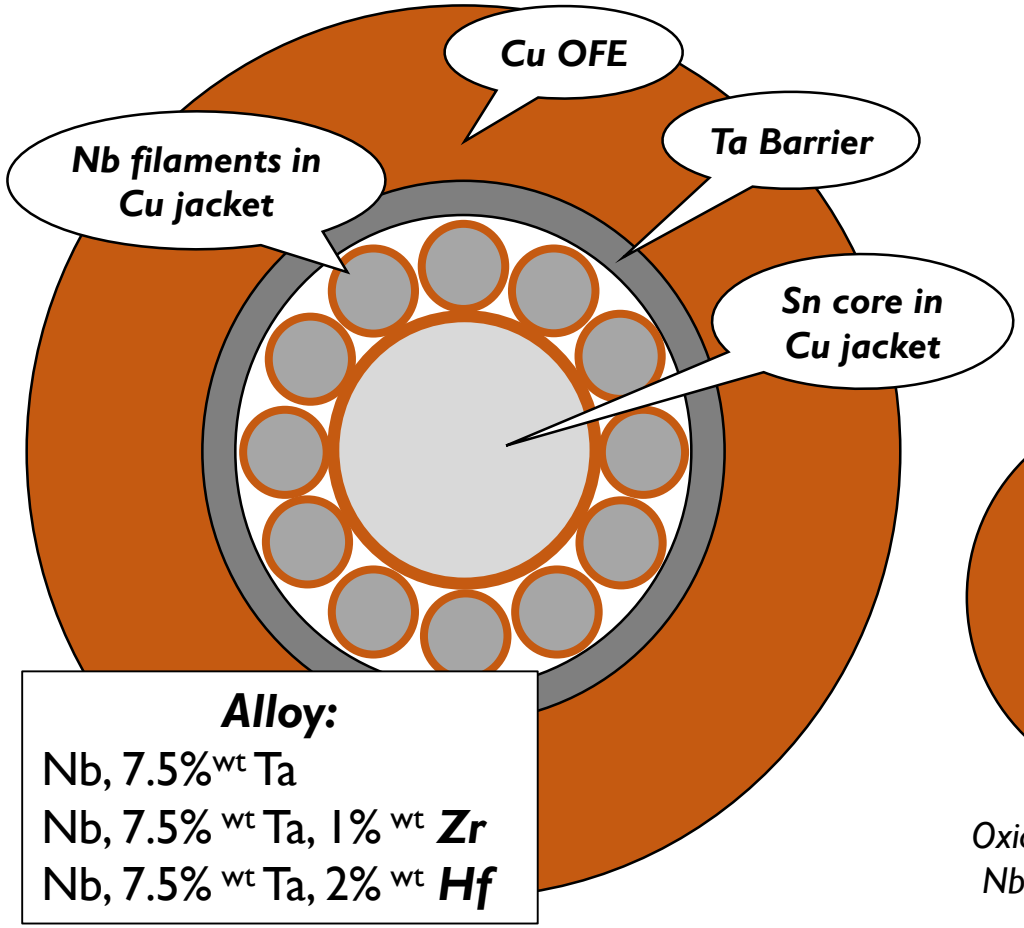
- Xu X. et al. *Applied Physics Letters* 104.8 (2014): 082602.
- Xu X. et al. *Superconductor Science and Technology* 36.3 (2023): 035012.

Simplified multifilamentary wires layout and fabrication process

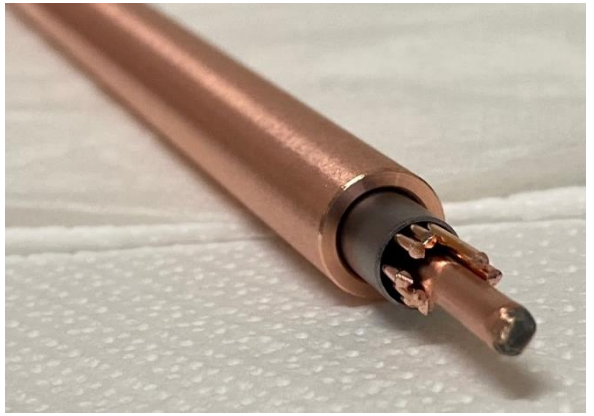


Alloy:

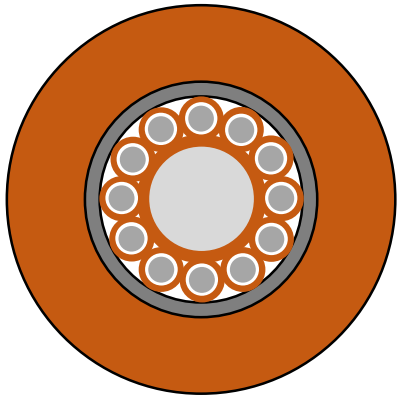
Nb, 7.5%^{wt} Ta

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

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

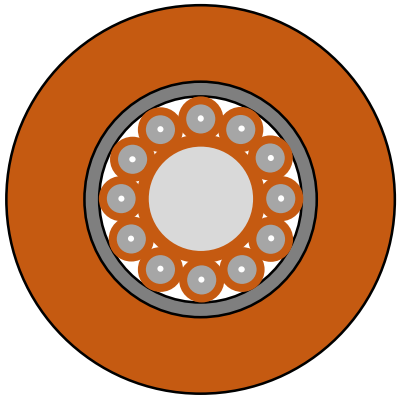


AnnularOS



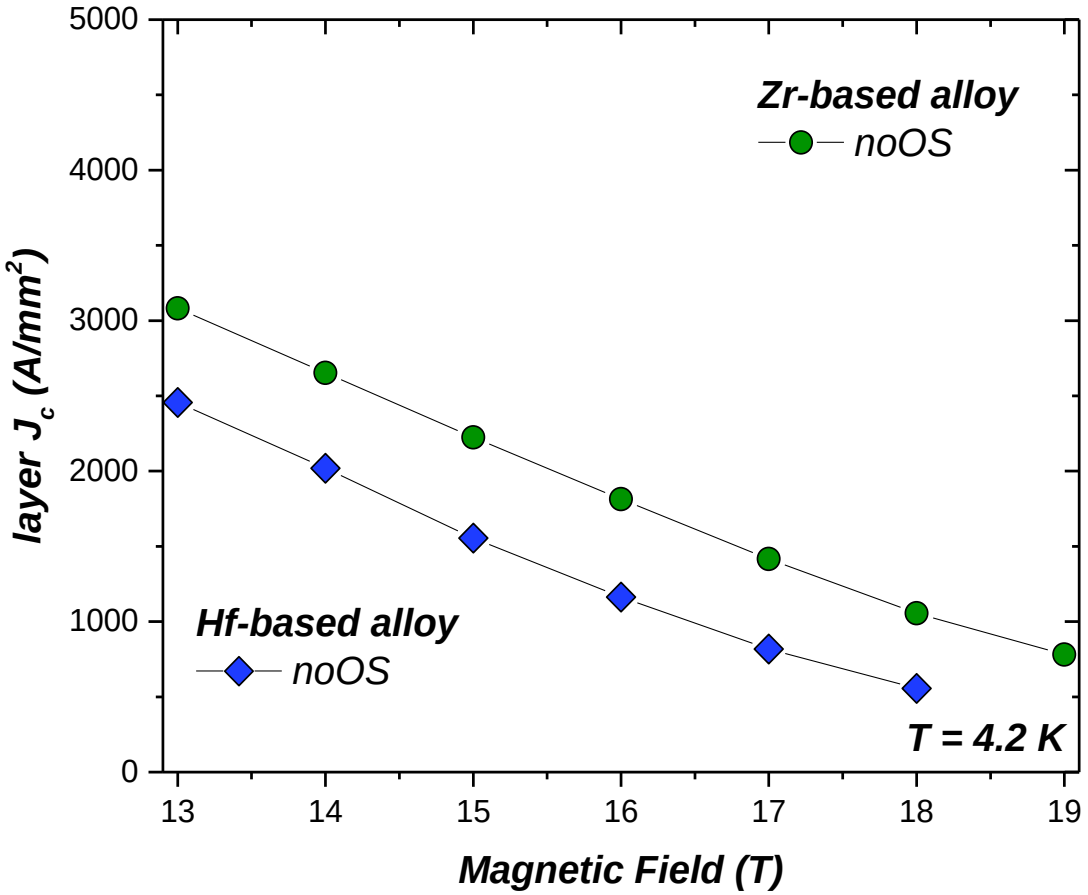
Oxide powder between the Nb-alloy filament and the copper jacket

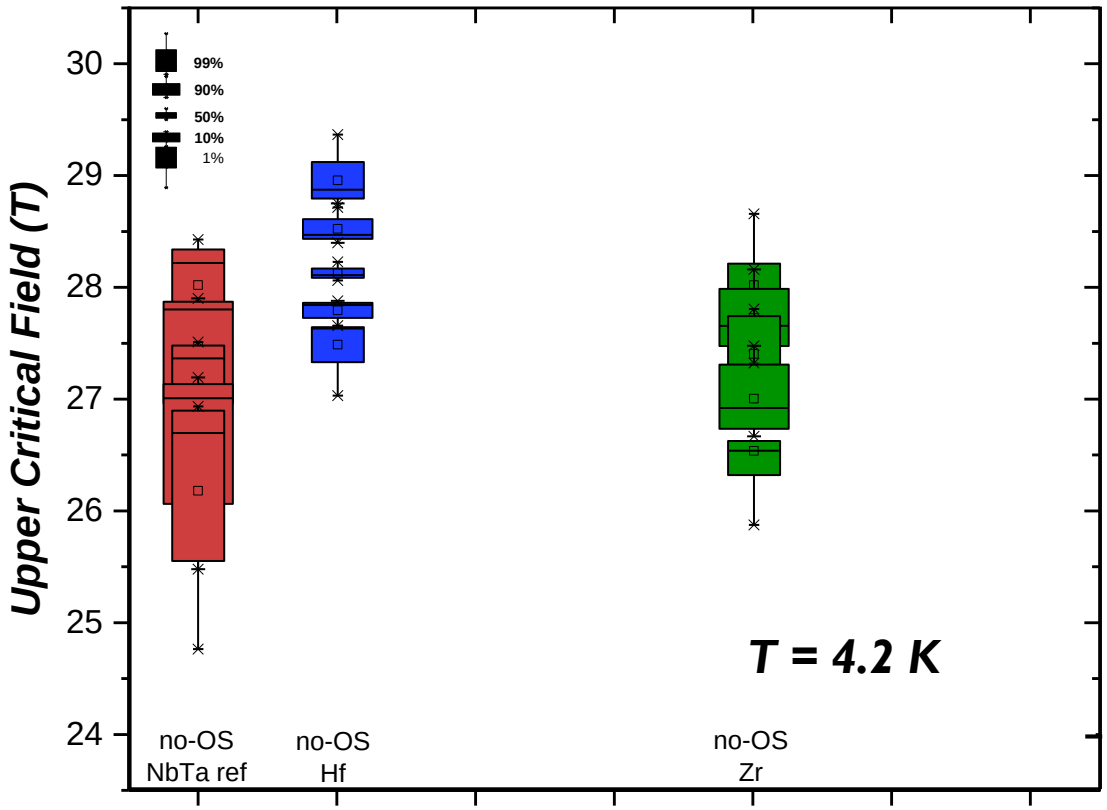
CoreOS



Oxide powder inside the Nb-alloy filaments

Effects of the internal oxidation on the superconducting properties





- Bovone G. et al. *Superconductor Science and Technology* (2023)

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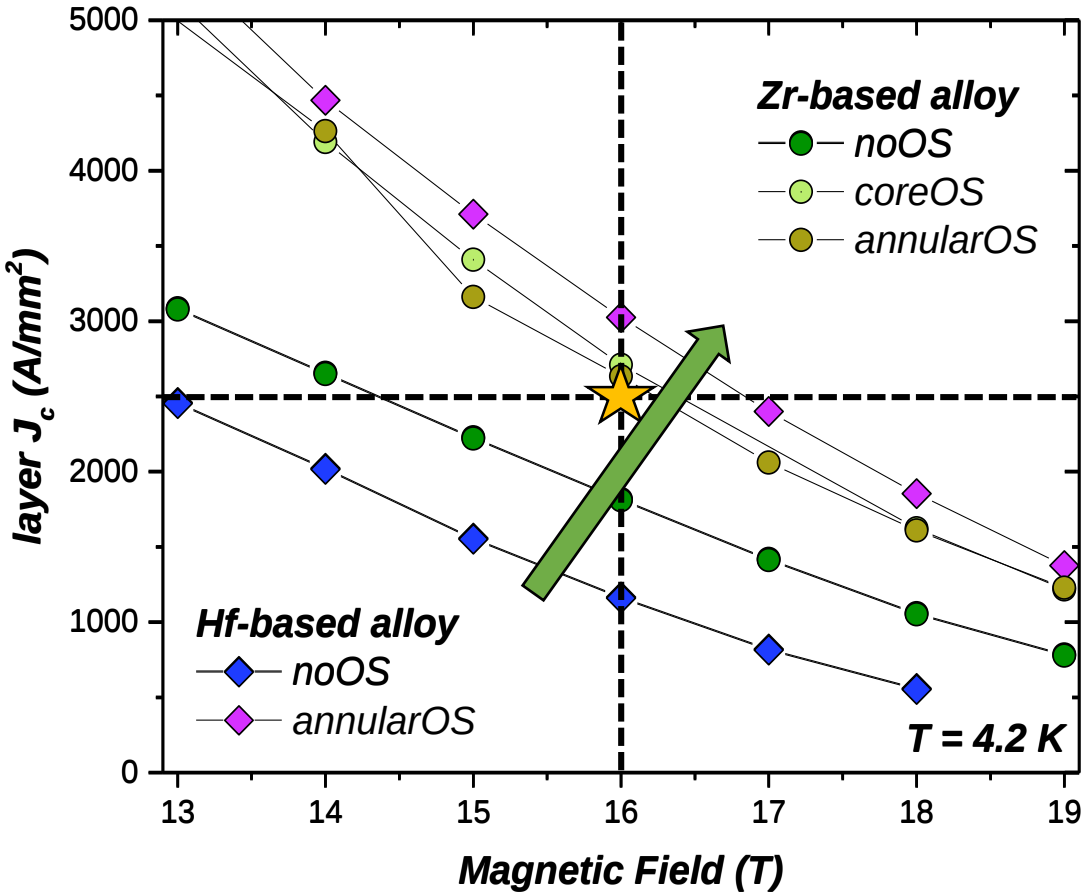

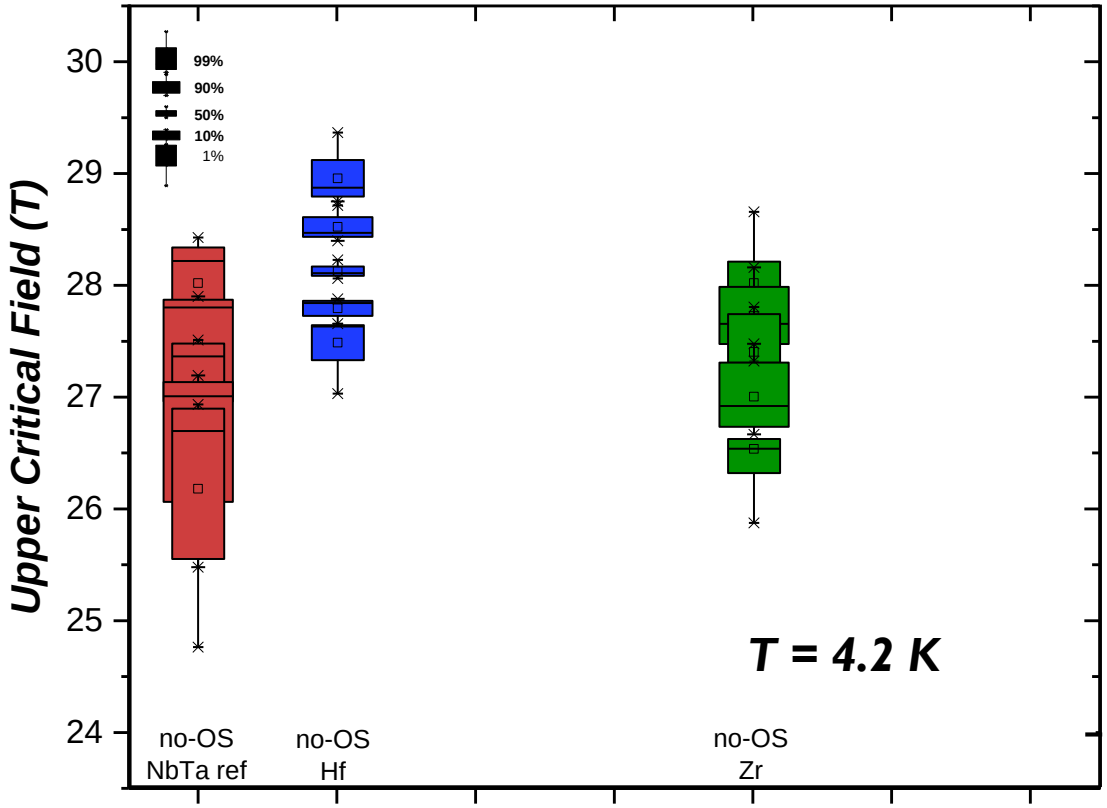

Layer J_c *above* ***FCC target*** *of* ***2500 A/mm^2***, *at 4.2 K and 16 T*

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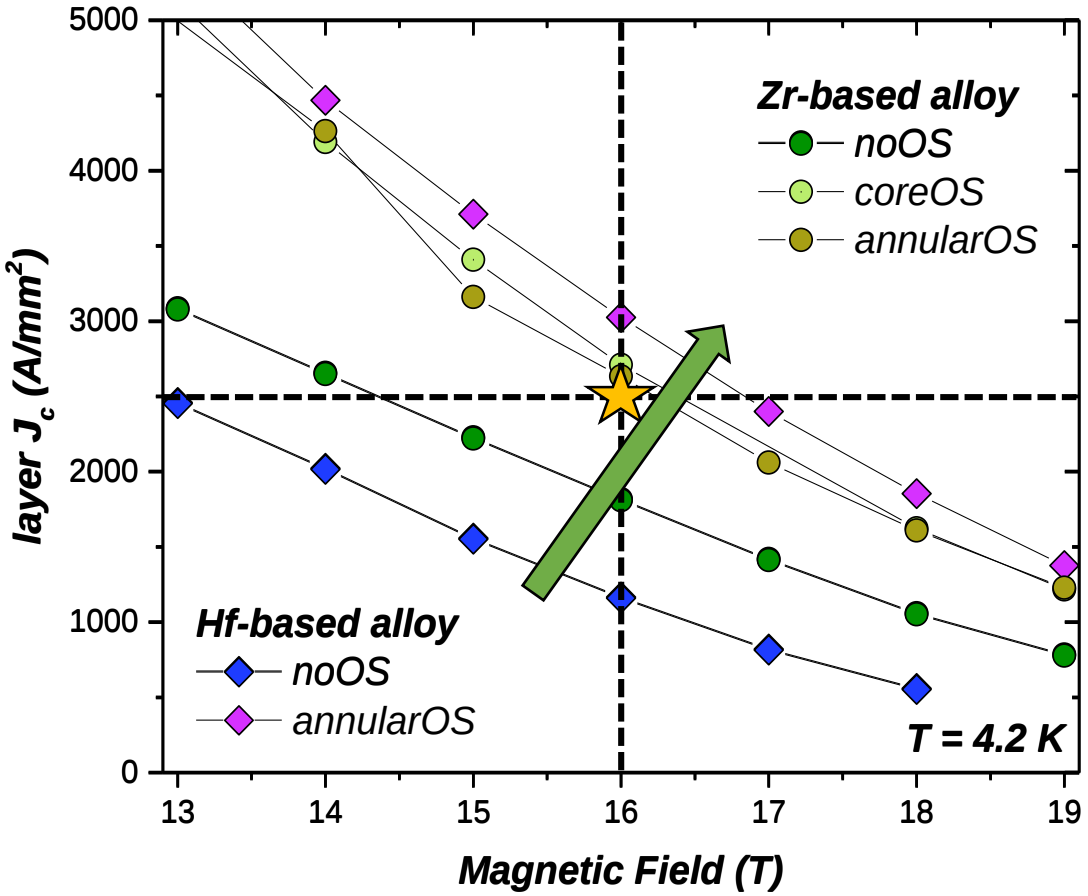

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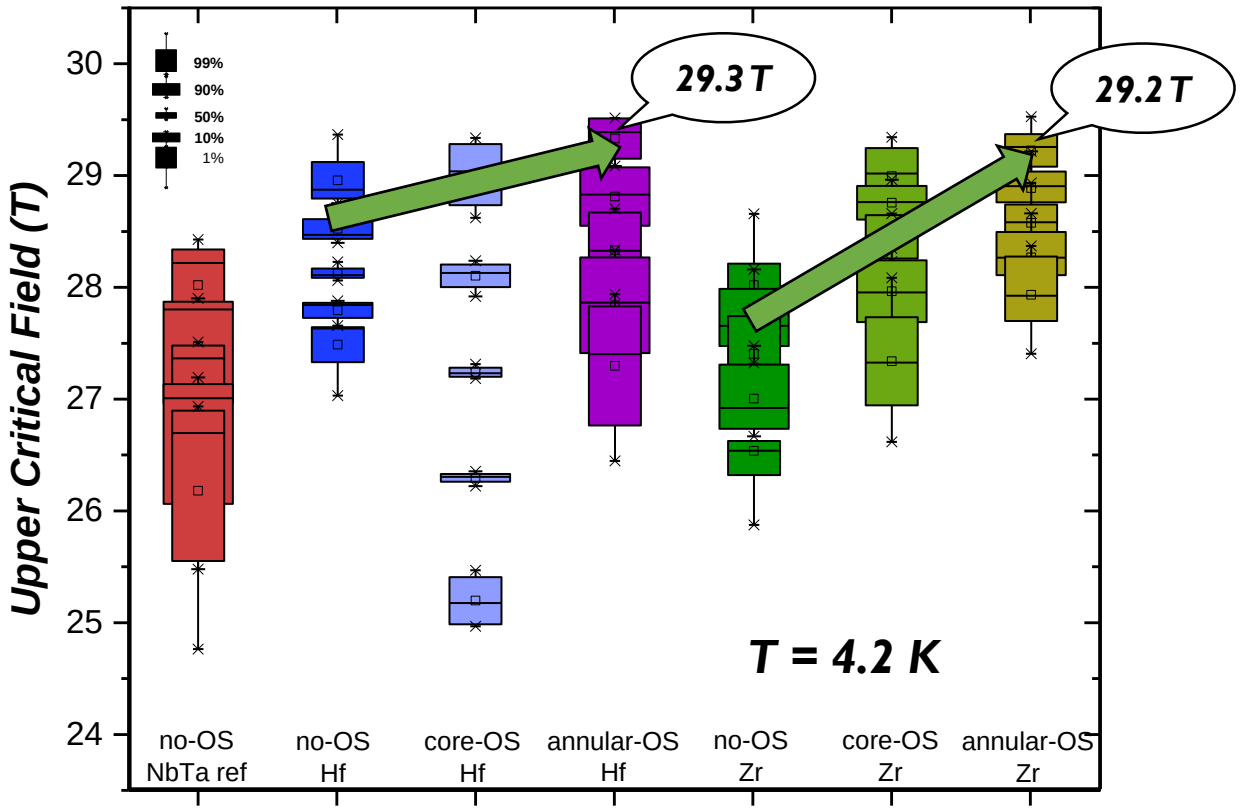

B_{c2} *enhanced above* ***29 T*** *for* ***annularOS*** *samples in both* ***Hf and Zr****-based wires*

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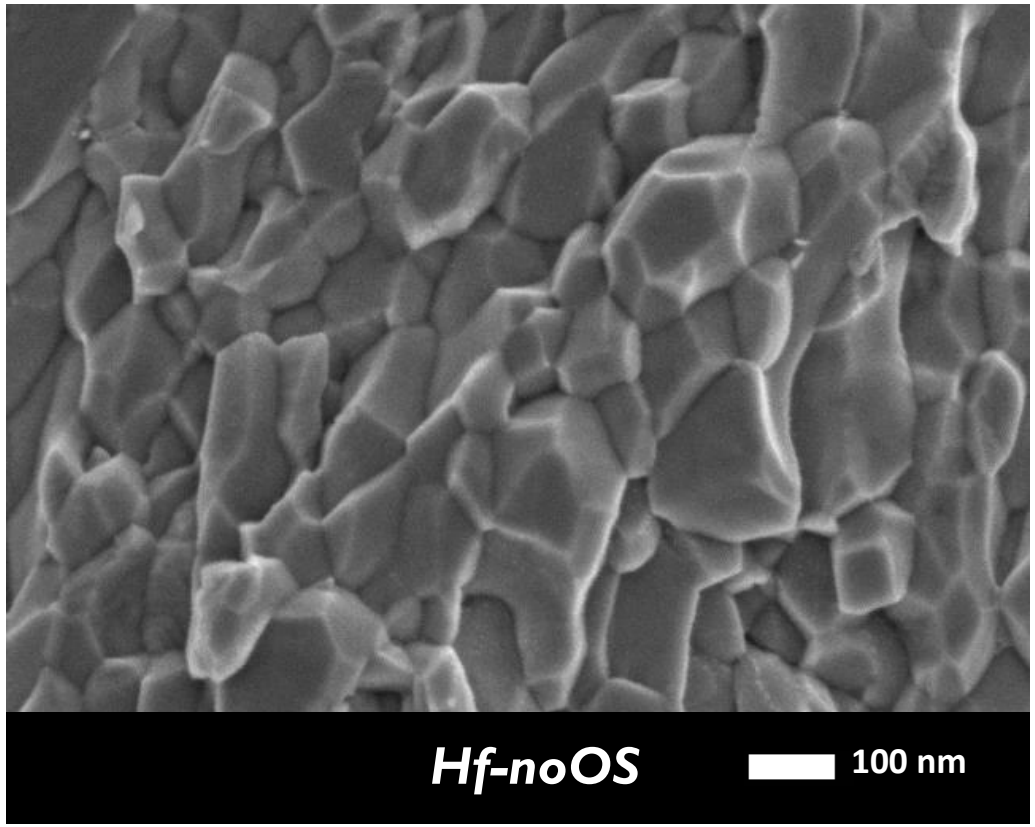
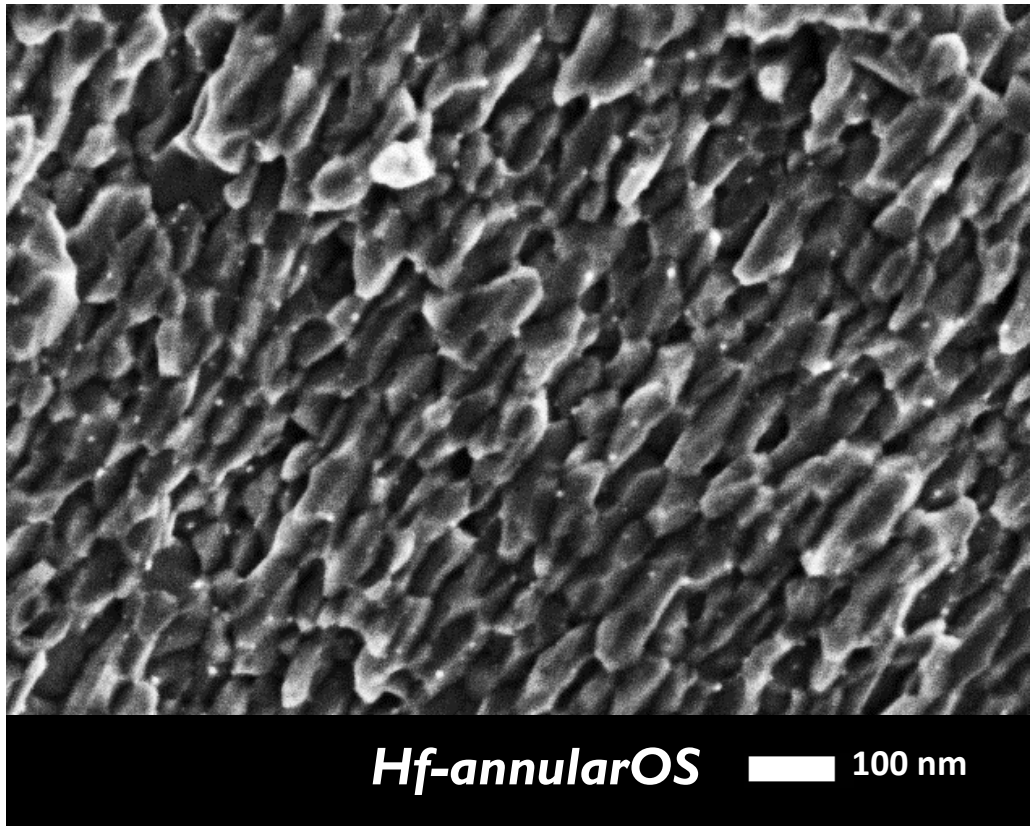
What drives the enhancement?





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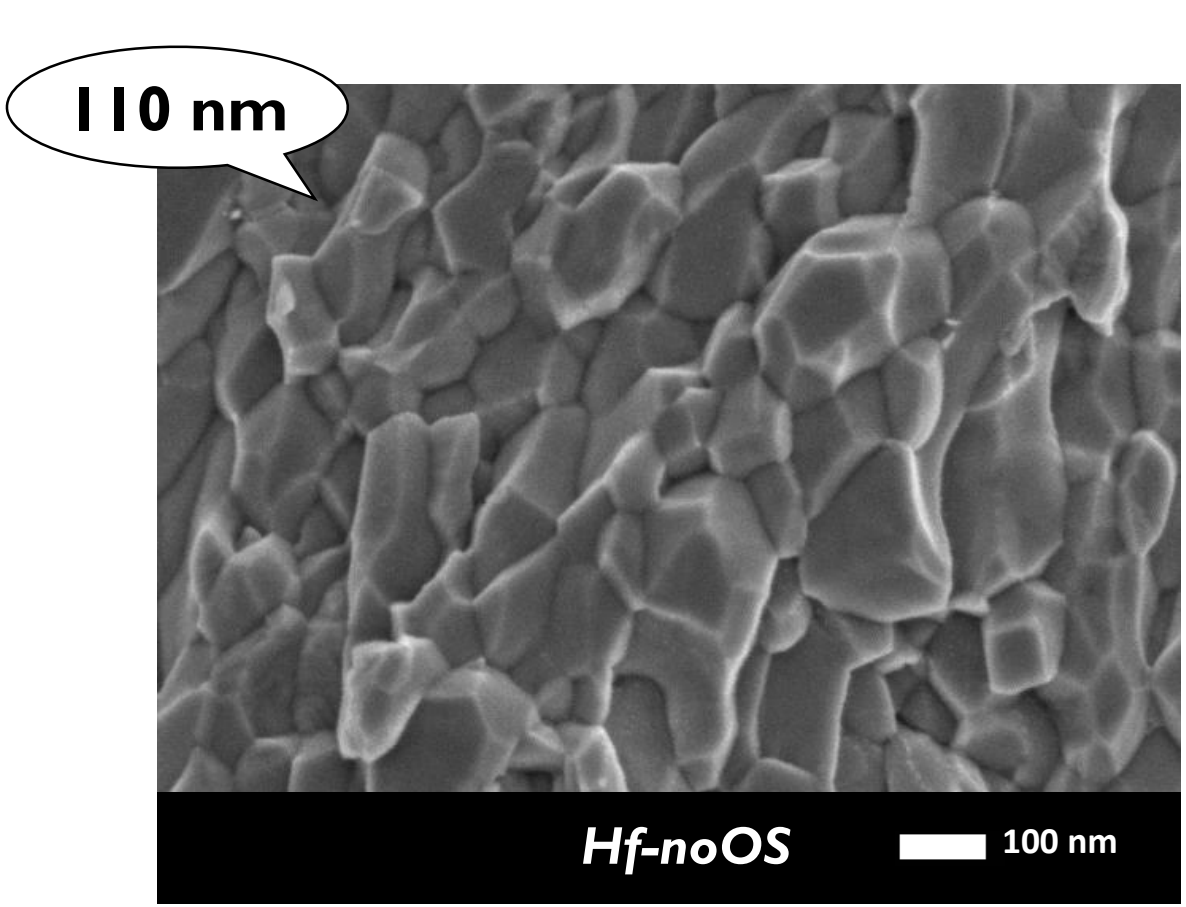

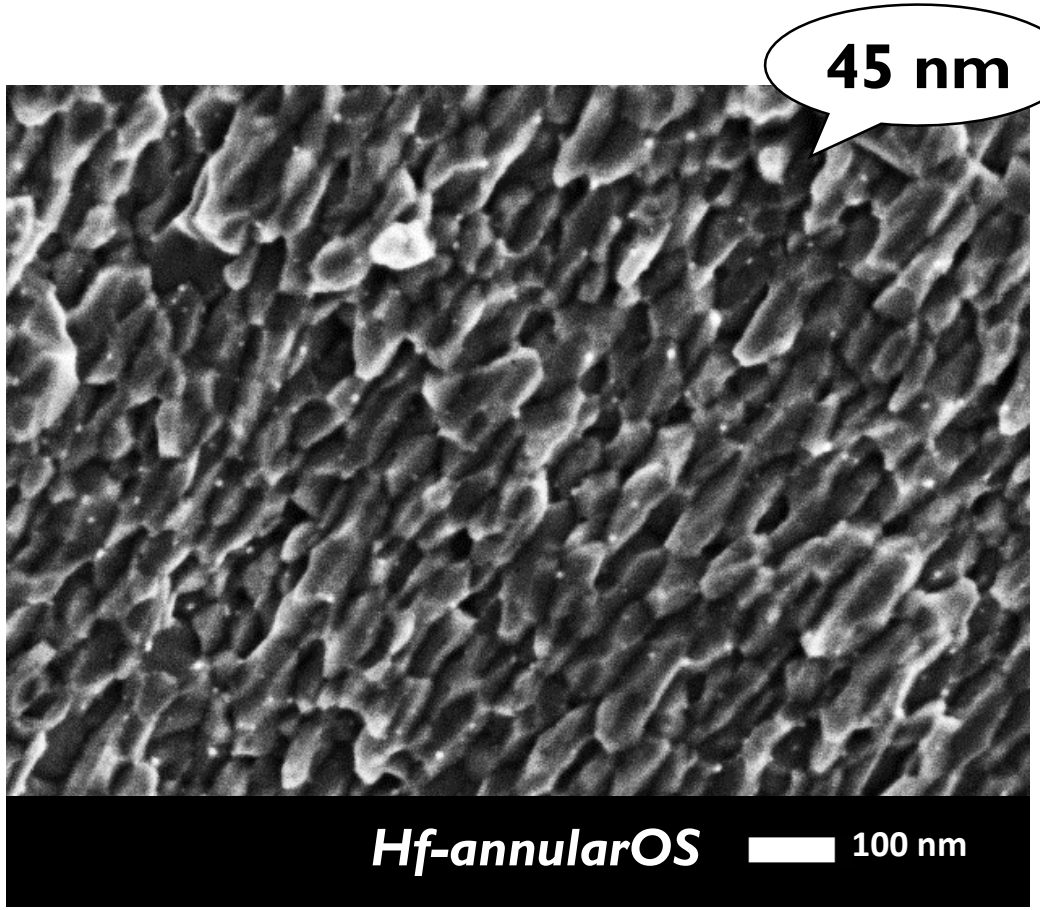

Nb_3Sn ***grain size reduction*** *induced by internal oxidation leads to* ***J_c enhancement***

- Bovone G. et al. *Superconductor Science and Technology* (2023)

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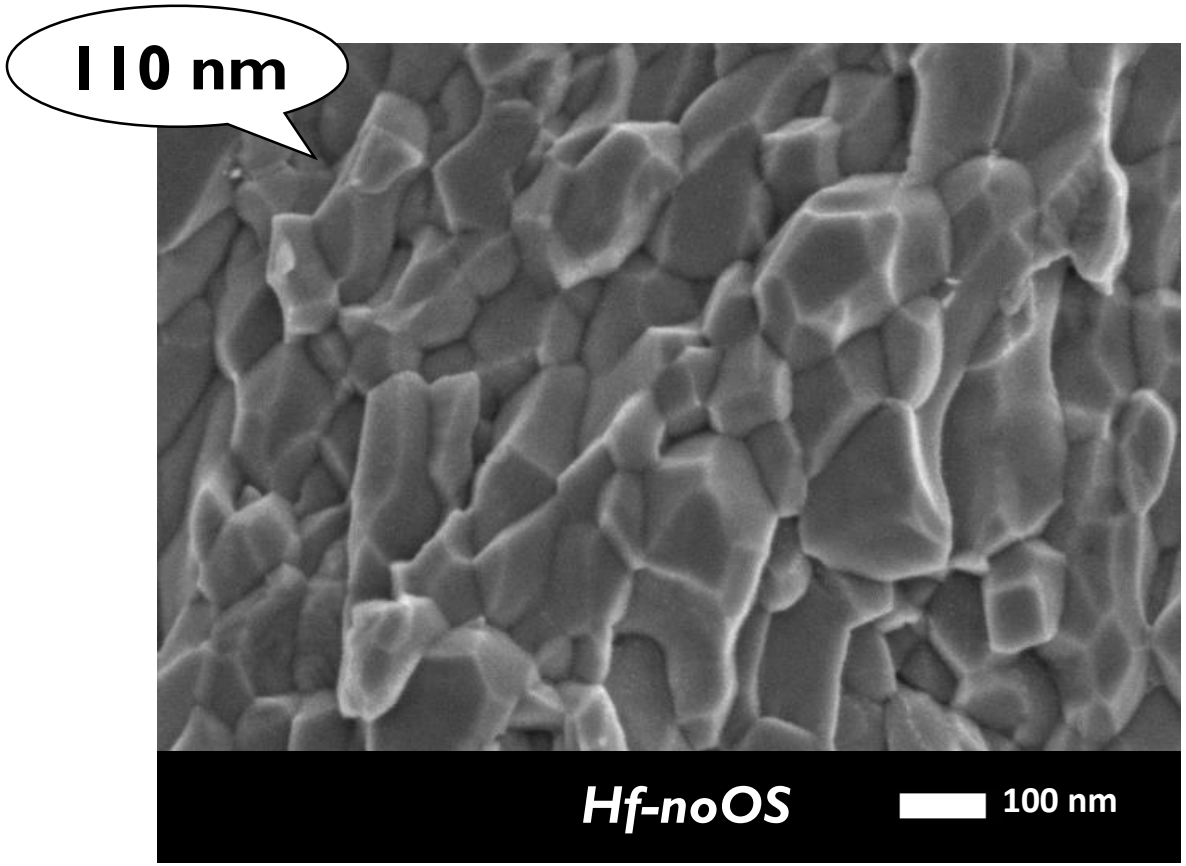

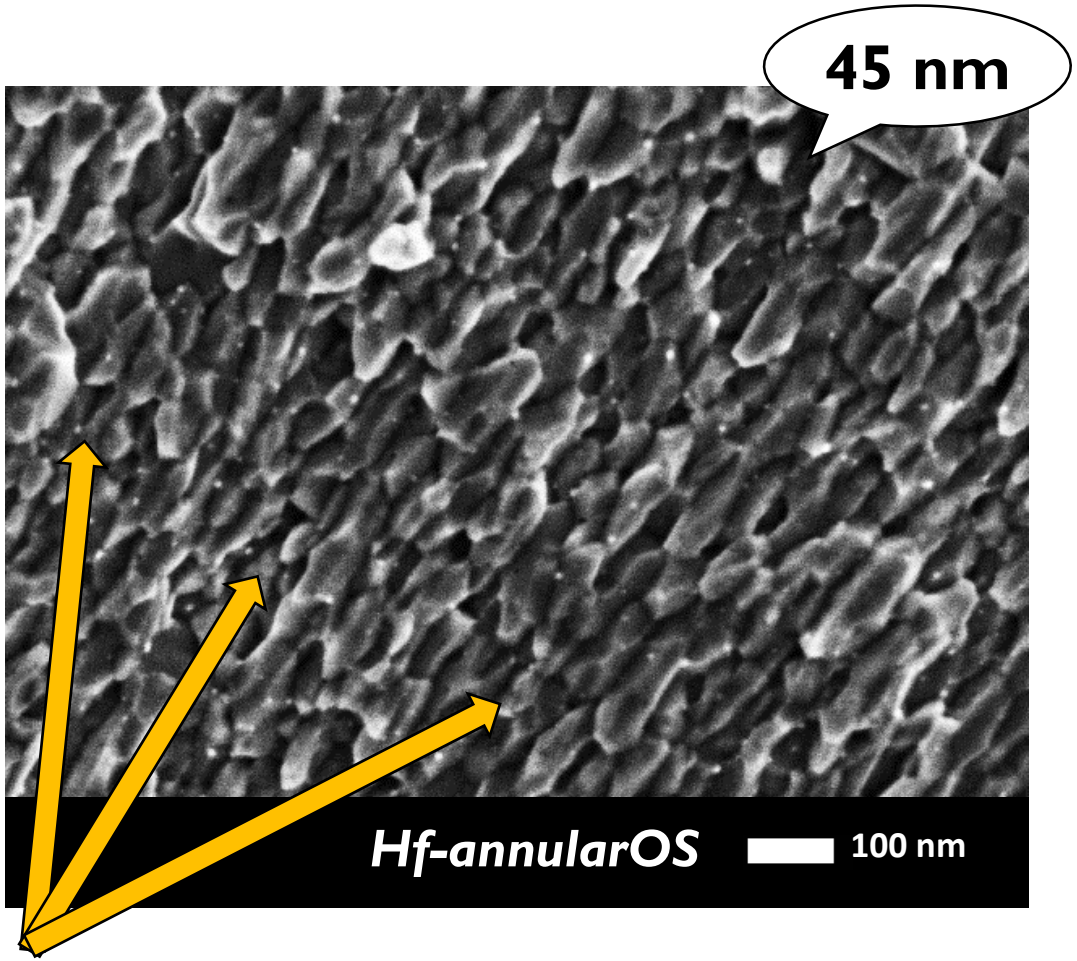

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Presence of precipitates *(presumably HfO_2)*

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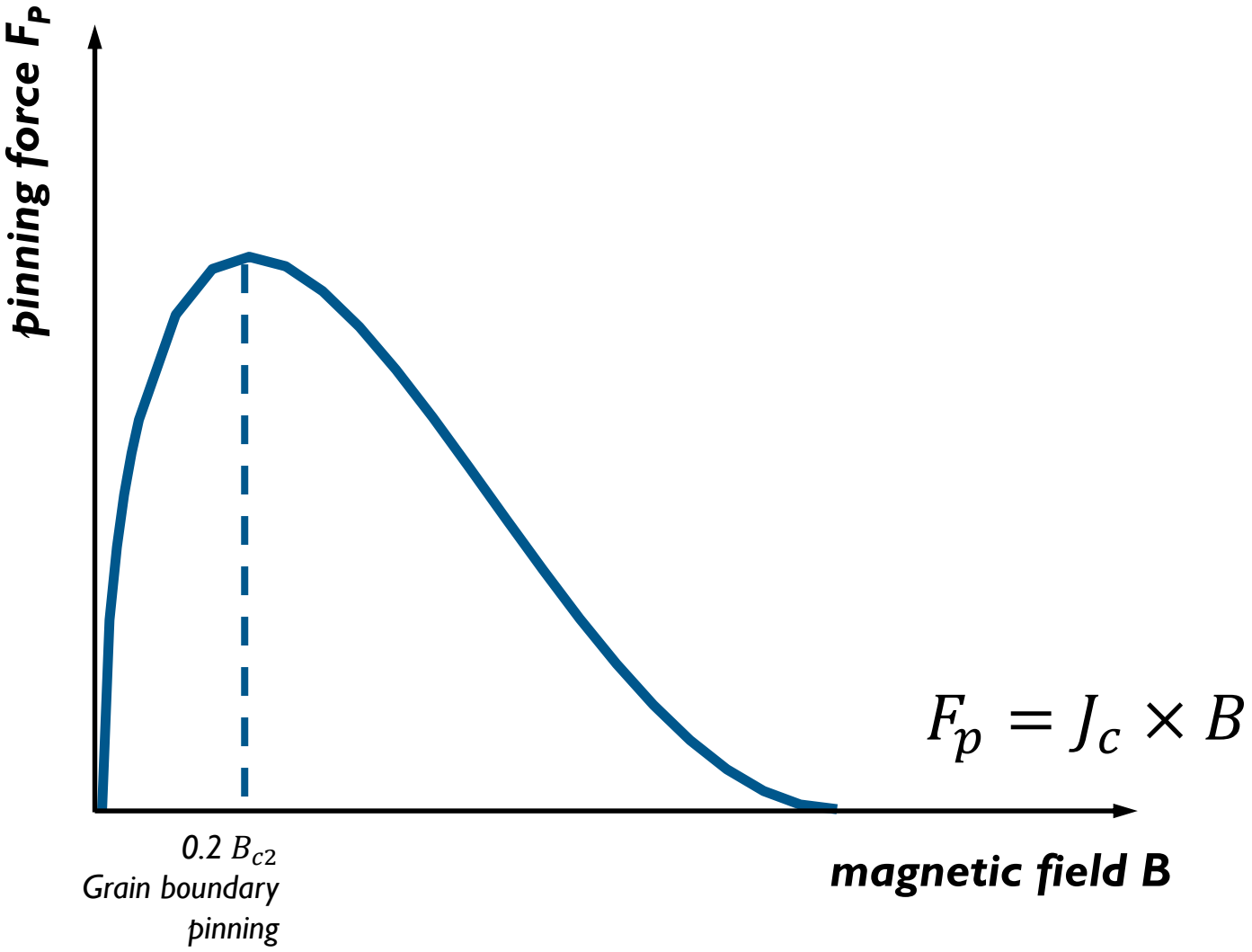
Precipitates and pinning contribution



$$F_p = J_c \times B$$

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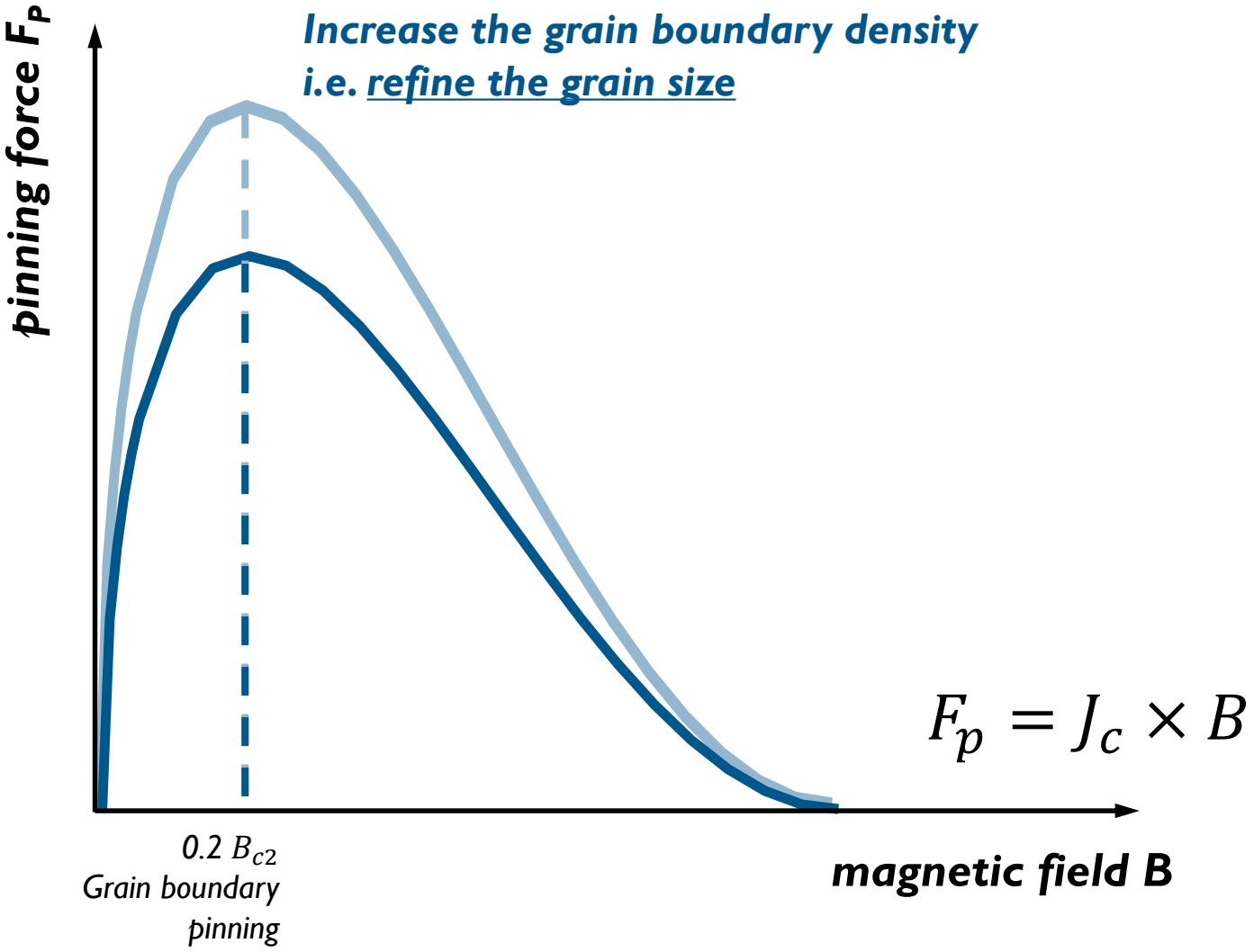

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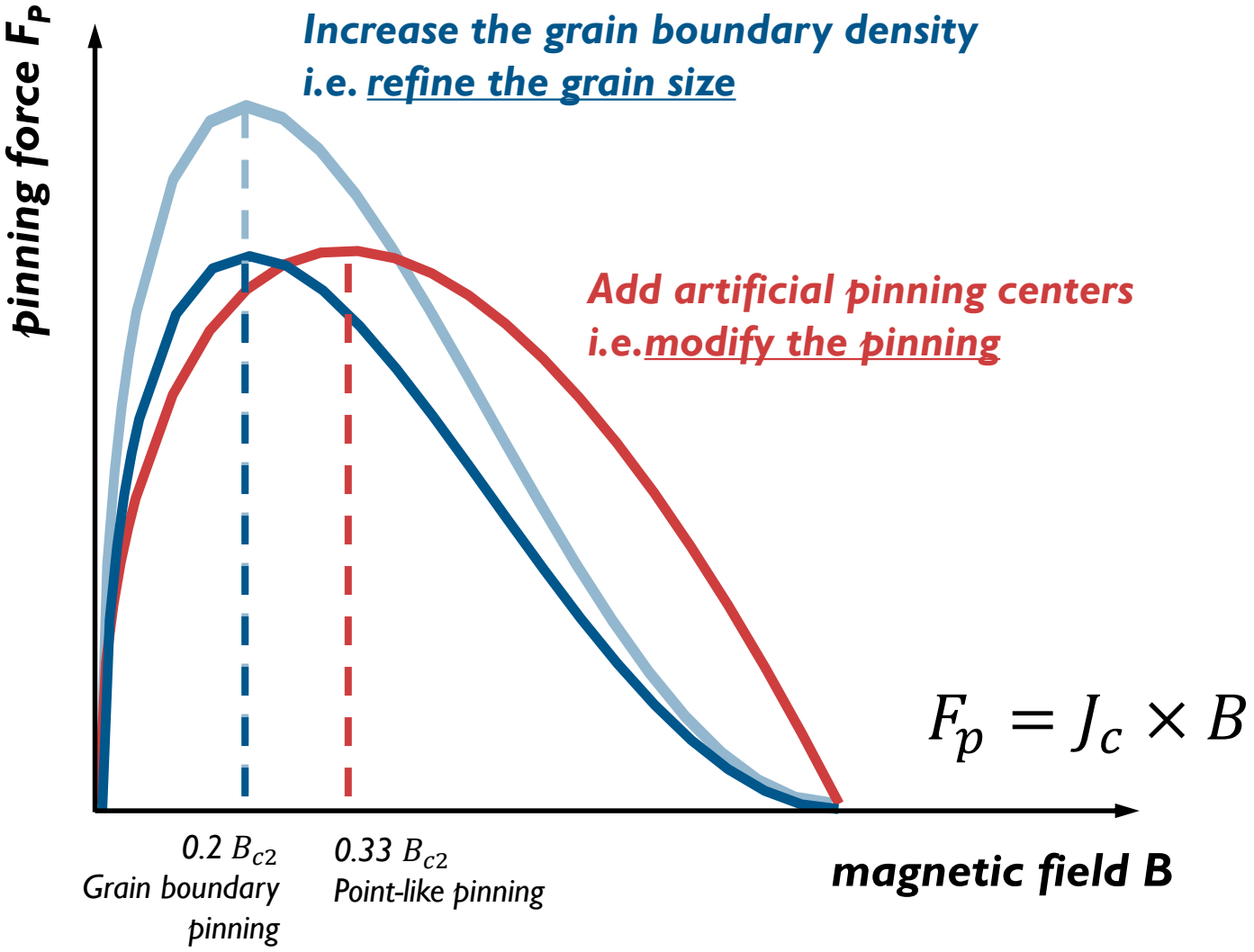

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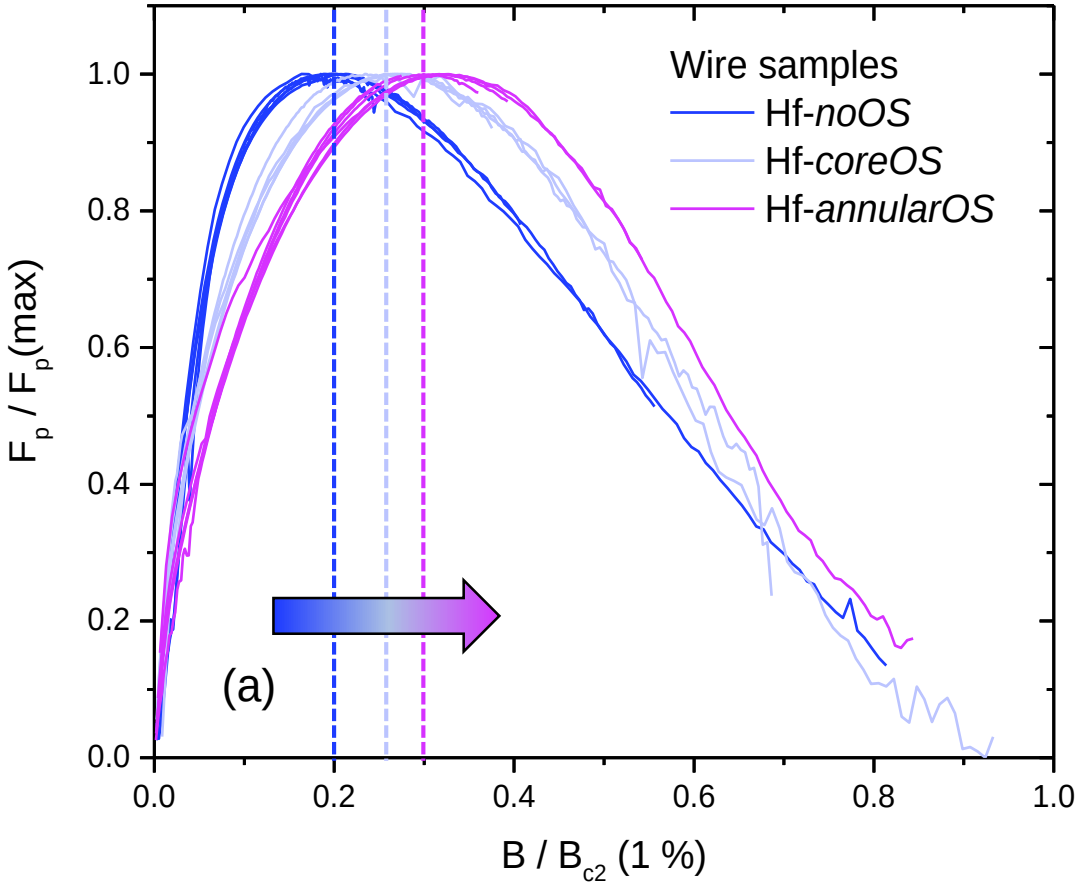

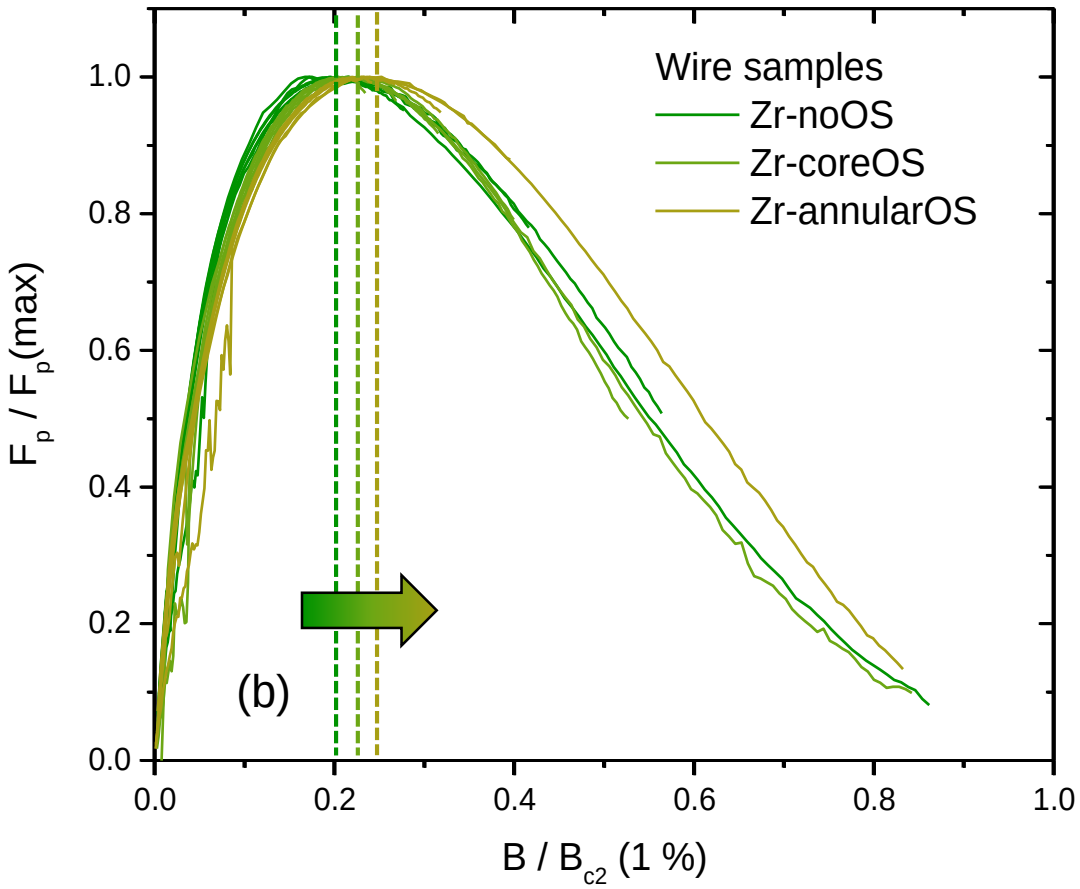

Change of dominant pinning mechanism *induced by the presence of* ***oxide precipitates*** *for both* ***Hf and Zr-****bases wires (shift induced by Hf is larger)*

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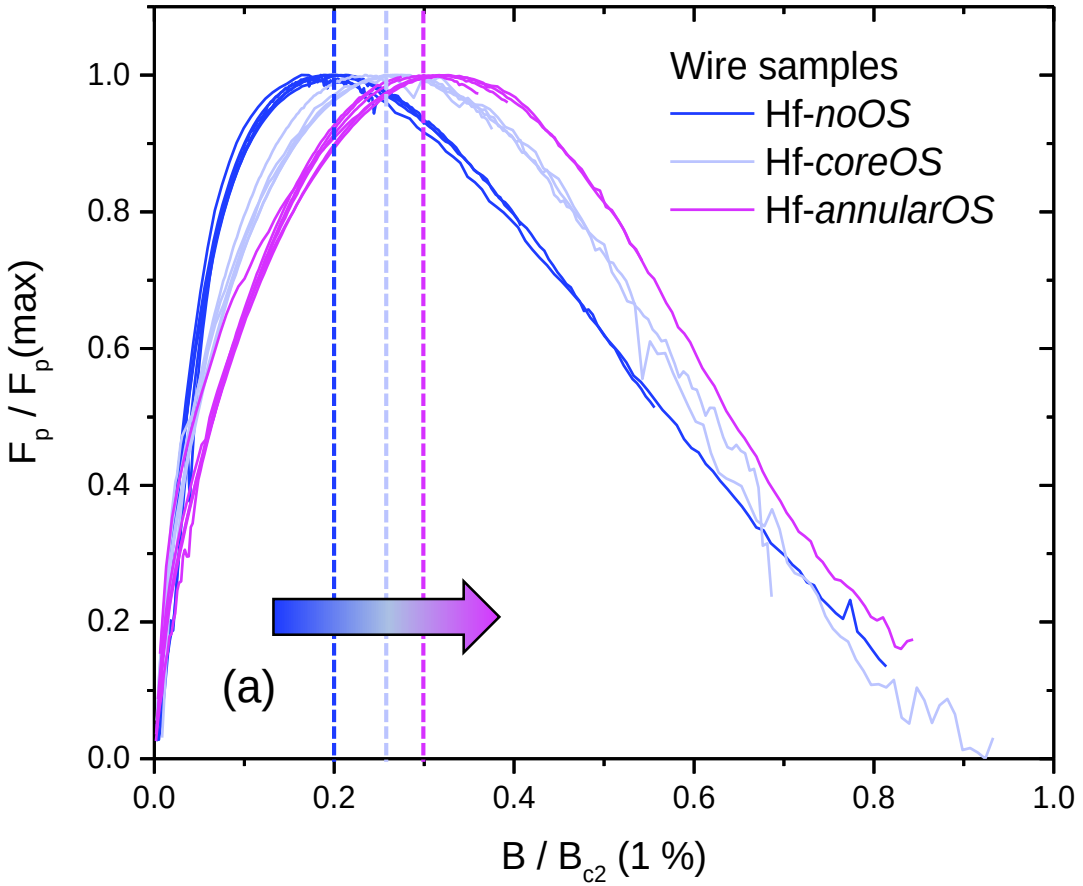

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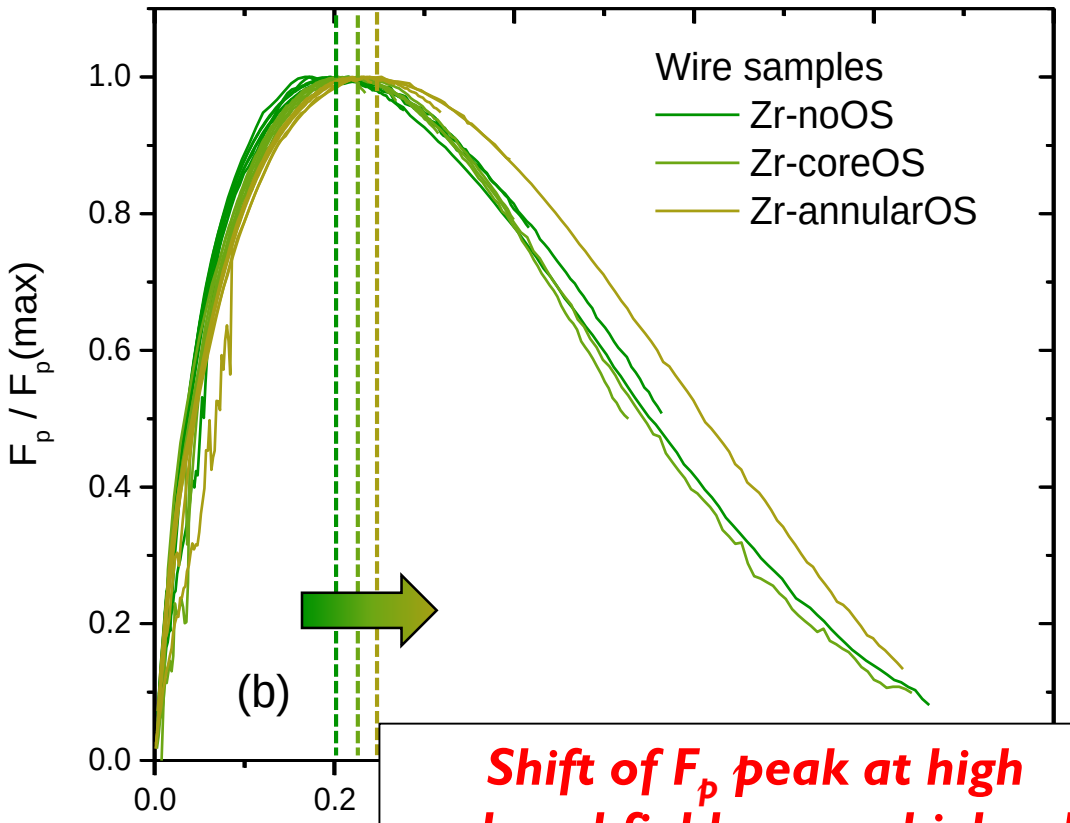

Shift of F_p peak at high reduced field means higher J_c at high magnetic field

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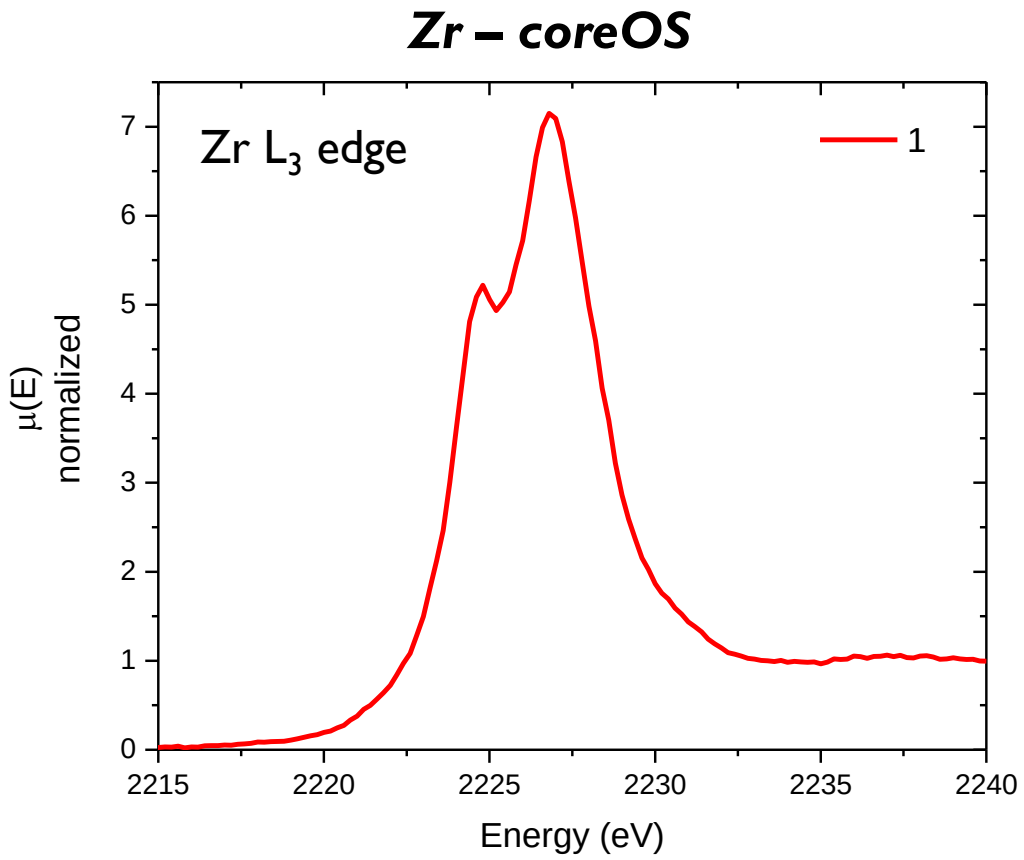
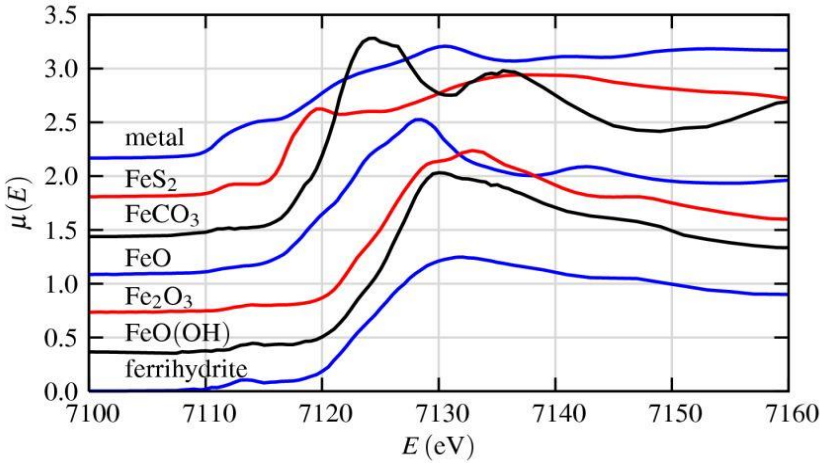
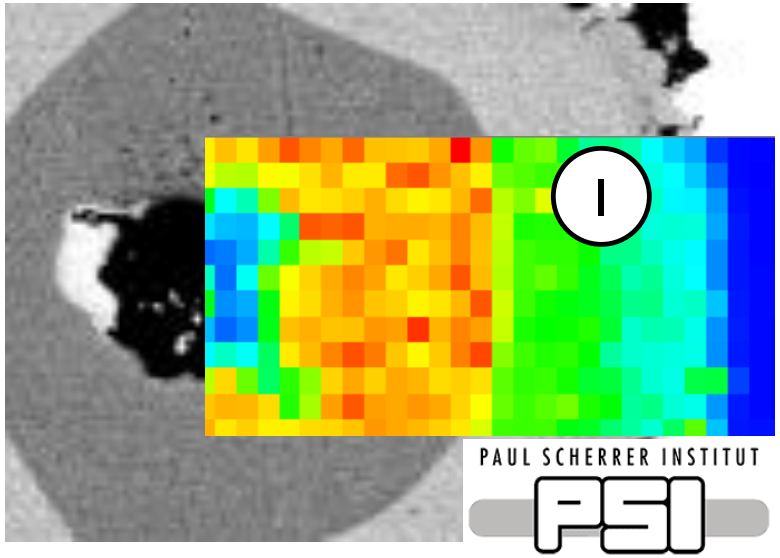
An X-Ray Absorption Spectroscopy study at the PHOENIX beamline of PSI





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



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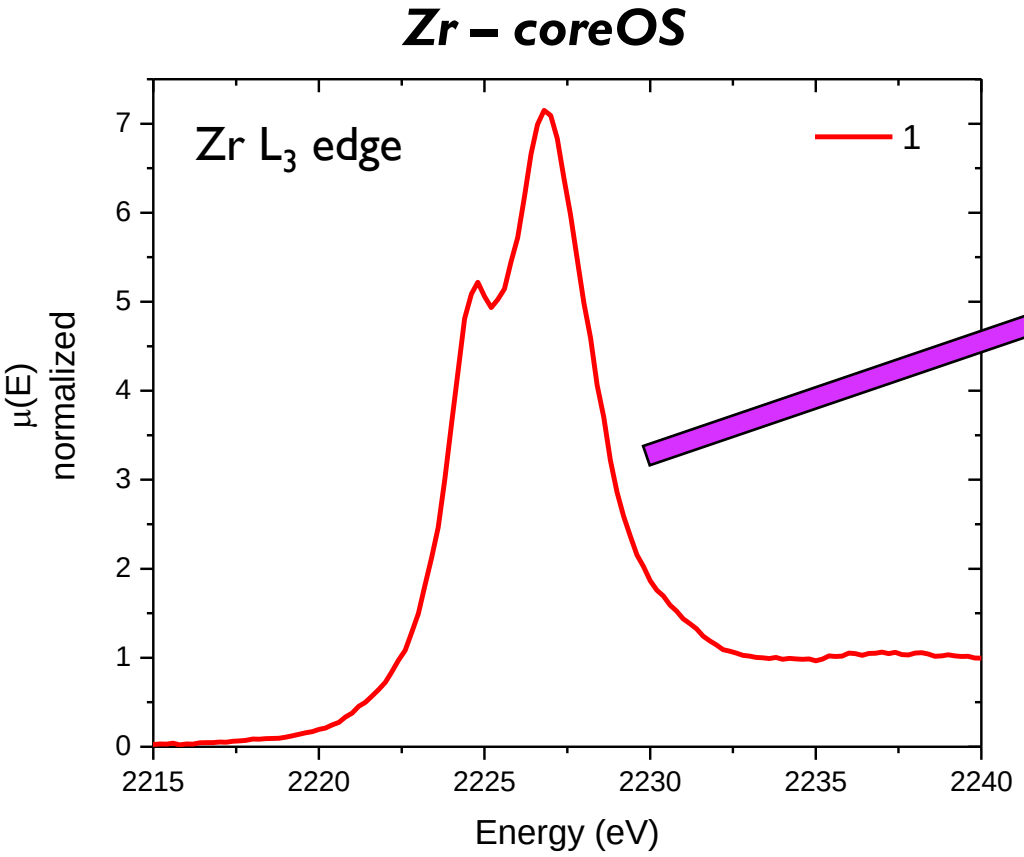

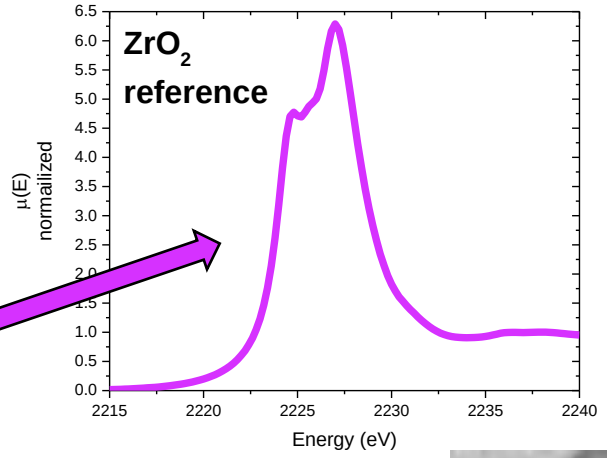

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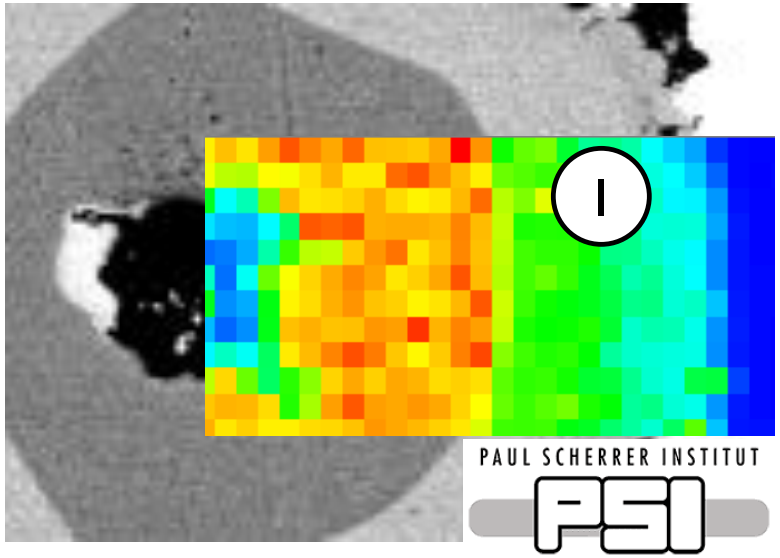

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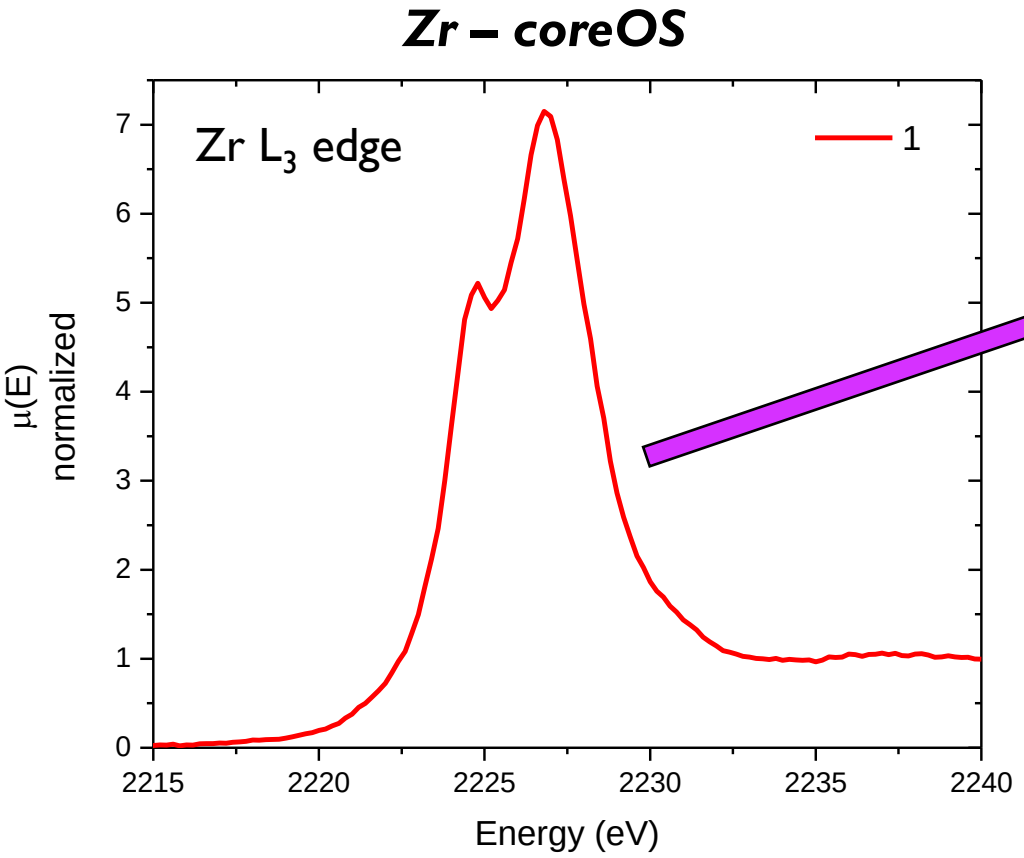

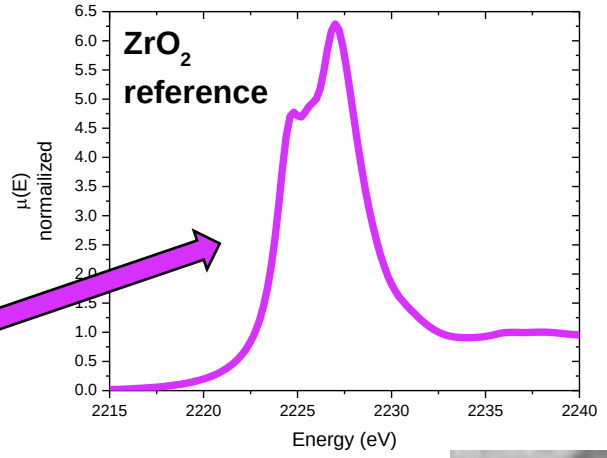

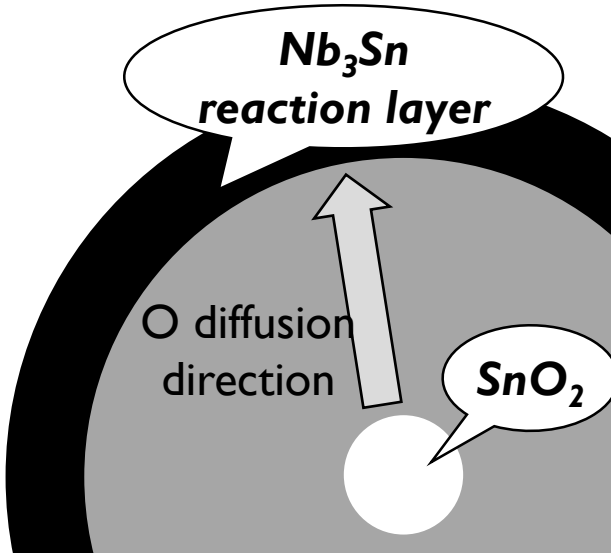

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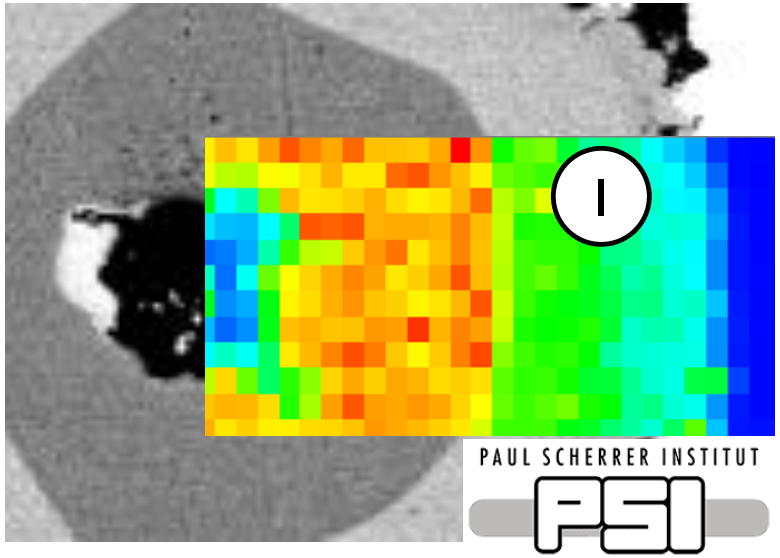

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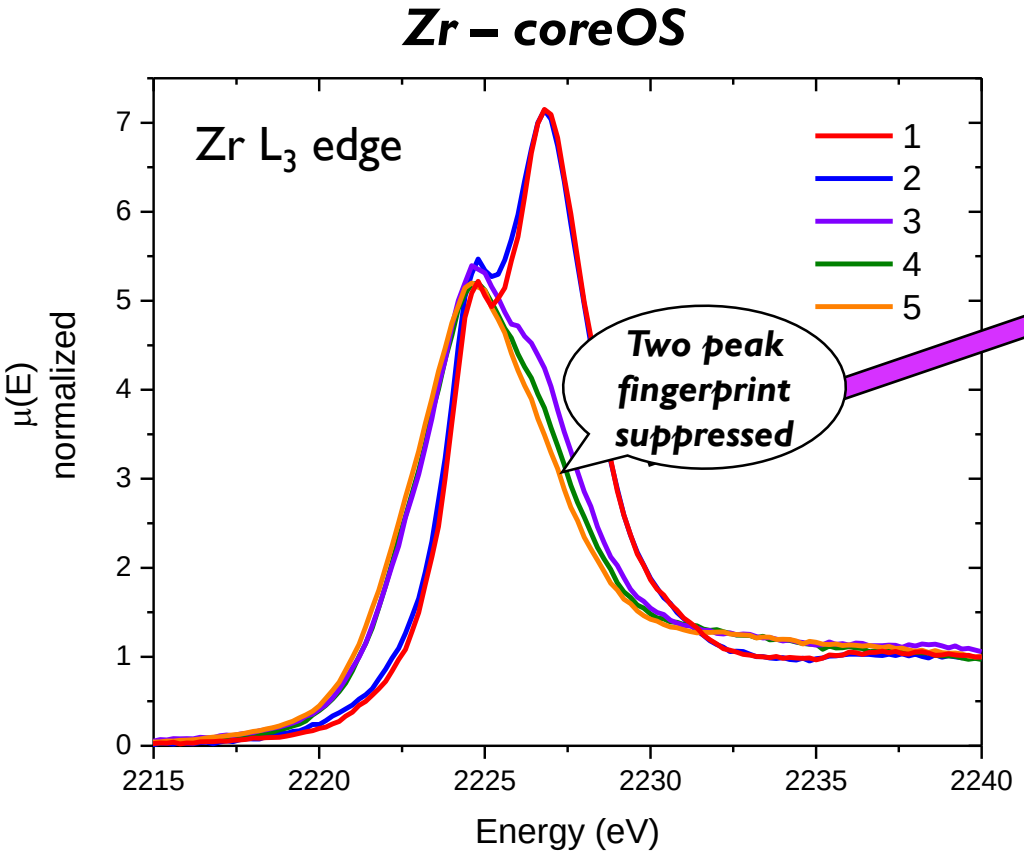
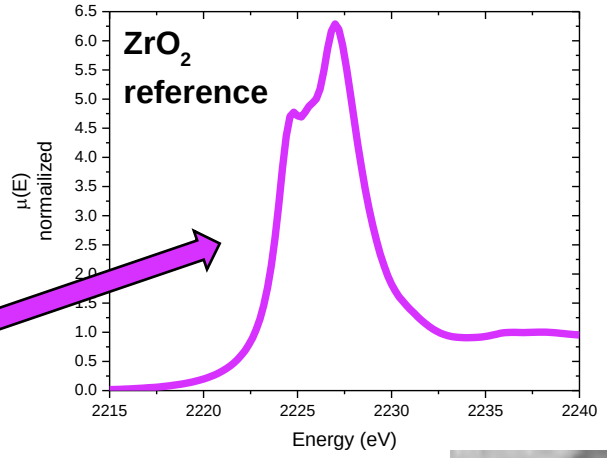
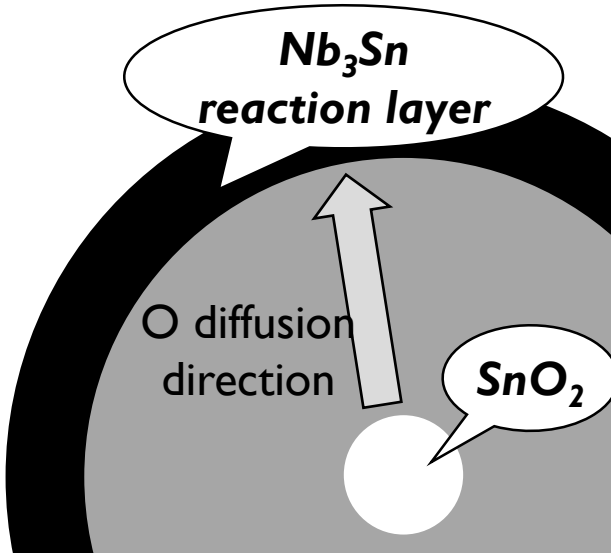
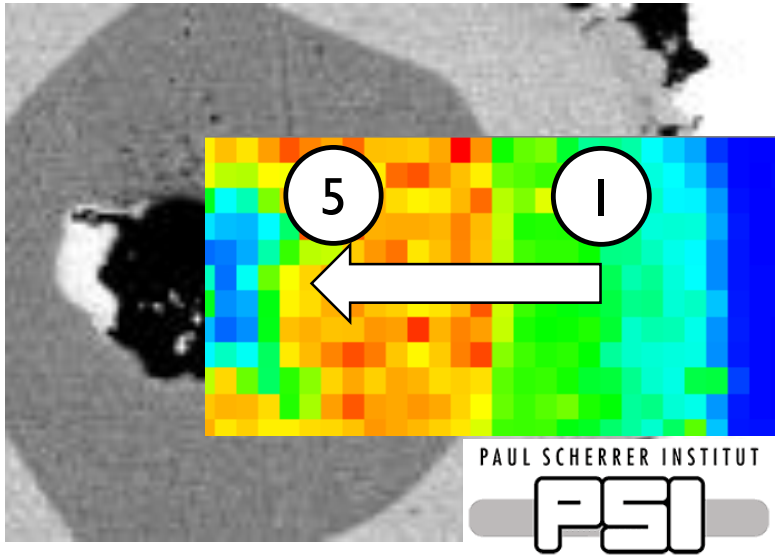


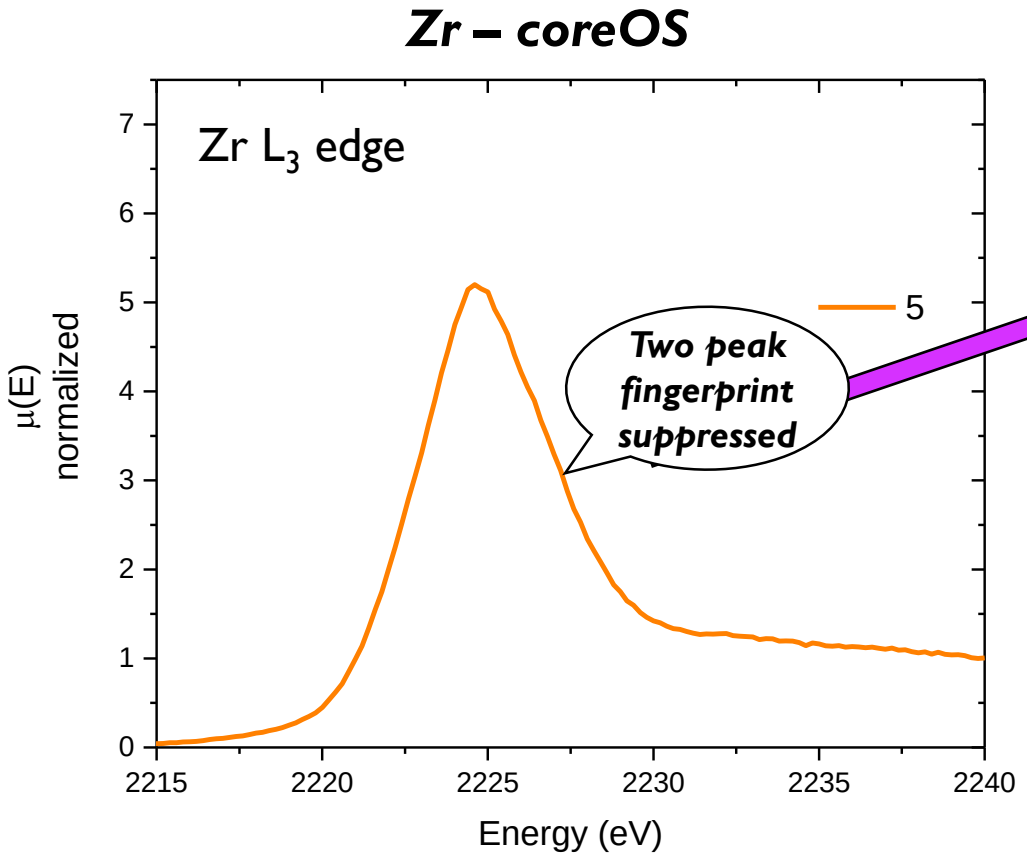


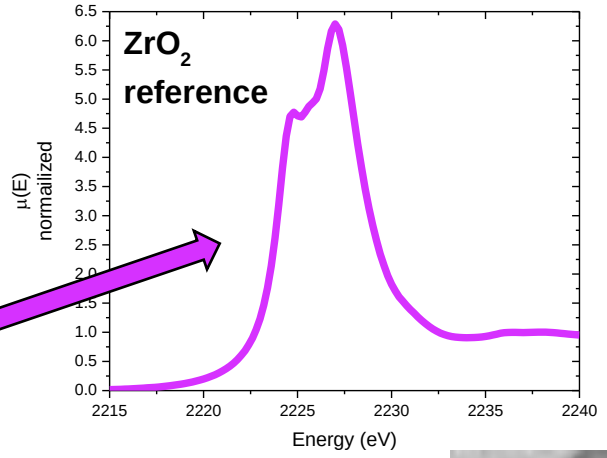


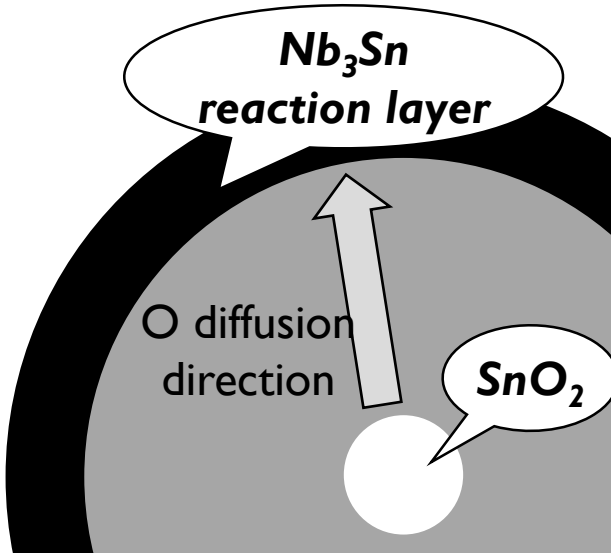
Atomic specific *technique, sensitive to the* ***chemistry*** *and to* ***crystal environment***



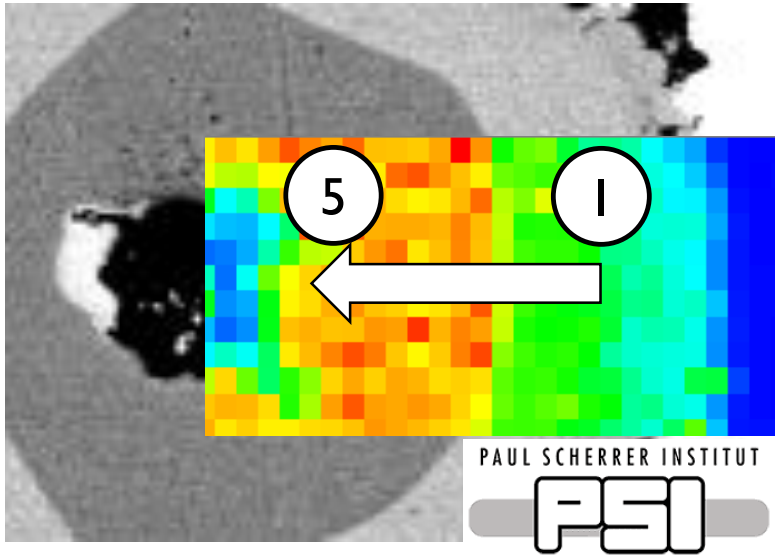
An X-Ray Absorption Spectroscopy study at the PHOENIX beamline of PSI





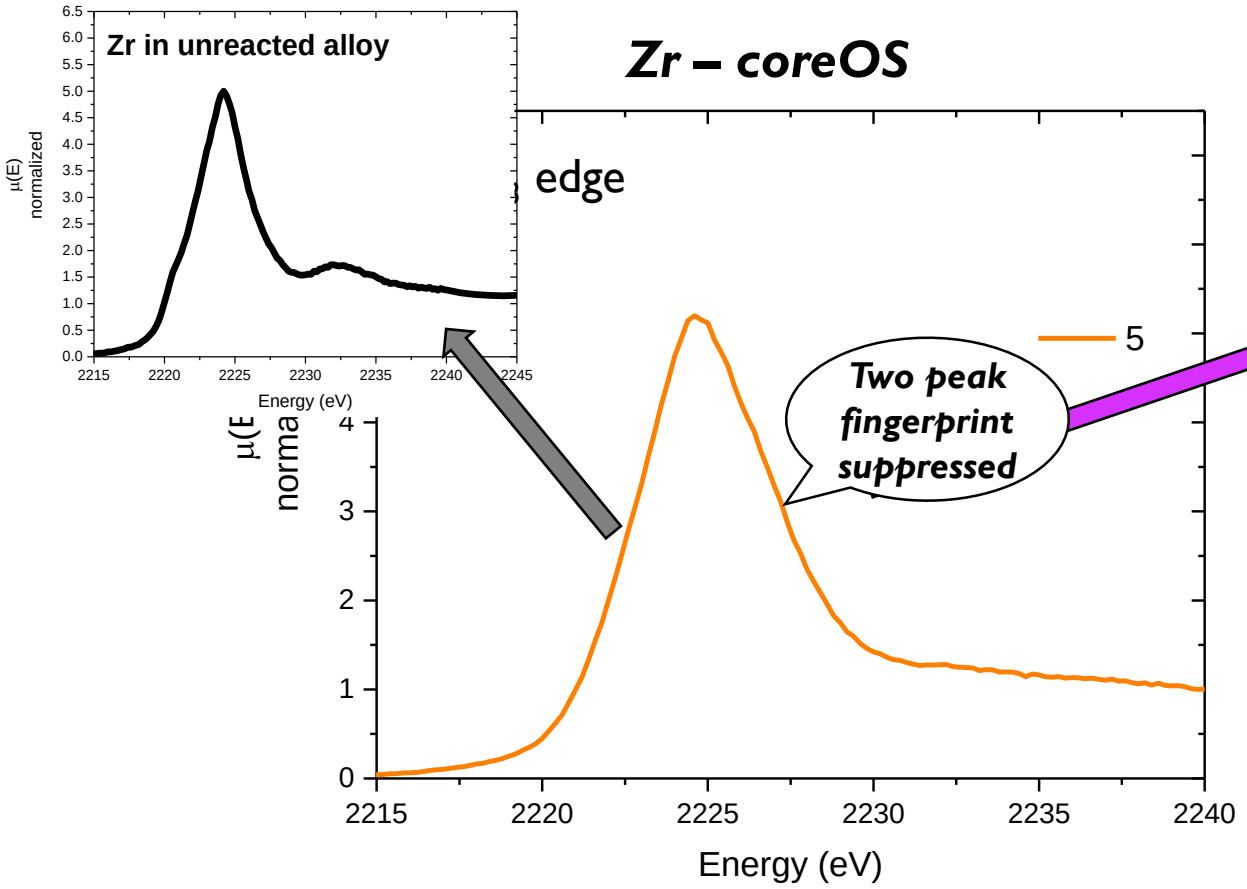


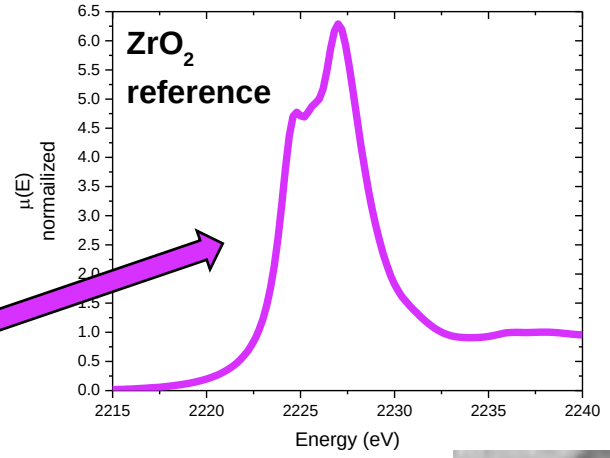
Atomic specific *technique, sensitive to the* ***chemistry*** *and to* ***crystal environment***

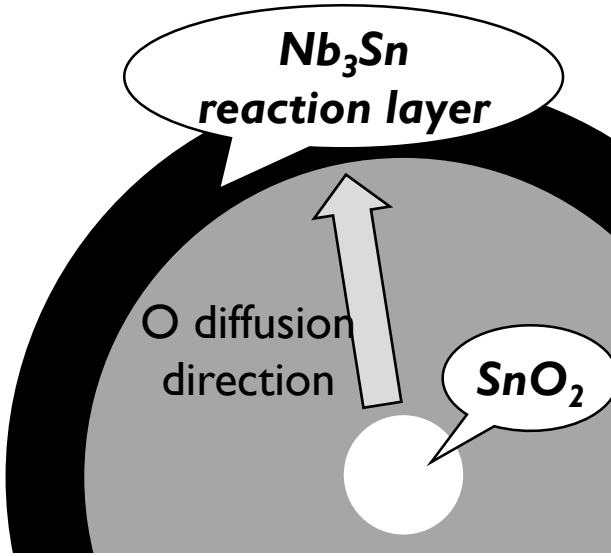


PAUL SCHERRER INSTITUT

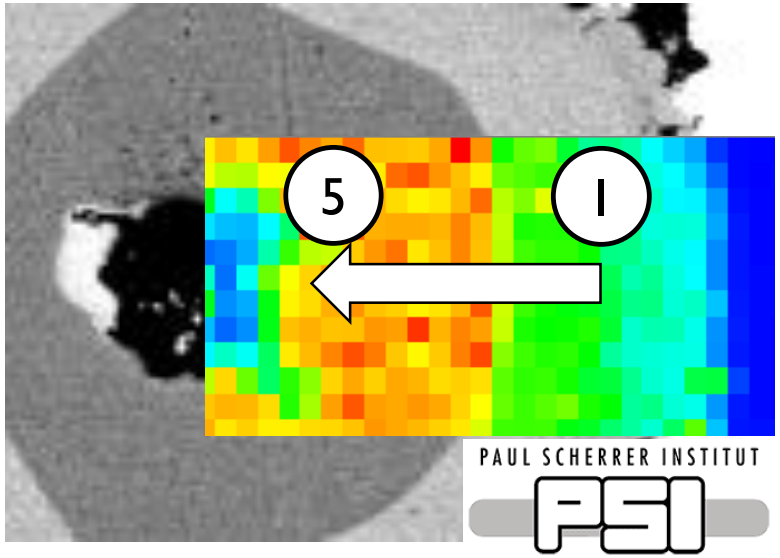
An X-Ray Absorption Spectroscopy study at the PHOENIX beamline of PSI





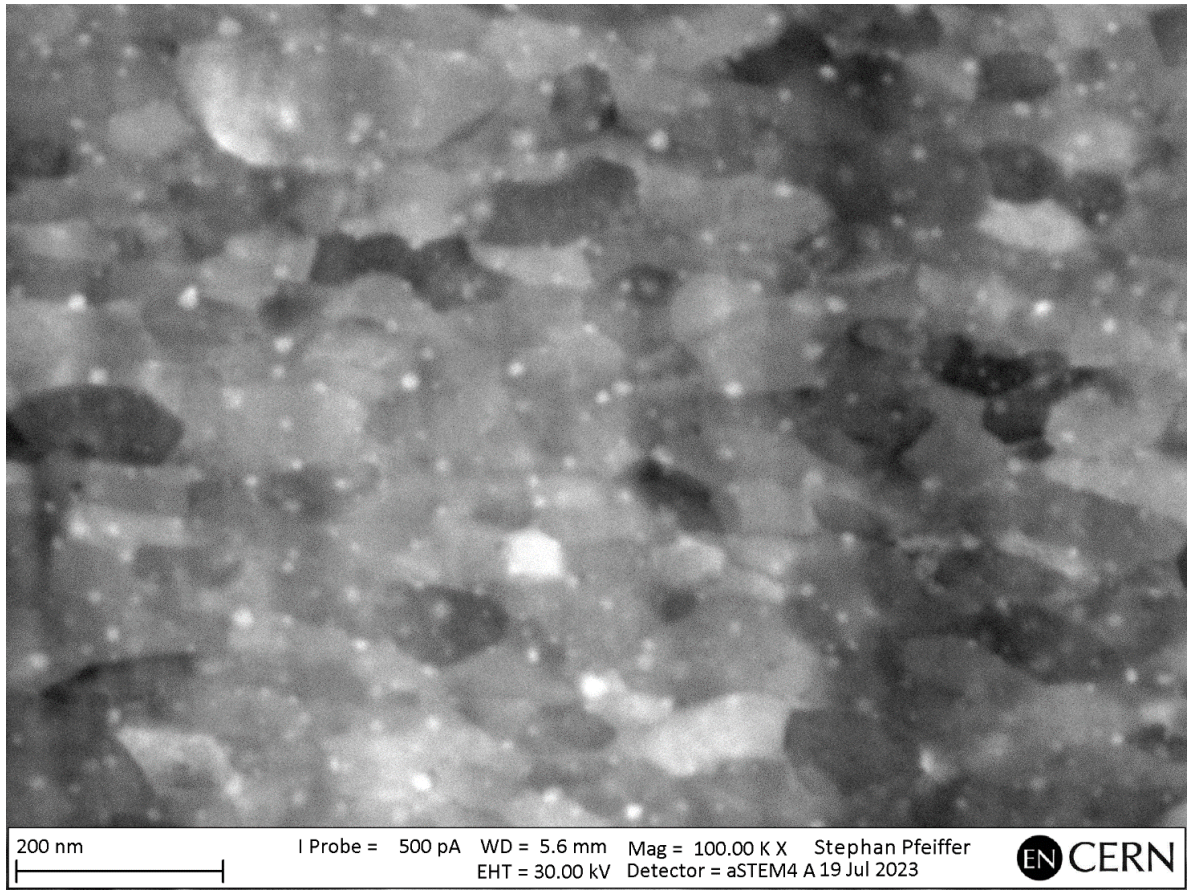


Atomic specific *technique, sensitive to the* ***chemistry*** *and to* ***crystal environment***



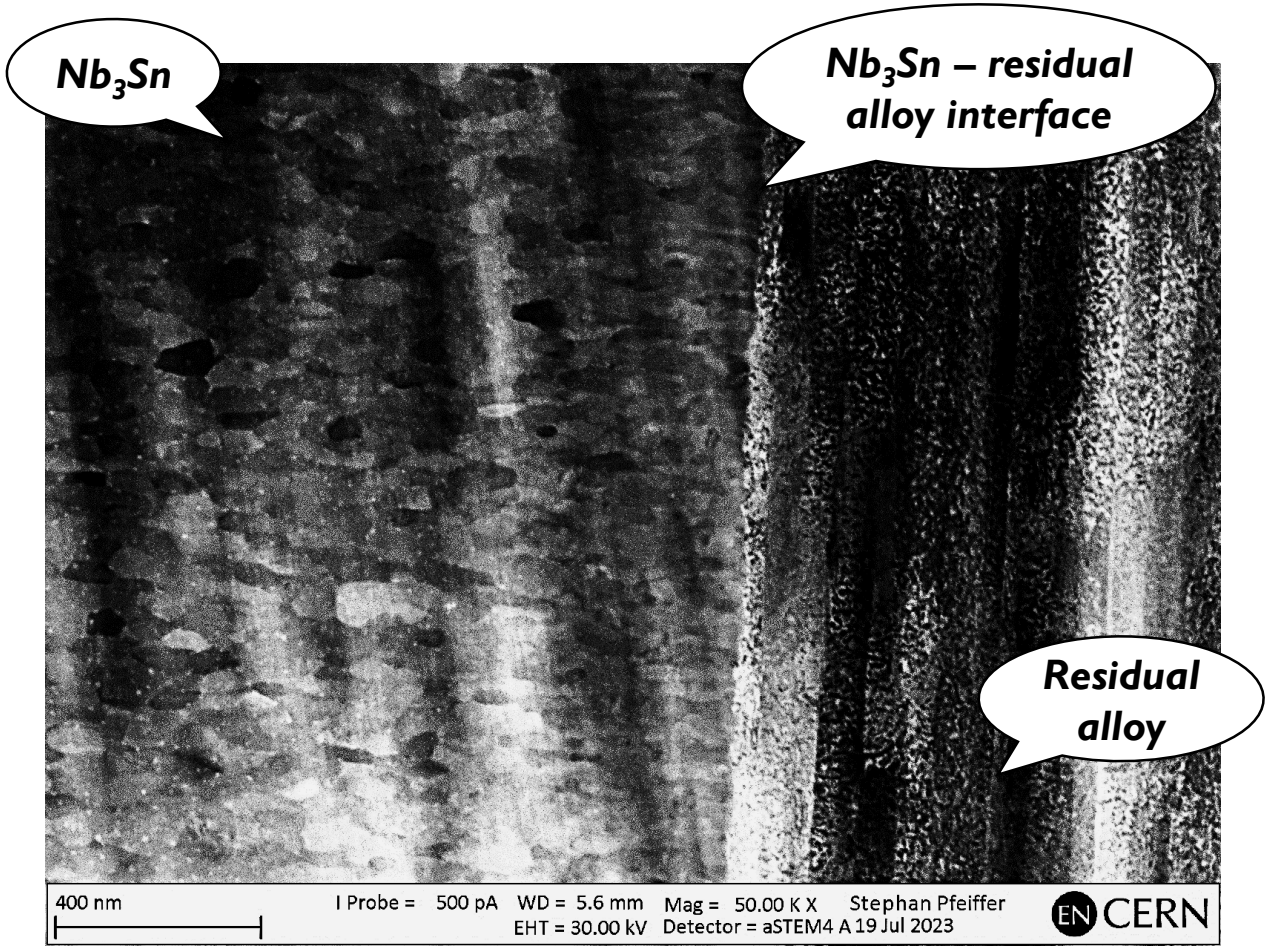
ZrO_2–like spectrum found only in Nb_3Sn!
Different Zr spectrum in residual alloy, despite oxygen diffusion

Precipitates in Nb_3Sn



Nb_3Sn grain STEM image of Zr-annularOS sample

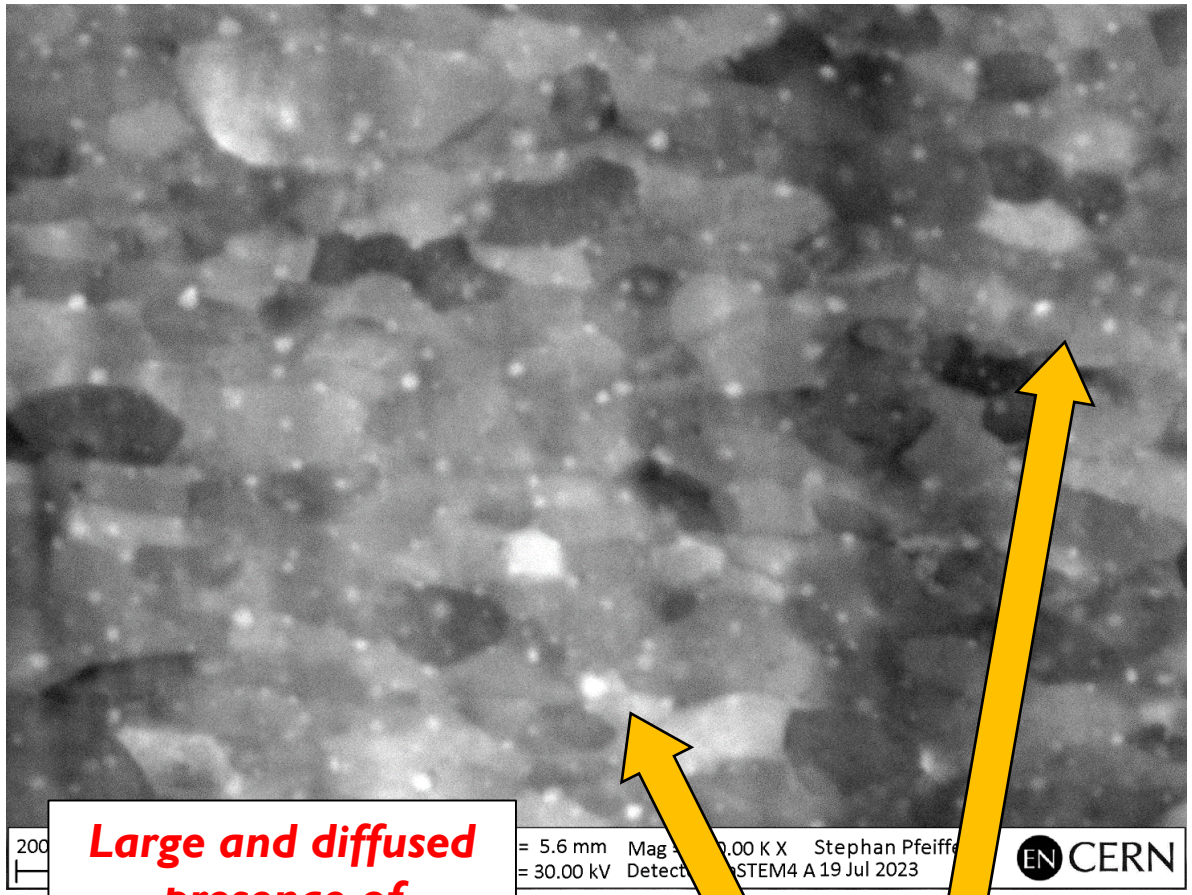
Courtesy of Stephan Pfeiffer, CERN

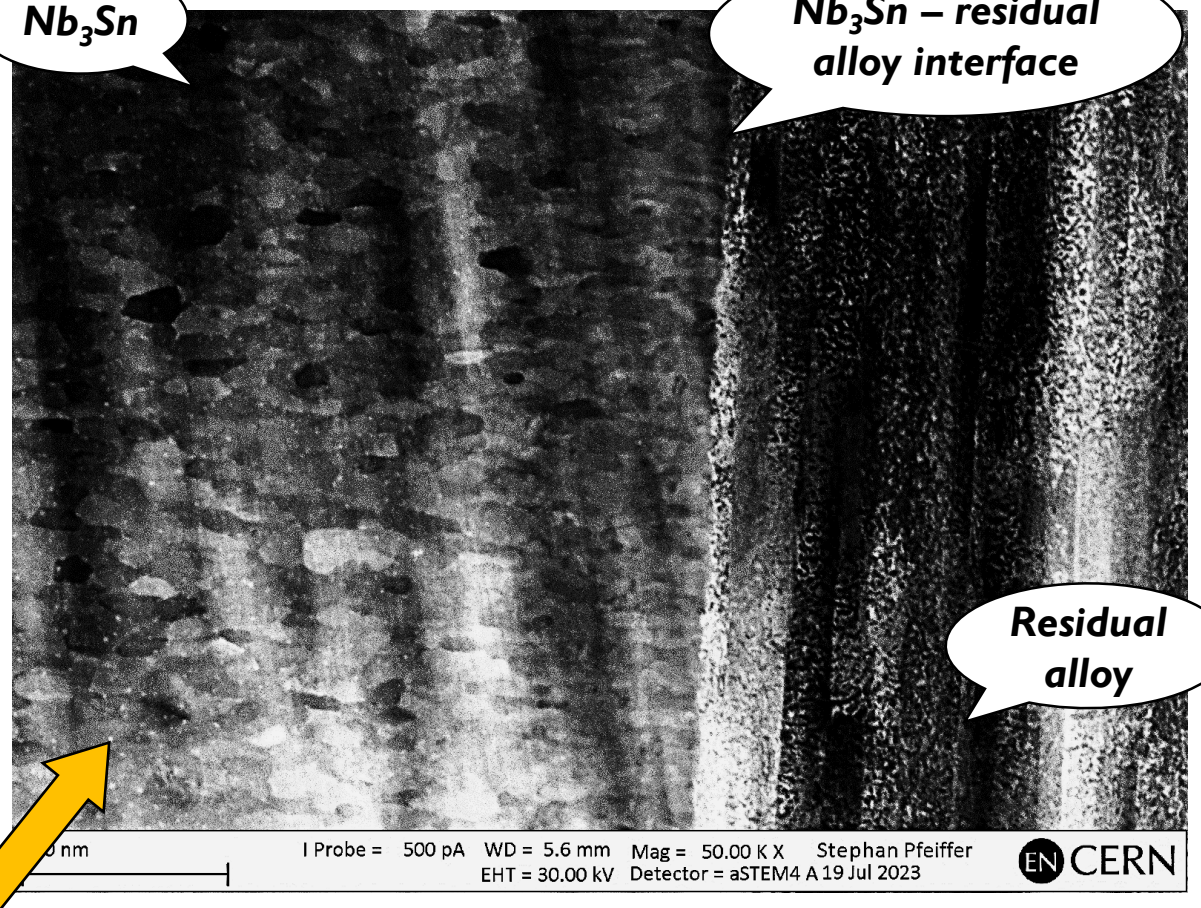


Precipitates in Nb_3Sn

Nb_3Sn grain STEM image of Zr-annularOS sample

Courtesy of Stephan Pfeiffer, CERN



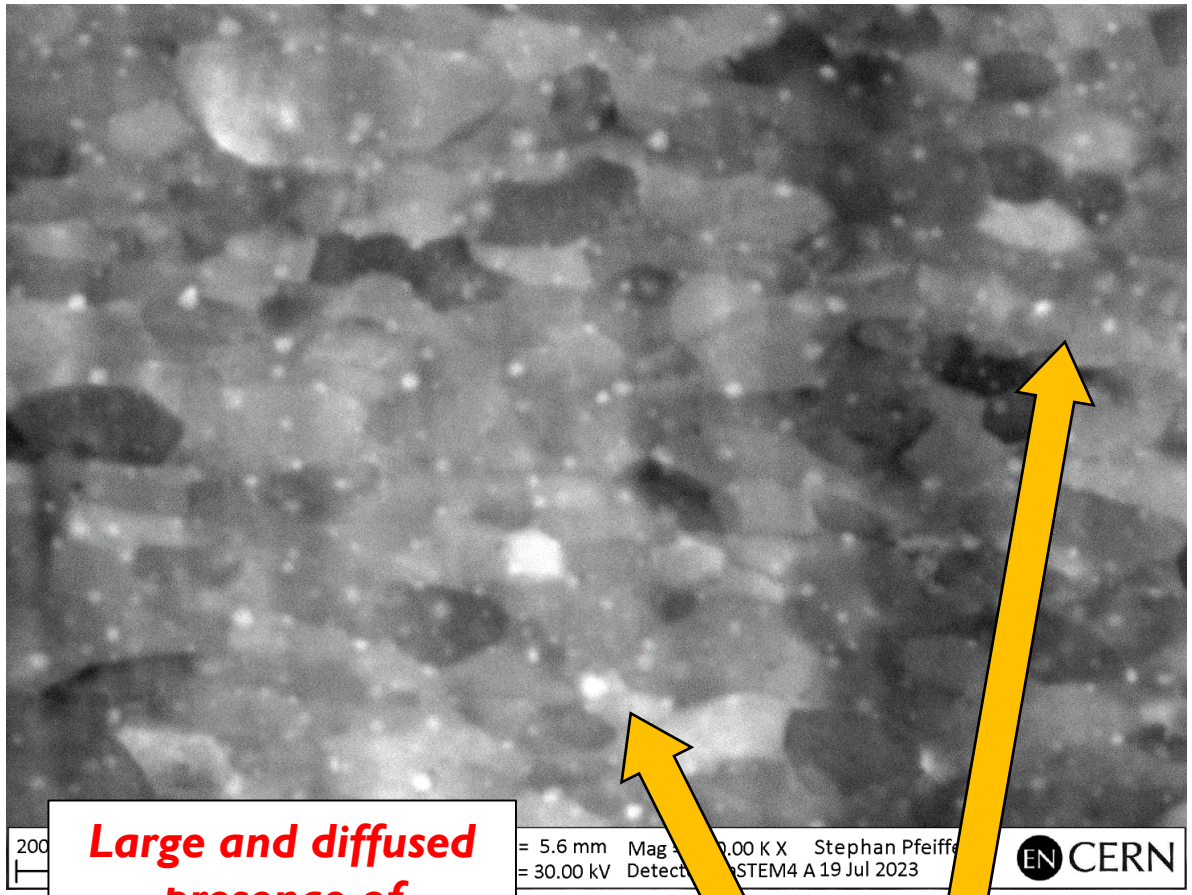


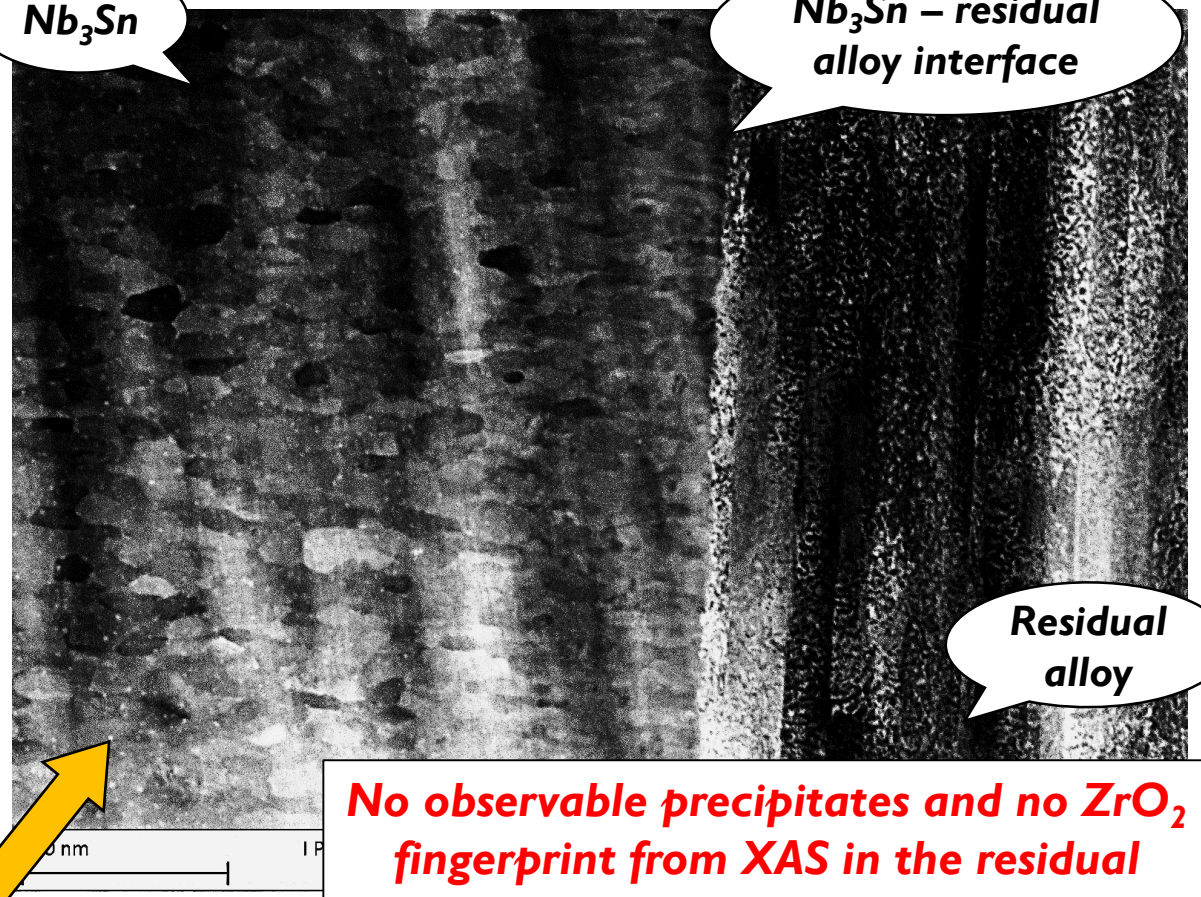
Large and diffused presence of precipitates, ZrO_2

ZrO_2 precipitates

Precipitates in Nb_3Sn

Nb_3Sn grain STEM image of Zr-annularOS sample
Courtesy of Stephan Pfeiffer, CERN





Large and diffused presence of precipitates, ZrO_2

ZrO_2 precipitates

No observable precipitates and no ZrO_2 fingerprint from XAS in the residual alloy. Zr-O clusters may be present

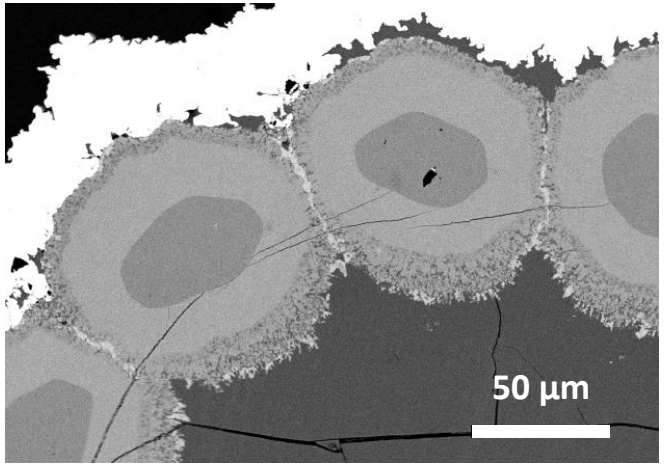
- Hall, Ernest L., et al. *MRS Online Proceedings Library (OPL)* 205 (1990).

Heat treatment optimization: reaction layer thickness

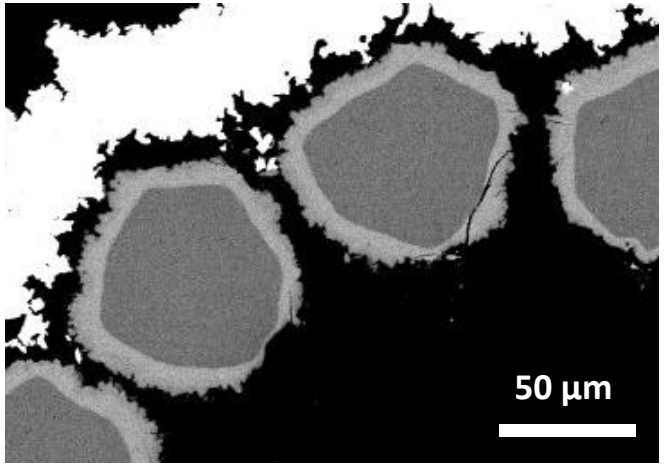
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS



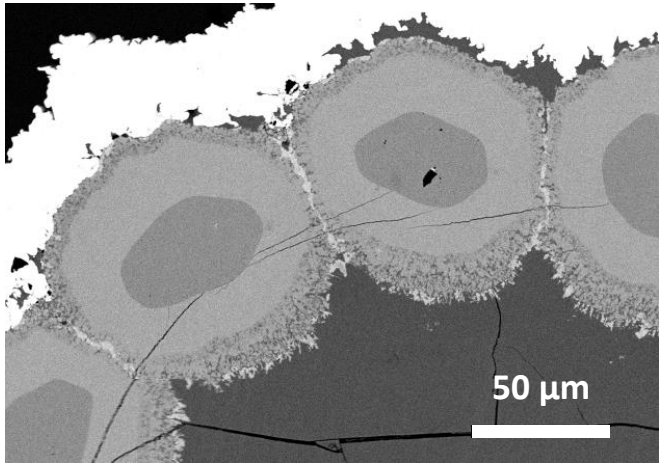
*Drastic reduction of **Nb_3Sn layer thickness** when OS is added*

Heat treatment optimization: reaction layer thickness

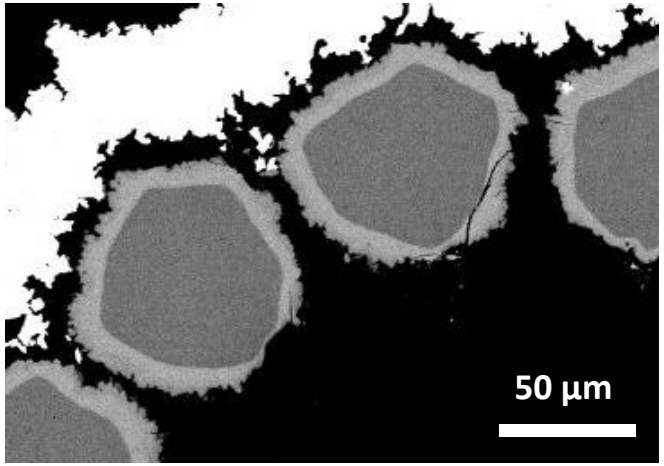
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS



*Drastic reduction of **Nb_3Sn layer thickness** when OS is added*

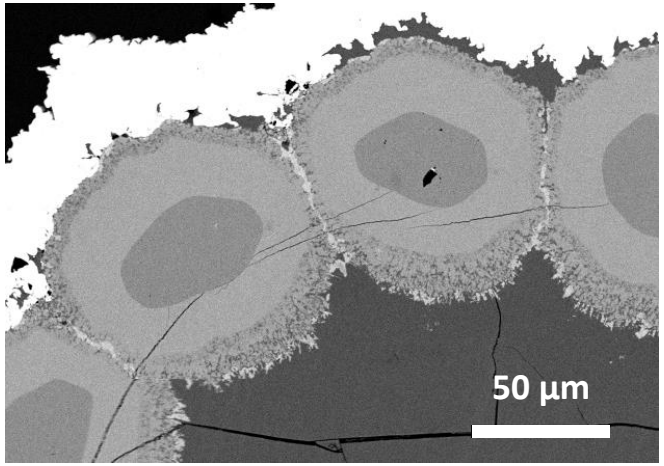
Higher temperature to enlarge
Nb_3Sn layer thickness
*keeping **grain size** low*

Heat treatment optimization: reaction layer thickness

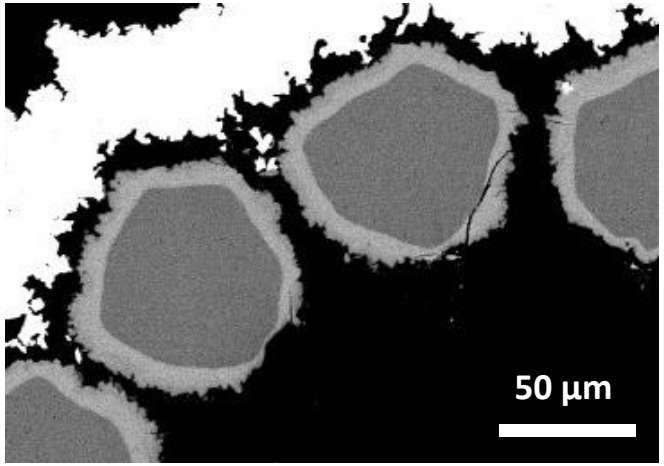
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS

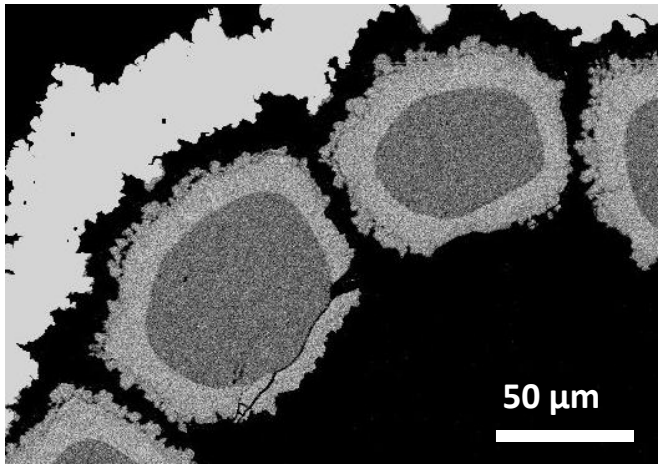


*Drastic reduction of **Nb_3Sn layer thickness** when OS is added*

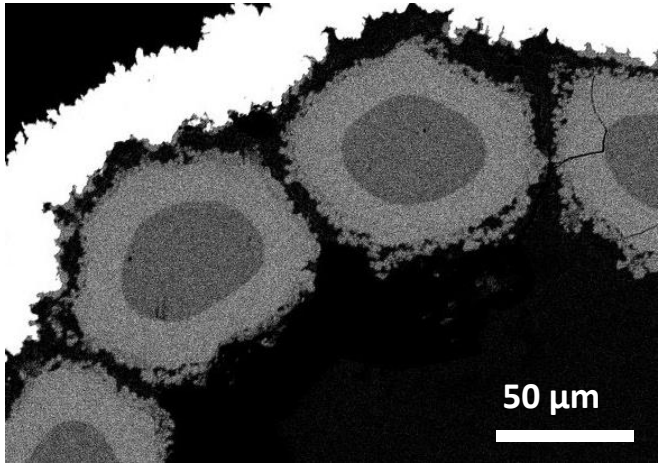
Higher temperature to enlarge
Nb_3Sn layer thickness
*keeping **grain size** low*

With OS

700 °C × 50 h



700 °C × 100 h

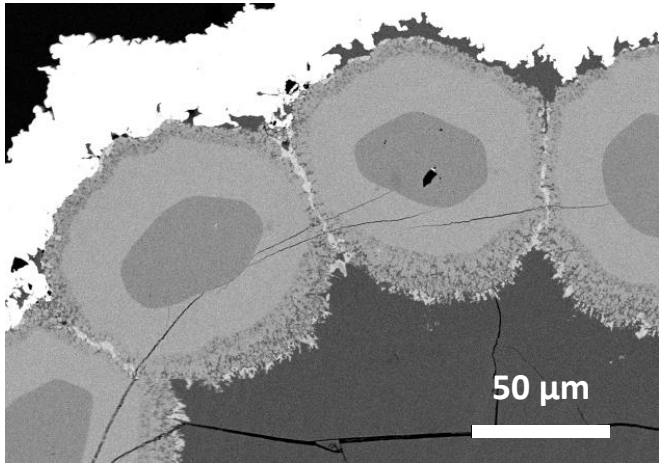


Heat treatment optimization: reaction layer thickness

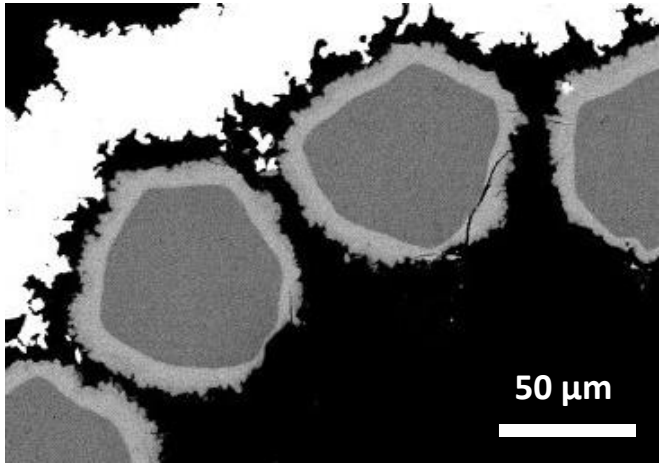
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS



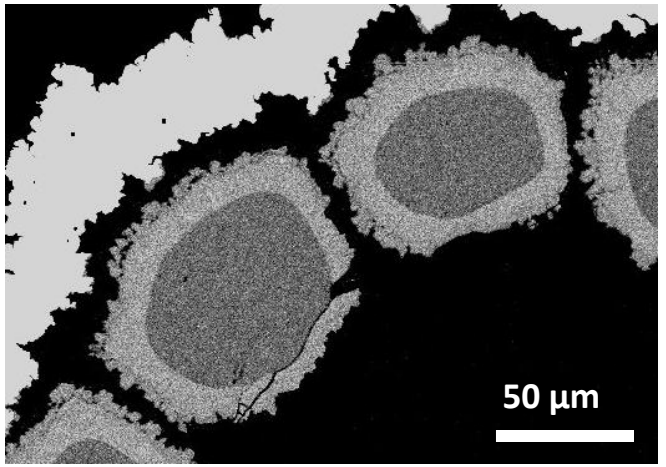
*Drastic reduction of **Nb_3Sn layer thickness** when OS is added*

*Higher temperature to enlarge Nb_3Sn layer thickness keeping **grain size** low*

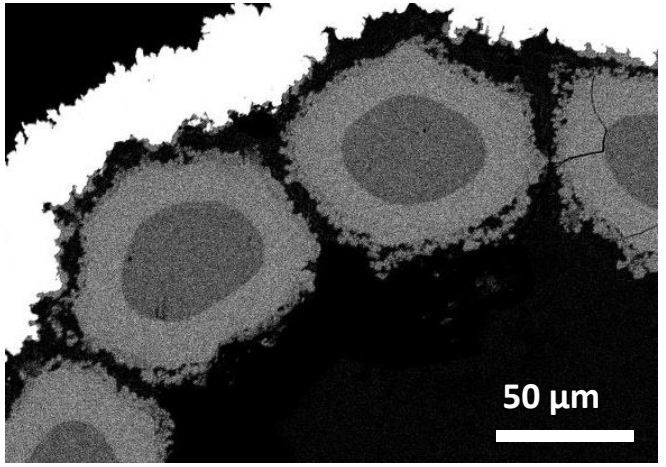
Significant increase of layer thickness at 700 °C

With OS

700 °C × 50 h



700 °C × 100 h

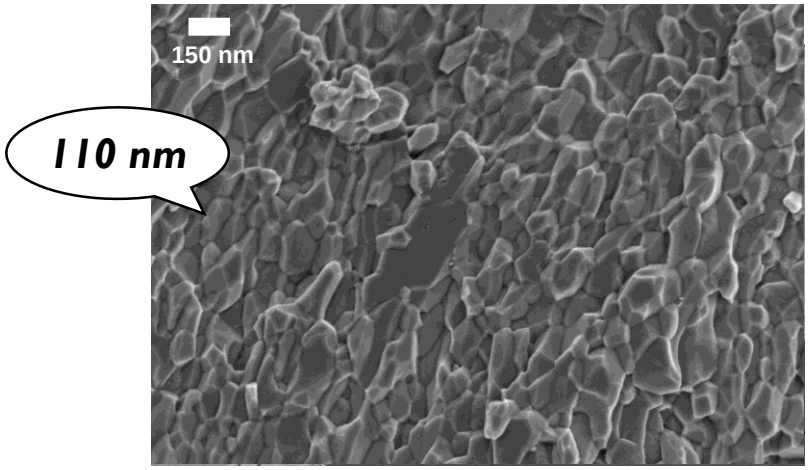


Heat treatment optimization: reaction layer thickness

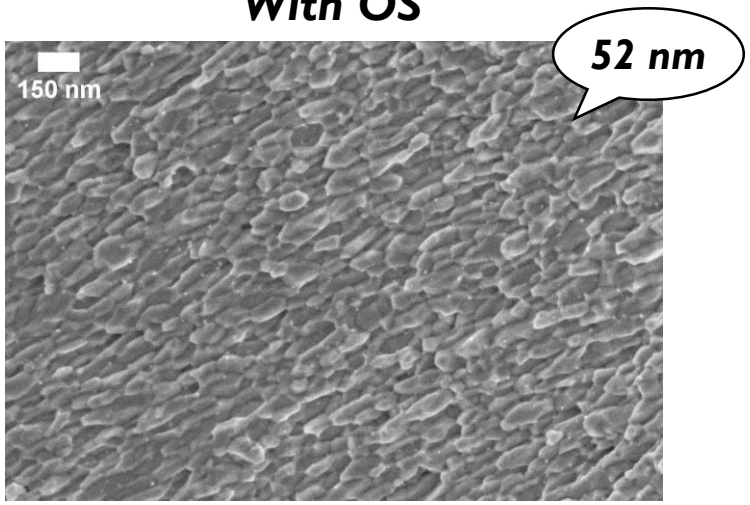
Heat treatment (HT):

550°C × 100 h + 650°C × 200 h

No OS



With OS



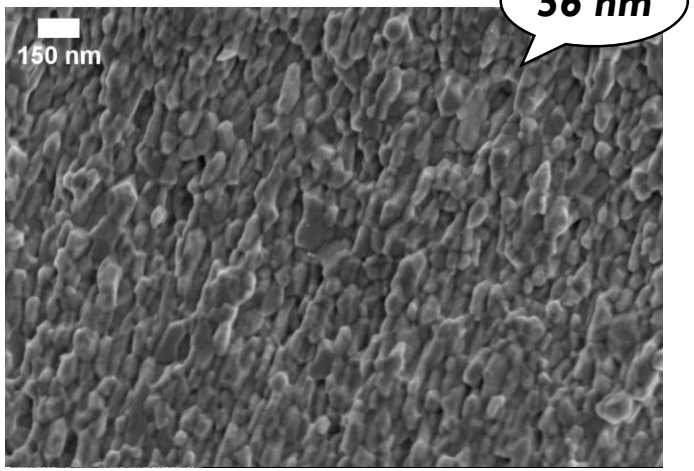
*Drastic reduction of **Nb_3Sn layer thickness** when OS is added*

*Higher temperature to enlarge Nb_3Sn layer thickness keeping **grain size** low*

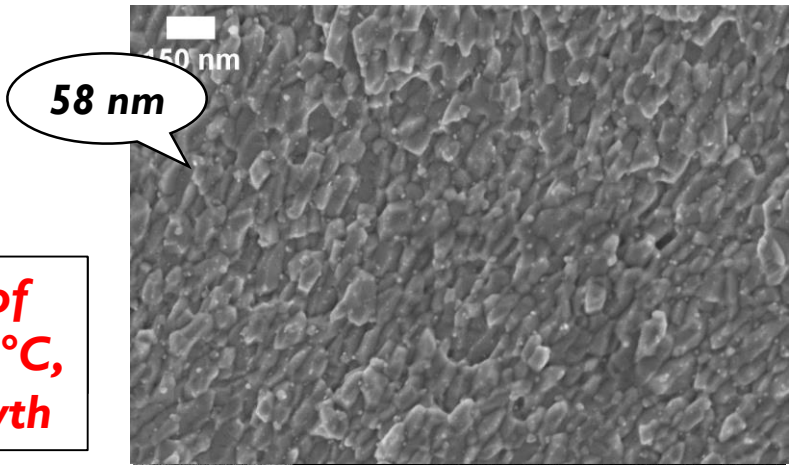
Significant increase of layer thickness at 700 °C, but modest grain growth

With OS

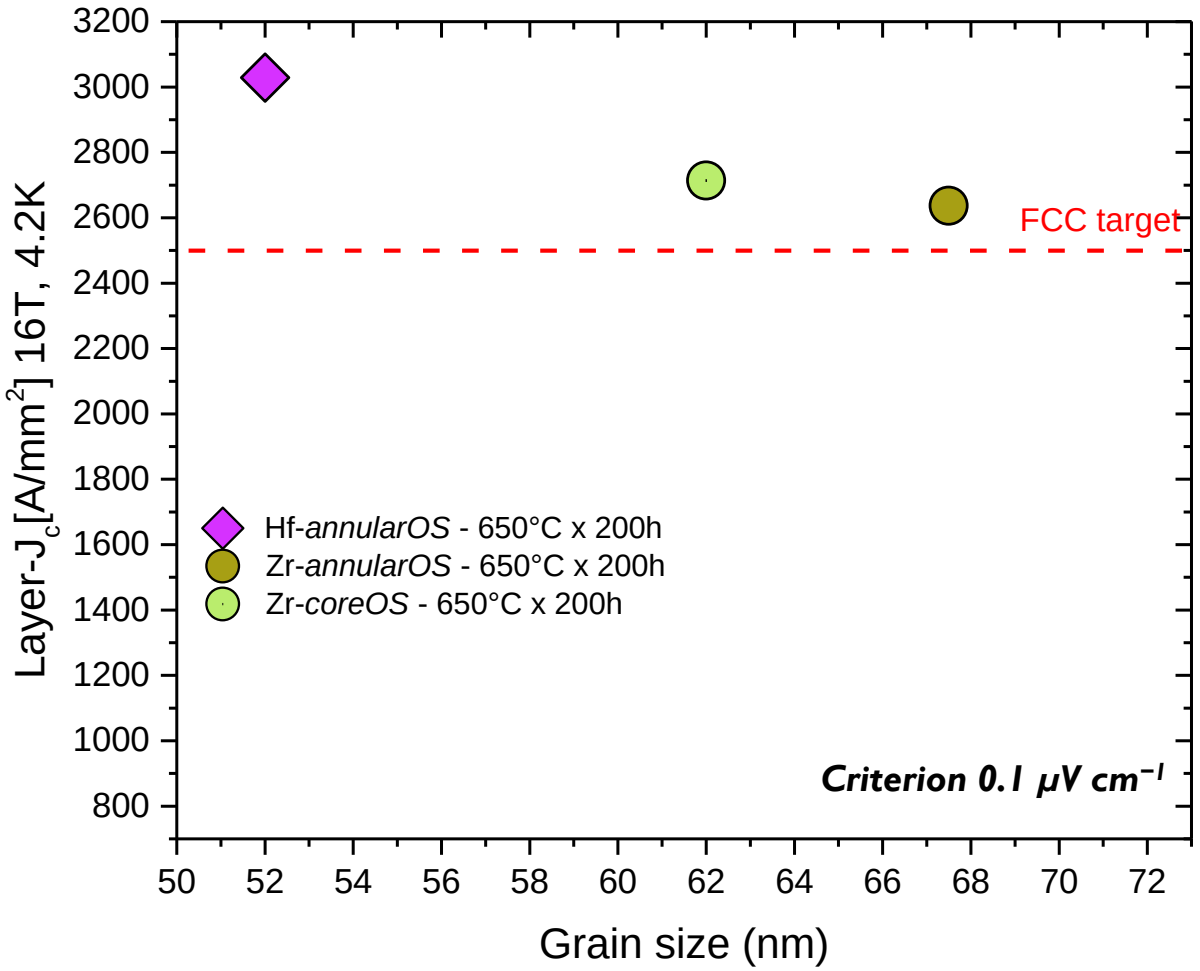
700 °C × 50 h

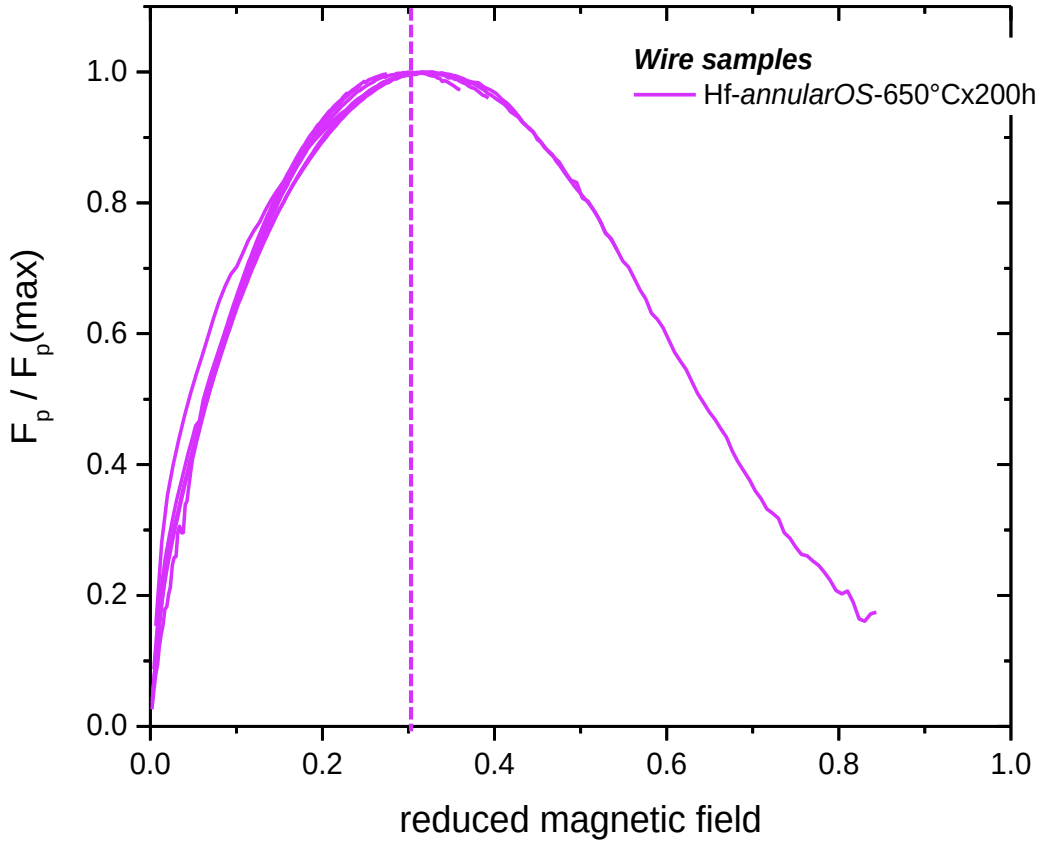


700 °C × 100 h

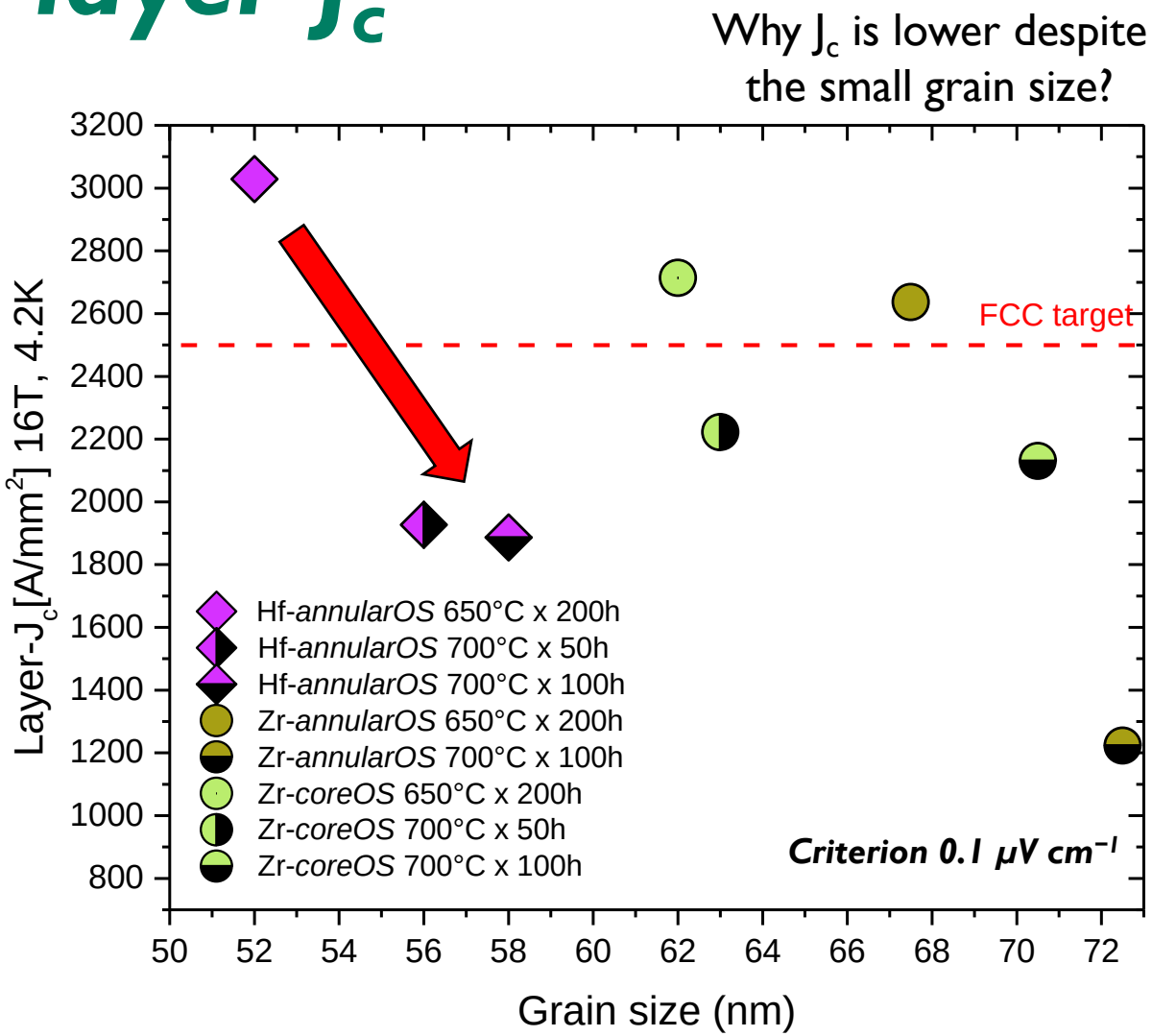


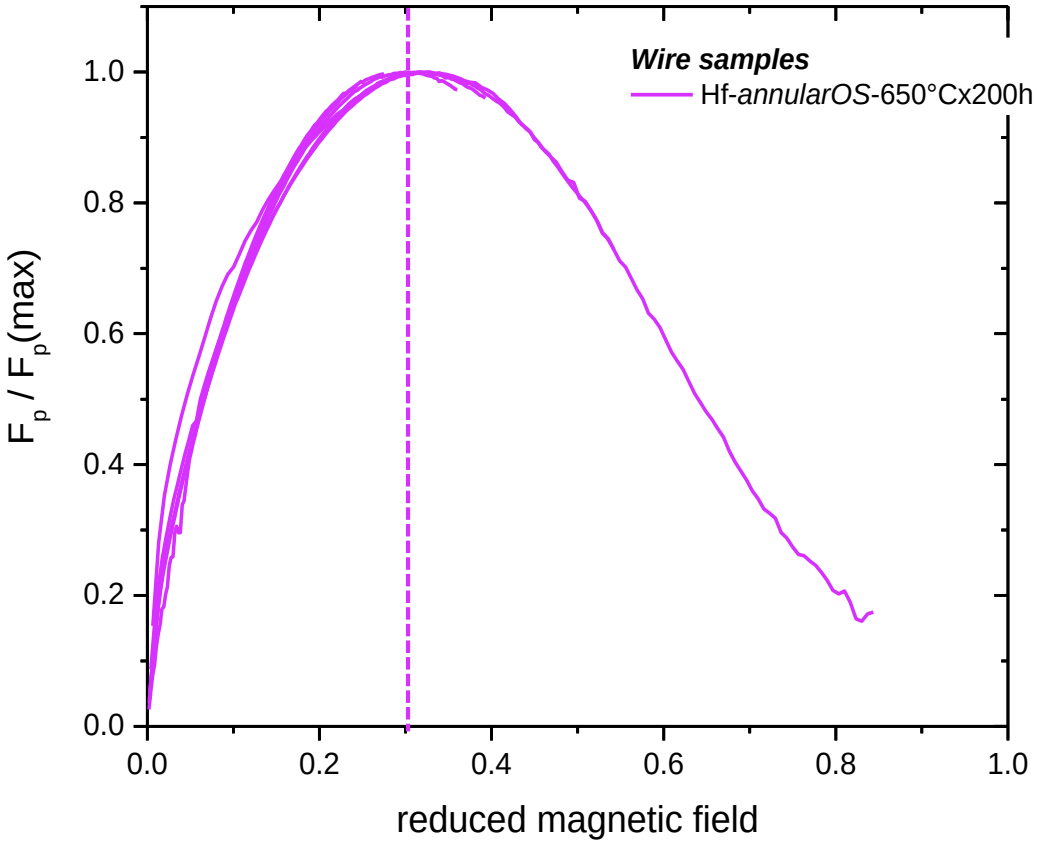
Heat treatment optimization: layer-J_c



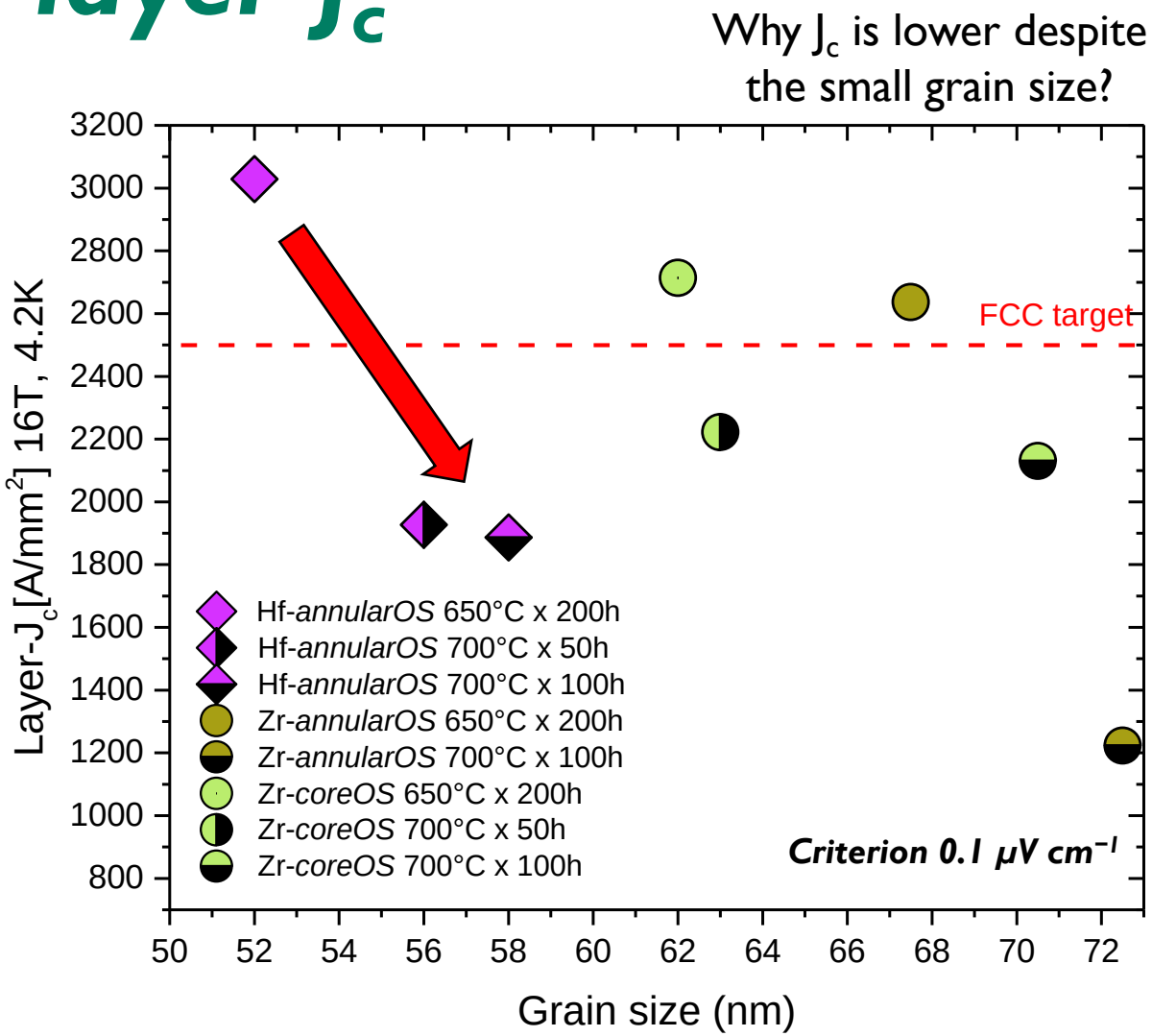


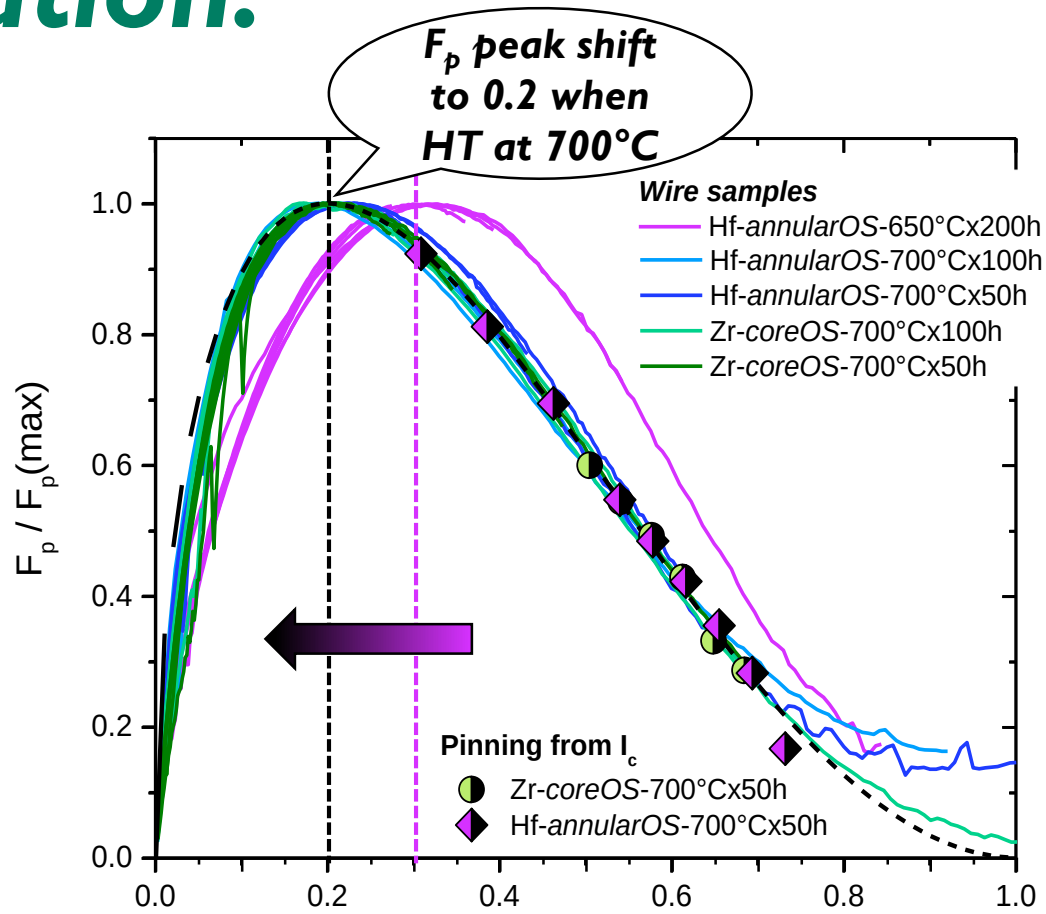
Heat treatment optimization: layer-J_c





Heat treatment optimization: layer-J_c



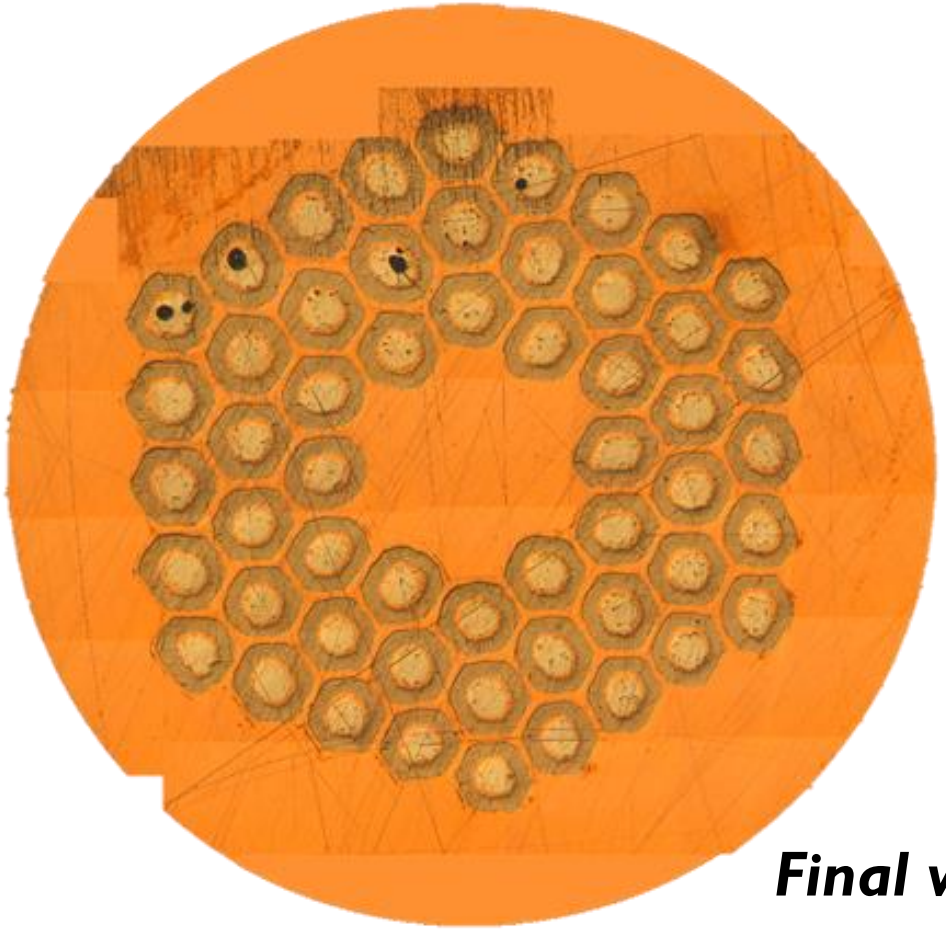


Excessive growth of precipitates at 700 °C, suppressing their contribution to pinning force

- Lonardo F. et al. *Under Submission on IEEE TAS* (2023)

On the road to multifilamentary wires

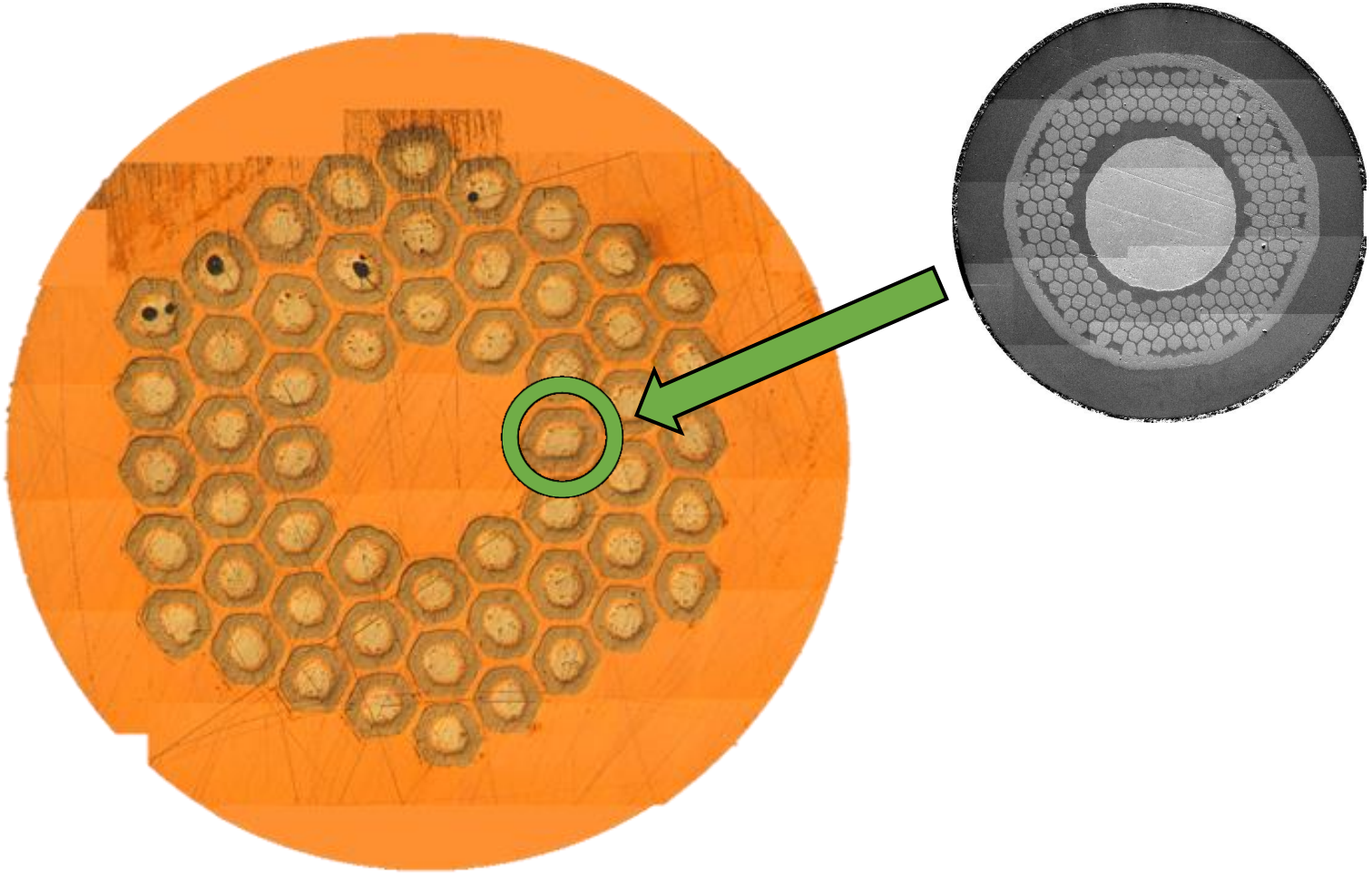
Manufacturing of multifilamentary RRP wire, starting from sub-elements made of 192 filaments.



Final wire

On the road to multifilamentary wires

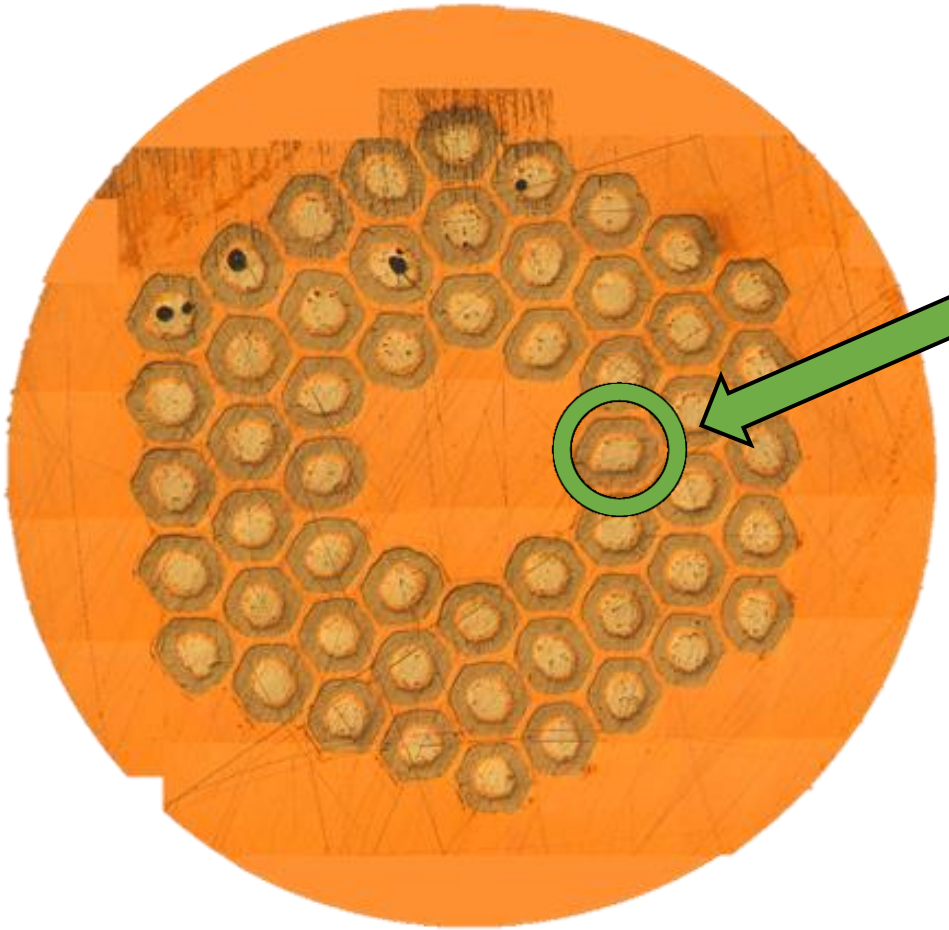
Manufacturing of multifilamentary RRP wire, starting from sub-elements made of 192 filaments.

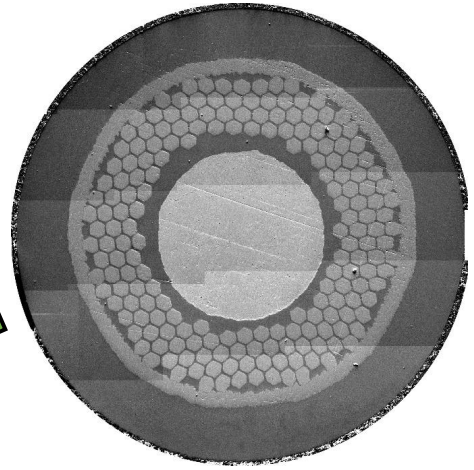


NbTa - noOS
Cu/Nb-7.5%^{wt}Ta
No Oxygen Source

On the road to multifilamentary wires

Manufacturing of multifilamentary RRP wire, starting from sub-elements made of 192 filaments.

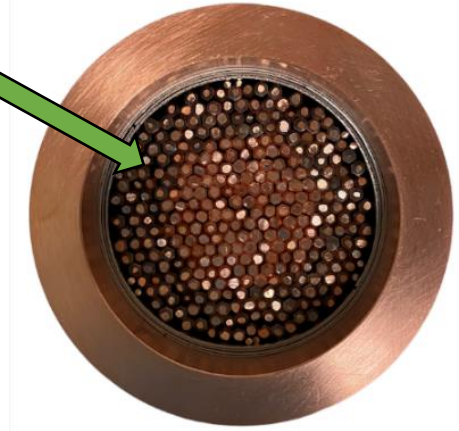




NbTa - noOS
Cu/Nb-7.5%^{wt}Ta
No Oxygen Source

Multifilamentary wire

Cu and Cu/Nb filaments cut and assembled at UNIGE



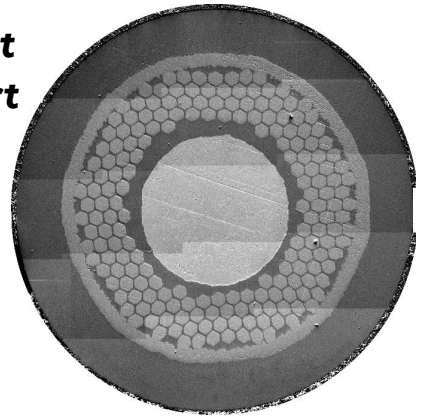
Billet e-Beam weld at CERN

Hot Isostatic Press to remove voids

Hot Isostatic Extrusion

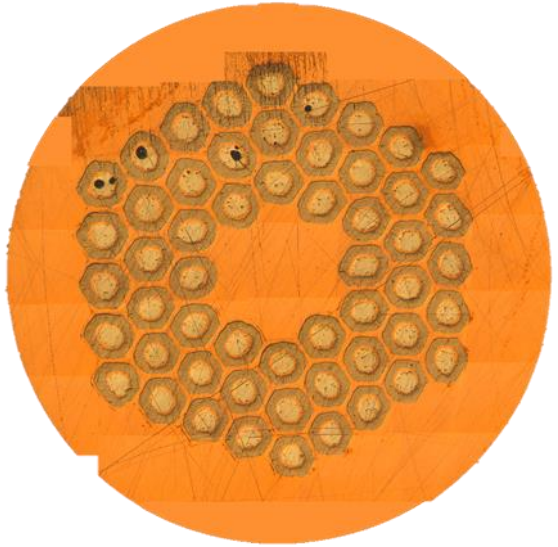
Cold deformation to diameter for next step

Gun-drilling of the Sub-element Cu core to insert Sn rod



Cold deformation to reach restack size

2.45 mm



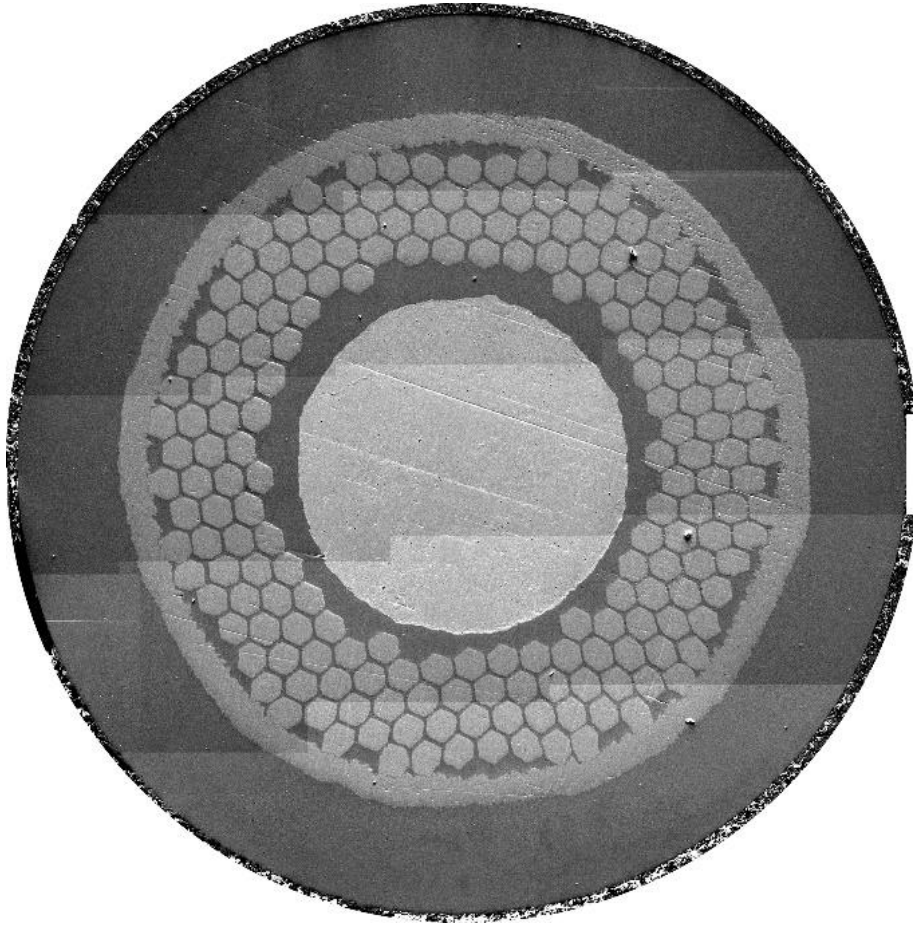
54/61

design

Restack of the sub-element in a Cu tube, and cold deformation to final wire size (1 – 0.7 mm)

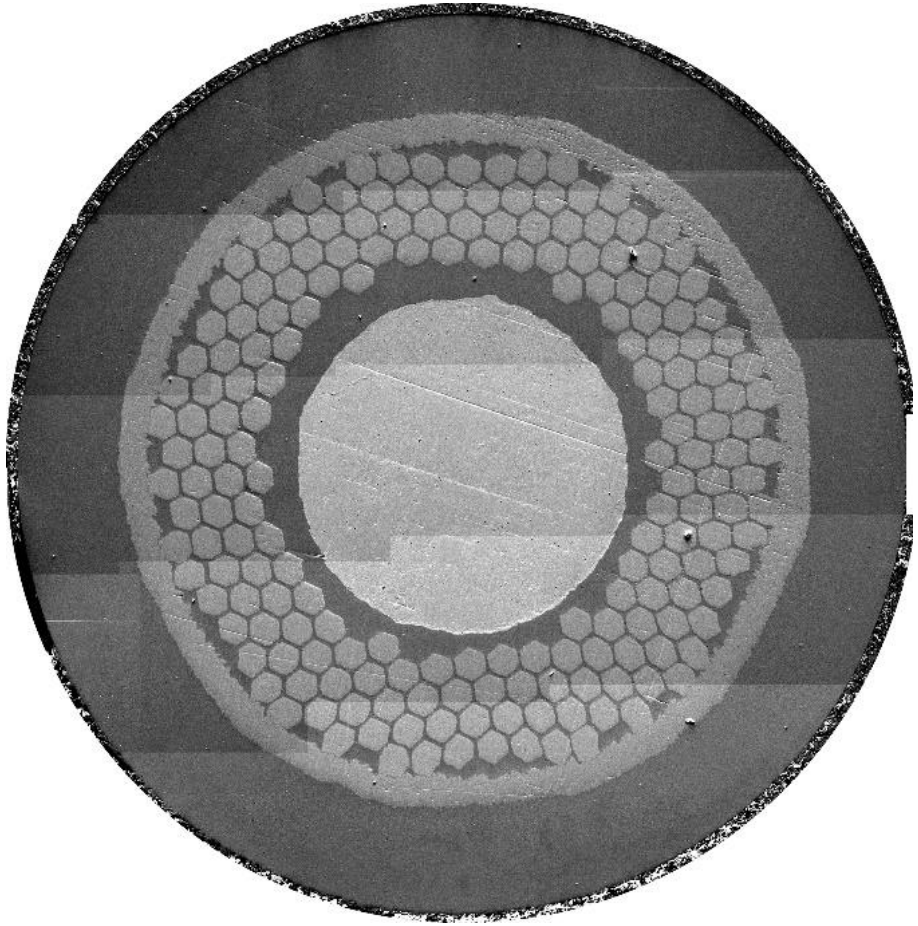
Approaches to implement internal oxidation

Where should we place the OS in a RRP wire?

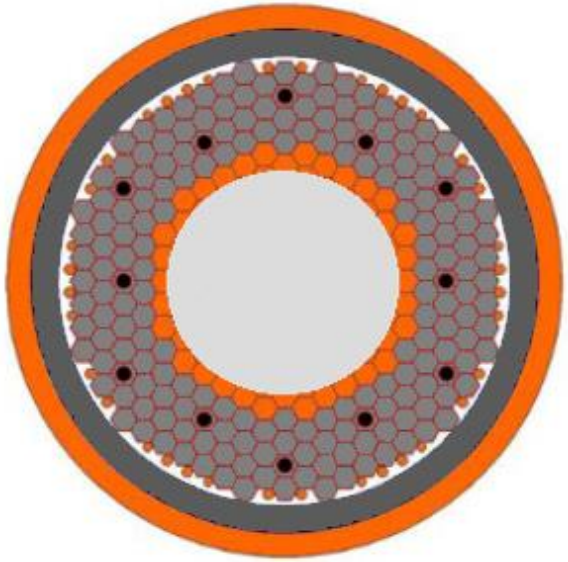


Approaches to implement internal oxidation

Where should we place the OS in a RRP wire?



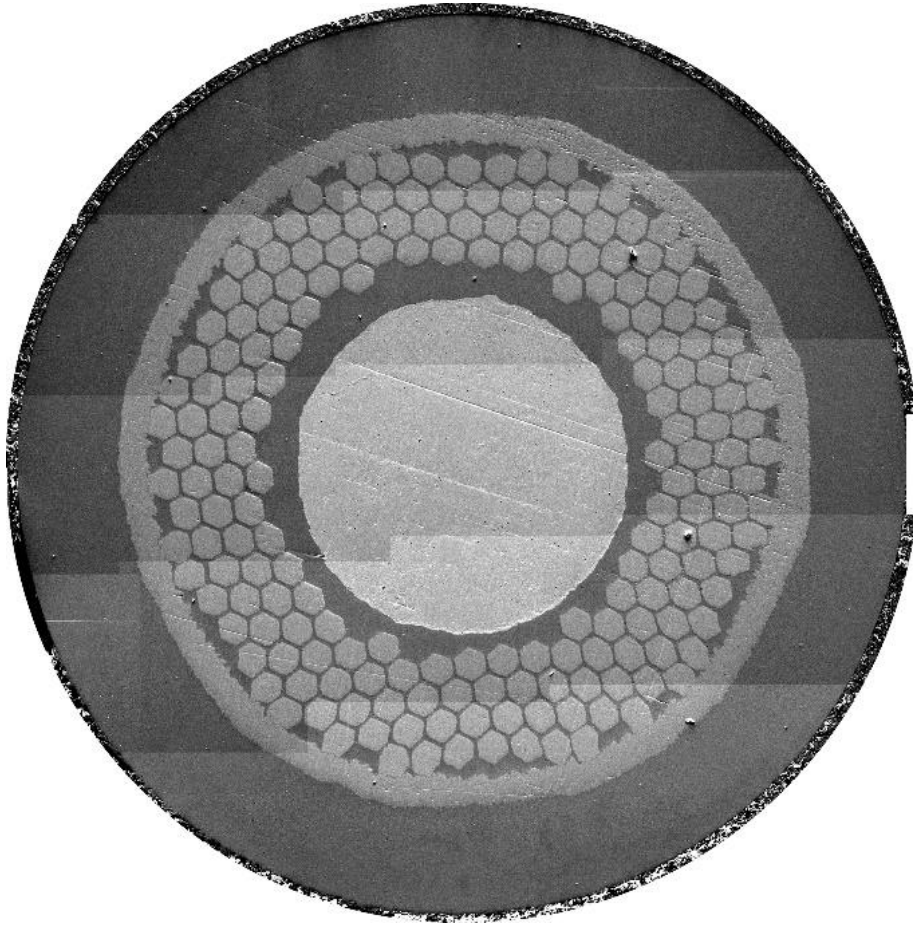
OS at subelement level



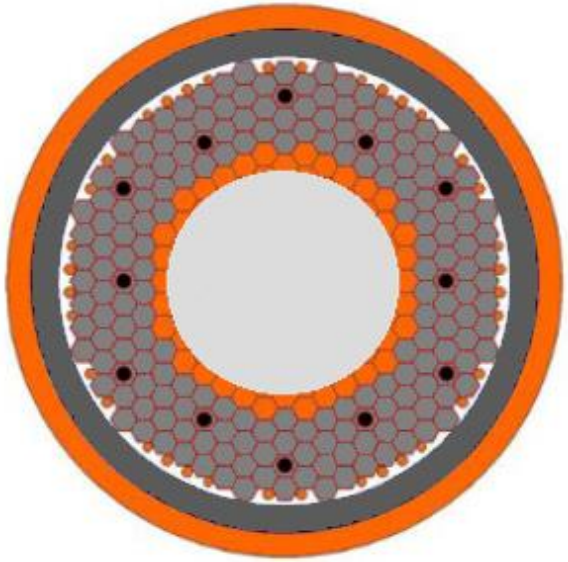
Nb filaments
substituted with OS

Approaches to implement internal oxidation

Where should we place the OS in a RRP wire?

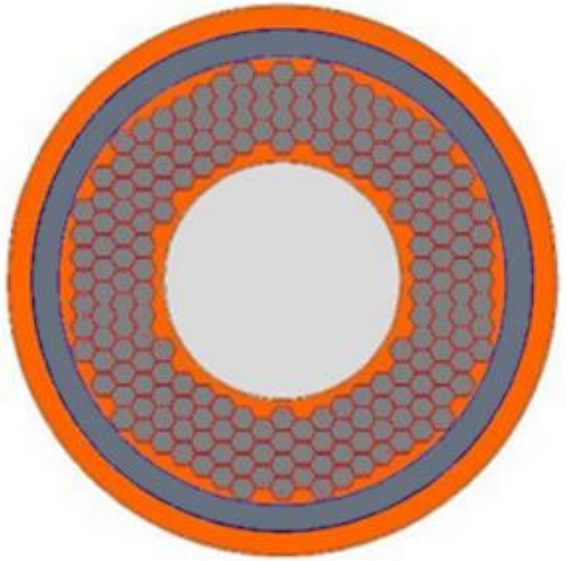


OS at subelement level



Nb filaments
substituted with OS

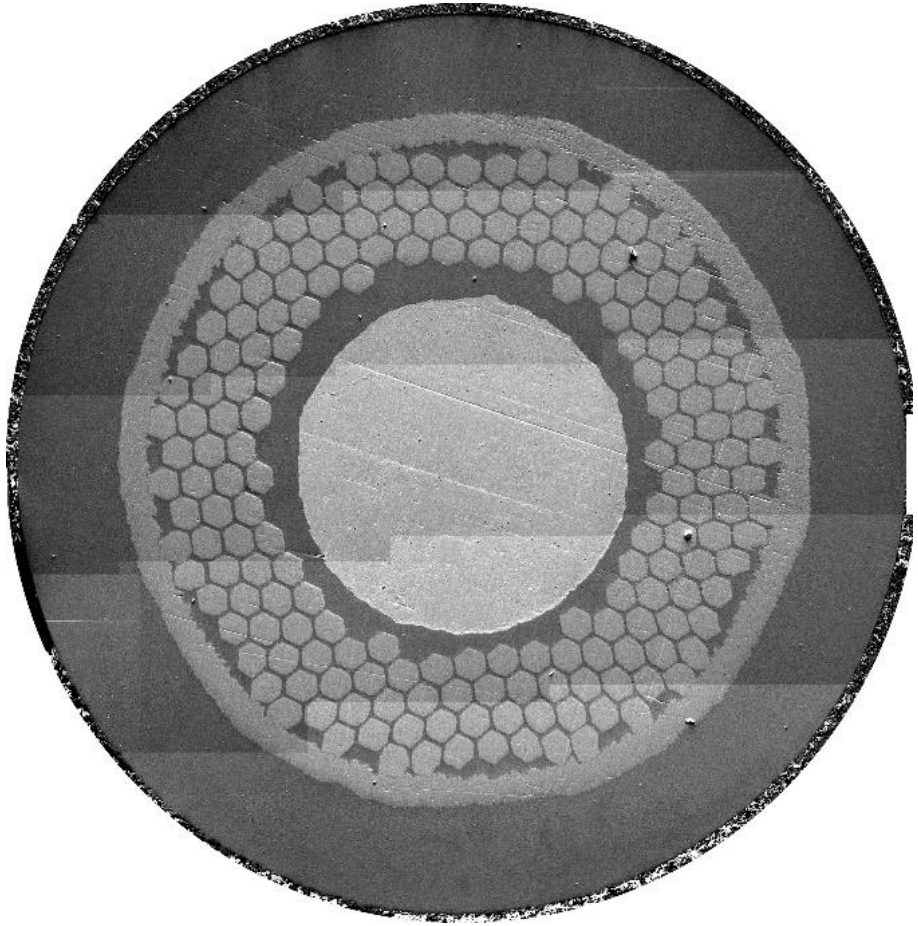
OS at filament level



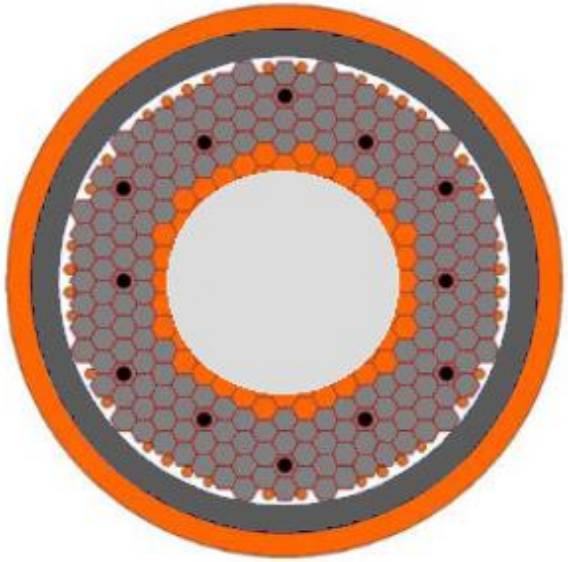
OS inside each filament
of the subelement

Approaches to implement internal oxidation

Where should we place the OS in a RRP wire?

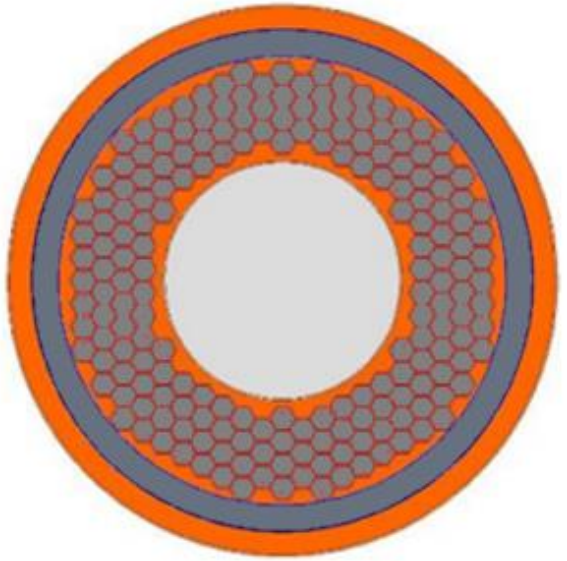


OS at subelement level



Nb filaments
substituted with OS

OS at filament level



OS inside each filament
of the subelement

Billets of the filaments extruded for both approaches; ongoing deformation to reach subelement assembly size

Conclusions

- ***Enhancement of J_c*** *above the* ***FCC specifications***, ***record-high B_{c2}*** *and change of pinning mechanism (****point defect****) when* ***Oxygen Source (OS)*** *is present, for wires* ***reacted at 650 °C***
- ***X-Rays Absorption Spectroscopy (XAS)*** *on Nb_3Sn wires* ***with and without OS*** *show the presence of* ***ZrO_2 only in the Nb_3Sn layer*** *and not in the unreacted alloy*
- ***Suppression*** *of the* ***point defect*** *pinning mechanism in samples* ***reacted at 700 °C*** with consequent drop of ***layer−J_C below the FCC target***
- *The billets* ***containing OS*** *in the layout have undergone* ***extrusion*** *and are currently being* ***prepared*** *for the* ***next stages of deformation***

Thank you for your attention