APPLIED SUPERCONDUCTIVITY CENTER
NATIONAL HIGH MAGNETIC FIELD LABORATORY
FLORIDA STATE UNIVERSITY

Advances in Nanoscale Analysis of Hf doped Nb_3Sn wires using Atom Probe Tomography

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Research at University of Oxford is supported by EPSRC, the main Research Council for Physical Sciences in the UK

Research at FSU is supported by the US Department of Energy, Office of Science, and Office of High Energy Physics under Award Number DE-SC0012083, and was performed at the National High Magnetic Field Laboratory, which is supported by National Science Foundation Cooperative Agreements NSF- DMR-1644779 and by the State of Florida

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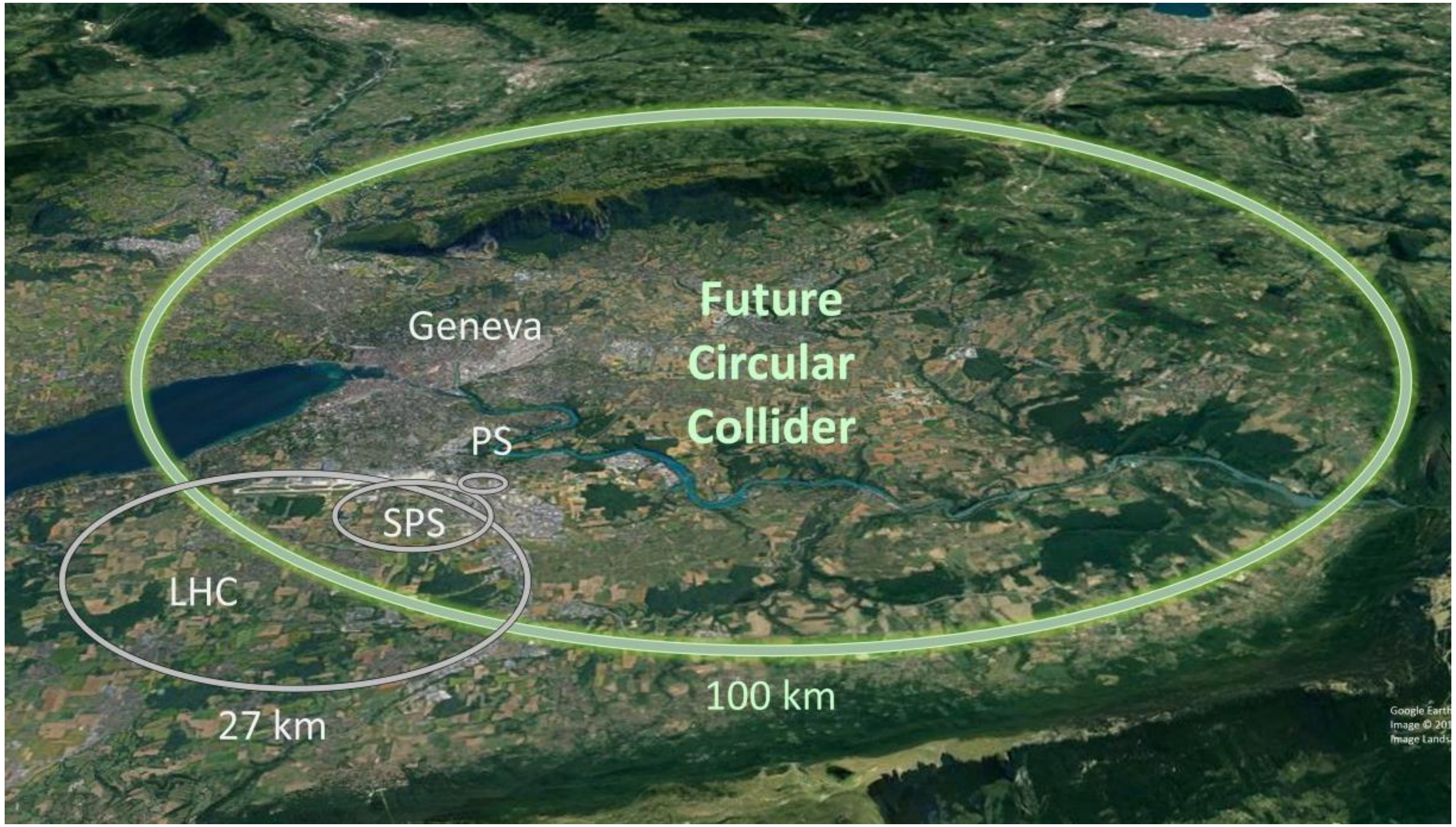
Presentation given at Applied Superconductivity Conference, Honolulu, HI, USA, October 2022.

Requirement: High Performance Nb_3Sn for FCC

- Operational J_c in Nb_3Sn superconductor: 1500 Amm^{-2} and RRR > 150 at 16 T (4.2 K)
- Needs radical improvements in performance of Nb_3Sn filaments



Schematic of the FCC
www.home.cern/science/accelerators/future-circular-collider

Optimising the Superconducting Properties

- For high J_C at 16 T the pinning force function will require both grain boundary and secondary point pinning

$$F_P = A_{GB}\left(\frac{H}{H_{IRR}}\right)^{0.5}\left(1-\frac{H}{H_{IRR}}\right)^2 + A_{PD}\left(\frac{H}{H_{IRR}}\right)\left(1-\frac{H}{H_{IRR}}\right)^2$$

- Grain boundaries and optimised point defects are on the scale of the coherence length (3-4 nm in Nb_3Sn)
- Nanostructural analysis is required to visualise grain boundaries, secondary phases and local chemistry changes on this scale

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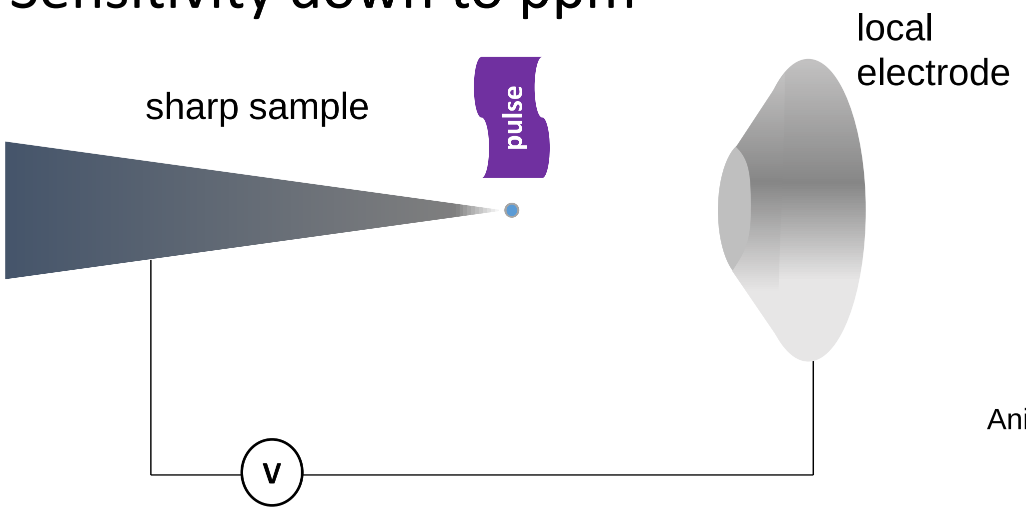
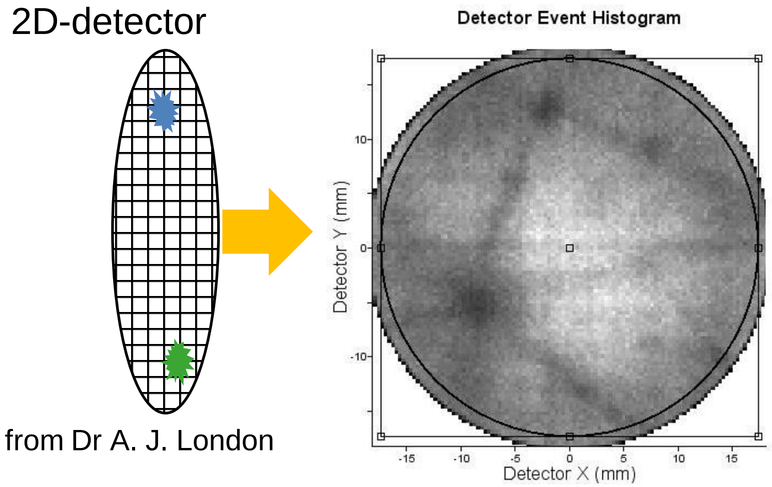
What is Atom Probe Tomography?

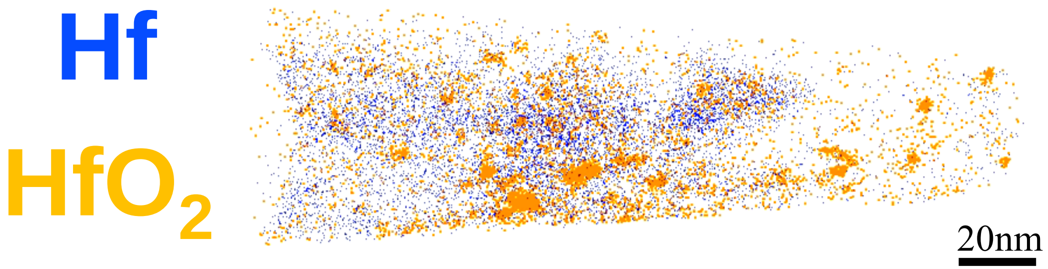
- 3-Dimensional characterisation technique
  - High spatial and chemical resolution
  - Sensitivity down to ppm

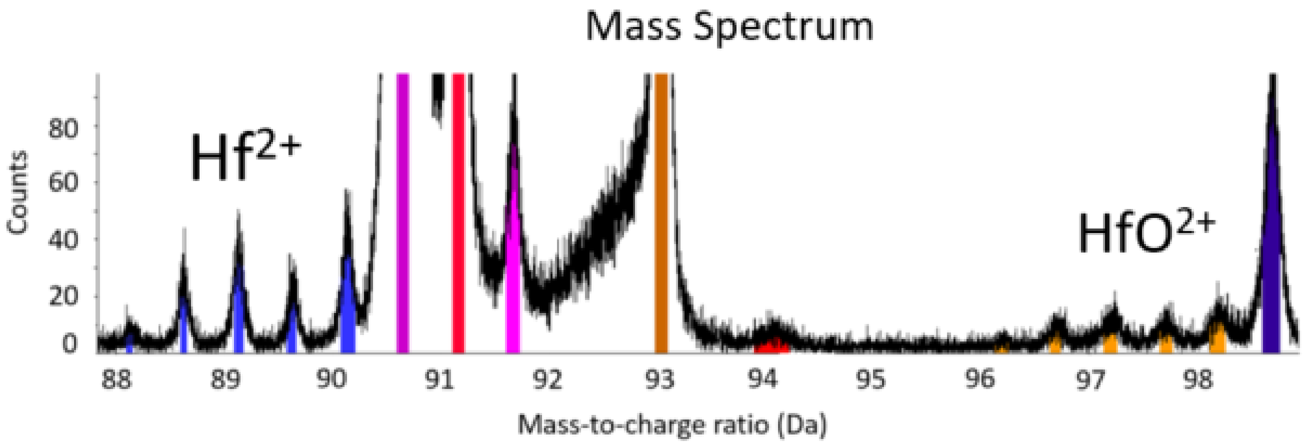
$$\frac{m}{n} \approx 2eV\left(\frac{t_{flight}}{L}\right)^2$$





Animation adapted from Dr A. J. London





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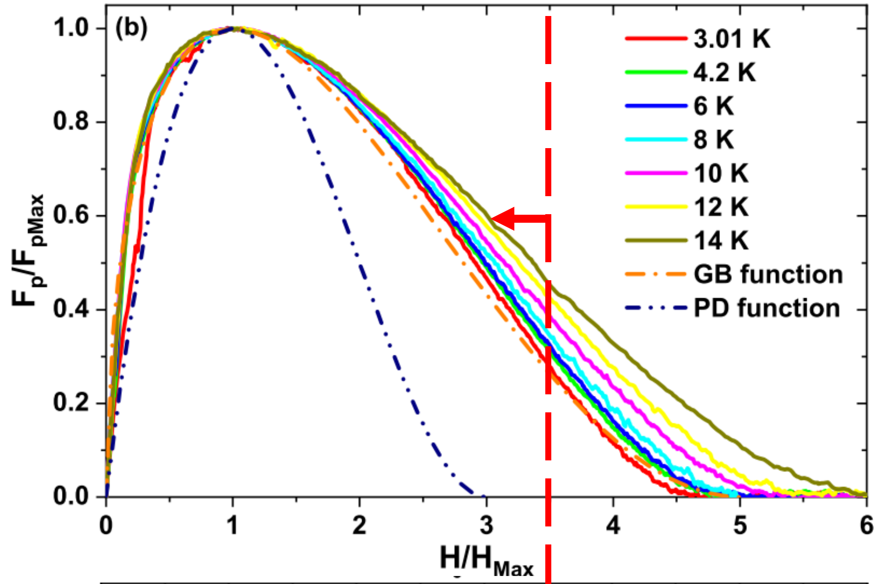
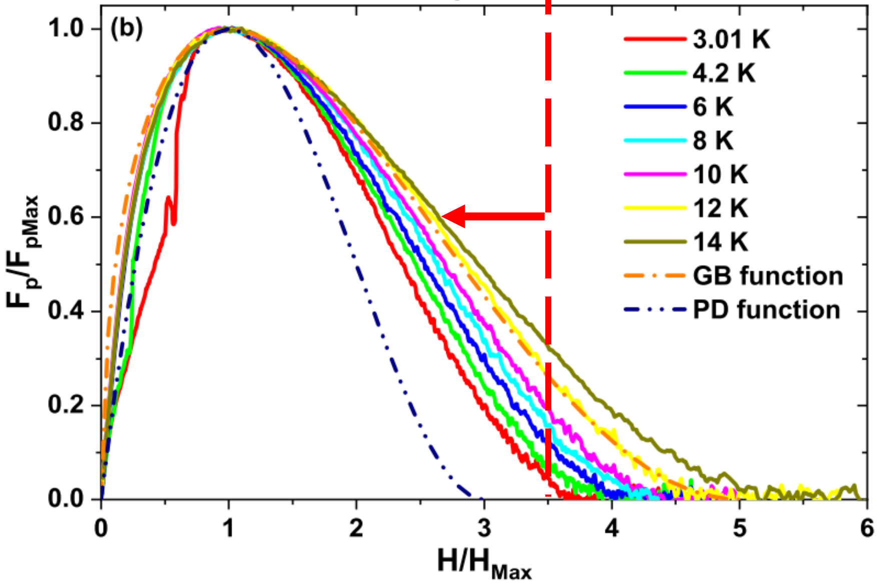
Additions for Point Defects

- Addition of Group IVB elements Zr and Hf to produce oxide nanoparticles [1,2]
- Ta known addition to increase the upper critical field
- Obvious shift to point pinning function seen in Hf doped sample

Nb-Ta4 sample



Nb-Ta4-Hf1 sample



[1] S. Balachandran et al. 2019 Supercond. Sci. Technol. 32 044006
[2] X. Xu et al. 2020 Scripta Materialia 186 317-320.
[3] C.Tarantini et al .2019 Supercond. Sci. Technol. 32 124003

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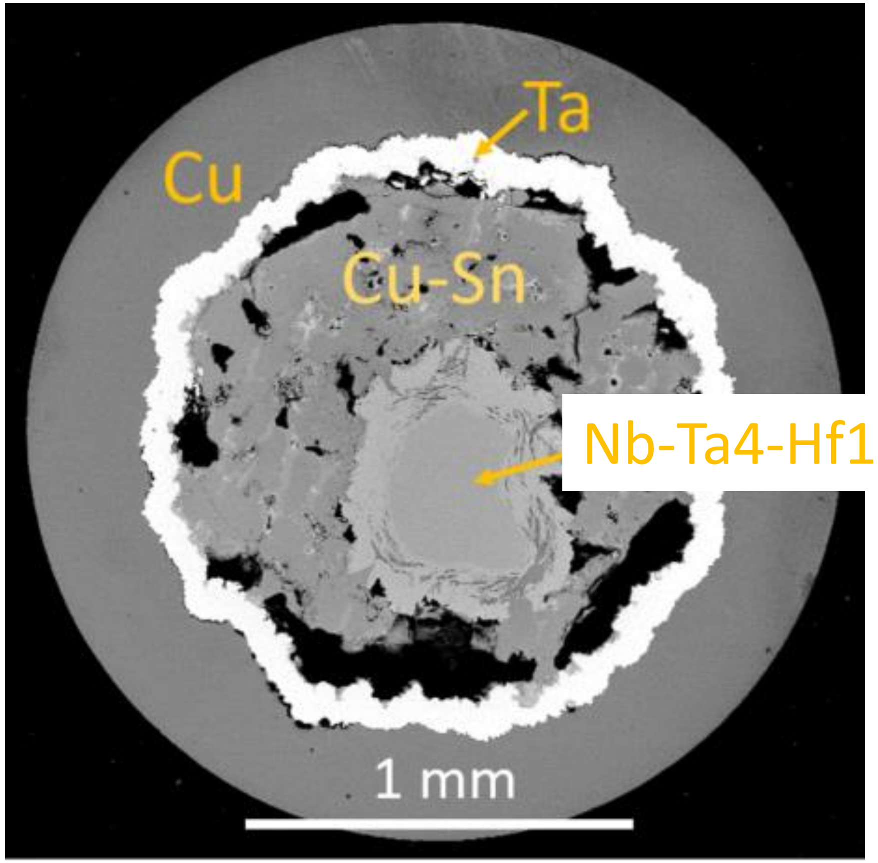
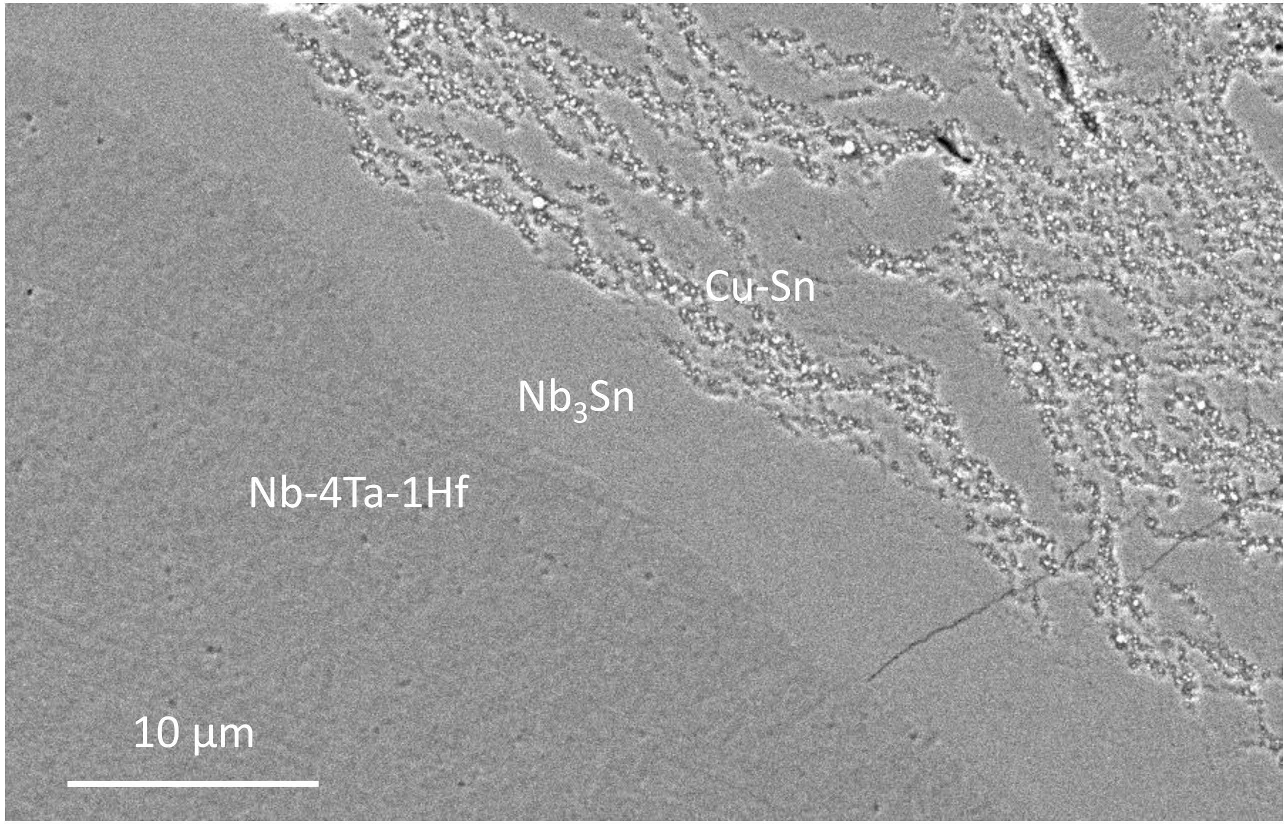
Sample studied in this work



BSD image of reacted wire



Are nanoparticles present in the Nb_3Sn region?

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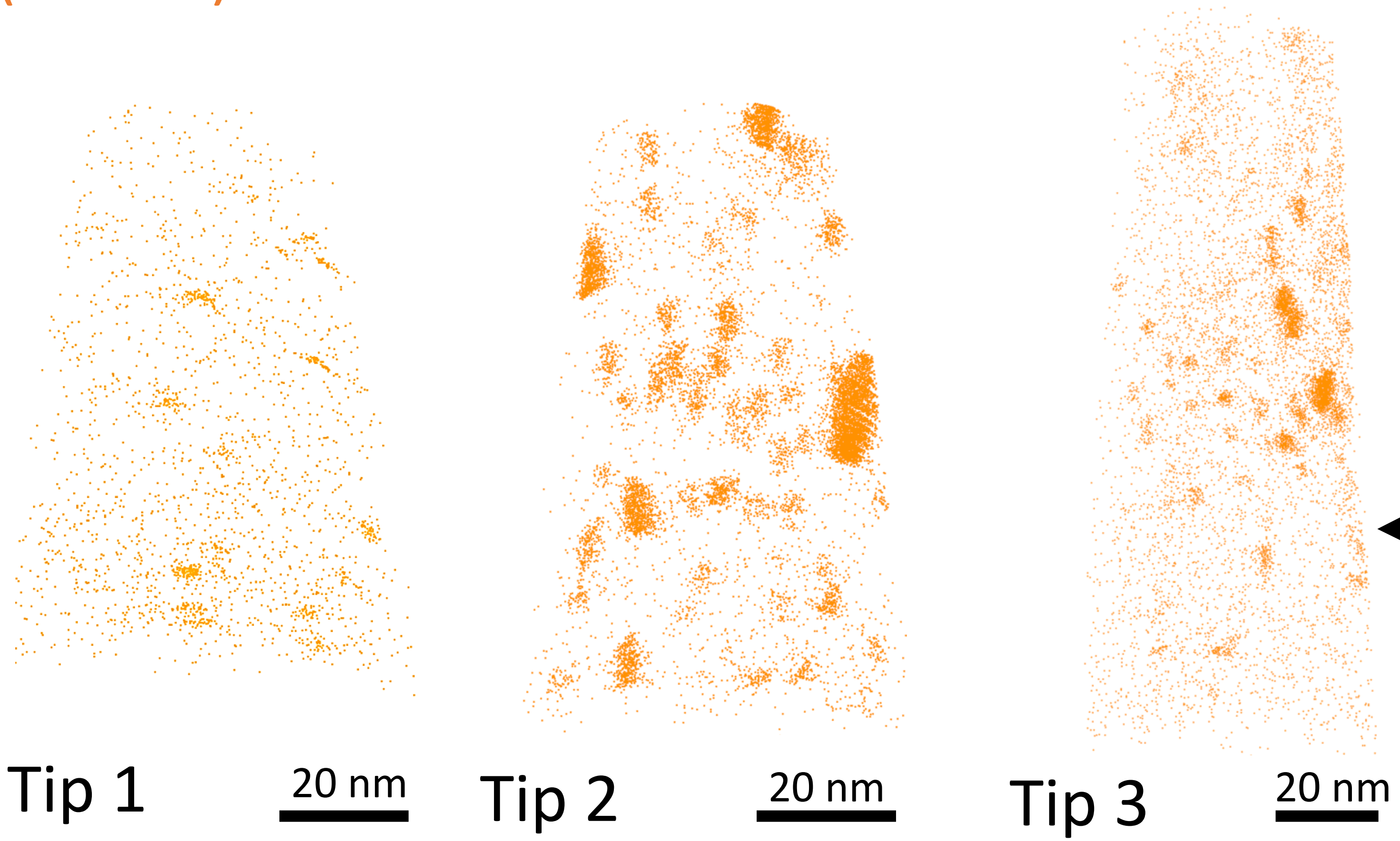
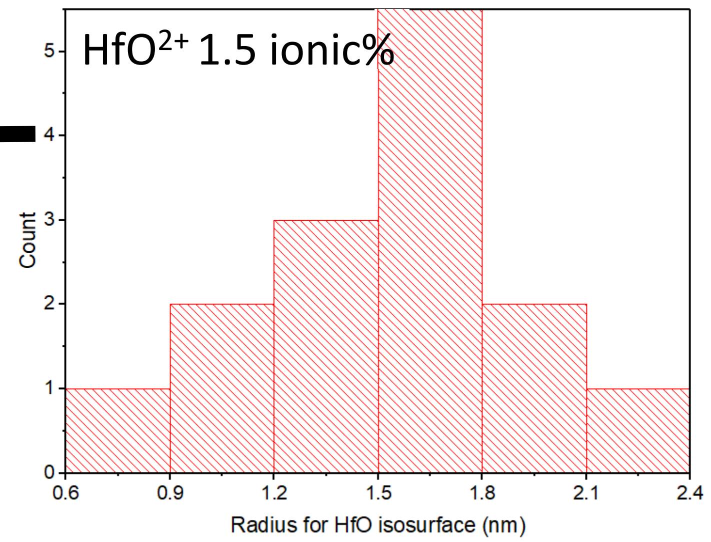
Atom Probe Tips from Nb_3Sn layer

HfO_2 molecular (cluster) ions



Tip	Average HfO_2 Diameter (nm)
1	2.7
2	3.1
3	2.9

Tip 3 Isoconcentration surface HfO^{2+}



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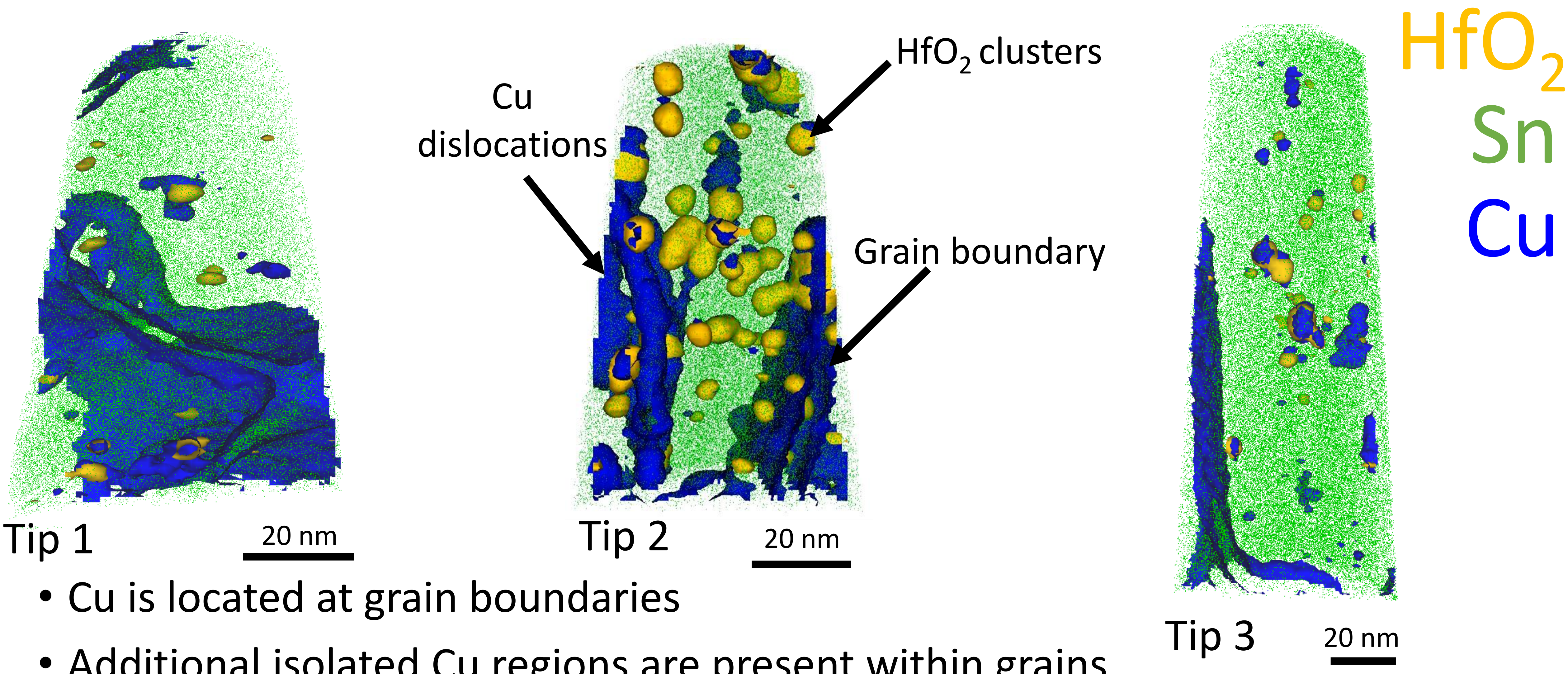
Nb_3Sn region: Location of oxides



- Cu is located at grain boundaries
- Additional isolated Cu regions are present within grains

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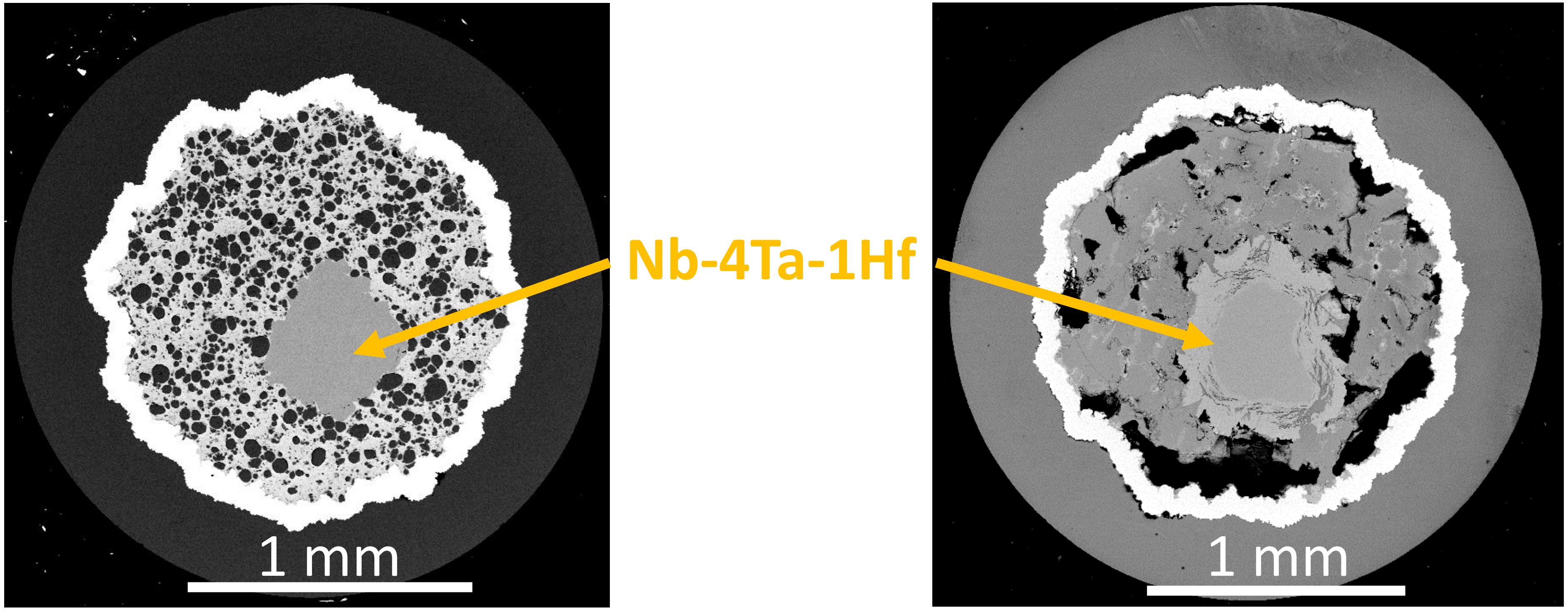
Oxide Source

- **<u>No oxygen</u>** added to the alloy
- Are HfO_2 nanoparticles present within the Nb-Ta-Hf alloy before reaction?



Pre-heat treatment

Post-heat treatment

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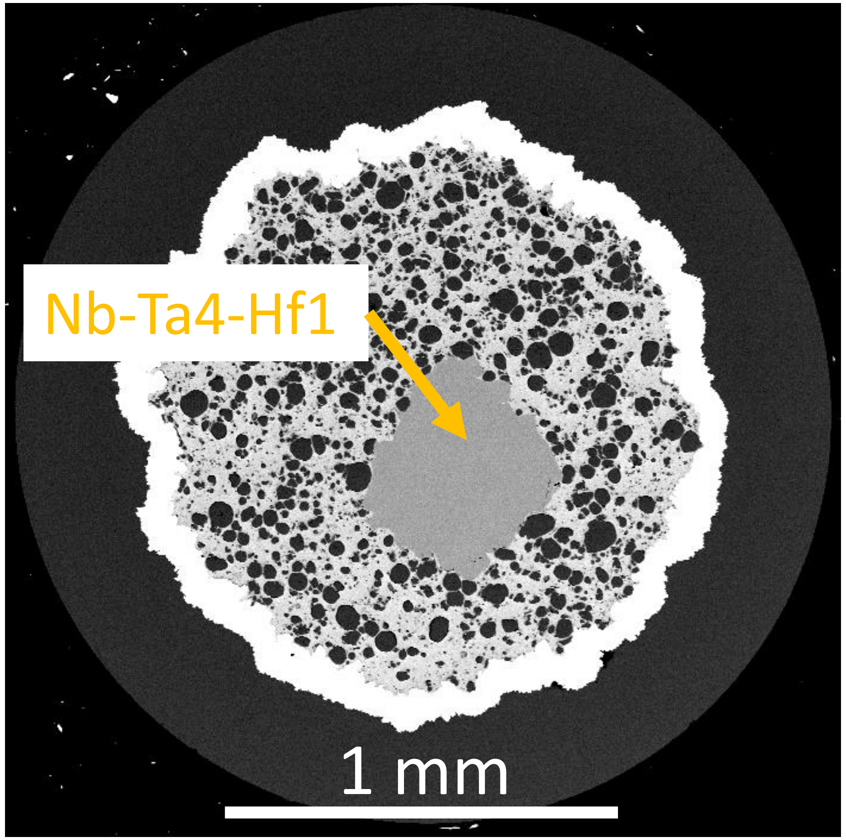
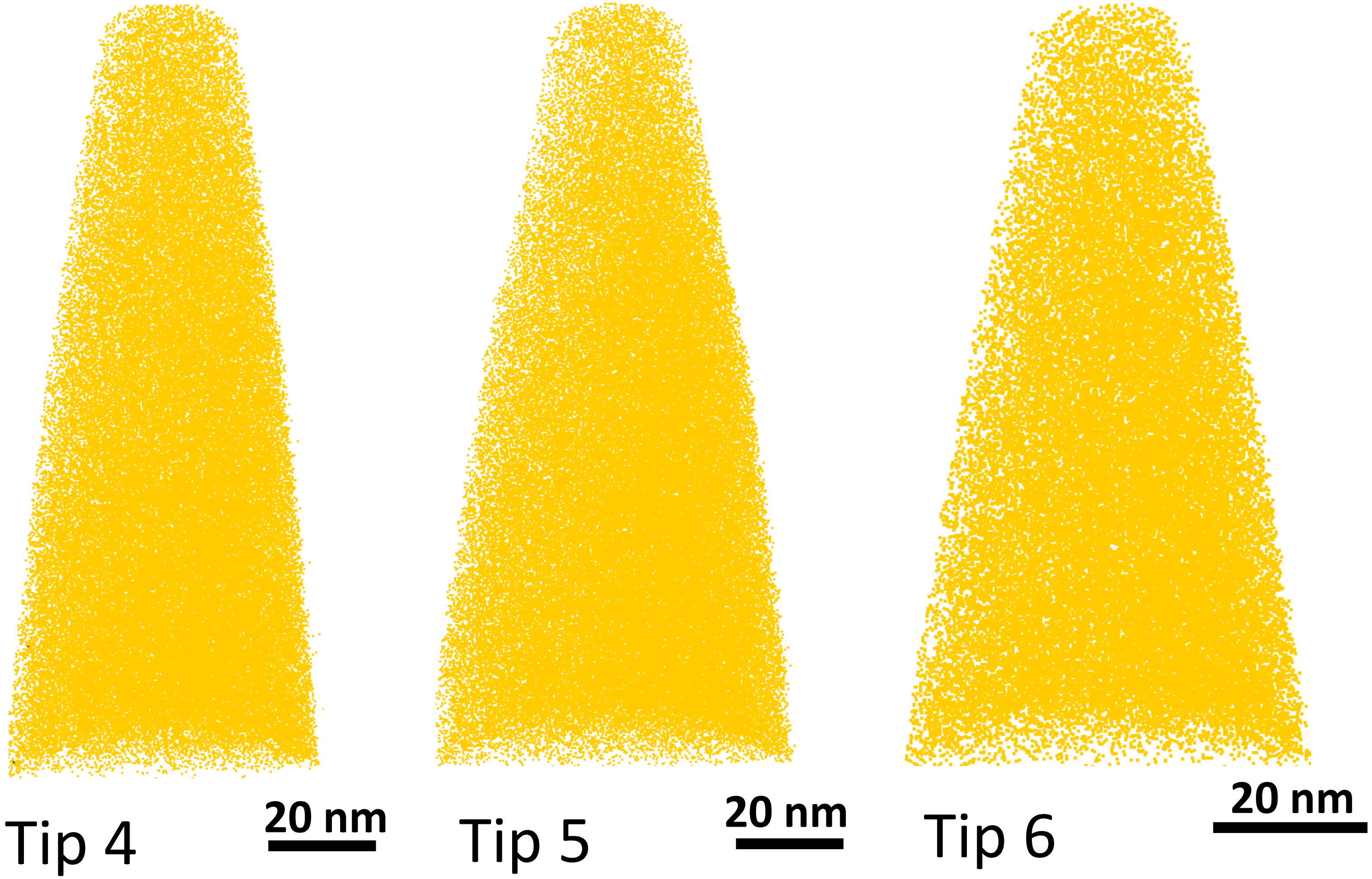
Pre-Heat Treatment Nb-Ta-Hf alloy: Hf distribution



There is no sign of HfO_2 clusters

Hf ions



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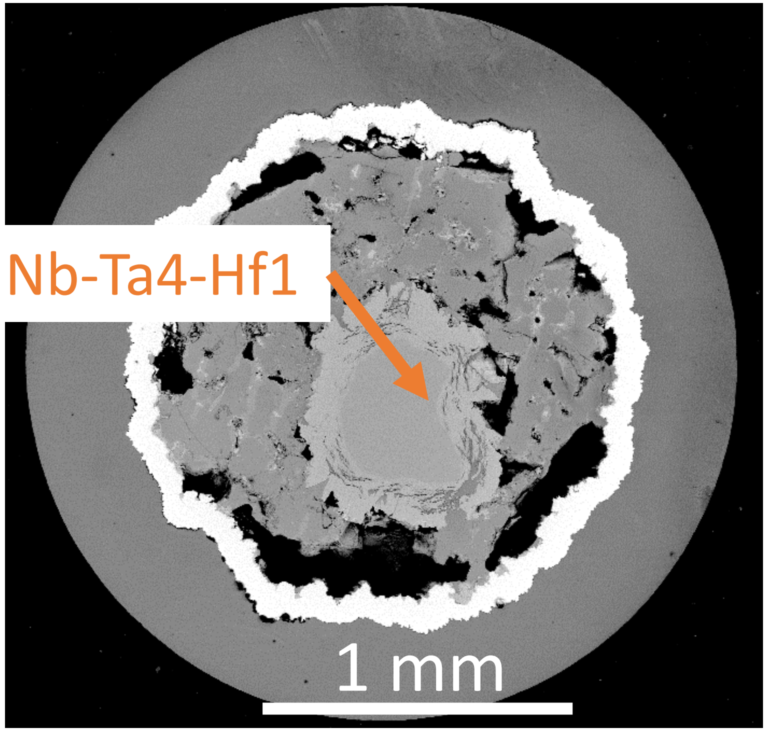
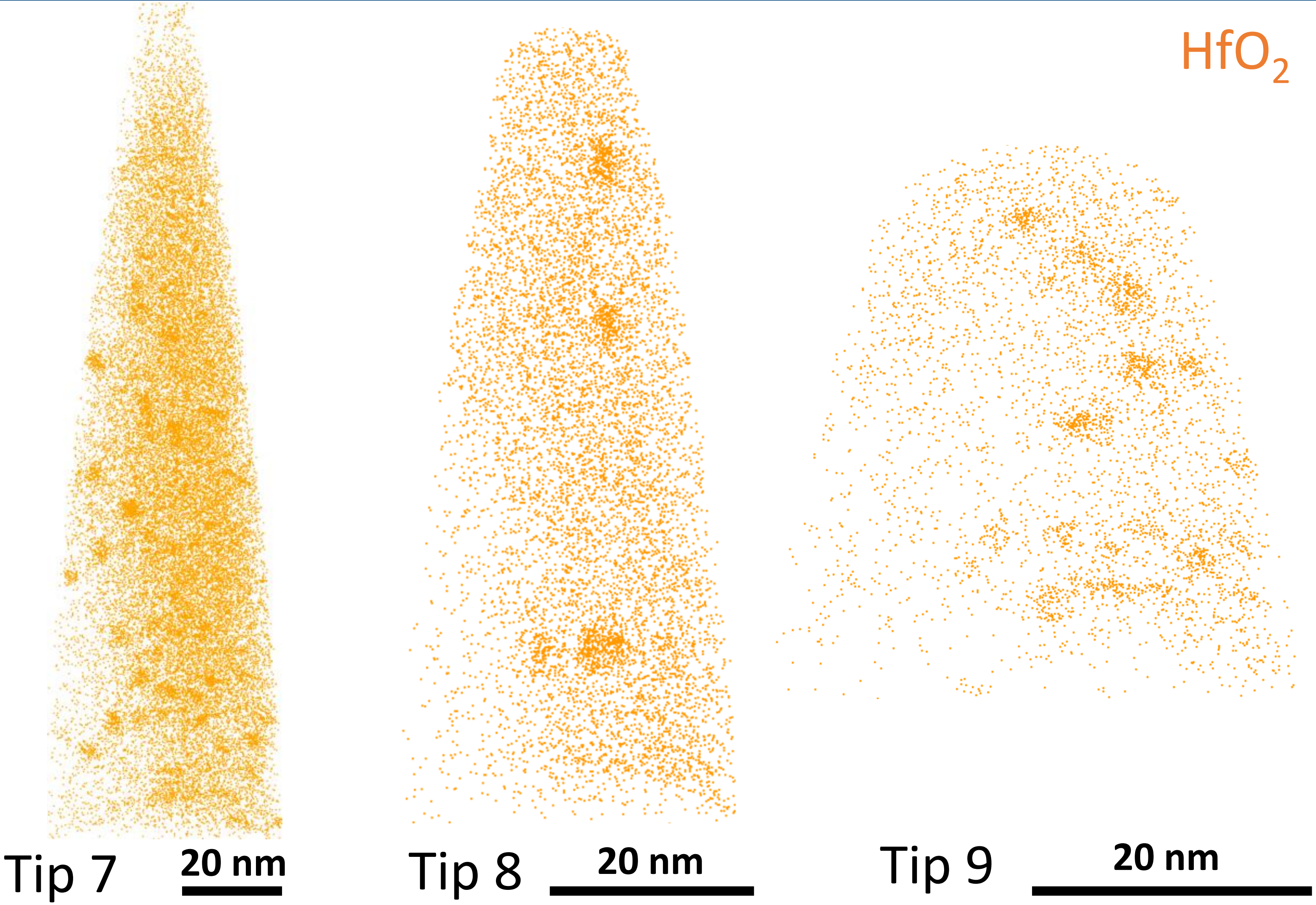
Post Heat Treatment Nb-Ta-Hf alloy

Now the unreacted regions of metallic alloy do contain HfO_2 nanoparticles



Post-heat treatment



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Oxygen Content

Pre-heat treatment Nb alloy (Oxygen at%)	Post-heat treatment Nb alloy (Oxygen at%)	Nb_3Sn (Oxygen at%)
2.59	3.66	0.49
1.95	3.61	0.87
3.83	1.36	0.16

Oxygen content in pre-heat and post-treatment Nb-Ta-Hf alloy is very similar, with far lower oxygen in the Nb_3Sn layer which is confined to purely oxide clusters.

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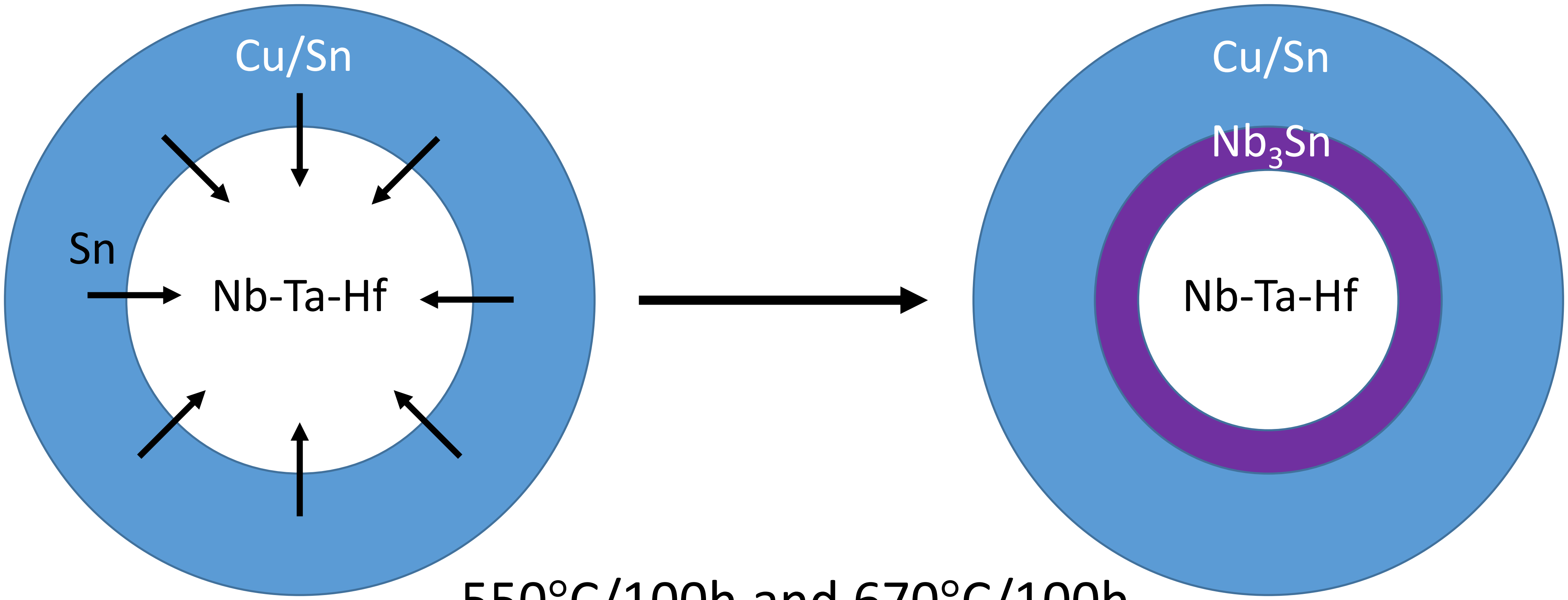
We can also study the reaction process



550°C/100h and 670°C/100h

Sn diffuses in → $NbSn_2$ → Nb_6Sn_5 → Nb_3Sn

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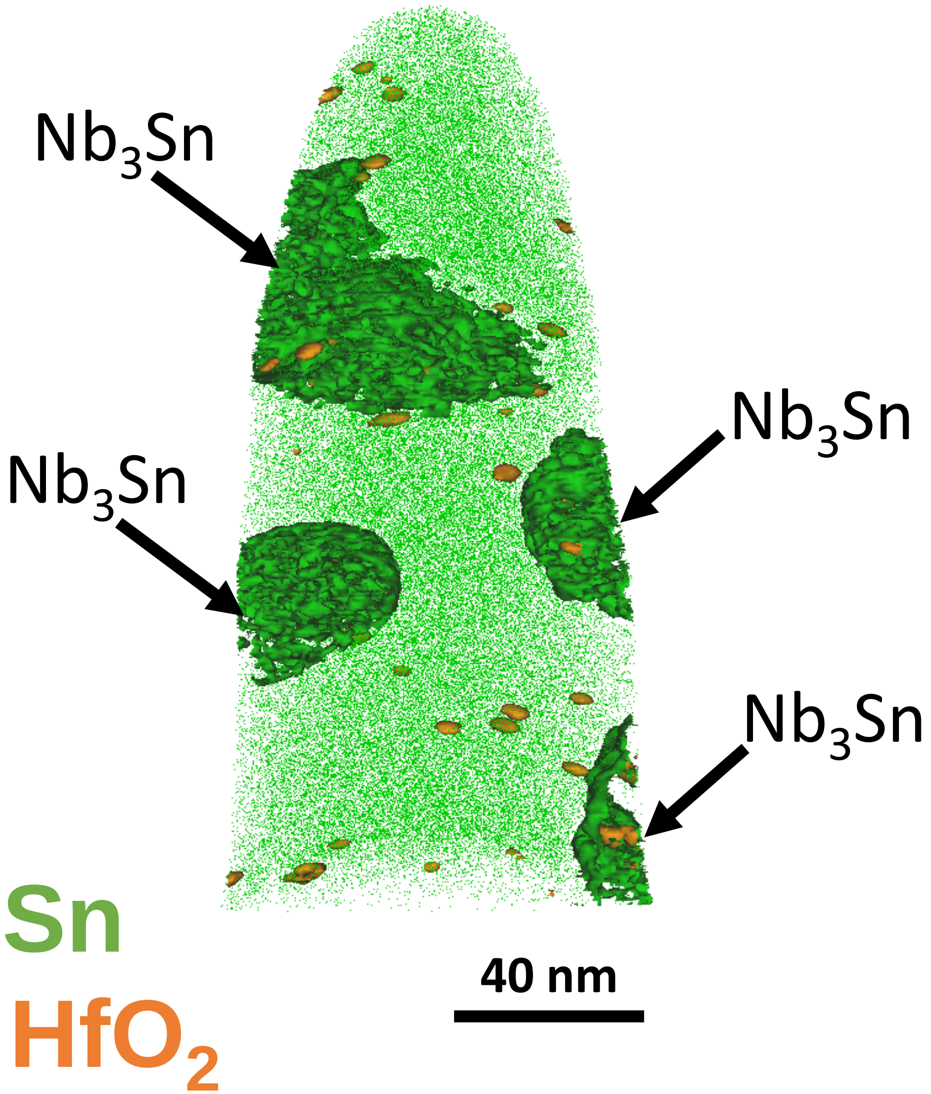
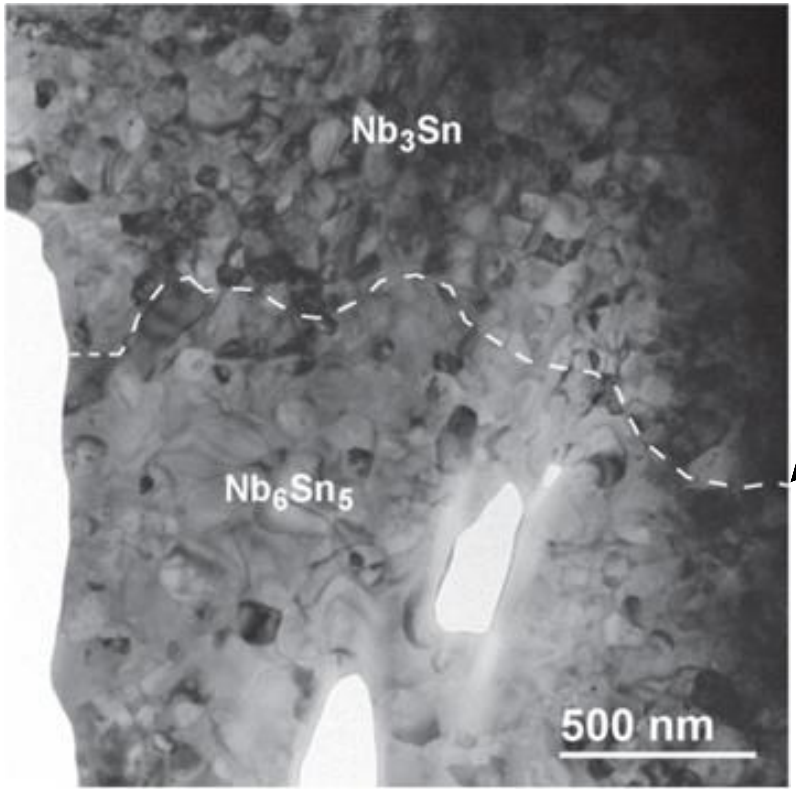
Residual Nb_6Sn_5



- Nb_3Sn shown with the dark green surface
- The rest of the tip is Nb_6Sn_5
- HfO_2 clusters seen in Nb_6Sn_5 as well as the Nb_6Sn_5



TEM image of Nb_3Sn-Nb_6Sn_5

S. Balachandran et al. 2019
Supercond. Sci. Technol. 32 044006

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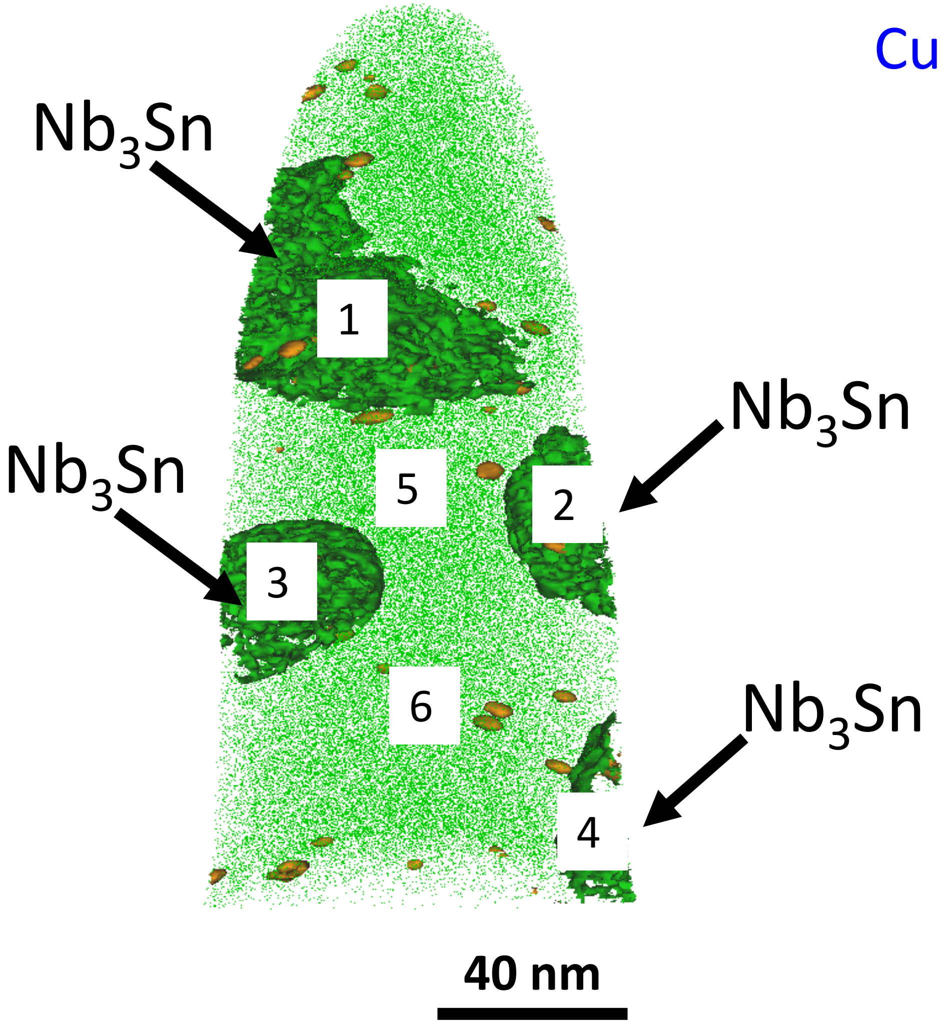
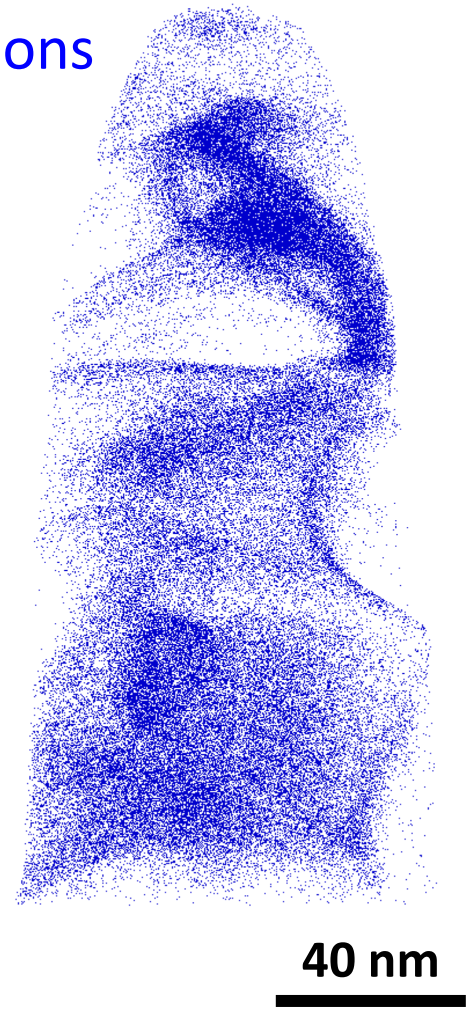
Cu preferentially partitioned into the Nb_6Sn_5





Location	Nb_3Sn Copper at%
1	0.47
2	0.39
3	0.07
4	0.56

Location	Nb_6Sn_5 Copper at%
5	1.38
6	2.41

C. Segal, et al. IOP Conference Series: Materials Science and Engineering. Vol. 279. No. 1. IOP Publishing, 2017.

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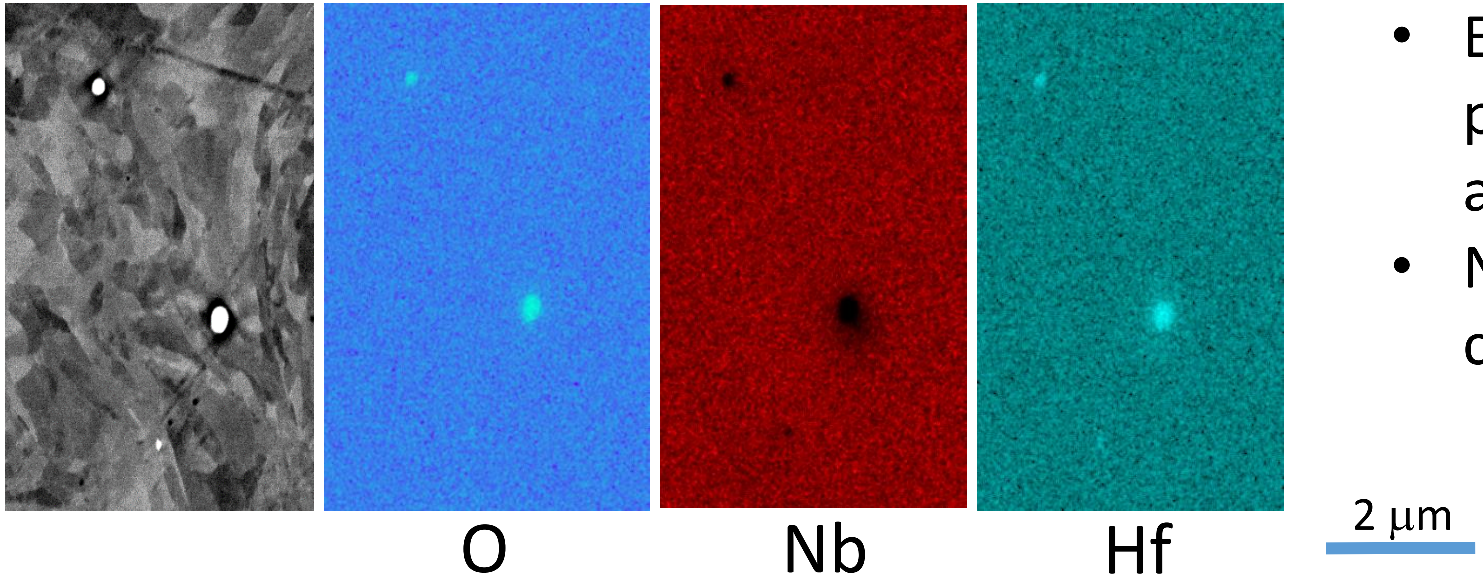
Next Steps: Comparison of Oxygen content to a Commercial Alloy

Pre-heat treated Monofilament Nb-4Ta-1Hf alloy (at%)	Commercial Nb-4Ta-1Hf alloy (at%)
2.59	2.17
1.95	1.09
3.83	1.04

BSD image of a commercial alloy



- Evidence of larger HfO_2 precipitates in the commercial alloy
- Next step is to compare oxygen content across alloys using EPMA

EDX courtesy of Junliang Liu

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Conclusion

- HfO_2 nanoparticles are found in the post-heat treatment Nb-Ta-Hf alloy, the Nb_6Sn_5 and the Nb_3Sn layer
- The oxygen was originally dissolved in the Nb-Ta-Hf alloy, leading to the formation of these oxides during heat treatment
- Nanoscale Cu islands are also present in the Nb_3Sn (and may contribute to pinning)
- Nb_6Sn_5 contains a larger concentration of Cu than the Nb_3Sn that forms from it (approx. 2at%)
- Different Nb-Ta-Hf alloy compositions can be compared to determine the best starting material for producing optimal superconducting properties