Studies of Internal Oxidation in Ternary Nb_3Sn at the University of Geneva

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FCC WEEK 2019
BRUSSELS, BELGIUM
24 - 28 JUNE 2019
Crowne Plaza Brussels Le Palace

Objective

Evaluate the potential of increasing the critical current densities in Nb_3Sn by reducing the grain sizes with ZrO_2 nanoparticles at the same time as increasing the upper critical field by Ta addition.
Study the kinetics of formation and the microstructure of the Ta-doped Nb_3Sn grown in the presence of internally oxidized ZrO_2.
Evaluate different oxygen sources for the internal oxidation of Zr atoms present in the Nb filaments.
Investigate the flux pinning mechanisms in Nb_3Sn that was grain refined with ZrO_2 nanoparticles.

Sample preparation

0.22 mm diameter wires of Nb alloy with a metal oxide core were prepared by cold deformation of a 12 mm diameter rod with nanosized powders compacted in a central axial hole.

In the past (Eucas 2017) we tested MoO_3 was as an oxygen source, along with SnO_2. CuO is also interesting for this purpose because it has high Gibbs free energy of formation (actually higher than SnO_2) and is compatible with Nb_3Sn.

The Nb alloy wire was then electroplated successively with: Cu, Sn, Cu.
The thicknesses were varied to achieve the desired Cu/Sn and Nb/Sn ratios.

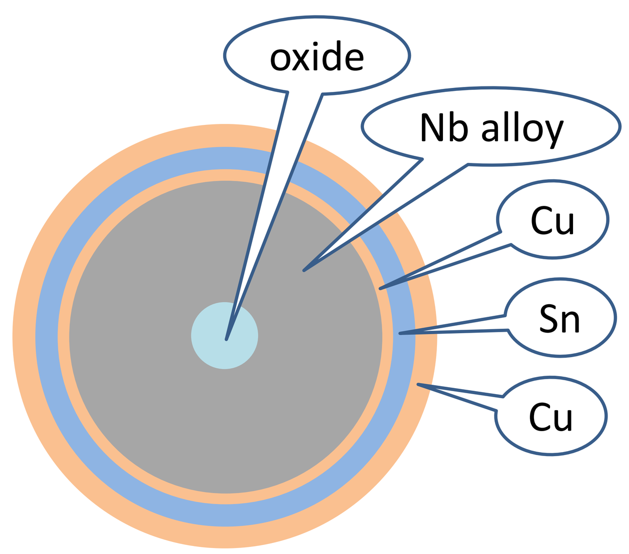
650°Cx200h heat treatments were applied on all samples under vacuum or argon.

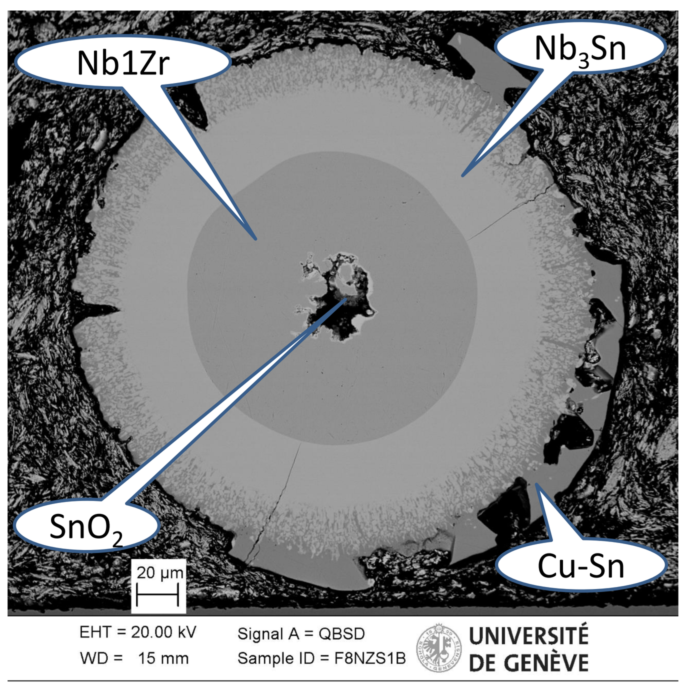
Filament material - oxygen source combinations

Nb alloy	Metal oxide
Nb-1wt%Zr	SnO_2
Nb-7.5wt%Ta-1wt%Zr	SnO_2
Nb-7.5wt%Ta-2wt%Zr	SnO_2
Nb-1wt%Zr	CuO
Nb-7.5wt%Ta	None

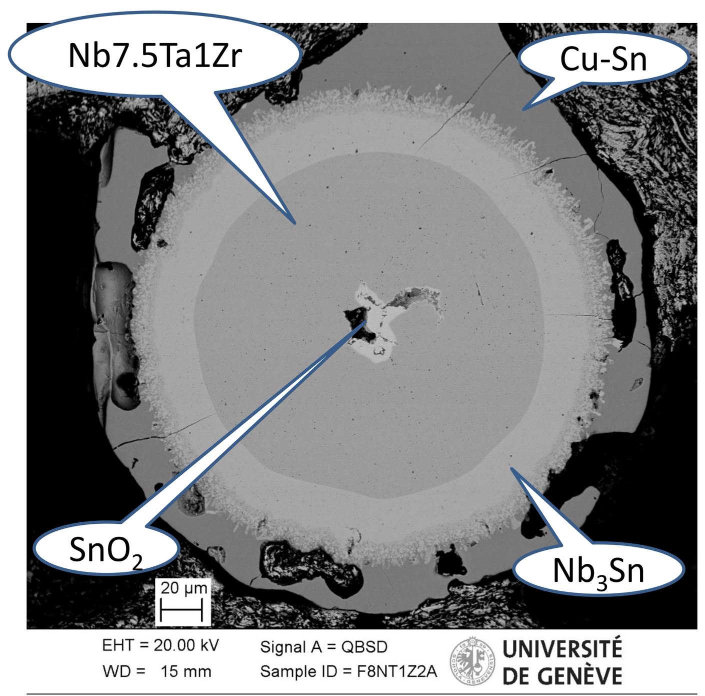
Metal oxide to be reduced by oxidizing Zr in the Nb alloy.
Fine ZrO_2 particles keep Nb_3Sn grains small.

Precursor wire configuration

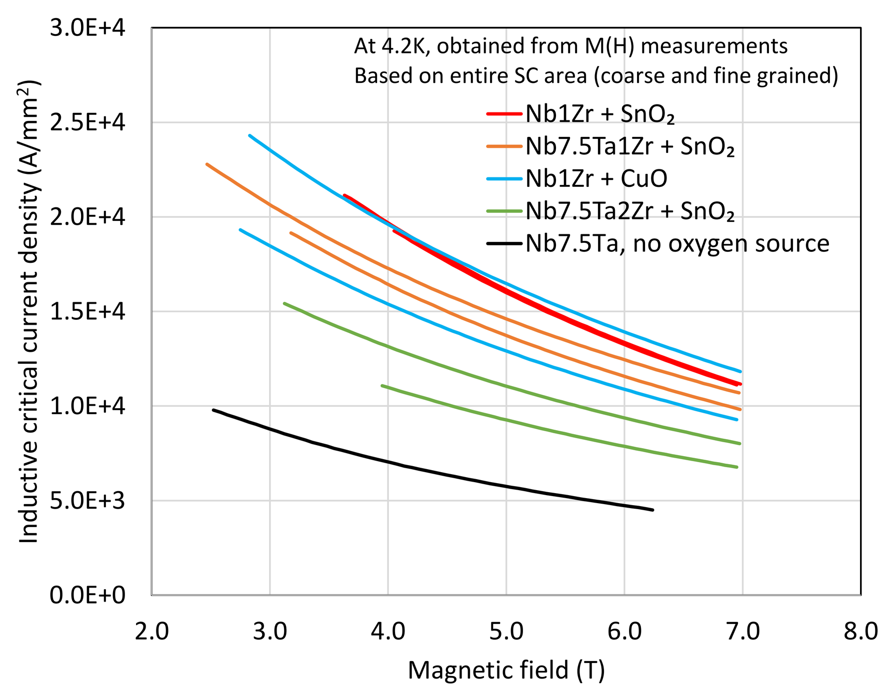


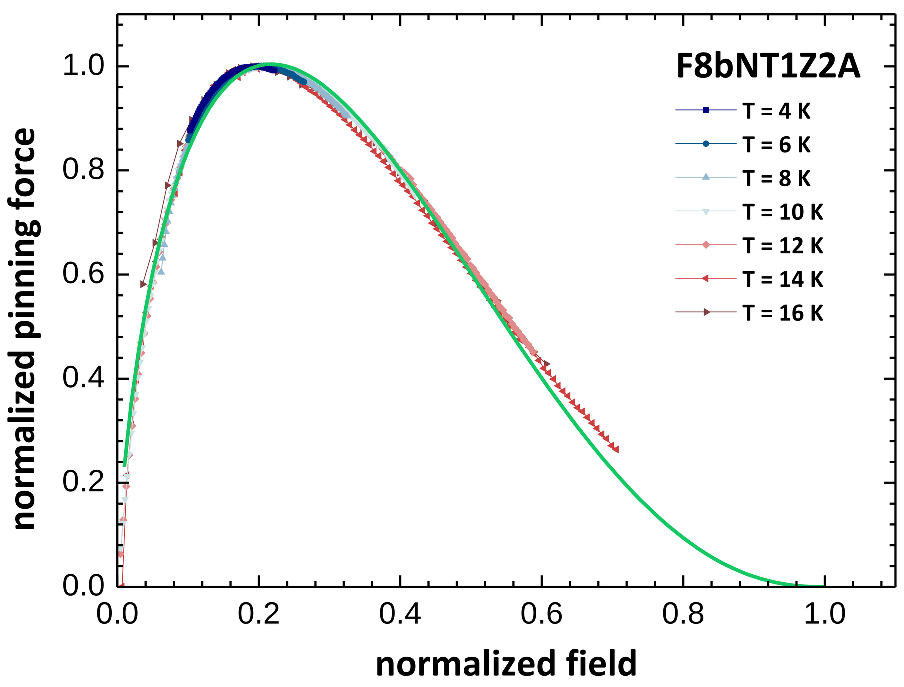


The reaction layer is thinner for the Nb-Ta-Zr filament material than for Nb1Zr. However, our layers are much thicker than in practical wires.



Results and analysis





Critical current densities (J_c) were calculated from M(B) curves, based on hollow cylinders in perpendicular magnetic field. (Eikelboom, J. A., et al. (1989). IEEE Trans. Magn. 25(2): 1968-1971)

For the most recent batch (reacted under argon in the presence of liquid Sn to minimize Sn losses from our samples) the superiority of the samples based on Nb7.5Ta1Zr is challenged at low magnetic fields but not at the values of interest for applications.

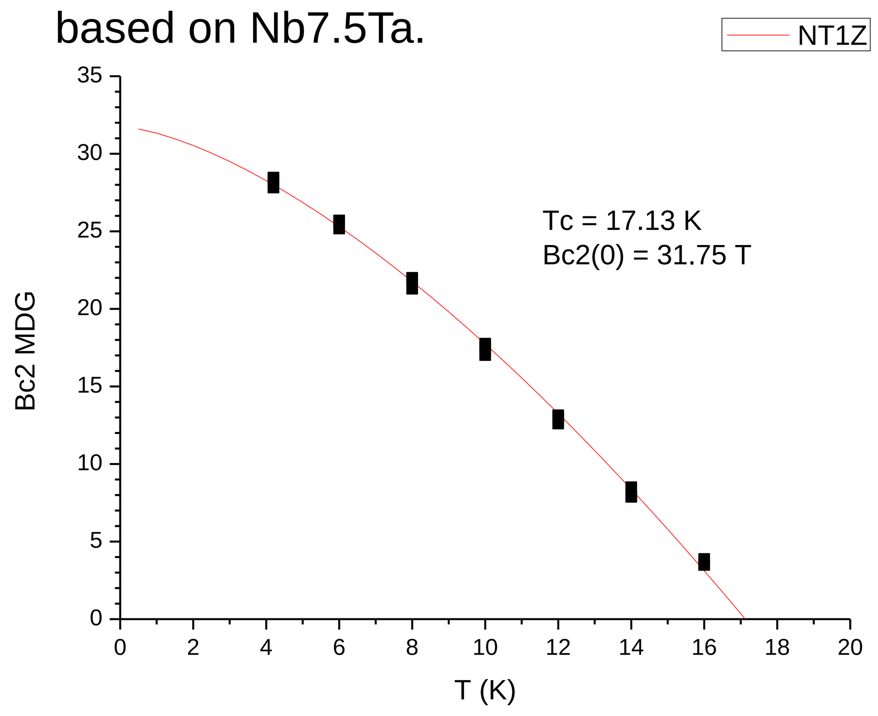
It is not clear why the samples based on Nb7.5Ta2Zr do not show J_c-s that are at least comparable with those based on Nb7.5Ta1Zr, despite showing even finer grains. Unalloyed Ta inclusions and other defects/impurities present in the starting material may be part of the explanation.

The normalized pinning force of the grain refined samples based on Nb-Ta-Zr or Nb1Zr maximizes at a highe reduced field which is very close to 0.2.

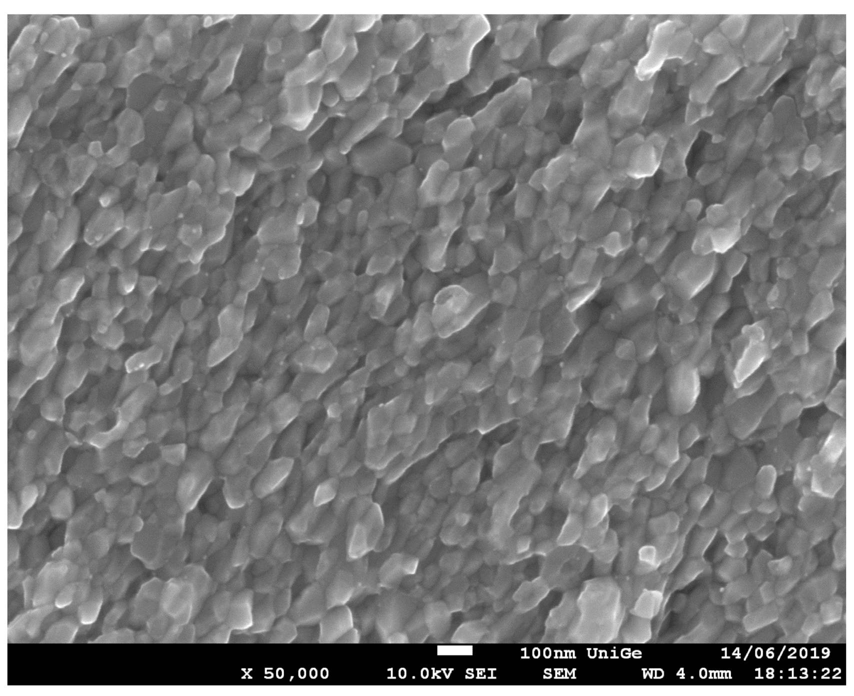
Resistive measurements under magnetic fields up to 35T at LNCMI in Grenoble allowed the determination of the critical magnetic fields as a function of temperature.

Nb alloy	B_{irr} (4.2 K) (T)	B_{c2} (4.2 K) (T)
Nb7.5Ta2Zr	27.2	28.8
Nb7.5Ta1Zr	27.1	28.2
Nb7.5Ta	26.3	27.5
Nb1Zr	25.2	26.5

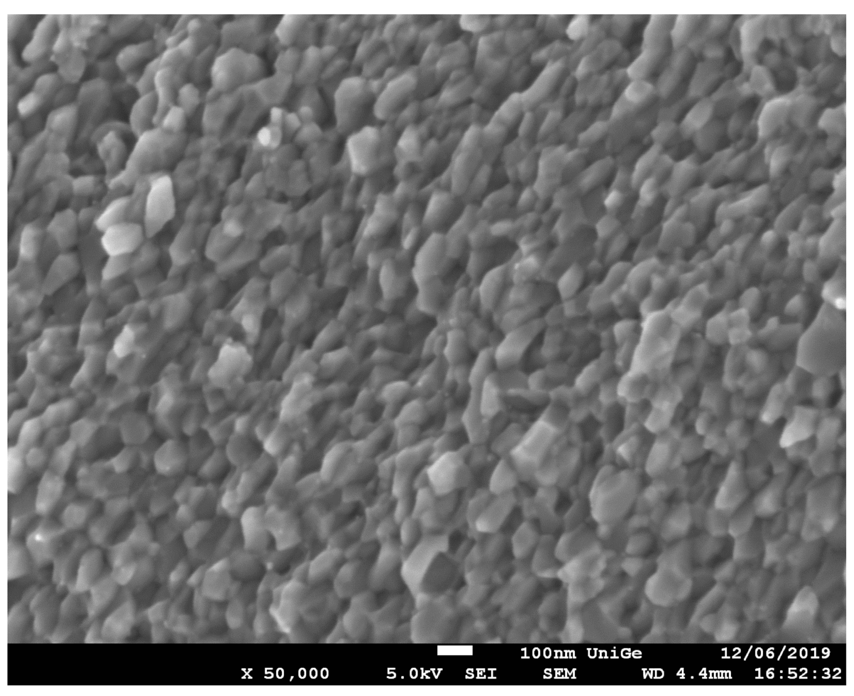
The irreversibility magnetic field is ~2 T higher for the samples based on the Nb-Ta-Zr alloys than for those based on Nb-Zr. Moreover the samples based on Nb7.5TaZr surpass by almost 1 T similar samples based on Nb7.5Ta.



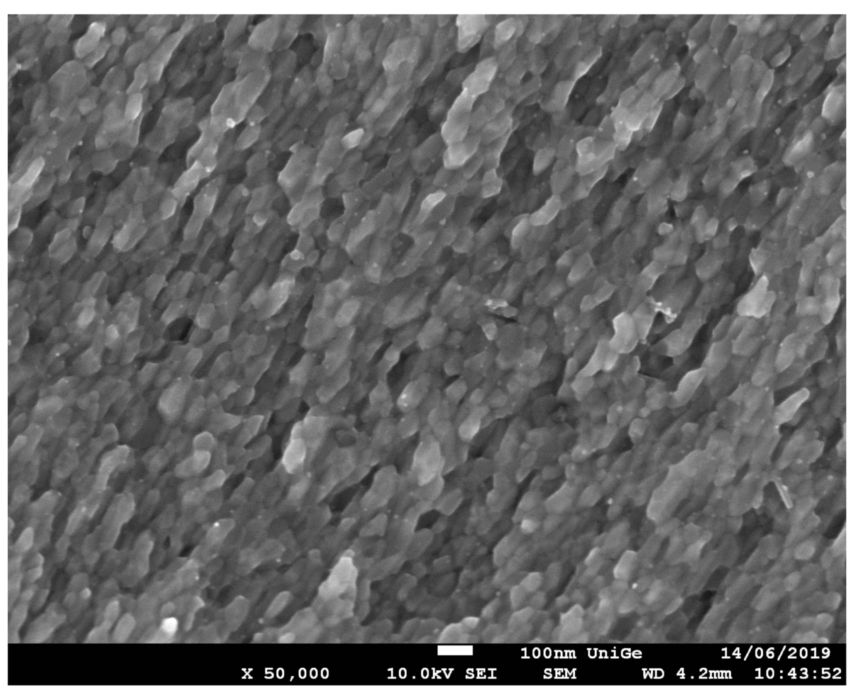
A Maki - de Gennes dependence is obeyed. The Tc-s of samples based on different Nb alloys show little variability.



FESEM image of Nb_3Sn observed on a fractured surface of a sample with Nb1Zr filament and SnO_2 core.



FESEM image of Nb_3Sn observed on a fractured surface of a sample with Nb7.5Ta1Zr filament and SnO_2 core.



FESEM image of Nb_3Sn observed on a fractured surface of a sample with Nb7.5T2Zr filament and SnO_2 core.

The samples based on Nb7.5Ta2Zr have significantly smaller grain sizes (under 100 nm) than those based on Nb7.5Ta1Zr and Nb1Zr, who are comparable.

They all have finer grains than similar samples without internally oxidized Zr.

Conclusion

- A combination of high upper critical field and enhanced J_c can be obtained by grain refining in Ta-doped Nb_3Sn with internally oxidized Zr.
- With value above 27T, the irreversibility magnetic fields of the samples based on Nb-7.5Ta-Zr exceed the values of similar samples based on Nb7.5Ta.
- The Nb_3Sn grain sizes were further reduced in the ternary alloy containing 2 wt% Zr as compared with 1 wt% Zr. Not yet confirmed as an increase in J_c.
- CuO can also be used as an oxygen source, with poor reproducibility until now. Further investigations needed.

Acknowledgments

We thank Dr.-Ing. Max Schlötter GmbH & Co. KG of Geislingen/Steige, Germany for supplying the electrolytic solutions used for electroplating.
The Nb-Ta-Zr alloys were acquired from the Materials Preparation Center at Ames Laboratory, which is supported by the US DOE Basic Energy Sciences.
We acknowledge the support of the LNCMI-CNRS, member of the European Magnetic Field Laboratory (EMFL).
This work was funded by the FCC Study of the European Organization for Nuclear Research (CERN).

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