Correlation of the microstructure and magnetic properties of neutron irradiated Nb₃Sn superconductors



S. Pfeiffer¹, J. Bernardi, M. Stöger-Pollach¹, T. Baumgartner², M. Eisterer², J. Hecher², A. Ballarino³, L. Bottura³, C. Scheuerlein³

¹ University Service Centre for Transmission Electron Microscopy, TU Wien, Vienna, Austria

- ² Atominstitut, TU Wien, Vienna, Austria

³ CERN, Geneva, Switzerland

Contact: stephan.pfeiffer@tuwien.ac.at



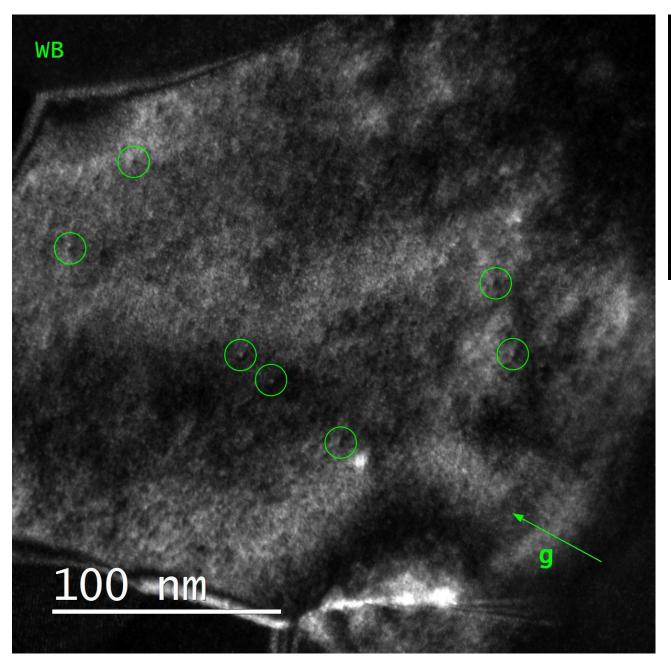
Introduction

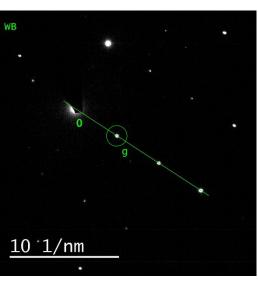
An increase of the high field critical currents in commercial Nb₃Sn wires by about 50 % is required for the design of FCC-hh superconducting magnets. This target has already been reached by creating additional flux pinning centers through fast neutron irradiation that induces defects in the crystal structure [2].

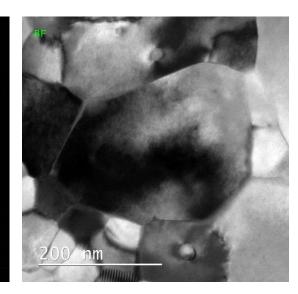
In this study, the underlying mechanisms are investigated through combined microstructural and magnetic analyses. A correlation is made to develop a better understanding of the influence of the microstructure on local superconducting properties and ultimately on the macroscopic performance of the superconductor.

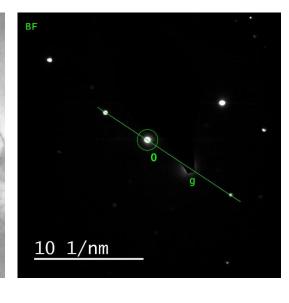
Weak-beam dark-field microscopy

FIB (focused ion beam) was used to prepare transmission electron microscopy (TEM) specimens of subelements before and after irradiation in the nuclear research reactor of TU Wien. Using weak-beam dark-field microscopy, neutron impact sites can be made visible. For this, the electron beam is tilted to shift the excited g-reflection (two-beam case) into the optical axis which loses intensity and is used to form the image. Defects in the crystal structure that fulfil the Bragg condition will then show high contrast.





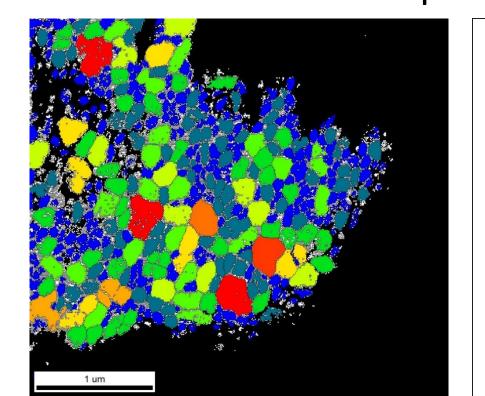


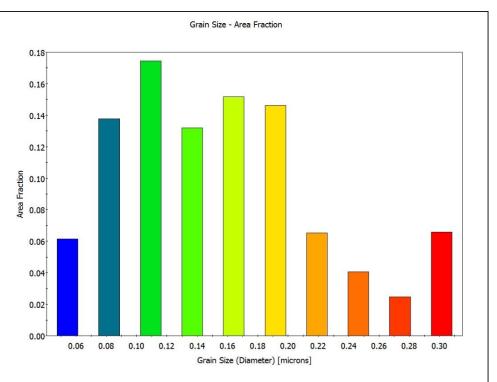


Weak-beam dark-field image obtained using TEM. The diffraction patterns show the used beam geometry. Several locations of a few nm in size with high contrast changes parallel to the g-vector could be identified, as predicted by simulations.

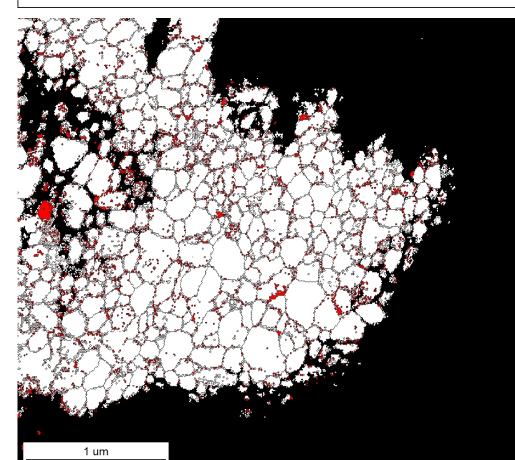
Transmission Kikuchi diffraction

Transmission Kikuchi diffraction (TKD) conducted on a TEM specimen using the scanning electron microscope (SEM) has the advantage of a higher spatial resolution compared to conventional electron back-scatter diffraction (EBSD) because of a smaller specimen tilt angle.



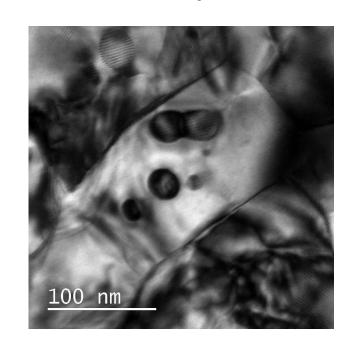


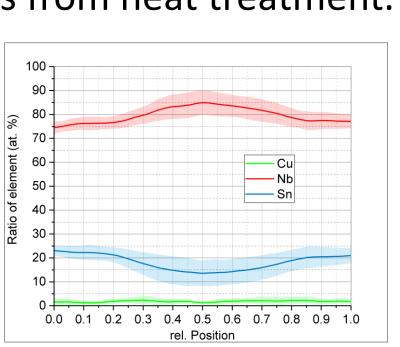
information yields about size grain distribution, orientation and phase distribution. Grain size distribution map with corresponding statistic (top). Average grain size of examined RRP-Ti sample is 104 nm. Phase map combined with grain boundary map shows that Cu (red) is mainly located at grain boundaries (black) as a result of the heat treatment (right).

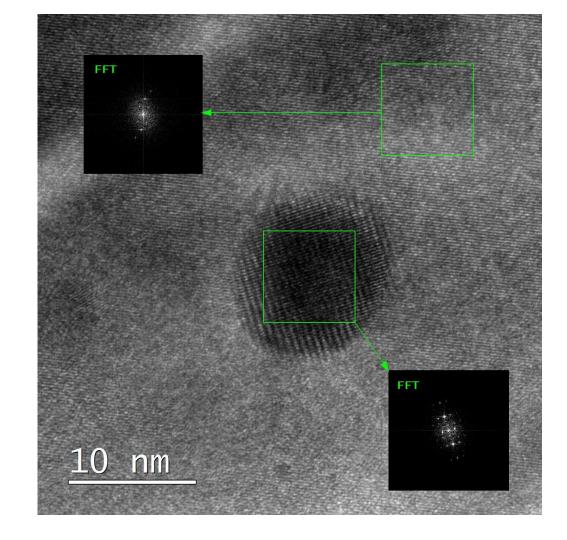


Local texture

Numerous structures 10 nm in diameter were found in specimens of RRP-Ti wire. FFT shows different crystal orientation in these areas. EDX and EELS analyses reveal higher Nb/Sn ratio. These inclusions are most likely Nb leftovers from heat treatment.







References

[1] T. Baumgartner, M. Eisterer, H. W. Weber, R. Flükiger, C. Scheuerlein, and L. Bottura, 'Effects of neutron irradiation on pinning force scaling in state-of-the-art Nb₃Sn wires', Supercond. Sci. Technol. 27 (1): 015005, 2014.

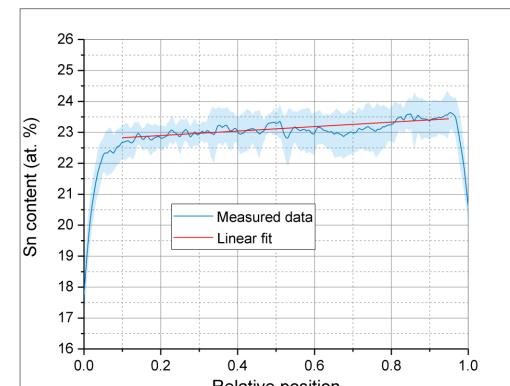
[2] T. Baumgartner, M. Eisterer, H. W. Weber, R. Flükiger, C. Scheuerlein, and L. Bottura, 'Performance boost in industrial multifilamentary Nb₃Sn wires due to radiation induced pinning centers', Sci. Rep. 5: 10236, 2015.

[3] T. Baumgartner, J. Hecher, J. Bernardi, S. Pfeiffer, C. Senatore, and M. Eisterer, 'Assessing composition gradients in multifilamentary superconductors by means of magnetometry methods', Supercond. Sci. Technol. 30 (1): 014011, 2017.

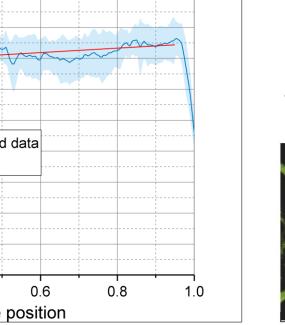
[4] A. Godeke, 'A review of the properties of Nb3Sn and their variation with A15 composition, morphology and strain state', Supercond. Sci. Technol., 19 (8): R68, 2006.

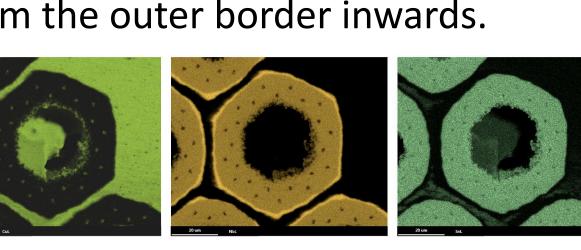
Elemental composition analysis

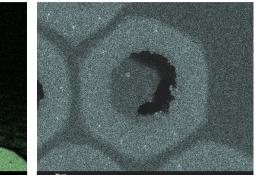
The elemental content of Sn inside Nb₃Sn subelements highly impacts the superconducting performance. EDX linescans performed using SEM and TEM reveal not only a Sn gradient inside subelements but also inside single grains.



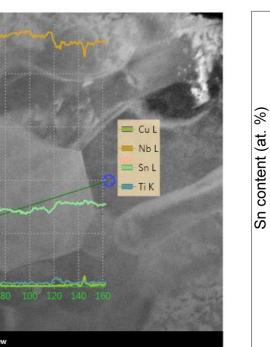
Statistic of EDX linescans over subelements of RRP-Ti wire. A fit through the linear region of the Sn gradient yield a content change of 0.07 at%/µm from the outer border inwards.

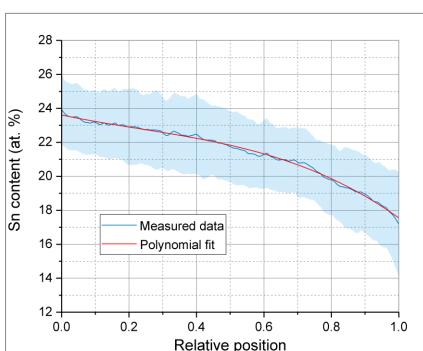


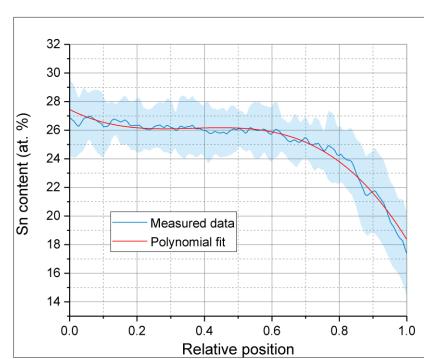




EDX mapping of RRP-Ti wire reveals Cu accumulation at locations of Ti rods before heat treatment, where residual Ti can also be found.



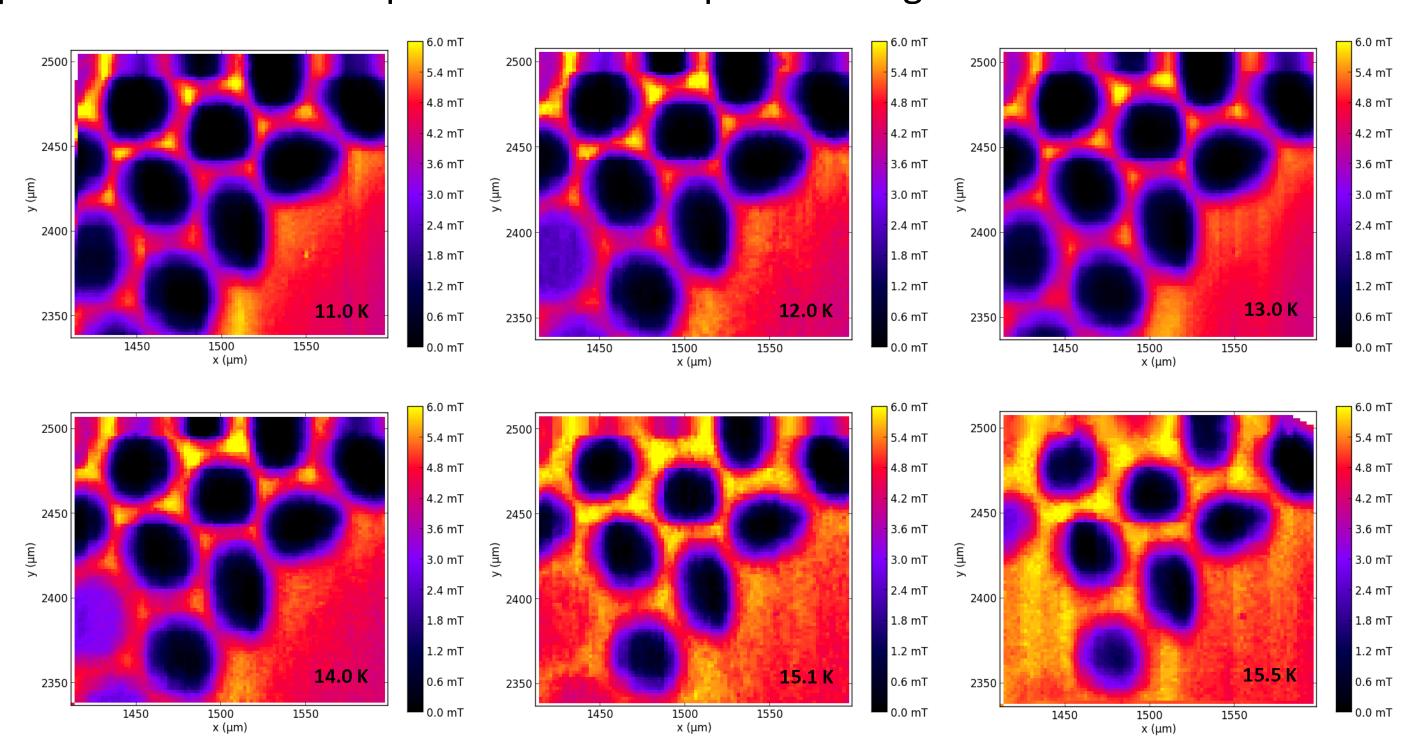




Statistic of EDX linescans over grains of PIT-Ta (center) and RRP-Ti (right) wires from the grain boundary to the center. The highest Sn content can be found at grain boundaries while at the grain center it drops to 18 %.

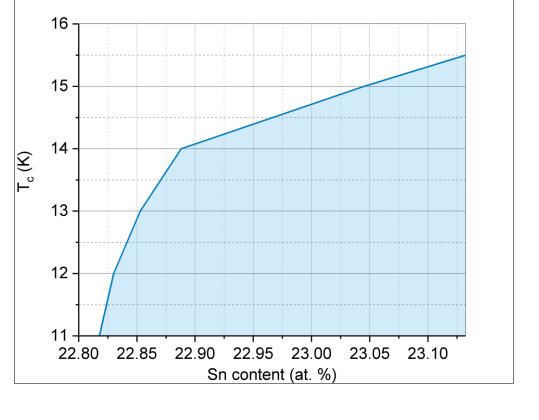
Scanning Hall probe microscopy

Using a self-built scanning Hall probe microscope, specimens with a thickness of less than 10 µm were magnetized before scanning over the surface with a Hall probe. The result is a spatial resolved map of the magnetic flux distribution.



Hall scans in the Meißner state after zero field cooling and applying 5 mT at different temperatures. The shielding radius decreases with increasing temperature, revealing a T_c gradient inside the subelements stemming from a varying Sn content.

Conture plot shows the paths of shielding currents at 2.5 mT inside subelements of RRP-Ti wire at different temperatures at an applied field of 5 mT (right).



Correlation of the shielding radius at different temperatures with EDX scans yield the dependency of T_c on the Sn content (left). The determined dependency is stronger than the one found in literature [4], which could possibly arise due to the addition of Ti and the intragranular Sn gradient.

Outlook

Attempts of quantifying irradiation damage as function of neutron flux will be made to correlate defect density with critical current. Hall scans at higher applied fields will hopefully provide information about inhomogeneities of the critical current of single subelements by inversion of the Biot-Savart law. Irradiating scanning Hall microscopy specimens could allow a comparison of local critical currents before and after irradiation.

Acknowledgements









