Analysis of the Surface Morphology and Chemical Composition of Zr-Nb_3Sn Alloys with Zr Different Concentrations

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FERMILAB-POSTER-24-0243-

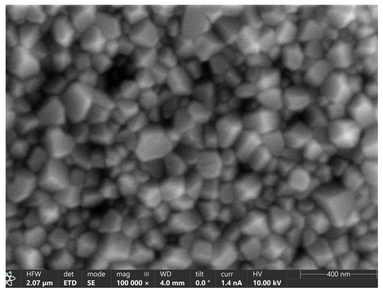
Introduction

The Zr inclusion in Nb_3Sn can improve the high magnetic field performance of Nb_3Sn radio frequency cavities. The focus of this project is to characterize the surface properties and chemical composition of Zr-Nb_3Sn. Nb_3Sn was doped with two different concentrations of Zr. ~0.5%, and ~24% Zr. Various spectroscopy techniques were used to examine how the surface morphology and chemical composition changes with increasing Zr fraction in Nb_3Sn.

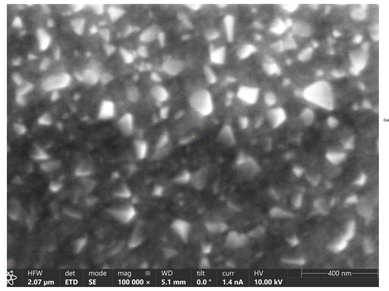
Materials and Methods

To image the surface, Scanning Electron Microscopy (SEM) was used at various magnifications (greater than 25,000x). X-Ray Photoemission (XPS) was used to determine the approximate elemental and various oxidation states of the samples. Energy Dispersive Spectroscopy (EDS) was used to analyze the elemental composition. Finally, ImageJ was used to quantify the grain distribution.

SEM



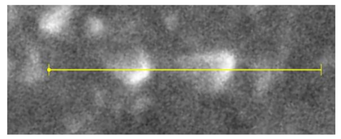
SEM image of Zr-Nb_3Sn, Zr ~ 0.5%



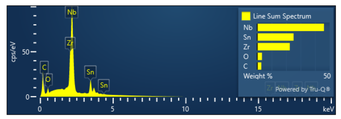
SEM image of Zr-Nb_3Sn, Zr ~ 24%

These two SEM images highlight the differences in grain formation of two samples with different Zr fractions. The image for Nb_3Sn with ~ 0.5% Zr shows clear and distinct grains, whereas with the ~24% Zr, the grains are completely unobservable at the same magnification. However, in the case of ~24% Zr sample, we observed some large particles which were further examined via EDS.

EDS

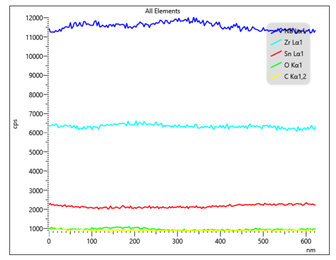


Above, Line Scan of Zr-Nb_3Sn, Zr ~ 24%



EDS Elemental Spectrum of Zr-Nb_3Sn, Zr ~ 24%-line scan

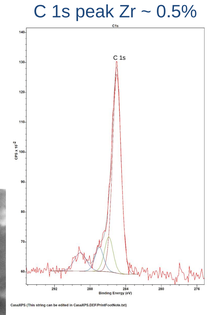
Below, Signal Comparison of Zr-Nb_3Sn, Zr ~ 24%



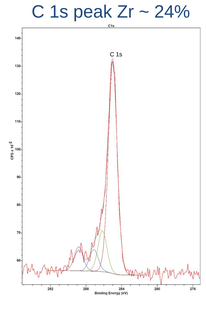
To further investigate these nanometer sized particles an EDS line scan was conducted. The results of this scan reveal slightly elevated amounts of oxygen meaning that these particles could be oxides. However, further testing would need to be conducted to confirm.

XPS

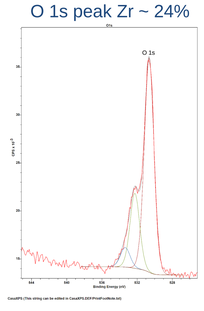
C 1s peak Zr ~ 0.5%



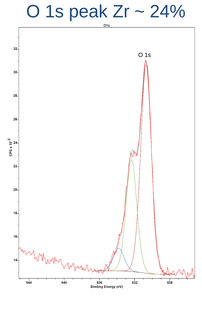
C 1s peak Zr ~ 24%



O 1s peak Zr ~ 24%

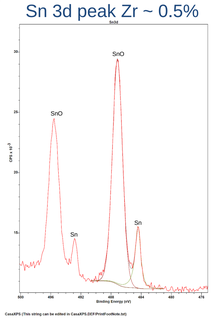


O 1s peak Zr ~ 24%

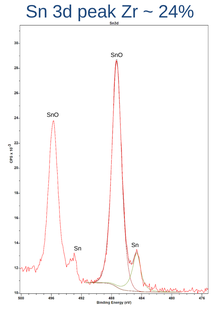


XPS data was calibrated with the Carbon 1s peak. For line shape a combination of Gaussian-Lorentzian and Lorentzian Asymmetric line shapes were used for the different peaks.

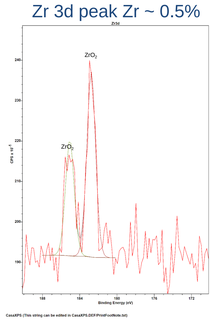
Sn 3d peak Zr ~ 0.5%



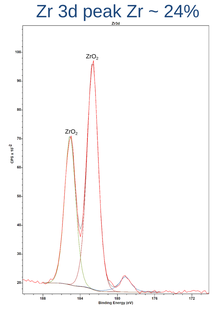
Sn 3d peak Zr ~ 24%



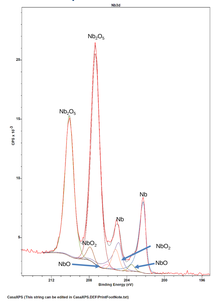
Zr 3d peak Zr ~ 0.5%



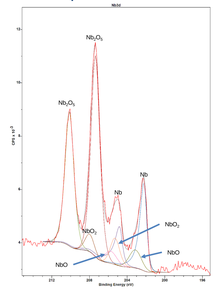
Zr 3d peak Zr ~ 24%



Nb 3d peak Zr ~ 0.5%



Nb 3d peak Zr ~ 24%



Relative Atomic %

Sample	Zr ~ 0.5%	Zr ~ 24%
Nb_2O_5 3d 5/2	71.52	61.11
Nb 3d 5/2	21.29	23.57
NbO 3d 5/2	1.56	7.19
NbO_2 3d 5/2	5.63	8.14

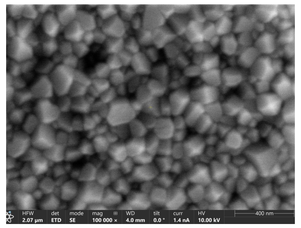
Ratio of Metal to Oxide Nb

Sample	Zr ~ 0.5%	Zr ~ 24%
Nb/NbO_x	0.270487	0.308346

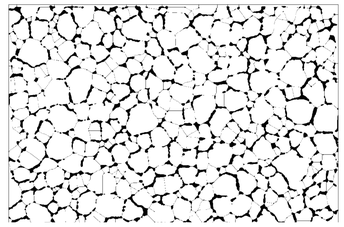
Ratio of Metal to Oxide Sn

Sample	Zr ~ 0.5%	Zr ~ 24%
Sn/SnO_x	0.191697	0.175748

ImageJ



SEM image of Zr-Nb_3Sn, Zr ~ 0.5%



Grain Boundary Map of Zr-Nb_3Sn, Zr ~ 0.5%

Grain Distribution Analysis

	Zr ~ 0.5%
Number of Grains	474
Average Area per Grain (nm^2)	5298.6
Number of Grains per Area (μm^2)	164.87

A grain distribution analysis was performed using ImageJ. The method to create this map was as follows: Set Scale > Bandpass filter > Threshold > Watershed > Particle Distribution. This analysis provided size of the grains per scan area. However, there is still scope of improvement for the method in order to account the surface voids .

Conclusion

- The grain size decrease with increasing Zr concentration and additionally at 24% concentration we observed nanoscale particles on the surface.
- EDS scan revealed the formation of possible oxides on the ~ 24% Zr sample.
- XPS shows that the NbO_x decreases in thickness as the concentration of Zr increases, conversely the SnO_x slightly increases in thickness with increasing Zr concentration.
- ImageJ analysis shows the average grain area for sample with ~ 0.5% Zr to be around 5300 nm^2

Acknowledgments

This manuscript has been authored by Fermi Research Alliance, LLC under Contract No. DE-AC02- 07CH11359 with the U.S. Department of Energy, Office of Science, Office of High Energy Physics.
This work was supported in part by the U.S. Department of Energy, Office of Science, Office of Workforce Development for Teachers and Scientists (WDTS) under the Community College Internship (CCI)

References

[1] Godeke, A. "A Review of the Properties of Nb3_33Sn and Their Variation with A15 Composition, Morphology and Strain State." Ernest Orlando Lawrence Berkeley National Laboratory, Berkeley, CA, 94720, 2006.
[2]. C. A. Schneider, W. S. Rasband, and K. W. Eliceiri, "NIH Image to ImageJ: 25 years of image analysis," Nature Methods, vol. 9, no. 7, pp. 671–675 (2012).