Contaminant analysis of polycrystalline and single crystal niobium used in accelerator cavities

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Introduction

The surface of the niobium in superconducting accelerating cavities used in particle accelerators is of significant interest since the range of the interaction of the electromagnetic fields used only encompasses the first 60nm of the niobium. Cavity performance is still not as reproducible as desired and further characterization is warranted, especially in light of the introduction of single crystal niobium cavities.

Experimental

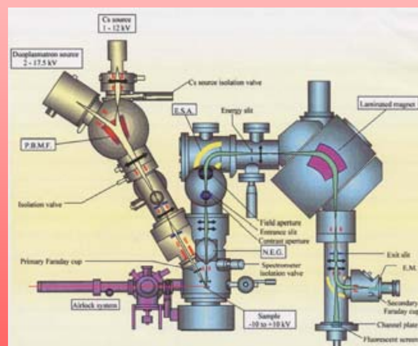
Analyses to a depth of at least 100nm have been accomplished using Secondary Ion Mass Spectrometry (SIMS) on un-annealed and annealed polycrystalline and single crystal niobium samples. Hydrogen, carbon, nitrogen, oxygen, and several metallic contaminants have been studied. Chemical state information on one sample was obtained using X-ray Photoelectron Spectroscopy (XPS).

- Niobium Specimens

Sample	*Description*	*Vendor*
Xtal	Single crystal Nb, no anneal	A
Xtala	Single crystal Nb, annealed 1250ºC	A
Polyp	Polycrystalline Nb, no anneal	B
Polyw	Polycrystalline Nb, annealed 1250ºC	B
Polyk	Polycrystalline Nb, another batch, no anneal	B
Polyt	Polycrystalline Nb, annealed 1250ºC	C

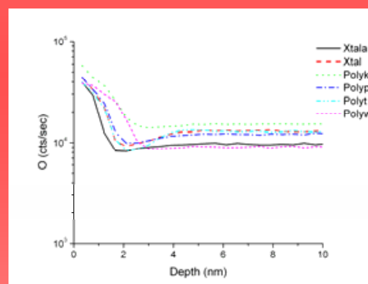
- SIMS: CAMECA IMS-6f
  - O_2^+ 1.25keV impact energy and Cs^+ 14.5keV impact energy
  - Raster size 180µm x 180µm to 220µm x 220µm
  - 60µm optically gated analyzed area
- XPS: Riber LAS-3000
  - Mg Kα x-rays with 75º take off angle from the sample surface to the electron energy analyzer, 2mm diameter analyzed area

Cameca IMS-6f Magnetic Sector Mass Spectrometer

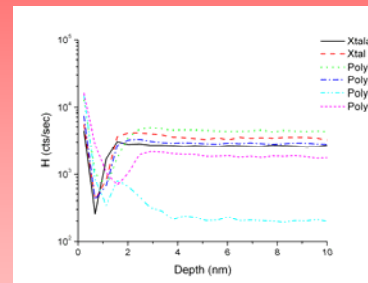


Results and Discussion

- Oxide thickness

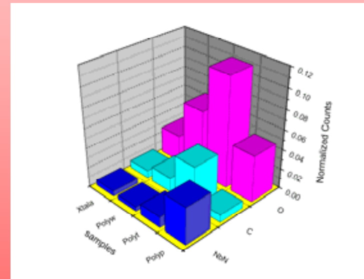


- Hydrogen profile



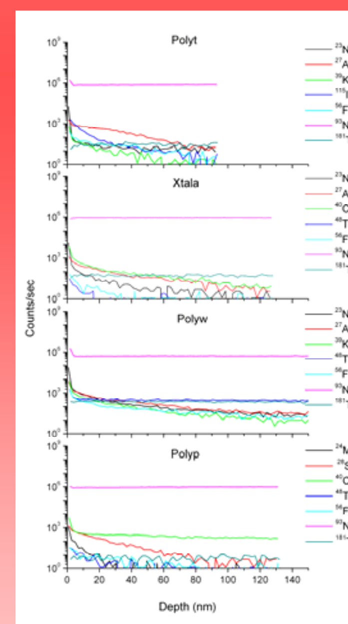
SIMS Oxygen profiles show that the niobium samples have an oxide layer thickness of 2~3 nm. Hydrogen profiles show a depletion of hydrogen in the surface oxide layer, and the polyt specimen from Vendor C has significantly less hydrogen.

- C, N, O (measured just below oxide)



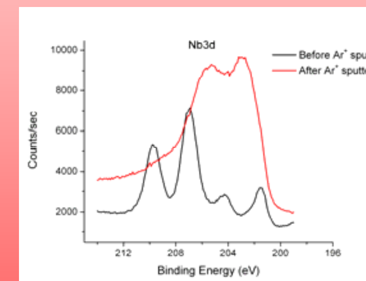
C, N, O concentrations as indicated by impurity counts normalized to Nb were lowest in the annealed single crystal Nb sample (Xtala)

- Other contaminants



Although still detectable in the bulk Nb, the highest concentrations of contaminants were detected on the surface. Tantalum appears to be uniformly distributed over the analyzed depth.

- XPS analysis of unannealed Nb single crystal (Xtal)



XPS analysis shows primarily Nb and Nb_2O_5 on the surface and Nb with a lower oxide (NbO) after sputtering to remove the approximately 2nm surface oxide.

Summary

- Use of a low impact energy O_2^+ primary beam provided sufficient depth resolution to allow determination of Nb oxide to be in the 2-3nm range depending on the sample
- Hydrogen depth profiles show a depleted hydrogen concentration in the surface oxide as compared with the bulk, and a significantly lower hydrogen concentration in a polycrystalline sample from one supplier
- Nitrogen, oxygen, and carbon are lower in the annealed single crystal sample compared with the polycrystalline samples. The concentrations of these three elements vary for the different polycrystalline samples
- Highest concentration of metallic impurities was found at the surface, but tantalum appears to be uniformly distributed through the region measured
- SIMS has shown the capability to provide useful analysis of these important impurities in this ongoing study
- XPS analysis of one sample shows significant change in oxidation state after light sputtering.

Acknowledgement

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