

CONTAMINANT ANALYSIS OF POLYCRYSTALLINE AND SINGLE CRYSTAL NIOBIUM USED IN ACCELERATOR CAVITIES*

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Abstract

Secondary Ion Mass Spectrometry (SIMS) can characterize the surface and near surface of Nb used in accelerator cavities. Results show Nb oxide in the 2-3 nm range, a depleted H concentration in the oxide compared with the bulk, and N, C, O lower in an annealed single crystal sample than several polycrystalline samples. Other metallic contaminants are primarily at the surface, but tantalum is distributed uniformly through the material.

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 electromagnetic fields 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. This study is devoted to analysis of the surface oxide and interstitial and metallic contaminants for polycrystalline and single crystal niobium.

EXPERIMENTAL

Analysis to at least 100 nm has been accomplished using Secondary Ion Mass Spectrometry (SIMS) on unannealed and annealed polycrystalline and single crystal niobium samples as listed in Table 1.

Table 1: Samples analyzed

xtala	Single crystal niobium, annealed at 1250°C
xtal	Single crystal niobium, no anneal
polyp	Polycrystalline niobium, no anneal
polyw	Polycrystalline niobium, annealed 1250°C
polyk	Polycrystalline niobium, another batch, no anneal
polyt	Polycrystalline niobium, another vendor, no anneal

SIMS analyses were obtained using a CAMECA IMS-6F magnetic sector SIMS instrument. The raster size was 180 μm x 180 μm and the detected area was a 60μm diameter circle positioned at the center of the raster. SIMS measurements were obtained using both O²⁺ at 1.25keV impact energy and Cs⁺ at 14.5keV impact energy. Chemical state information on annealed single crystal Nb was also obtained using X-ray Photoelectron Spectroscopy (XPS). XPS measurements were made

using a Riber LAS3000 with Mg Kα x-rays and 58° secondary electron take off angle.

RESULTS AND DISCUSSION

It has been known for some time that the surface of the polycrystalline Nb used in accelerator cavities has a surface oxide over a suboxide [1, 2]. Figure 1 shows XPS analysis of an unannealed Nb single crystal. The oxidation state for the surface matches that for Nb₂O₅ with some Nb, and after ~2 nm sputter the Nb changes to a lower oxidation state (apparently NbO, but altered stoichiometry and/or changes in Nb oxidation state resulting from Ar⁺ sputtering cannot ruled out).

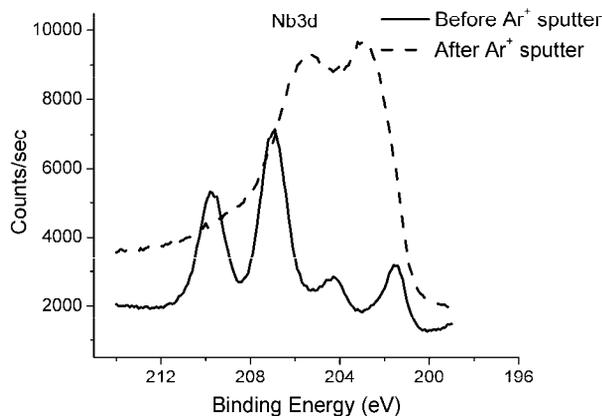


Figure 1: XPS analyses of unannealed niobium single crystal before and after sputtering to remove ~2 nm. The peaks at 202 and 204.7 eV binding energy correspond to Nb, 207 and 209.7 to Nb₂O₅, and 203.5 and 206 to lower Nb oxide.

Oxygen profiles obtained using SIMS and shown in Fig. 2 indicate that the niobium samples have variable oxide layer thickness of ~2-3 nm. All depth axes were calibrated using the sputter rate obtained for the polished single crystal sample. There were no major differences between single crystal and polycrystalline samples, with the exception that the annealed single crystal Nb sample had the thinnest Nb oxide layer. Hydrogen SIMS depth profiles shown in Fig.3 indicate a depletion of hydrogen in the surface oxide layer in all samples. It is notable that the polyt specimen from a different vendor has significantly less hydrogen in the bulk of the sample.

*Supported in part by DOE contract DE-AC05-84ER40150

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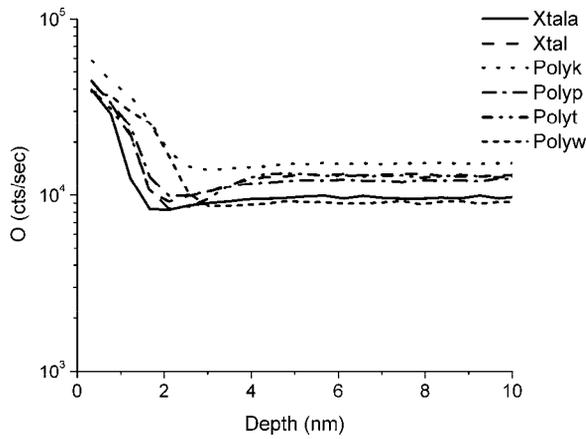


Figure 2: SIMS depth profiles for O on polycrystalline and single crystal.

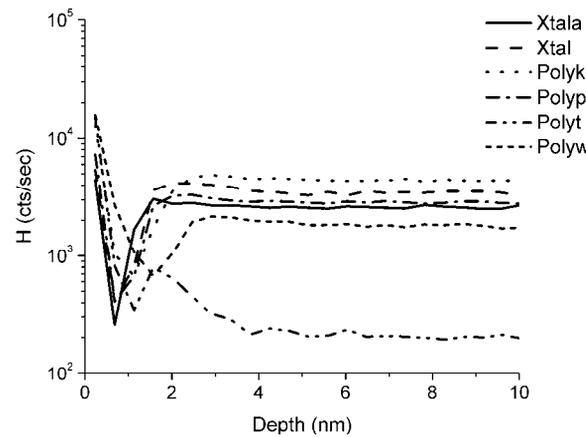


Figure 3: SIMS depth profiles for H on polycrystalline and single crystal.

C, N, and O are contaminant species considered to act as interstitials. SIMS analysis for these elements summarized in Fig. 4 shows the lowest contaminant levels for the single crystal sample. Of the metallic contaminants studied (Na, Al, Ca, Ti, Fe, Ta), the highest concentration was found at the surface of the samples. These impurities were detected for all samples as shown in Fig. 5.

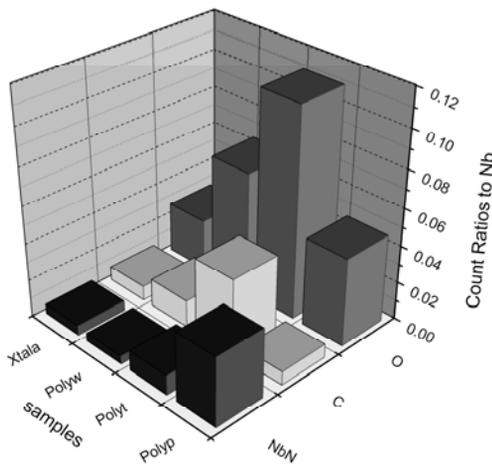


Figure 4: SIMS data for C, N, and O on polycrystalline and single crystal.

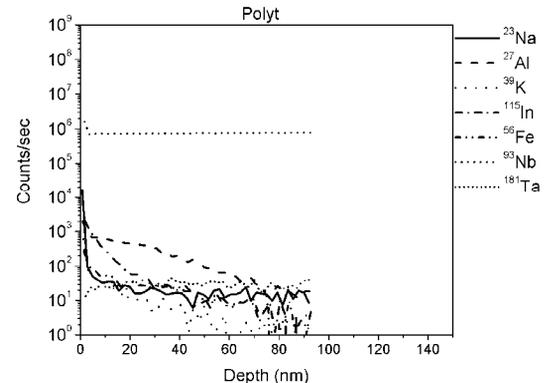
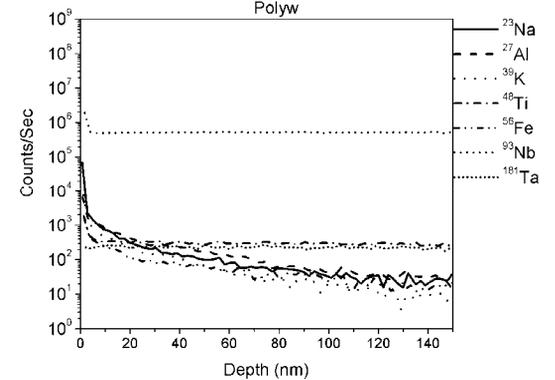
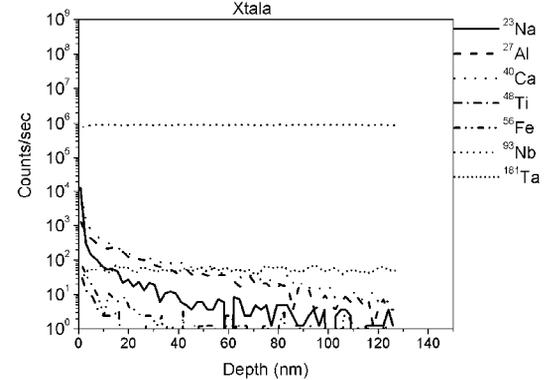
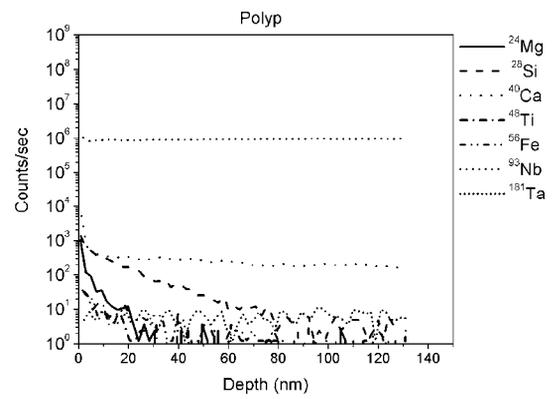


Figure 5: SIMS data for metallic impurities on polycrystalline and single crystal.

SUMMARY

Use of a low impact energy O_2^+ primary beam provided sufficient depth resolution to allow measurement of the

Nb oxide. While most metallic impurities are found near the surface, tantalum is distributed uniformly throughout all samples. Hydrogen depth profiles show a depleted hydrogen concentration in the surface oxide as compared to the bulk for all samples, and a significantly lower hydrogen concentration in a polycrystalline sample from one supplier (polyt specimen). Nitrogen, oxygen, and carbon are lower in the annealed single crystal sample as compared to the polycrystalline samples. SIMS has shown the capability to provide valuable analysis of these important impurities in Nb.

REFERENCES

- [1] M. Strongin et al., Particle Accelerators 3, 209 (1972).
- [2] M. Grundner and J. Halbritter, J. Appl. Phys. 51, 397 (1980).