DEVELOPMENT OF SIMS STANDARDS FOR MEASUREMENT OF H, C, O, AND N IN NB

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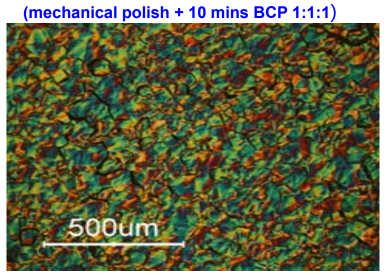
Abstract:

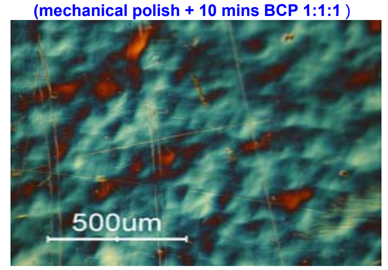
Performance of SRF cavities is strongly dependent on the interior surface due to the shallow RF penetration depth. Therefore, study Nb surface characteristics and contamination which could affect the performance of the SRF Nb cavity is of great importance. C, N, and O interstitial contaminants are of particular importance and earlier work suggested very high H concentration [1]. Secondary Ion Mass Spectrometry (SIMS) has the depth resolution and sensitivity to quantitatively measure these species in the region of interest. However, standards for quantitative SIMS analysis of these elements in Nb did not exist. Initial attempts to develop an ion implanted standard were unsuccessful because of the roughness of the Nb surface and the inability to polish the soft metal Nb surface with traditional methods. In this study, Nb samples were specially chemical mechanical polished followed by a light buffered chemical polish (BCP). The result is a surface finish suitable for SIMS analysis and implantation standards. Ion implants of C, N, O, and deuterium (D) were obtained in Nb (and simultaneously in Si for dose verification). D was implanted to characterize H to avoid the high H background. The results show that D is apparently also very mobile in Nb, and another approach will be required to quantify this H in Nb. This multi-element implant standard has already been of great benefit in characterization of C, O, and N in polycrystalline and large grain Nb [2].

Surface Topography of Nb Samples for SIMS

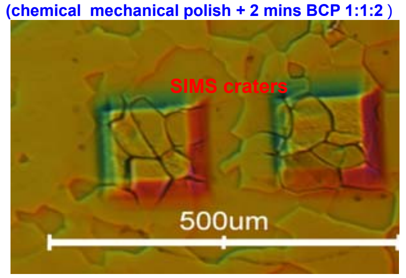
Polycrystalline Nb
(mechanical polish + 10 mins BCP 1:1:1)



Single Crystal Nb
(mechanical polish + 10 mins BCP 1:1:1)



Polycrystalline Nb
(chemical mechanical polish + 2 mins BCP 1:1:2)



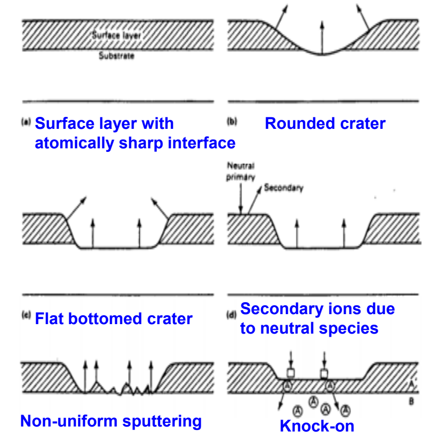
Single Crystal Nb
(chemical mechanical polish + 2 mins BCP 1:1:2)

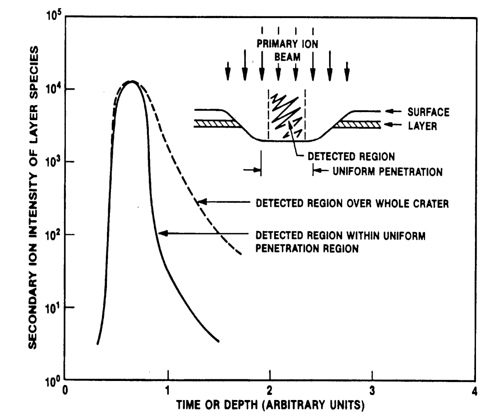
- •BCP treated polycrystalline and single crystal Nb surfaces are unsuitable for SIMS implant quantification standards because of degraded depth resolution and the difficulty of making crater depth measurements resulting from surface roughness.
- •Chemical mechanical polished Nb single crystal samples with light BCP have nm order surface roughness and are suitable for implantation standards.

Secondary Ion Mass Spectrometry (SIMS) Measurements

- •All SIMS analyses were obtained with a CAMECA IMS-6F. Analyzer vacuum was kept at ~2×10^{-10} Torr to minimize background contributions from H, C, N, O residual gas species present in the analysis chamber.
- •A Cs^+ beam with a beam current of 15-20nA and a raster size of 120-130μm was used for analysis. SIMS secondary ions were detected from a 30μm diameter optically gated region at the center of the crater.
- • Higher energy (14.5keV impact) was used for initial measurements on the standards. Lower energy (6keV impact) was then used to obtain better depth resolution to characterize the near surface region.

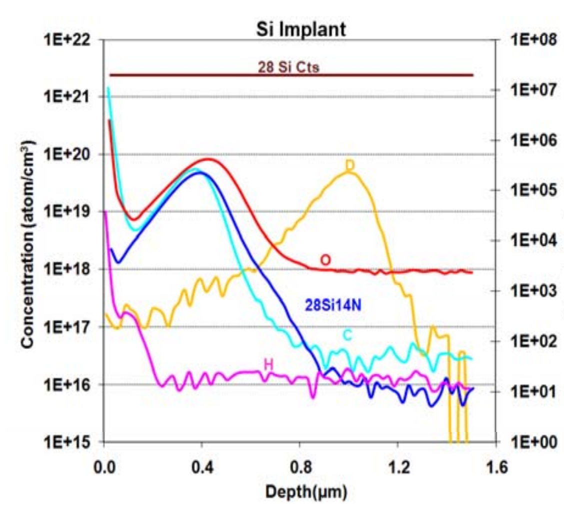
Artifacts On Surface Can Distort SIMS Depth Profile [3,4]

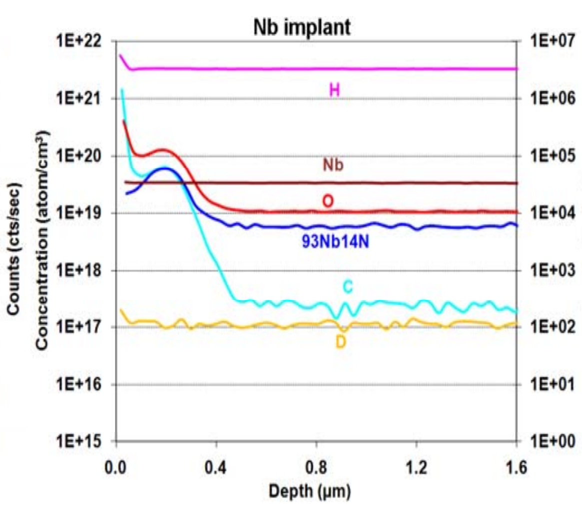




- •Accurate crater measurement requires a flat surface.
- •Poor crater shape and rough surfaces will distort profiles (loss of depth resolution) .
- •Reduce side-wall contributions by detecting ions from center of larger crater.
- •Optimize distance from wall to detected area

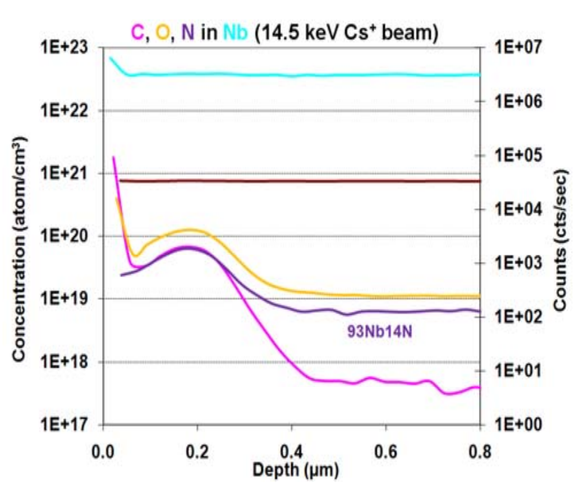
H, D, C, N, O Implants in Si and Nb (Single Crystal)

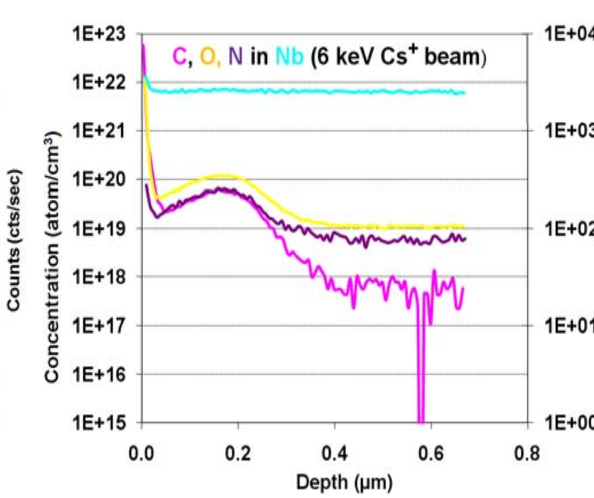




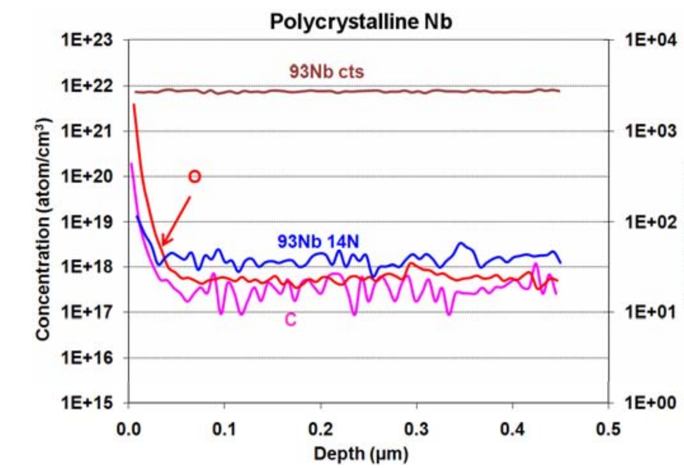
- •Pieces of Si and Nb were implanted simultaneously to take advantage of well characterized implants in Si available at NCSU/AIF to check the implanted dose.
- •D is very mobile in Nb as indicated by the inability to obtain a depth profile of implanted D in Nb. C, O, and N show typical implant profiles in both matrices.

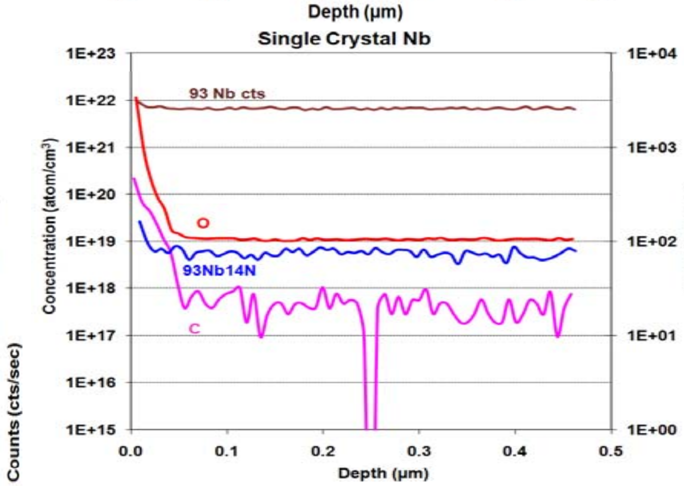
Low Impact Energy Shows Improved Depth Resolution





SIMS Depth Profiles at 6keV Polycrystalline *vs.* Single Crystal Nb





- •Measurements on polycrystalline and single crystal
- •Nb showed N and O were lower in the polycrystalline sample and C was at a low level for both samples.

Conclusion:

Quantitative SIMS analyses of near surface C, N, and O in SRF Nb were made possible by use of ion implantation into nano-smoothed single crystal Nb. Measurements show N and O were lower in the polycrystalline sample than the single crystal sample, and both samples showed low C. Analysis of a D implant in Nb showed D was very mobile. SIMS based and other methods are being explored to measure H and D in Nb. SIMS analyses of C, N and O were shown to be useful to improve our understanding of the near surface distribution of these elements in Nb and increase our understanding of Nb for use in SRF cavities.

References

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