# TOF SIMS Temperature Study of Hydrogen in Niobium

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## *INTRODUCTION*

• Previous work has shown that H content in Nb can be related to the efficiency of SRF cavities.[1]

• A heat treated (800ºC : 3hrs; 120ºC : 24hrs) large grain Nb sample shows less H near surface and higher RRR compared with a non heat treated sample.

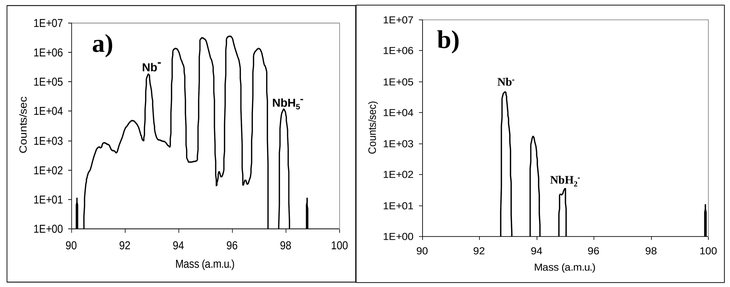
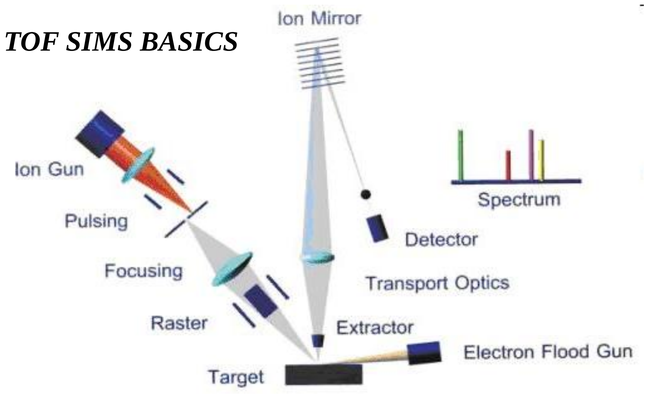



*Fig.1 SIMS Mass spectra of Nb large grain samples. a) non heat treated, b) heat treated Significant reduction in H containing species due to heat treatment[2]*

## *TOF SIMS BASICS*




## *OBJECTIVE*

To study the behavior of H in Nb at low temperatures (160 - 300ºK) and high temperatures (300-475ºK).

## *SAMPLES & INSTRUMENTATION*

• Large grain Nb non heat treated (control) samples were used for both the high and low temperature studies.

• ION-TOF 5 Time of Flight Secondary Ion Mass Spectrometer (TOF-SIMS) with a cold/hot stage was used to study the top 200nm surface of the specimens for H levels.

• All TOF-SIMS analyses were made using a 25keV $Bi_3^+$ analysis beam and a 10keV $Cs^+$ sputter beam.

• The sputter area was 120x120 µm$^2$ and the detected area was 50x50 µm$^2$ with analysis current of 0.7pA and sputter current of 20nA

## *LOW TEMPERATURE ANALYSES*

• Analyses were performed first decreasing temperature from 300 to 160ºK and then increasing to 300ºK.

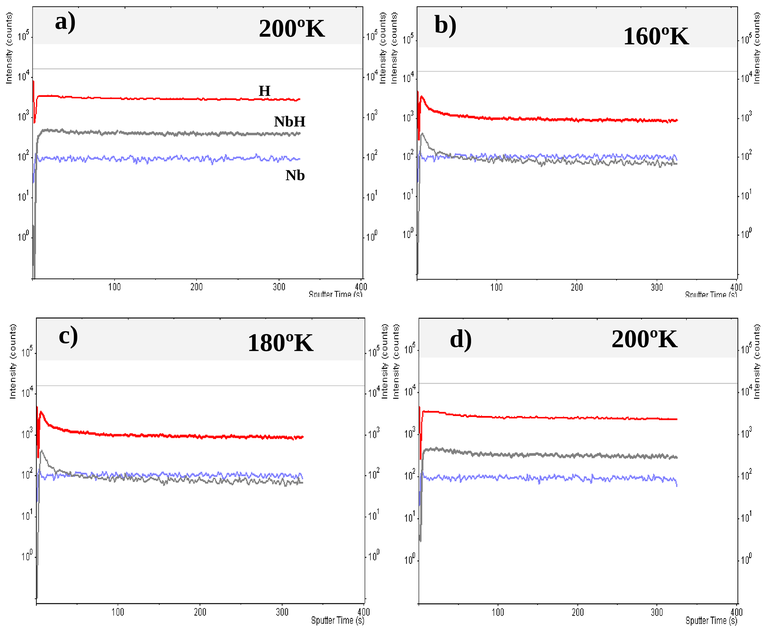
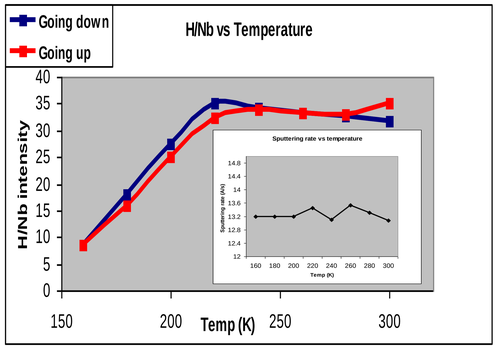



*Fig.2 H signal vs time as temperature is decreased from 200-160ºK (Fig. 2 a and b) and increased from 180-200ºK (Fig. 2 c and d). Results indicate that H signal decreases as temperature reduced below 200ºK*

*Fig. 3 H/Nb ratios obtained in the temperature range 160-300ºK. The difference in the two curves shows thermal hysteresis. H seems to follow a trend of first increasing and then decreasing as temp is decreased. This effect was reproducible. Enclosed fig. shows sputtering rate vs temperature*




## *HIGH TEMPERATURE ANALYSES*

• TOF SIMS was also used for high temperature analyses (300-475ºK) on a control sample.

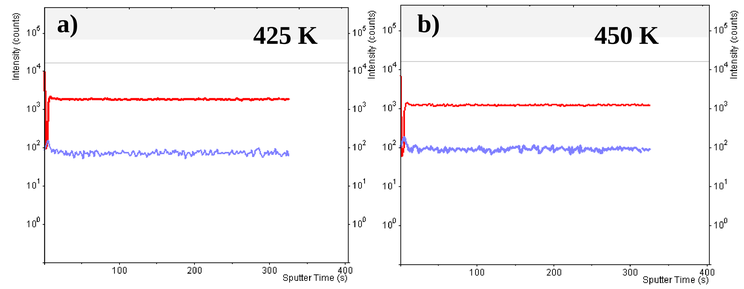



*Fig. 4 H signal vs time data shows a significant decrease in H/Nb intensity as specimen is heated from 425 to 450ºK*

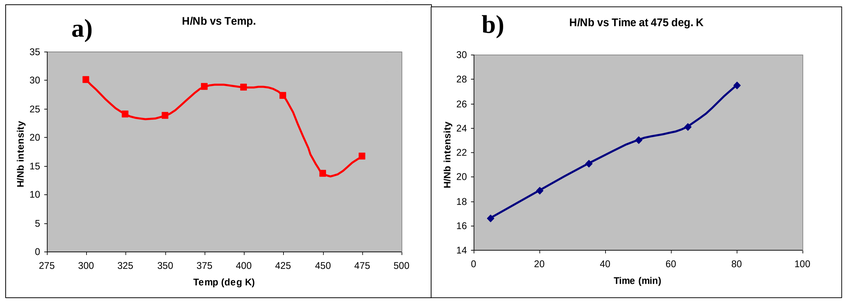



*Fig. 5 a) H/Nb intensity extracted from the TOF SIMS data in the temperature range 300-475ºK. The ratio shows a trend of first decreasing, then increasing and then significantly decreasing for T > 425ºK. Fig. 5 b) shows the H/Nb intensity vs time at 475ºK which shows increase with time*

## *CONCLUSION*

• TOF SIMS analyses of H in Nb were performed both at low and elevated temperatures.

• Low temperature analyses showed that H/Nb intensity decreased significantly for T < 200ºK and that it reaches a maximum for 200> T > 250ºK. H/Nb intensity also seems to decrease at T> 425ºK in the elevated temp analyses.

• Future work is planned to study the phase transformations of H in Nb using Differential Scanning Calorimetry and X-Ray Diffraction techniques and correlate this data to the H-Nb phase diagram.

### *REFERENCES*

1. *G. Ciovati, G. Myneni, F. Stevie, P. Maheshwari, D. Griffis, Physical Review Special Topics-Accelerators and Beams 13, 022002 (2010)*
2. *P. Maheshwari, et al., Surf. Int. Anal. 43, 153 (2011)*

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