6^{th} SRF Materials Workshop, Tallahassee, FL 2/18/2010

Fundamental Surface Chemistry of Nb Oxidation

Miki Nakayama, Tuo Wang, Nataliya Yufa,
Dan Killelea and Steven J. Sibener
The James Franck Institute & Dept. of Chemistry, U. of Chicago

Today's Presentation:
Polishing & XPS Oxidation Involving Technical Substrates
Next: Moving to Single Crystal Studies – XPS and STM

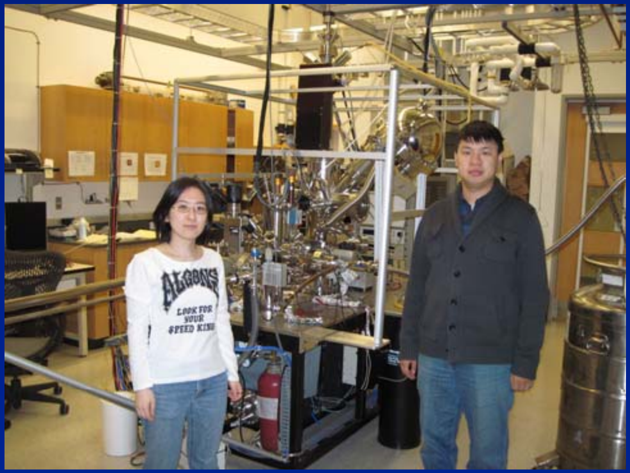
Collaborators:
Lance Cooley, SRF Materials Group, FNAL
Cabot Microelectronics - Polishing

Acknowledgments:
University of Chicago – FNAL Seed Grant Program
DOE-HEP

Key Issues

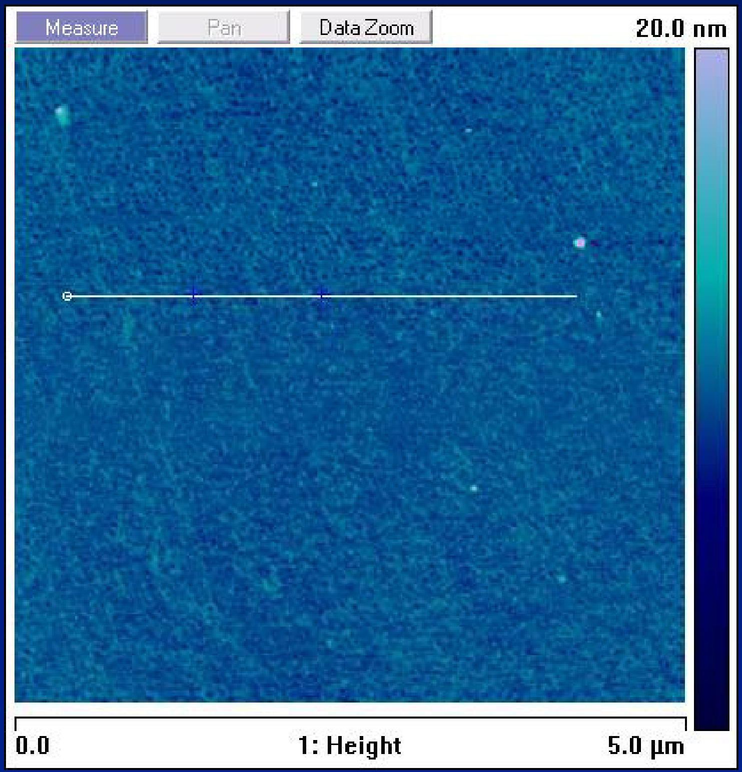
- Nature of the clean Nb surface
- Oxidation
  - Mechanism and kinetics
  - Effect of crystal face, polycrystallinity, grain boundaries
  - Effect of other species (e.g. H_2O, H_2)
  - Stability of oxides
  - "Communication" with bulk
- Role of defects
- Role of C and NbC
- Effect of various treatments on interfacial chemistry
  - Baking / cooldown
  - Polishing / cleaning
- Can we design an optimal chemical/ mechanical polishing procedure?
- Effect of above on superconductivity

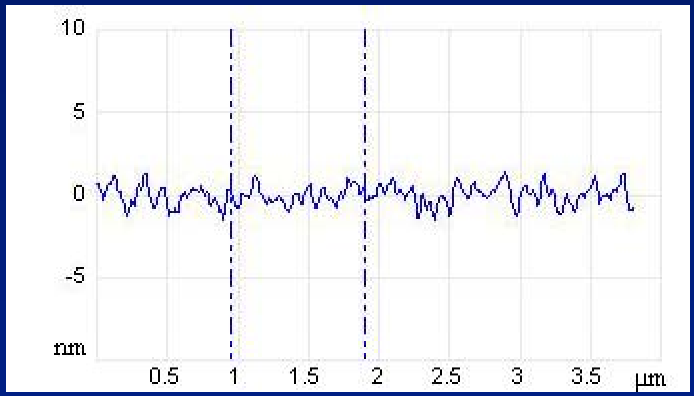
UHV Instrument: equipped with STM, XPS, Ion Sputtering, AES
Graduate Students: Miki Nakayama and Tuo Wang



Cavity-grade Niobium Samples Can Be Polished to Sub-nanometer RMS Roughness

Atomic Force Microscopy Image of As-Received Sample





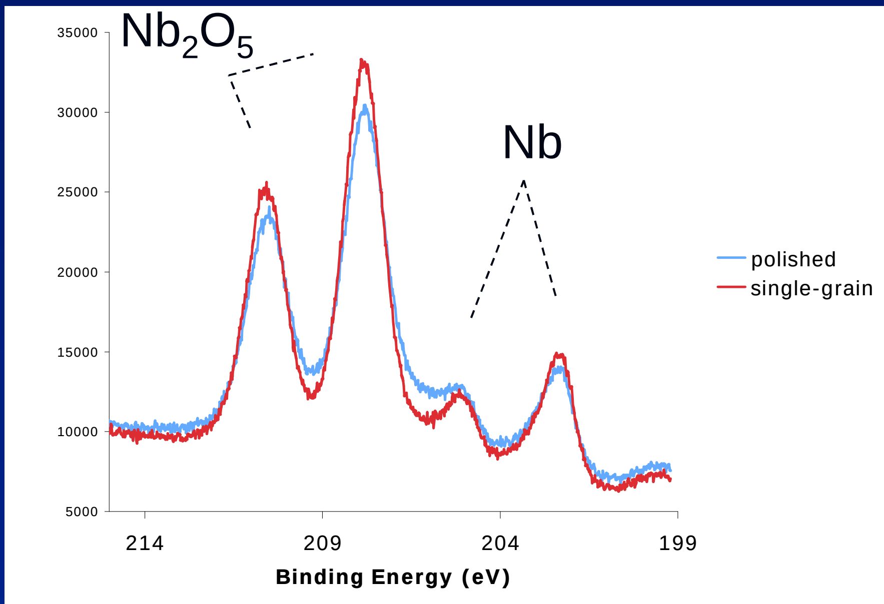
Average roughness is <0.5 nm

Polishing: Cabot Microelectronics, Aurora, IL

Imaging: Nataliya Yufa, Graduate Student, U. of Chicago

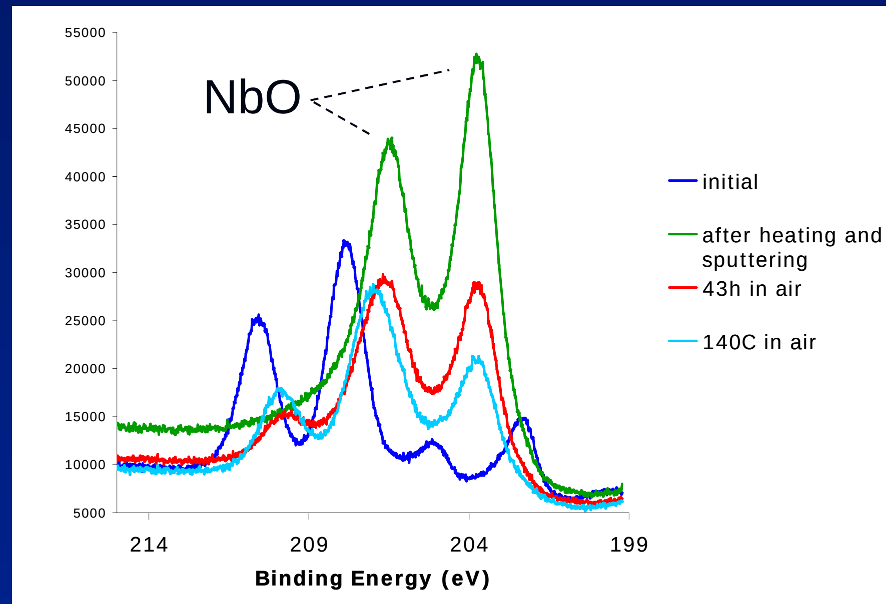
XPS of Niobium in Pristine State and Following Heating (in Air or UHV) & Sputtering

XPS of Pristine Sample – Nb 3d
Nb_2O_5 Dominated Interface



- Oxide thick as Nb XPS peaks from underlying Nb are relatively weak

Heating in Air and Aging
Thermal Treatment 1 Hour at 140 ˚C

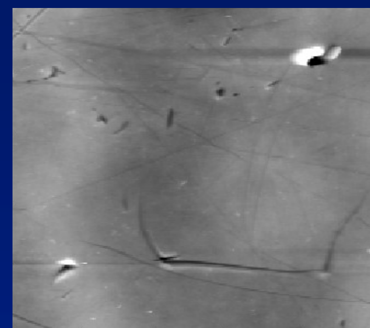


- NbO robust and persistent in UHV after sputtering and heating
- Surface region reincorporate O upon air exposure or heating in air

AFM and SEM of Sample w/ Welds

- Polished side of large sample supplied by FNAL
  - Many scratches seen with AFM as well as a few pits
  - Defects span nano- through micron-sized features

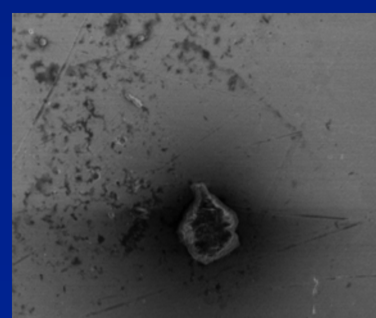
50 x 50 µm^2



25 x 25 µm^2

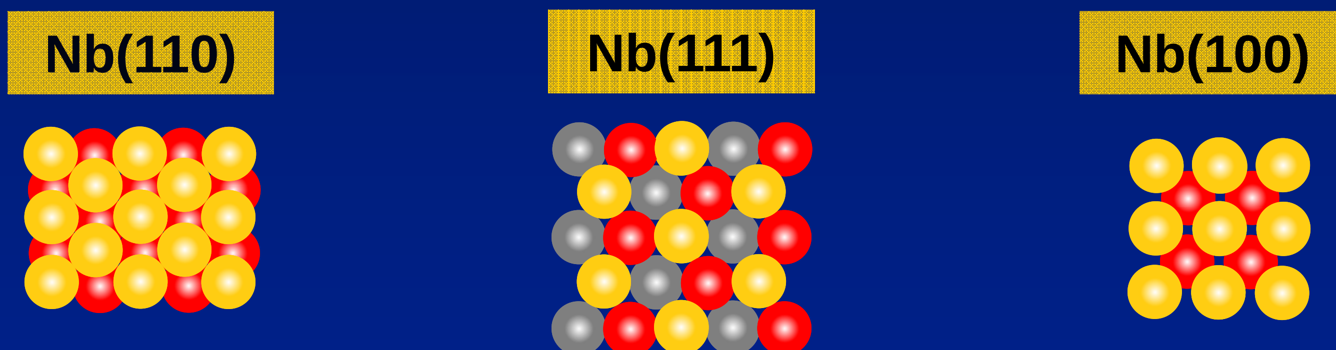
5.75 x 5.75 µm^2

  - One area observed with SEM with many pits and a large pit that seems to cause charging (black area shown below)



20 µm

Niobium Single Crystals
Nb(110), Nb(111) and Nb(100)



- Three crystal faces, material 99.99% purity
- Polished to ~0.1º of crystallographic orientation
- Final polish 0.05 microns
- Source: Surface Preparation Lab, Zaadam, The Netherlands

= top layer Nb
= second layer Nb
= third layer Nb

Summary and Plans Going Forward

Early work: emphasis on precision studies of oxidation mechanisms on technical substrates

- FNAL supplied & Cabot Microelectronics polished samples used at UC for initial oxide studies; polishing achieves < 1nm rms roughness
- Initial exploration of the efficacy that different heating, sputtering, and polishing preparations have on the quality of the interface

Program: emphasis shifts to precision studies of oxidation mechanisms on single crystal substrates

- Ion sputtering, annealing, oxidation in UHV to simulate cavity etching, baking, aging under controlled conditions
- Vibrational and STM studies will complement XPS
- Experiments to assess oxidation kinetics (including O_2, H_2, C, H_2O), structure & defects, and correlations with physical properties
- Other Issues: Stability of the Interface? Passivation vs. Conditioning?