Structural and Compositional Analysis
Of
Single Crystal Niobium

via
Transmission Electron Microscopy (TEM)
And
Secondary Ion Mass Spectrometry (STEM)

Phil Russell

FIB Prepared TEM Cross Sections for Jefferson Labs Nb (100), (110), (111)

Phil Russell
Dale Batchelor
Donovan Leonard

Analytical Instrumentation Facility
North Carolina State University

Overview

- Samples sputter coated with 60nm of Au-Pd to prevent surface damage from Ga Focused Ion Beam (FIB)

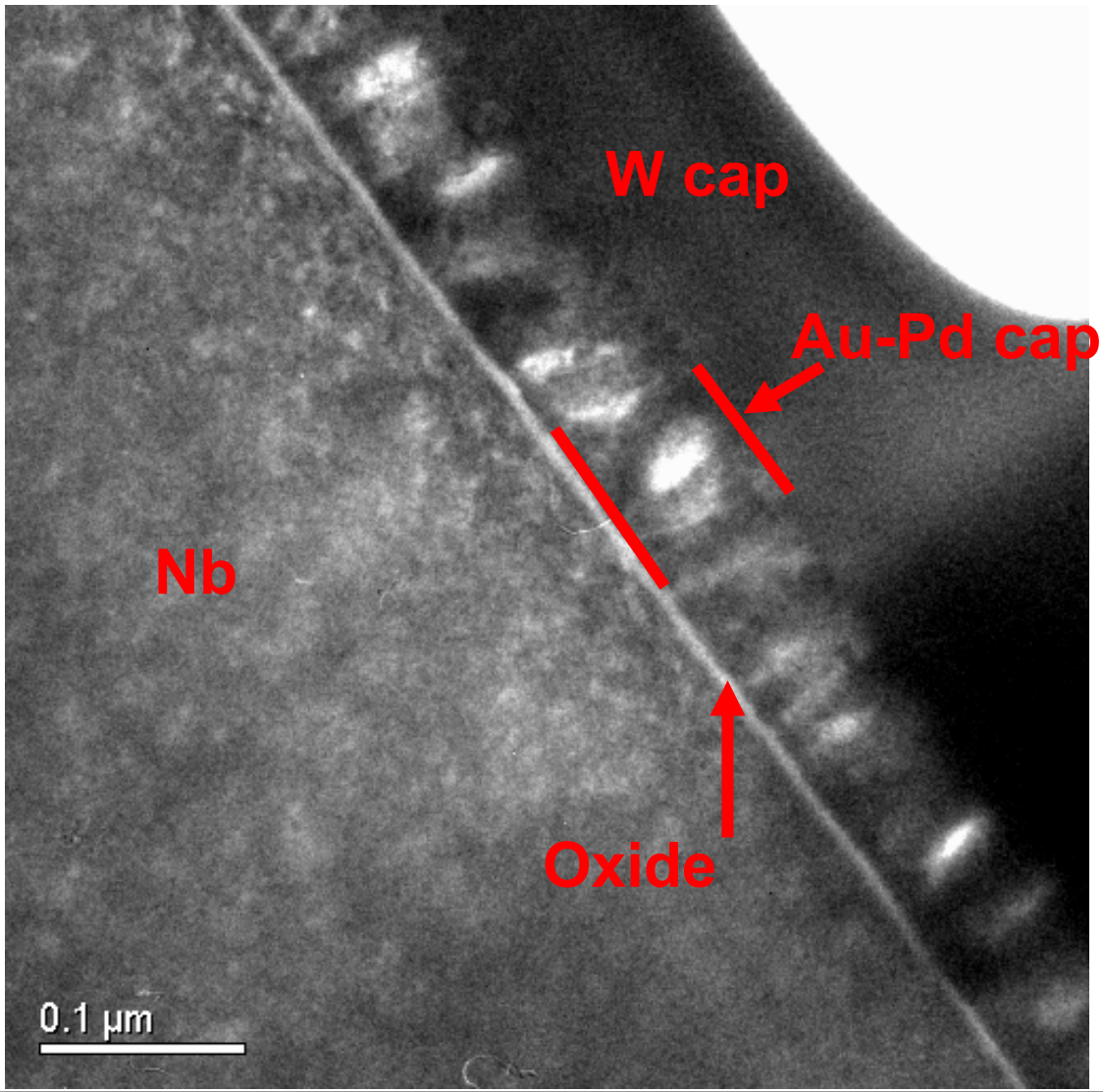
- Samples additionally coated by in situ deposition with 2µm of W in region of analysis

- FIB preparation performed with an Hitachi FB-2100 Focused Ion Beam System

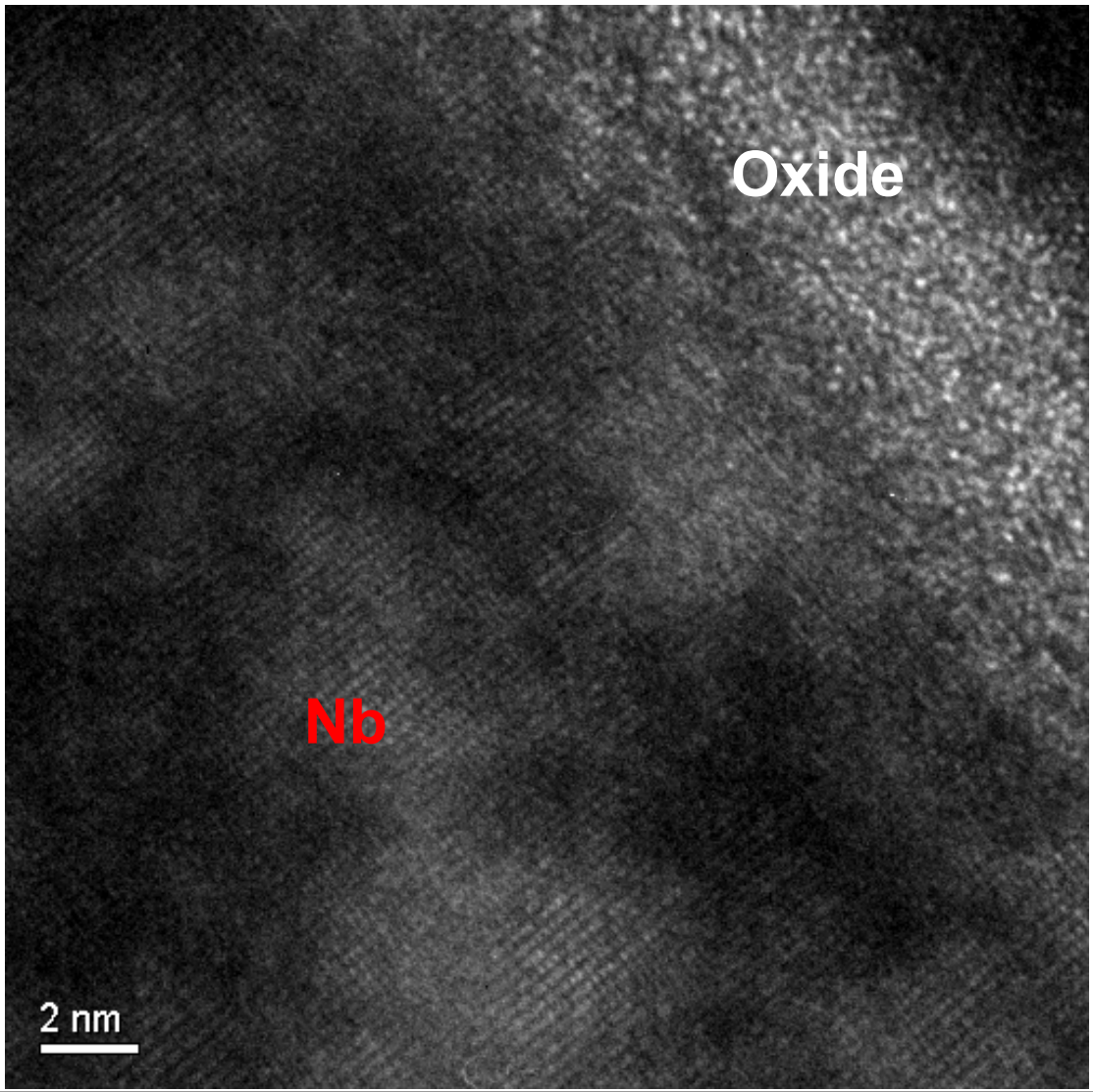
- TEM micrographs captured with a Gatan Digital Camera on a JEOL JEM2010F High Resolution TEM/STEM

- Oxide Nb(100) ~4.9nm, Nb(110) ~8.3nm, Nb(111) ~7.5nm

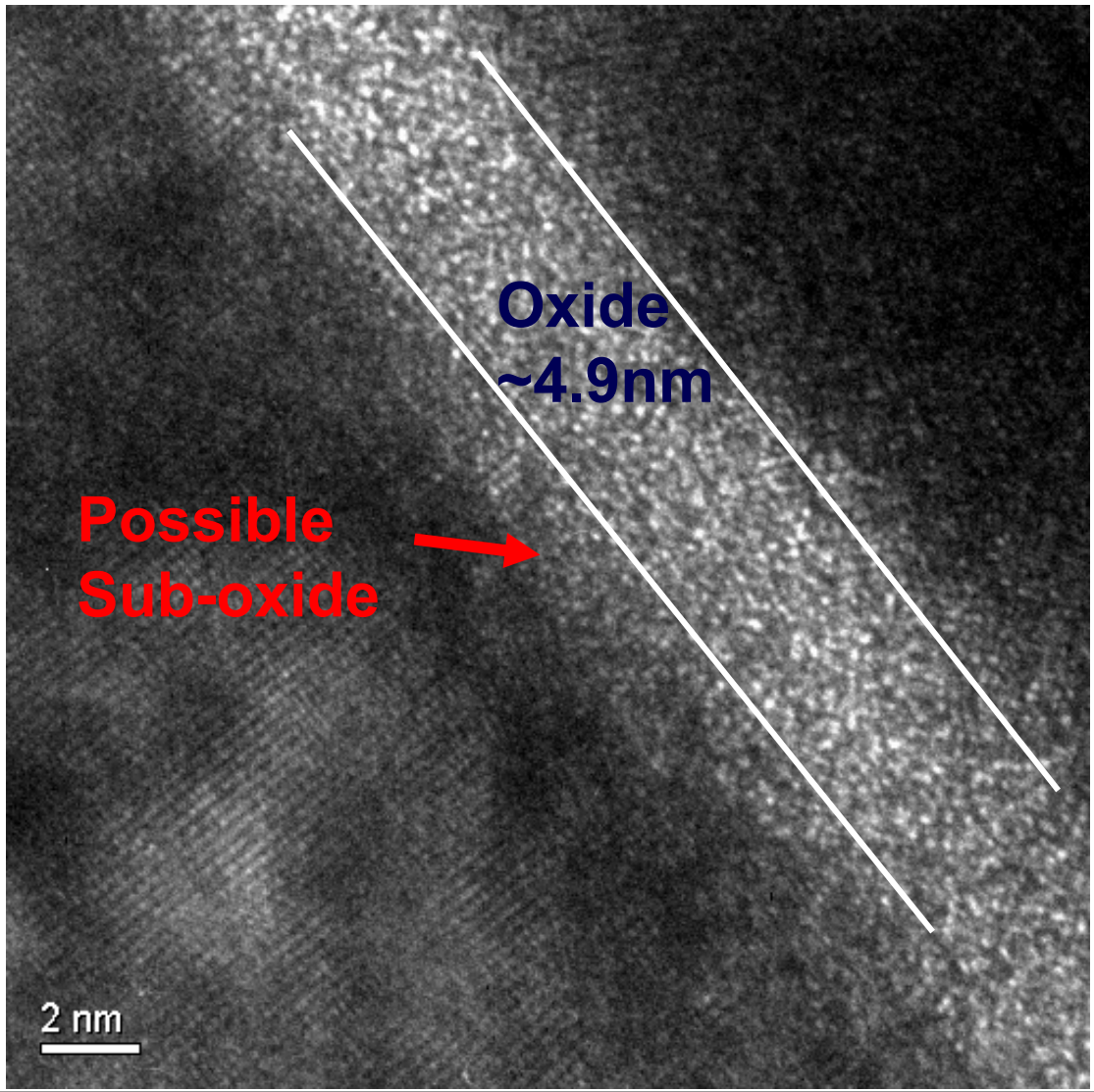
Nb (100)



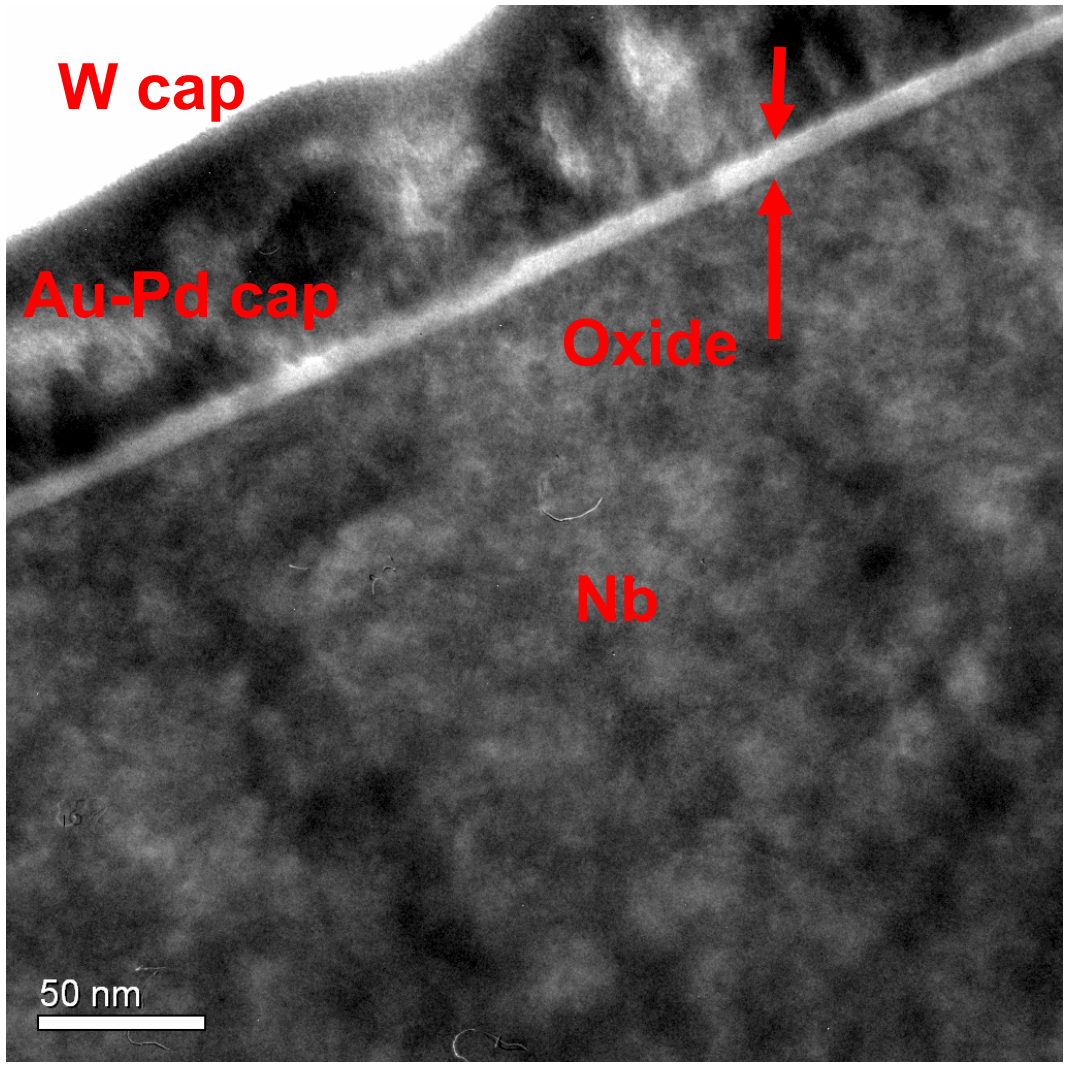
Nb (100)



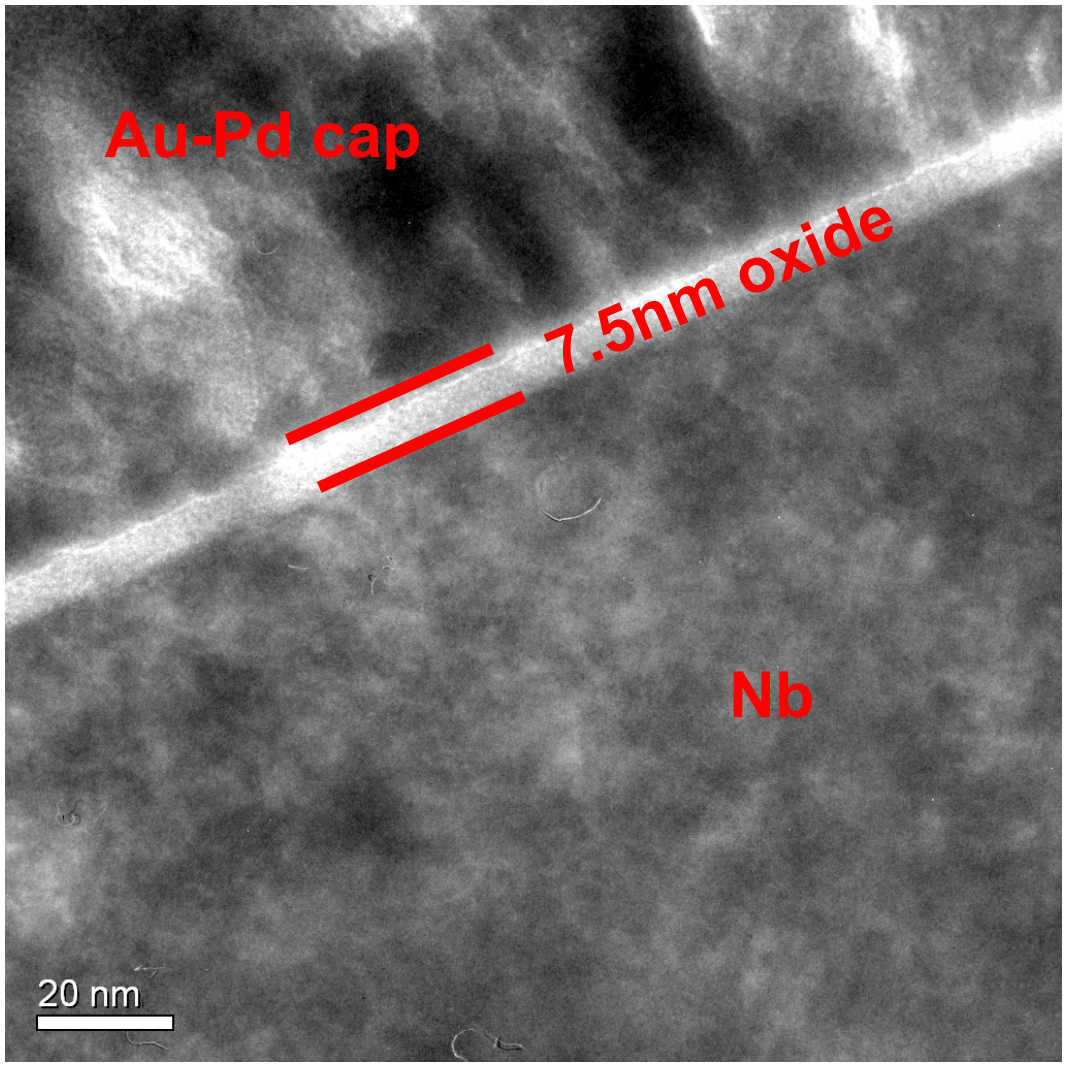
Nb (100)



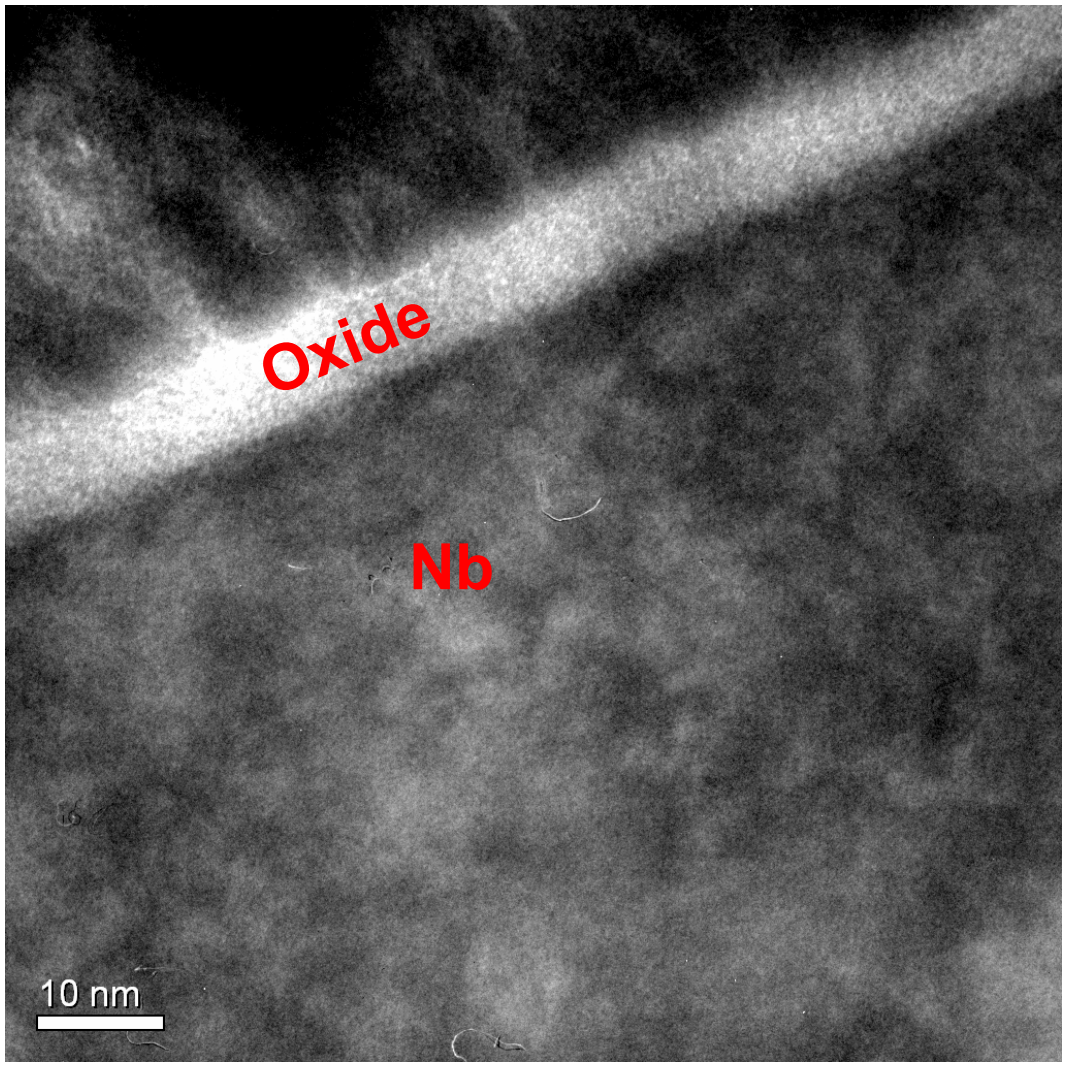
Nb (111)



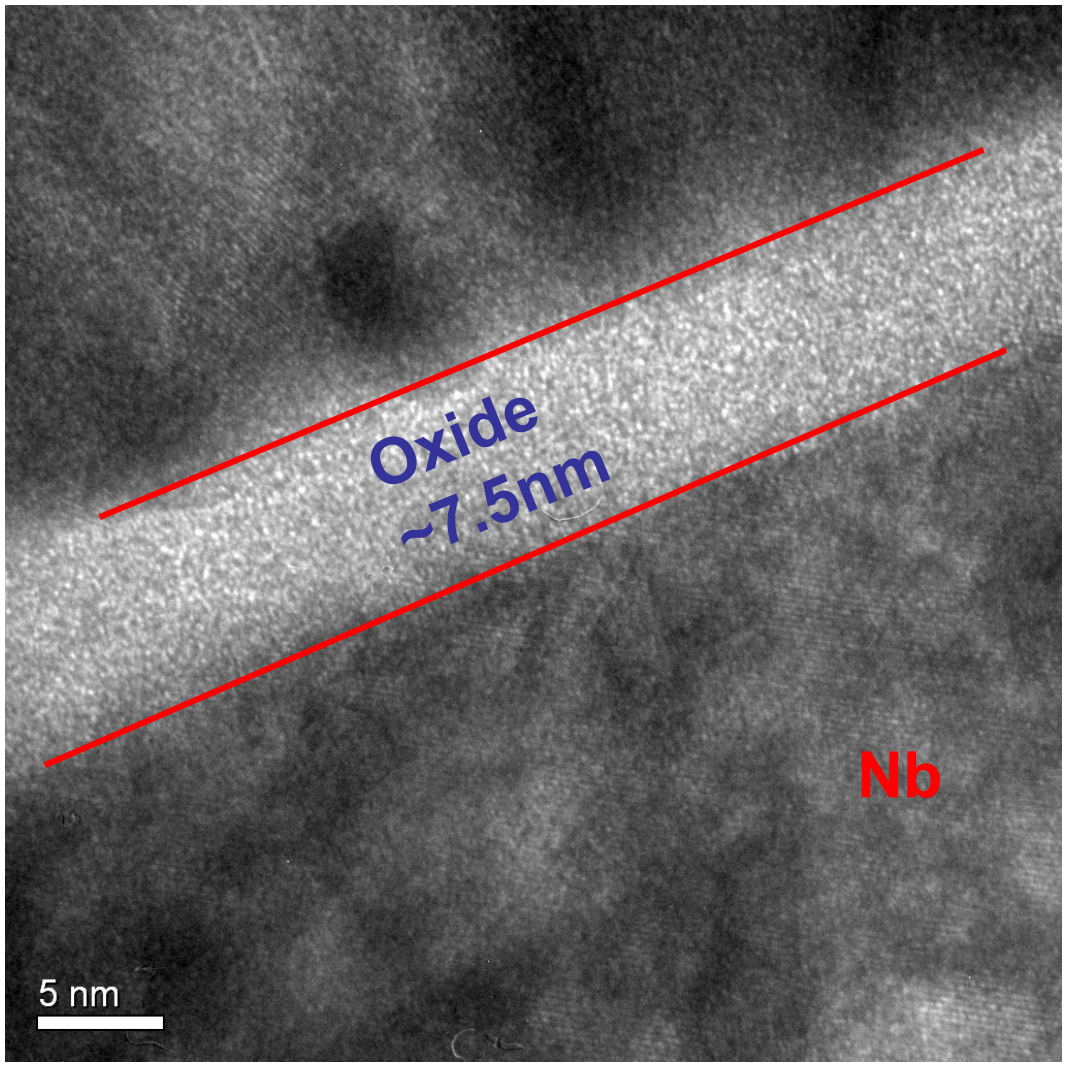
Nb (111)



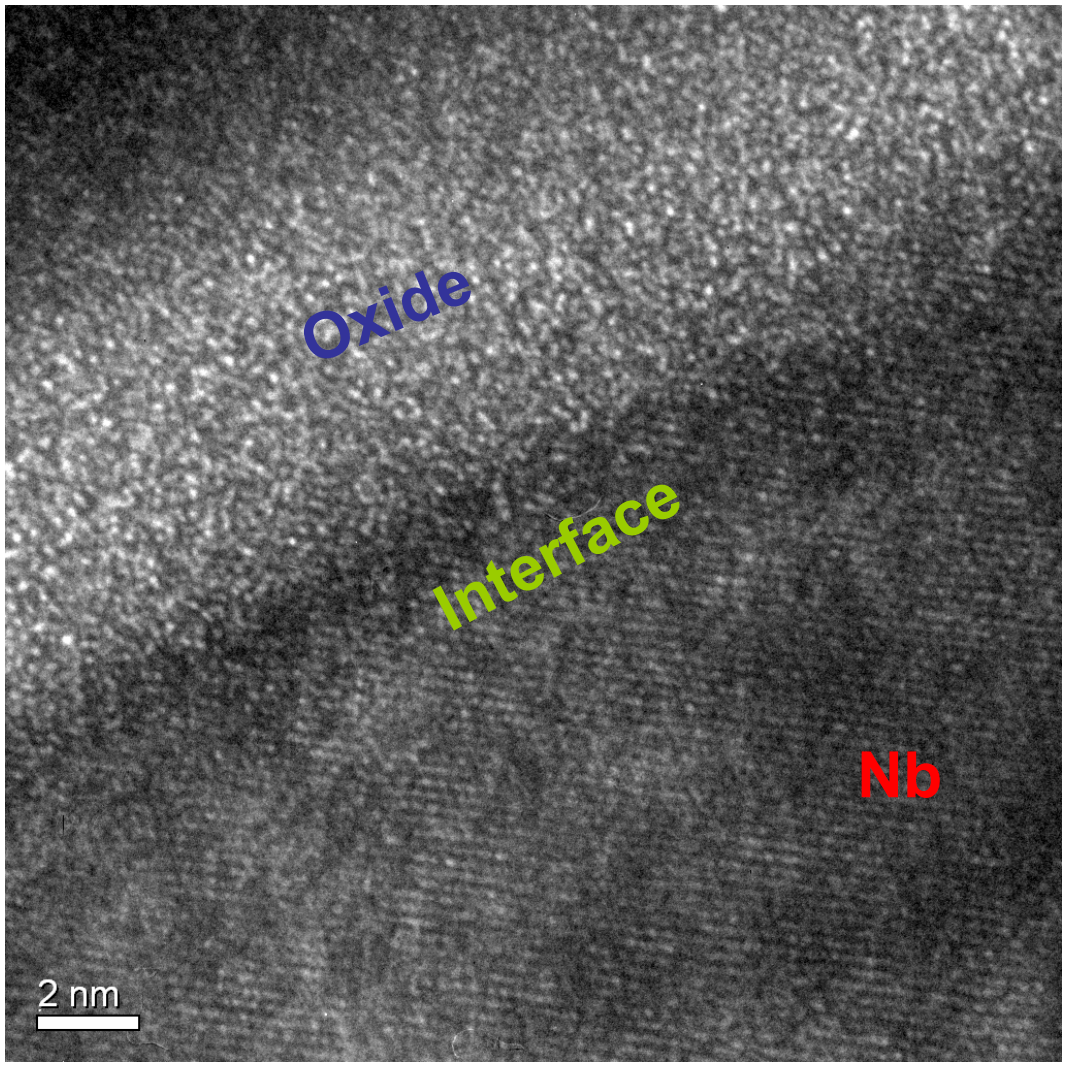
Nb (111)



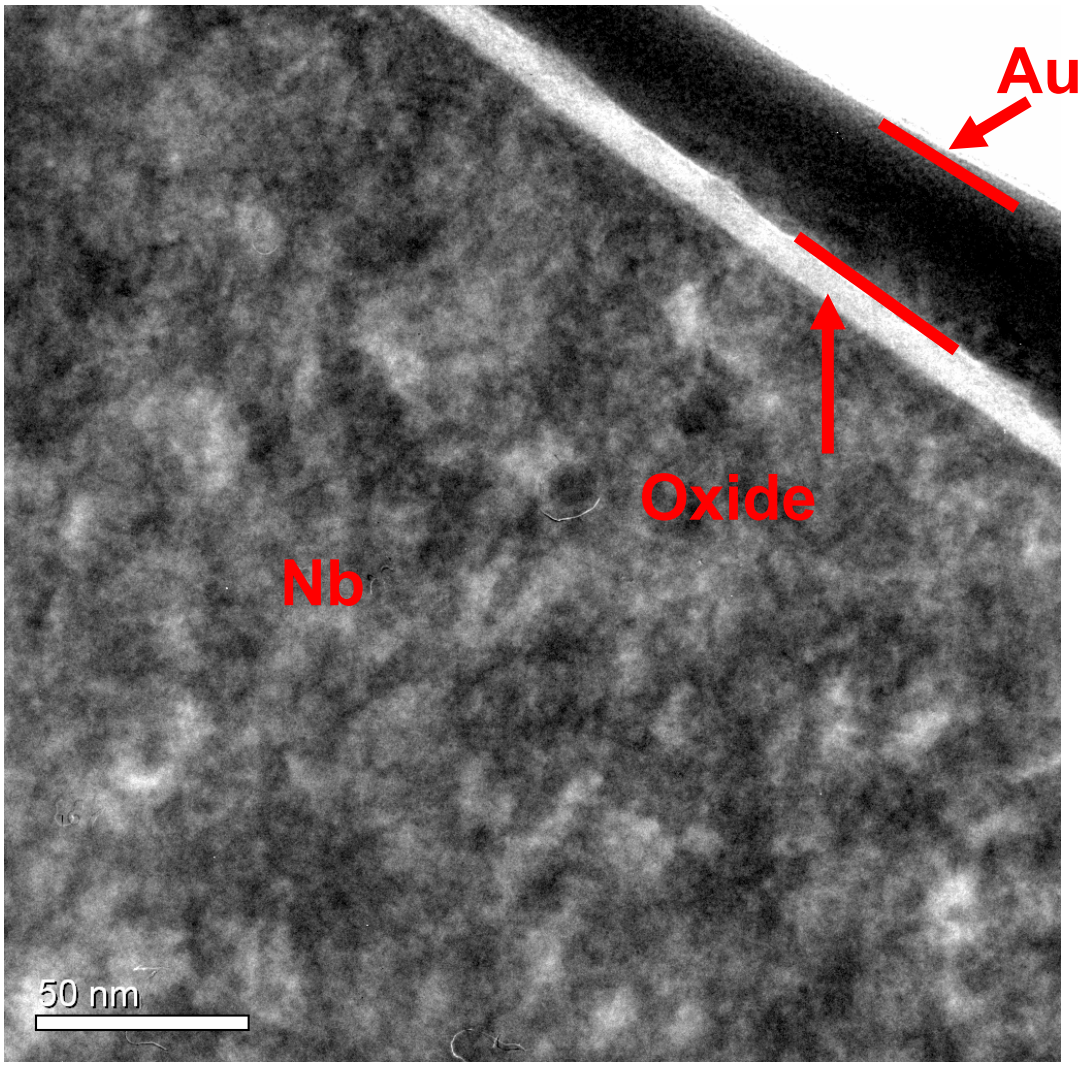
Nb (111)



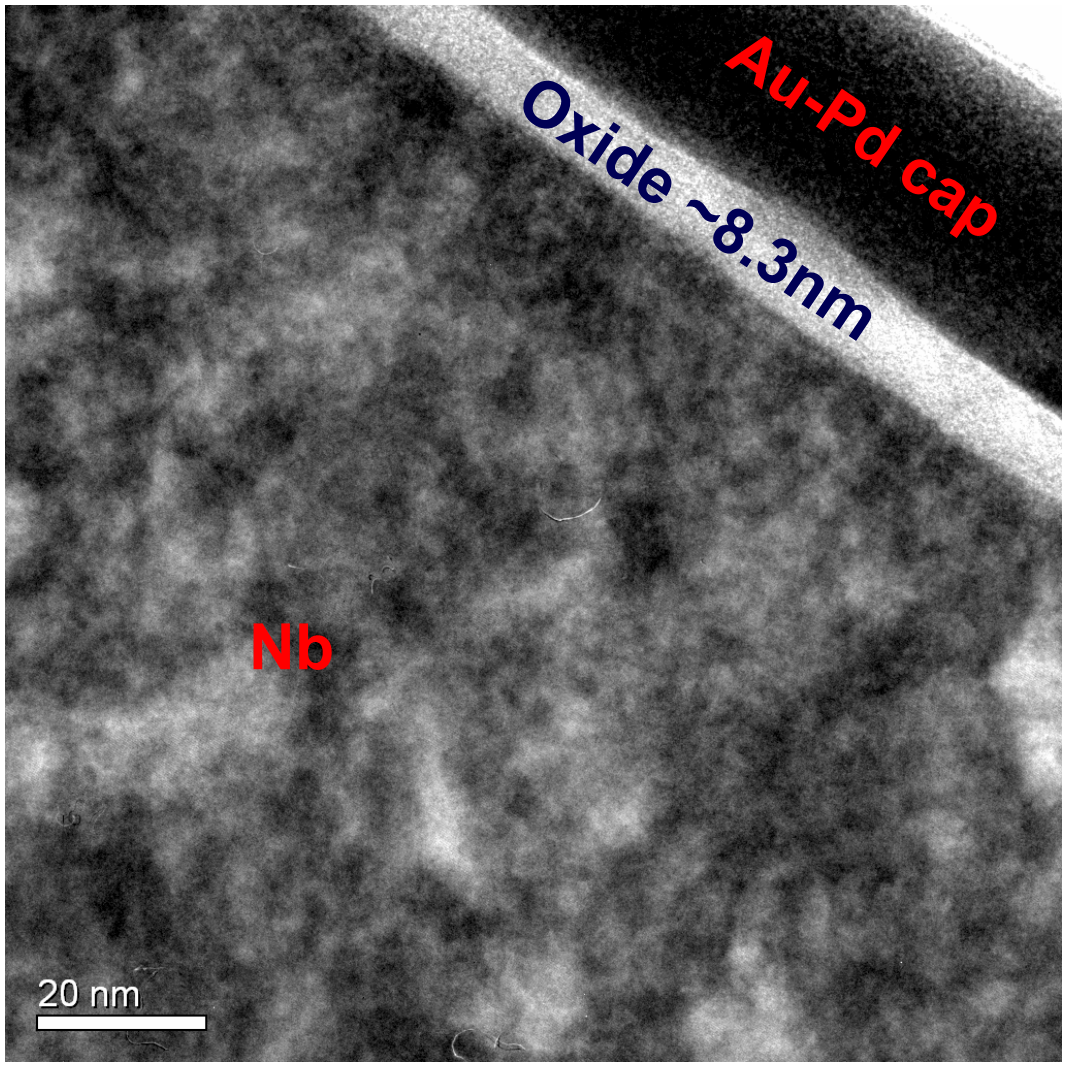
Nb (111)



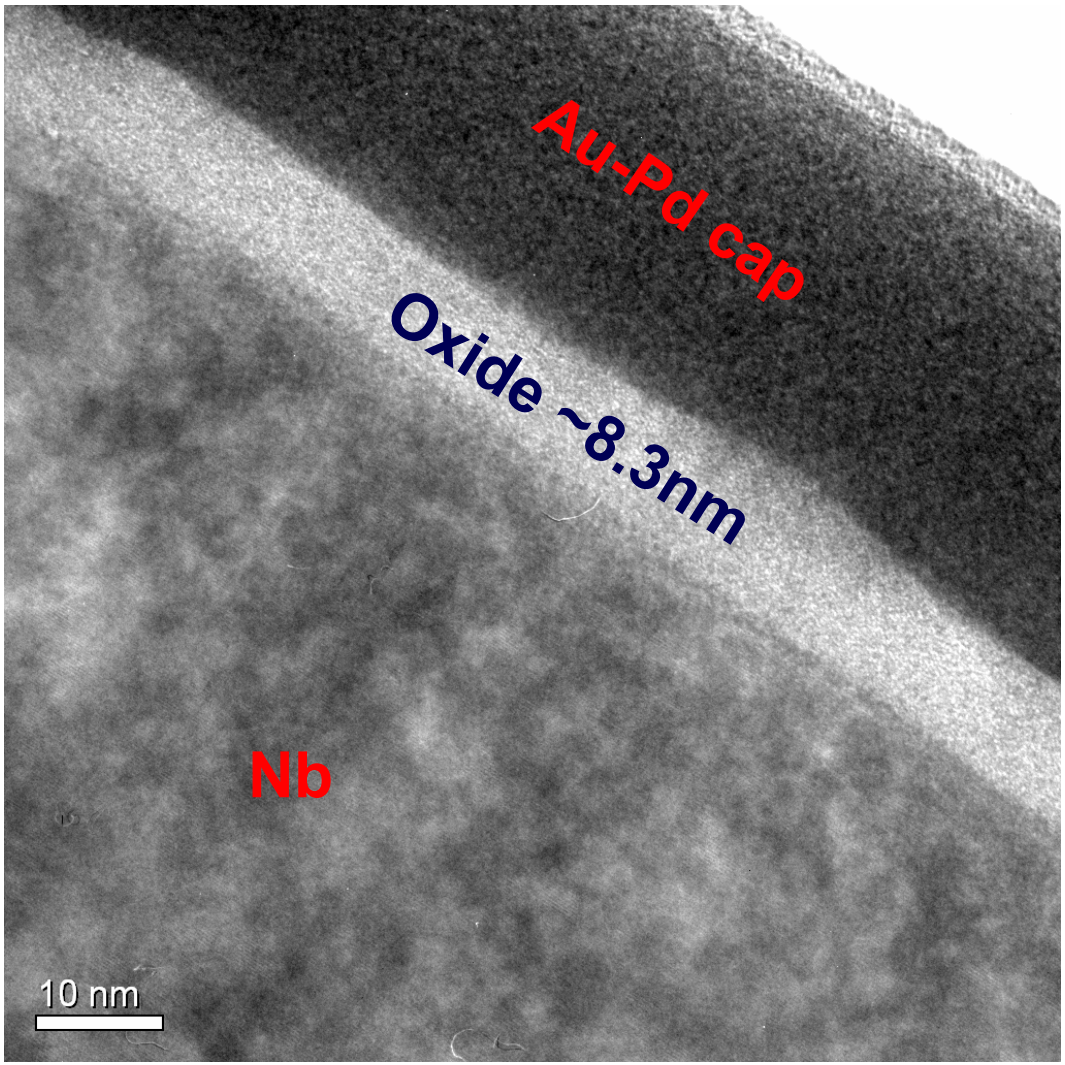
Nb (110)



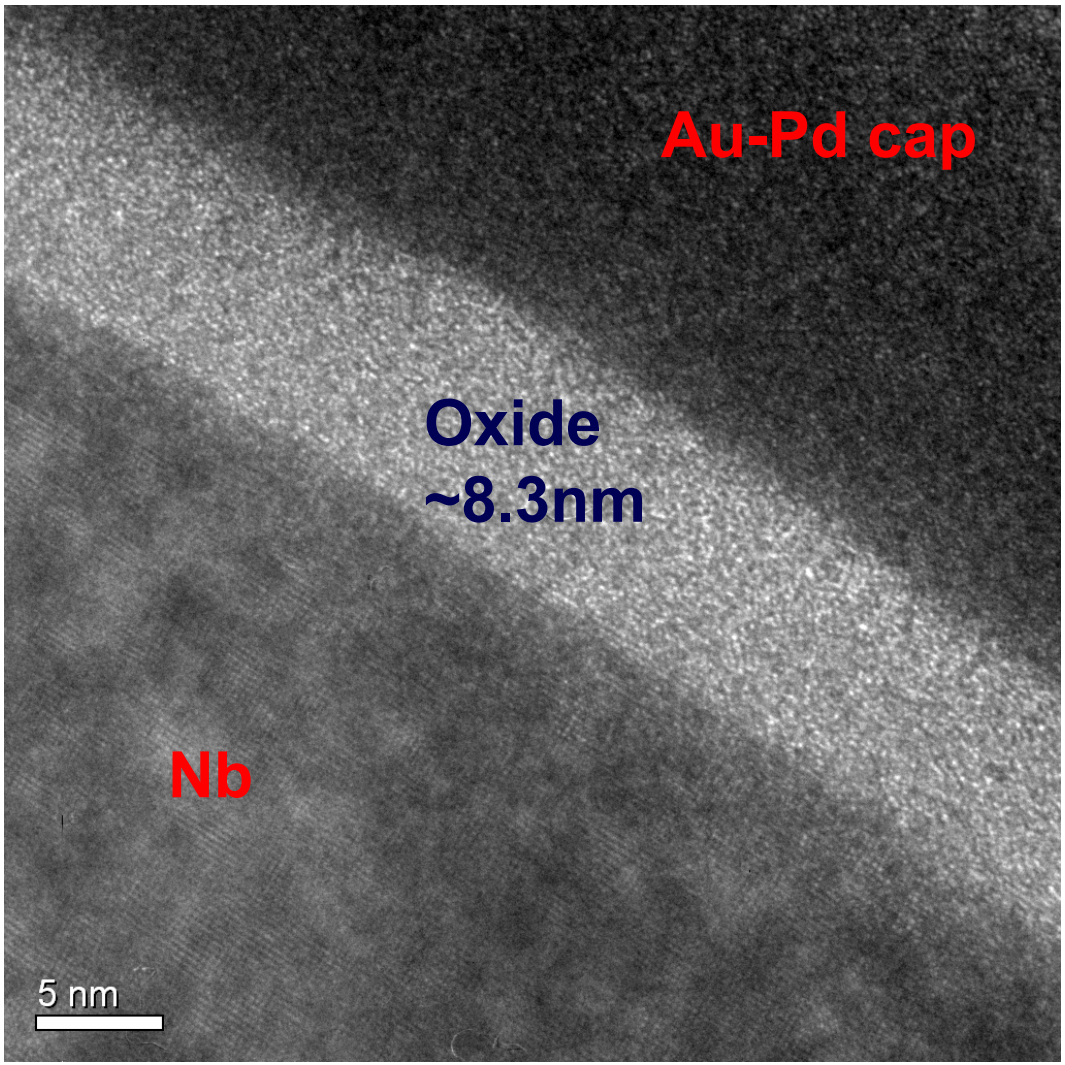
Nb (110)



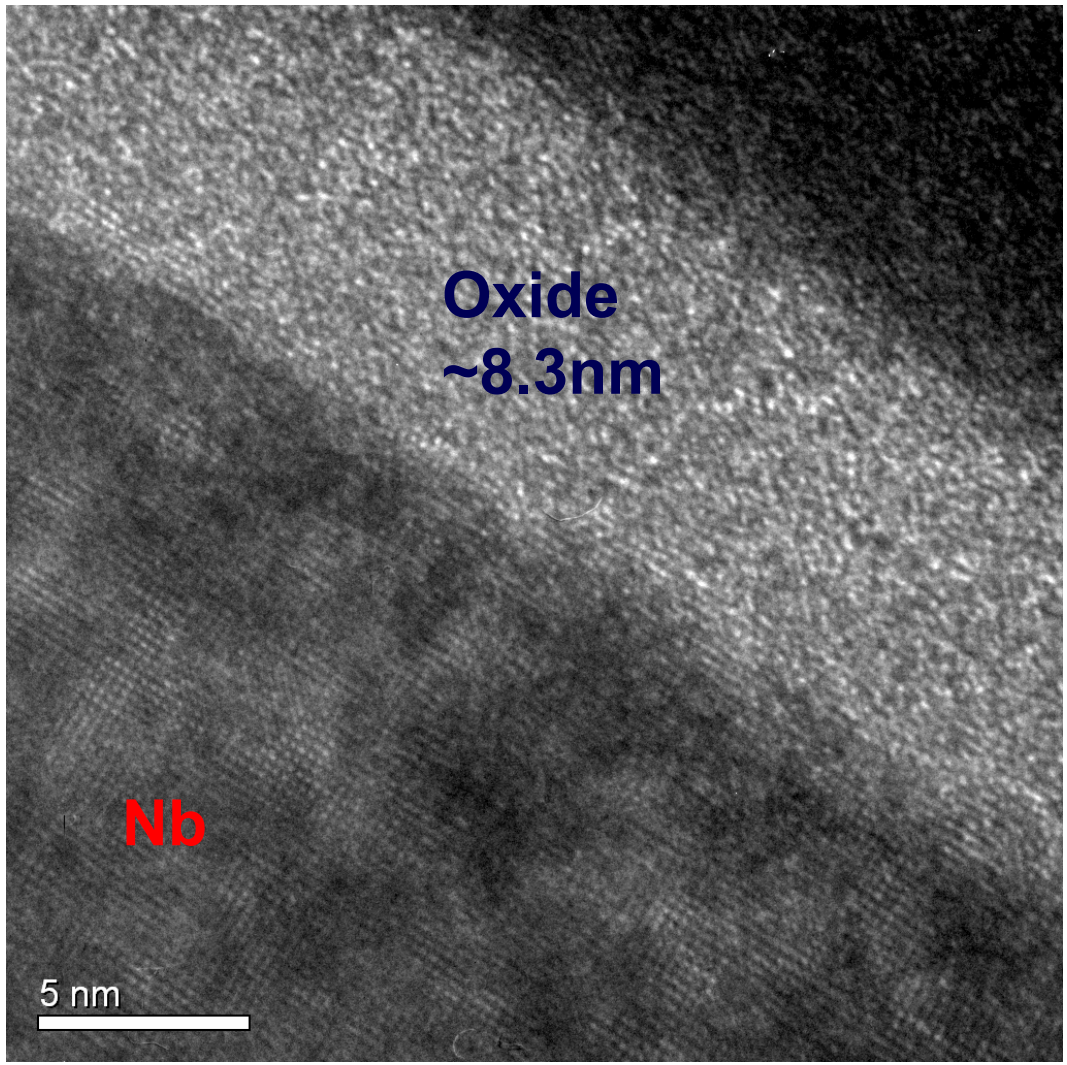
Nb (110)



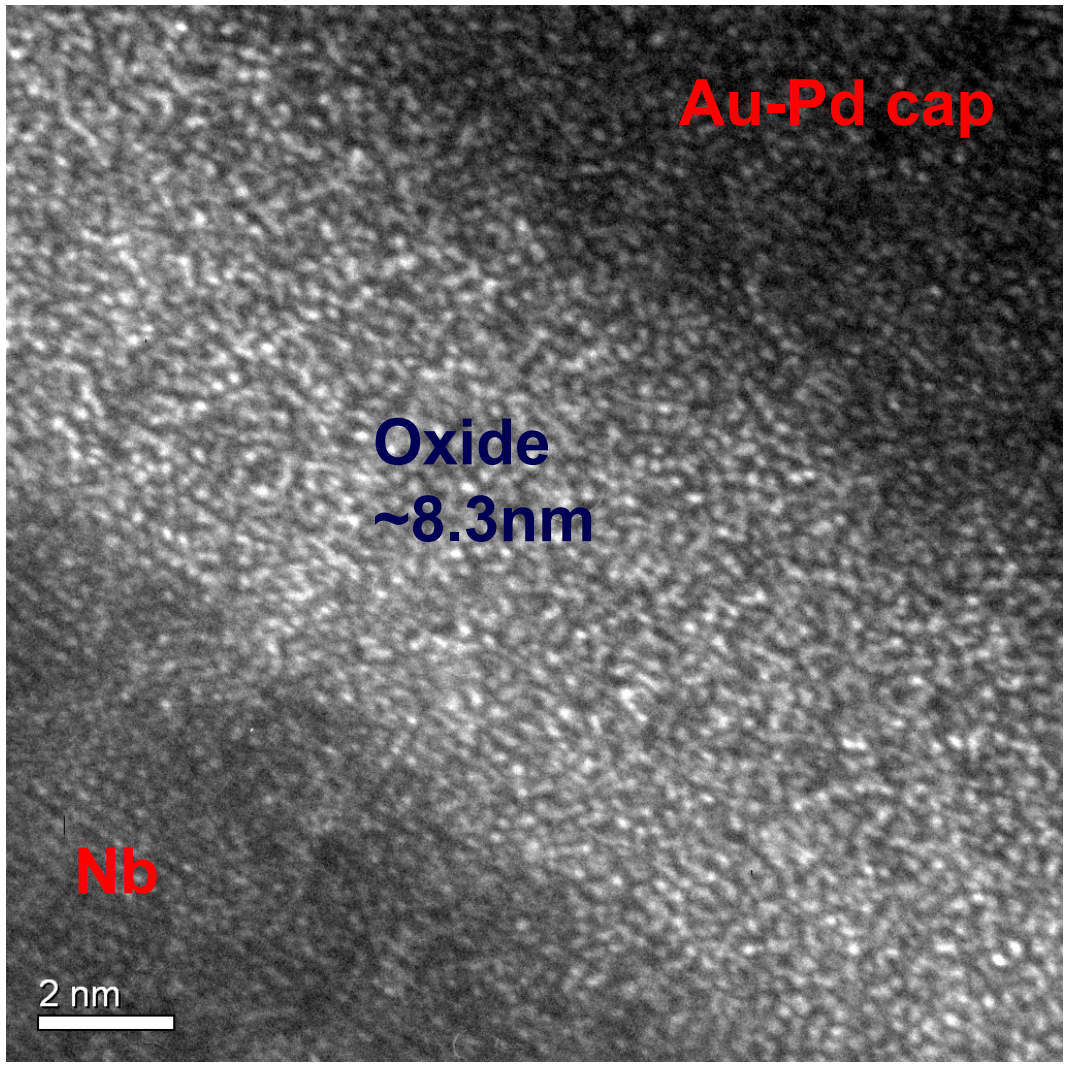
Nb (110)



Nb (110)



Nb (110)



TEM Summary

Oxide Thickness measurements:

Nb(100) ~4.9nm,

Nb(110) ~8.3nm,

Nb(111) ~7.5nm

Sub-oxides? More investigation needed

SIMS: Secondary Ion Mass Spectrometry

SIMS Analysis of
H, C, O
in single crystal Nb

- with 100, 110, 111 orientations
- before and after anneals

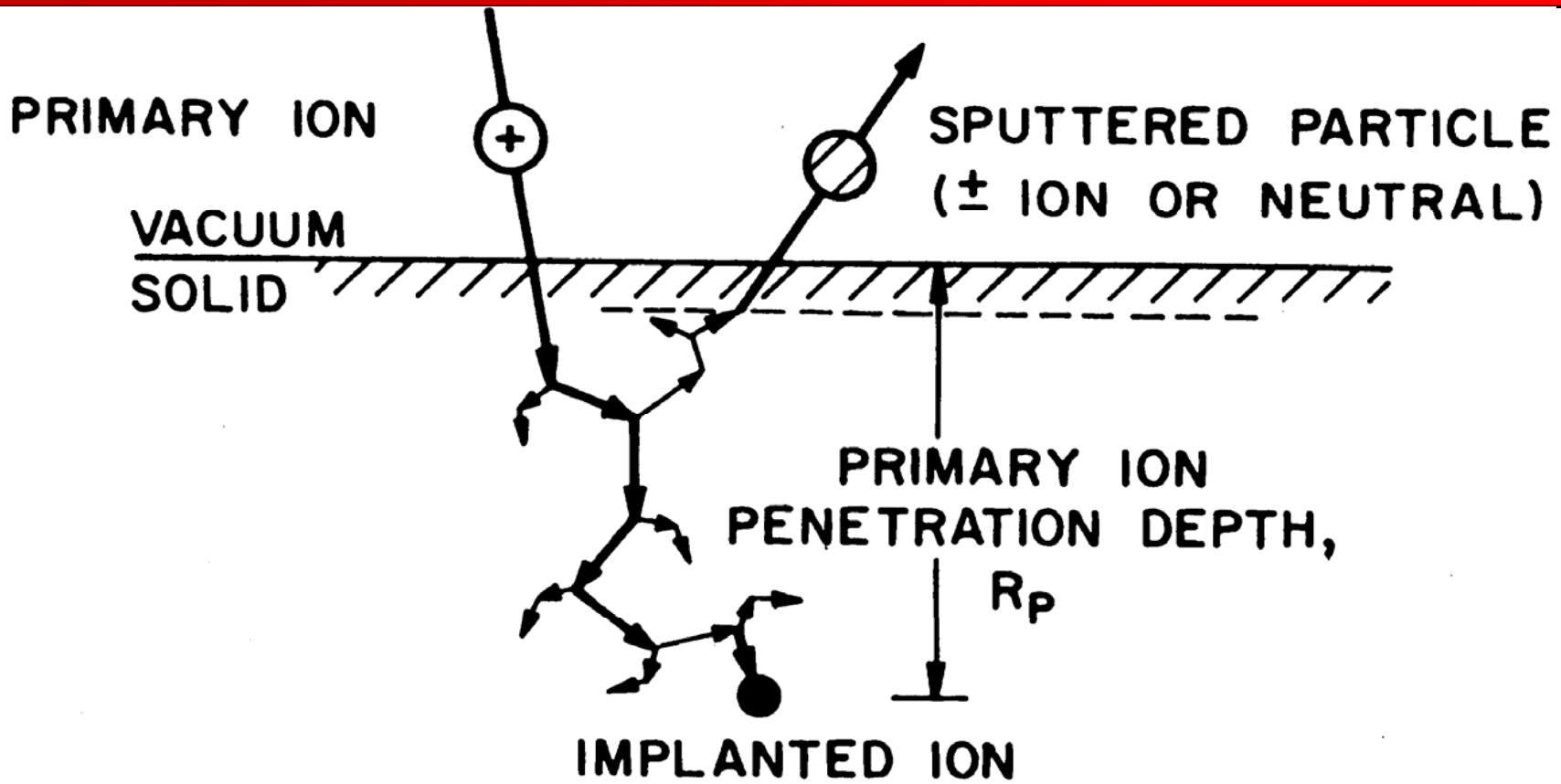
Fred Stevie
Dieter Griffis

Secondary Ion Mass Spectrometry (SIMS)

- Ion bombardment of surface
- Sputtered species analyzed
- Detection of mass resolved secondary ions
- Good sensitivity (ppm) and nm depth resolution

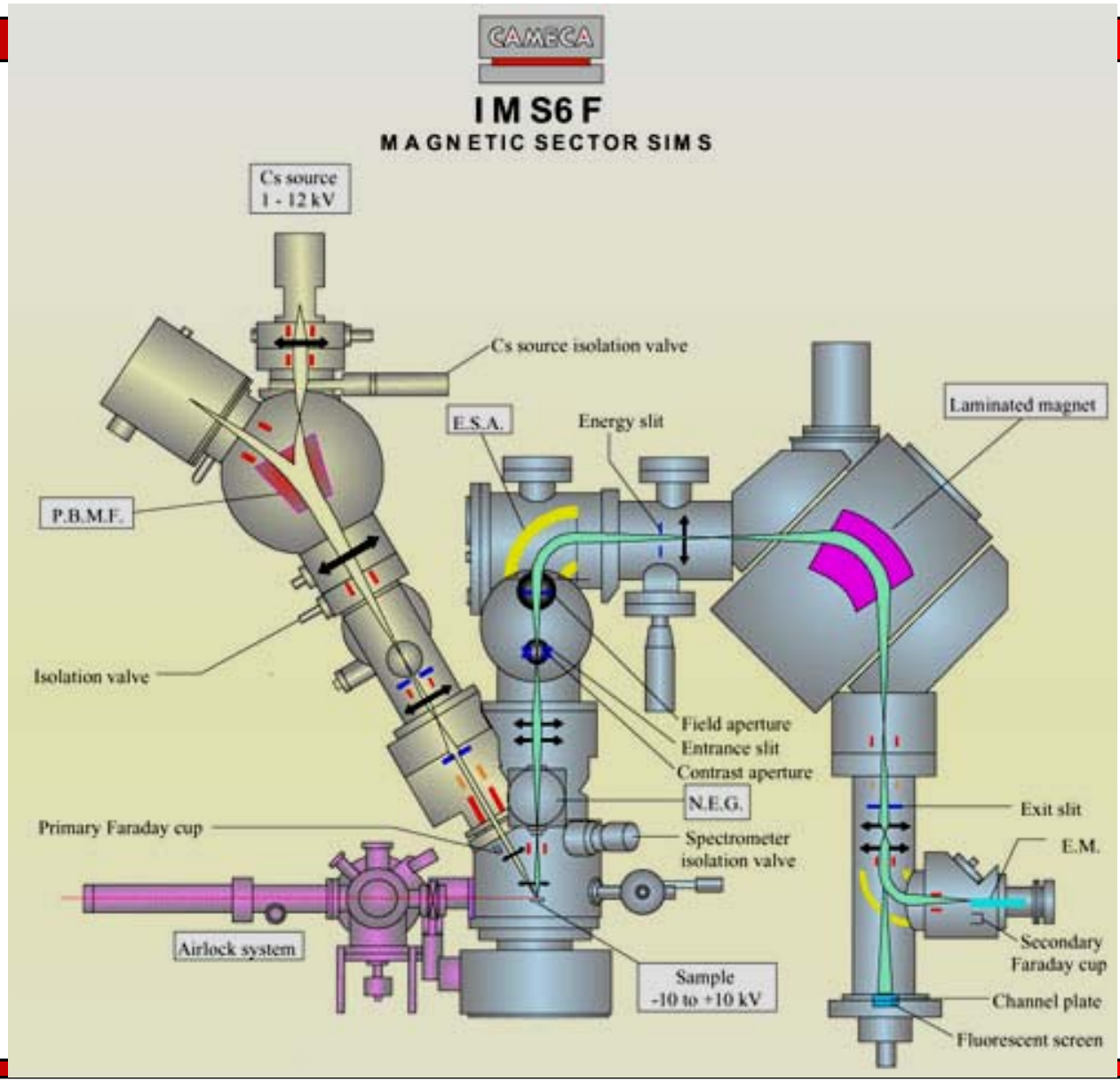
•Analyzed region is near surface only!

SIMS Process

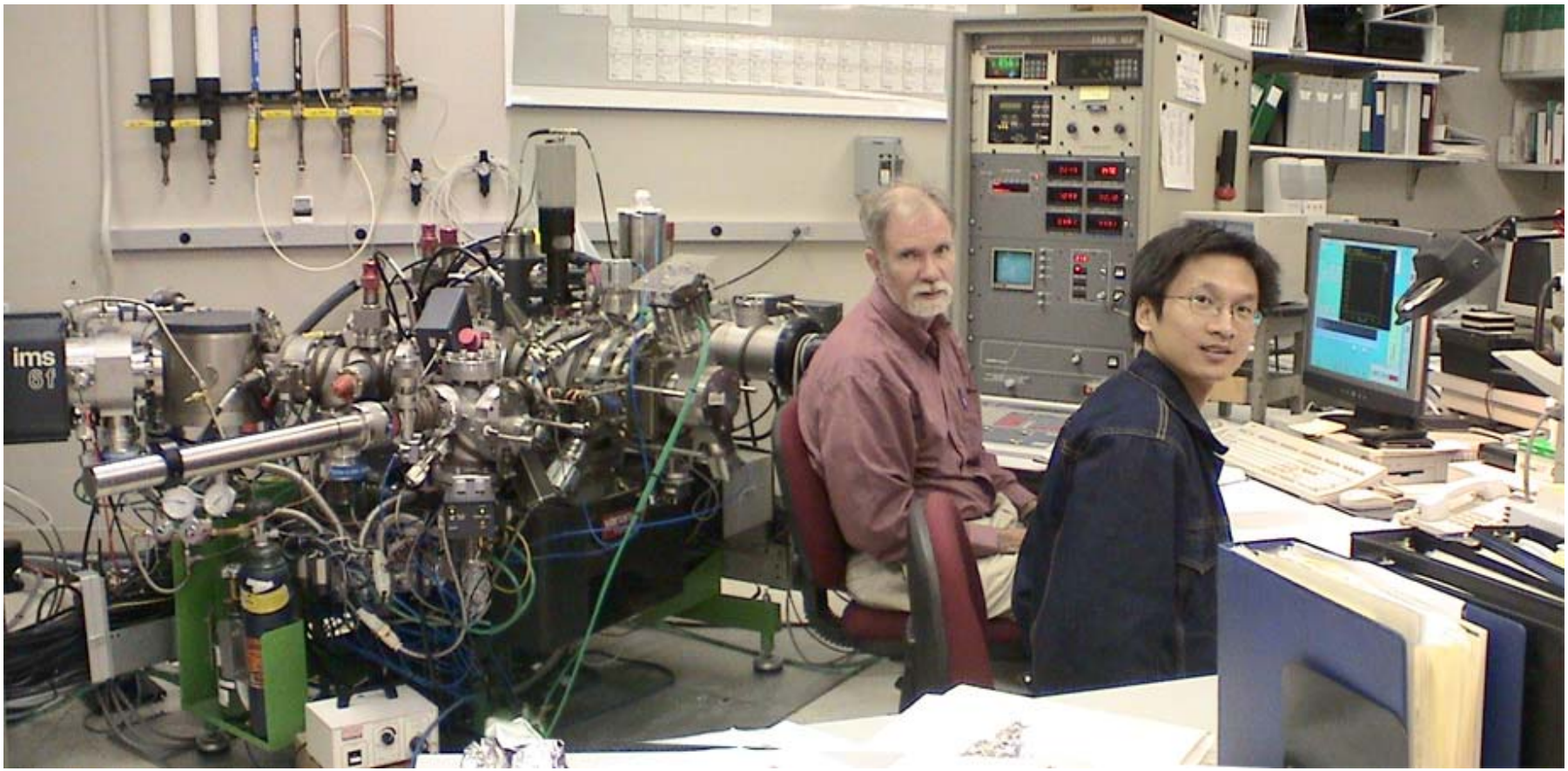


Primary ion penetrates surface, energy lost through collision cascade, primary ion implanted into solid, secondary particles (including ions) leave surface at low energy

CAMECA IMS-6F SIMS Instrument



AIF CAMECA IMS-6F SIMS Facility



SIMS Analysis Conditions

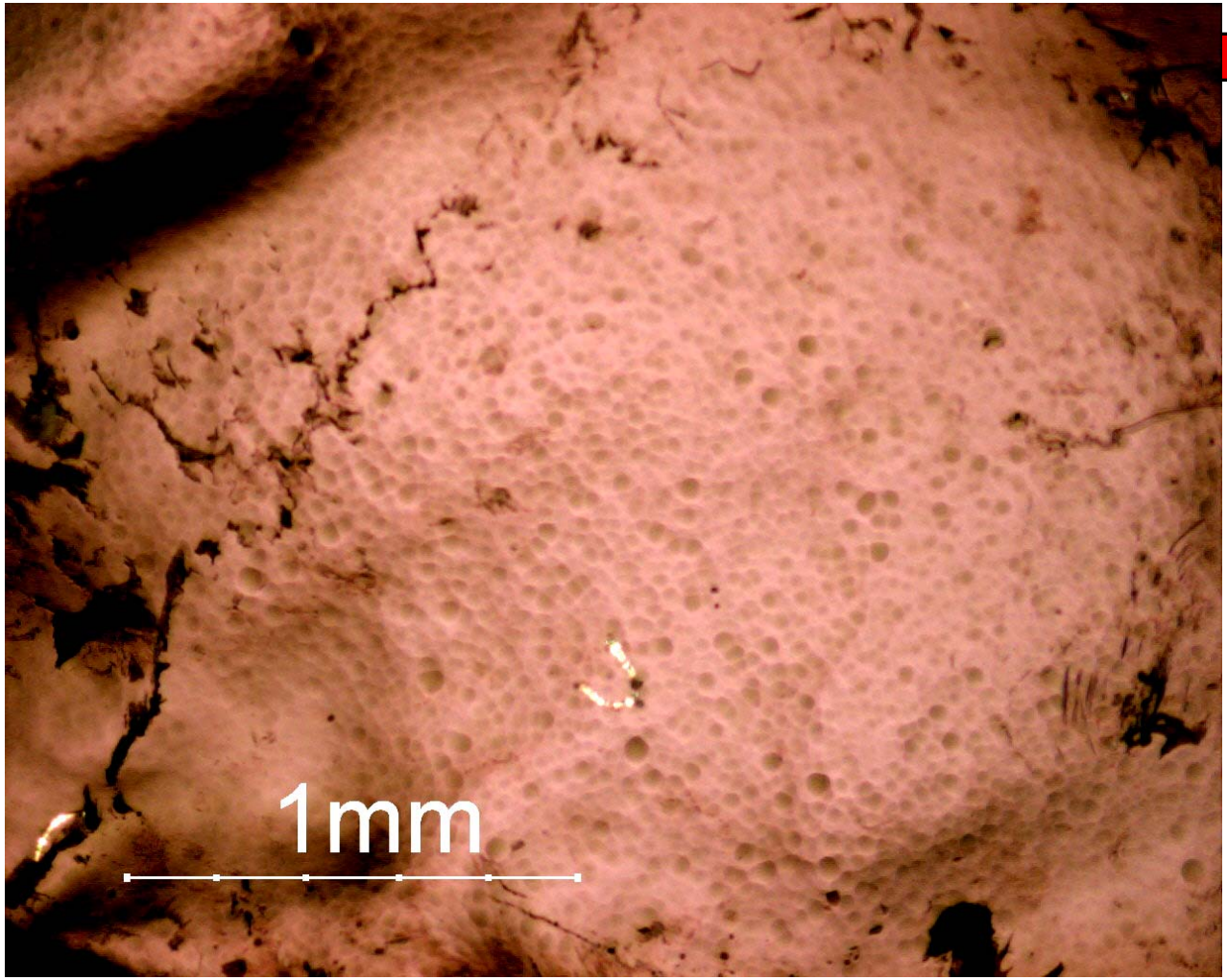
- CAMECA IMS-6F magnetic sector instrument
- Cs^+ primary beam 6keV impact energy, 3nA
- Incidence angle 24º from normal
- 160µm x 160µm raster
- 30µm diameter detected area
- Mass resolution~500 (M/ΔM)
- Samples pumped to ~$1x10^{-9}$ Torr before analysis
- Chamber pressure $2x10^{-10}$ Torr during analysis
- At least two sites measured per sample

Specimen Considerations

Samples submitted for analysis by Ganapati

- **Samples are 100, 110, 111 Nb from MaTecK**
  - **Float zone?**
- **Nb samples have a rough surface as received**
- **Roughness varies with sample**
- **Polycrystalline metals do not sputter uniformly but single crystal metals should**

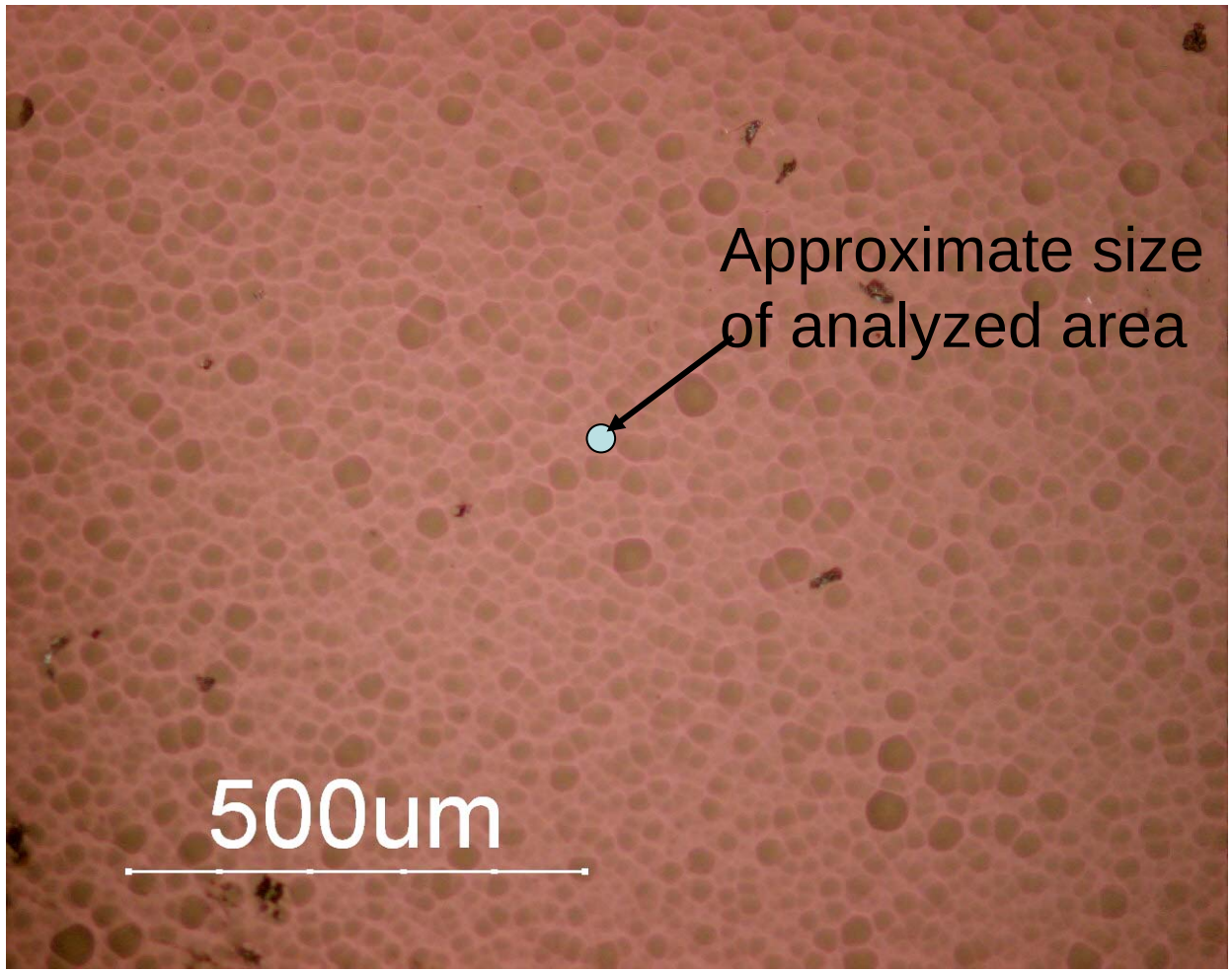
Reflected light optical image



(100) as received

Profilometer roughness measurement:
Peak-valley 0.6μm, RMS 0.13μ

100 after 600C anneal (optical)

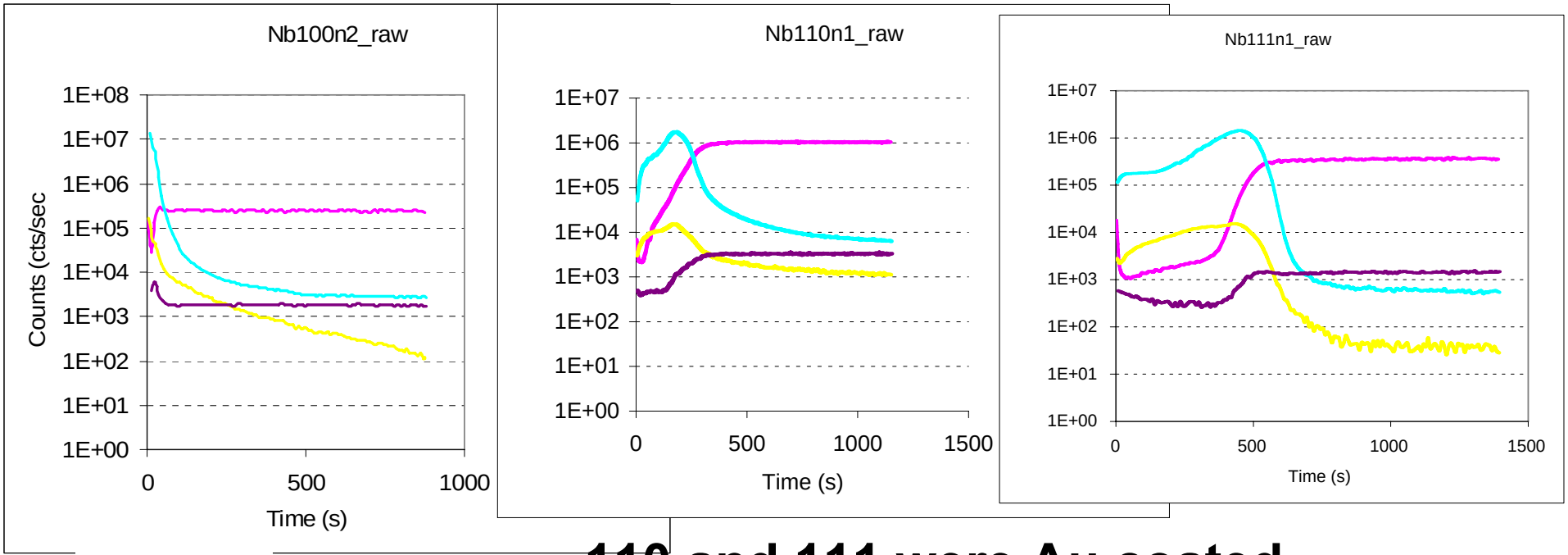


Peak-valley 1.0µm RMS 0.1µm

Non-Annealed

100 110 111

QuickTime™ and a TIFF (LZW) decompressor are needed to see this picture.



QuickTime™ and a TIFF (LZW) decompressor are needed to see this picture.

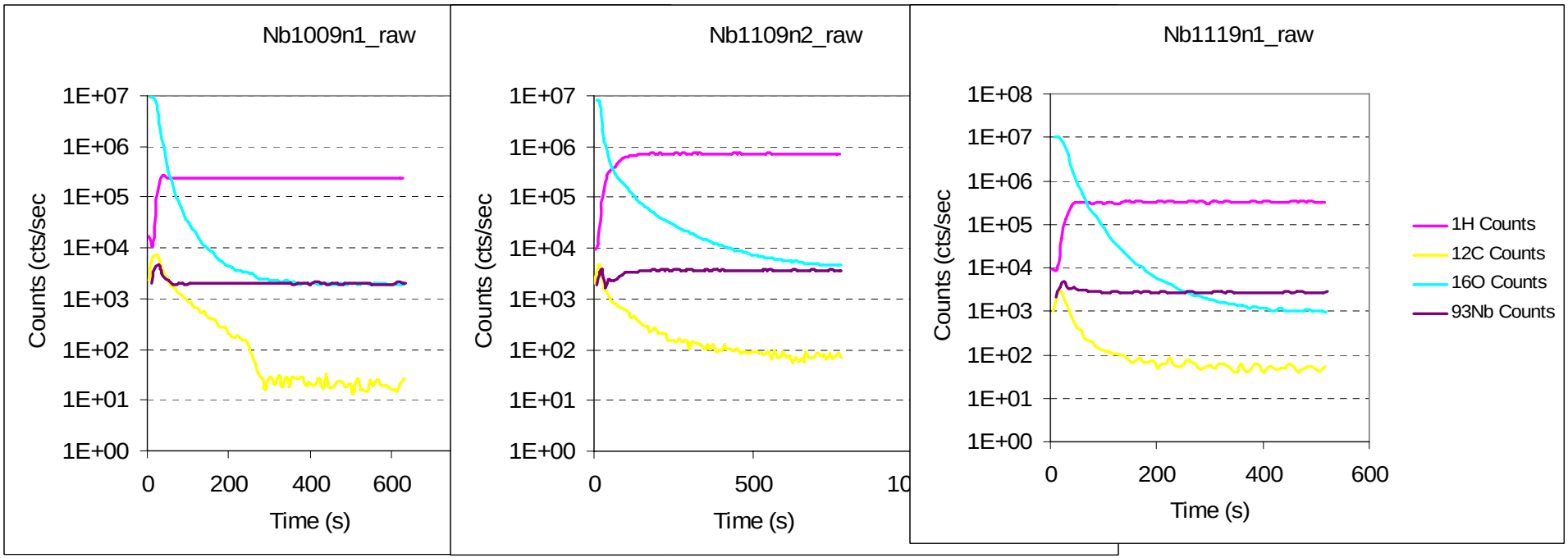
110 and 111 were Au coated as part of preparation for TEM analysis

Sputter rate ~0.5A/sec for analyses shown: 1000sec~50nm

After 90C Anneal

100 **110** **111**

(110 and 111 were Au coated)

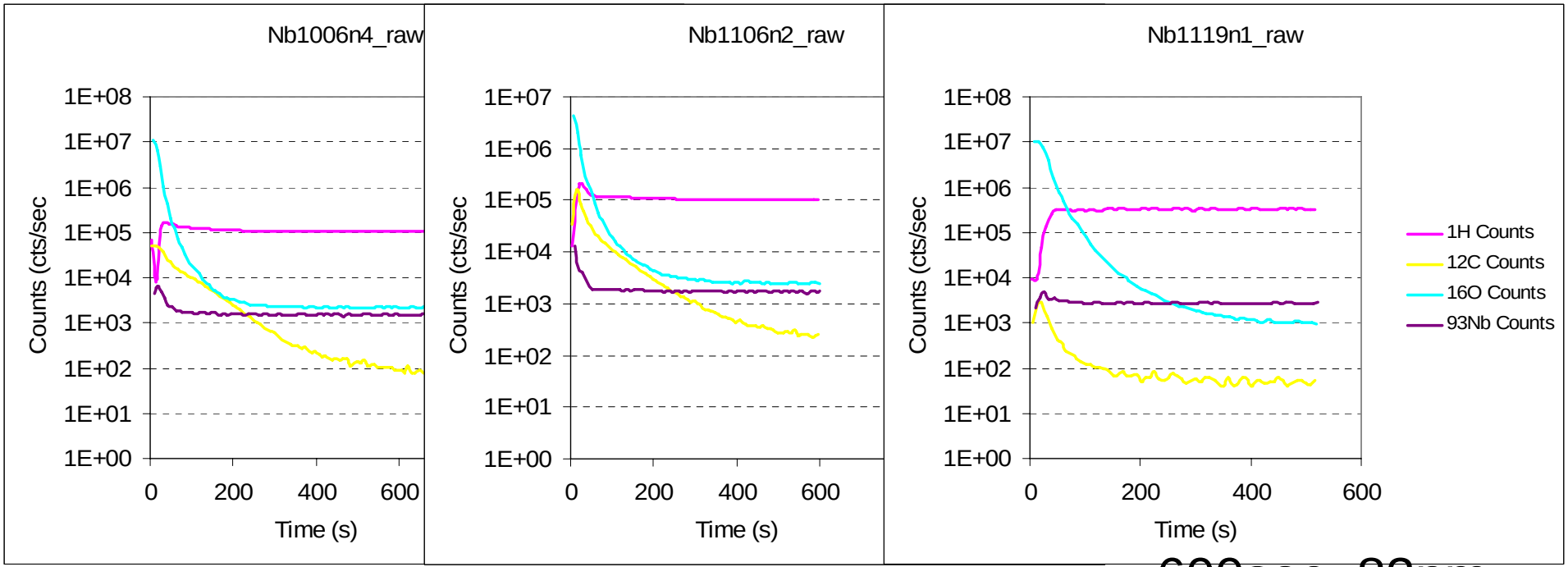


Note H in 100 oxide

600sec~83nm

After 600C Anneal

100 110 111

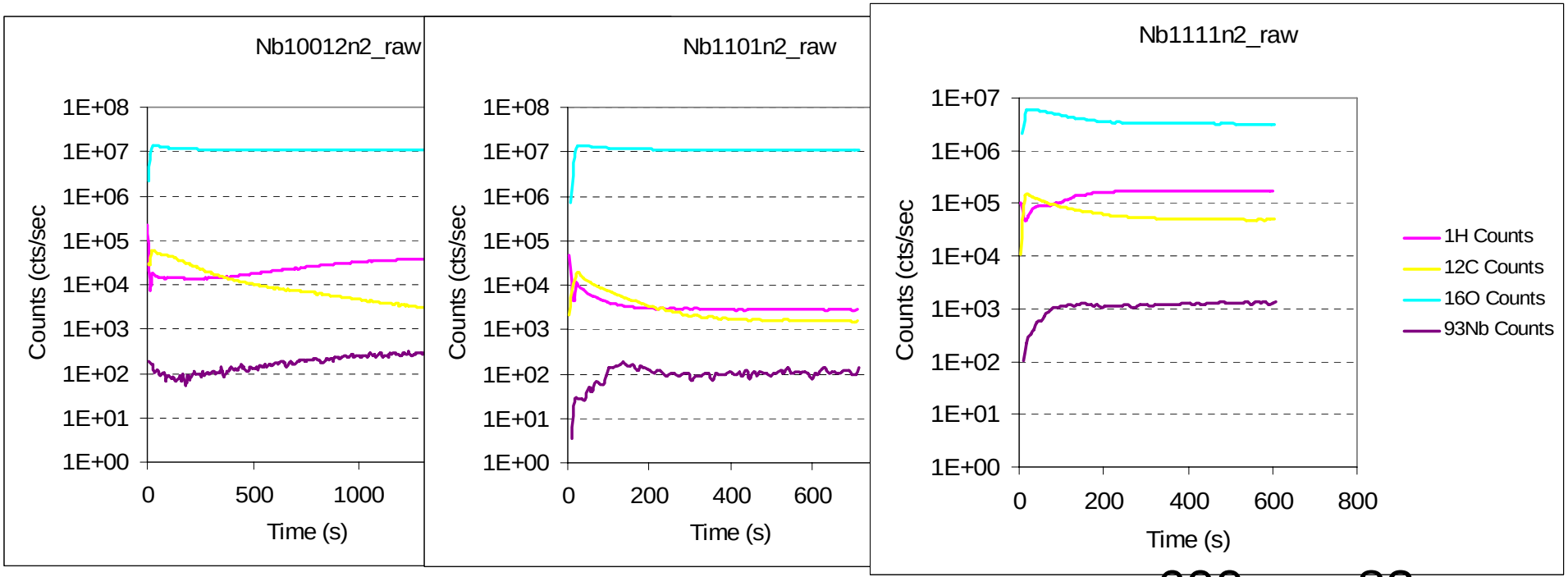


600sec~83nm

(110 and 111 were Au coated)

After 1250C Anneal

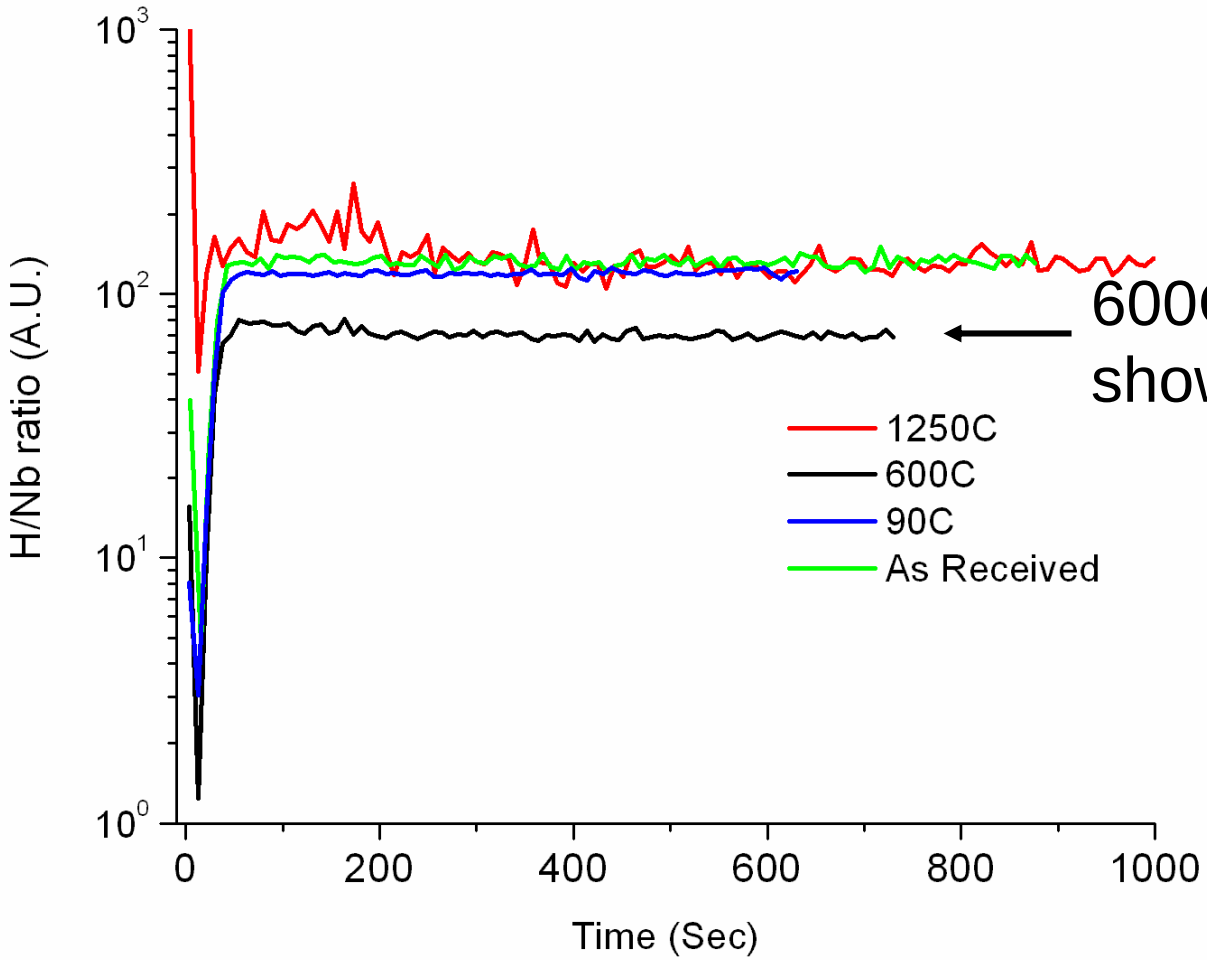
100 110 111



600sec~83nm

100 Nb sample:
after 700nm, still in oxide

100 Nb H/Nb Ratio



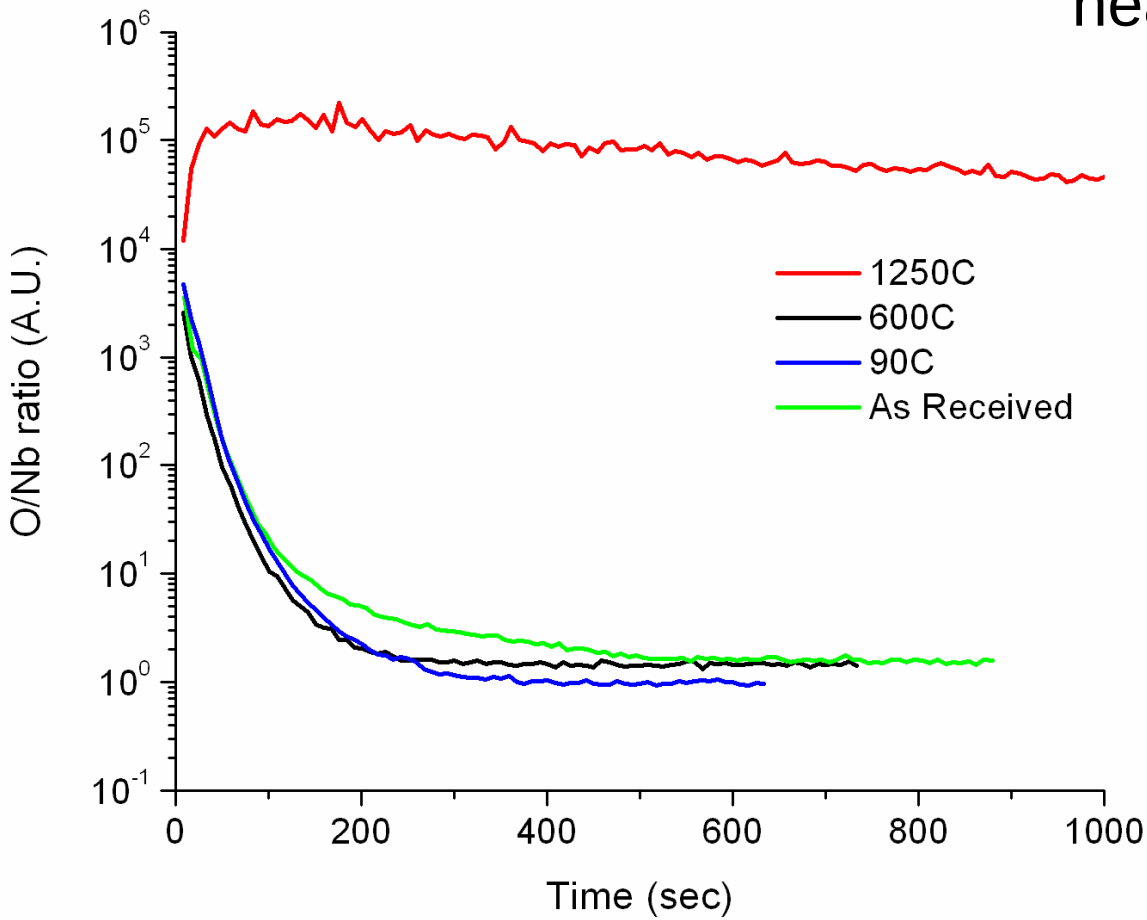
600C annealed sample shows significantly less H

1000sec~50nm

1250C sample is heavily oxidized (this is a measurement in oxide)

100 Nb O/Nb Ratio

1250 C annealed is heavily oxidized



SIMS Results

- H, C, O detected and oxide present on all samples
- H after 90C anneal similar to un-annealed
  - H higher in 100 oxide
- H after 600C anneal lower than un-annealed
  - H out diffuses
- After 1250C anneal, samples heavily oxidized
- No evidence of high oxygen content region below oxide
- C high at surface and then decreases for all samples

SIMS Summary and Future Work

- H, C, O can be analyzed in single crystal Nb
- Surface roughness limits depth resolution
- Some differences between crystal orientations
- 1250ºC annealed samples heavily oxidized

- Need surface treatment to reduce surface roughness
- Need implant standards to quantify results

TEM Summary

Oxide Thickness measurements:

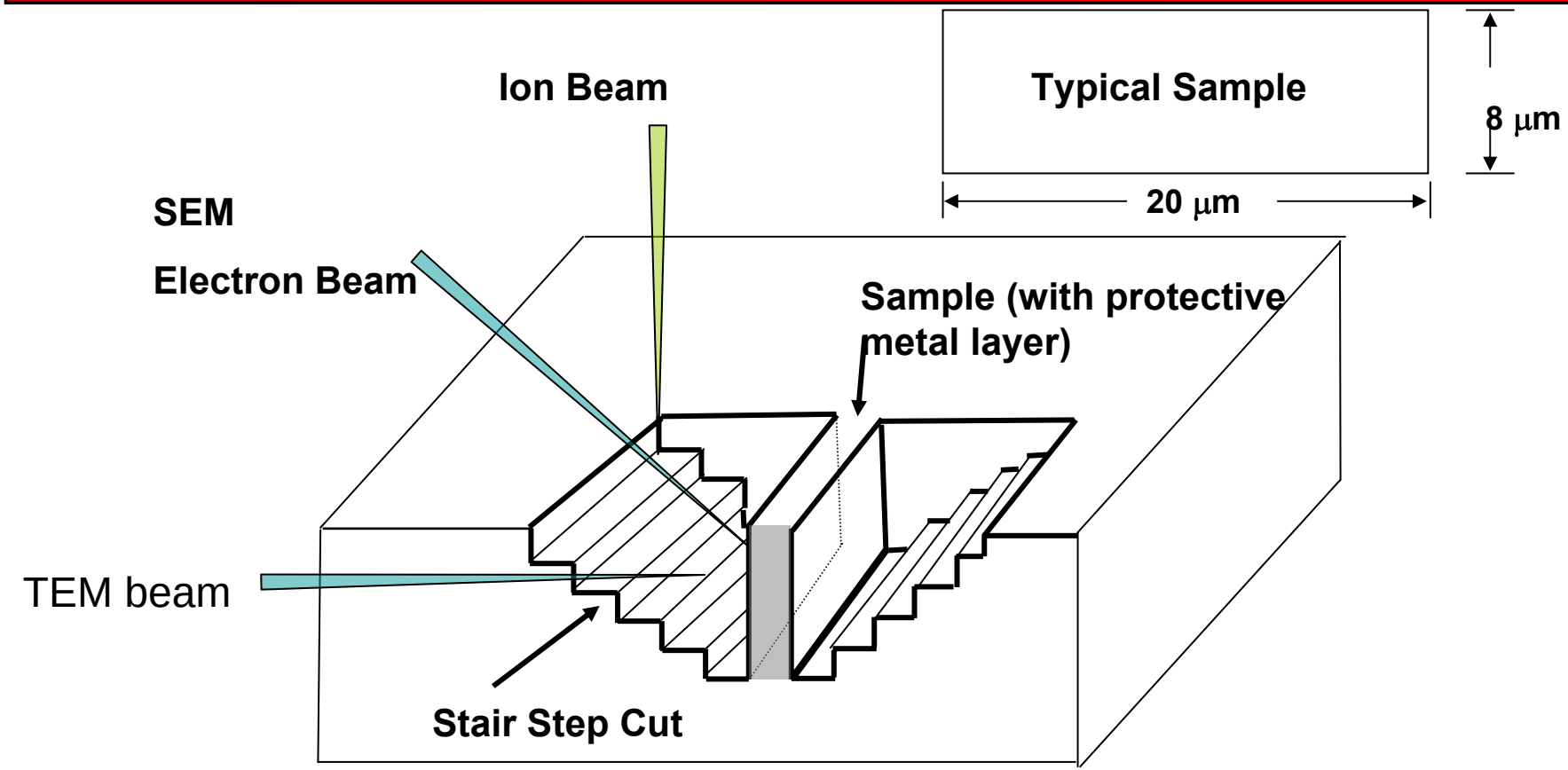
Nb(100) ~4.9nm,

Nb(110) ~8.3nm,

Nb(111) ~7.5nm

Sub-oxides? More investigation needed

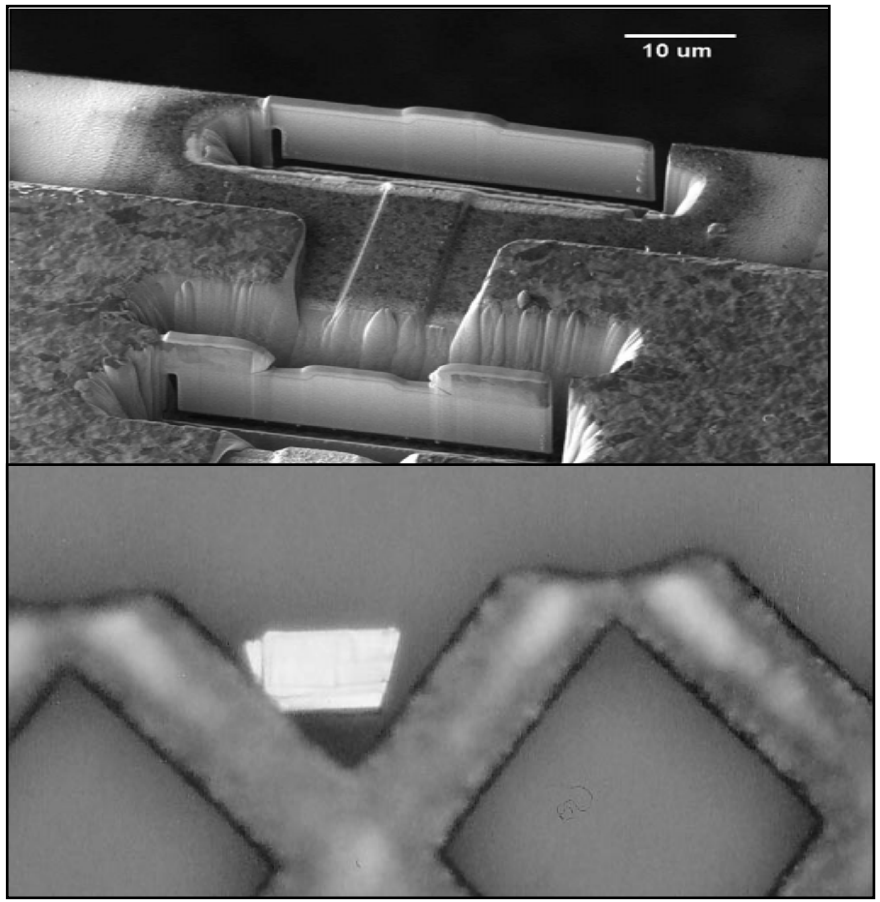
FIB Micromachining to Produce TEM Cross Sections



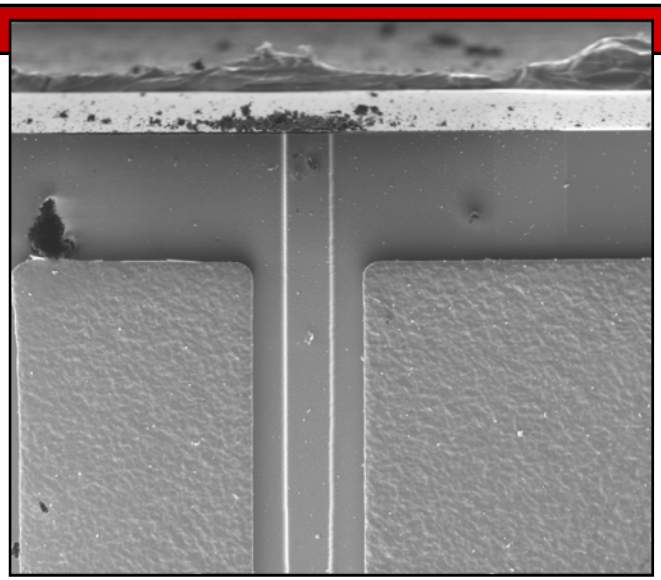
Called lift-out sample as final sample must be lifted out of the trench and mounted on a TEM grid. (or all material removed from TEM beam path)

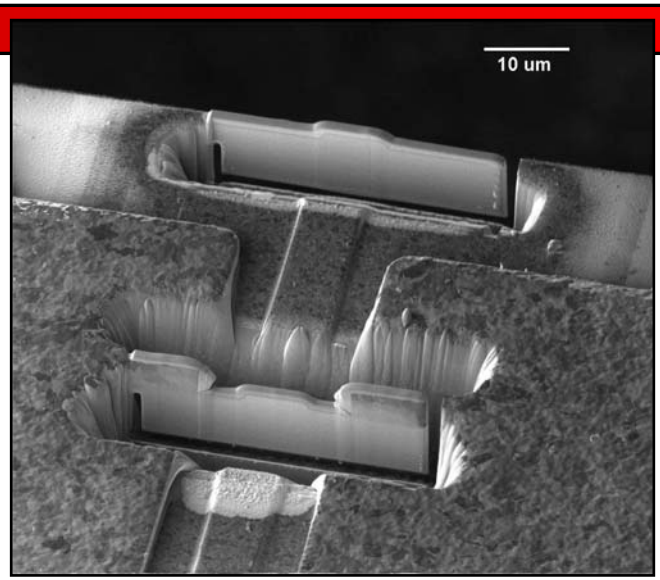
Lift-out methods are my favorite

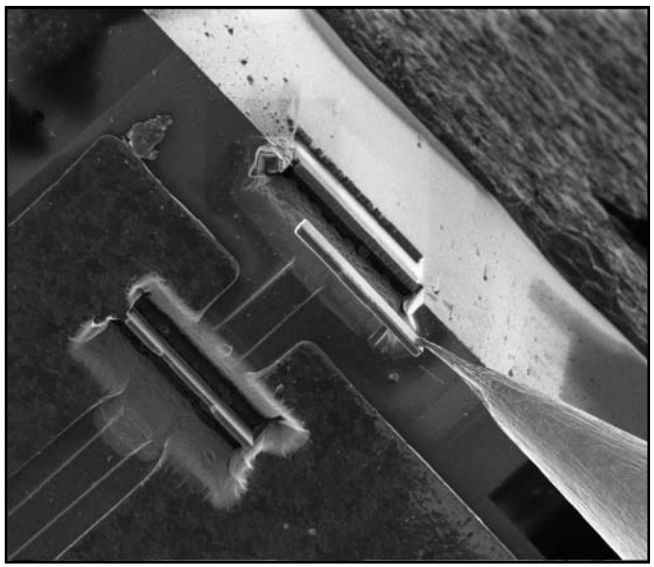
- Foil(s) cut out from sample in FIB
- Foil(s) attached to TEM grid
  - Outside FIB (external)
  - Inside FIB (internal)
    - ***USING FIB INDUCED DEPOSITION***



Locate areas, FIB thin and notch, attach needle, lift out and transfer

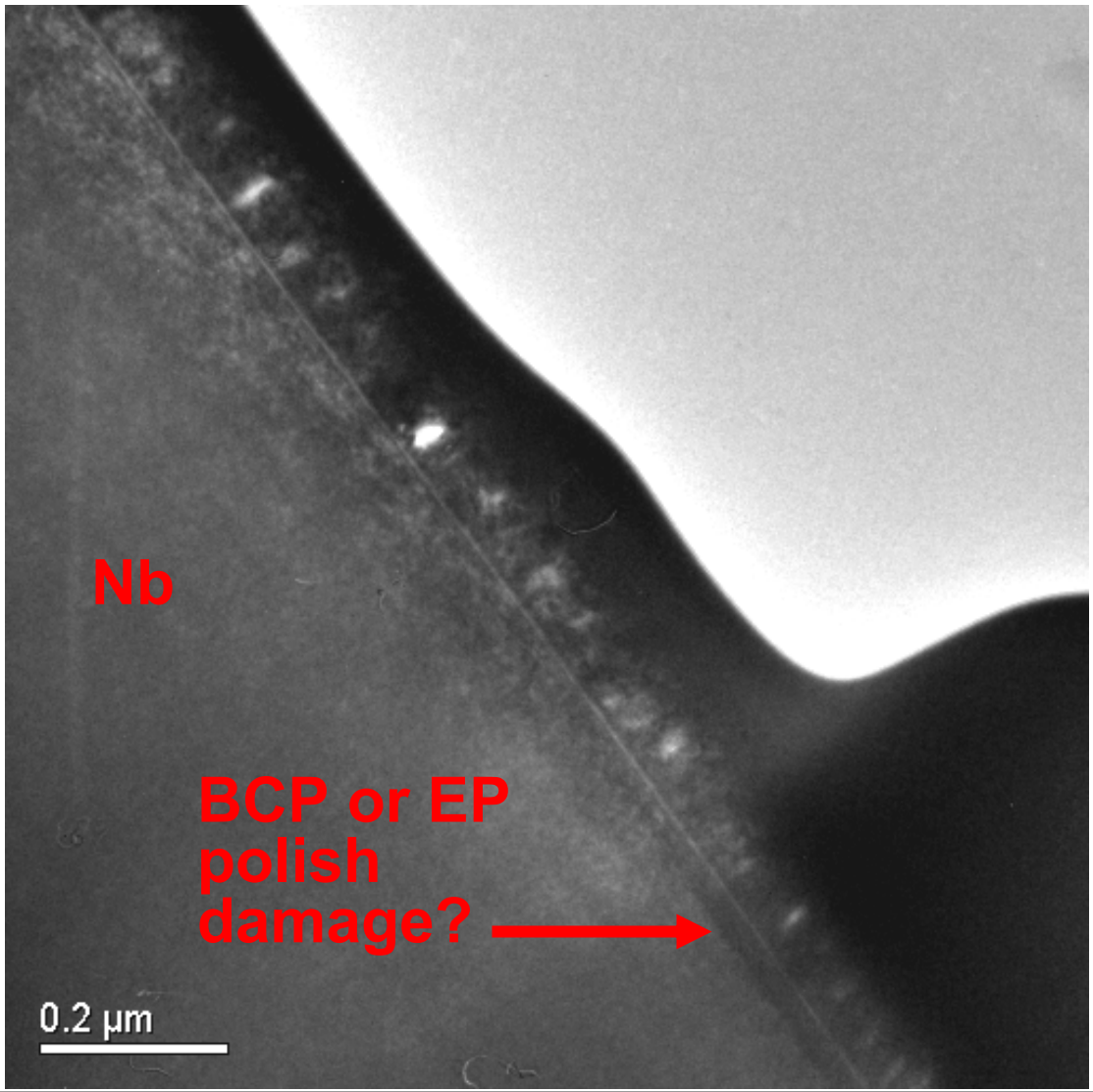




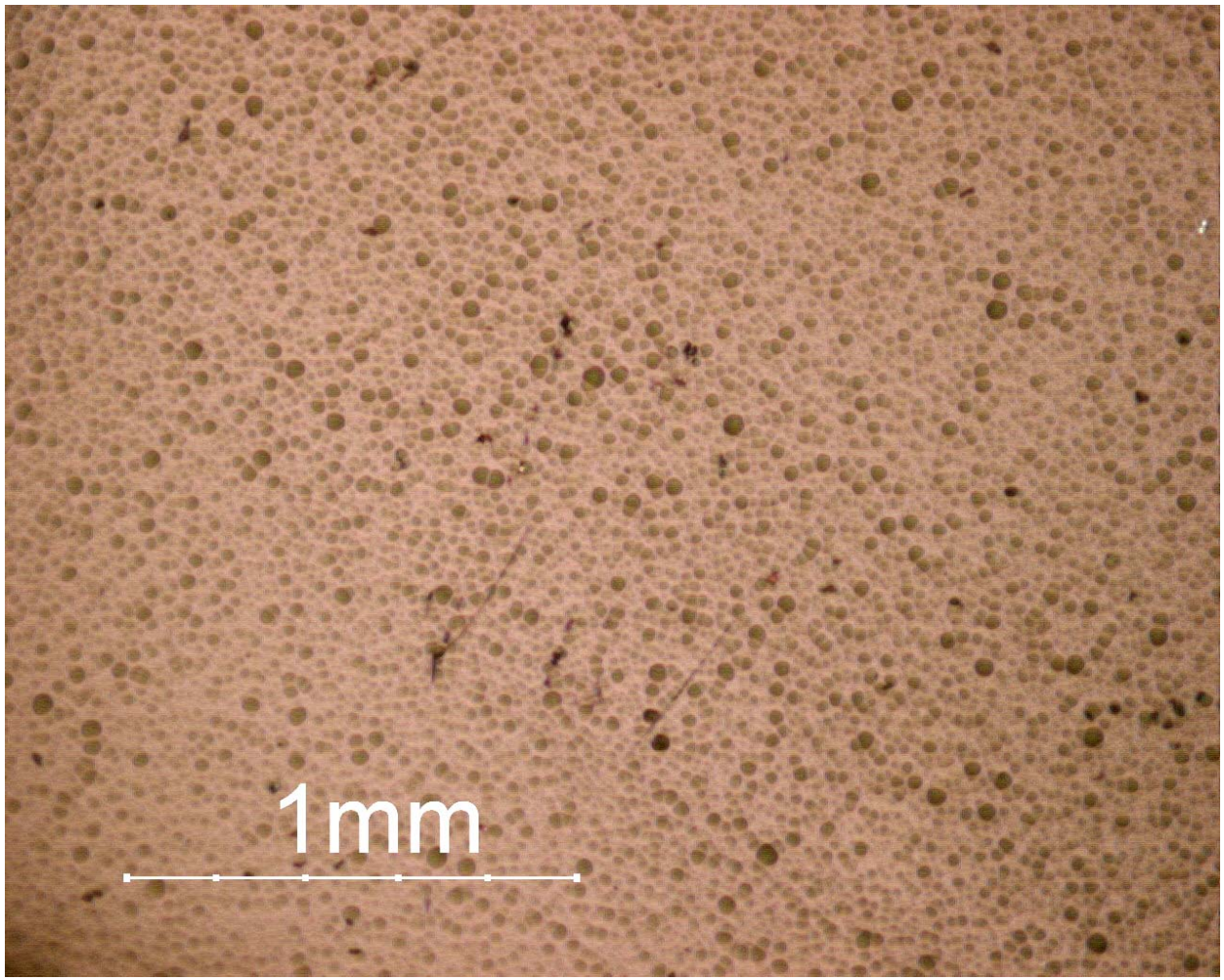


Why stop we only one or two samples?

Nb (100)



(100) after 600C anneal



Peak-valley 1.0µm RMS 0.1µm