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Thomas Jefferson National Accelerator Facility

Some On-going R&D Activities on Niobium at Jefferson Lab

Andy T. Wu

Thomas Jefferson National Accelerator Facility, 12000 Jefferson Avenue, Newport News, VA23606
USA

Symposium on Surface Science 2007   Las Arcs 2000

CEA Saclay, France, Mar.19, 2007

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Content of the Talk

- Very brief introduction to JLab accelerator systems: CEBAF & FEL
- Nb SRF cavities   Why surface is critical
- Some challenges that SRF community (surface related) is facing
- Surface Science Lab at JLab
- Some surface R&D activities on Nb at JLab
- Summary

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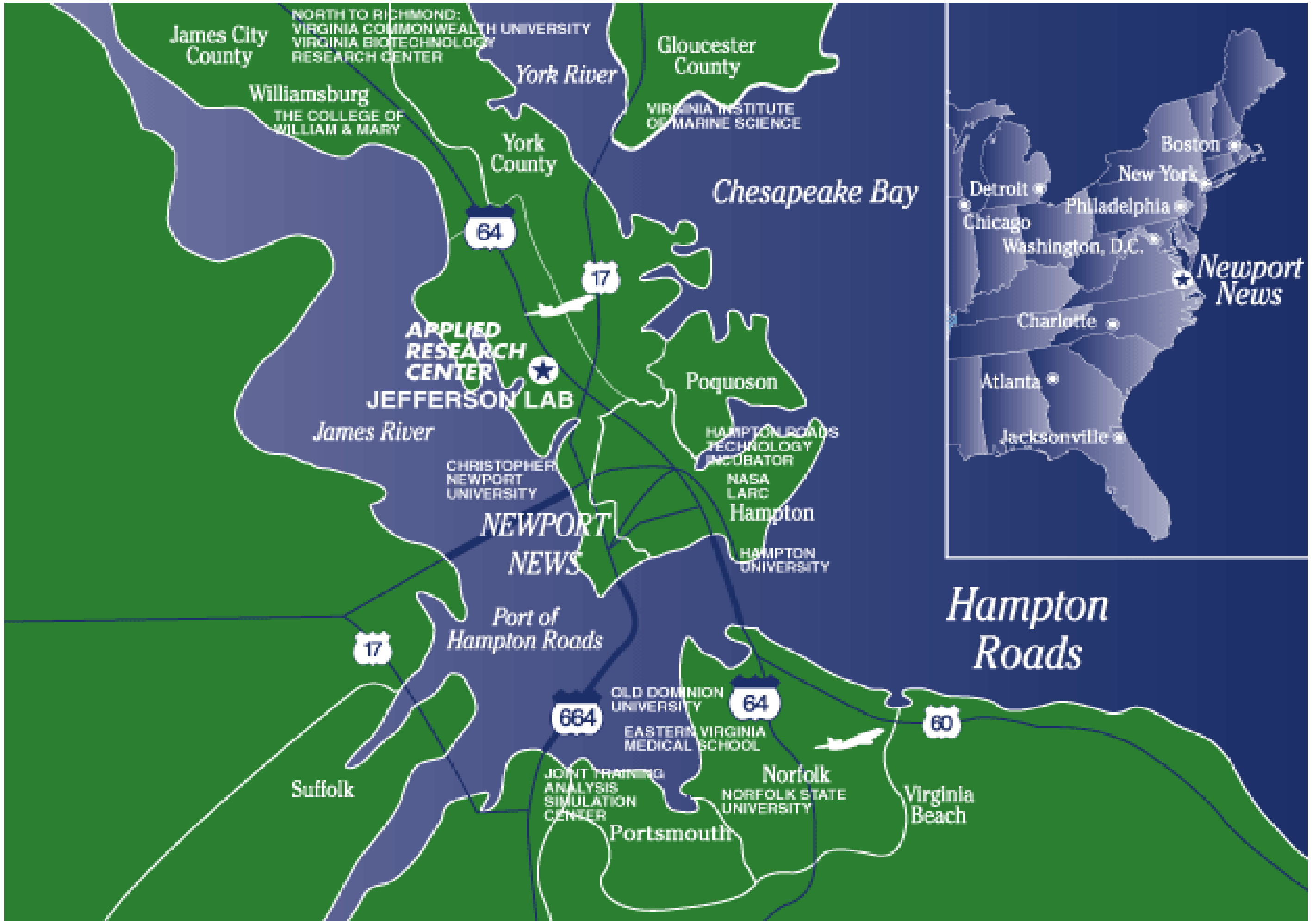

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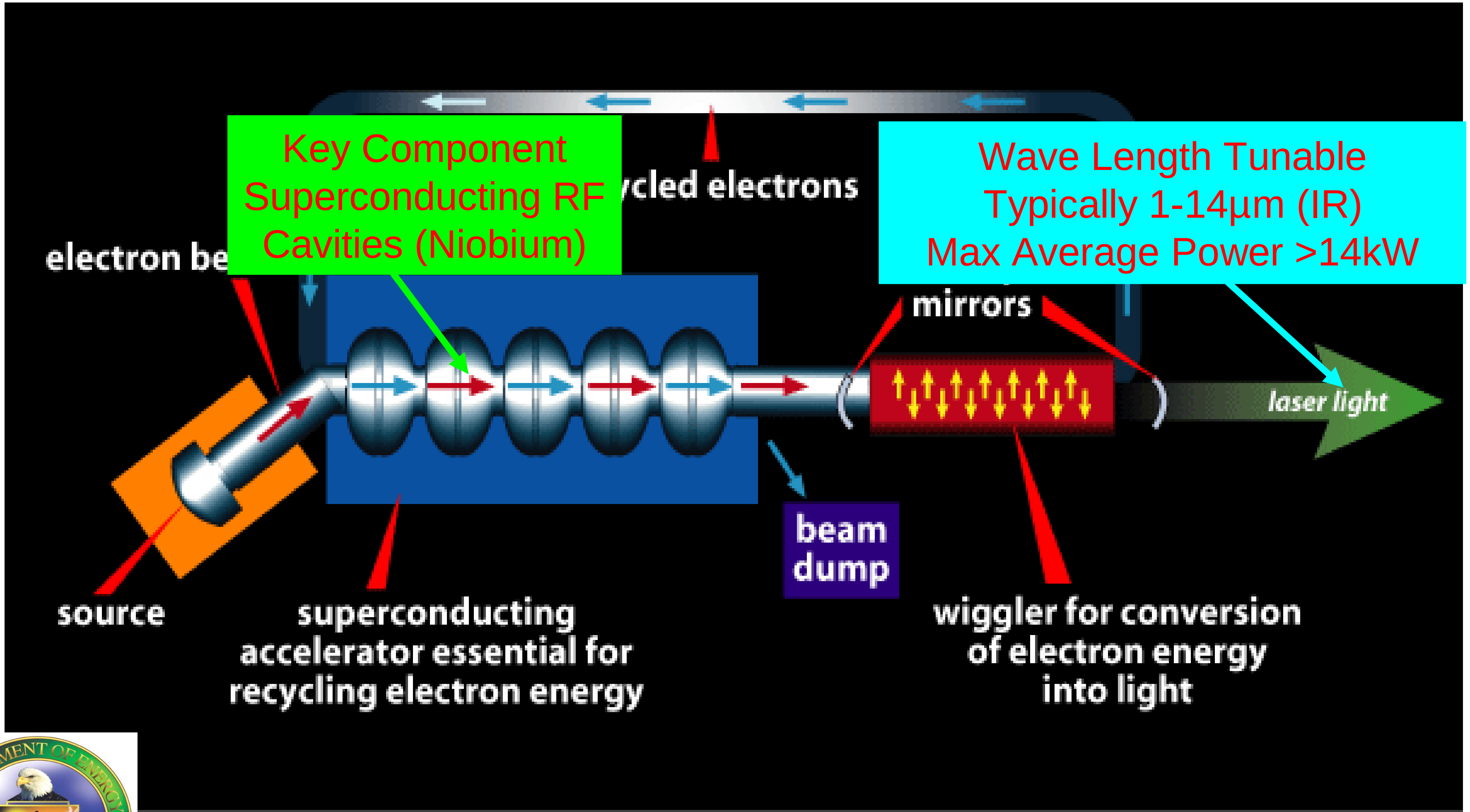
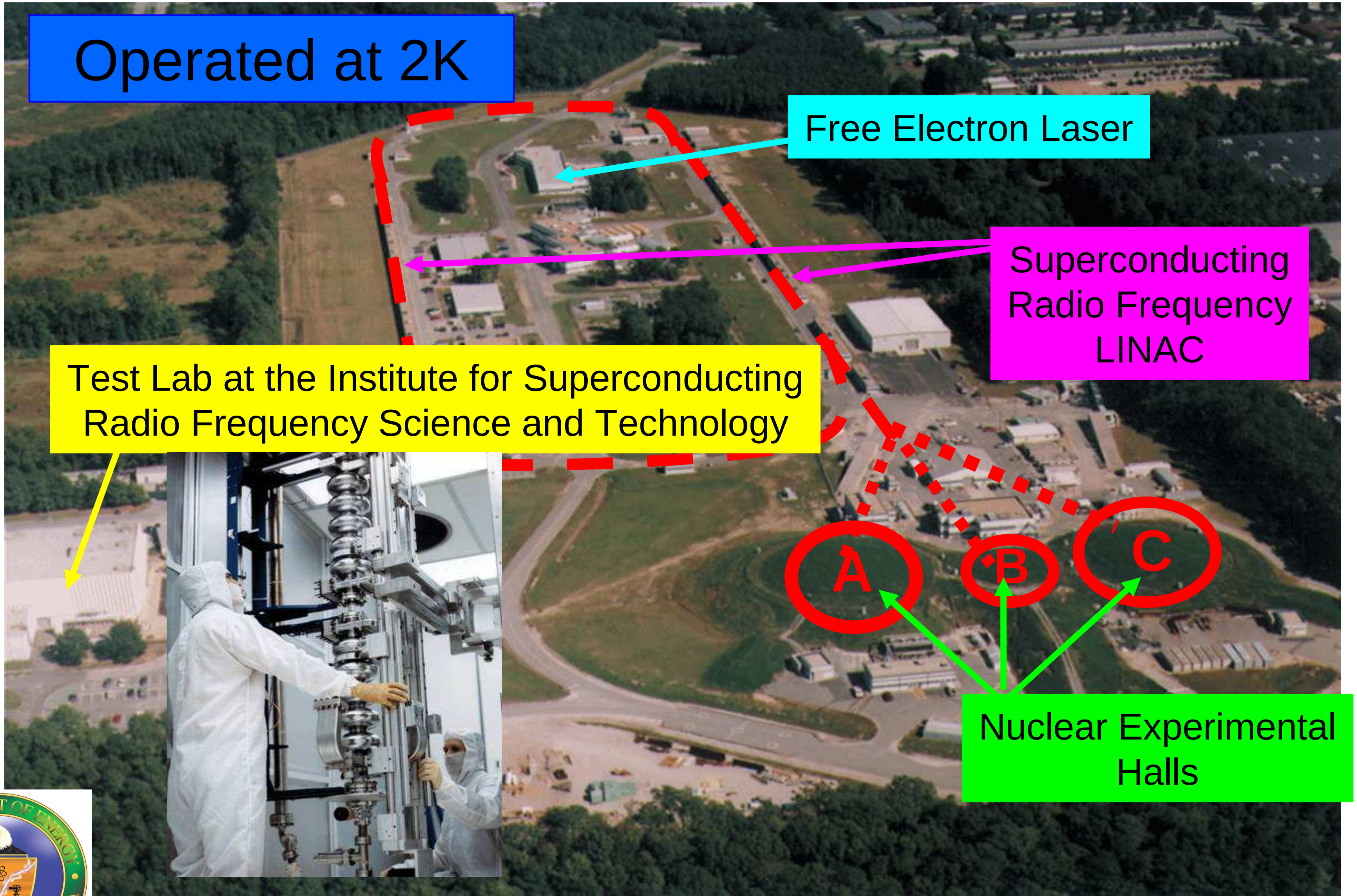
Free Electron Laser (FEL)



Continuous Electron Beam Accelerator Facility (CEBAF)



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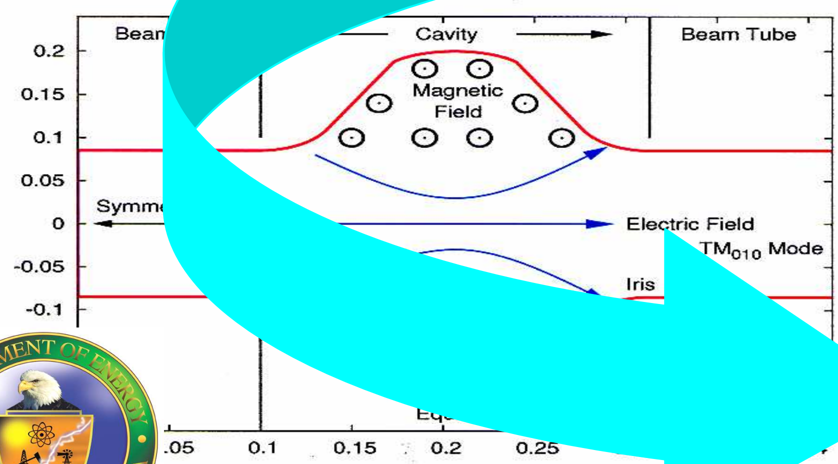
Nb SRF Cavities

Nb Single Cell SRF Elliptical Cavity

7 C... Cavity



matic of an elliptical cavity with field line ... M_{010} mode.

Why Niobium?

Tc=9.25K (highest among pure metals), best thermal, mechanical, and magnetic properties.

The performance of a SRF cavity ... **excitation**

...)/P_{diss}

Accelerating Gradient V_{acc}

U stored energy in the cavity

P_{diss} energy lost in the cavity

Typical RF frequency 1.5GHz

Typical RF penetration depth: 50 nm

Surface is very critical

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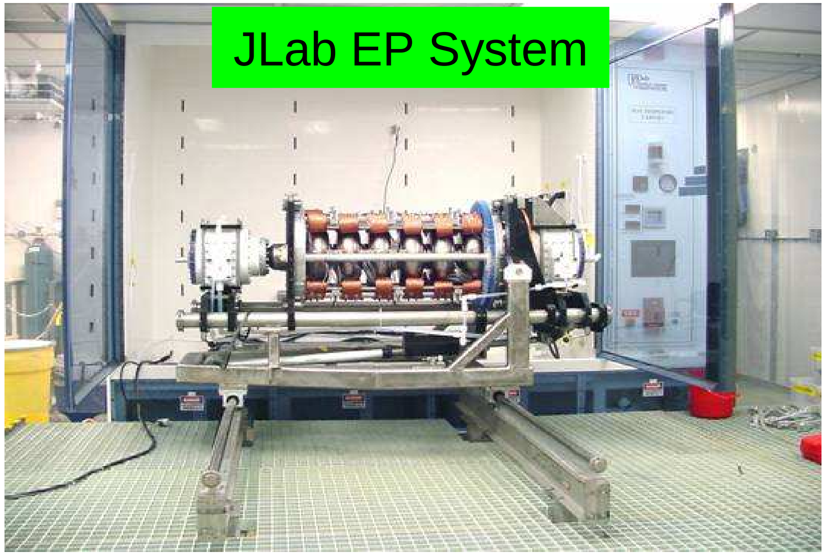
Surface Chemical Treatments on Nb

- Degreasing (ultrasound + soap+water, solvents)
- Buffered Chemical Polishing (BCP) ($HF:HNO_3:H_3PO_4$ as 1:1:1, 1:1:2,1:1:4)
- Electropolishing (EP) (HF/H_2SO_4 Siemens-KEK-Recipes)

High pressure Ultrapure Water Rinsing



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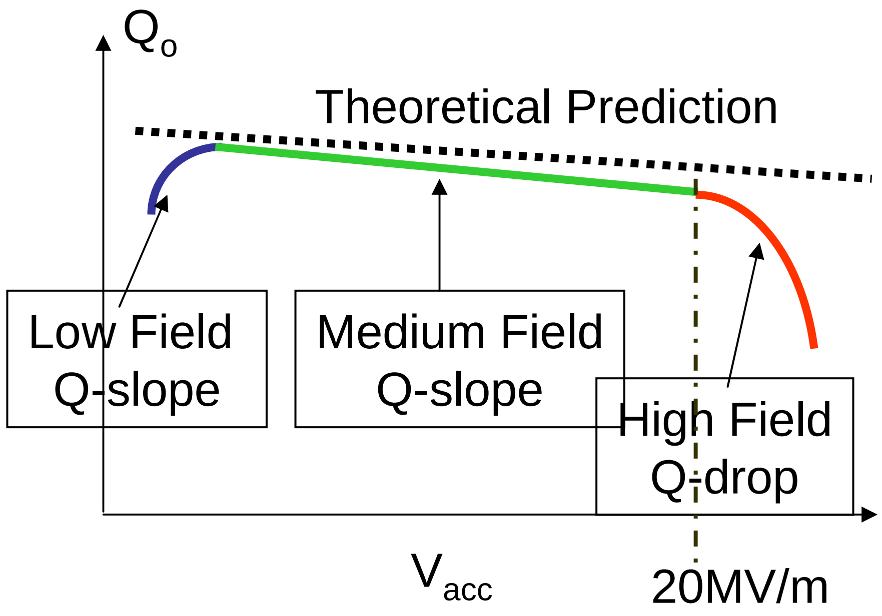
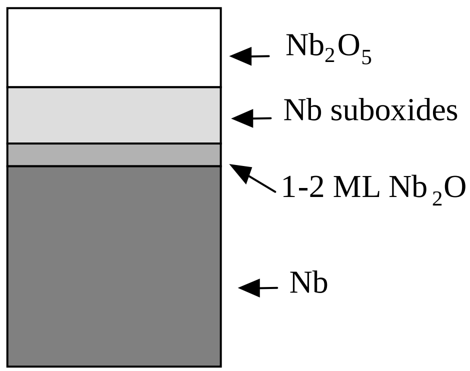
Q Degradation at High Gradients

- For high purity Nb, often a degradation in Q is found at gradients >20MV/m
- Baking of the cavities at 120C for certain periods of time (~10 hrs) improves the Q-values in the Q-drop regime
- The improvement is often more pronounced for EP cavities, but is also observed for BCP'd cavities
- It is believed that this effect is related mainly to the change of the oxide layer structure on Nb surface after the baking (H too)



Oxide Layer Structure on Nb Surface



Defect Free Nb Surface

- To reach the predicted excitation curve requires an ideal defect free Nb surface
- Nb surface is always covered by an oxide layer
- There are always some defects on Nb surface (dislocations, interstitial atoms, stacking faults, vacancies, etc)
- Defects change the lattice structure of Nb locally, leading to worse thermal conductivity, superconductivity, and magnetic properties, which eventually degrades cavity performance

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Surface Field Emission

- Field emission is mainly caused by surface contaminants of micron- to submicron particles
- Many cavities show onset of field emission at E_{acc}=10-20MV/m
- Emission current I can be described by modified Fowler-Nordheim equation

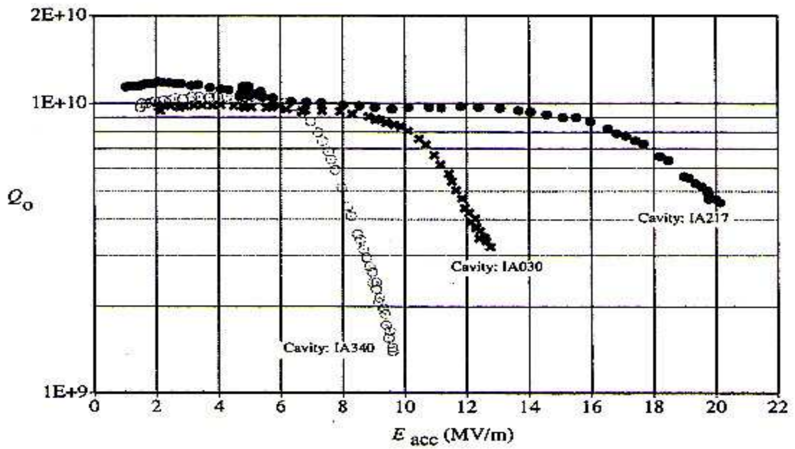
$I \sim A_e(\beta_{fn}E)^{2.5}Exp(-C\Phi^{3/2}/\beta_{fn}E)$

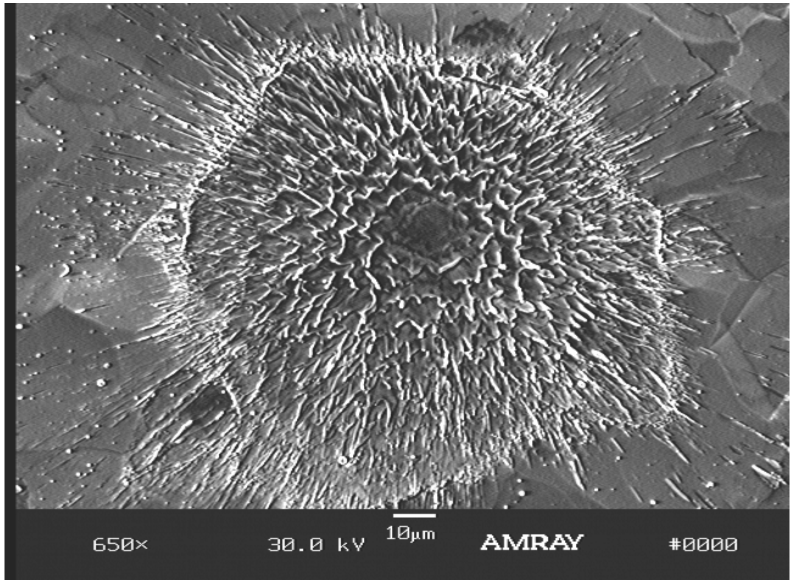
A_e effective emission area

β_{fn} field enhancement factor

Φ surface work function

- Emitter density depends strongly on processing and handling
- FE is sensitive to adsorbates, gas exposure, chemical residue, and particulate contamination.





Creating Better Surfaces

- Buffered Electropolishing (BEP) HF/H_2SO_4/Lactic Acid
- Sputtered Nb film on Cu
- Surface etching by Gas Cluster Ion Beam (GCIB) technique
- MgB_2
- Nb_3Sn
- Large or single grain Nb cavities
- High T_c Superconductors (cuprate)

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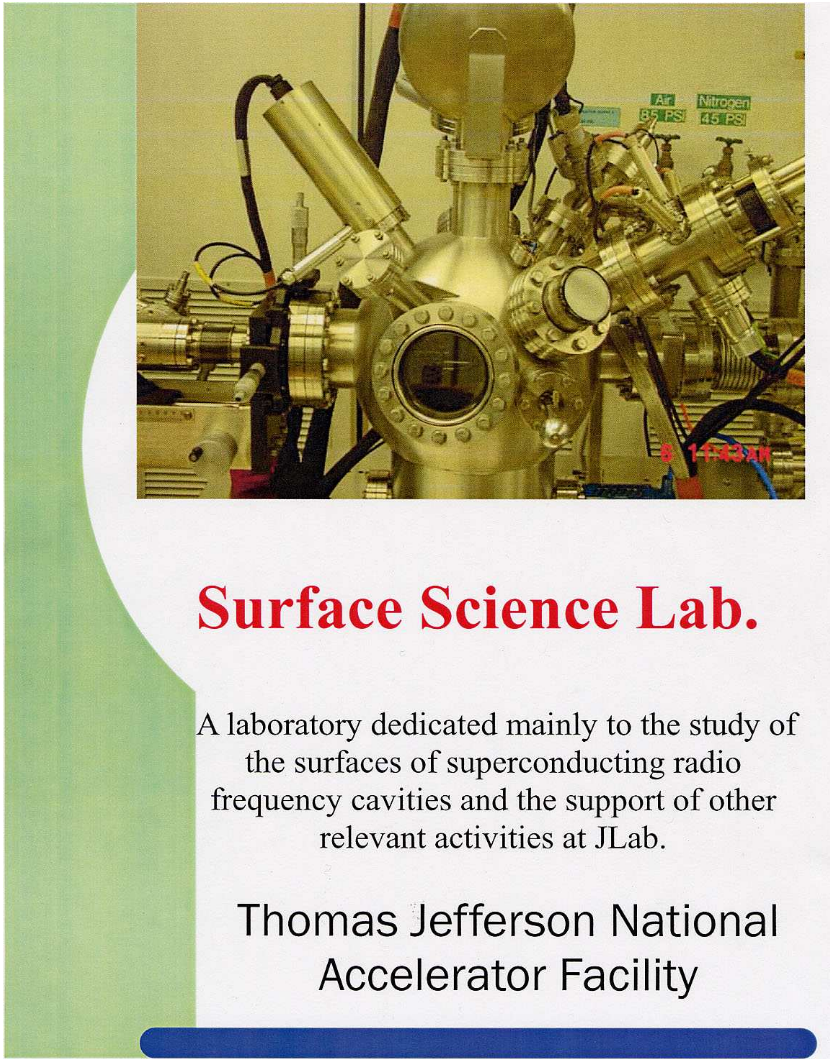
Surface Science Lab.

- Formally established in 2003
- Study the surfaces of SRF cavities in general, niobium in particular
- Study surfaces prepared by various techniques and procedures in terms of texture, morphology, oxide structure, defect, impurity, and level of tolerable contamination will enable us to make cavities with highest performance and reproducibility at the lowest possible cost



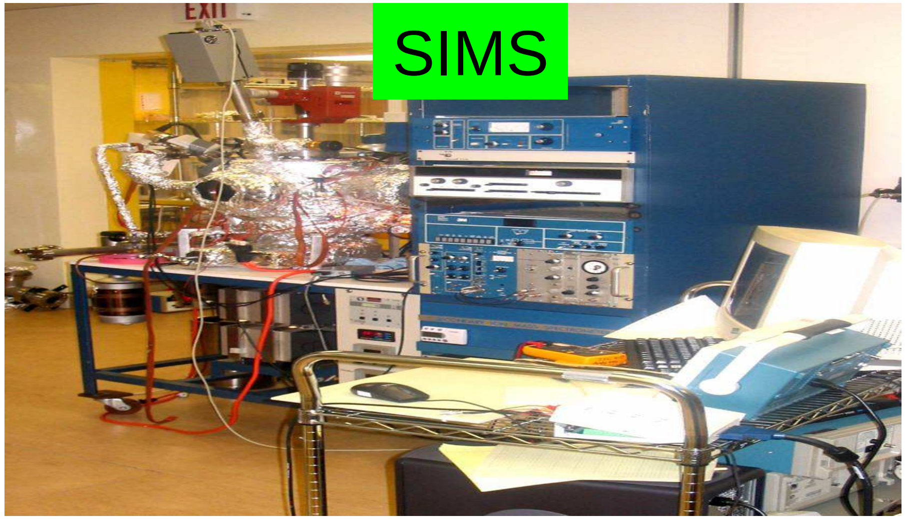
Surface Equipment 1

Scanning Field Emission Microscope

- Coupled with SEM and EDX
- Large scan area 25mm
- Voltage gradient 140MV/m
- 2.5µm lateral resolution
- In situ heat treatment up to 1400C at different environments

Secondary Ion Mass Spectrometry

- Vertical resolution < 3nm
- Mass range 1-300AMU
- Mass resolution >2
- Whole spectrum
- Depth profiling
- RGA



Surface Equipment 2

Scanning Auger Microscope

- Auger electron spectroscopy
- Depth profiling
- Scanning electron microscope
- Surface chemical mapping
- In-situ cooling -230C and heating up to 1000C

Transmission Electron Microscope and Scanning Transmission Electron Microscope

- Accelerating voltage 100kV
- Lattice resolution 0.14nm
- Point resolution 0.3nm

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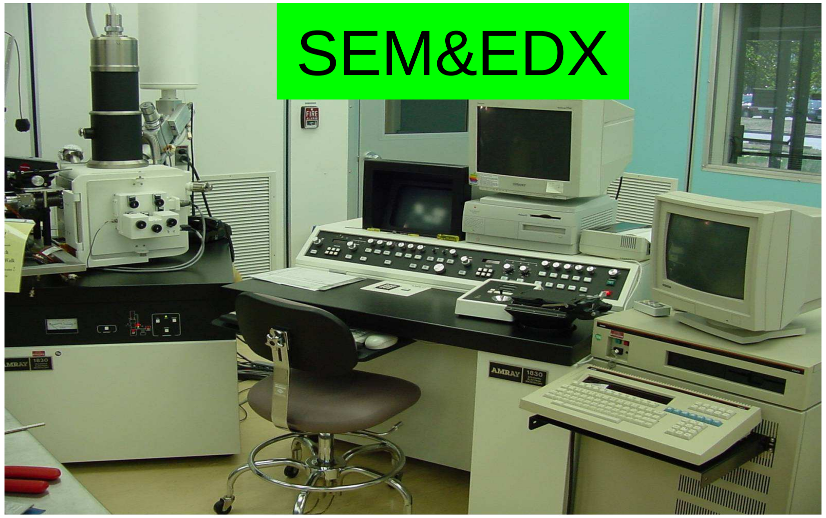
Surface Equipment 3

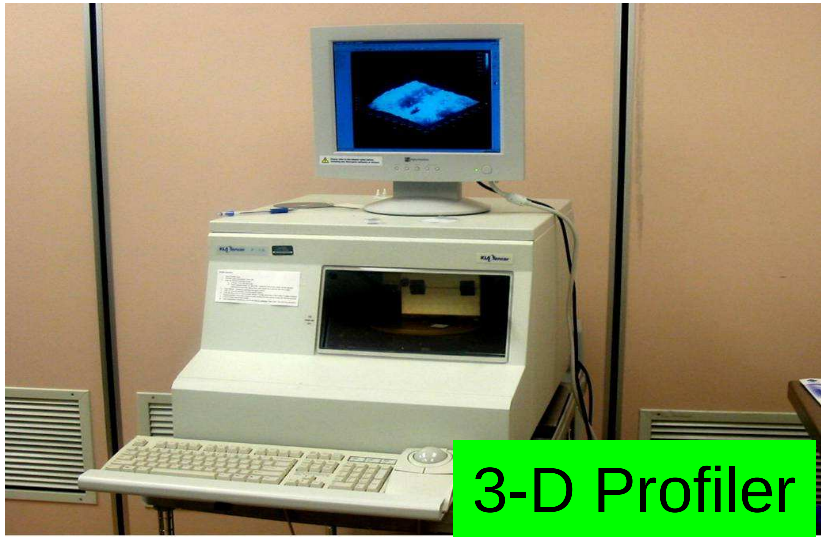
Scanning Electron Microscope & Energy Dispersive X-ray

- Topography and morphology
- Cross section
- All elements heavier than B
- Sampling depth ~ 1μm
- Sensitivity ~ 0.5% atomic weight
- 100,000 X

3-D Profilometer

- Scan area 8X20 cm^2
- Vertical resolution 0.1nm
- Guaranteed reproducibility 0.75nm
- Quantitative surface topographic information


SEM&EDX


3-D Profiler

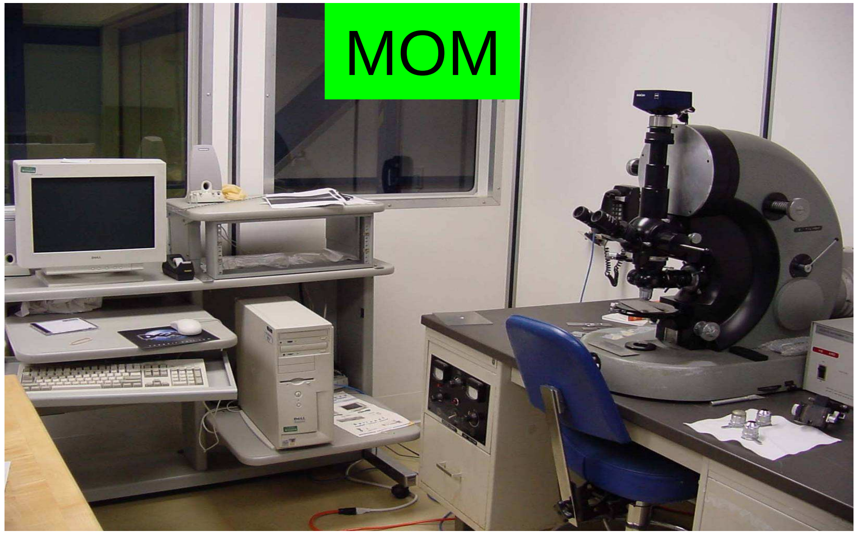
Surface Equipment 4

Metallographic Optical Microscope

- Computer controlled image capture and processing (Carl Zeiss)
- Magnification 8000X
- Resolution ~ 120nm

A Fully Equipped Sample Preparation Room

- TEM, SEM, MOM sample preparation
- Lapping, polishing, and cutting
- Surface and cross-section
- Heat treatments



Applications of Surface Equipment

- **SAM (LT baking, surface oxide layer structure, surface contamination, depth profiling)**
- **SFEM (field emission)**
- **SEM/EDX (field emitters, surface contaminants)**
- **SIMS (surface oxide layer structure, depth profiling)**
- **TEM/STEM (defects, interstitial atoms, secondary phases, stacking faults, etc)**
- **P-15 3-D Profilometer (surface morphology, topography)**
- **MOM (surface general inspection)**

Buffered Electropolishing (BEP) on Nb

- Electrolyte HF/H_2SO_4/Lactic Acid
- Add organic acid into the conventional EP acid mixture

A.T.Wu et al, Applied Surface Science 253 (2007) 3041-3052

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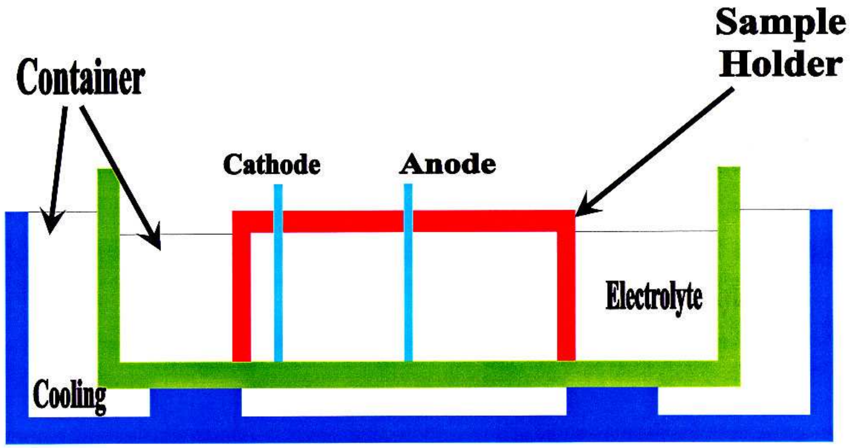
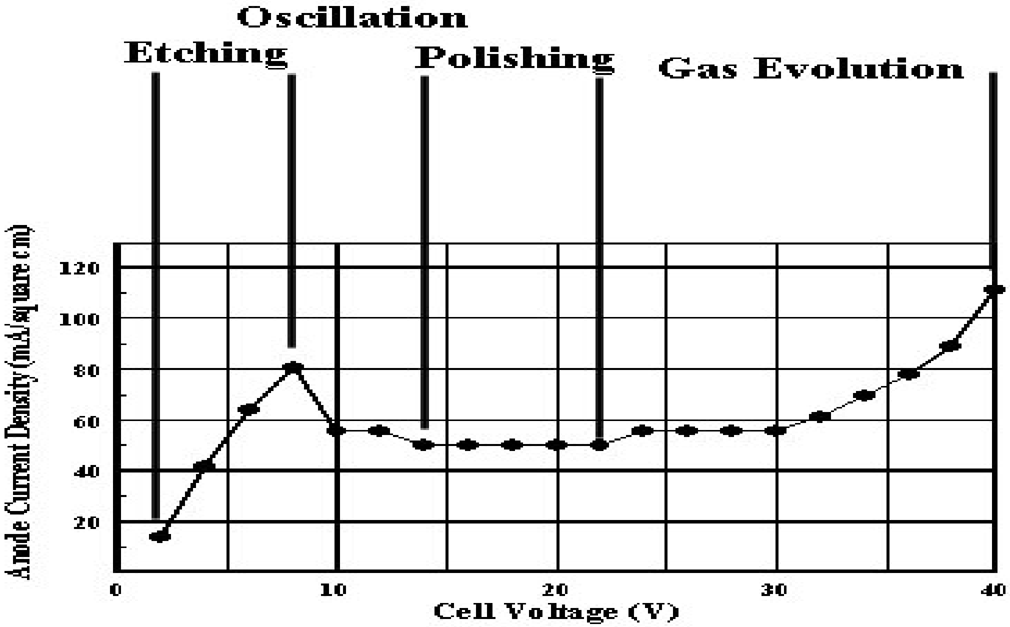
Experimental Set-up & I-V Curve

- Rectangular Nb flat samples 40X110mm^2
- 90mm immersed
- Temperature <30C
- 9 slots in sample holder
- 25 mm between neighbor slots
- Four I-V regions





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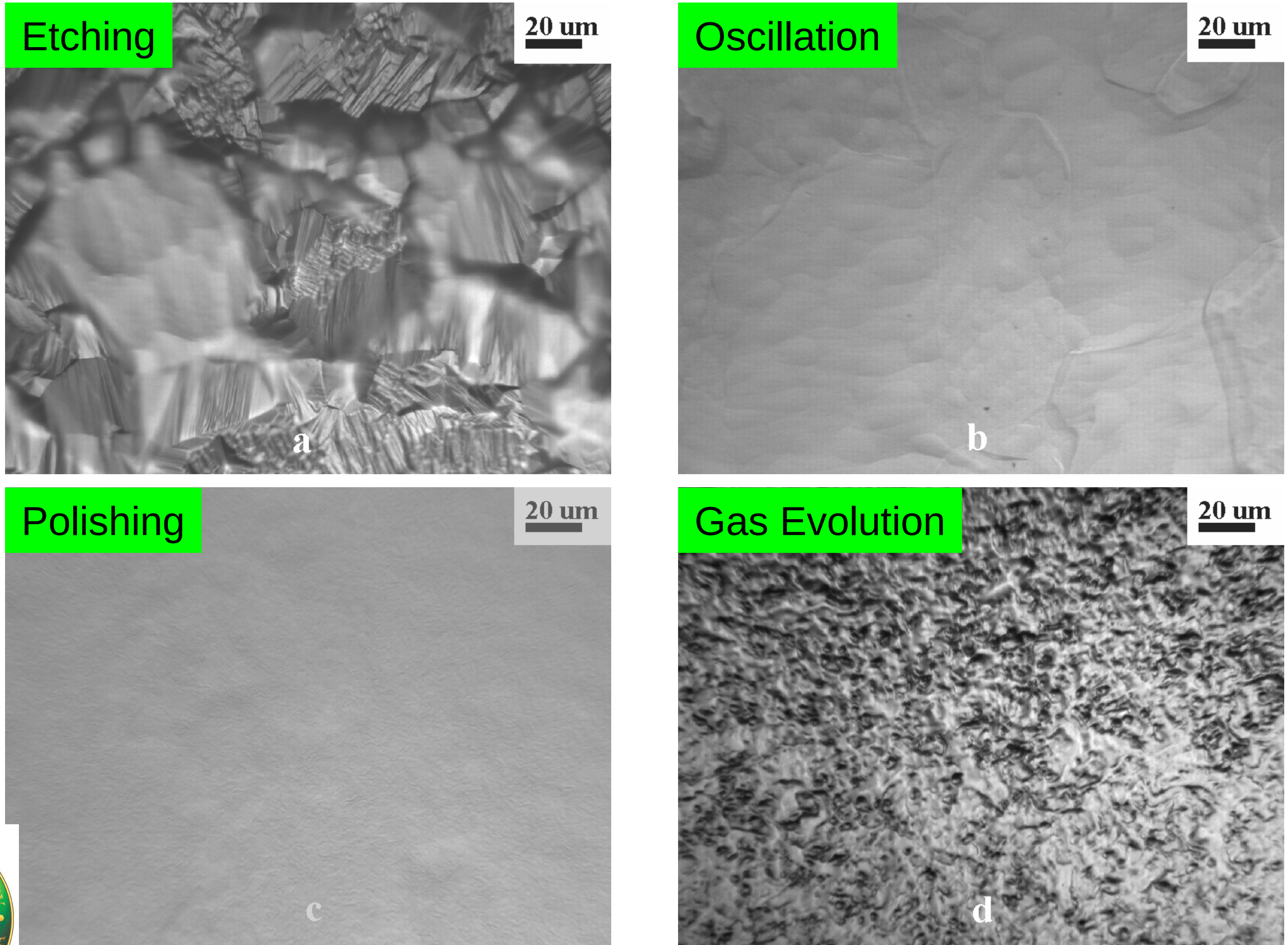
MOM Observation



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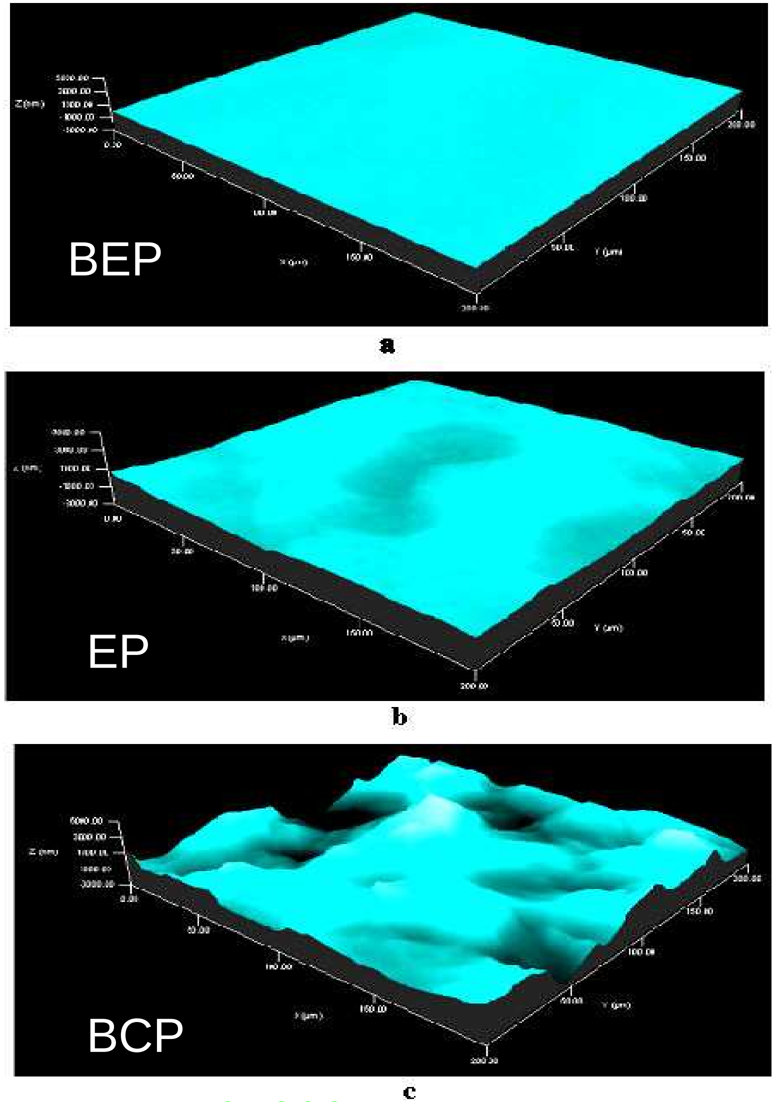
Profilometer Measurements

Measured Parameter \ Treatment Method	BCP	EP	BEP
TIR (nm)	7081	1561	290
RMS (nm)	1274	251	35
AMD (nm)	1019	198	25



A.T. Wu et al, Applied Surface Science 253 (2007) 3041-3052

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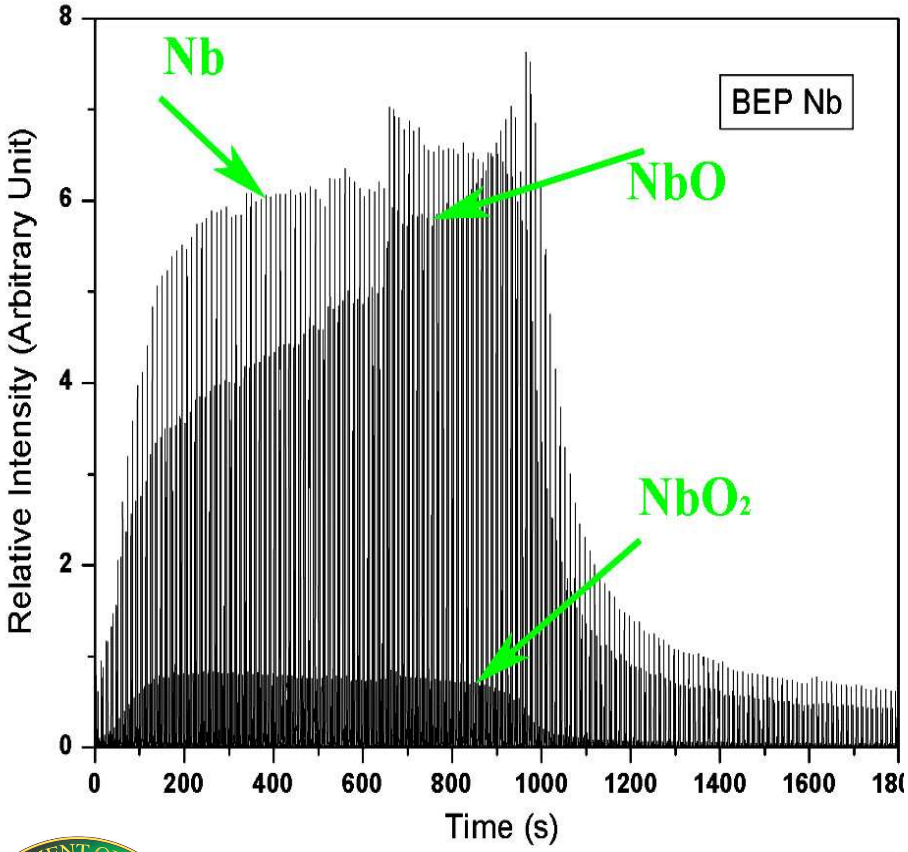
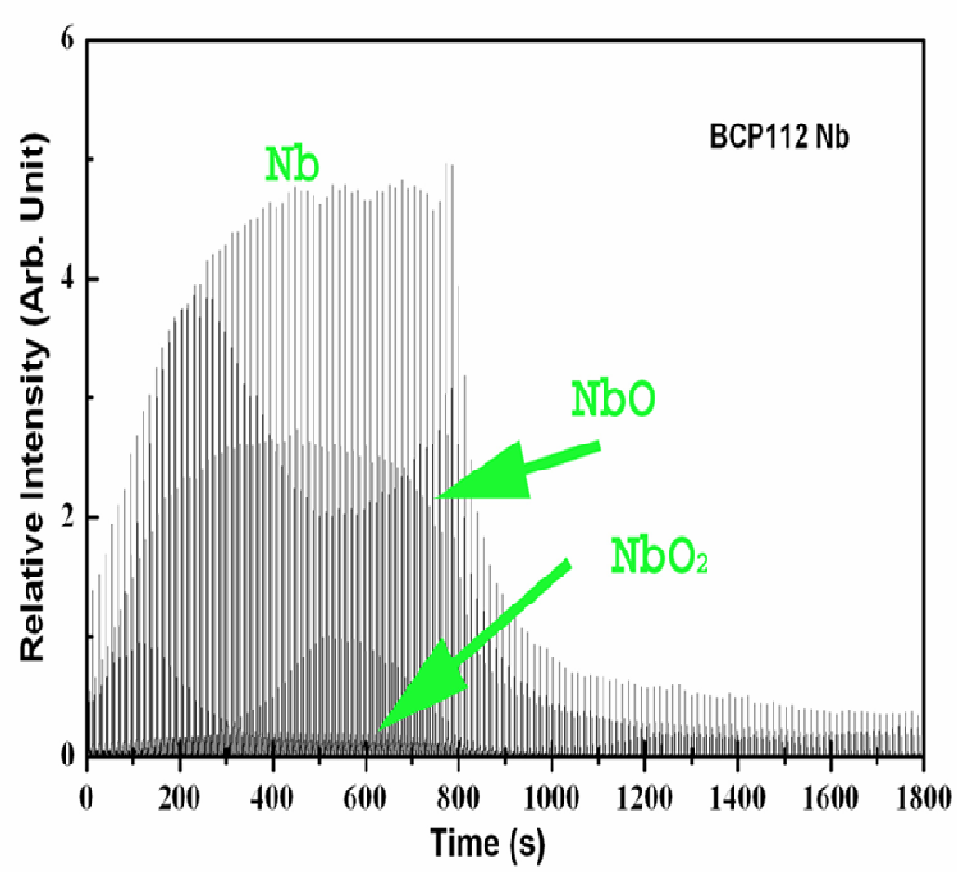
SIMS Depth Profiling





BEP on Nb Cells

Non uniform electric field

More uniform electric field

Buffered Electropolishing (BEP) on Nb

- Surface is an order of magnitude smoother than that by the conventional EP
- Much higher polishing rate. BEP 646nm/min Vs. EP 381nm/min
- Acid mixture is safer to handle.
- The life of the acid mixture is longer than the conventional mixture
- Acid mixture is cheaper
- Less sulphur precipitation
- Easy to be adopted to the existing EP setups

A.T. Wu et al, Applied Surface Science 253 (2007) 3041-3052

Summary of BEP

- Highly promising for polishing on Nb SRF cavities
- Huge capital gain
- A project has been launched to do BEP on Nb single cell cavities

Single Cell Vertical BEP Set-up

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Investigation of Oxide Layer Structure on Niobium Surface Using a Secondary Ion Mass Spectrometry

A.T. Wu, Physica C 441 (2006) 79-82

CEA Saclay, France, Mar.19, 2007

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Niobium Surfaces

- **Complications:**

Known

Top most layer Nb_2O_5

Unknown

suboxides Nb_2O_3, NbO_2 NbO Nb_2O etc (thickness and chemical states) and interstitial atoms (O, H, N, C, S, etc). Variations with surface preparation techniques

- **Experimetal Techniques**

1. X-ray photoelectron spectroscopy (XPS) = electron spectroscopy for chemical analysis (ESCA)

*Ambiguity in background subtraction and curve fitting, too many fitting parameters and uncertainty about suboxides

*Poor Sensitivity   typical 0.1 at %

*Chincarini's Principal Component Analysis   SRF2001

2. Secondary ion mass spectrometry (SIMS)

More experimental results only recently   1ppm~1ppb

Conventional Commercial SIMS Systems

- Primary ion beam: Oxygen (positive secondary ions) or Cesium (negative secondary ions) most common
- Depth profiling: Monitor the intensity of a selected peak as a function of time
- Not constructed or purchased for studying niobium (wait for explanation later)

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Typical SIMS Spectrum from Conventional SIMS System

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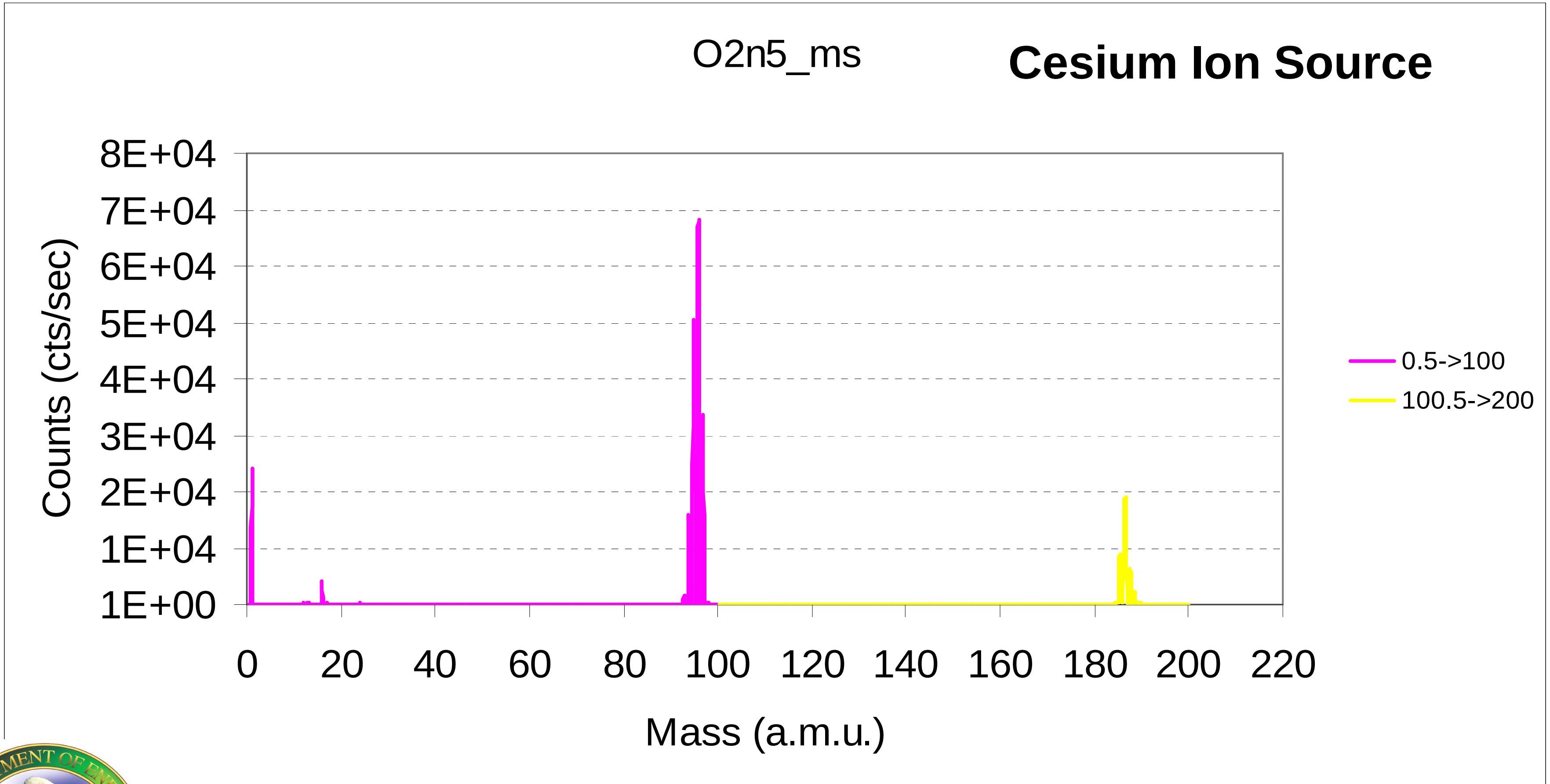

By Courtesy of Gigi

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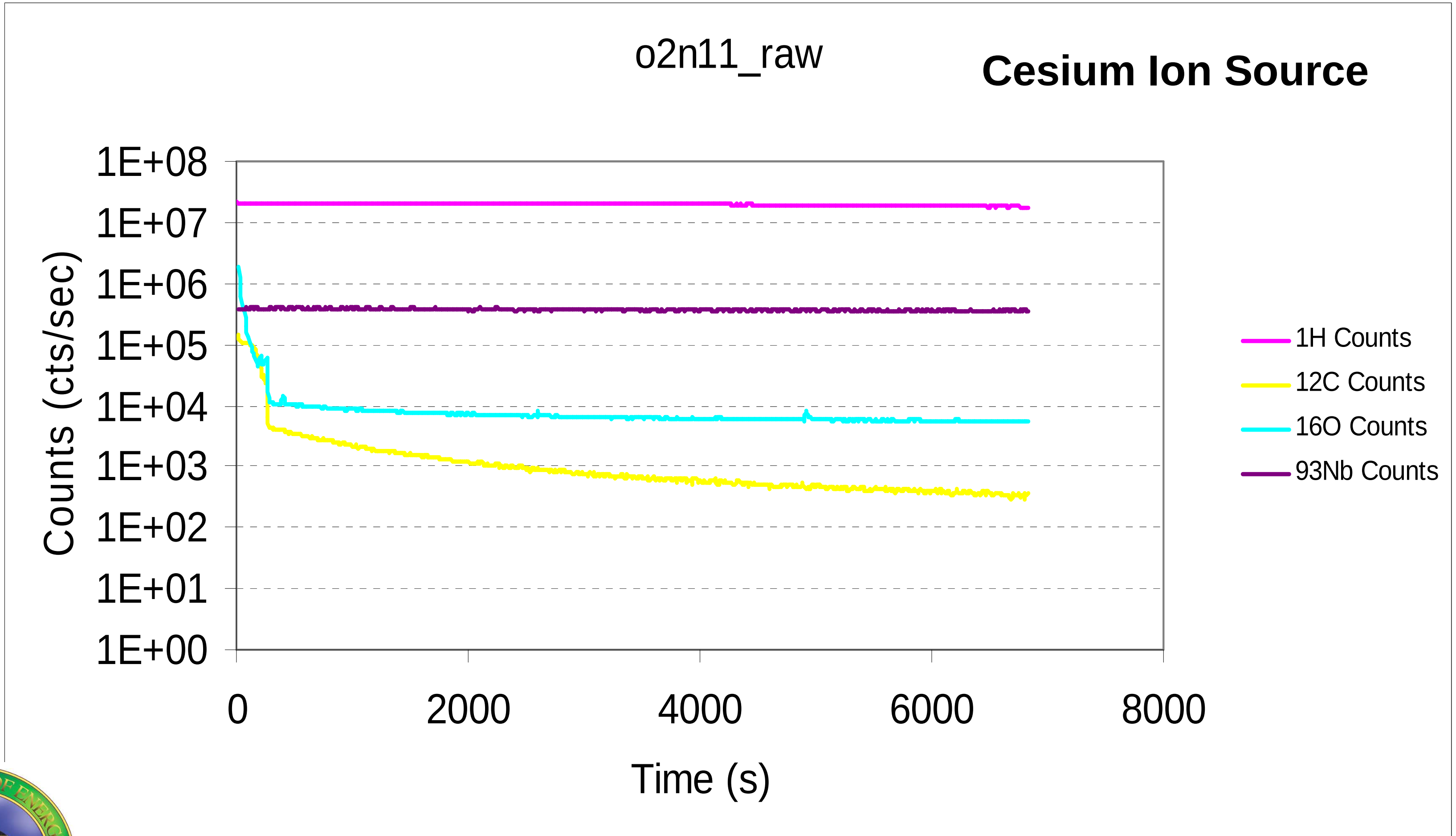
Typical SIMS Depth Profiling from Conventional SIMS System



By Courtesy of Gigi

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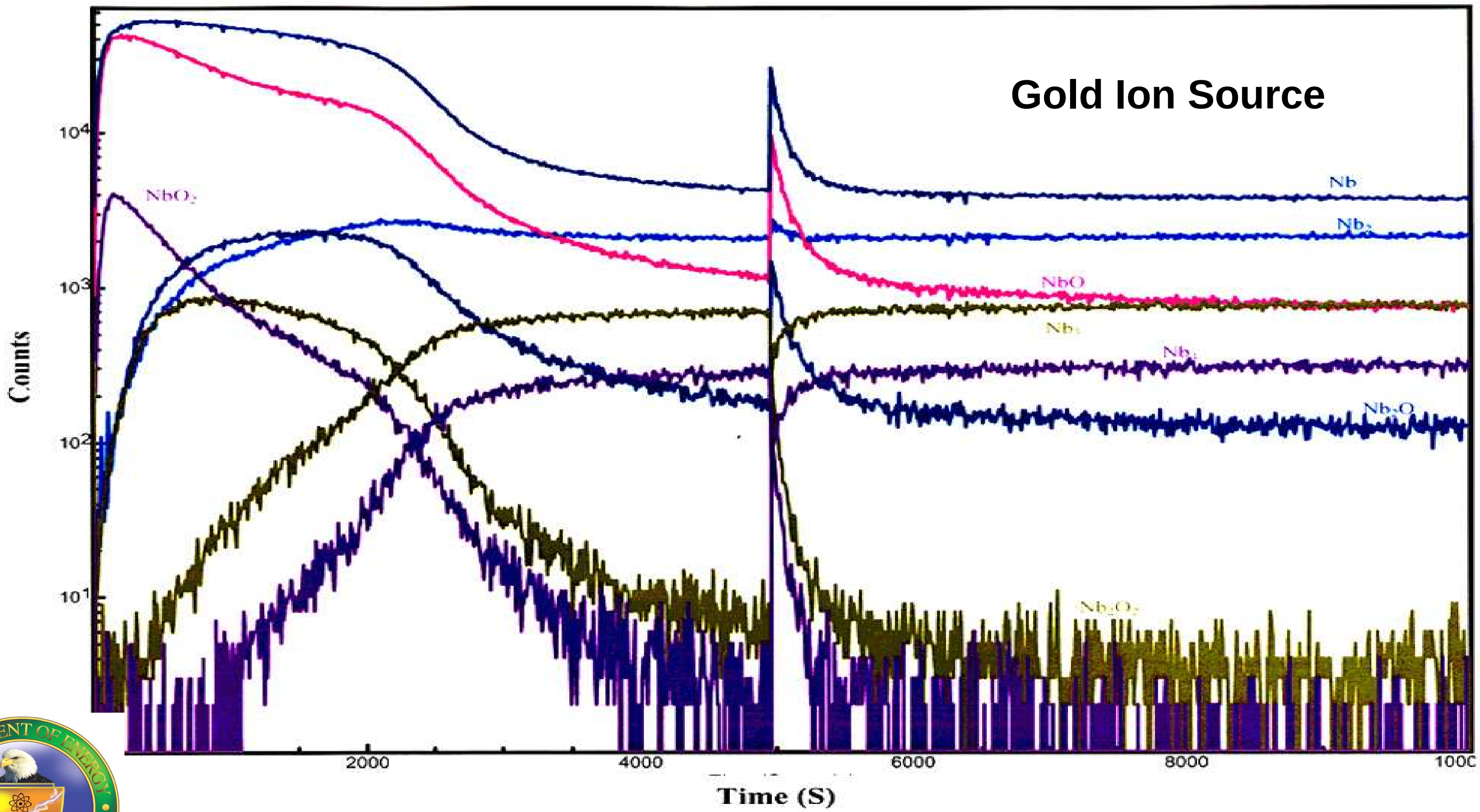
Typical SIMS Depth Profiling from Conventional Static SIMS System



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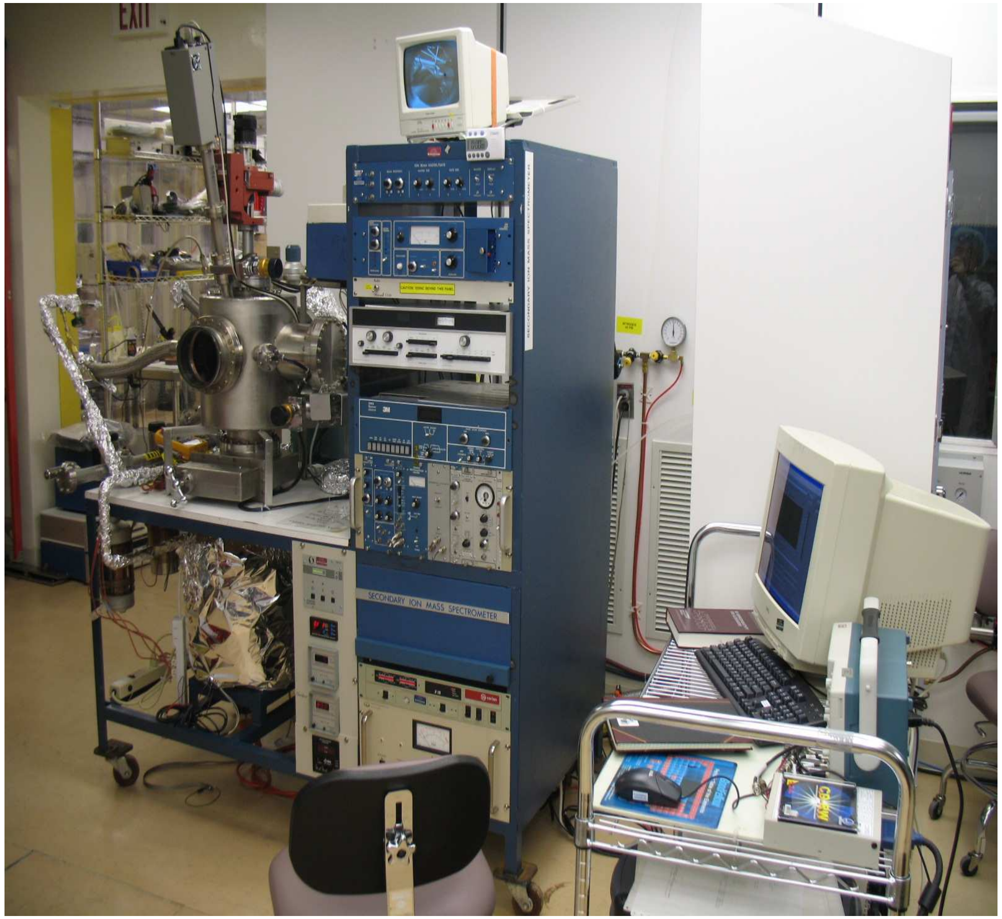
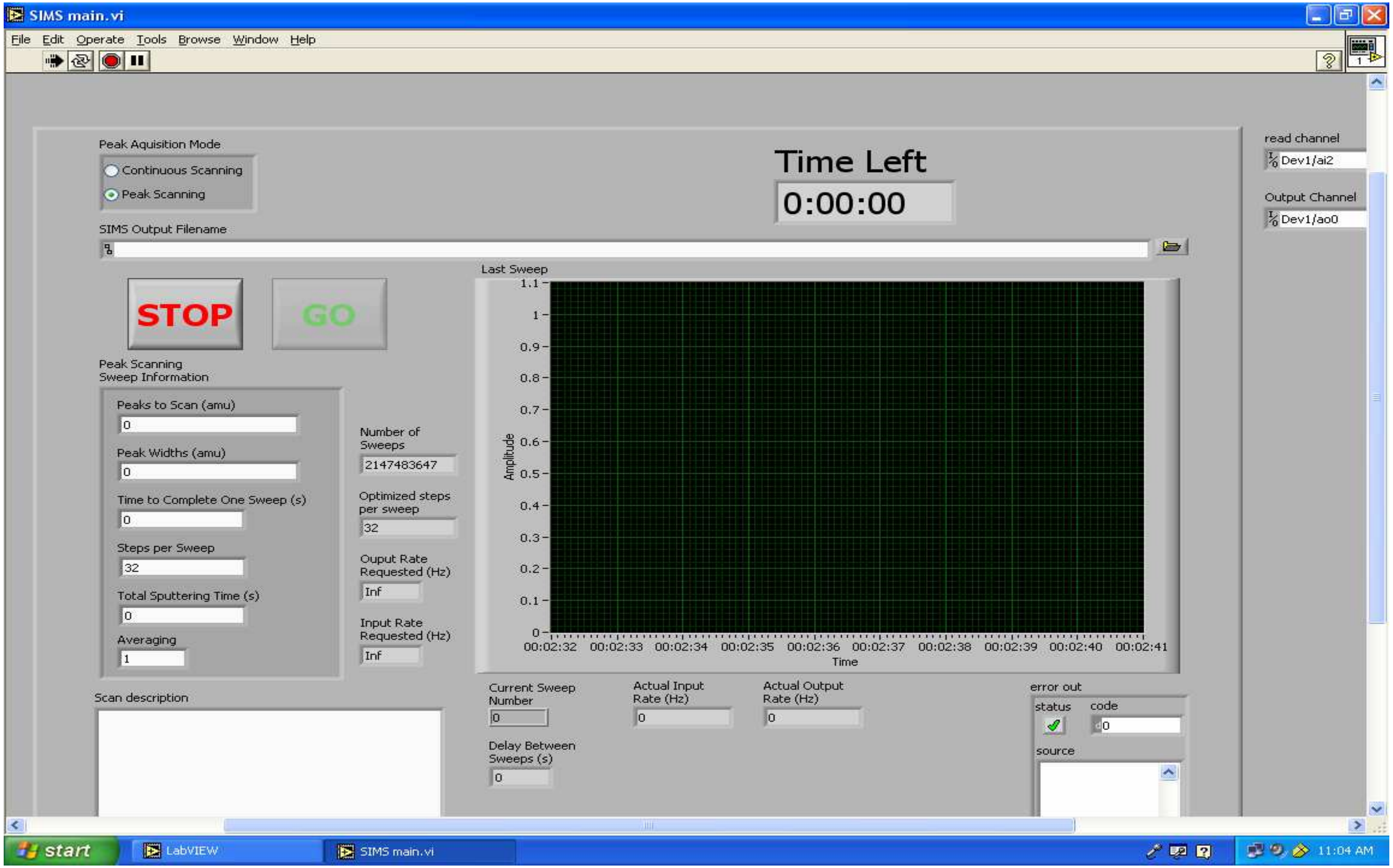
Our Dynamic SIMS System

- Built to study Nb surface mainly
- Depth profiling through peak scans
- More accurate in depth profiling
- Able to do real elemental depth profiling and semi-quantitative measurements



DEPARTMENT OF ENERGY
UNITED STATES OF AMERICA

Our SIMS


SIMS main.vi
File Edit Operate Tools Browse Window Help
Peak Aquisition Mode
Continuous Scanning
Peak Scanning
Time Left
0:00:00
SIMS Output Filename
STOP
GO
Last Sweep
Peak Scanning
Sweep Information
Peaks to Scan (amu)
Peak Widths (amu)
Time to Complete One Sweep (s)
Steps per Sweep
Total Sputtering Time (s)
Averaging
Number of Sweeps
2147483647
Optimized steps per sweep
Ouput Rate Requested (Hz)
Input Rate Requested (Hz)
Amplitude
Time
Scan description
Current Sweep Number
Delay Between Sweeps (s)
Actual Input Rate (Hz)
Actual Output Rate (Hz)
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read channel
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LabVIEW
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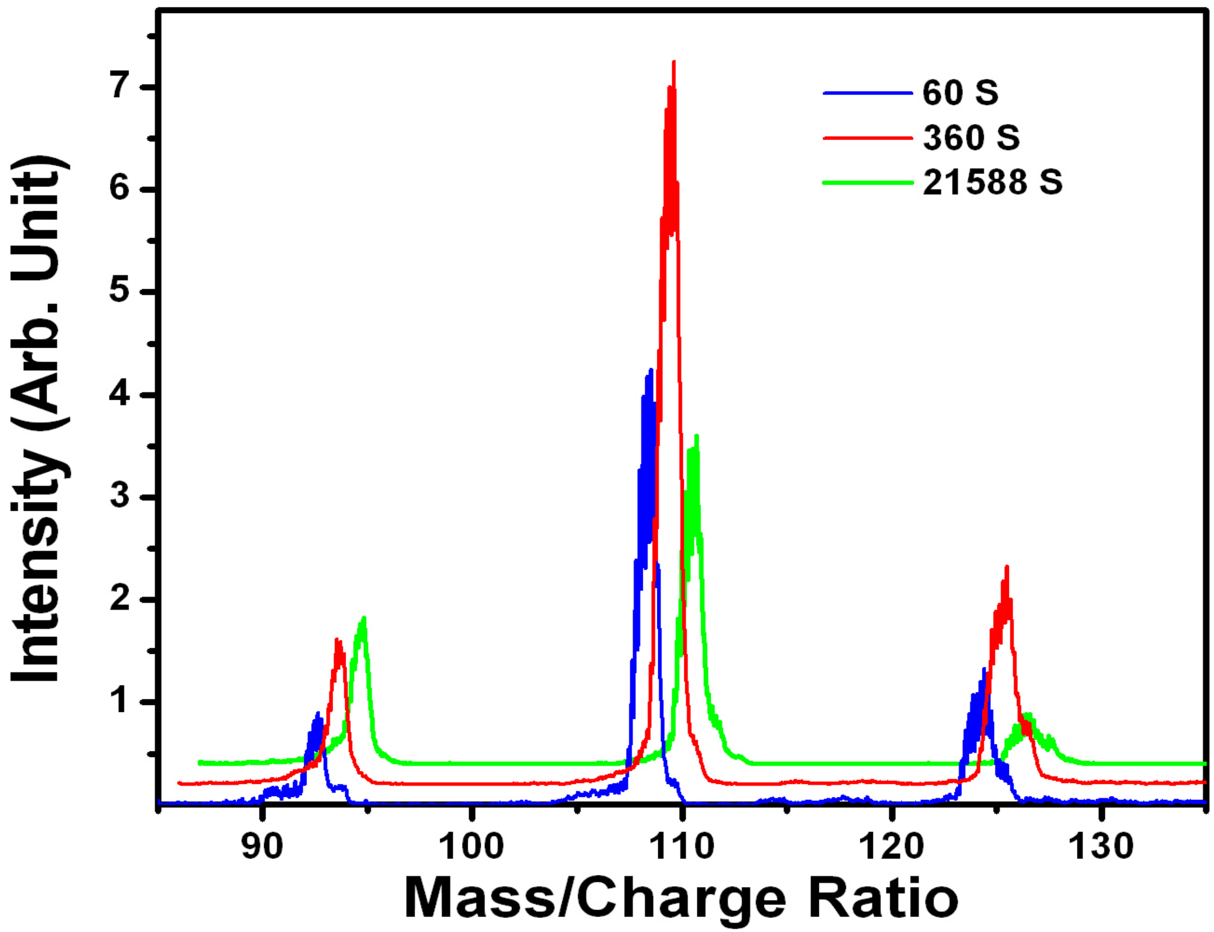
Our SIMS Spectra



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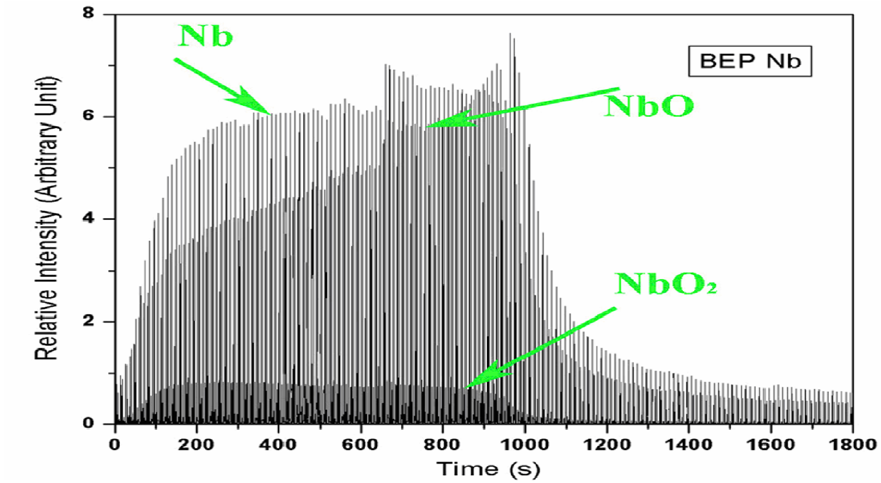
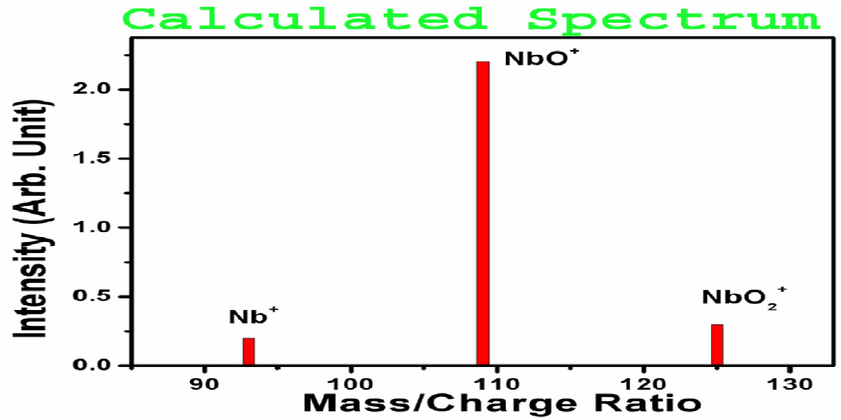
SIMS Study of Nb Surface Oxide Layer Structure

- Oxide layer structure is studied by continuously monitoring peaks of secondary ions of Nb and its relevant oxides of a function of time during depth profiling
- Different Nb oxides have different cracking patterns
- Changes in oxide layer structure are reflected from changes in the ratio between Nb and its relevant oxide peaks

A.T. Wu, Physica C 441 (2006) 79-82





C.Plog et al, Surface Science, 67 (1977) 565

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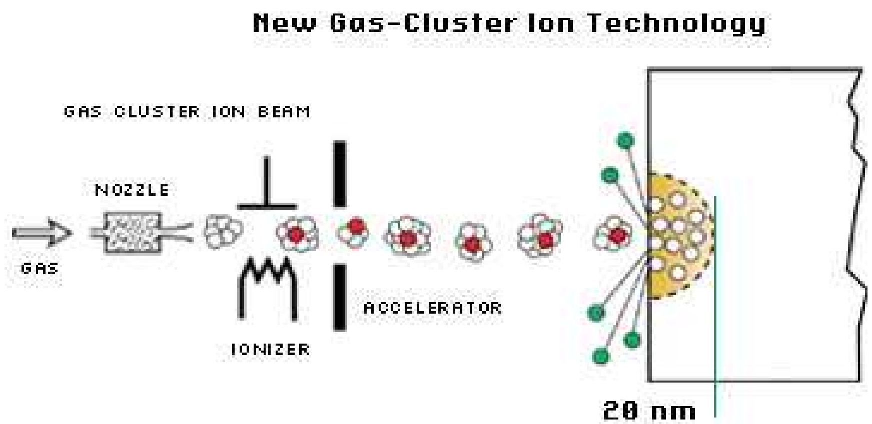
GCIB Treatments on Nb

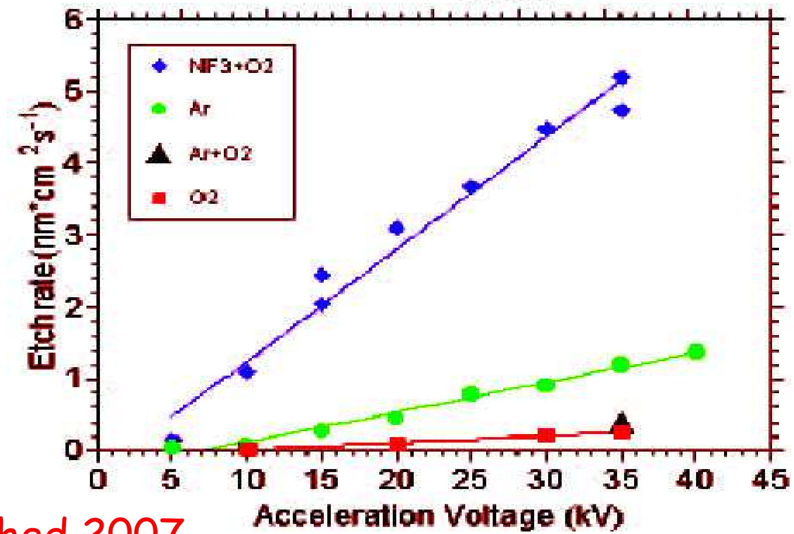
Typical Parameters of GCIB:

- >10,000 atoms
- Accelerating Voltage 30kV
- Average Charge State 3.2
- Impact Velocity 6.7 km/s
- Beam Current >200μA

Various gas species: Ar, O_2, N_2, NF_3, SF_6, B_2H_6, BF_3 and GeH_4

Etch rate can be as high as 5.5 nm/s





D.R. Swenson & A.T. Wu et al, to be published 2007

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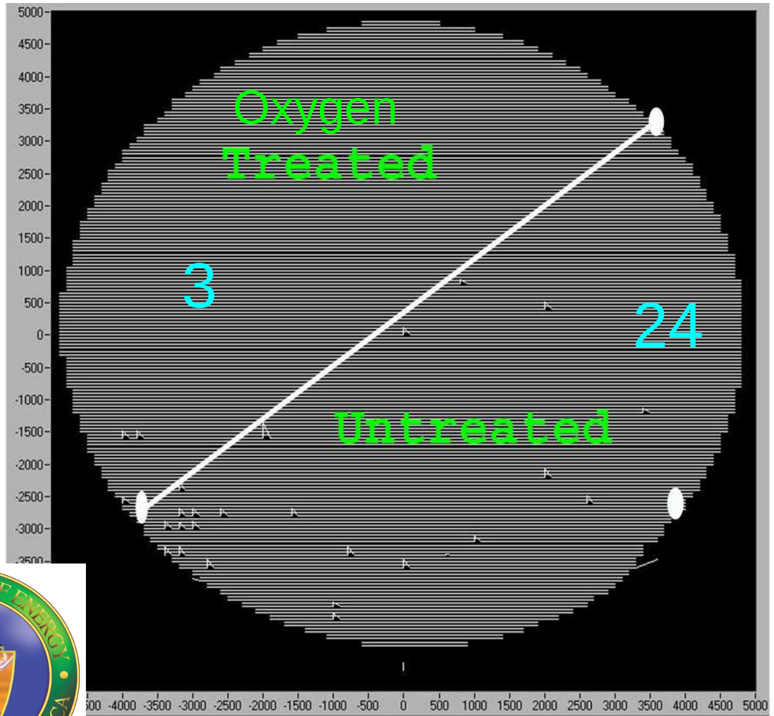
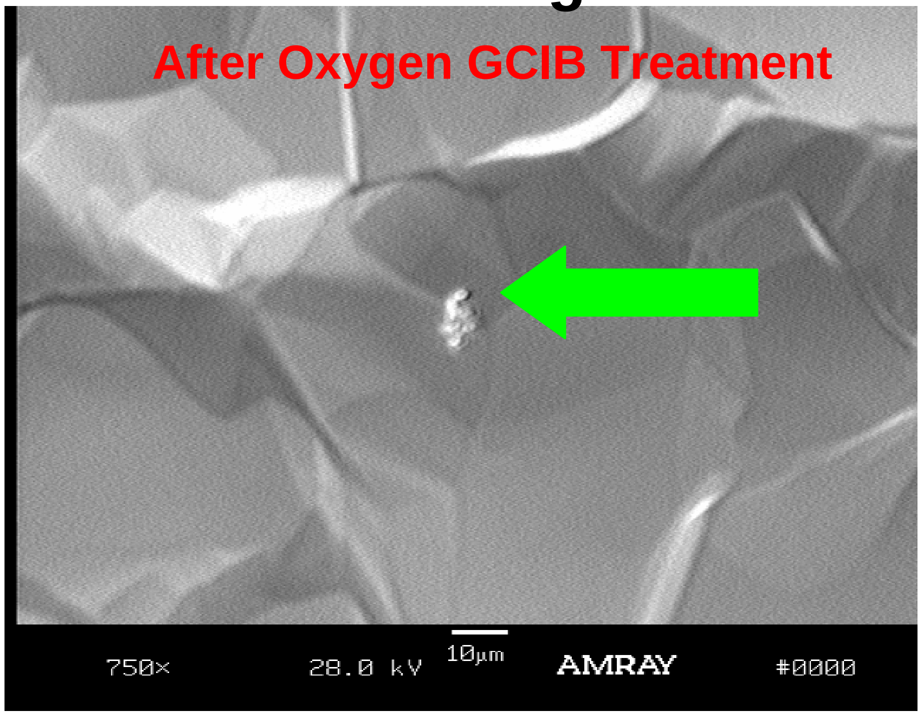
Benefits of GCIB

- Significant reduction in the number of emitters
- Dry chemistry
- Possibly new surface oxide layer structure
- GCIB on Nb single cell cavities (collaboration EPION D.R. Swenson JLab A.T. Wu/P. Kneisel)

SFEM Scans



SEM Image



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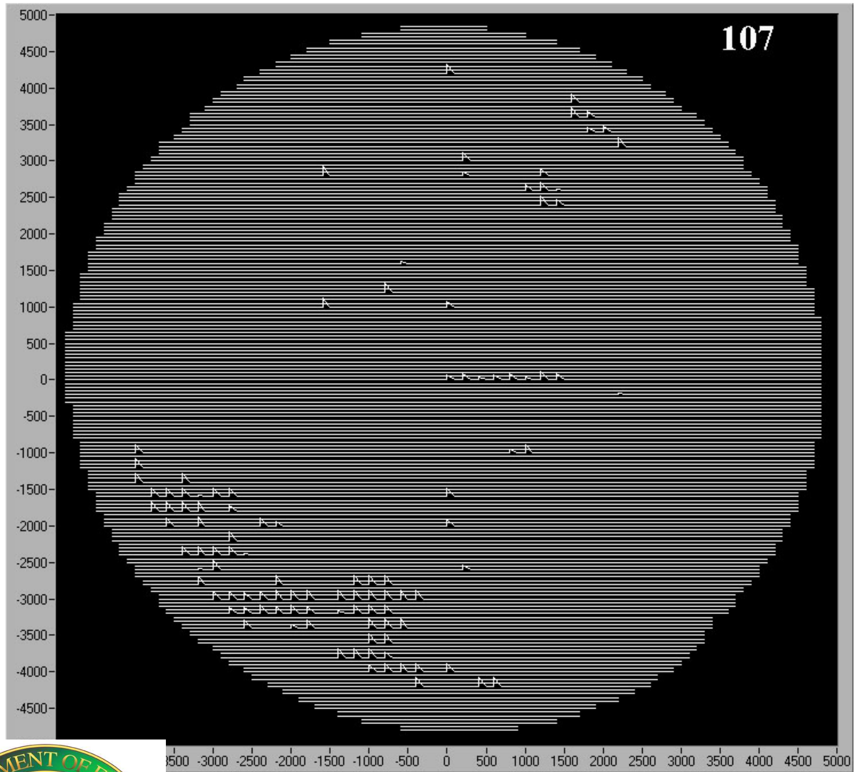
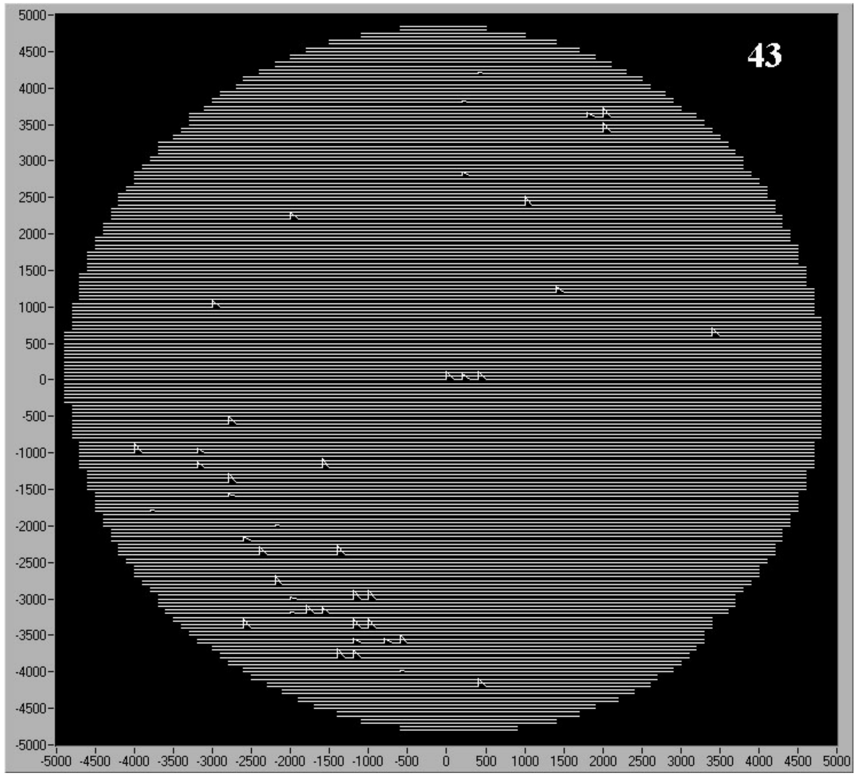
The Low Temperature Baking Effect on Nb

Before Baking at 120C for 48hrs



After Baking at 120C for 48hrs



A.T. Wu et al, to be published 2007

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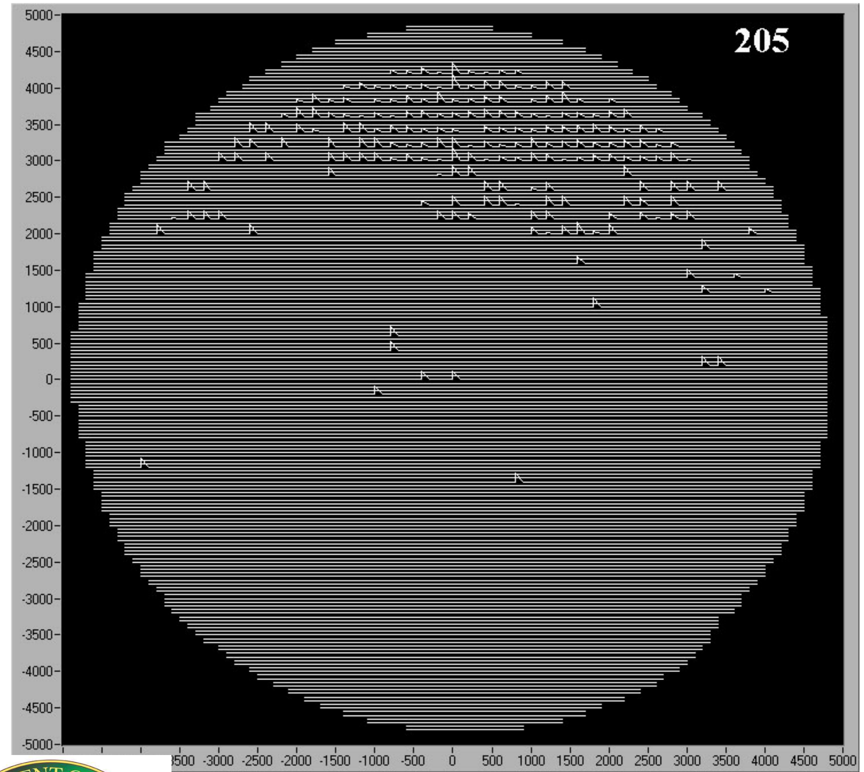
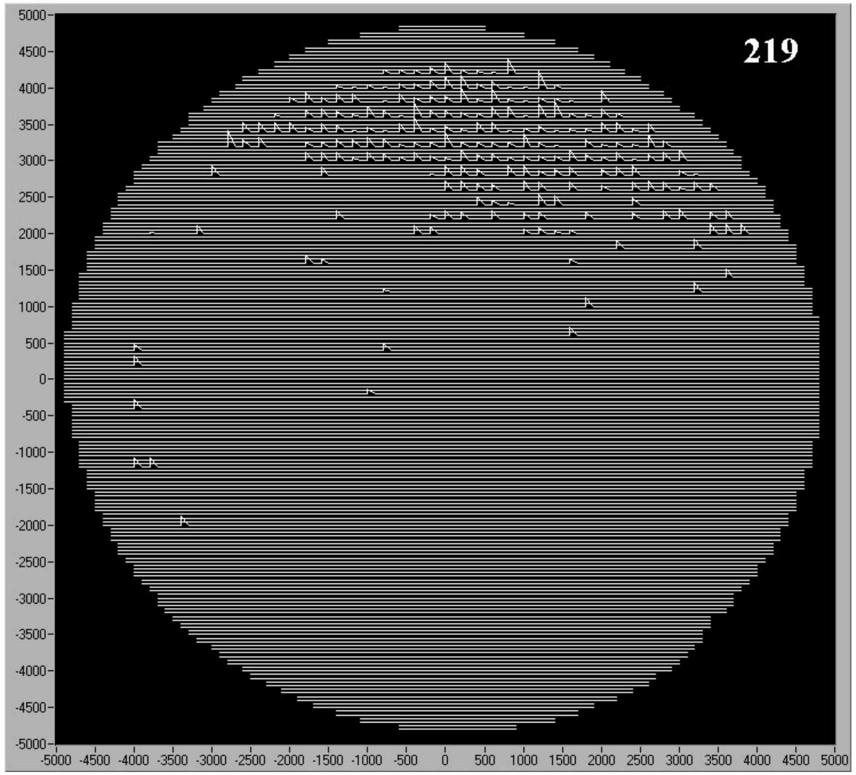
SFEM Scans of Unbaked Nb

First Scans



Second Scans



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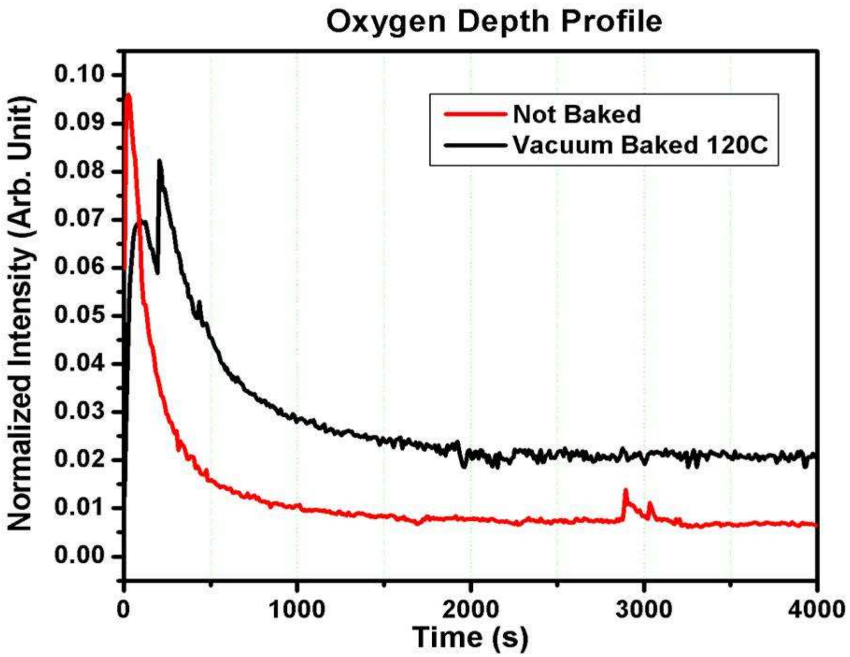
SIMS Depth Profiling



- LTB reduces number of emitters (new result)
- LTB changes surface oxide layer structure (on-going)

A.T. Wu et al, to be published 2007

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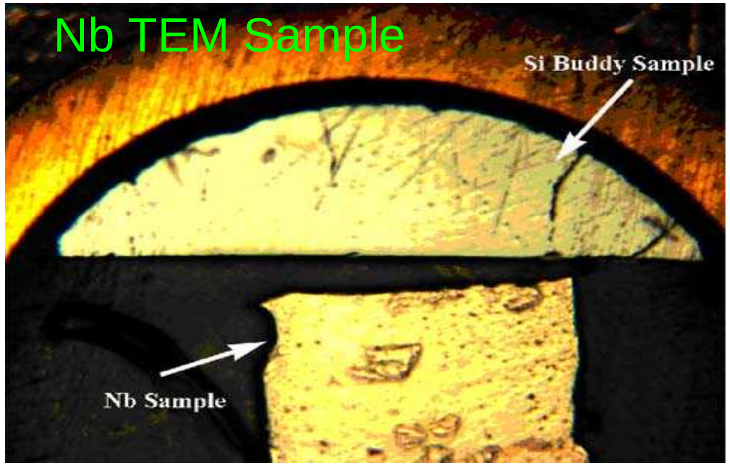
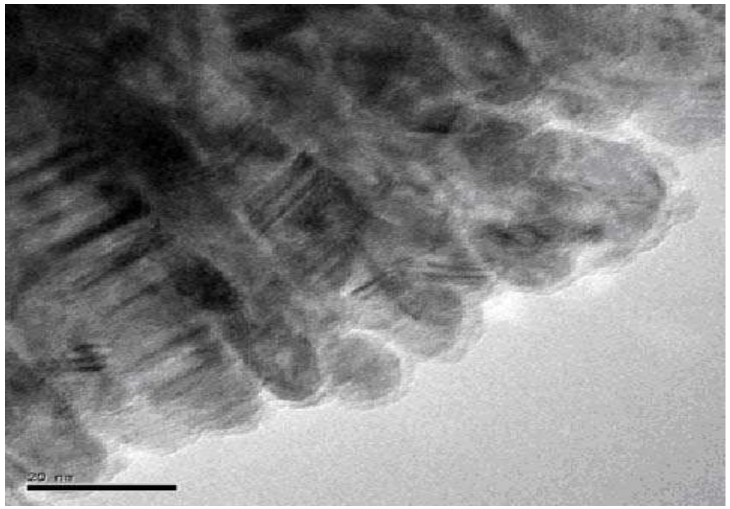
TEM Observation of Cross Section of Nb

- Attempts have been made to observe the surface oxide layer structure of Nb by TEM at JLab
- Surface oxide layer structure appears to be inhomogeneous



First TEM Cross-section of Nb



A.T. Wu, Proc. of 11th workshop on SRF, Germany 2003

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Summary

- Surface condition is very critical for the performance of SRF cavities
- A surface science lab has been established at JLab to study the surfaces of SRF cavities.
- Some on-going R&D activities on Nb at JLab such as, BEP, SIMS study of surface oxide layer structure of Nb, GCIB treatments on Nb, study of LTBE, and TEM cross-section of Nb, are briefly shown and discussed.
- Surfaces of SRF cavities are unique and complicated and need more attention.

Collaborations are welcome!!!