Review of SRF Materials Workshop

Genfa Wu, Lance Cooley, Claire Antoine,
and Helen Edwards

Fermilab

Outline

- Overview of the workshop
- Highlights of new approaches, new results and new ideas
- Highlights of the debate

Fermilab

RF Superconducting Materials Workshop at Fermilab

Registered Participants

Dinner Information

Agenda and **Proceedings**

PHOTO Album

First Announcement

Hotels

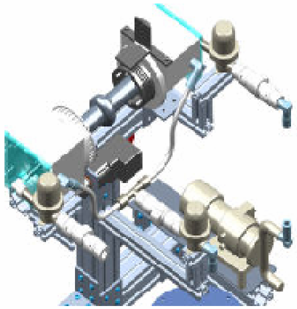
Hotel Locations
Transportation and Accommodations at Fermilab
Visa Application Guidance
Other Useful Links

Instructions for Accessing the Fermilab Wireless Network

Abstract Submission

Abstracts Submitted

May 23 & 24, 2007



The goal of the workshop is to form a coordinated SRF materials research activity, promote inter-disciplinary studies, further expand SRF research to more universities, invite early industry participation and strengthen the collaboration between national laboratories and academic institutes. As such the focus will be different from the SRF international workshop.

The workshop will be organized in the form of invited talks and working group discussions. Below are some topics of discussion:

- Fundamentals of RF superconductivity
- Material properties of superconductor
- Fabrication of superconducting materials
- Processing of materials
- Surface Characterizations
- Industrial collaboration

The workshop will be held in Curia II located on the 2nd floor of Wilson Hall.

Past Midwestern SRF materials research meetings can be found here

| Contact Us | Fermi National Accelerator Laboratory

Legal Notices

http://tdserver1.fnal.gov/project/workshops/RF_Materials/

Overview of workshop

Workshop pushed by H. Edwards, D. Larbalestier, …

SRF Materials Workshop

The need to bring together SRF practitioners, materials and surface scientists, experts from universities, national labs, and industry - teaming of ideas and expertise

why it is effective/ necessary/useful… to do basic R&D along with technological developments

R&D is a way to confirm/ optimize (/choose among) empiric improvements

The physics behind ultimate limitations is still not well understood. It is of basic scientific interest

Better understanding will lead to new ideas and to better paths for development and improved preformance

- H. Edwards

Overview of workshop

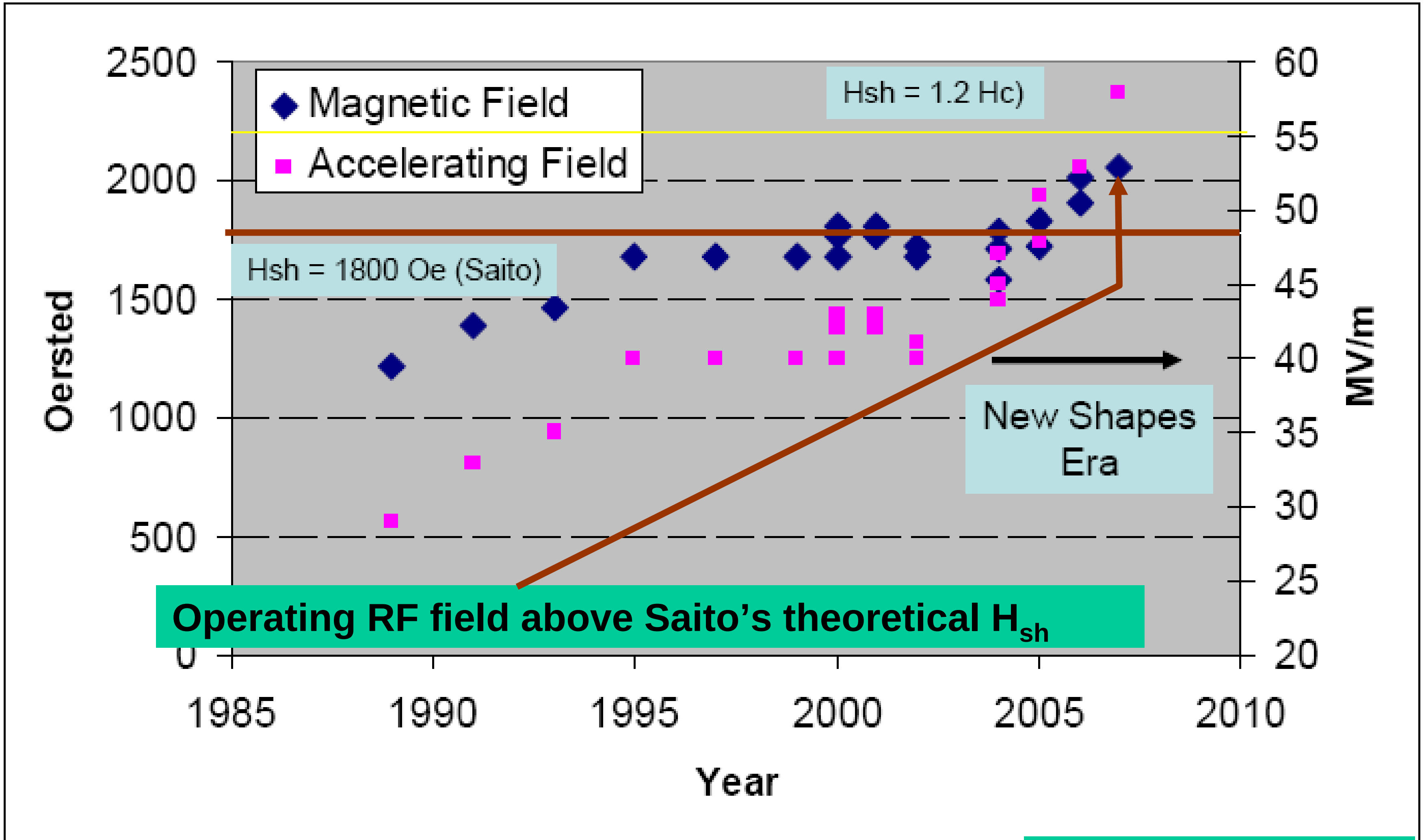
Workshop organized by <u>H. Edwards, C. Antoine,</u> G. Wu, supported by Technical Division, Fermilab

- 73 registered participants from 5 national labs, 11 universities, 1 oversea national lab and 7 private businesses.
- 5 main topics.
  - Fundamentals of RF superconductivity
  - Materials properties and surface characterizations
  - New materials
  - Innovative processing of materials
  - Production of niobium
- 34 talks (9 review talks) in two days.

Highlights of new things

- Fundamental limitations
  - Framework for superheated critical field by Sethna and Padamsee
  - High field vortex dynamics by Gurevich
- Multi-layer theory and experiment is converging
  - Gurevich, Pellin et al., and Xi
- Oxygen as a pollutant, and the role of baking
  - Tian, Romenenko, and Eremeev
- New approaches to surface characterization
  - Microwave microscopy (Anlage et al., Wu et al.)
  - Tunneling spectroscopy (Zasadzinski et al.)
  - Studies of isolated grain boundaries (Sung et al.)
- Alternative processing techniques
  - Gas Cluster Ion Beam (GCIB) processing (Swenson et al.)
  - Plasma processing for dry etching (Raskovic et al., Wu et al.)
  - Acid-free surface polishing solutions (Crooks et al.)
  - Ultra-smooth Chemical mechanical polishing (Muftu et al.)

High light 1: Physics of the Ultimate RF critical magnetic field (superheating field)



Operating RF field above Saito's theoretical H_{sh}

Padamsee, Sethna

High light 1: Physics of the Ultimate RF critical magnetic field (superheating field)

Energy balance argument:

- Vortex line nucleation model gives an upper bound on the *equilibrium* critical field for vortex penetration, $\propto H_{c1}$
- Energy balance does not discuss meta-stability
- Model is useful for inhomogeneities on the scale of the coherence length, but not as a fundamental limit for uniform, flat, pure superconductors

Line nucleation model inappropriate:

- Superheating is a prediction from the Abrikosov, G-L theory. It applies for $T \sim T_c$.
- Hysteresis (pinning) in magnetization gives unreliable answers for H_{c1} and H_c
- Hsh-rf = $\sqrt{2}$ Hsh-dc incorrect for phase transition field

Padamsee, Sethna

High light 1: Physics of the Ultimate RF critical magnetic field (superheating field)

Eilenberger/Gorkov for H_{sh} in Nb

James P. Sethna, Gianluigi Catelani

$$\left[\omega + \boldsymbol{n}\cdot\left(\boldsymbol{\nabla} - i\frac{2\pi\xi_0}{\phi_0}\boldsymbol{A}(\boldsymbol{r})\right)\right] f(\omega,\boldsymbol{n},\boldsymbol{r}) = \Delta(\boldsymbol{r})g(\omega,\boldsymbol{n},\boldsymbol{r})$$

$$\left[\omega - \boldsymbol{n}\cdot\left(\boldsymbol{\nabla} + i\frac{2\pi\xi_0}{\phi_0}\boldsymbol{A}(\boldsymbol{r})\right)\right] \bar{f}(\omega,\boldsymbol{n},\boldsymbol{r}) = \Delta^\dagger(\boldsymbol{r})g(\omega,\boldsymbol{n},\boldsymbol{r})$$

- Equations of motion for the (anomalous) Green's functions $f(\omega,\boldsymbol{n},x)$ and $g(\omega,\boldsymbol{n},x)$

$$\Delta(\boldsymbol{r})\log\left(\frac{T}{T_c}\right) + 2\pi T\sum\left[\frac{\Delta(\boldsymbol{r})}{\omega} - \int\frac{d\boldsymbol{n}}{4\pi}f(\omega,\boldsymbol{n},\boldsymbol{r})\right] = 0$$

- Self-consistent equation for gap Δ

$$\frac{2\pi\xi_0}{\phi_0}\boldsymbol{\nabla}\times\boldsymbol{H} + i\left(\frac{\xi_0}{\lambda_0}\right)^2 2\pi T\sum\int\frac{d\boldsymbol{n}}{4\pi}3\boldsymbol{n}g(\omega,\boldsymbol{n},\boldsymbol{r}) = 0$$

- Maxwell equation for H from current

$$g^2(\omega) + f(\omega)\bar{f}(\omega) = 1$$

- Constraint on the Green's function

Matsubara frequencies ω and Fermi wavevector direction $\boldsymbol{n}$

Framework set to solve it …nducting state

Find 2D instability threshold H_{sh} (functional eigenvalu…

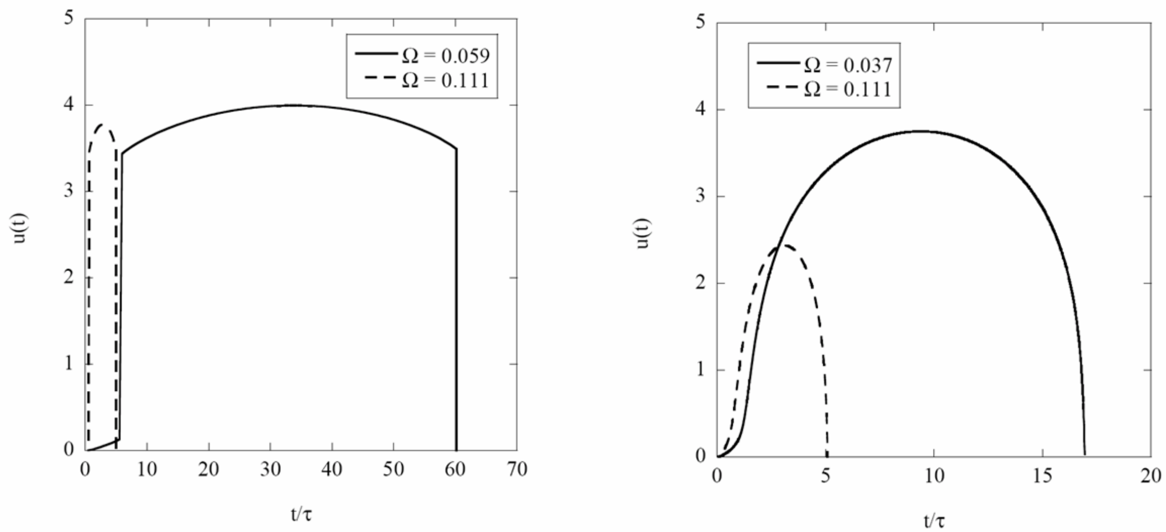
Padamsee, Sethna

Highlight 1: High field vortex dynamics

Jumpwise vortex penetration and exit

- Vortex jumps over the distance $\sim(3\text{-}4)\lambda \sim 120\text{-}160$ nm
- Dissipation is dominated by the jumps

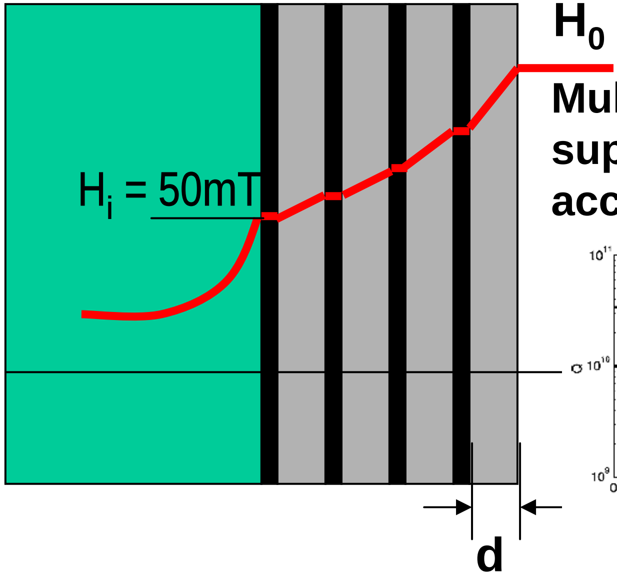
Stable vortex penetration may occur
Only at low fields $H \ll H_c$



viscous vortex drag is not applicable for high fields
Dissipation is dominated by the jumps

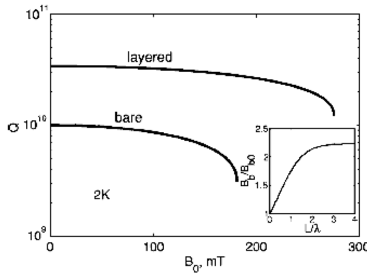
A. Gurevich

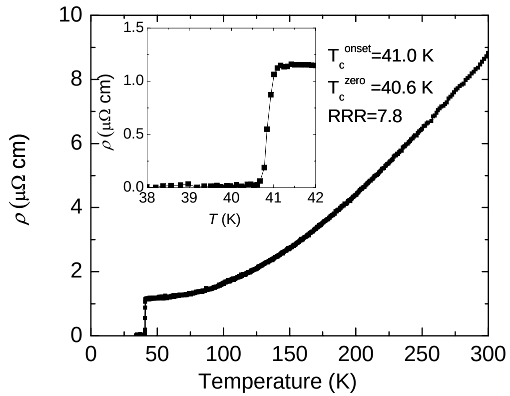
Highlight 2: Multi-layer theory and experiment is converging



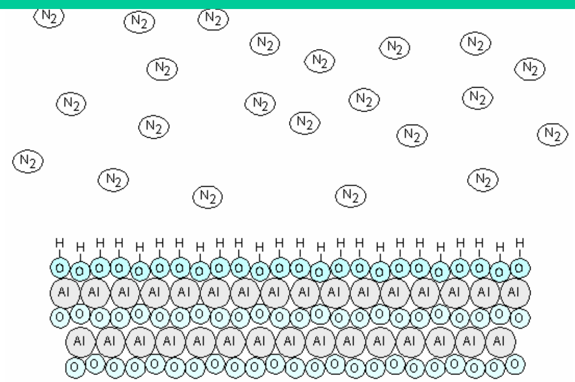
H_0 = 2T

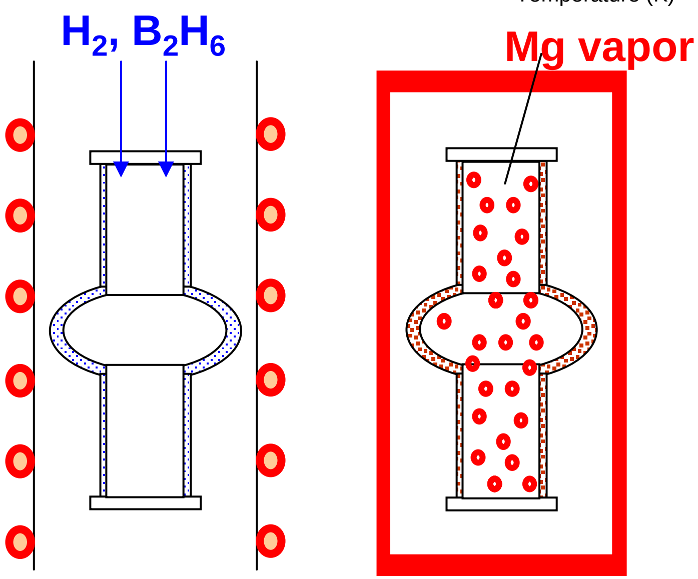
Multi-layer superconductor supports exceptionally high accelerating gradient with higher Q_0





Atomic layer deposition is very attractive for multi-layer coating –ANL (TUP64)

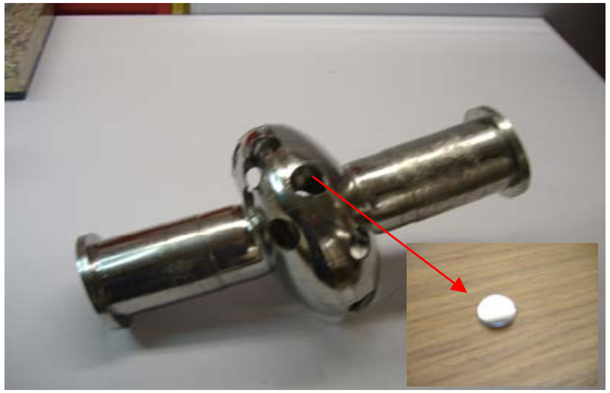




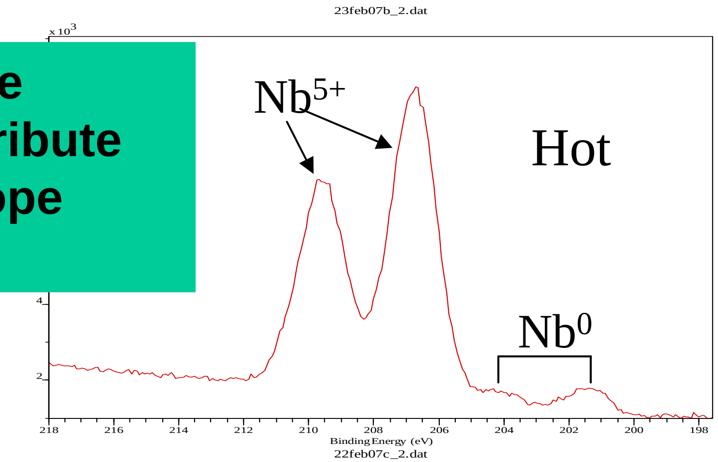
Two step MgB2 coating possible –Penn State

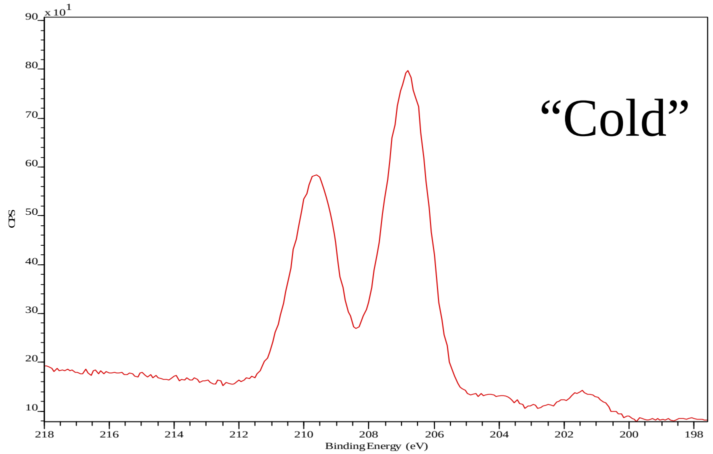
Gurevich, Pellin, Xi

Highlight 3: Oxide, Baking and High field Q-slop

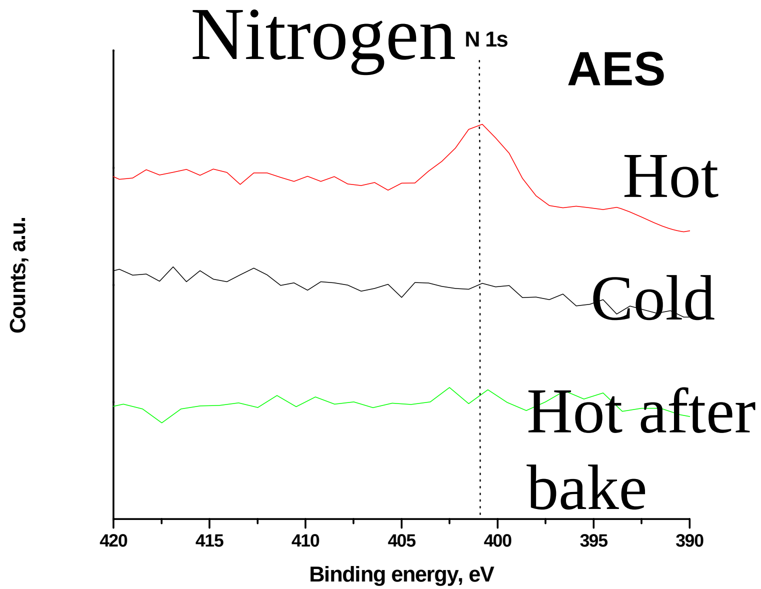


Oxide and metal/oxide interface do not contribute to the high field Q-slope losses





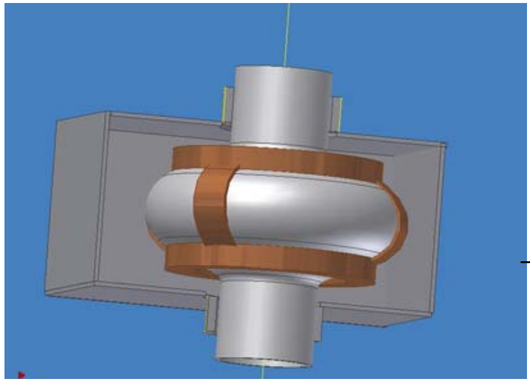
XPS



Nitrogen signal noticed

Cut out studies by Romanenko

Highlight 3: Oxide, Baking and High field Q-slop



Mainly Nb and a little NbO after HT baking

Sample verification

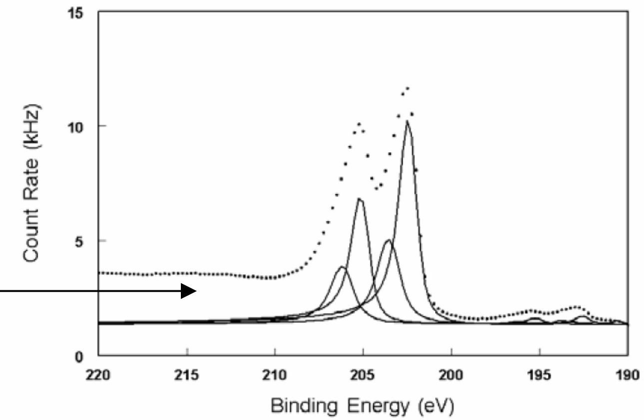
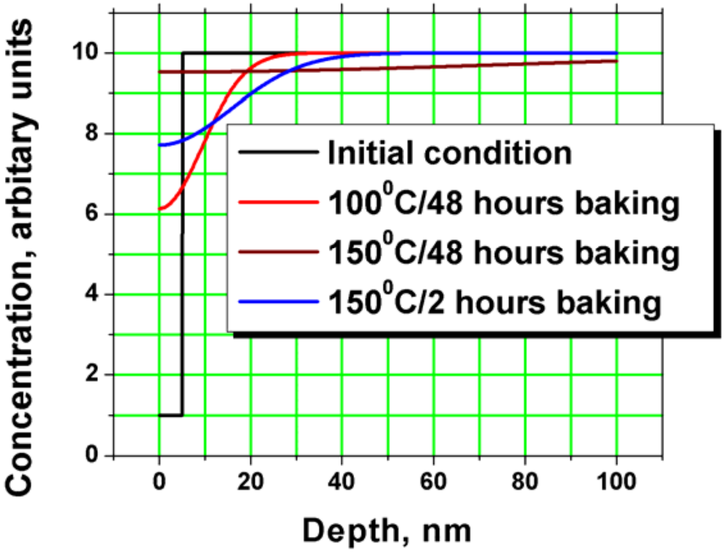
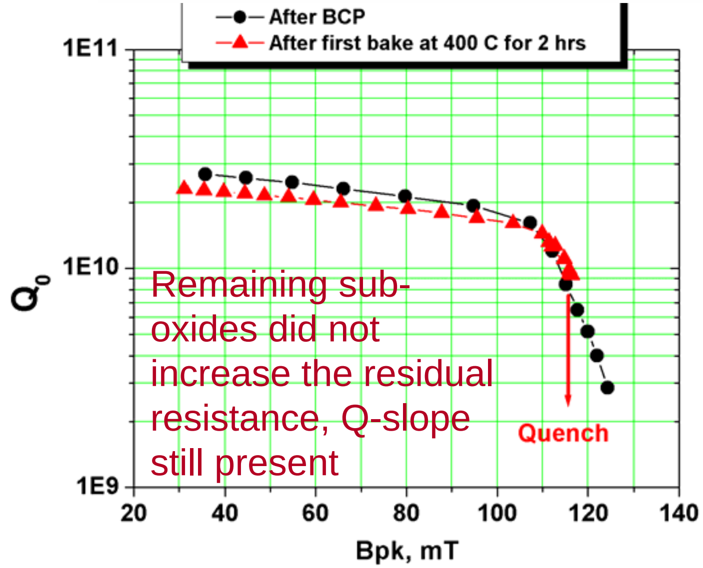


Figure 10. Curve fitted spectrum of sample #2, at 500°C, showing just two components, Nb^0 (metal) and Nb^{1+}.

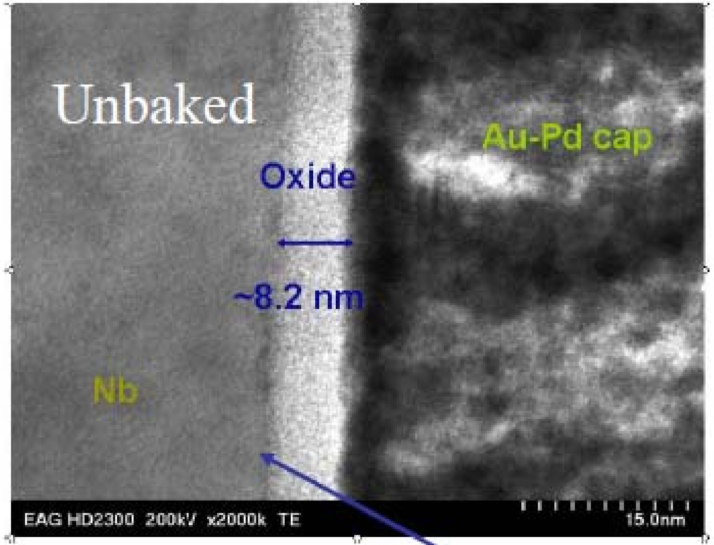


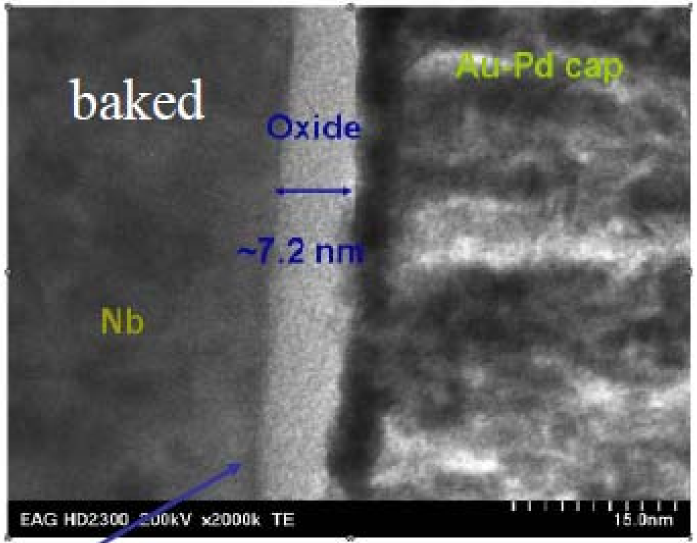


The bulk diffusion rates may not be applicable for diffusion near the surface

High T baking studies by Eremeev

Highlight 3: Oxide, Baking and High field Q-slop





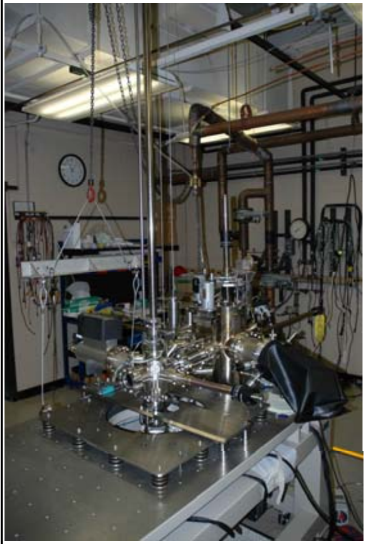
No clear structure change information can be observed (sub-oxide , interstitial oxygen)…

Preliminary

In-situ baked cavity remains good after air-exposure. Coupled with XPS data, one concludes sub-oxide not contributing High field Q-slope

Baking studies and TEM, XPS by Tian

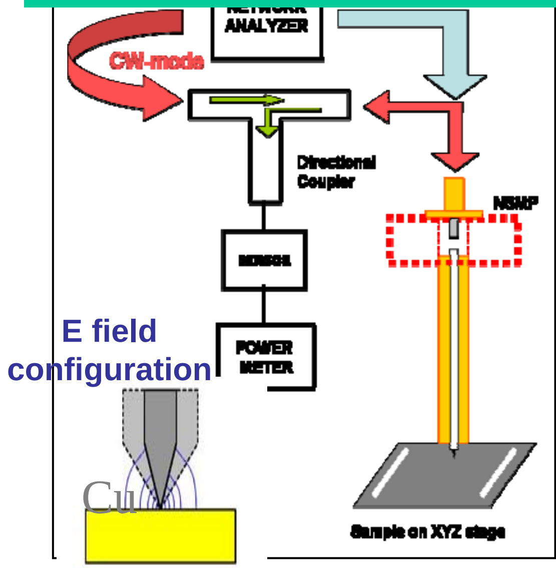
Highlight 4: New approaches for surface characterization

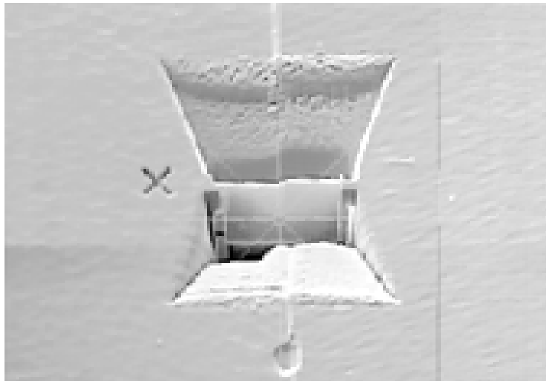


Low Temp. STM at Argonne:
- T=4.2 K, H=7 T
- e-beam for tip preparation
- Ion gun for surface preparation
- Cleaving stage in UHV
- in-situ transfer of sample and tip
- LEED/Auger
- Multi-source e-beam evaporator

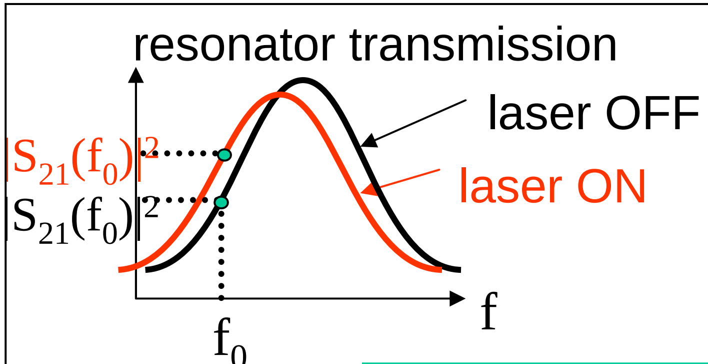
Tunneling spectroscopy
Zasadzinski et al., ANL

Near-field scanning microwave microscopy, Wu et al., Kansas State Univ





FIB prepared sample for GB studies
EELS for Oxides or micro-chemical Analysis
Sung et al. at Florida State University, WE105

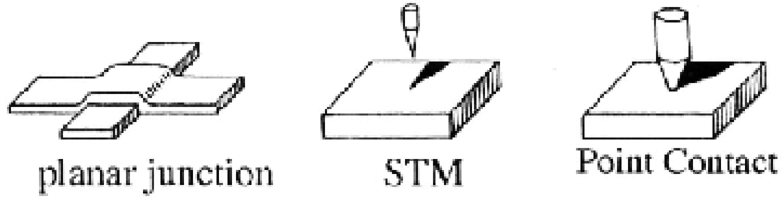


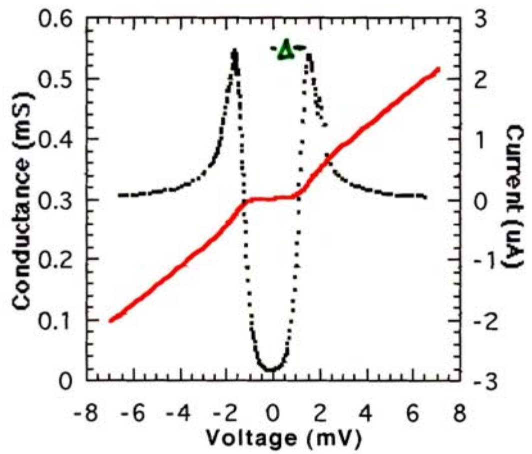
$\Delta|S_{12}|^2 \sim [J_{RF}(x,y)]^2$

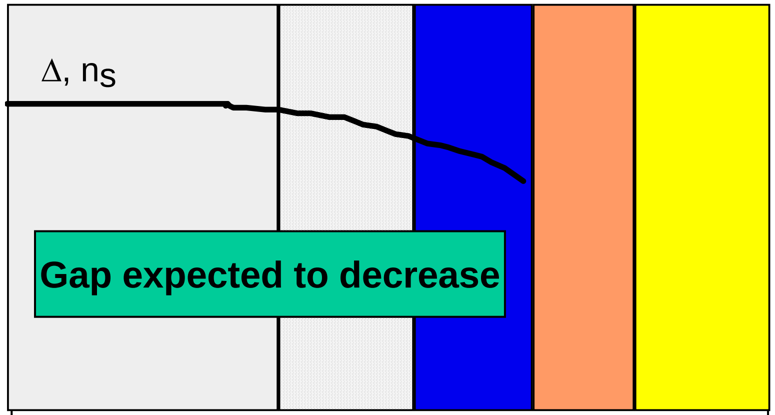
Laser Scanning Microscope
Anlage et al., University of Maryland

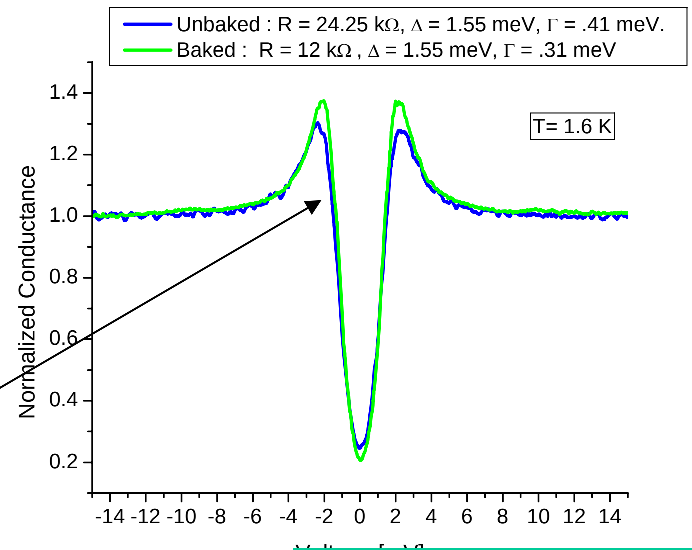
Highlight 4: New approaches for surface characterization

Tunneling spectroscopy









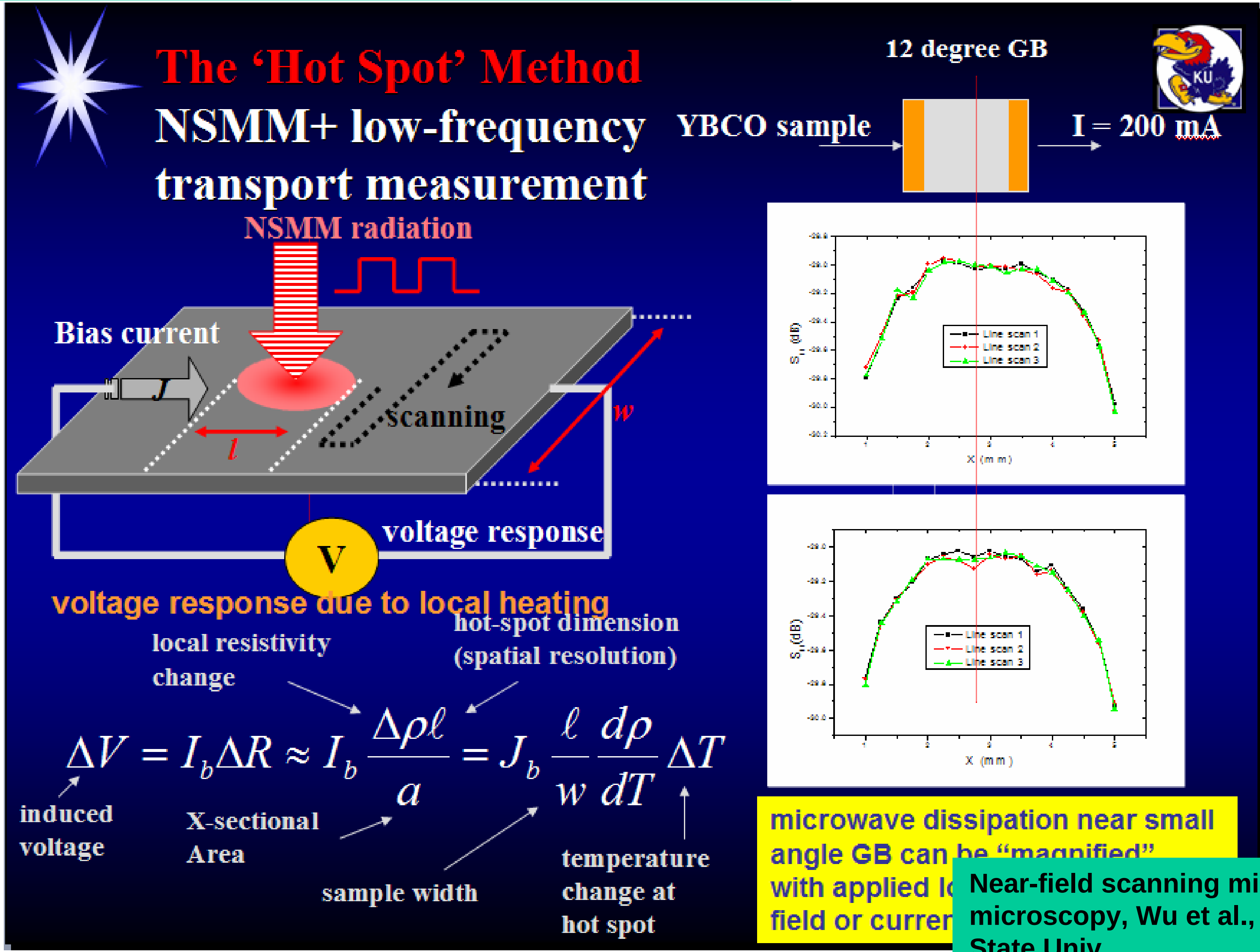
Studies on baked/unbaked sample

Preliminary result show: change in gap was not evident. Inelastic behavior of interface changed.

Oxide layer limited or no role?

J. Zasadzinski

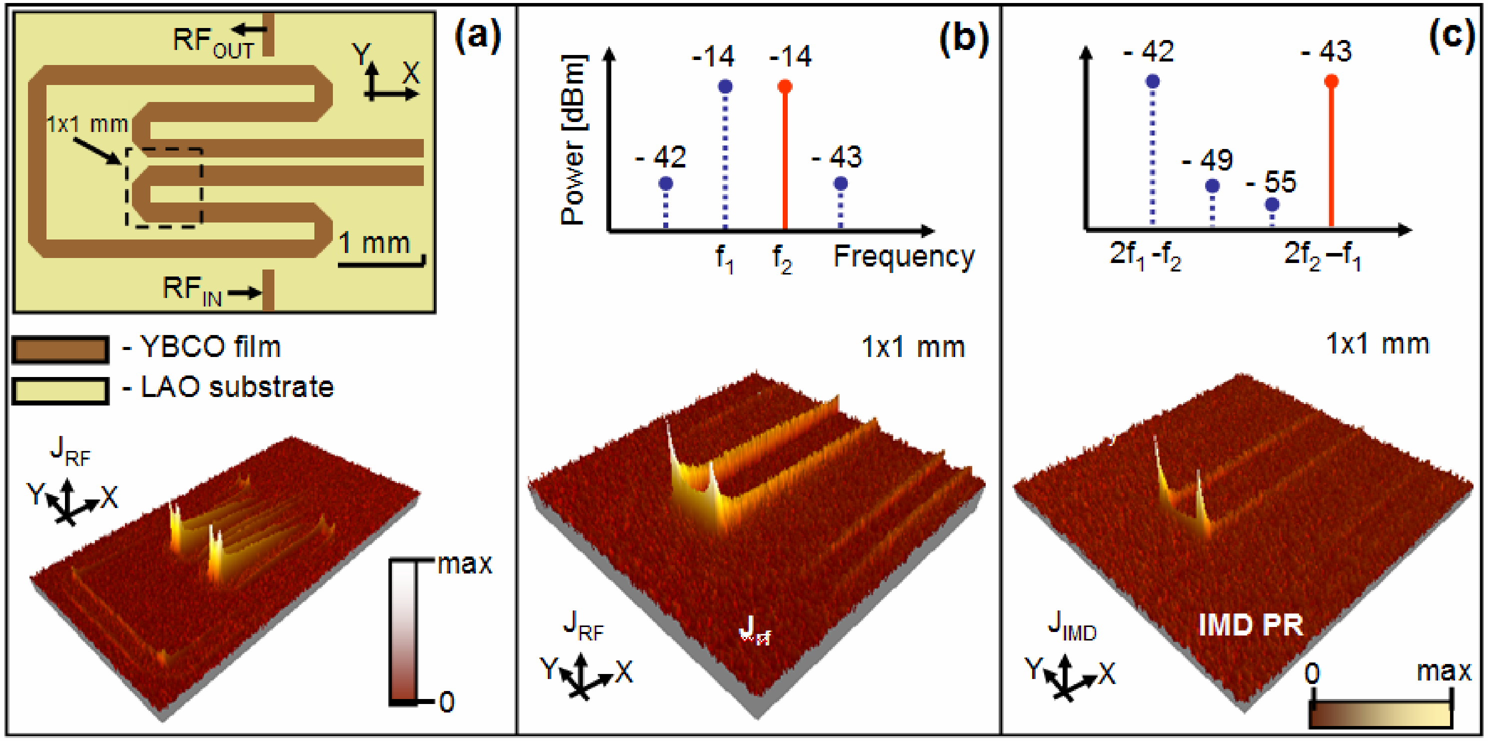
Highlight 4: New approaches for surface characterization



Near-field scanning microwave microscopy, Wu et al., Kansas State Univ

Highlight 4: New approaches for surface characterization

Spatially-resolved microwave photo-response



(a) Top view of the resonator topology along with overall and (b, c) detailed 1x1 mm 3-d LTLSM plots (bottom images) showing (b) $J_{RF}(x,y)$ and (c) IMD PR distribution. The upper part of (b) shows the two input tones at –14 dBm as well as the output tones. The upper part of (c) shows the signals entering the spectrum analyzer after the primary tones have suffered partial cancellation.

A.P. Zhuravel, S. M. Anlage and A.V. Ustinov

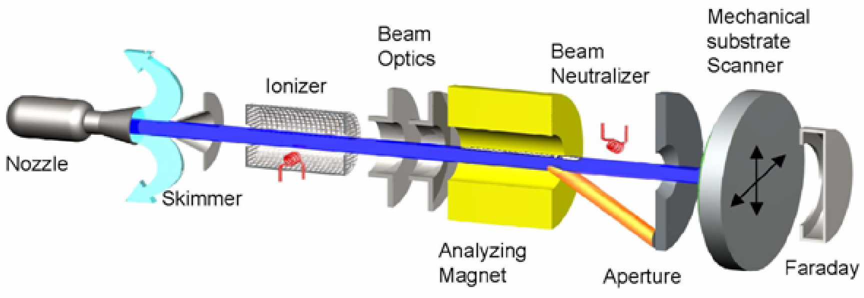
Imaging of Microw
Sources of Nonlin
Resonators

20

Laser Scanning Microscope
Anlage et al., University of Maryland

Highlight 5: Alternative processing techniques

Gas Cluster Ion Beams



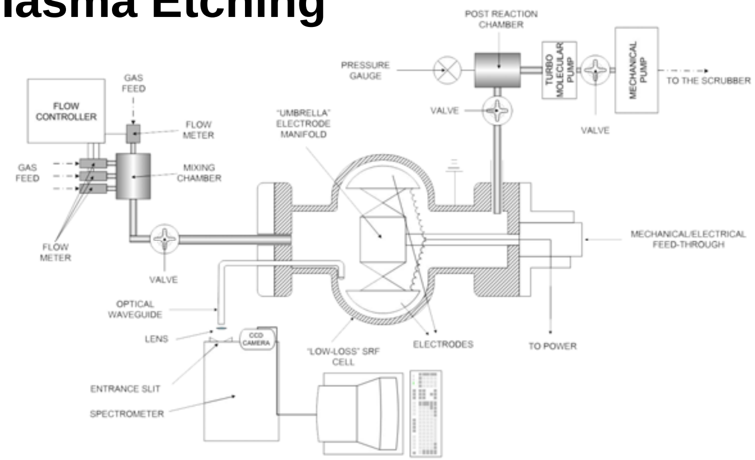
D. Swenson et al., TEL E-pion Inc

Alternative EP

Sample	Chemistry
BK-2	5.3g LiCl, 11g $Mg(ClO_4)_2$,500ml CH3OH, 250ml b.c.*
G1	11g $Mg(ClO_4)_2$,500ml CH_3OH
EP	$HF:H_2SO_4$; 1:9 (by volume) [Compton & Saxton (MSU), Fermilab, 2005]
ECUA	Choline Chloride, Urea, NH_4F + lower viscosity organic solvent

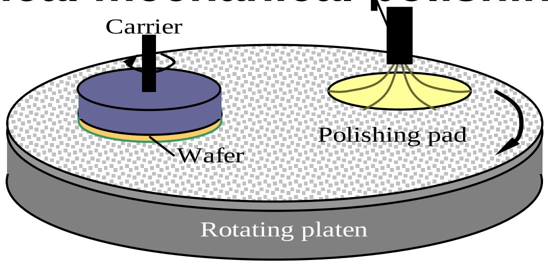
R. Crooks, Black Laboratory, LLC

Plasma Etching



M. Raskovic et al., Old Dominion University

Chemical mechanical polishing

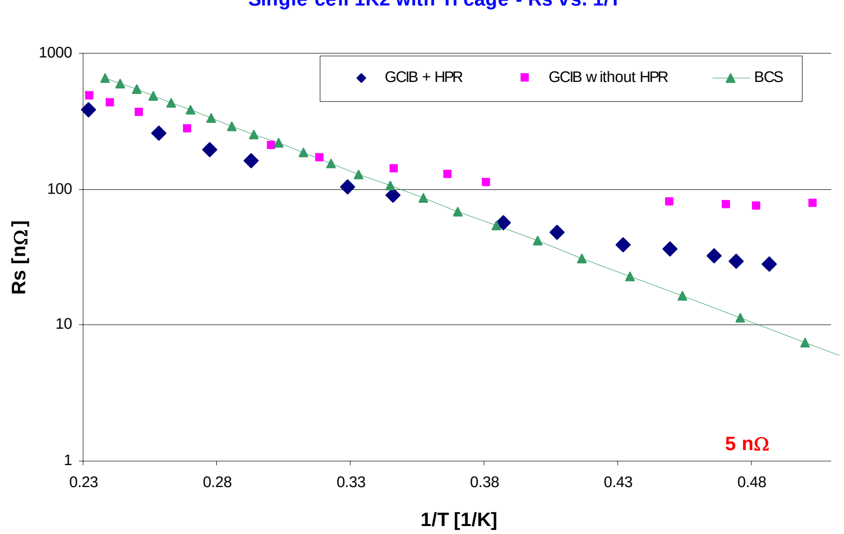


Schematic of CMP operation

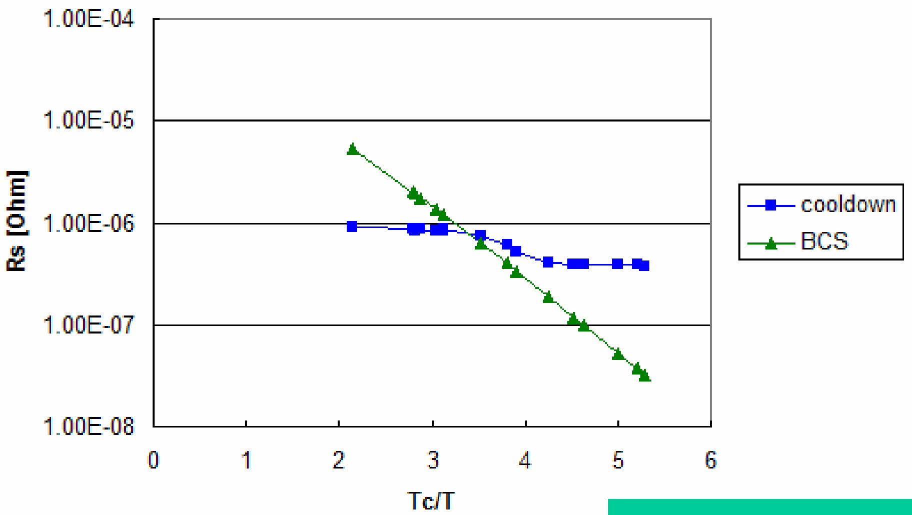
Material removal occurs due to ***particle abrasion*** of the ***chemically passivated*** *wafer surface*

S. Muftu et al., Northeastern University

Highlight 5: Alternative processing techniques



1.3GHz at Jlab



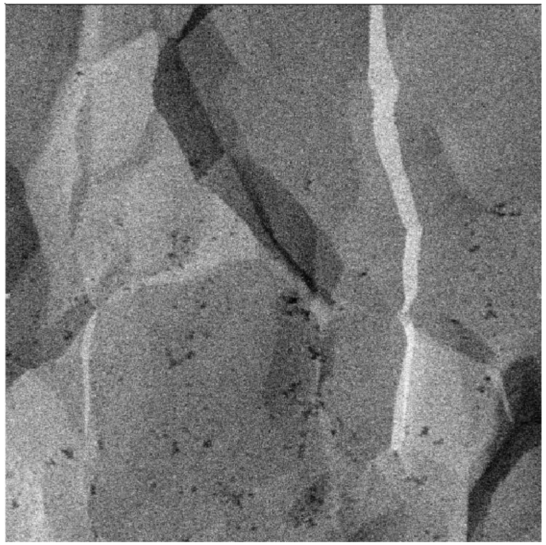
3.9GHz at Fermilab

D.R. Swenson, et. al, SRF Materials Workshop, Fermilab, 23 May 2007

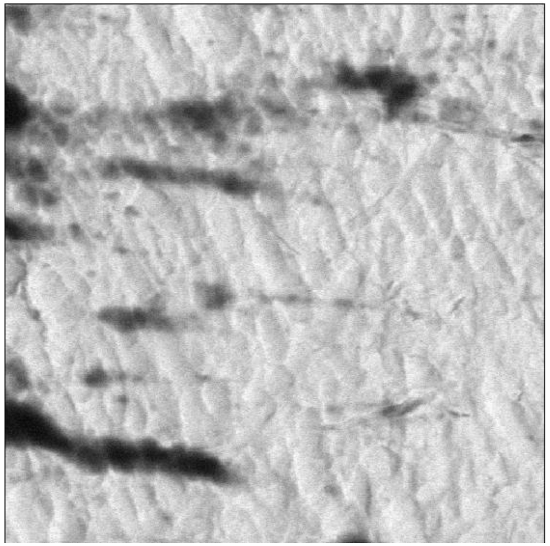
Gas Cluster Ion Beams, D. Swenson

Highlight 5: Alternative processing techniques

Morphology



BCP-treated



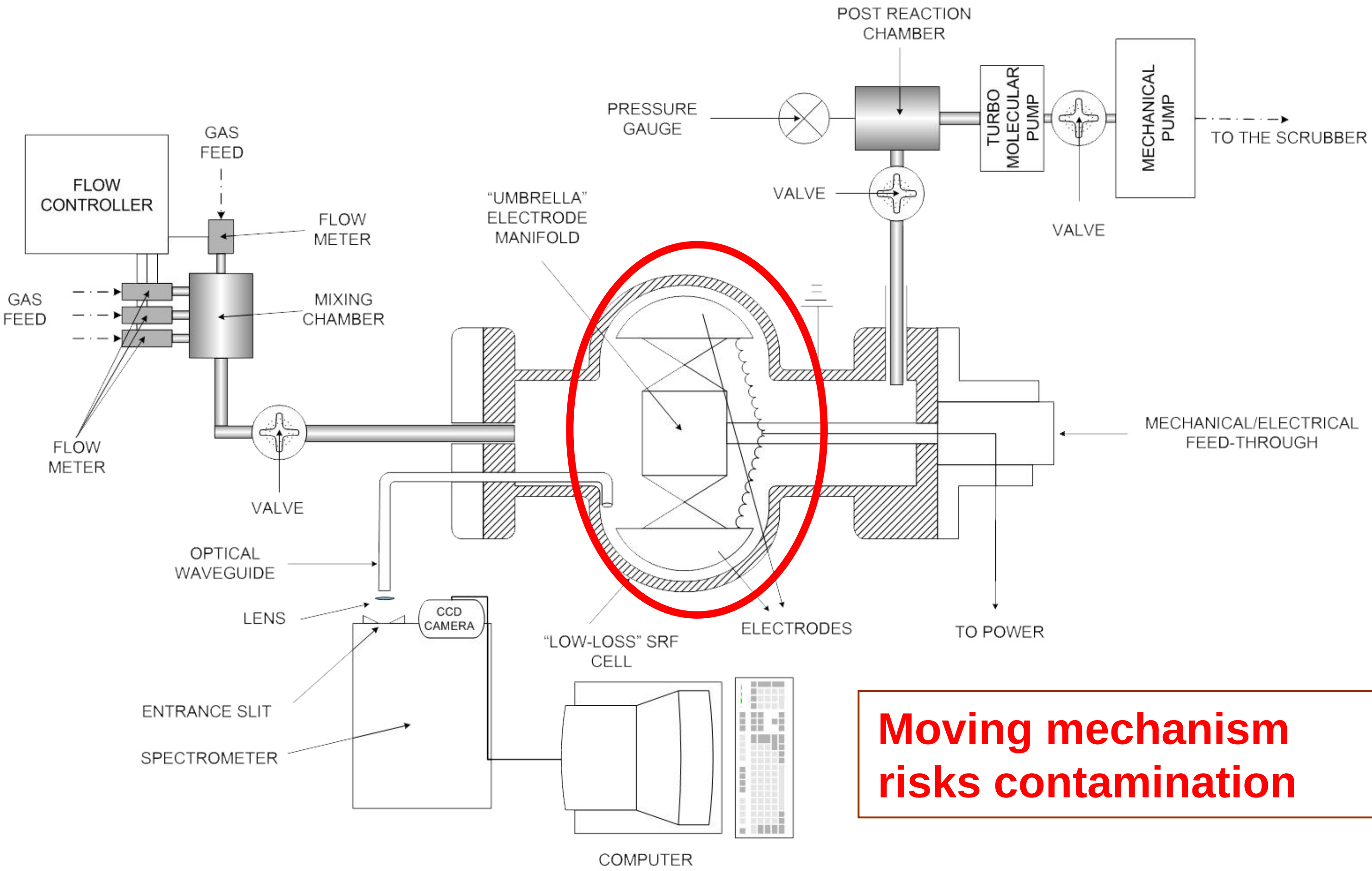
Plasma-treated (Sample No.1)

Waviness (W_a) and roughness (R_a) of the samples' surface determined by profilometer.

Sample	W_a (Å)	W_{RMS} (Å)	R_a (μm)	R_{RMS} (μm)
untreated	235	285	0.930	1.125
BCP	141	169	0.323	0.476
1 - plasma	20	26	0.246	0.309
2 - plasma	23	27	0.195	0.240
3 - plasma	11	16	0.295	0.353

Plasma etching, M. Raskovic (TUP73)

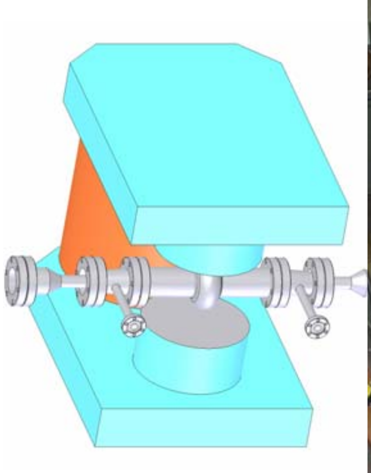
Highlight 5: Alternative processing techniques

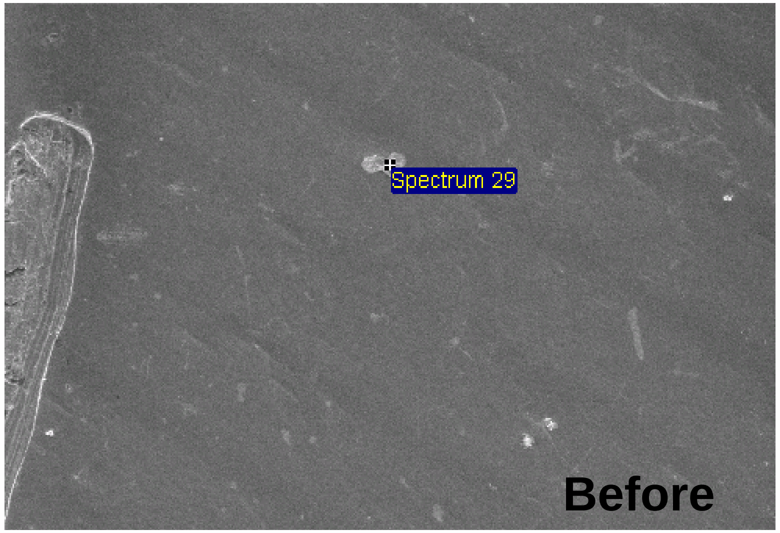


Moving mechanism risks contamination

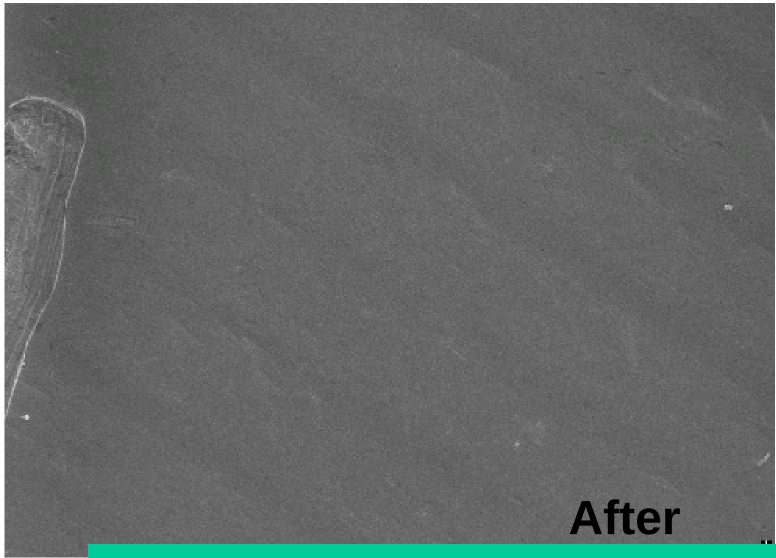
Plasma etching, M. Raskovic et al.

Highlight 5: Alternative processing techniques





Marked contaminated witness sample inserted into the cavity



ECR plasma cleans away most of the sulfur in just 30 minutes

Plasma cleaning, G. Wu et al.

Highlight 5: Alternative processing techniques

Experimental Conditions for Electropolishing

	Sample	Chemistry	T, ºC	V	mA/cm2
Salt 1	BK-2	5.3g LiCl, 11g $Mg(ClO_4)_2$,500ml CH3OH, 250ml b.c.*	-78	60	50
Salt 2	G1	11g $Mg(ClO_4)_2$,500ml CH_3OH	-78	30	40
	EP	$HF:H_2SO_4$; 1:9 (by volume) [Compton & Saxton (MSU), Fermilab, 2005]	40	10	50
Salt 3	ECUA	Choline Chloride, Urea, NH_4F + lower viscosity organic solvent	70	12	50

Electropolishing was performed under current control, with simple two-electrode System with a stainless steel cathode. Degreased in soapy water. Temperature control (dry ice/methanol bath) was used for first two.

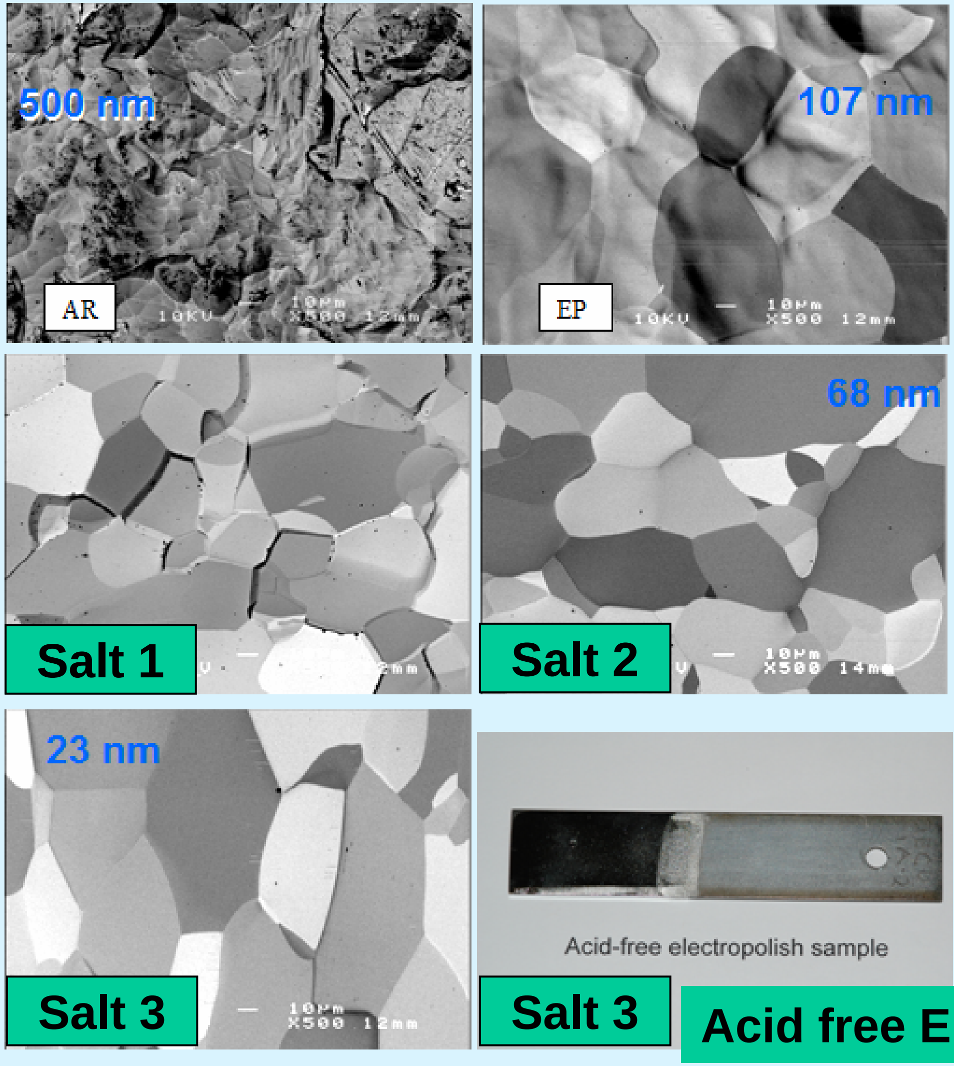
Current density of 50 mA/cm^2 corresponded to a removal rate of ~ 1 μm/min

Acid free EP, R. Crooks

Highlight 5: Alternative processing techniques

Channeling Contrast (backscattered) Electron Images and Ra values

Rolling Plane
(plan view)



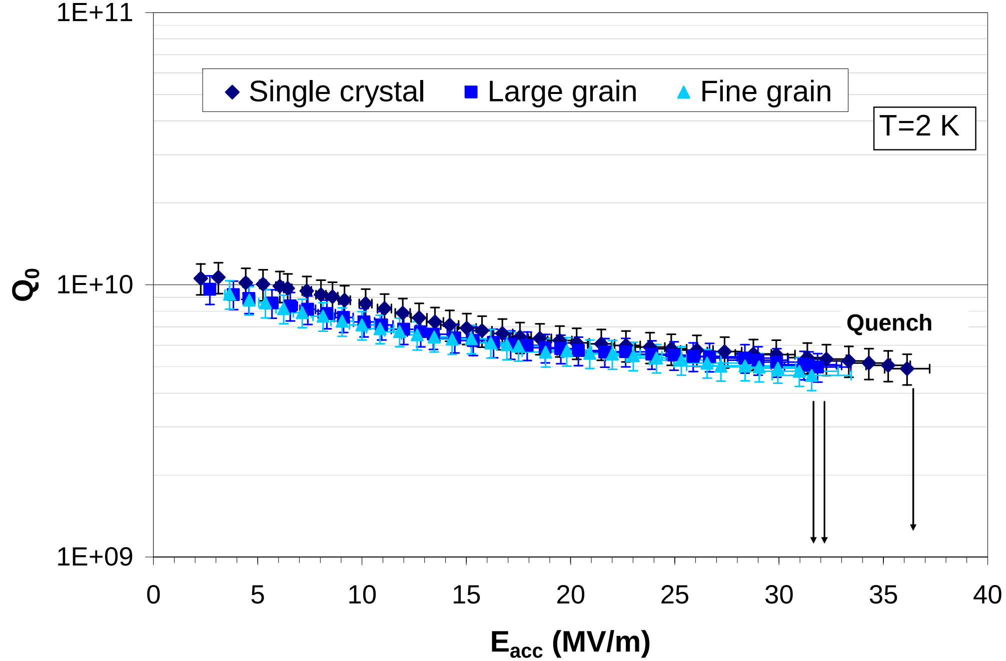
1 hour
Electropolish
AR RRR Nb

Several cm^2
electropolished

Acid free EP, R. Crooks

Highlights of debate

- Flux line penetration (AG) vs superheating (HP)
- GB problem or not?
- Heat transfer across interfaces?
- Are there ways to reduce the present requirements of near perfection?



G. Ciovati et al. Proc. of the 2006 LINAC, paper TUP033

Highlights of debate

- Medium field region (Linear) is still interesting subject
- Lack of broad understanding vs. interesting individual results
- Problem areas are local, but over what scale? … and what probe is best?
- Samples vs. cavities
- Will new material/coating ever deliver comparable results to solid niobium?

Highlights of debate

- Are oxides bad?
- Processing: show-and-tell, but no in-depth debate
- Single crystal vs. very small grains
- Are there advantages for certain Nb texture?
- Are specifications for niobium adequate?
  - Does the specification determine a starting point from which imperfections in the process subtract?

- New scientific results at SRF2007: ANL, FSU, MSU, W&M, FNAL, JLab, etc.

- Next meeting will be in spring 2008

  - Thanks to C. Antoine, L. Cooley

- Thank you

- Back up slides

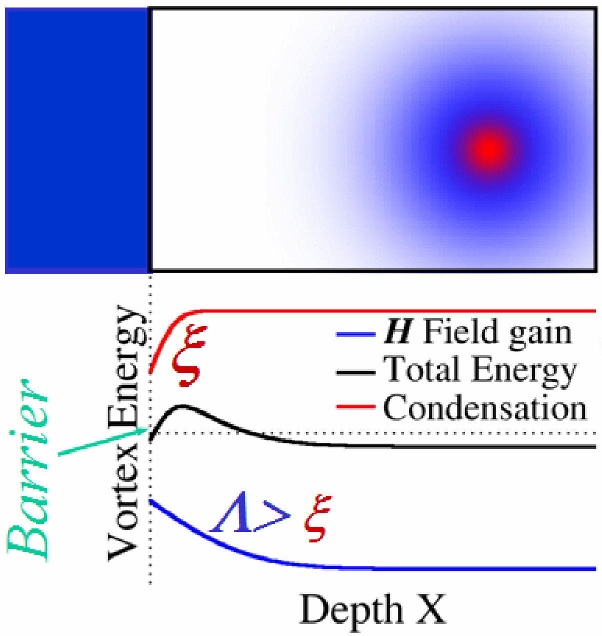
Physics of the Ultimate RF critical magnetic field (superheating field)

Fundamental limits to H_{sh} in Niobium

What can theory tell us?

James P. Sethna, Gianluigi Catelani

Why a superheating field?

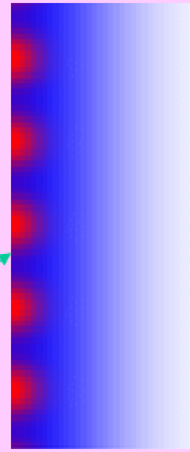


Costly core ξ enters first; gain from field Λ later

How to calculate H_{sh}?

- Field where barrier vanishes
- Linear stability analysis *determines* nucleation mechanism: vortex array
- At large κ and $T \sim T_c$, 1-D analysis

$$H_{sh} = 0.745\, H_c$$



"Line nucleation" *wrong*

- Yogi, Saito $H_{sh} \sim H_c/\kappa$ discouraging
- Via "energy balance", no barrier calculation
- Does not work for large κ,

$$H_{sh} < H_{c1} = H_c \ln(\kappa)/(\sqrt{2}\,\kappa)$$

- Correct balance theory gives H_{c1} not H_{sh}

Physics of the Ultimate RF critical magnetic field (superheating field)

Eilenberger/Gorkov for H_{sh} in Nb

James P. Sethna, Gianluigi Catelani

$$\left[\omega + \boldsymbol{n}\cdot\left(\boldsymbol{\nabla} - i\frac{2\pi\xi_0}{\phi_0}\boldsymbol{A}(\boldsymbol{r})\right)\right] f(\omega,\boldsymbol{n},\boldsymbol{r}) = \Delta(\boldsymbol{r})g(\omega,\boldsymbol{n},\boldsymbol{r})$$

$$\left[\omega - \boldsymbol{n}\cdot\left(\boldsymbol{\nabla} + i\frac{2\pi\xi_0}{\phi_0}\boldsymbol{A}(\boldsymbol{r})\right)\right] \bar{f}(\omega,\boldsymbol{n},\boldsymbol{r}) = \Delta^\dagger(\boldsymbol{r})g(\omega,\boldsymbol{n},\boldsymbol{r})$$

- Equations of motion for the (anomalous) Green's functions *f(ω,**n**,x)* and *g(ω,**n**,x)*

$$\Delta(\boldsymbol{r})\log\left(\frac{T}{T_c}\right) + 2\pi T\sum\left[\frac{\Delta(\boldsymbol{r})}{\omega} - \int\frac{dn}{4\pi}f(\omega,\boldsymbol{n},\boldsymbol{r})\right] = 0$$

- Self-consistent equation for gap Δ

$$\frac{2\pi\xi_0}{\phi_0}\boldsymbol{\nabla}\times\boldsymbol{H} + i\left(\frac{\xi_0}{\lambda_0}\right)^2 2\pi T\sum\int\frac{dn}{4\pi}3\boldsymbol{n}g(\omega,\boldsymbol{n},\boldsymbol{r}) = 0$$

- Maxwell equation for H from current

$$g^2(\omega) + f(\omega)\bar{f}(\omega) = 1$$

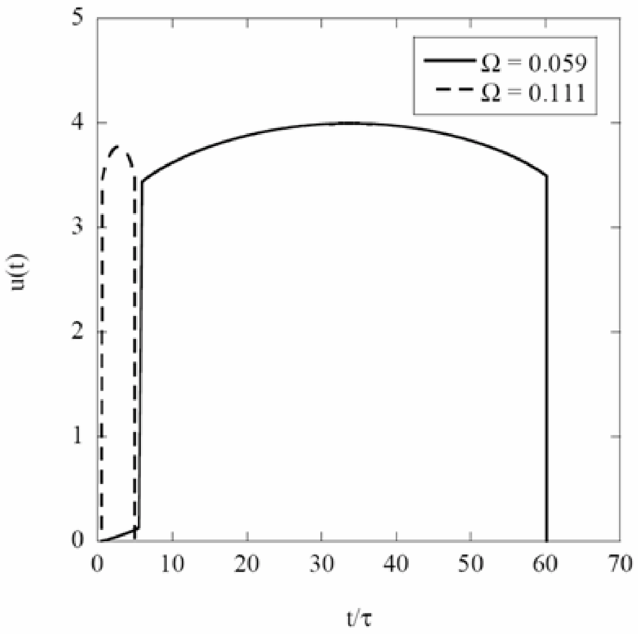
- Constraint on the Green's function

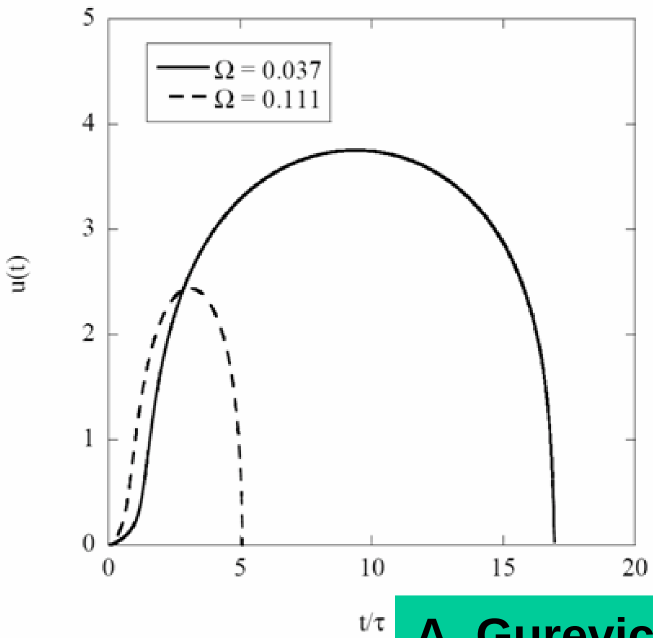
Matsubara frequencies ω and Fermi wavevector direction $\boldsymbol{n}$
Solve 1D ODE for uniform, superconducting state
Find 2D instability threshold H_{sh} (functional eigenvalue crosses zero)

Jumpwise vortex penetration and exit

- Vortex jumps over the distance $\sim(3\text{-}4)\lambda \sim$ 120-160 nm
- Dissipation is dominated by the jumps

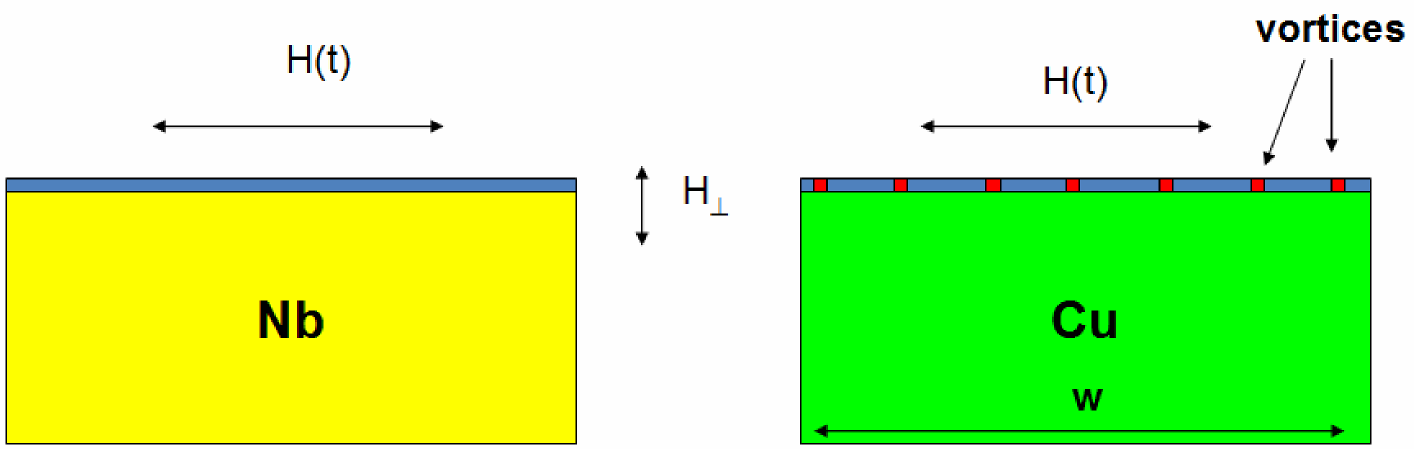
Stable vortex penetration may occur Only at low fields $H << H_c$





A. Gurevich

Why is Nb_3Sn on a Nb cavity much better than Nb_3Sn on a Cu cavity?



Nb_3Sn/Nb cavity is much better protected against vortices produced by small transverse stray fields $H_\perp$ than Nb_3Sn/Cu cavity

Meissner state persists up to $H_\perp < H_{c1}^{(Nb)}$

Meissner state is destroyed for small $H_\perp < (d/w)H_{c1}^{(Nb_3Sn)} \ll H_{c1}^{(Nb_3Sn)}$ due to large demagnetization factor $w/d \sim 10^3$-10^5

A. Gurevich

- **Highlights of new things**

Session 2: Material properties of superconductor & Surface Characterizations

- XPS studies suggested oxide layer not a major role in low-T baking (by Tian)
- Oxide plays no significant role in hot spot (Eremeev, Romanenko)
- Tunneling Spectroscopy studies of niobium baking (Zasadzinski)
- Grain boundary studies by TEM,EELS and FIB prep (Sung)
- Microwave photo response by Laser Scanning microscope (Anlage)
- Mushroom cavity for high power measurement (Tajima)
- Near-field Scanning Microwave Microscopy (J.Wu)
- Phonon peak recovery can provide added insurance for unexpected cavity quenches. (Chandrasekaran)

Surface oxide studies on solid Niobium for SRF Accelerators using variable photon energy XPS

Summary about low-T baking from XPS studies

- After baking , Nb_2O_5 is partially transformed into suboxides, the total oxide layer becomes thinner; but once air-exposure , the change of oxide layer observed after baking almost disappear –the suboxides created by in situ bake must be irrelevant to SRF cavity performance (cavities still kept good performance.)
- Comparing with 120 °C baking , higher baking temperature (160 °C) helps more oxide transformation ; 3 hrs , 160 °C baking almost same oxide transformation as that of 12 hrs , 120 °C baking.
- Air baking produce thick oxide layer (large grain cavities air baked at 120 °C about 3 ~ 6 hrs shows improvement of Q- drop at the high field)-ref .Gigi report @ j-lab
- Longer baking time at 120 °C (3hrs ~12hrs) increases Nb_2O_5 reduction & transformation (Large grain cavities after 3 hrs baking already shows performance improvement)– ref . Gigi 's report @ J-lab
- No observable change can be "detected " by XPS about metal Nb (expected to observe the contribution of interstitial oxygen) from above studies at unbaked, baked and air-exposure status . (besides XPS , no direct experimental results show clear evidences about observable interstitial oxygen changes between oxide layer and metal Nb available now)

So , from XPS studies , the changes in the Niobium oxide layer caused by low -T baking appear to not play a big role in cavities performance improvement

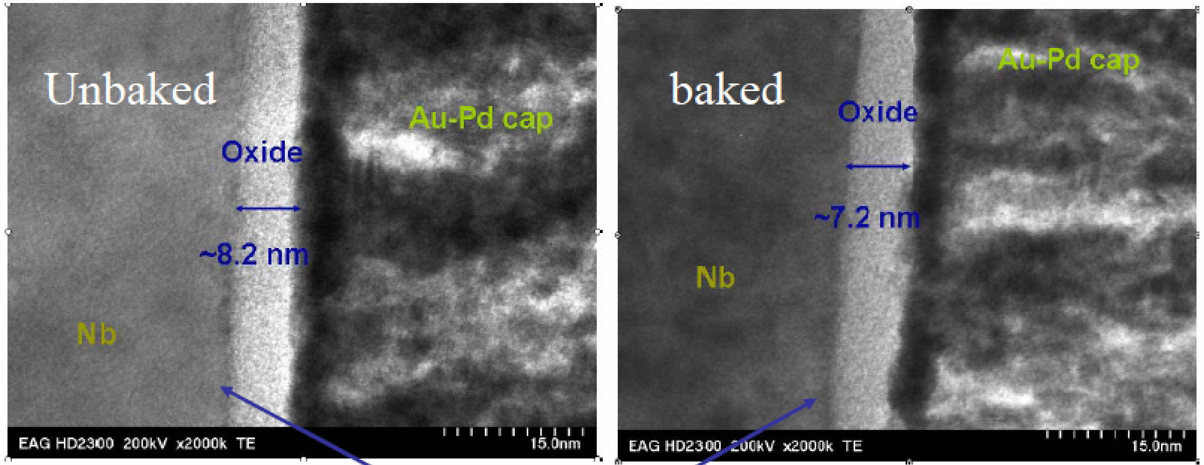
Jefferson Lab Thomas Jefferson National Accelerator Facility

Hui Tian , SRF Materials workshop , Fermi-lab, May 23-24, 2007

Surface oxide studies on solid Niobium for SRF Accelerators using variable photon energy XPS

Questions remain, further work is needed…

Oxides structure (unbaked /baked)- preliminary TEM results



No clear structure change information can be observed (sub-oxide , interstitial oxygen)…

Hydrogen ? Or something else?

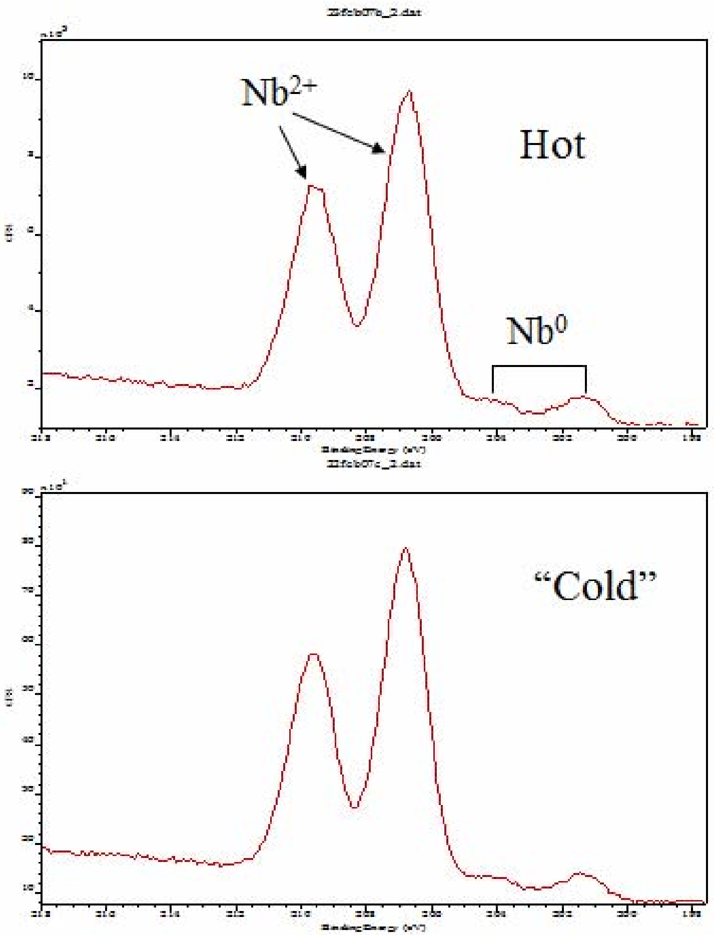
Jefferson Lab — Thomas Jefferson National Accelerator Facility

Hui Tian , SRF Materials workshop , Fermi-lab, May 23-24, 2007

Surface Analysis of samples dissected from a cavity with high-field Q slope

Cornell University
Laboratory for Elementary-Particle Physics

XPS Nb Hi Resolution



- Absolutely identical Nb high resolution spectra for all 19 samples (hot and "cold")
- No role of oxide and oxide/metal interface in the Q-slope

- Al Kα 1486.6 eV X-ray source
- Information depth ~7 nm, which includes oxide and interface

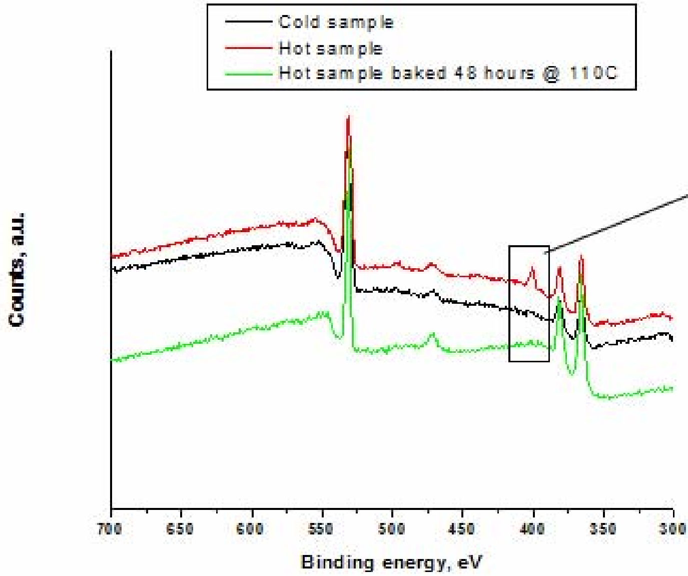
October 7, 2007 Alexander Romanenko 16

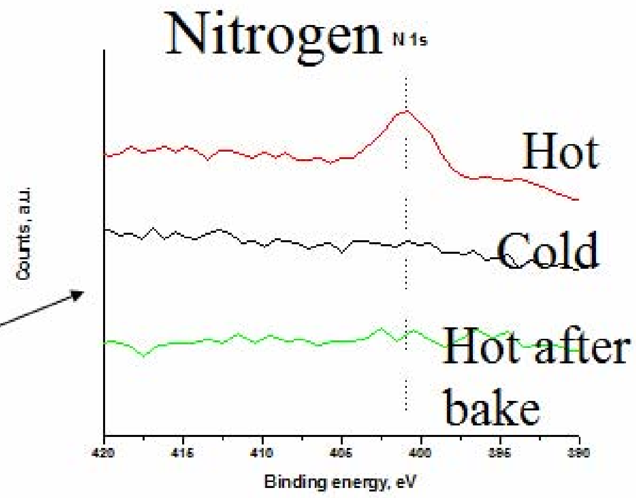
Surface Analysis of samples dissected from a cavity with high-field Q slope

Cornell University
Laboratory for Elementary-Particle Physics

XPS Results

- Al Kα 1486.6 eV X-ray source
- Information depth ~7 nm



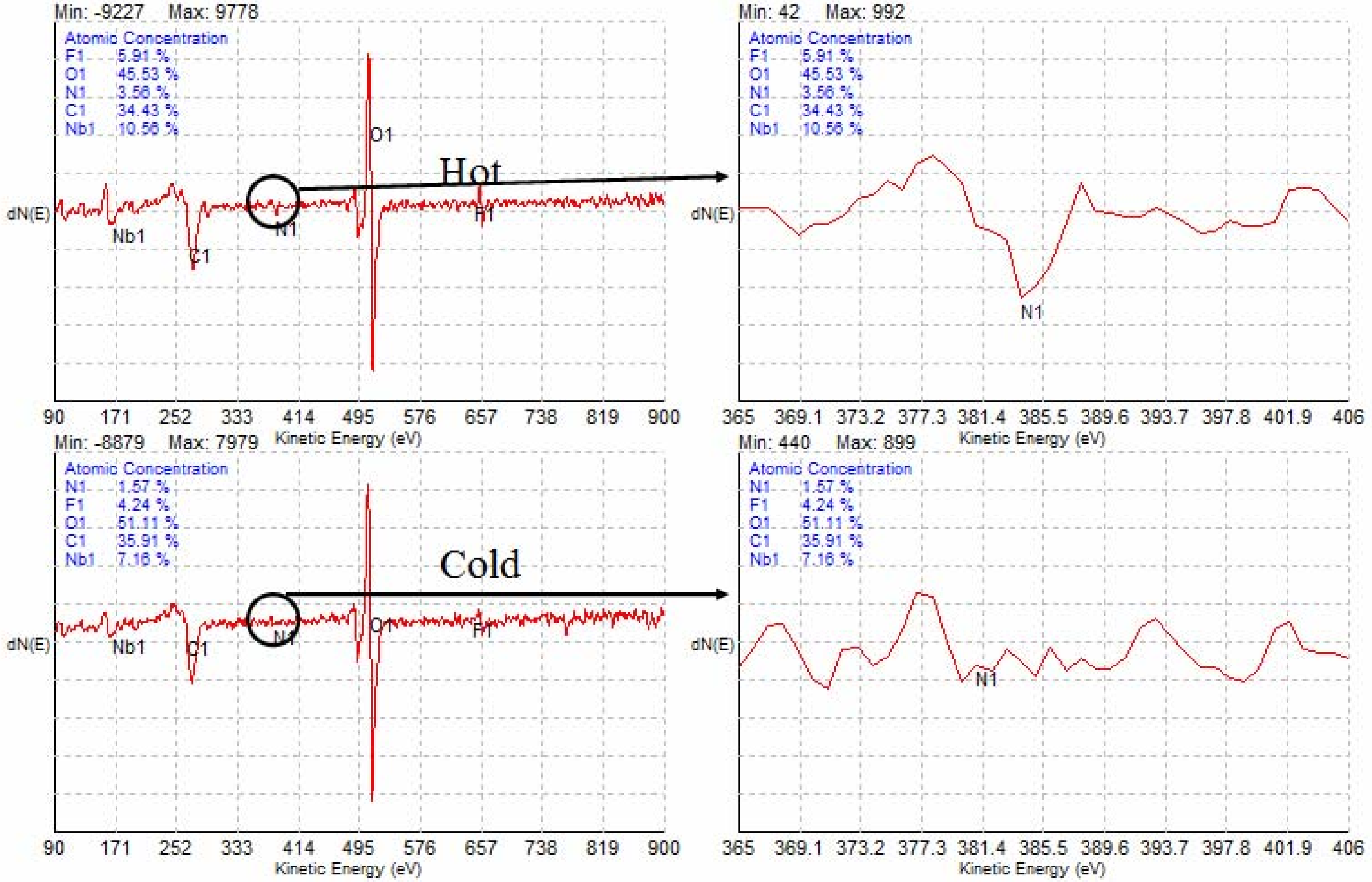


- Four different spots 1 mm^2 analyzed on each sample
- Three hottest samples out of 10 total revealed nitrogen presence in all 4 spots each
- Out of 9 "cold" samples one spot showed nitrogen
- Possible N chemical state – NO_3 (399 eV BE for free nitrogen, 401 eV for NO_3)
- Baking cavities helps reducing Q-slope
- Baking hottest sample @110^0C for 48 hours eliminated nitrogen signal

Surface Analysis of samples dissected from a cavity with high-field Q slope

Cornell University
Laboratory for Elementary-Particle Physics

AES Results



October 7, 2007 Alexander Romanenko 17

Correlation between XPS and temperature maps for nearly oxide-free niobium

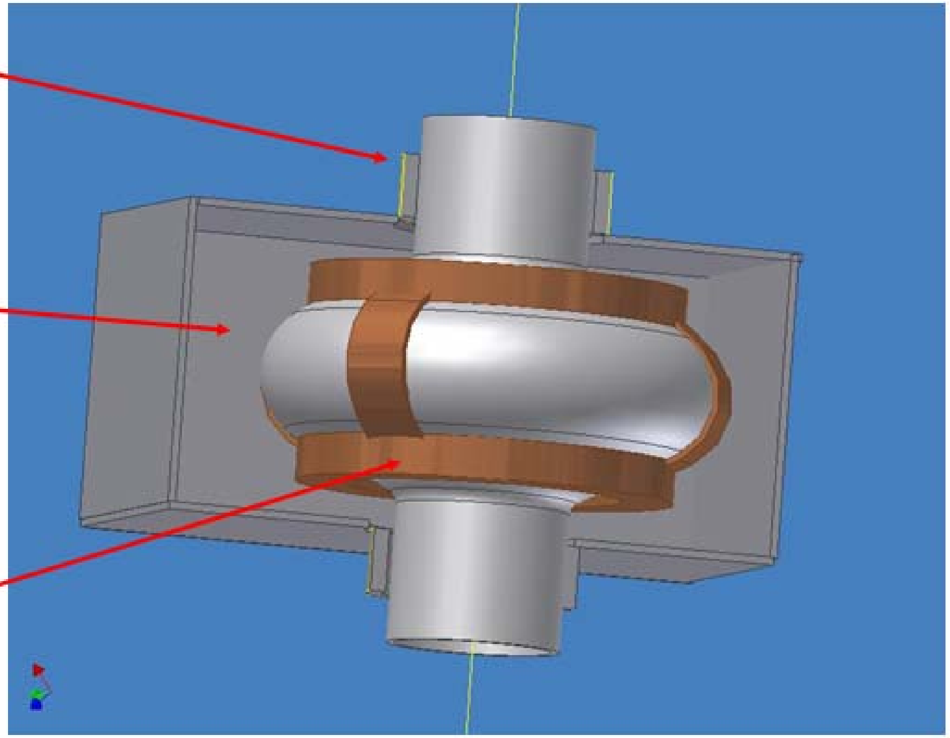
Cornell University
Laboratory for Elementary-Particle Physics

Baking setup

Water cooled copper clamps to keep temperature low on the flanges

Steel box filled with argon under pressure

Two 1.5 kW band heaters



05/23/2007 Grigory Eremeev 9

Correlation between XPS and temperature maps for nearly oxide-free niobium

Cornell University
Laboratory for Elementary-Particle Physics

Conclusion

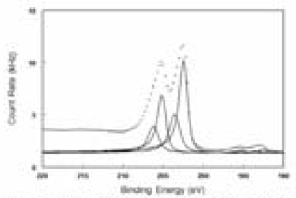
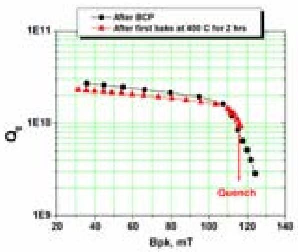
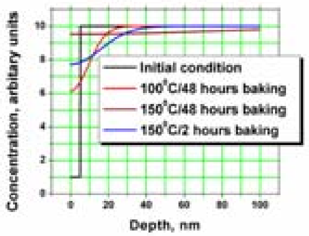


Figure 10. Curve fitted spectrum of sample #2, at 500°C, showing just two components, Nb^0 (metal) and Nb^{1+}.



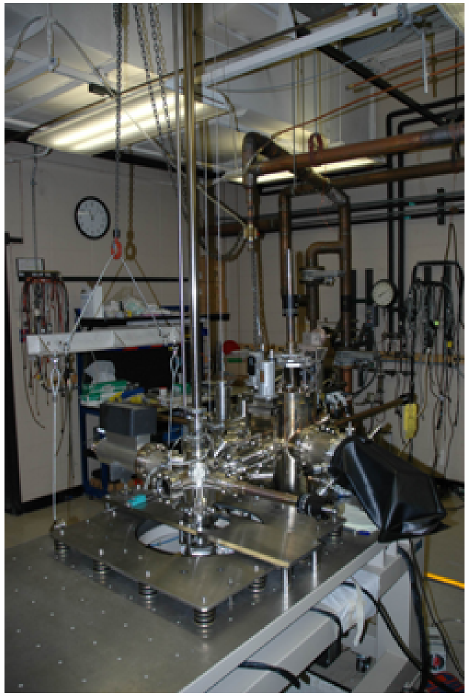


- High field Q-slope does not change after 400^{0}C baking, but calculations show, that the oxygen concentration in penetration depth should be small, so oxygen diffusion explanation for high field Q-slope improvement is contradicted
- Remaining sub-oxides after 400^{0}C baking did not increase the residual resistance(10 nOhm)
  - Other diffusion mechanisms may be involved in the baking effect.

XPS, SIMS measurements are necessary, which will not concentrate only on oxygen

05/23/2007 Grigory Eremeev 20

Low Temperature STM at Argonne



- T=4.2 K, H=7 T
- e-beam for tip preparation
- Ion gun for surface preparation
- Cleaving stage in UHV
- in-situ transfer of sample and tip
- LEED/Auger
- Multi-source e-beam evaporator

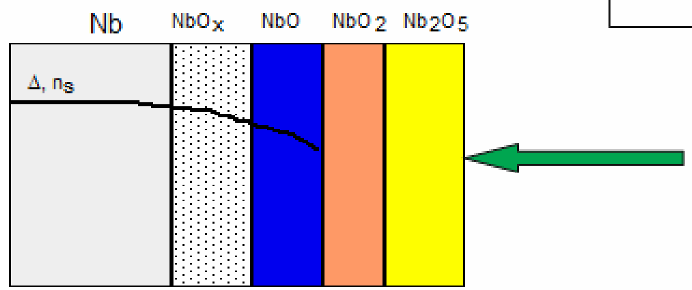
J.F. Zasadzinski, M. Iavarone, T. Proslier, K.E. Gray

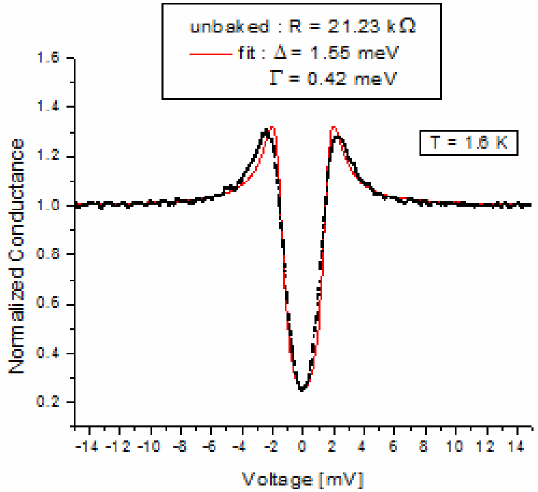
Argonne NATIONAL LABORATORY

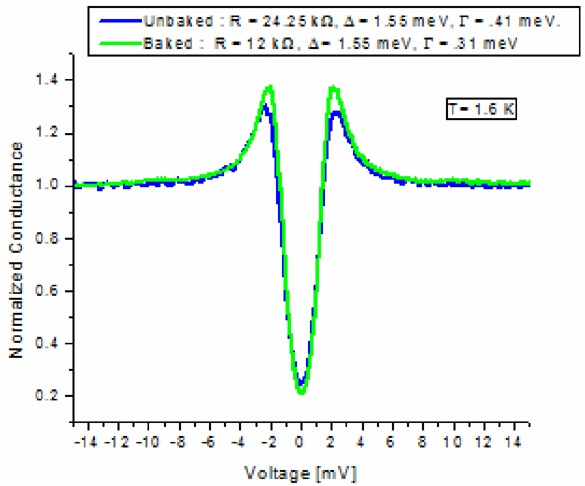
ILLINOIS INSTITUTE OF TECHNOLOGY

Tunneling Spectroscopy and Surface Modification of Nb for SRF Cavity Development

PCT Results







Unbaked Nb single crystal

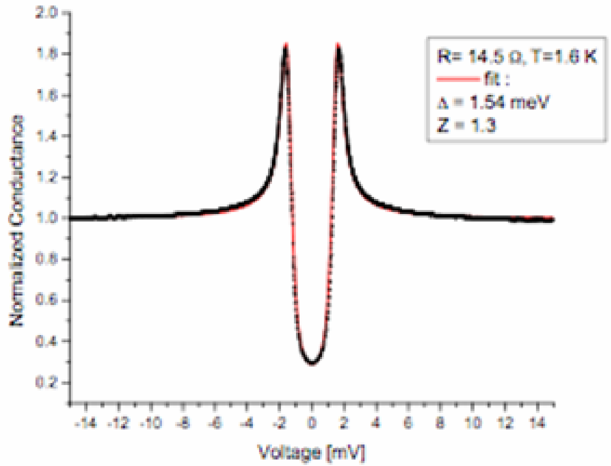
J.F. Zasadzinski, M. Iavarone, T. Proslier, K.E. Gray

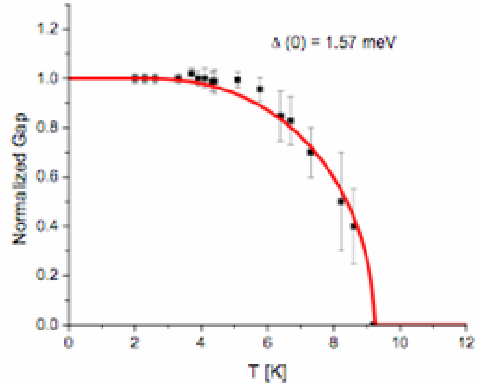
Argonne National Laboratory

Illinois Institute of Technology

Tunneling Spectroscopy and Surface Modification of Nb for SRF Cavity Development

Low Resistance Point Contacts on Nb





Baked Nb single crystal:

124 C during 15 h in air

J.F. Zasadzinski, M. Iavarone, T. Proslier, K.E. Gray

Analytical electron microscopy studies and transport characteristics of large grain niobium for SRF cavity

Key questions

1. Does BCP produce changes to GB structure and composition different than their effect in the bulk?

2. Is there any evidence that GBs, especially those showing preferential flux penetration, possess a lowered superconducting order parameter or penetration field?

Experiment of Zu-Hawn Sung aims to answer this question

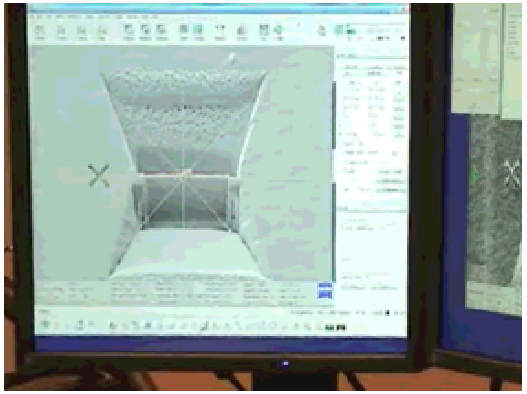
Z. H. Sung

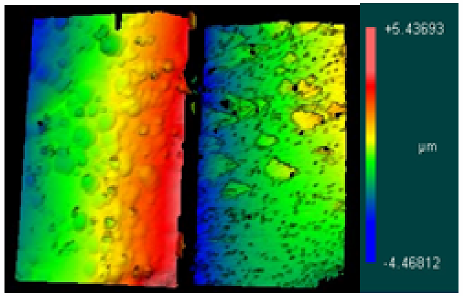
The Applied Superconductivity Center
The National High Magnetic Field Laboratory - FSU

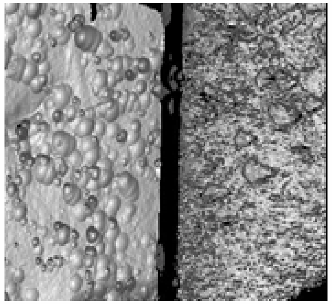
RF superconducting Material workshop at Fermi lab
May. 23 — 24, 2007

FIB'ing of Bi X-tal pure niobium

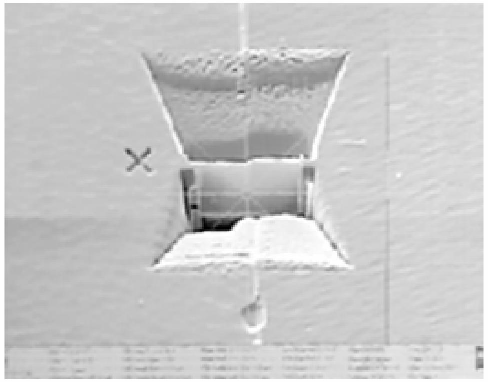
For Micro-structural analysis of I-shape







Movie for TEM prep at G.B



TEM preparation by FIB (Carl Zeiss 1500 EsB) for Bi X-t
at Grain Boundary

Z. H. Sung

Analytical electron microscopy studies and transport characteristics of large grain niobium for SRF cavity

Electron Energy Loss Spectroscopy

Niobium Oxide Phases (NbO, NbO_2, Nb_2O_5)

Successful GB TEM foils allow us to perform μ-chemical comparisons between the GB region and the Grain.

Nb – $M_{2,3}$; white line ~ 362 – 378 eV / Nb – $M_{4,5}$; delayed maxima ~ 204 eV / O – K; ~532 eV

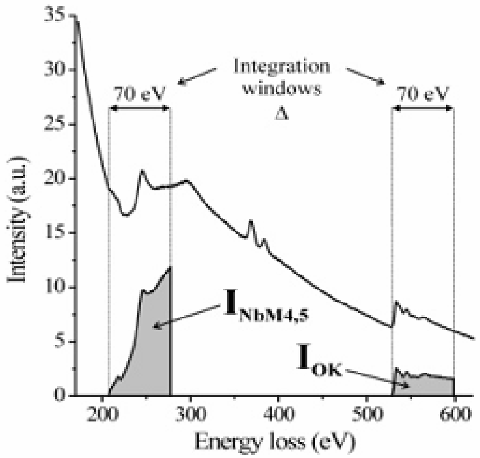


Figure 4. Intensities of the Nb-$M_{4,5}$ ($I_{NbM4,5}$) and the O-K (I_{OK}) edges measured after background subtraction over two 70-eV integration windows (LEO 922 Omega).

$$\frac{N_{Nb}}{N_O} = \frac{I_{Nb}^{M}(\beta\cdot\Delta)}{I_O^{K}(\beta\cdot\Delta)}\cdot\frac{1}{k_{Nb,O}}$$

Integration window	k-factor			
	NbO	NbO_2	Nb_2O_5	Average k-factor
25	0.298 ± 0.017	0.375 ± 0.009	0.329 ± 0.007	0.334 ± 0.075
50	0.107 ± 0.005	0.115 ± 0.002	0.103 ± 0.002	0.108 ± 0.012
75	0.069 ± 0.003	0.073 ± 0.001	0.069 ± 0.001	0.070 ± 0.004
100	0.054 ± 0.002	0.055 ± 0.001	0.053 ± 0.001	0.054 ± 0.002
125	0.047 ± 0.002	0.047 ± 0.001	0.047 ± 0.001	0.047 ± 0.002
150	0.043 ± 0.002	0.041 ± 0.001	0.042 ± 0.001	0.042 ± 0.002

D.Bach. et al., *Microsc. Microanal. 12, 2006* & M.J.Olszta. et al., *J. Micr 2006*

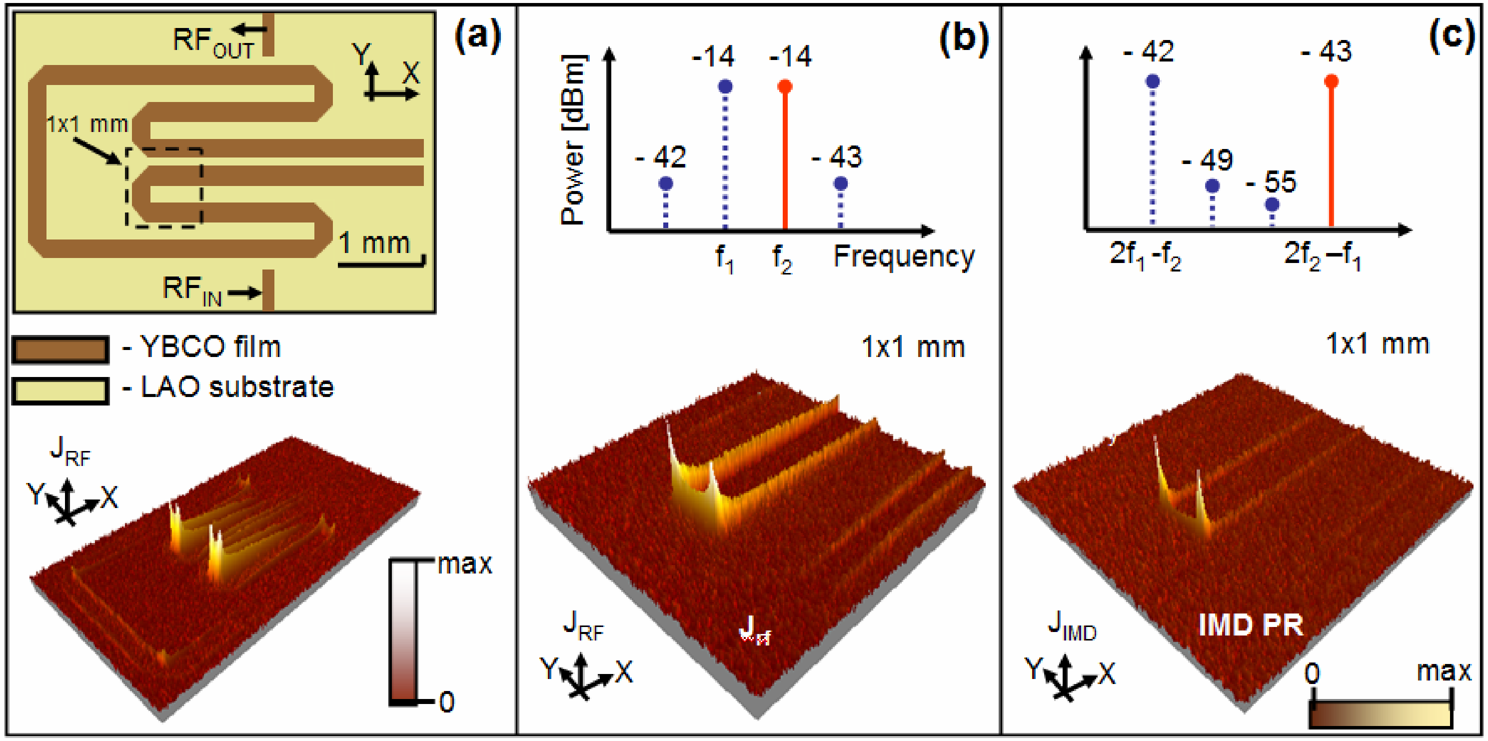
Z. H. Sung

The Applied Superconductivity Center
The National High Magnetic Field Laboratory - FSU

RF superconducting Material workshop at Fermi lab
May 23 — 24, 2007

Imaging of Microwave Currents and Microscopic Sources of Nonlinearities in Superconducting Resonators

Spatially-resolved microwave photo-response



(a) Top view of the resonator topology along with overall and (b, c) detailed 1x1 mm 3-d LTLSM plots (bottom images) showing (b) $J_{RF}(x,y)$ and (c) IMD PR distribution. The upper part of (b) shows the two input tones at –14 dBm as well as the output tones. The upper part of (c) shows the signals entering the spectrum analyzer after the primary tones have suffered partial cancellation.

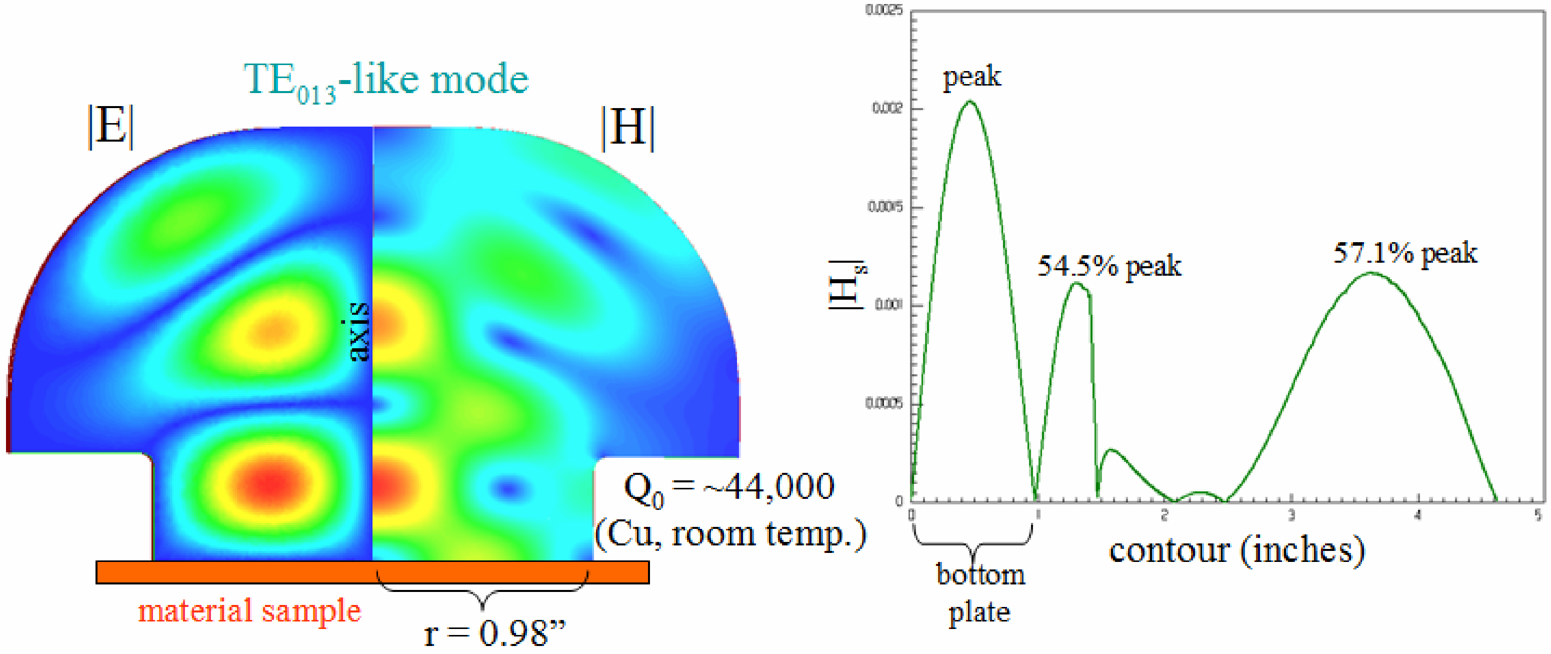
A.P. Zhuravel, S. M. Anlage and A.V. Ustinov

Imaging of Microwave Currents and Microscopic Sources of Nonlinearities in Superconducting Resonators

20

Critical magnetic field measurement of MgB2

The Mushroom Cavity



Why X-band (~11.424 GHz)?:

- •high power & rf components available
- •fits in cryogenic dewar
- •small (3”) samples required

Features:

- •No surface electric fields (no multipactor)
- •Magnetic field concentrated on bottom (sample) face (75% higher than anywhere else)
- •Purely azimuthal currents allow demountable bottom face (gap).

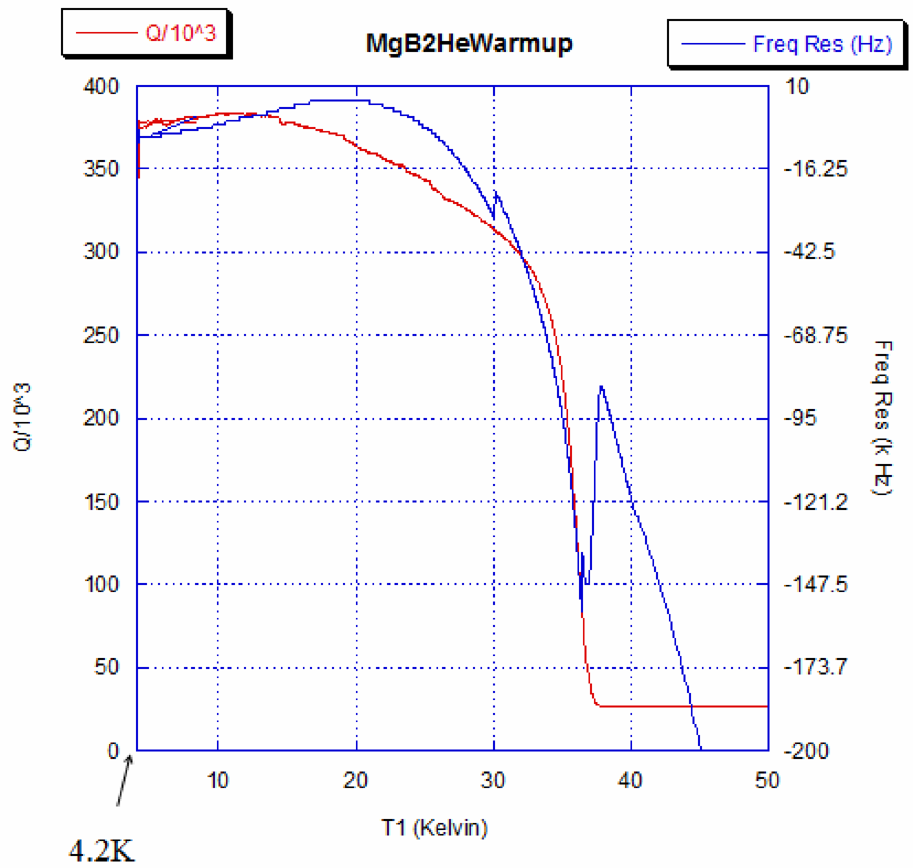
4

SRF Materials Workshop, FNAL, 23-24 May 2007

Tsuyoshi Tajima

Critical magnetic field measurement of MgB2

MgB_2 film (~500 nm) by STI



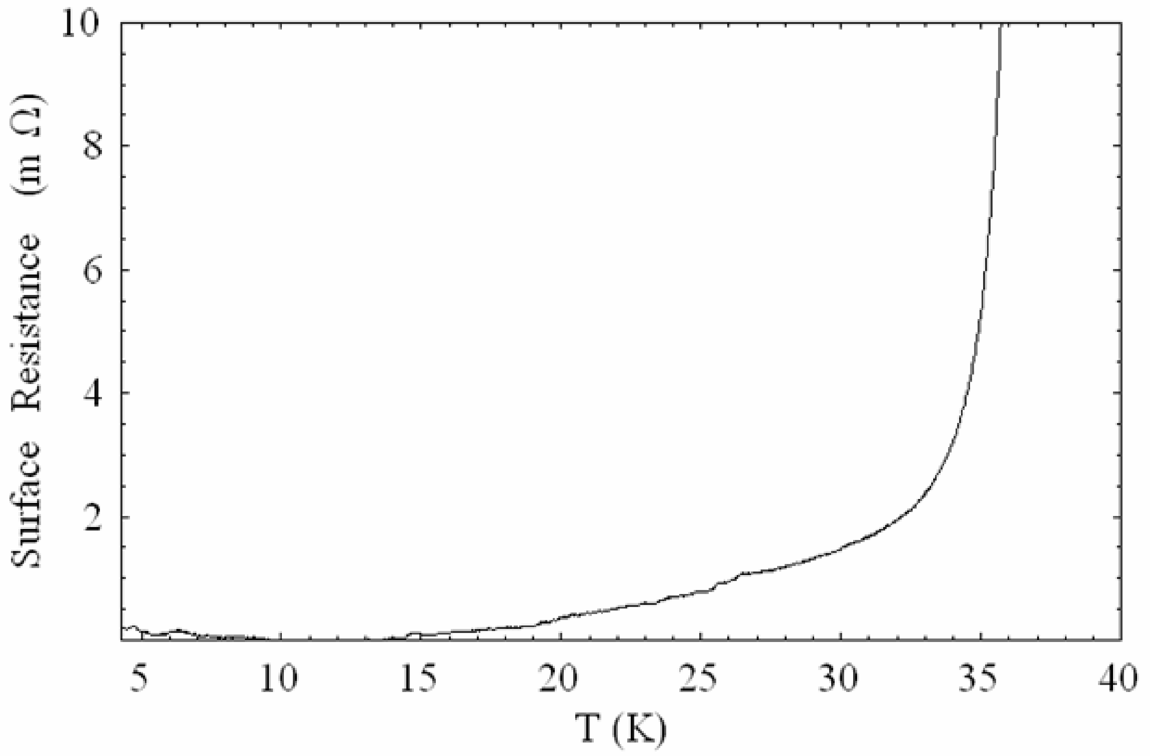
SRF Materials Workshop, FNAL, 23-24 May 2007

13

Tsuyoshi Tajima

Critical magnetic field measurement of MgB2

MgB_2 surface resistance calculated from the difference betw. Cu and MgB_2 tests (preliminary)

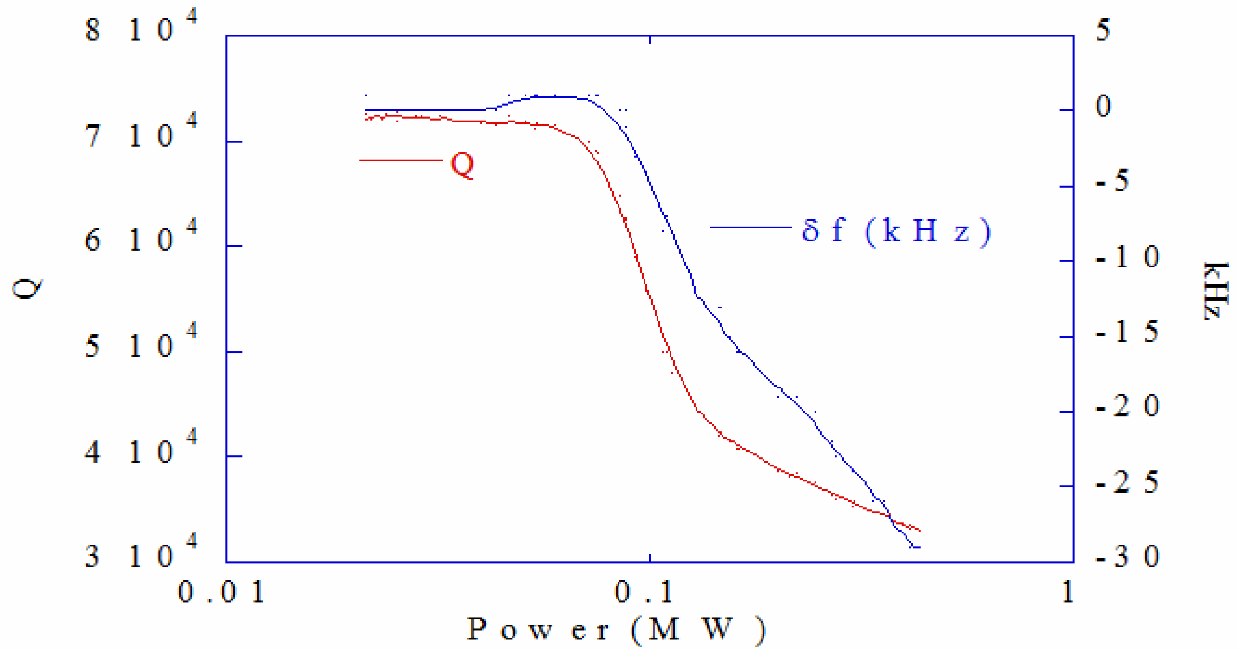


SRF Materials Workshop, FNAL, 23-24 May 2007

Tsuyoshi Tajima

Critical magnetic field measurement of MgB2

High Power Test of Niobium at 4.2K (reactor grade Nb)



New test setup for inexpensive accurate characterization of high-field RF properties of materials and processing techniques

SRF Materials Workshop, FNAL, 23-24 May 2

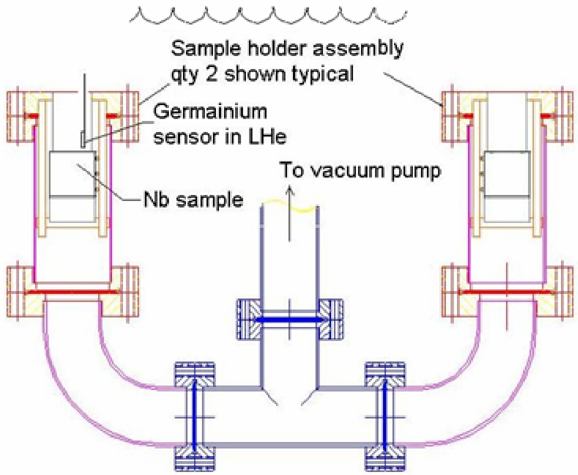
Tsuyoshi Tajima

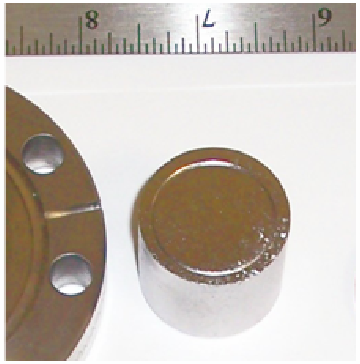
Heat transfer measurements of niobium for SRF cavities

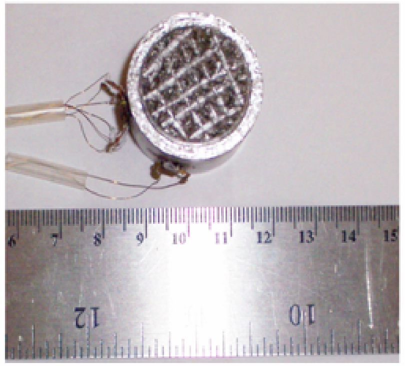
NSCL

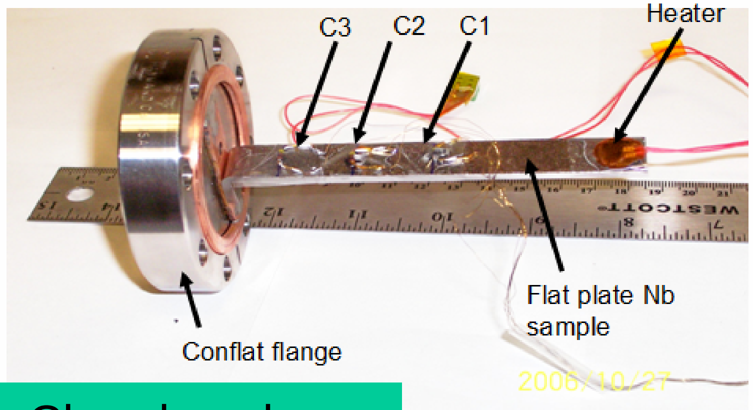
Experimental Apparatus

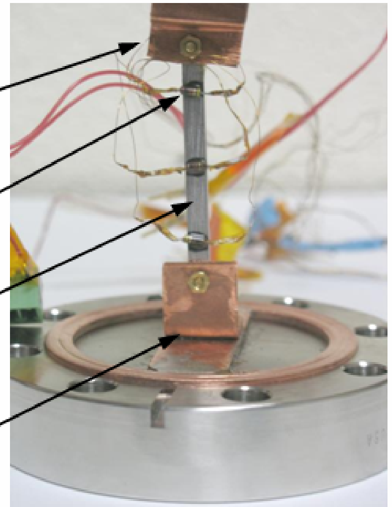
NATIONAL SUPERCONDUCTING CYCLOTRON LABORATORY AT MICHIGAN STATE UNIVERSITY











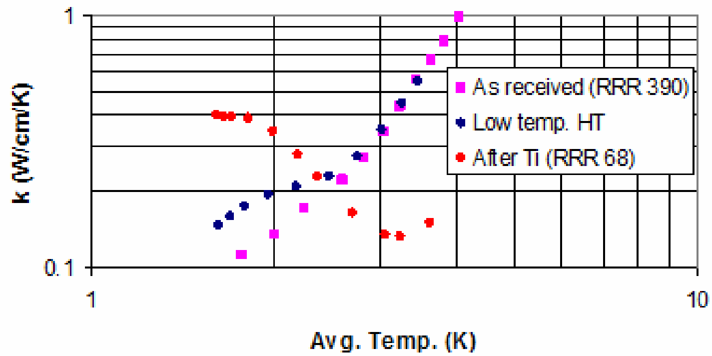
S.K. Chandrasekaran

Adapted from Aizaz, 2006

NSCL
NATIONAL SUPERCONDUCTING CYCLOTRON LABORATORY AT MICHIGAN STATE UNIVERSITY

Results (cont.)

Flat plate samples



Adapted from Aizaz, 2006

	As received	Post-heat treatment 750 °C (2 hrs)	Post-titanification 1300 °C (2 hrs), 1200 °C (4 hrs)
k	No phonon peak	Slight increase <2.5 K	Phonon peak recovered; significant decrease >2.5 K

- Post-titanification thermal conductivity measurements suggest RRR ~ 80
- Post-titanification expectation of RRR ~ 600
- Measured RRR in samples cut from same plate indicate RRR 67 (+/- 20%) ...ts by Fermilab)

S.K. Chandrasekaran

- **Highlights of new things**

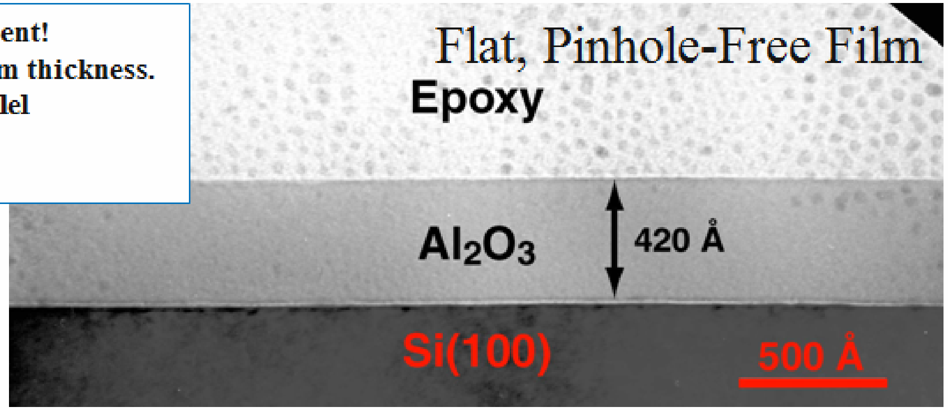
Session 3: New Materials for the Future

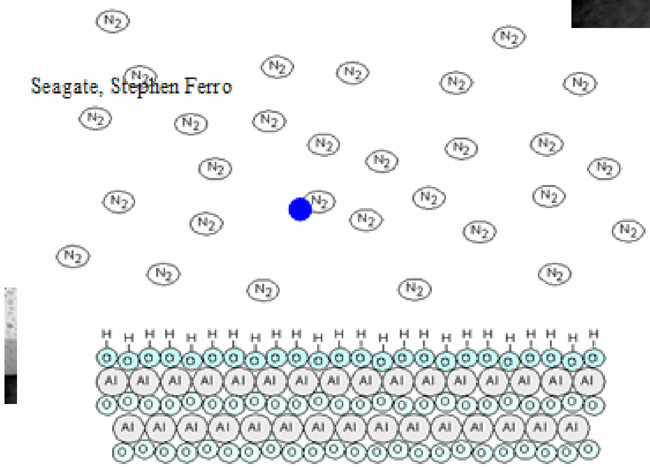
- Atomic layer deposition: an attractive method for surface engineering (Pellin)
- Highest quality MgB_2 cavity coating is feasible (Xi)

Atomic layer deposition

ALD Reaction Scheme

- No uniform line of sight requirement!
- Errors do not accumulate with film thickness.
- Fast! (µm's in 1-3 hrs) and parallel
- Pinholes seem to be removed.
- Bulk





- ALD involves the use of a pair of reagents.
  - each reacts with the surface completely
  - each will not react with itself
- This setup eliminates line of site requirements
- Application of this AB Scheme
  - Reforms the surface
  - Adds precisely 1 monolayer
- Pulsed Valves allow atomic layer precision in growth
- Viscous flow (~1 torr) allows rapid growth
  - ~1 µm / 1-4 hours

Argonne NATIONAL LABORATORY

SRF Materials Workshop
FNAL, May 23,24,2007

M. Pellin, J. Moore

Atomic layer deposition

Proposal for bringing ALD to SCRF fabrication

- Investigate the properties of Al_2O_3 capping layers for Nb surfaces
  - Al_2O_3 is the ALD "poster child"
  - Stabilize Nb surface oxides
  - Anneal to reduce oxide thickness
  - We are already using it to cap FePt particles (resist oxidation to 1000 C @ 10 nm thick film)
- Synthesize Nb "bulk" layer on Al or Cu cavities
  - Develop the chemistries for "clean" deposition and crystallite control
  - In situ capping
- Grow NbN, Nb_3Sn, MgB_2 layers on the Al_2O_3 capped Nb
  - Develop the chemistries for "clean" deposition and crystallite control
  - Test for enhanced field behavior
- Scale-up
  - ALD was made for growing films on the inside of tubes – precision layer by layer growth at aspect ratios exceeding 10000
  - Coating is of all surfaces, no field penetration at the corners or edges

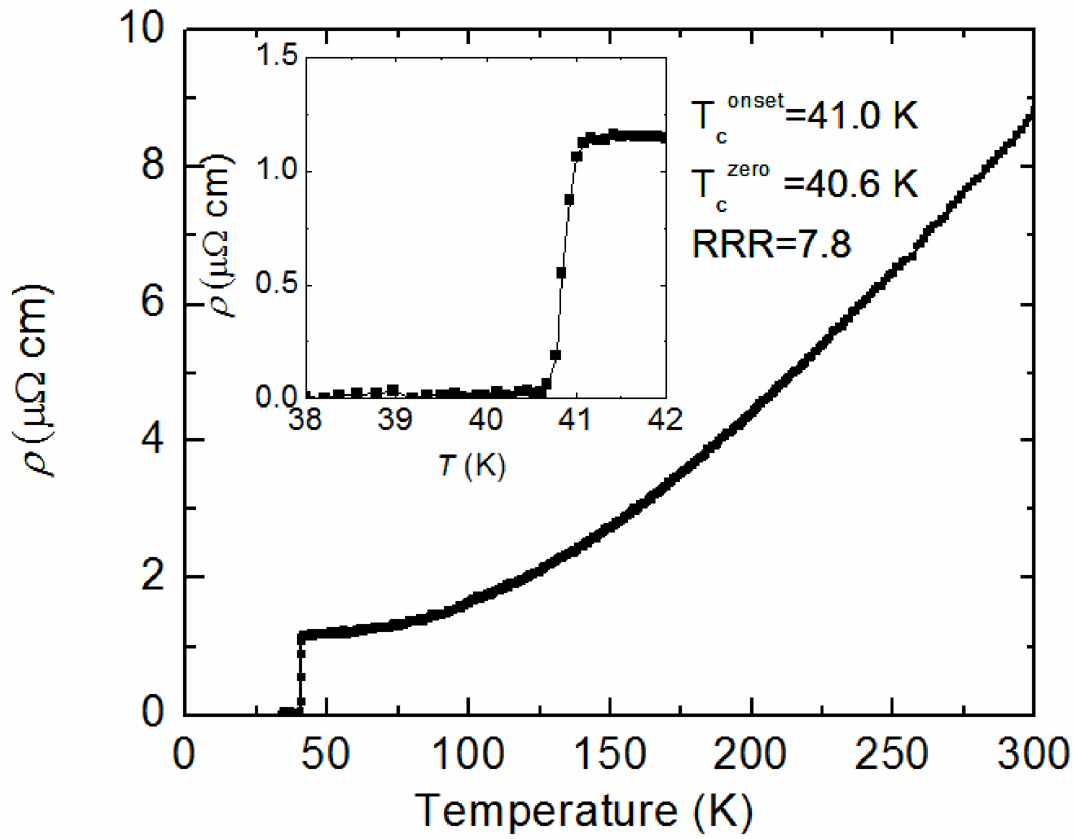
Argonne NATIONAL LABORATORY

SRF Materials Workshop
FNAL, May 23,24,2007

M. Pellin, J. Moore

MgB2 thin film and its application to RF cavities

MgB_2 Film by Reaction of CVD B Film

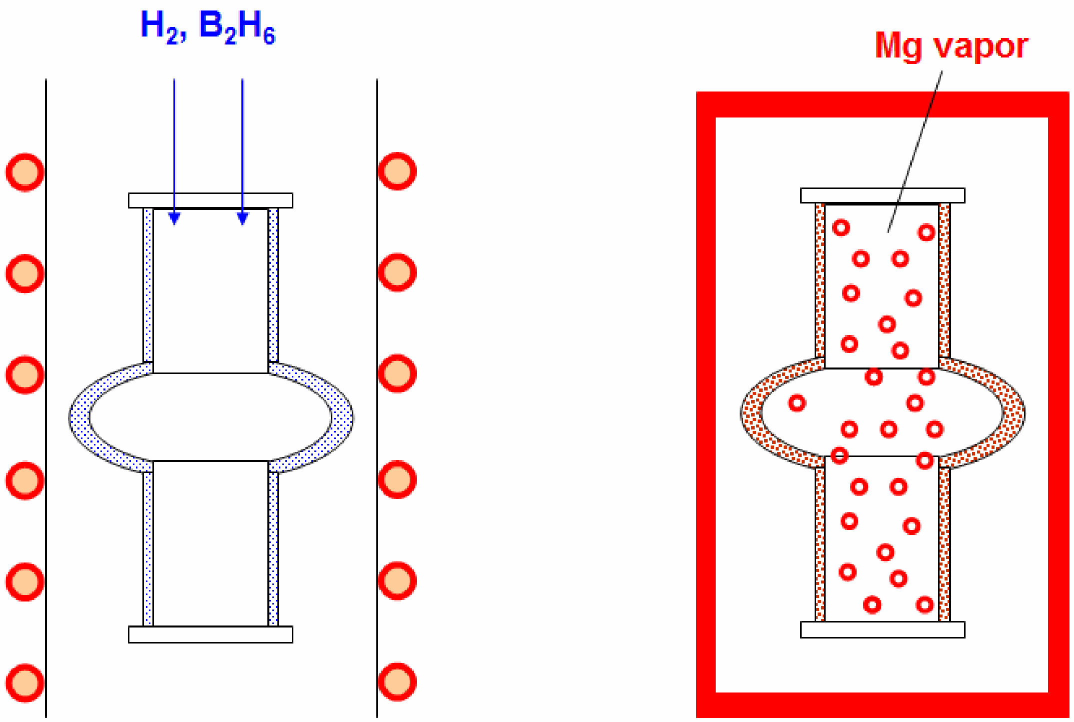


Clean B precursor layer leads to clean MgB_2 film.

Xiaoxing Xi

MgB2 thin film and its application to RF cavities

Coating SRF Cavity with a Two-Step Process



Coating cavity with B layer at ~400-500° C using CVD

Reacting with Mg to form MgB_2 at ~ 850-900 ° C in Mg vapor

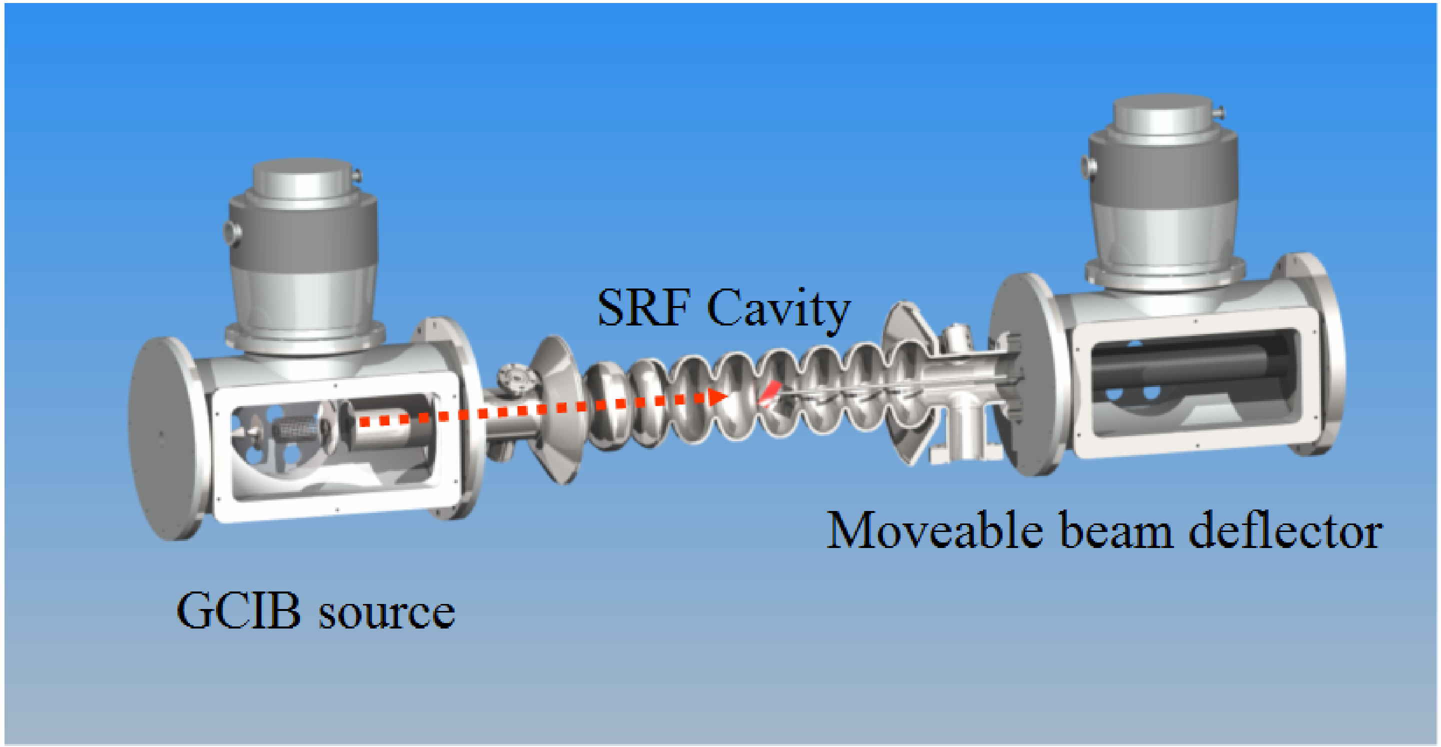
Xiaoxing Xi

- **Highlights of new things**

Session 4: (innovative) Processing of materials

- GCIB process was successful on other applications, can it be successful to SRF? (Swenson)
- Plasma etching, an alternative for surface etching (Raskovic, G.Wu)
- Alternative EP processes possible (Crooks)
- Chemical mechanical polishing as a pre-etching polishing (Muftu)

GCIB treatment of SRF cavity



In-situ high vacuum surface treatment

D.R. Swenson, et. al, SRF Materials Workshop, Fermilab, 23 May 2007

TEL epion

Why Oxygen treatment?

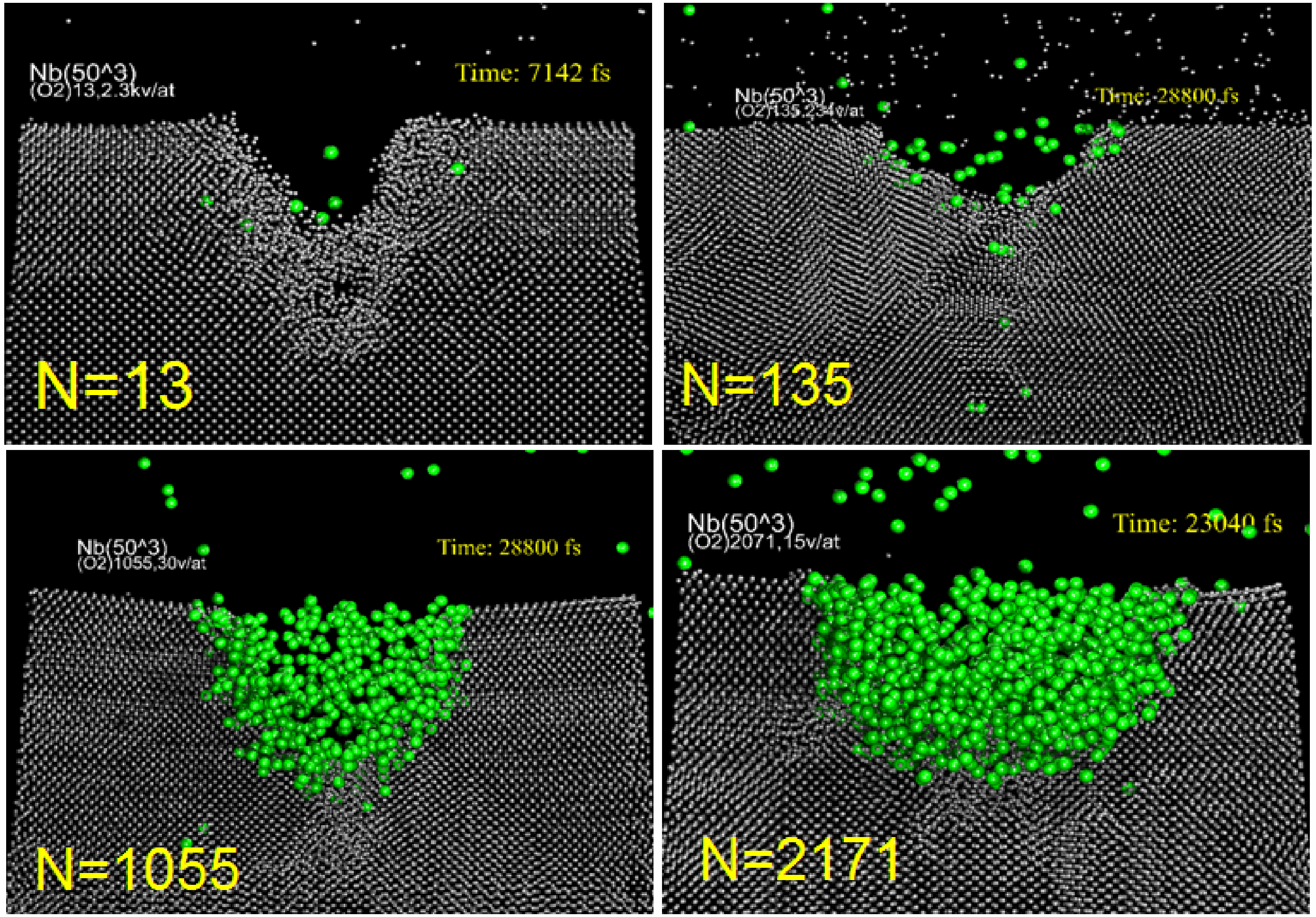
- Smoothing
  - Reduce field emission
- Etching
  - Should remove 30 nm
- Cleaning
  - Disrupt particles and contamination on the surface
- Hardening
  - High pressure impacts will increase density and hardness of surface layer
- Oxygen Infusion
  - Oxidize sub-oxides to pent-oxide
  - Sharp interface between pent-oxide and Nb

D.R. Swenson, et. al, SRF Materials Workshop, Fermilab, 23 May 2007

TEL epion

surface treatment of SRF cavities with Gas Cluster Ion Beams

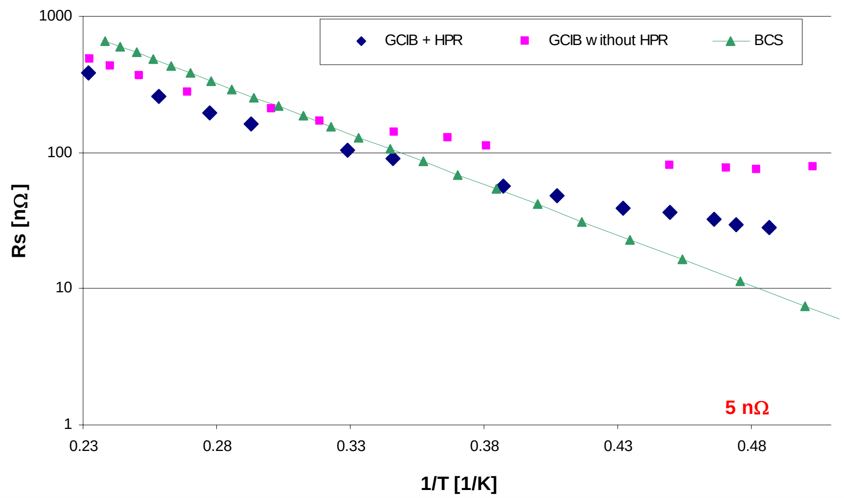
30kV (O2)n (n=13-3000) cluster Infusion in Nb (100)



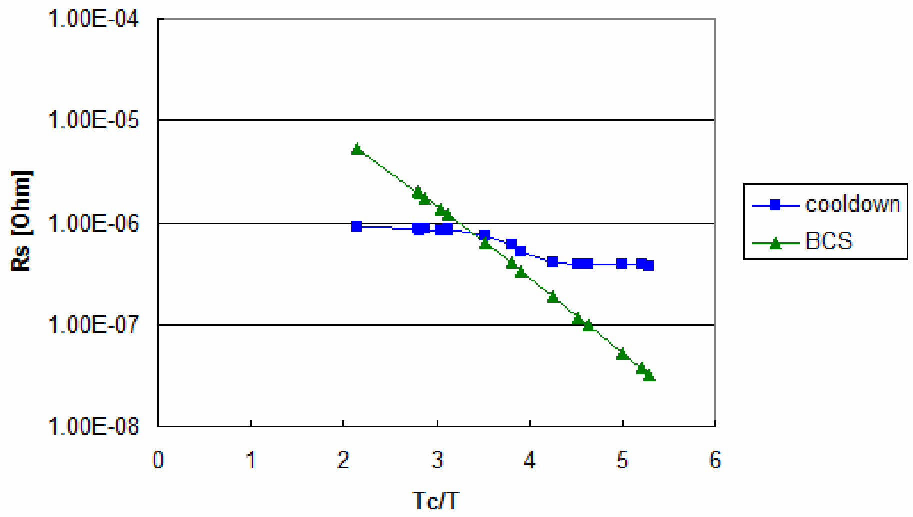
Argonne NATIONAL LABORATORY

Zeke Insepov 17

surface treatment of SRF cavities with Gas Cluster Ion Beams



1.3GHz



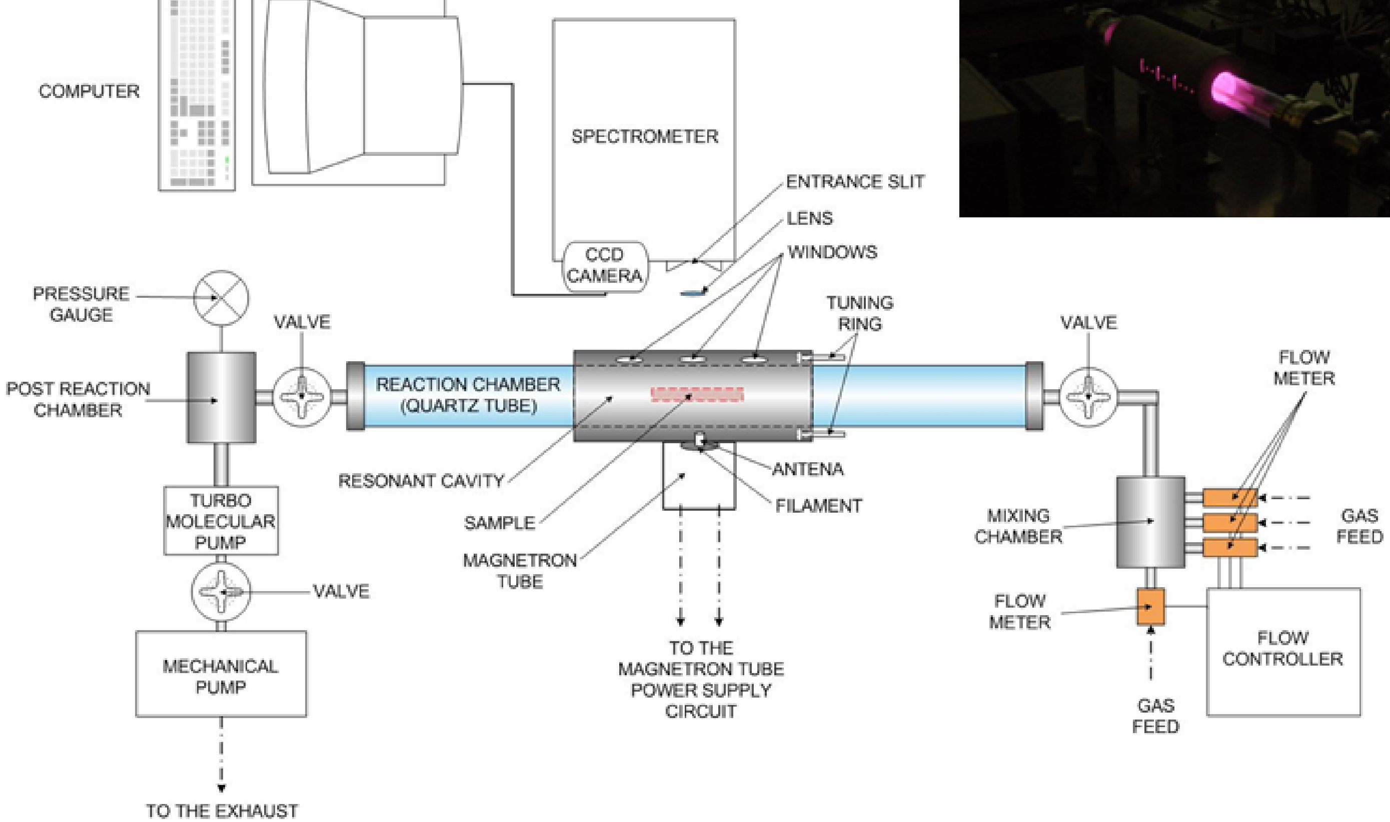
3.9GHz

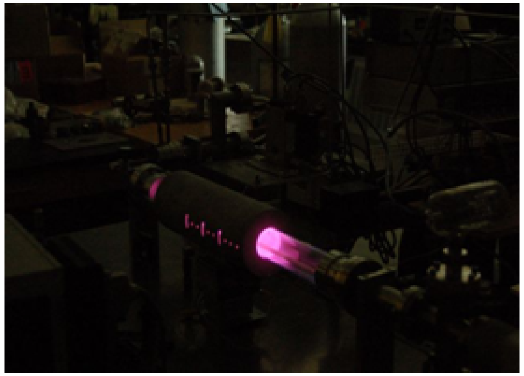
D.R. Swenson, et. al, SRF Materials Workshop, Fermilab, 23 May 2007

Plasma etching of Niobium surface

Microwave (MW) glow discharge system

Reactive gas: Cl_2 in Ar
Reactor: barrel, MW radiation at 2.45 GHz



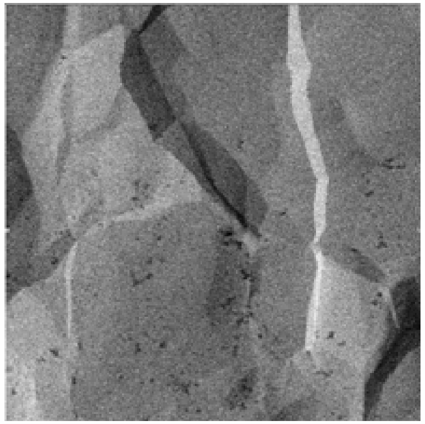


M. Rašković, L. Vušković, S. Popović, L. Phillips, A.-M. Valente-Feliciano, S. B. Radovanov, and L. Godet, Proceedings of EPAC 2006, Edinburgh, Scotland, MOPCH184.

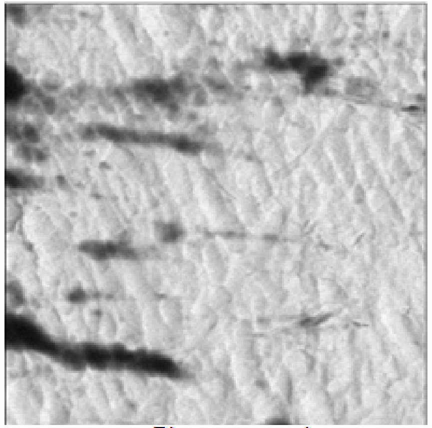
Plasma etching of Niobium surface

3. Scanning electron microscope (JSM-6060LV)

a) Morphology



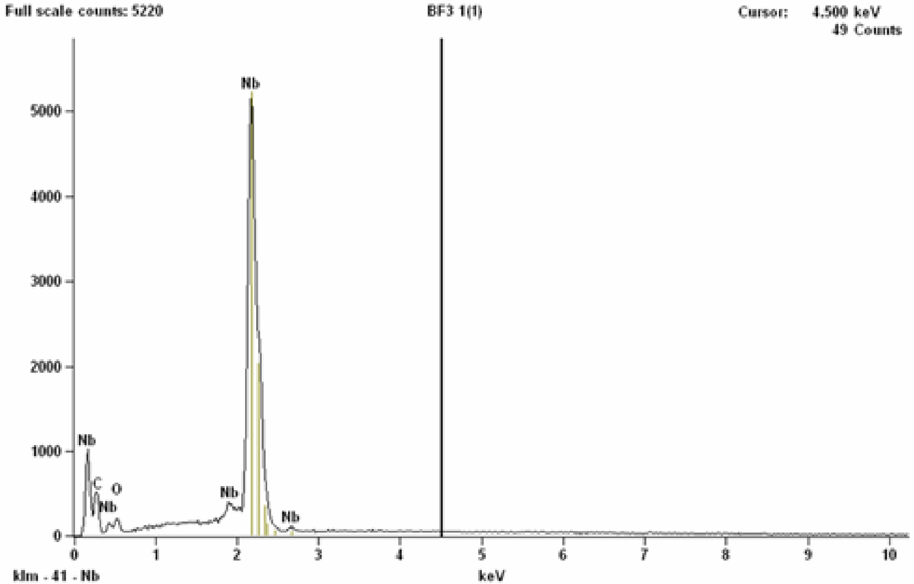
BCP-treated



Plasma-treated
(Sample No.1)

b) Composition analysis

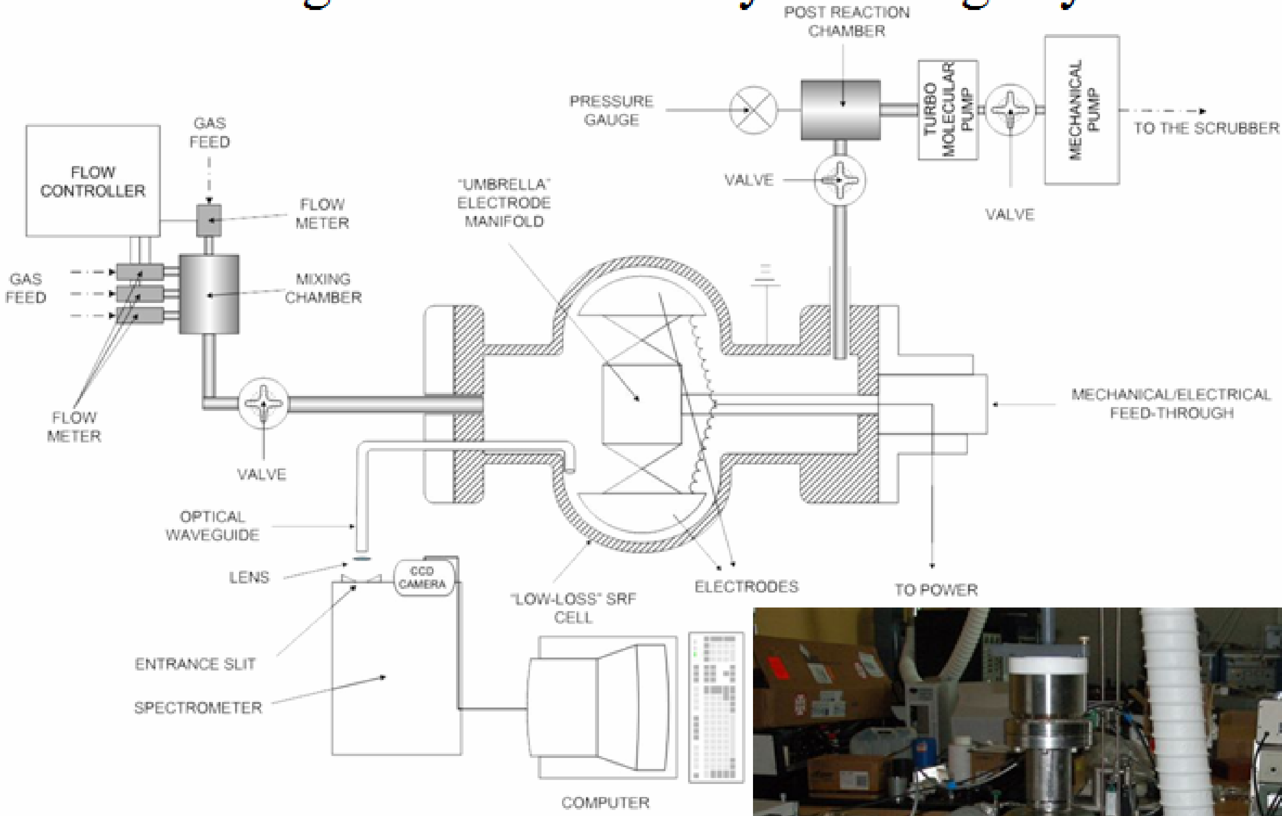
Average area EDX spectra of the plasma-treated sample

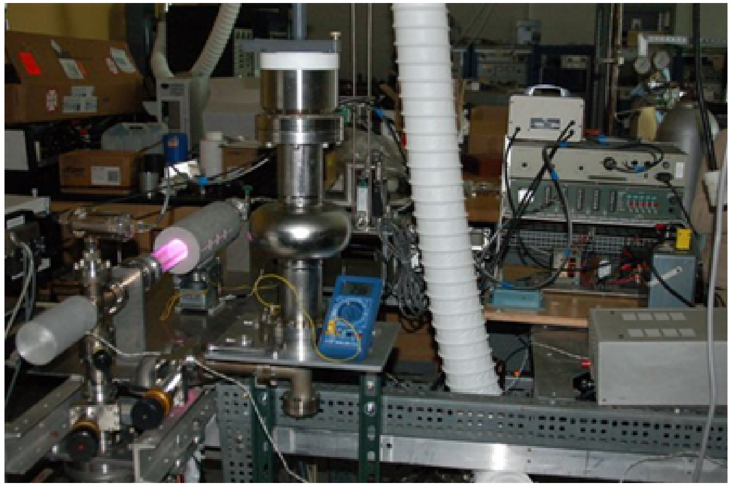


Sample	W_a (Å)	W_{RMS} (Å)	R_a (μm)	R_{RMS} (μm)
untreated	235	285	0.930	1.125
BCP	141	169	0.323	0.476
1 - plasma	20	26	0.246	0.309
2 - plasma	23	27	0.195	0.240
3 - plasma	11	16	0.295	0.353

Plasma etching of Niobium surface

Single cell SRF cavity discharge system



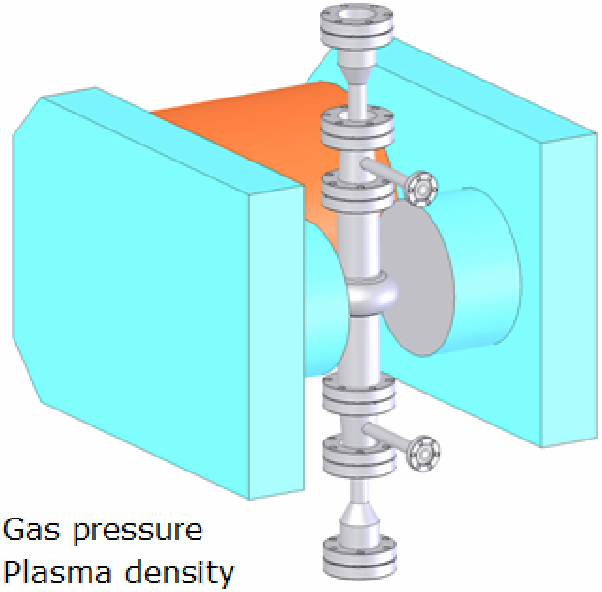


ECR plasma: a possible in-situ cavity processing technique

ECR plasma: a possible in-situ cavity processing technique

Experimental plan and the issues to be addressed

- Usual cleaning
- Cold RF test to find FE limit
- Plasma processing at room temperature
- Cold RF test to verify improvement
- Gas mixtures: Ar, H_2, O_2, He, Kr.



- Surface contamination removal?
- Ar implantation? Ar ion creates surface defects?
- Dry-oxidation afterwards?
- If Hydrogen, how about Q-disease?
- If Oxygen, oxidation compound?
- He also effective?

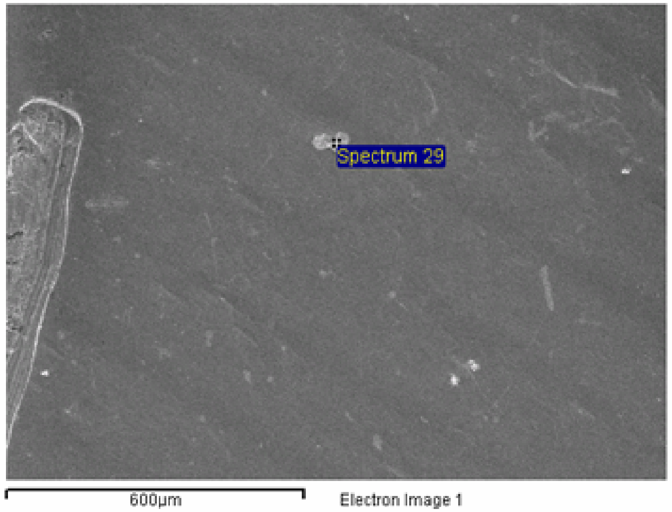
- Gas pressure
- Plasma density
- Temperature distribution
- Ion energy distribution
- Ion flux rate
- Chemical reaction

2007/05/24 G. Wu, SRF materials workshop 11

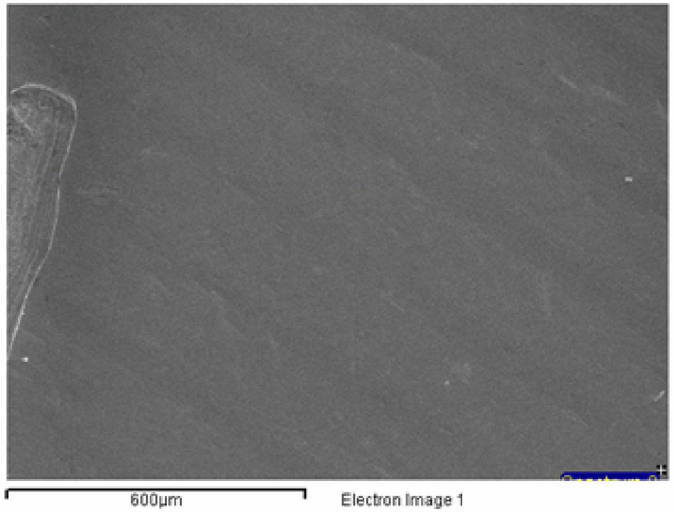
ECR plasma: a possible in-situ cavity processing technique

ECR plasma: First result

ECR plasma results



Before plasma treating



After plasma treating

Ar plasma 20 minutes, Air plasma 30 minutes, highest temperature 48°C

Experimental Conditions for Electropolishing

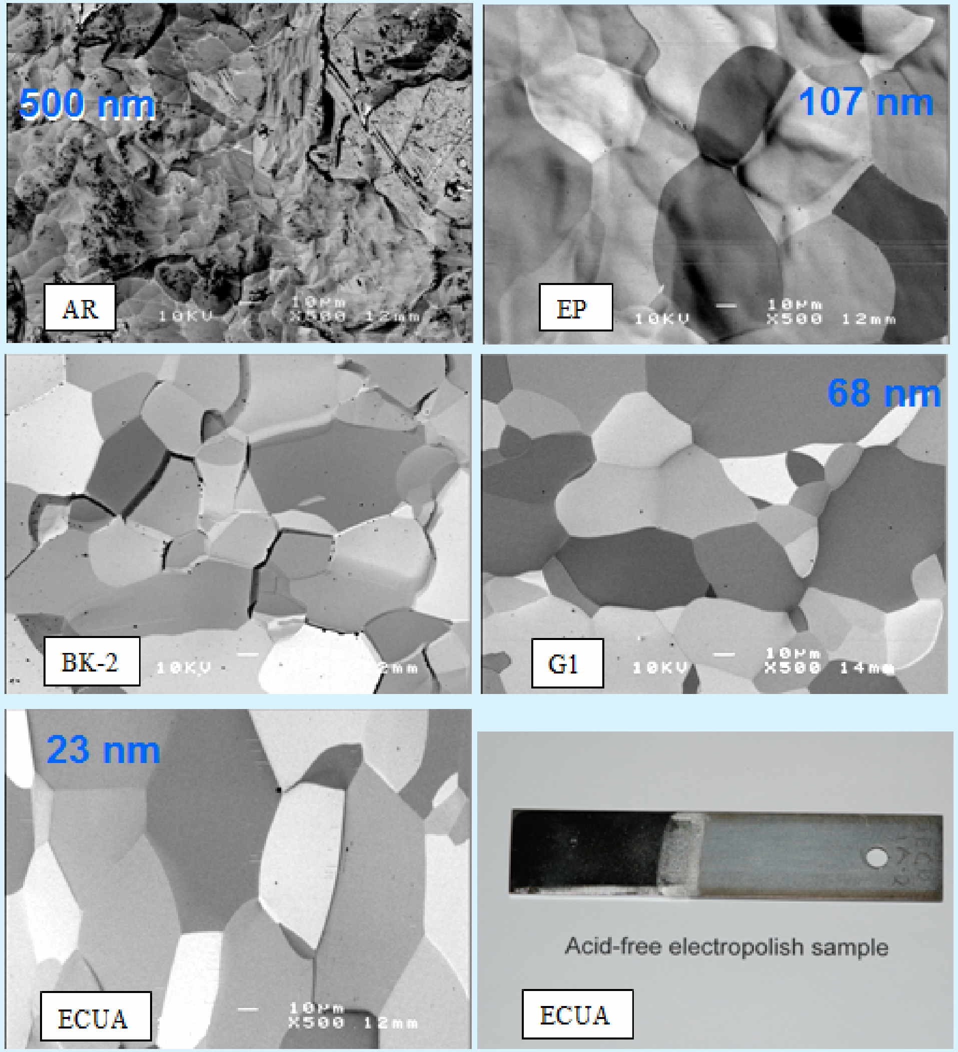
Sample	Chemistry	T, ºC	V	mA/cm2
BK-2	5.3g LiCl, 11g $Mg(ClO_4)_2$,500ml CH3OH, 250ml b.c.*	-78	60	50
G1	11g $Mg(ClO_4)_2$,500ml CH_3OH	-78	30	40
EP	$HF:H_2SO_4$; 1:9 (by volume) [Compton & Saxton (MSU), Fermilab, 2005]	40	10	50
ECUA	Choline Chloride, Urea, NH_4F + lower viscosity organic solvent	70	12	50

Electropolishing was performed under current control, with simple two-electrode System with a stainless steel cathode. Degreased in soapy water. Temperature control (dry ice/methanol bath) was used for first two.

Current density of 50 mA/cm^2 corresponded to a removal rate of ~ 1 μm/min.

Roy Crooks

Channeling Contrast (backscattered) Electron Images and Ra values



Rolling Plane (plan view)

1 hour Electropolish AR RRR Nb

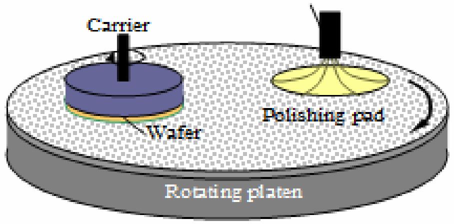
Several cm^2 electropolished

Roy Crooks

CMP Process

Process description:

- A *wafer* is pushed against a polymeric *polishing pad*
- (P_a = 1-10 psi)
- Pad and wafer rotate independently (~60 rpm).
- *Slurry*, containing *oxidizing chemicals* and *abrasive particles* is supplied into the interface.
- **Material removal** occurs due to ***particle abrasion*** of the ***chemically passivated*** *wafer surface.*



Schematic of CMP operation

Slurry

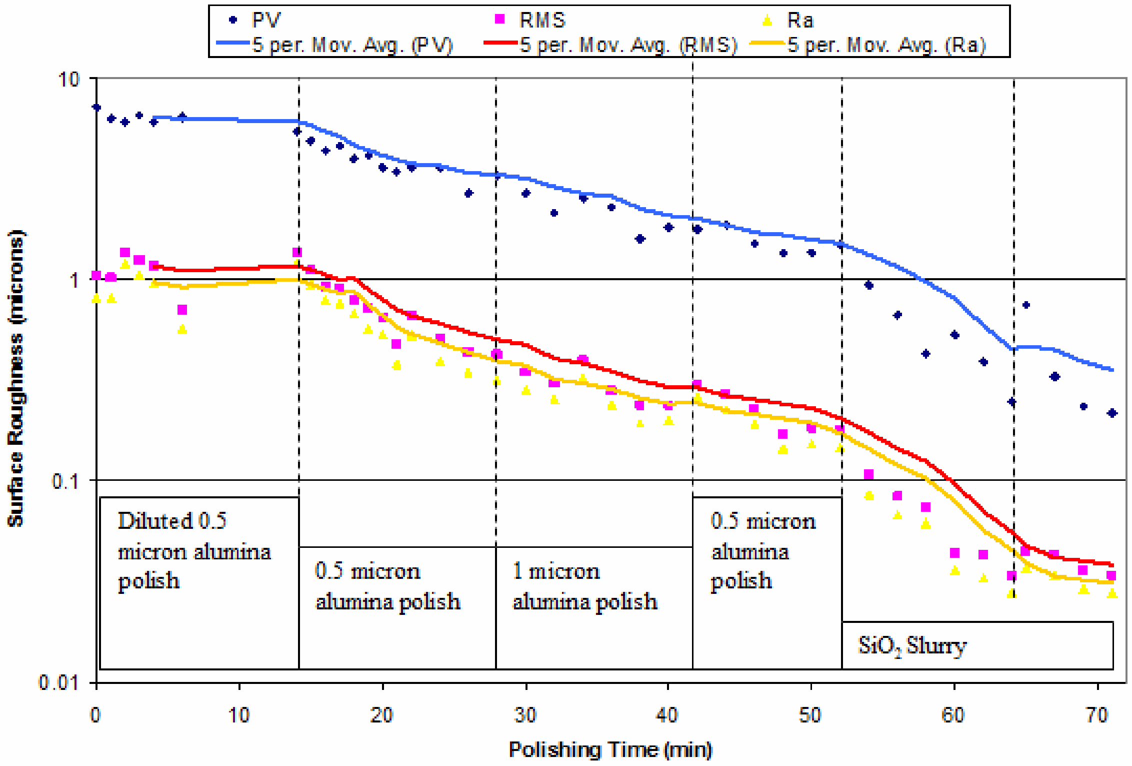
Designed to create (low-hardness) oxides.

Chemicals:
- Oxidizers
- Buffers
- Surfactants

Particles:
- Material:
  Silica (SiO_2), alumina (Al_2O_3, Ceria (CeO_2)
- Size: 50-150 nm
- Shape: Spherical

Chemical Mechanical Polishing for Obtaining Very Smooth Surfaces

Two step process



Sinan Muftu

...ering and Chemical Engineering

Northeastern University

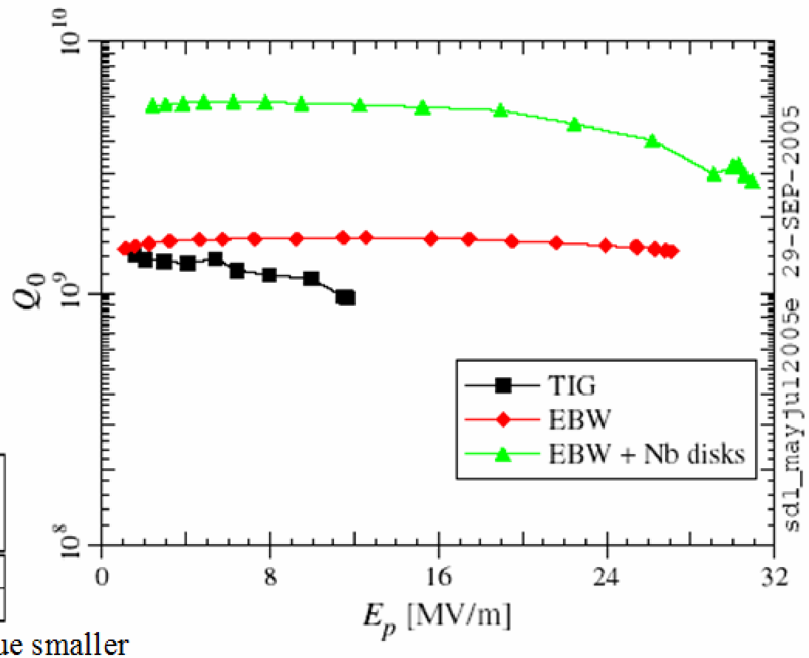
Highlights of new things

Session 5: Niobium production

Test Results of first TIG cavity

Cavity	Estimated R_{BCS} at 4.2 K (nΩ)	Inferred surface RRR
TIG	1600 to 1970	$\lesssim 25$
EBW	2300 to 2370	~ 100

Generally infer a surface RRR value smaller than bulk RRR specification



Tests results at 2 K

Advancing Knowledge. Transforming Lives.

Chris Compton

Highlights of new things

Session 5: Niobium production

Schedule

- Recheck RRR measurements
- Chamber modifications (80 ppb best to date)
  - Main seal (leak)
  - Torch purge gas
  - Add Titanium getter
- Weld improved niobium samples for RRR measurements
- Fabricate single cell cavity using TIG Chamber
  - 1.3 GHz, Beta = 0.81, Proton Driver Prototype
    - Dies and infrastructure complete
    - Niobium from Fermi
- Cold performance testing at MSU

NSF NSCL MICHIGAN STATE UNIVERSITY Advancing Knowledge. Transforming Lives.

Chris Compton