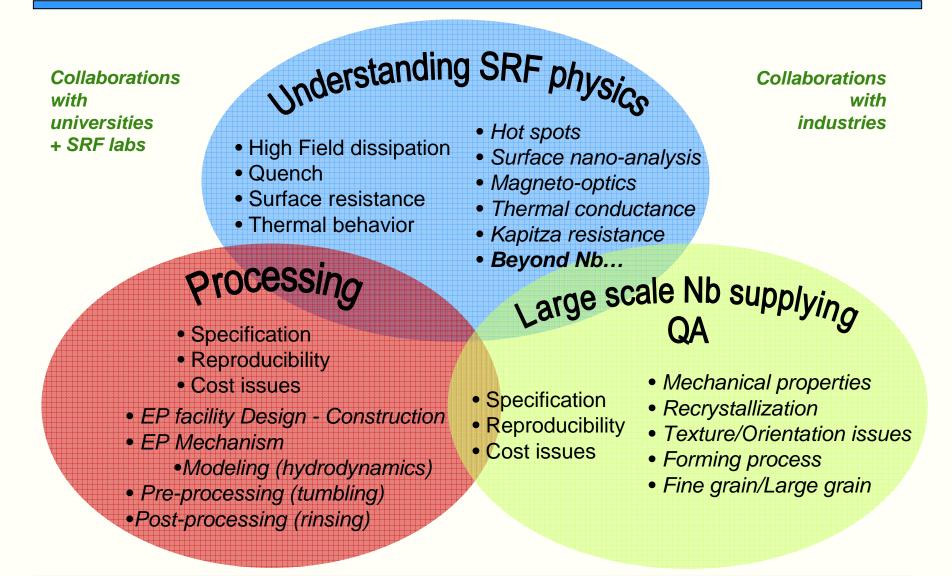
# **SRF Material R&D**

<u>Claire Antoine</u> Cristian Boffo Genfa Wu

# **SRF Materials Issues**





Feb 13-14, 2007

# **SRF Materials group**



•

	(Dressessing DOD University Colleb)				
C. Antoine	(Processing R&D, University Collab.)				
Engineer-Physicists:					
D. Hicks*	(Cavity, Nb processing)				
G. Wu	(Materials R&D, Processing R&D)				
Engineers:					
C. Boffo	(Processing R&D and Facilities, Materials R&D)				
C. Cooper*	(Processing Lab Safety, Processing R&D)				
N. Dhanaraj	(1 Cell Program)				
G. Galasso	(Processing R&D)				
Designers:					
K. Ewald	(Processing R&D)		FTE:		
F. McConologue*	(FTE Processing R&D)		1 SC		
Designer 1 cell*	( 1 Cell Program)		130		
Tech:			5 ENG		
D. Bice	(Processing R&D @ J-Lab)		3.5 TEC		
D. Burke*	(Processing Lab support)				
O. Frianeza	(Processing R&D, SRF Materials Lab)		2 DES		
R. Schuessler	(SRF Materials Lab)				

### Improvement of QC/QA and Support to projects



- <u>Ongoing</u>
  - Eddy Current Scanner, microscopy, mechanical measurements (collab. MSU)...
  - Cutting study
  - RRR measurement
- Short term activities
  - Cold tensile test (implementation of the Instron Machine)
  - Surface routine analysis\*
  - Thermal conductivity measurement
- <u>Mid-long term activities</u>
  - Squid Eddy Current scanning (sheets, cavities) ? \*\*.
  - Field emission scanner ? \*\*

\*Investment needed

\*\* developed else where

# **Process R&D Activities**



- <u>Ongoing</u>
  - Pre-processing (Tumbling) / Post-processing (dry/plasma cleaning)
  - Samples R&D (bath aging, Fluorine monitoring with ISE, process understanding,...)
  - 3.9 GHz 1-cell EP set-up
  - EP modeling (needs to be reinforced), BCP (thermal modeling)
  - EP/BCP facility @ ANL & FNAL
  - Assembly
- Short term activities
  - Upgrading the 3.9 GHz EP set-up to 1.3 GHz ?
  - Development of nine-cells EP set-up
  - Development of single/nine-cells RF test stand with diagnostic (*T-mapping, replicas...*)
- <u>Mid term activities</u>. They need to be first demonstrated on 1-cell before being applied to 9-cell. It supposes the 1-cell RF test stand to be running.
  - Reproducibility of the complete process: EP + HPR + Baking + RF test (1-then 9cells)
  - Alternative rinsing (ethanol, degreasing)
  - Baking study
  - Feasibility of online F monitoring on the EP set-up.

# **Materials R&D Activities**

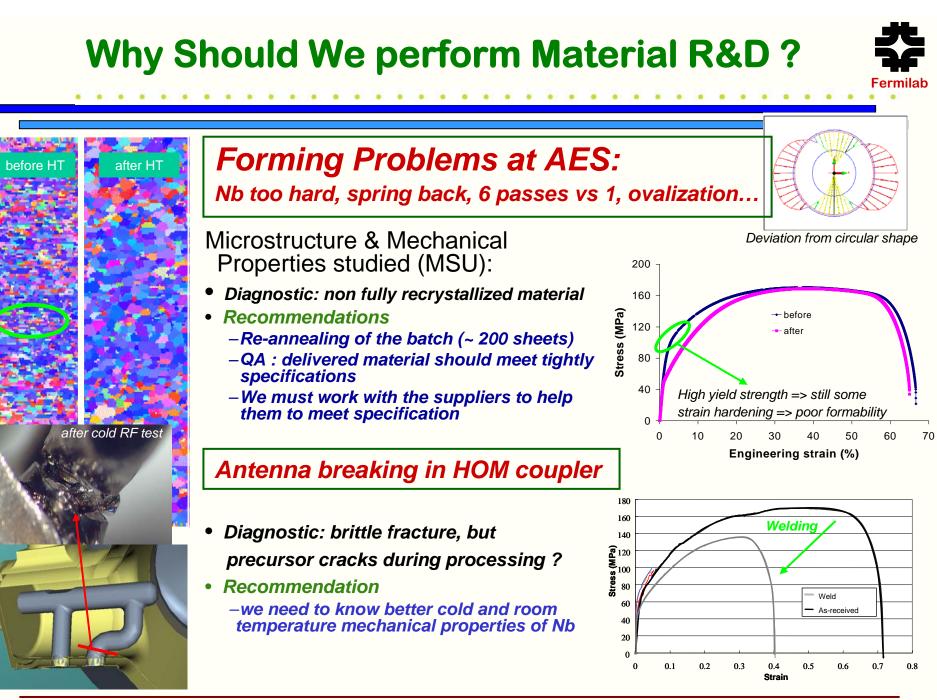


- <u>Ongoing</u>
  - Surface 3D microprobe analysis (with NU)
  - Magnetic characterization, magneto-optic, critical current, influence of grain boundary, baking (with FSU, ex Wisconsin)
  - Mechanical characterization, texture analysis (with MSU)
- <u>Short term activities</u>
  - Recrystallization study, cold and RT mechanical properties (1-2 year post doc)
  - Development of large grain/monocrystal cavity fabrication (project, not necessarily within the material's group)
  - Magnetometry on monocrystals (e.g. Fermi local PhD program), sensitivity of grain orientation to the processing \*
  - Rs low field measurement with RF microscope, Theory of SRF (at FSU\*\*)

### Mid/long term activities

- nm thin films of e.g. MgB2 on medium/large grain Nb cavities ; collaboration with Argonne, FSU and Penn State U \*.
- Superconducting Gap measurement by photoemission and STM ; collaboration with Argonne, IIT \*.

\* Investment needed \*\* developed else where



Feb 13-14, 2007

200

### Why Should We perform Process R&D?



### Surface processing 1

Large spread of results for electropolishing (EP)

#### Why are EP results are not reproducible ?

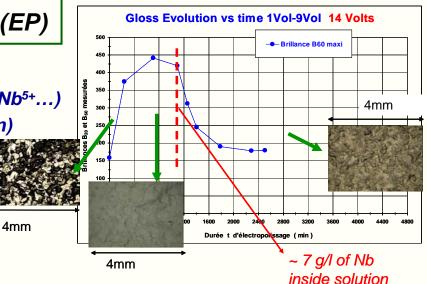
- -Aging of the solution, evolution of the composition (F, Nb<sup>5+</sup>...)
- -Impurities, particles generation (Sulfur vs field emission)
- Variation of the surface composition ?
- Variation of the surface roughness ?

#### Surface processing 2

Field emission is the major practical limitation

#### What are the possible sources?

- Poor control of the wet process : particle counting is not effective
- -Poor cleaning of the ancillaries : e.g. couplers
- -Contamination during assembly : long, complex, man-made
- -Absence of post processing solution

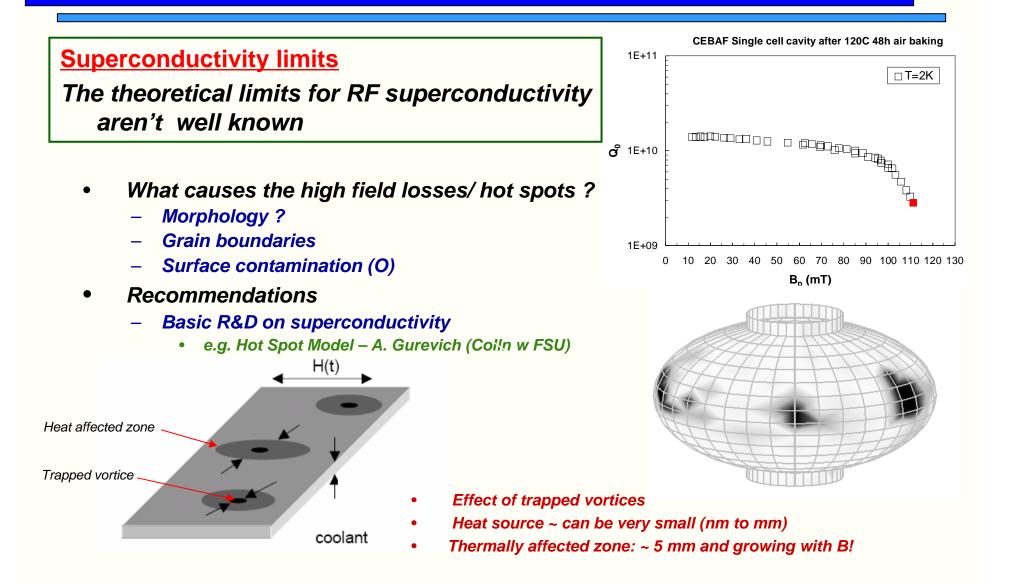


#### **Recommendations**

- Need to do R&D on samples, 1-cells before 9-cells
- Developing monitoring (F<sup>-</sup>, Nb<sup>5+</sup>...)
- Modeling
- Surface studies (composition, morphology)
- Develop new designs/tooling to ease assembling (Collabn Jlab)
- Develop post processing applicable to assembled cavities (e.g. Plasma cleaning w ECR plasma)

# Why Should We perform Advance Material R&D? (1/3)



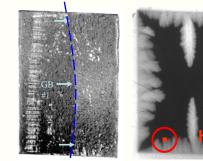


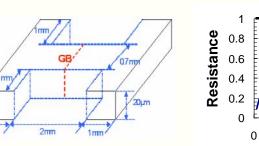
Why Should We perform Advance Material R&D? (2/3)



Morphological effect or depleted SC ?

### Flux penetration @ GB





[A. Polyanskii et al, WU/FSU]

[Sung Hawn]

Magnetic field Saturation-field H0 gives information on de-pairing J<sub>d</sub> of SC GB

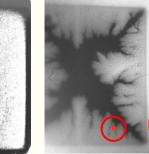
40

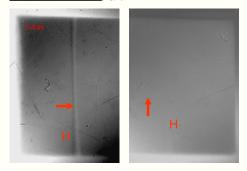
H0

20

### @ artificial notch







There is a local field enhancement due to roughness

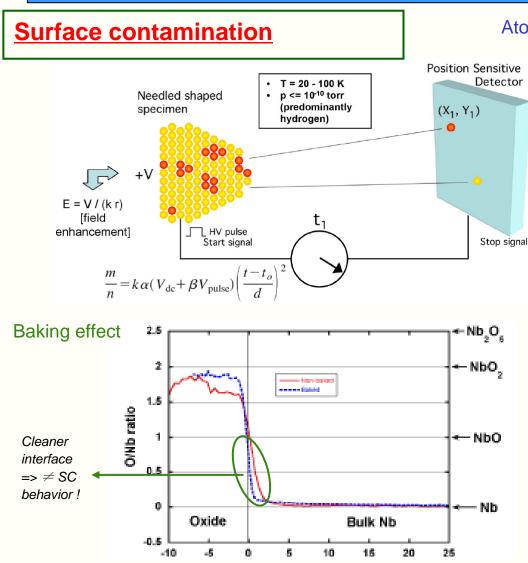
R0

60

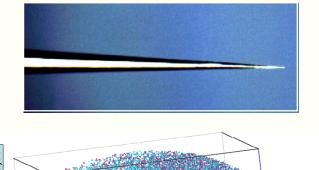
80

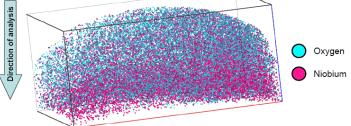
### Why Should We perform Advance Material R&D? (3/3)





#### Atom-probe tomography (APT) [Collbn University]





- Atomic resolution !!!
- Very sensitive

#### **But**

- No direct chemical information
- Complex => low turnover
- Need to be completed with other techniques

# Priority # 1 Single cell test program



#### R&D aims at 1 performances => ultimate test is cavity !

### Process R&D (ILC-S0)

- Reproducibility of the tight loop processing
- Alternative rinsing (ethanol, degreasing)
- Pre-processing (tumbling)
- Baking study

# SRF R&D (1/5 tests in the 1<sup>st</sup> 2 years)

- Post processing (plasma cleaning)
- Large grain
  - Grain size, orientation
  - Grain boundary dynamics
  - Processing optimization
- Beyond Nb (2-3 years from now)
  - e.g. MgB<sub>2</sub> on large grain
  - Collaboration W. U
  - Few tests



J-Lab Setup

# Single cell test program

	Priority Description	Manpower @ Fermi	M&S	Time scale	Comments
1	<ul><li>1 Cell test stand</li><li>Setup</li></ul>	1.00 FTE ENG 0.60 FTE DES	\$235K	1 year	Includes 3 1-cell cavities
	● Program ○S0 ○SRF R&D	0.50 FTE TEC 0.75 FTE ENG 0.50 FTE TEC	\$100K \$60K	1 year startup	Helium + small material 3 additional cavities
	Total FTE = 1.75 ENG + 1 TEC + 0.6 DES = 3.35 Total M&S \$395				

Fermilab

. .

.

14

### 9-cell processing facility at FNAL design

- tooling, flange design (reduce field emission risk)
- Cavity assembly automation
- Dry processing (plasma cleaning)

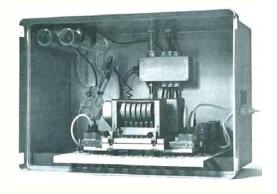
Along with the completion of the EP 9-cell infrastructure design and fabrication at ANL already financed...

**Priority # 2 Mid term / Process R&D – EP and...** 

• EP Modeling

(if HF work at FNAL = authorized...)

- Upgrading 1cell EP set up from 3.9 GHz to 1.3 GHz
  - Issues : e.g. New end parts
- Online Fluorine monitoring
  - Issues : large volume of consumables + wastes...









•

0.25 FTE DES 0.25 FTE TEC     0.25 FTE DES 0.25 FTE TEC     0.25 FTE TEC       ○Modeling     1.00 FTE STU     \$5K     1 year       ○Tumbling     0.25 FTE ENG     \$10K     1 year startup	nments
Samples R&D0.50 FTE SCI 0.25 FTE TEC\$5K6 monthPart of programo1Cell set up0.25 FTE ENG 0.25 FTE DES 0.25 FTE TEC\$40K1 year startupStart 3.9 GHz atoModeling1.00 FTE STU\$5K1 yearOngoingoTumbling0.25 FTE ENG\$10K1 year startupOngoing	
0.25 FTE DES     0.25 FTE DES       0.25 FTE TEC     0.25 FTE TEC       ○Modeling     1.00 FTE STU       ○Tumbling     0.25 FTE ENG       \$10K     1 year       Ongoing	n at J-Lab
oTumbling 0.25 FTE ENG \$10K 1 year startup	nd design 1.3GHz
Processing facilities     1.00 FTE ENG     2 years     ANI collab Eff	
0.50 FTE DES	ort and FNAL
Field emission reduction	
oNew tooling for assembly0.5 FTE ENG 1.0 FTE DES\$30K1 yearJ-Lab collab. 1 year	tech at J-lab for 1
• Dry processing 1.5 FTE TEC \$5K 5 month No basic resea	-
0.5 FTE ENG oriented 3.9GH	z single test

Total FTE: 0.5 SC + 2.5 ENG + 2 TEC + 1.75 DES + 1STU

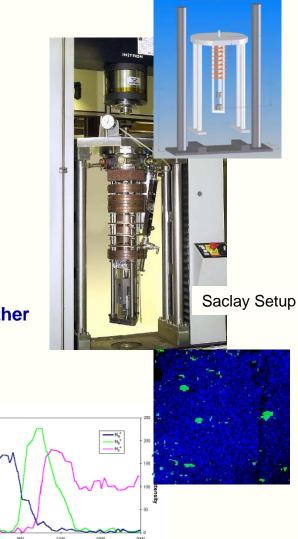
Total M&S \$95K

### # 3 Priority : Material Characterization : Mechanical, surface chemistry



#### Systematic testing of new batches (QA) + Failure analysis

- RT and Cold mechanical properties
  - data for modeling (forming, mechanical resistance, RF behavior...)
  - Recrystallization study (post doc student) => improving specifications for Nb
  - Crystal orientation/texture effects...
- Rapid SIMS characterization
  - High detection sensitivity (metal or non-metal)
  - Spatial resolution 10  $\mu$ m (horizontal) and 1 nm (depth)
  - Large size sample (100 mm round )
  - Very robust/reproducible analysis conditions => allows to gather statistics
- Additional benefit
  - Hydrogen, oxygen embrittlement at low temperature
  - Effect of welding (mechanical, chemical)
  - Grain boundary strength, composition
  - Oxide layer study
  - Weaker layer study/Coating study



Time / s

# **Material testing**



. .

	Priority Description	Manpower @ Fermi	M&S	Time scale	Comments
3	Material testing				
	<ul> <li>Cold test and recrystallization study</li> </ul>	1.0 FTE STU 0.25 FTE TEC	\$25K	1 year	6 month facility upgrade, open ended for material
	Surface analysis	0.25 FTE ENG	\$200K		study
	<ul> <li>Eddy Current</li> <li>Scanning &amp; RRR</li> </ul>	0.25 FTE ENG 1.00 FTE TEC	\$10K	ongoing	operation support M&S
	Total FTE = 1.0 STU + 1.25 TEC + 0.5 ENG = 2.75 Total M&S \$235				

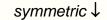
## # 4 Priority : monocrystal Nb program

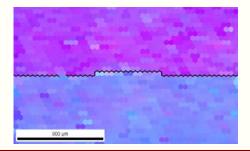
2 complementary goals :

- Developing local expertise on the fabrication process:
  - ~ 10-15 1-cell cavities project
  - 3.9 GHz then 1.3 GHz :
- R&D program on sample: (PhD or post Doc students)
  - Sensitivity of the crystalline orientation to :
    - Hydrogen loading
    - Formability ? e.g. (111) = more favorable for small grain textures
    - EP vs BCP, Oxygen diffusion ? (111) = close packed/ (001) = loose packed
    - EP vs. BCP, Oxide thickness ? (idem)
    - Surface B<sub>c</sub> ? (B<sub>c3</sub>), Superconducting gap ?
    - Recrystallization @ welding...
  - Can/ should be completed w surf Analysis

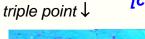
#### grain orientations:











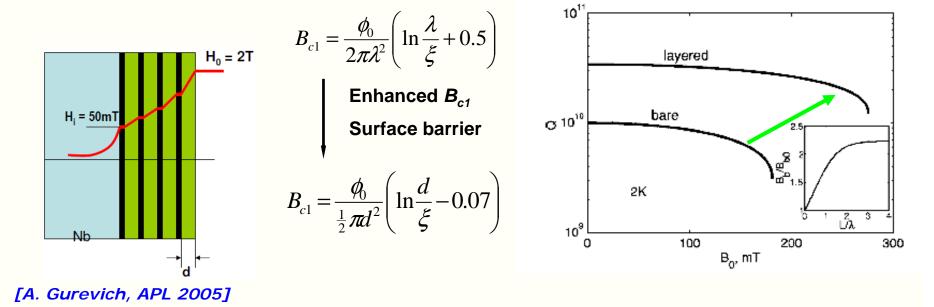
[collabn, MSU]



Feb 13-14, 2007

**DOE SCRF Review** 

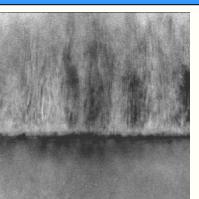
MgB<sub>2</sub> [X. Xi. Penn State]



Longer term / SRF R&D: S-I-S Multilayer

Single cell test program, collaboration with Universities

- **Theory (FSU-National High Magnetic Field** Lab)
- MgB<sub>2</sub> (ANL, Penn State)
- NbN (ANL, JLAB)
- Nb<sub>3</sub>Sn (ANL, JLAB)









.

	Priority Description	Manpower @ Fermi	M&S	Time scale	Comments
4	•	1.0 FTE STU ? 1.0 FTE SCI 0.25 FTE TEC	\$250K \$50 K	2 years	Magnetometer (external funding?) Collaborations (external funding?) Fermi contribution: •Sample and small material @ short term •1-cell testing @ mid term
	•Multilayer S-I-S				
	Total FTE: 1 SC + 1 STU + 0.25 TEC			Total M&S \$300K	

## Collaborations



Cavity Processing, assembling... (S0 for ILC ...) Jlab, Cornell, ANL **Chasing Hot Spots** Micro & macro scale Local variations in SC properties? Magneto-optics and Transport / ASC-FSU • **Defects**, Impurities? Local nano-chemistry – 3DAP / NU Local superconducting gap - ANL+ IIT **Thermal Properties?** Thermal conductivity and Kapitza – MSU **Beyond Niobium** Fundamentals of SC, theory / ASC-FSU **Multilayers SIS Deposition process ANL/ Penn state** Local nano-chemistry – 3DAP / NU, ANL <u>Organization of the 1rst extended SRF Material workshop</u> (Spring 2007) Fermi





Program	Total FTE	Total M&S
1-Cell RF test stand	1.75 ENG + 1 TEC + 0.6 DES = 3.35	\$395
Surface processing R&D	0.5 SC + 2.5 ENG + 2 TEC + 1.75 DES + 1 STU = 4.75	\$95K
Material testing	1.0 STU + 1.25 TEC + 0.5 ENG = 7.75	\$235
Advanced SRF R&D	1 SC + 1 STU + 0.25 TEC = 2.25	\$300K

### Total FTE: 15.10

### 1.5 SC + 4.75 ENG + 4.5 TEC + 2.35 DES + 2 STU

(comp. with present: 1 SC + 5 ENG + 3.5 TEC + 2 DES)

Total M&S:

\$690 K +\$ 300K AARD





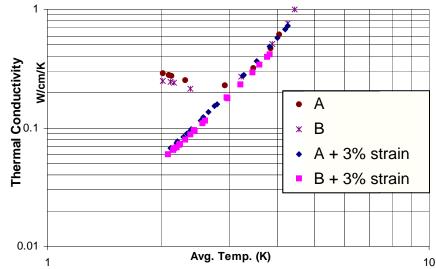
- SRF material issues affect all kind of SRF projects
- 3 main activities for the material group
  - Support to project : QA, failure analysis
  - Process R&D
  - Advanced SRF R&D
- Advanced SRF R&D is done mainly /collaborations
  - Grouped AARD proposal (ANL, FSU, MSU, NU...)
- Fermi needs to expand its advanced SRF R&D program:
  - Benefit from advanced knowledge on SC
  - Scientific leadership in SRF
  - Improvement of projects (cost, reliability, performance)
- The material group needs to be reinforced (Sc/Eng, Tec, Des)



Additionnal slides...

# **Thermal behavior**

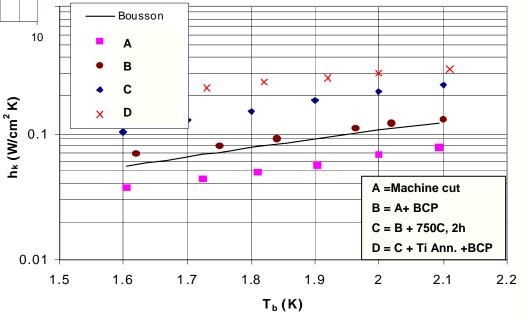




### Unexpected results !!!!!

- Kapitza conductance is improved with annealing
- It is not much affected by roughness

- Thermal conductivity @ 2 K decreases dramatically with strain (80%!)
- It is not recovered with 750C, 2h
- It is recovered with Ti annealing (1300-1400 C)
- Intermediate temp need to be tested



#### [CollnMSU]

### TIG Welding Study



Innovation:

Tig Welding experience @ MSU + Ultra pure Ar developed @ FNAL











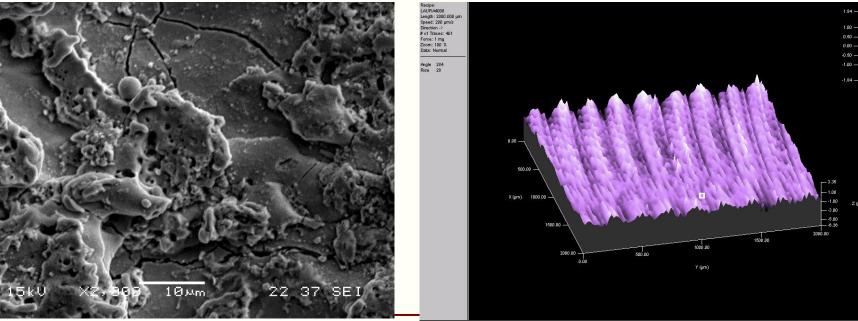


Surface Roughness and Chemistry:

Example: Study of different cutting techniques by C. Cooper / FNAL

# **SEM Images of EDM Wire Cut Sample at Various Magnifications**

EDM Cut Surface SEM



Milled Surface Topology

Feb 13-14, 2007

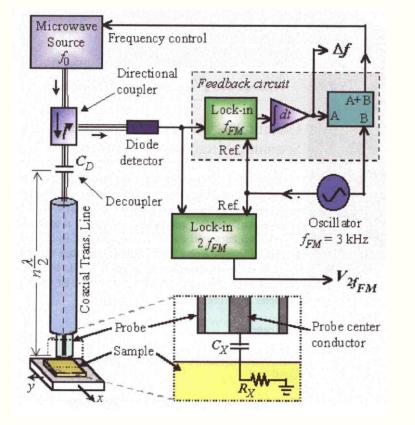
**DOE SCRF Review** 





.

### RF microscopy



### Superconducting gap measurement (Photoemission)

