

# SRF Material R&D

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# SRF Materials Issues



*Collaborations with universities + SRF labs*

## Understanding SRF physics

- High Field dissipation
- Quench
- Surface resistance
- Thermal behavior
- *Hot spots*
- *Surface nano-analysis*
- *Magneto-optics*
- *Thermal conductance*
- *Kapitza resistance*
- *Beyond Nb...*

*Collaborations with industries*

## Processing

- Specification
- Reproducibility
- Cost issues
- *EP facility Design - Construction*
- *EP Mechanism*
  - *Modeling (hydrodynamics)*
- *Pre-processing (tumbling)*
- *Post-processing (rinsing)*

## Large scale Nb supplying QA

- Specification
- Reproducibility
- Cost issues
- *Mechanical properties*
- *Recrystallization*
- *Texture/Orientation issues*
- *Forming process*
- *Fine grain/Large grain*

# SRF Materials group



## Scientists:

**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)

**F. McConologue\*** ( FTE Processing R&D)

**Designer 1 cell\*** ( 1 Cell Program)

## Tech:

**D. Bice** (Processing R&D @ J-Lab)

**D. Burke\*** (Processing Lab support)

**O. Frianeza** (Processing R&D, SRF Materials Lab)

**R. Schuessler** (SRF Materials Lab)

FTE:

1 SC

5 ENG

3.5 TEC

2 DES

\* Not full time

# Improvement of QC/QA and Support to projects

- Ongoing
  - 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*
- Mid-long term activities
  - *Squid Eddy Current scanning (sheets, cavities) ? \*\*.*
  - *Field emission scanner ? \*\**

\* Investment needed  
\*\* developed else where

# Process R&D Activities

- Ongoing
  - 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...*)
- Mid term activities. *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 9-cells)
  - Alternative rinsing (ethanol, degreasing)
  - Baking study
  - Feasibility of online F monitoring on the EP set-up.

# Materials R&D Activities



- Ongoing

- 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)

- Short term activities

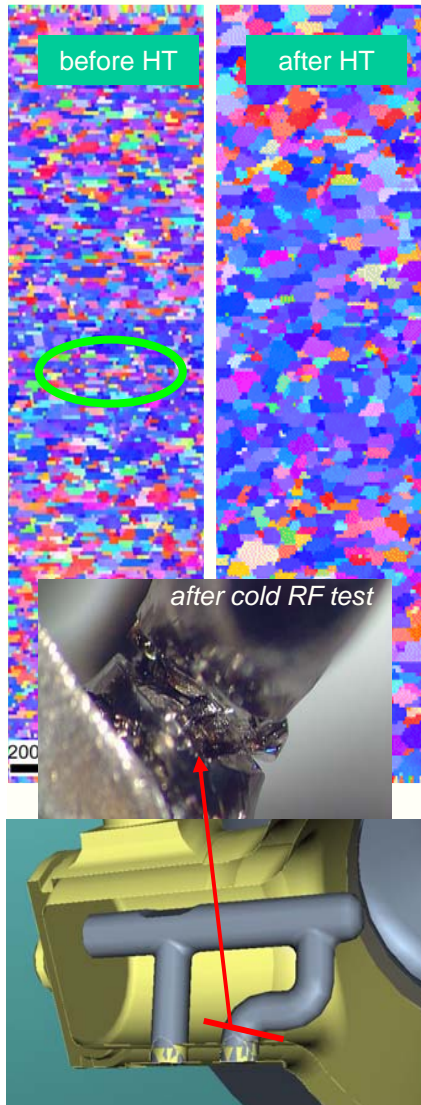
- Recrystallization study, cold and RT mechanical properties (1-2 year post doc)
- Development of large grain/monocrystal cavity fabrication (project, not necessarily within the material's group)
- Magnetometry on monocrystals (e.g. Fermi local PhD program), sensitivity of grain orientation to the processing \*
- Rs low field measurement with RF microscope, Theory of SRF (at FSU\*\*)

Mid/long term activities

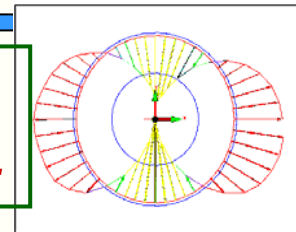
- nm thin films of e.g. MgB<sub>2</sub> on medium/large grain Nb cavities ; collaboration with Argonne, FSU and Penn State U \*
- Superconducting Gap measurement by photoemission and STM ; collaboration with Argonne, IIT \*

\* Investment needed  
\*\* developed else where

# Why Should We perform Material R&D ?



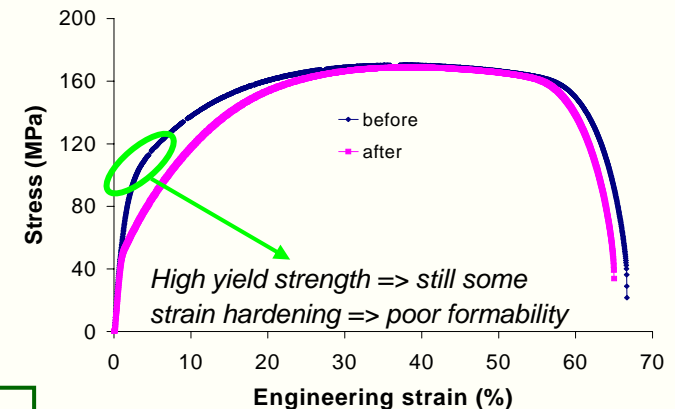
## Forming Problems at AES: Nb too hard, spring back, 6 passes vs 1, ovalization...



Deviation from circular shape

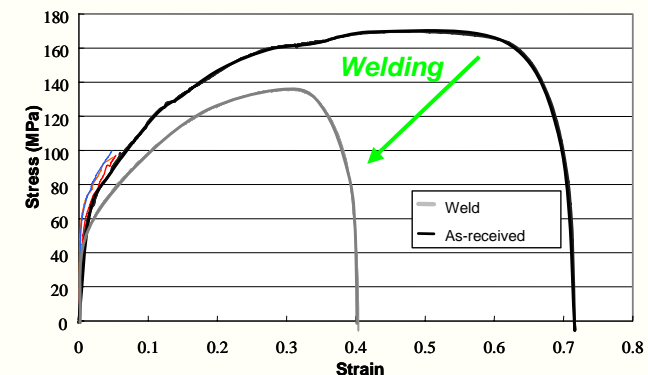
Microstructure & Mechanical Properties studied (MSU):

- **Diagnostic:** non fully recrystallized material
- **Recommendations**
  - Re-annealing of the batch (~ 200 sheets)
  - QA : delivered material should meet tightly specifications
  - We must work with the suppliers to help them to meet specification



## Antenna breaking in HOM coupler

- **Diagnostic:** brittle fracture, but precursor cracks during processing ?
- **Recommendation**
  - we need to know better cold and room temperature mechanical properties of Nb



# Why Should We perform Process R&D ?

## Surface processing 1

### *Large spread of results for electropolishing (EP)*

Why are EP results are not reproducible ?

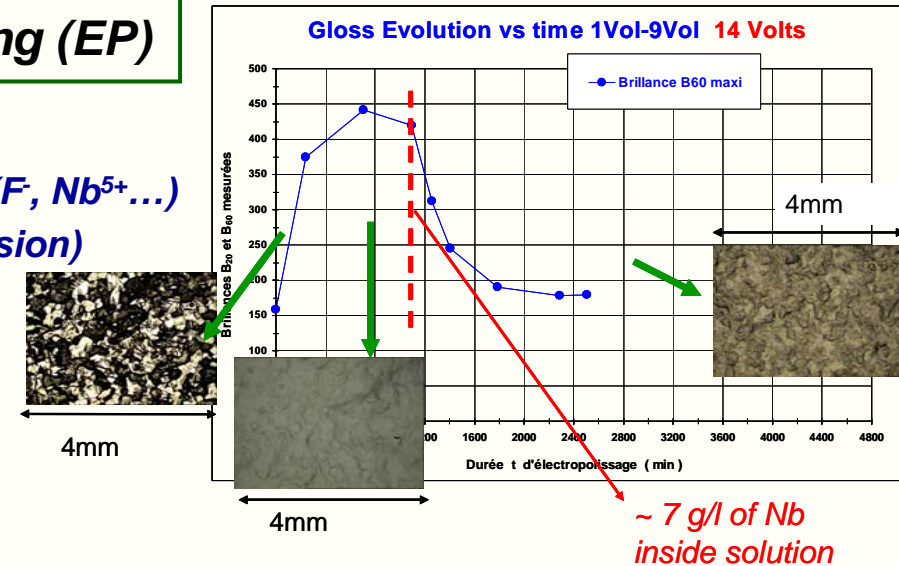
- Aging of the solution, evolution of the composition ( $F^-$ ,  $Nb^{5+}$ ...)
- Impurities, particles generation (Sulfur vs field emission)
- Variation of the surface composition ?
- Variation of the surface roughness ?

## 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 ( $F^-$ ,  $Nb^{5+}$ ...)
- 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)

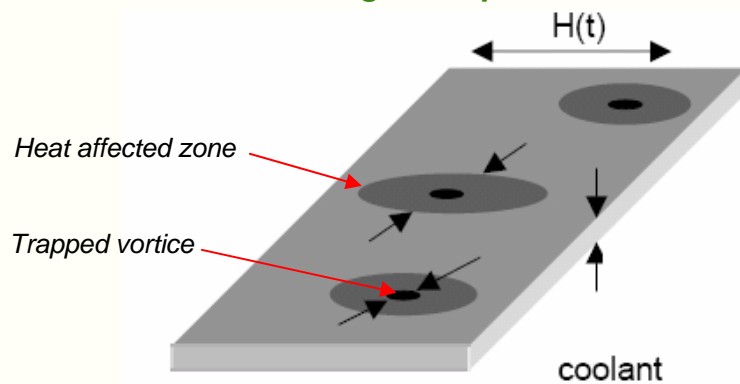


# Why Should We perform Advance Material R&D? (1/3)

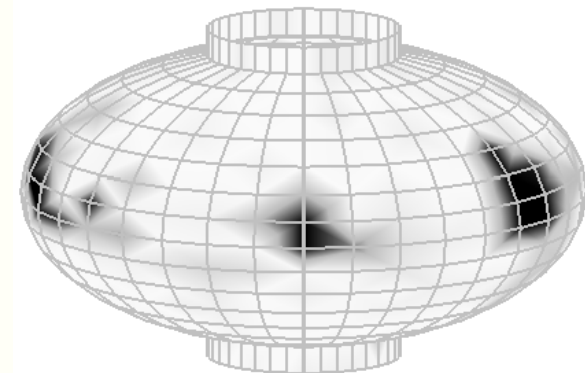
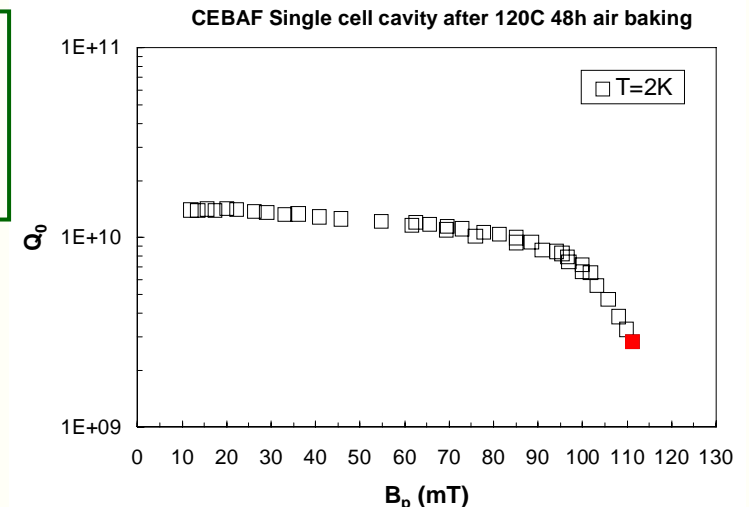
## Superconductivity limits

*The theoretical limits for RF superconductivity aren't well known*

- **What causes the high field losses/ hot spots ?**
  - Morphology ?
  - Grain boundaries
  - Surface contamination (O)
- **Recommendations**
  - Basic R&D on superconductivity
    - e.g. Hot Spot Model – A. Gurevich (Coil:n w FSU)



- **Effect of trapped vortices**
- **Heat source ~ can be very small (nm to mm)**
- **Thermally affected zone: ~ 5 mm and growing with B!**

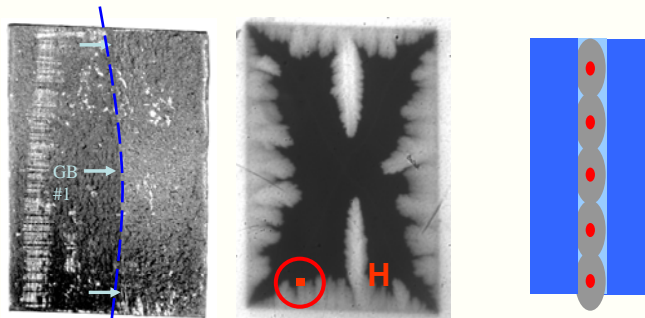


# Why Should We perform Advance Material R&D? (2/3)

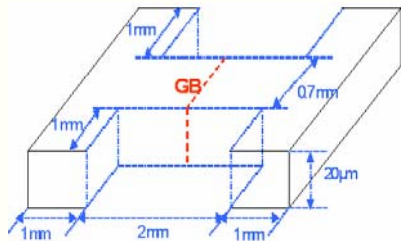
## What is the problem with GB ?

*Morphological effect or depleted SC ?*

Flux penetration @ GB

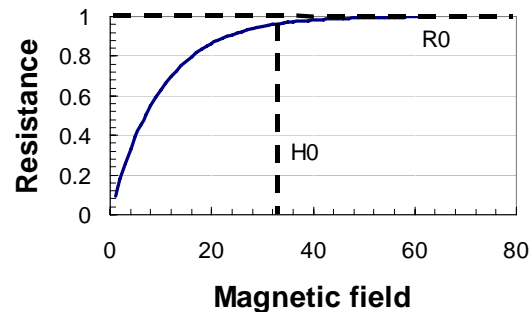


[Collabo. WU/FSU]



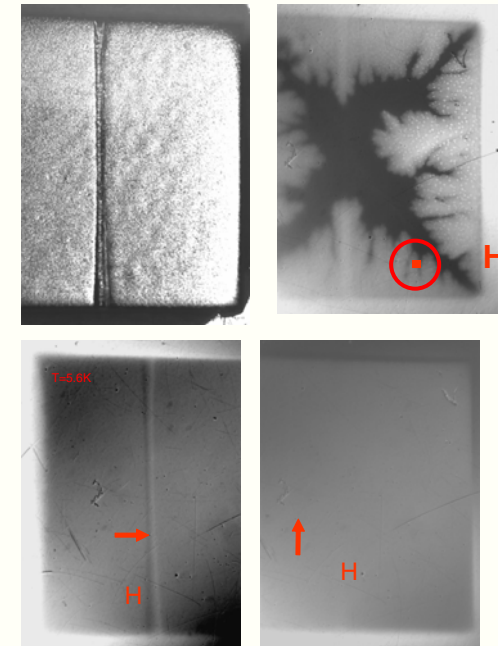
[A. Polyanskii et al, WU/FSU]

[Sung Hawn]



Saturation-field  $H_0$  gives information on de-pairing  $J_d$  of SC GB

@ artificial notch

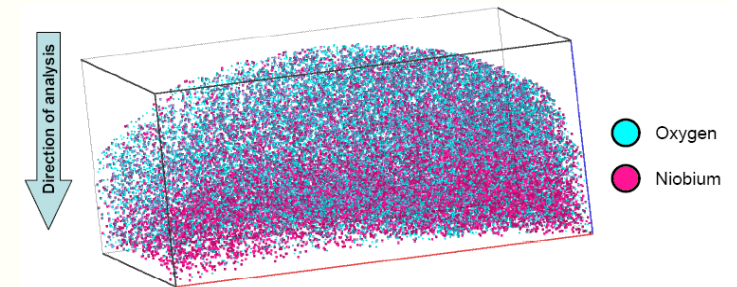
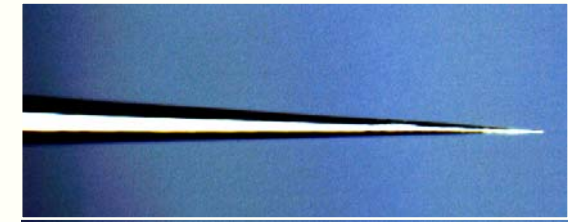
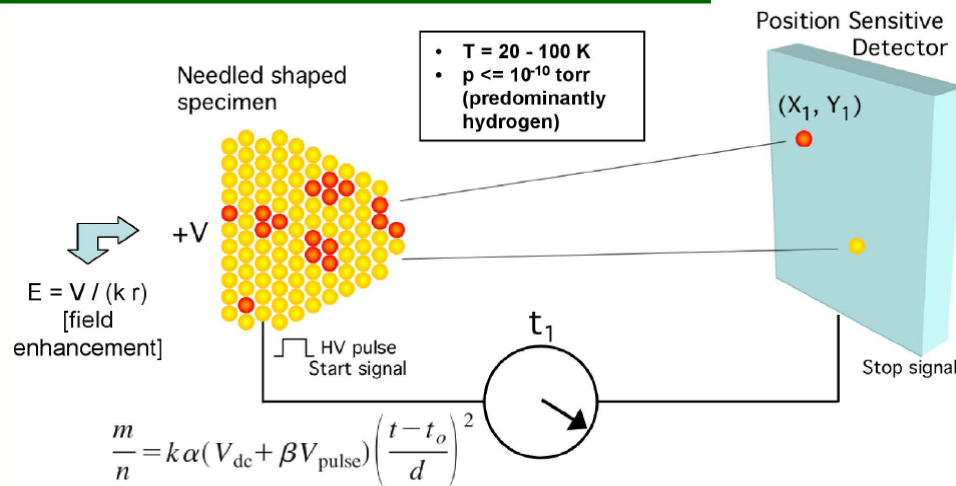


There is a local field enhancement due to roughness

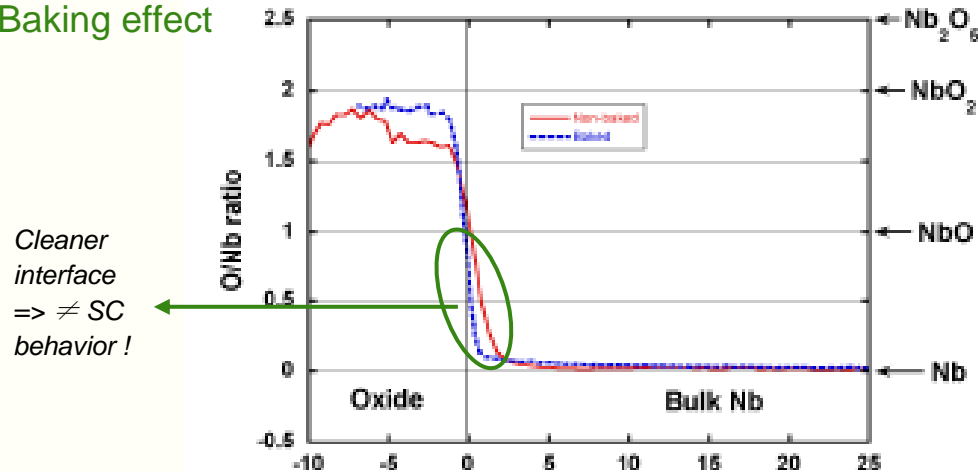
# Why Should We perform Advance Material R&D? (3/3)

## Surface contamination

Atom-probe tomography (APT) [Collbn University]



## Baking effect



- **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

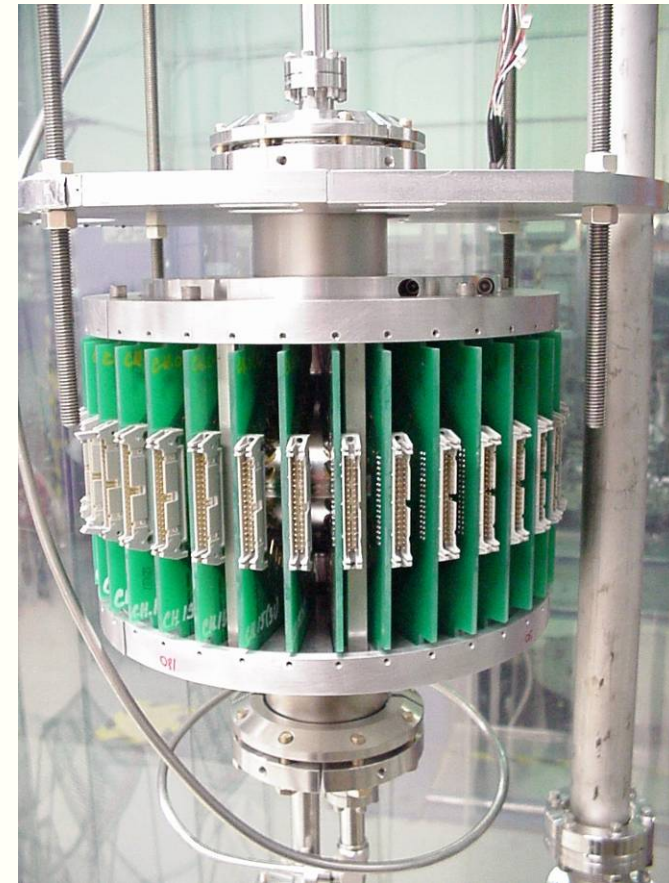
R&D aims at ↑ performances => ultimate test is cavity !

## 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

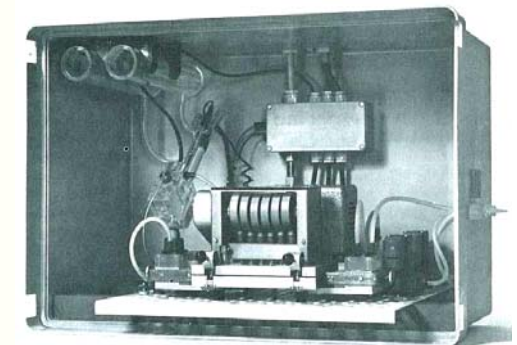
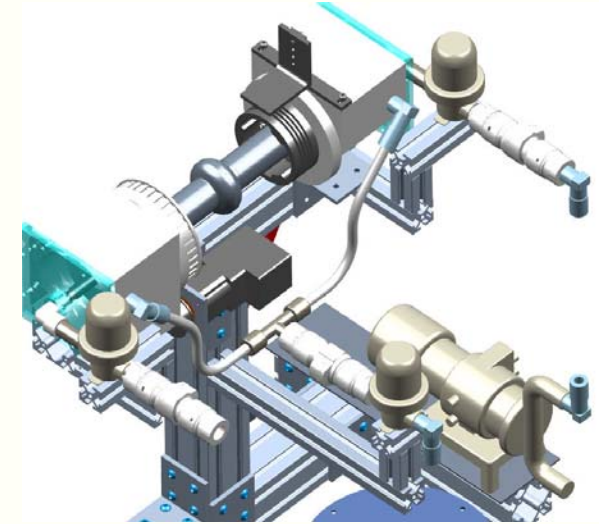


	Priority Description	Manpower @ Fermi	M&S	Time scale	Comments
1	1 Cell test stand				
	<ul style="list-style-type: none"> <li>• Setup</li>   <li>• Program                             <ul style="list-style-type: none"> <li>○S0</li> <li>○SRF R&amp;D</li> </ul> </li> </ul>	1.00 FTE ENG 0.60 FTE DES  0.50 FTE TEC 0.75 FTE ENG 0.50 FTE TEC	\$235K  \$100K \$60K	1 year  1 year startup	Includes 3 1-cell cavities  Helium + small material 3 additional cavities
<b>Total FTE = 1.75 ENG + 1 TEC + 0.6 DES = 3.35</b>					<b>Total M&amp;S \$395</b>

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

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

- **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...**
- **9-cell processing facility at FNAL design**
- **tooling, flange design (reduce field emission risk)**
- **Cavity assembly automation**
- **Dry processing (plasma cleaning)**



# Process R&D

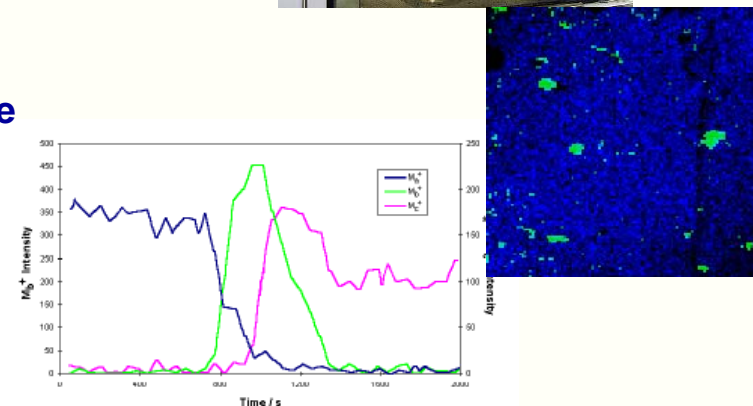


Priority	Description	Manpower @ Fermi	M&S	Time scale	Comments
2	Surface processing R&D				
	• EP processing				
	○ Samples R&D	0.50 FTE SCI 0.25 FTE TEC	\$5K	6 month	Part of program at J-Lab
	○ 1Cell set up	0.25 FTE ENG 0.25 FTE DES 0.25 FTE TEC	\$40K	1 year startup	Start 3.9 GHz and design 1.3GHz
	○ Modeling	1.00 FTE STU	\$5K	1 year	Ongoing
	○ Tumbling	0.25 FTE ENG	\$10K	1 year startup	
	• Processing facilities	1.00 FTE ENG 0.50 FTE DES		2 years	ANL collab. Effort and FNAL facility
• Field emission reduction					
○ New tooling for assembly	0.5 FTE ENG 1.0 FTE DES	\$30K	1 year	J-Lab collab. 1 tech at J-lab for 1 year	
○ Dry processing	1.5 FTE TEC 0.5 FTE ENG	\$5K	5 month	No basic research, but goal oriented 3.9GHz single test	
<b>Total FTE: 0.5 SC + 2.5 ENG + 2 TEC + 1.75 DES + 1STU</b>					<b>Total M&amp;S \$95K</b>

# # 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  $\mu\text{m}$  (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





# Material testing



Priority	Description	Manpower @ Fermi	M&S	Time scale	Comments
3	Material testing				
	<ul style="list-style-type: none"> <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 study
	<ul style="list-style-type: none"> <li>• Surface analysis</li> </ul>	0.25 FTE ENG	\$200K		
	<ul style="list-style-type: none"> <li>• Eddy Current Scanning &amp; RRR</li> </ul>	0.25 FTE ENG 1.00 FTE TEC	\$10K	ongoing	operation support M&S
<b>Total FTE = 1.0 STU + 1.25 TEC + 0.5 ENG = 2.75</b>					<b>Total M&amp;S \$235</b>

# # 4 Priority : monocrystal Nb program

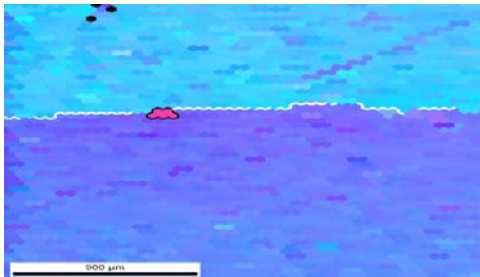
2 complementary goals :

- **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  $B_C$  ? ( $B_{C3}$ ), Superconducting gap ?**
    - Recrystallization @ welding...
  - **Can/ should be completed w surf Analysis**

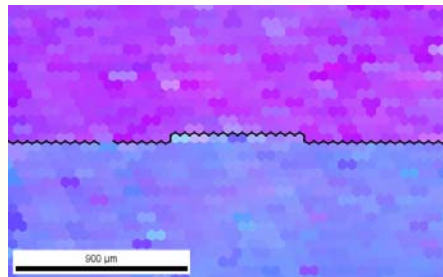


grain orientations:

asymmetric ↓

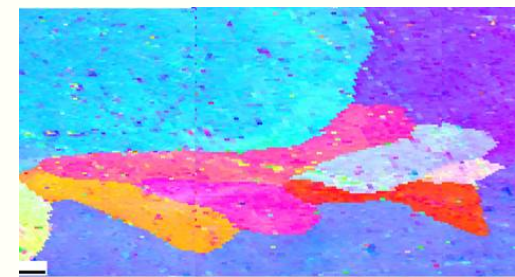


symmetric ↓



triple point ↓

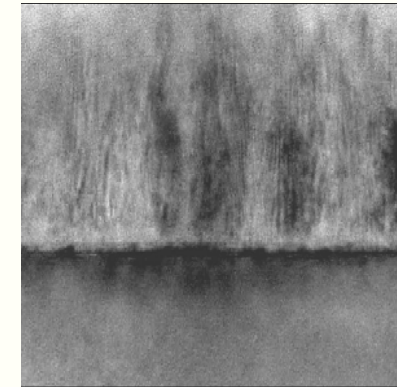
[collabn, MSU]



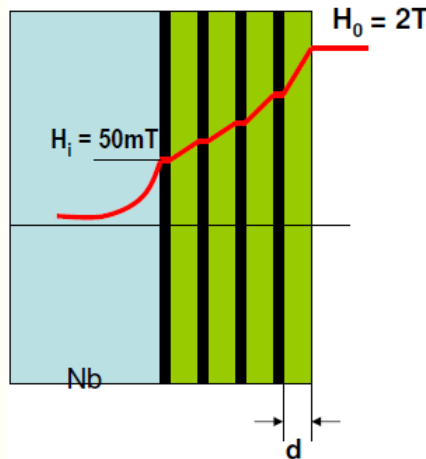
# Longer term / SRF R&D: S-I-S Multilayer

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)



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

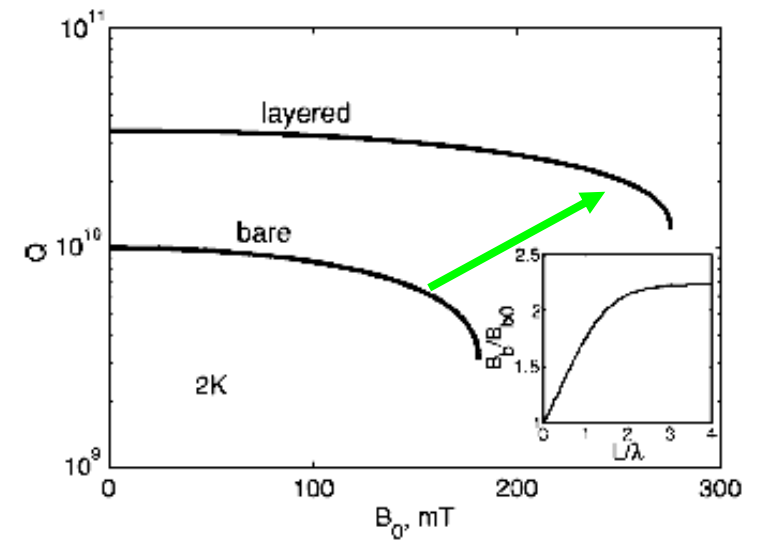


[A. Gurevich, APL 2005]

$$B_{c1} = \frac{\phi_0}{2\pi\lambda^2} \left( \ln \frac{\lambda}{\xi} + 0.5 \right)$$

Enhanced  $B_{c1}$   
Surface barrier

$$B_{c1} = \frac{\phi_0}{\frac{1}{2}\pi d^2} \left( \ln \frac{d}{\xi} - 0.07 \right)$$



# Advanced SRF R&D



Priority Description	Manpower @ Fermi	M&S	Time scale	Comments
<b>4</b> Advanced R&D <ul style="list-style-type: none"> <li>•Support to monocrystals prog</li> <li>•Superconducting Properties               <ul style="list-style-type: none"> <li>○Dissipation sources</li> <li>○Superconducting gap</li> </ul> </li> <li>•Surface fine studies               <ul style="list-style-type: none"> <li>○3D probe</li> <li>○Prime</li> </ul> </li> <li>•Multilayer S-I-S</li> </ul>	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
<b>Total FTE: 1 SC + 1 STU + 0.25 TEC</b>		<b>Total M&amp;S \$300K</b>		

# 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
- Organization of the 1st *extended SRF Material workshop* @ Fermi (Spring 2007)

# Summary



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**

# Conclusion

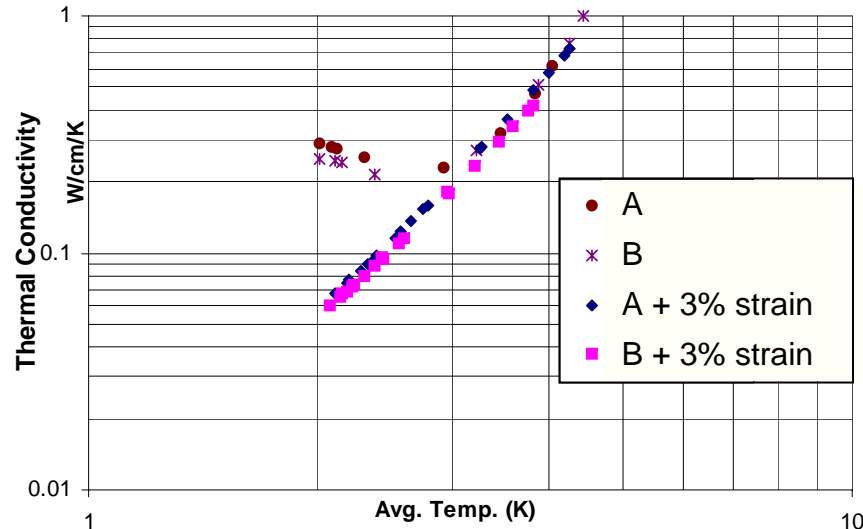
- **SRF material issues affect all kind of SRF projects**
- **3 main activities for the material group**
  - **Support to project : QA, failure analysis**
  - **Process R&D**
  - **Advanced SRF R&D**
- **Advanced SRF R&D is done mainly /collaborations**
  - **Grouped AARD proposal (ANL, FSU, MSU, NU...)**
- **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)**



**Additional slides...**



# Thermal behavior

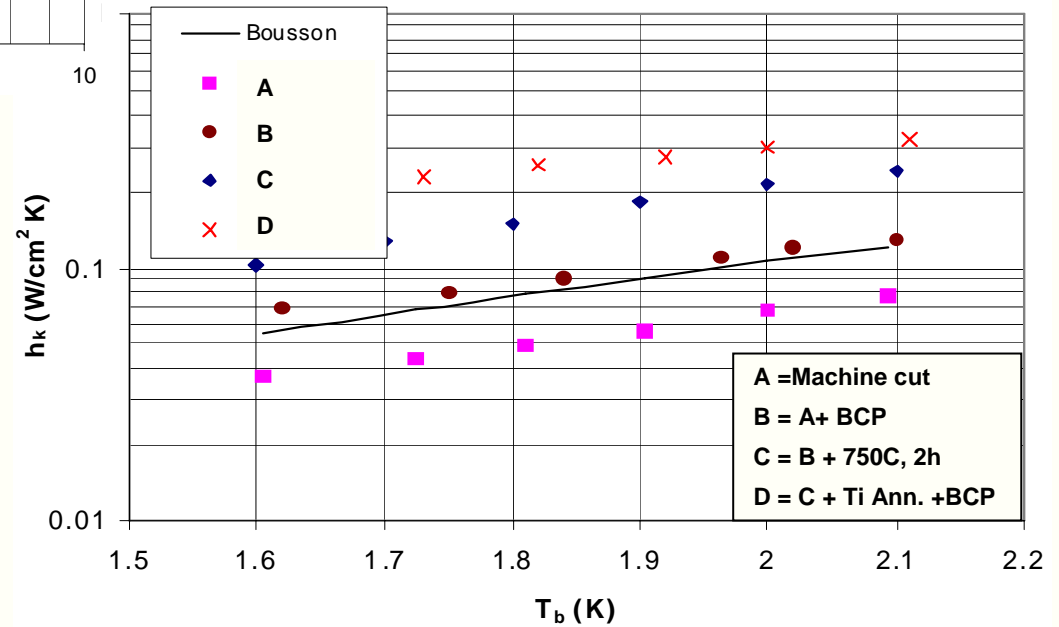


- Thermal conductivity @ 2 K decreases dramatically with strain (80%!)
  - It is not recovered with 750C, 2h
  - It is recovered with Ti annealing (1300-1400 C)
  - Intermediate temp need to be tested

## Unexpected results !!!!!

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

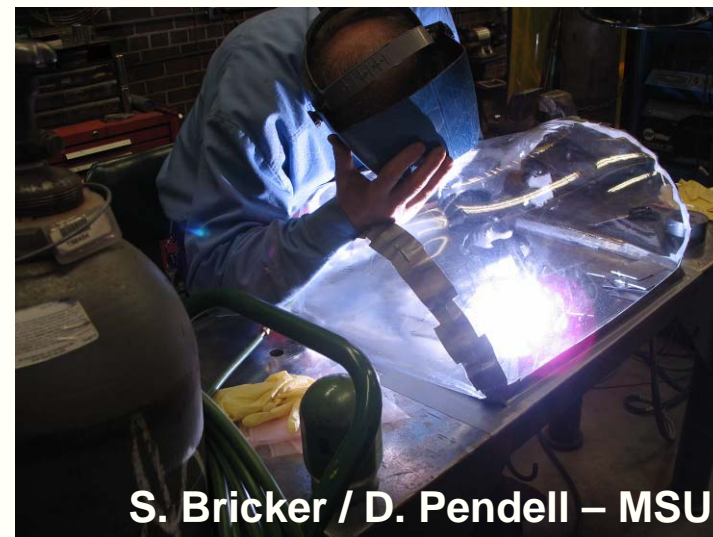
[CollnMSU]



# TIG Welding Study

Innovation:

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

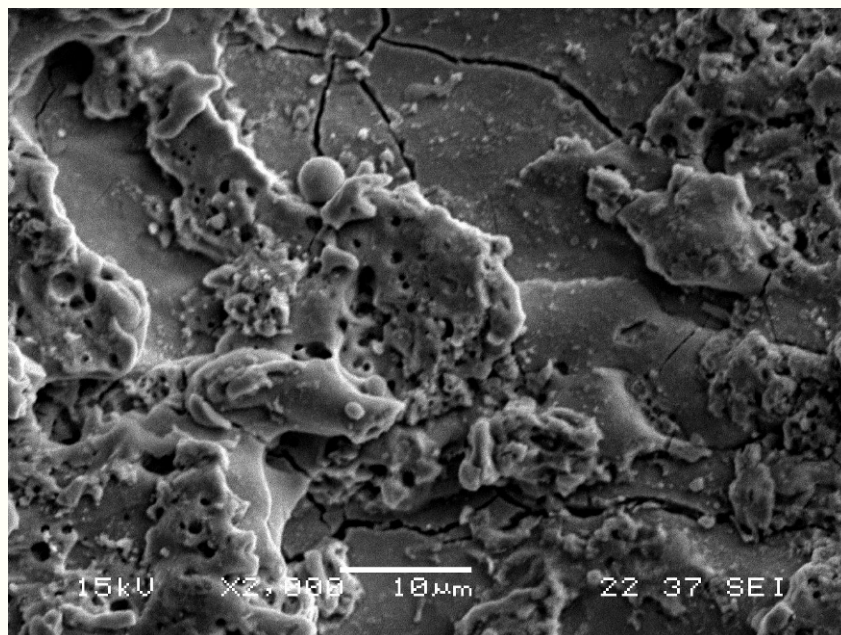


# Surface Topology

**Surface Roughness and Chemistry:  
Example: Study of different cutting techniques by C. Cooper  
/ FNAL**

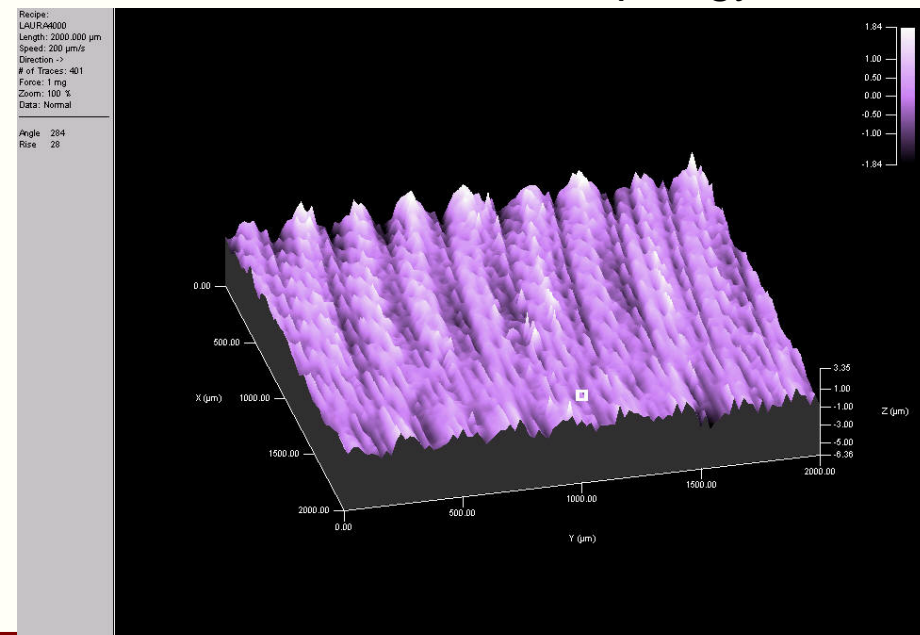
## **SEM Images of EDM Wire Cut Sample at Various Magnifications**

EDM Cut Surface SEM



Feb 13-14, 2007

Milled Surface Topology

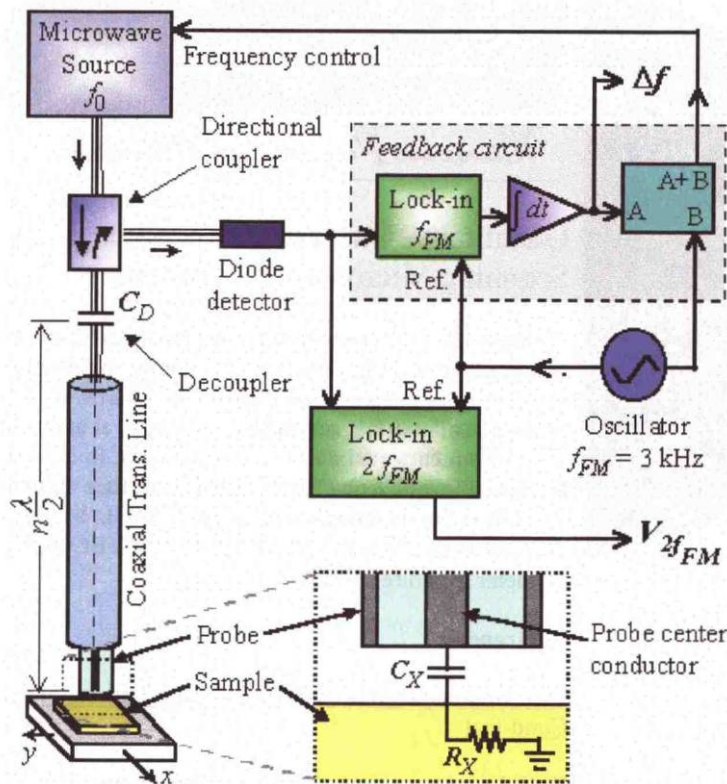


DOE SCRF Review

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# Others ideas ...?

## RF microscopy



## Superconducting gap measurement (Photoemission)

