An Investigation of the influence of grain boundaries on flux penetration in high purity large grain niobium for particle accelerators

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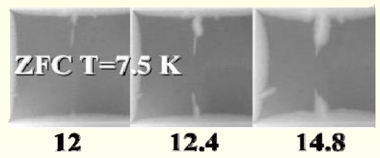
Motivation

- The performance of SRF Nb cavity is deeply influenced by the topmost surface layers (~40nm) of the material
- GBs in niobium cavities may be one of the important causes of extra power dissipation
- Our goal
  - To determine what BCP does both the surface and to the GBs
  - To understand whether or how grain boundary weakness can affect SRF cavity performance

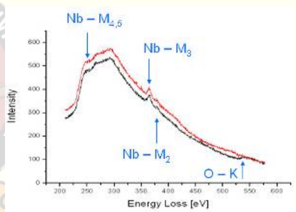
Experimental Procedure

- Selection of single & bi- xtals from large grain JLab source niobium sheet
- **I**-shape cutting & sample preparation

- **V-I** characterization (Transport measurement)
  - To measure the inter & intra - critical current density (J_b)
- Magneto Optical Visualization
  - To observe preferential flux penetration along grain boundary



- STEM and EELS (Electron Energy Loss Spectroscopy)
  - To investigate the topology of niobium oxides on the top surface

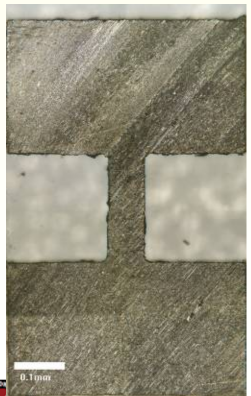


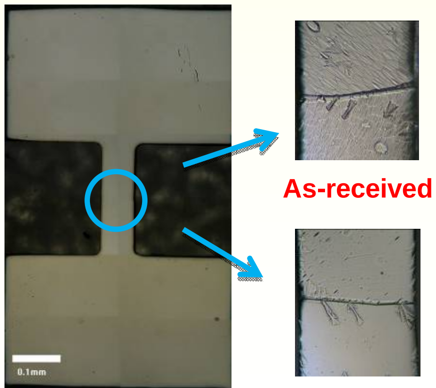
- All parts of the experiment have been done on <u>**ONE**</u> sample

Transport measurement

- **I**-shape Nb sample
  - ✓ Large grain JLab sample
  - ✓ Wire EDM
  - ✓ Pre-BCP (100µm) → mechanical polishing
  - ✓ Final thinning by BCP ~ 80-90µm
  - ✓ Dimension; Laser confocal microscope

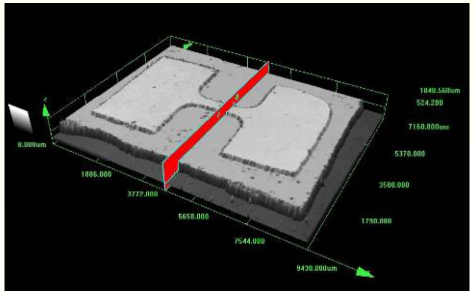
- Transport measurement
  - ✓ Sapphire plate for easy handling
  - ✓ Spring loaded pogo pins for connections
  - ✓ Measure V-I as a function of $H_{\perp}$



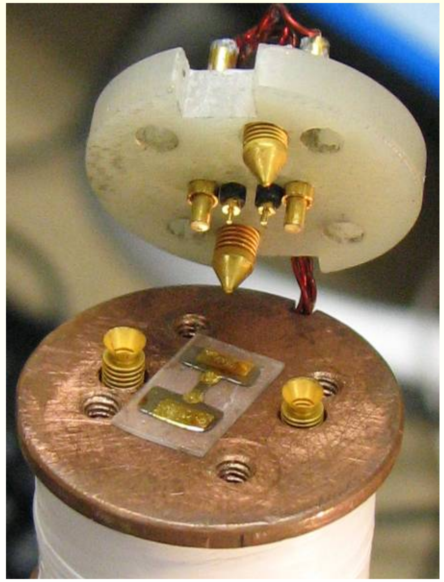


As-received side

EDM cutting side



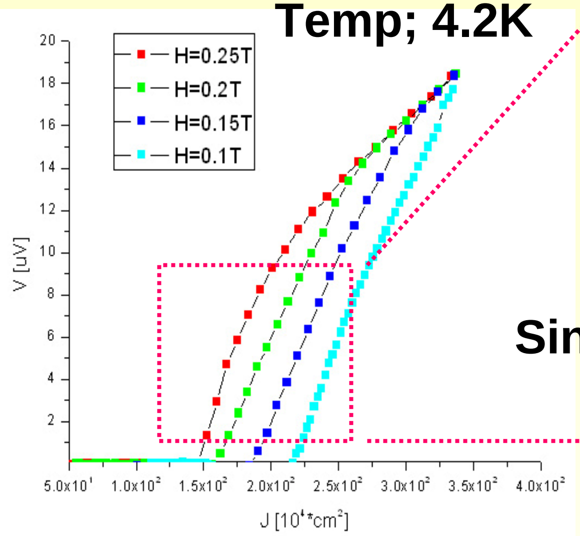
3D sample image

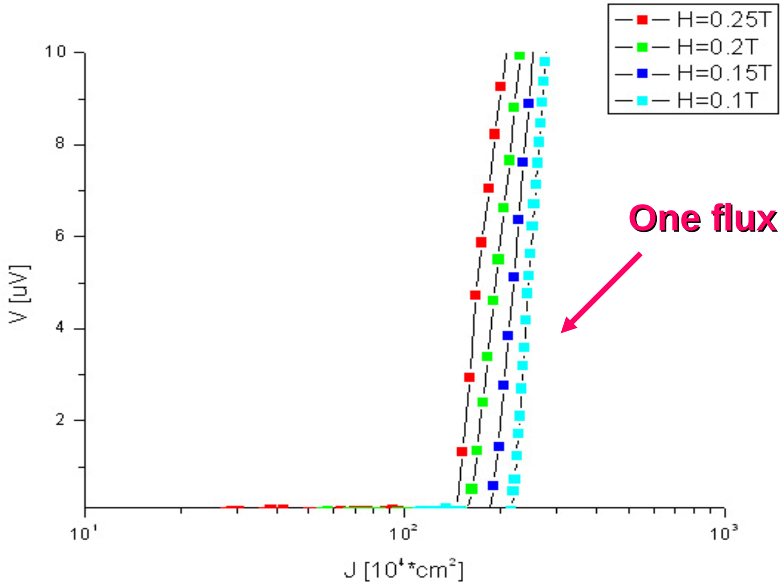


V-I response of Single & Bi- Xtals

Temp; 4.2K

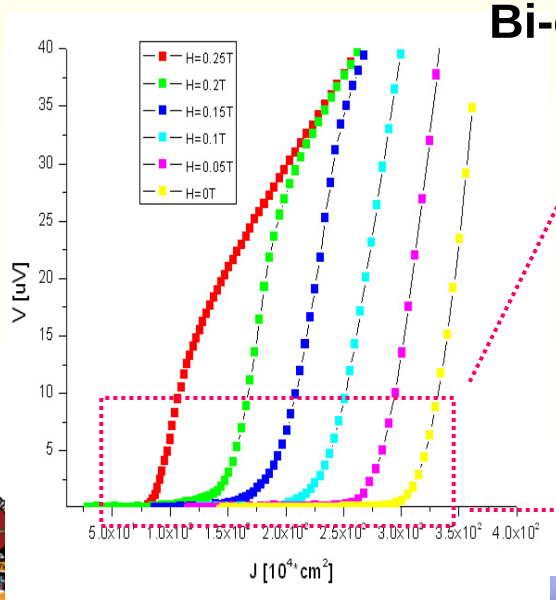
Single

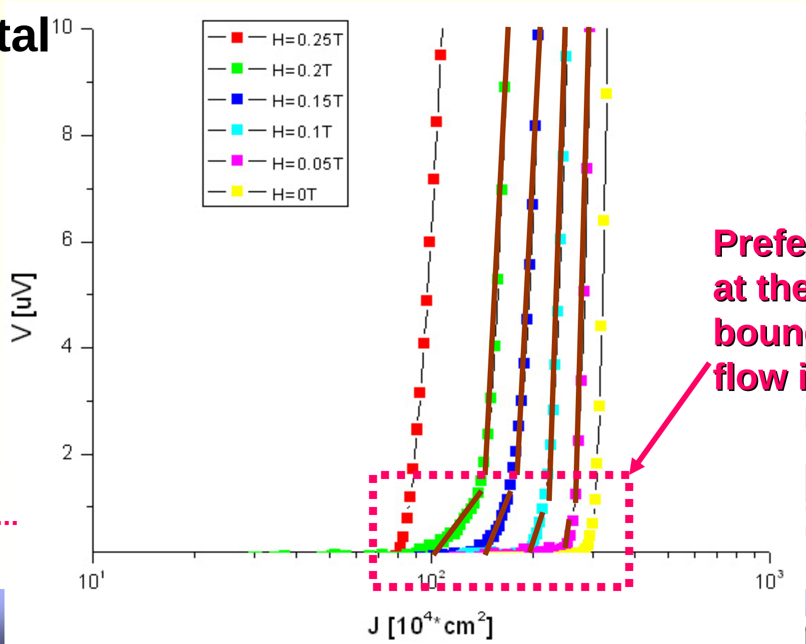




One flux flow event

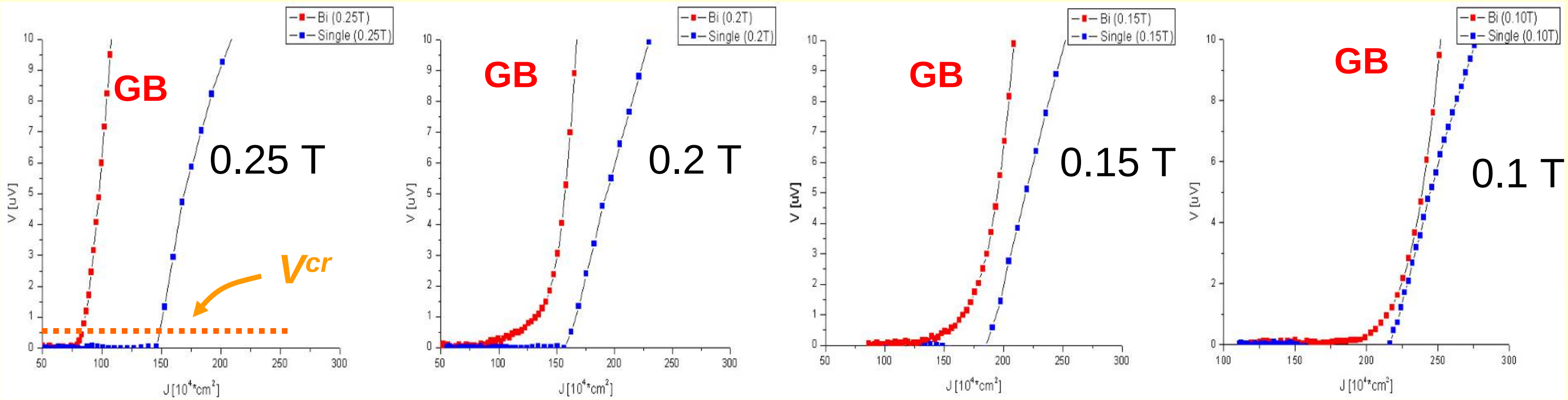
Bi-crystal





Preferential flux flow at the grain boundary before flux flow in the grain?

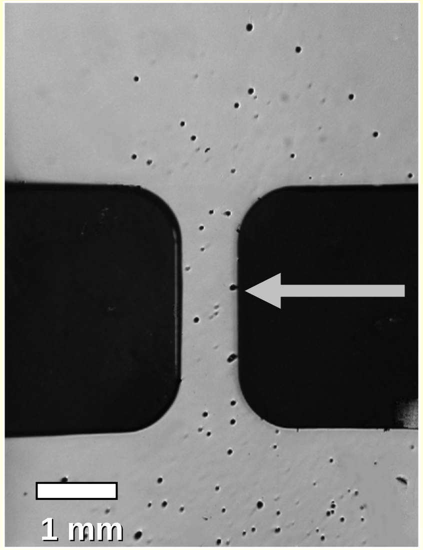
J_c comparison of Single & Bi X-tal



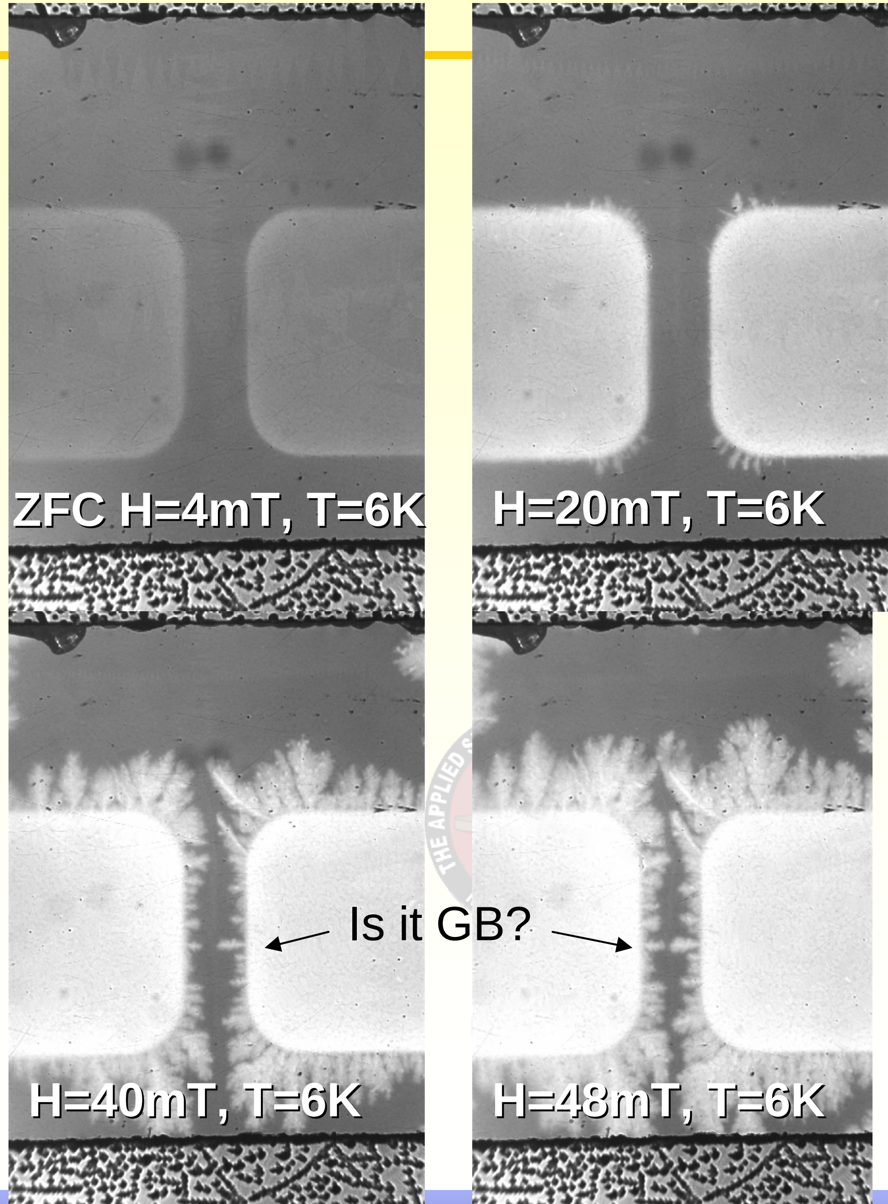
Field	Ic [mA] (Single)	Ic [mA] (Bi)	Jc [MA/cm^2] (Single)	Jc [MA/cm^2] (Bi)
H=0.25T	507.33	204.84	1.409	0.590
H=0.2T	588.93	374.89	1.636	1.079
H=0.15T	686.48	518.78	1.906	1.493
H=0.1T	793.17	682.97	2.203	1.965

$$J_{C,\,gb} < J_{C,\,grain}$$

MO: BEFORE final BCP thinning

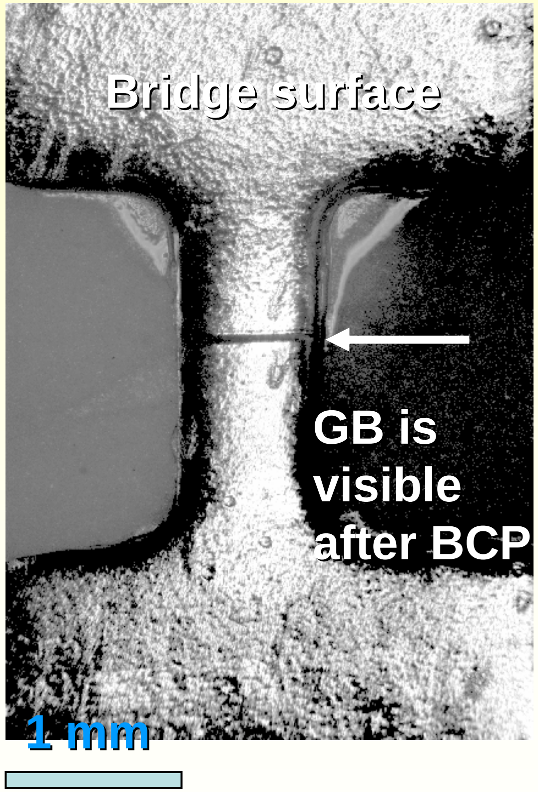


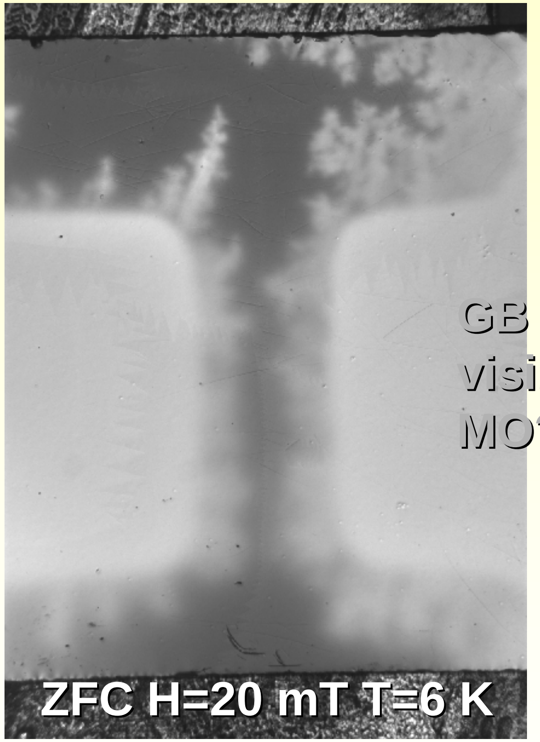
GB is not visible on bridge surface before BCP **(Thickness 127 µm, width ~ 536 µm)**



- ZFC MO images indicate dendritic flux penetration, perhaps induced by polishing defects.
- MO may show preferential flux penetrations at GB.

Magneto Optical Imaging of I–Sample After VI: No Clear Correlation





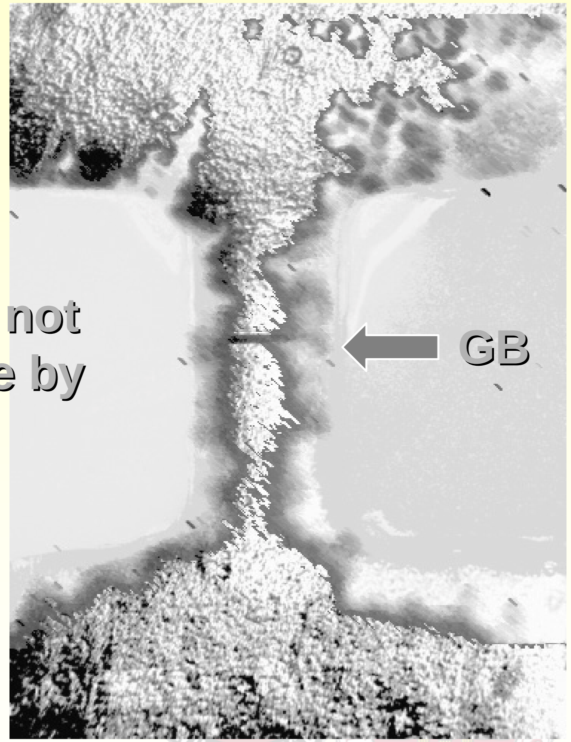
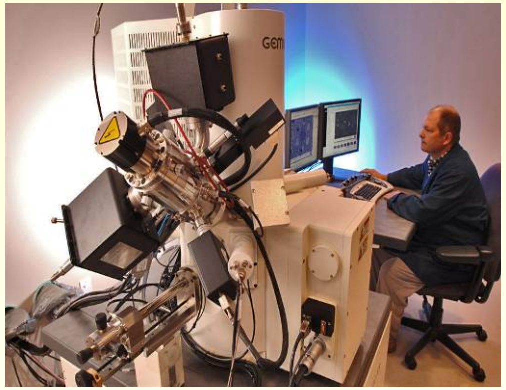


Image of flux penetration superimposed on surface do not show clear GB weakness

Micro structure and chemistry

- Sample preparation
  - BCP ($HF:HNO_3:H_3PO_4$ =1:1:2); ~ 100 µm (100 min)
  - Coating protective layers
    - Au-Pd; ~ 500 nm & Carbon; ~ 700 nm
  - Another protective layer (Pt); ~300 nm
    - Deposited by GIS (Gas Injection System) in FIB
  - TEM lamella formed by Focused Ion Beam



- Micro structure by STEM; Philips 200 UT (FEG)
  - Dr. Jim Bentley (SHaRE program at ORNL)
- Electron Energy Loss Spectroscopy (EELS)
  - Gatan Image Filter (GIF); 1.0 eV resolution & 0.5 eV/channel

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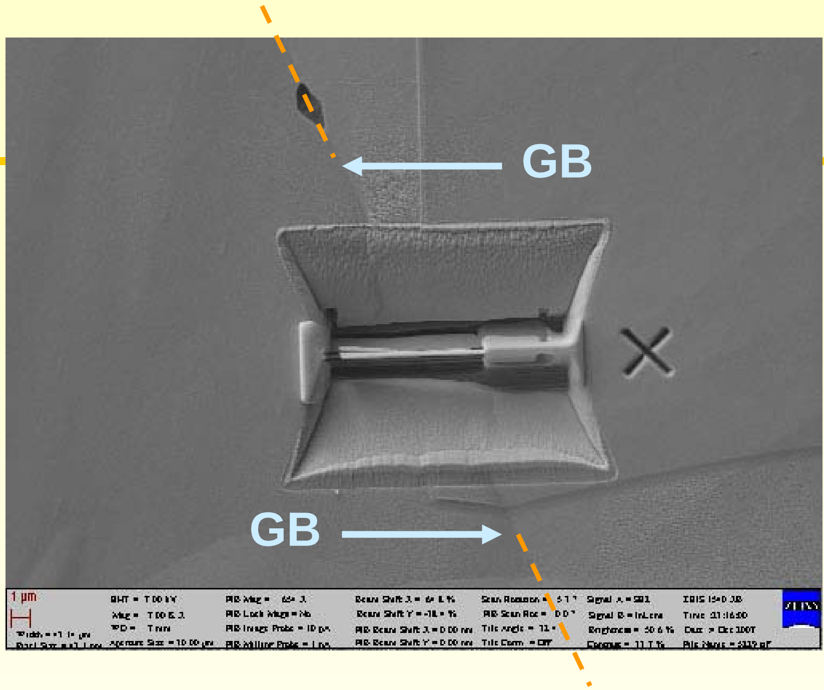
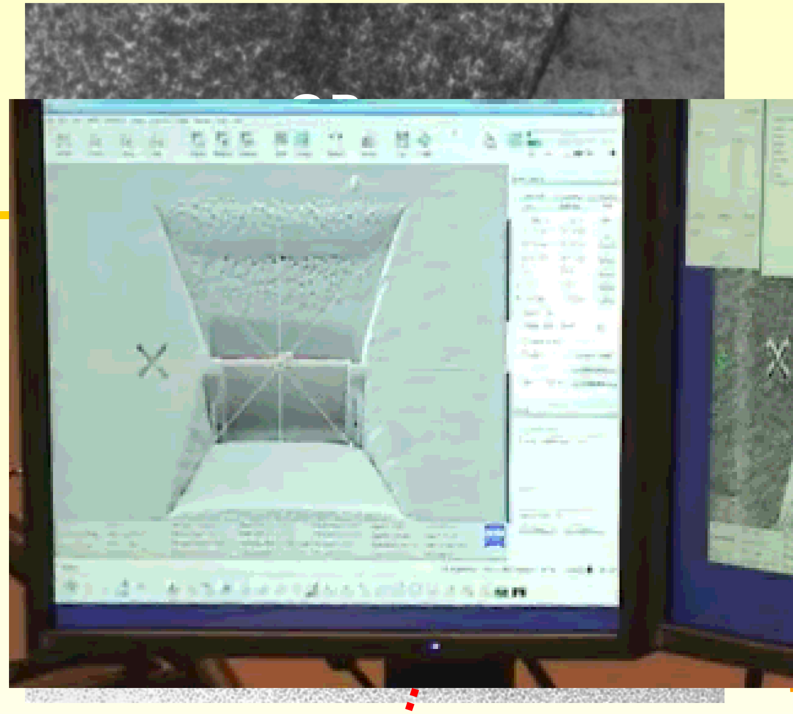
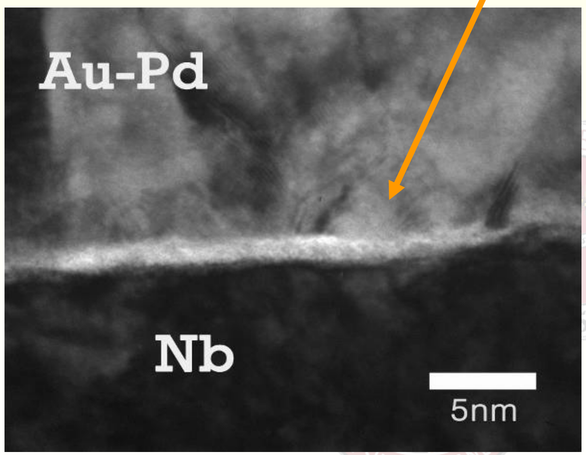




Niobium oxide layer

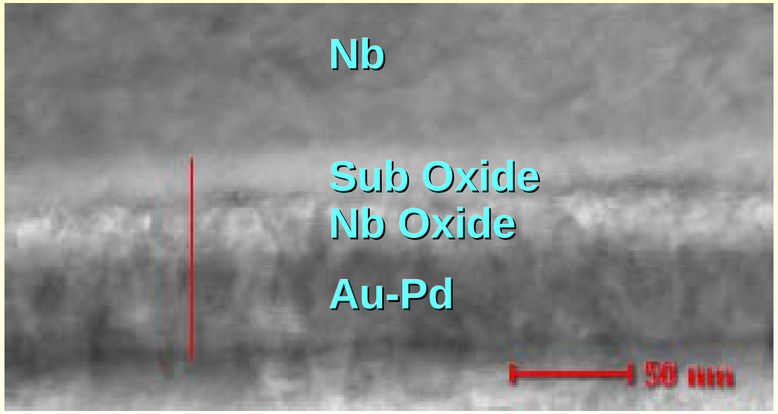


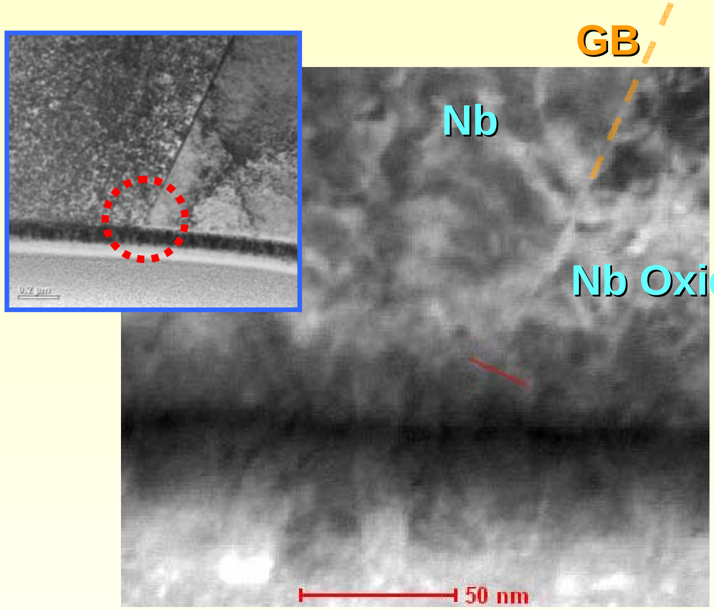
TEM Bright Field Image

- FIB was used to cut TEM foil at GB
- BCP'ed surface protected by Au-Pd, C and Pt layers.
- Variation in niobium oxide thickness observed for the two grains.
  - No evidence of large grain boundary oxide penetration.

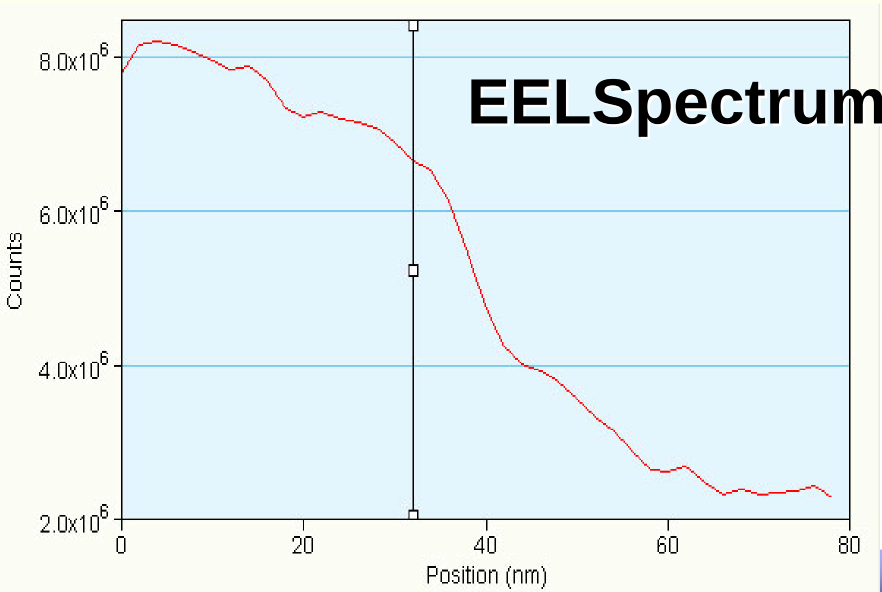
STEM: Niobium Oxide Surface Layer

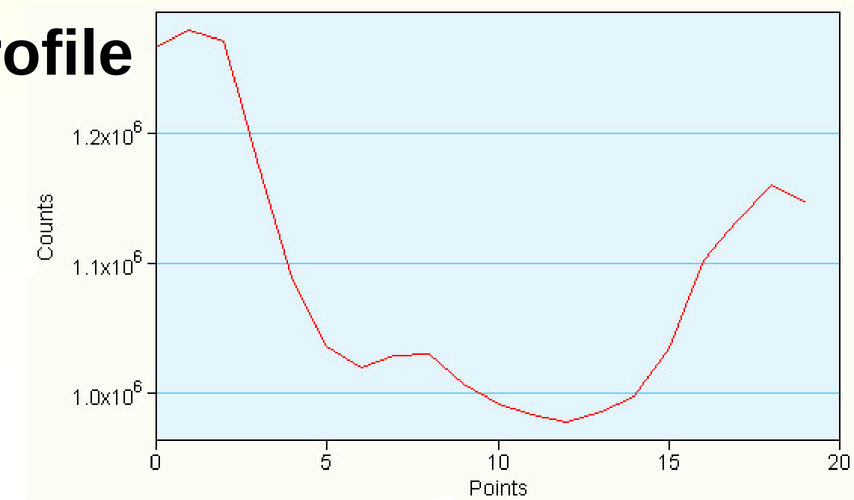
ADF





EELSpectrum Profile





Summary

- Can now perform all tests on one single sample with and without GB
- J_C (H) measurement reveals that $J_{C,\ gb} < J_{\ C,\ grain}$
  - But, suspect to preferential flux flow at the grain boundary
- MO visualization may indicate preferential flux penetration on GB of thin Nb sample
- Micro structural (STEM) and chemical analysis (EELS)
  - FIB (Focused Ion Beam) technique make possible to observe the microstructure of BCP'ed GB.
  - Sub-oxide layer locally exit at the interface between niobium matrix and niobium oxide layer.
  - Variation in niobium oxide thickness observed for the two grains.

Acknowledgments

- We would like to thank Peter Kneisel and Ganapati Rao Myneni and their colleagues at TJNL for providing the Nb slice.
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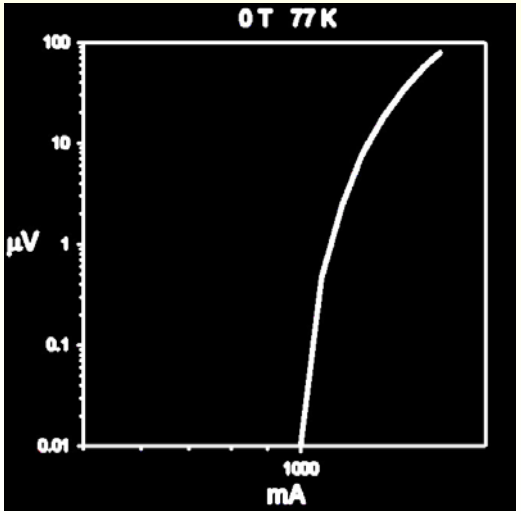
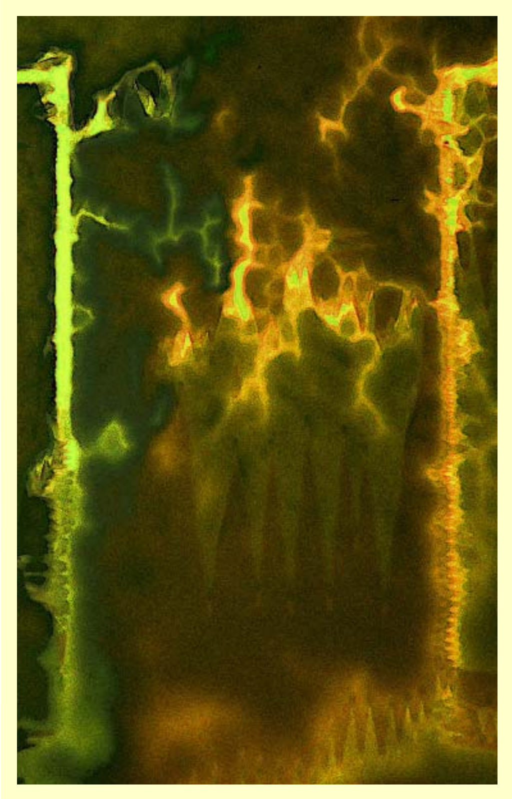
Coupled V-I data and Transport Magneto-Optics on YBCO coated conductor

- Current percolates from before onset of dissipation to well after
- J_C of link limited by only a small number of existing GBs

$I_{APPLIED}$ = 1.5 A

J = 1.5 J_c

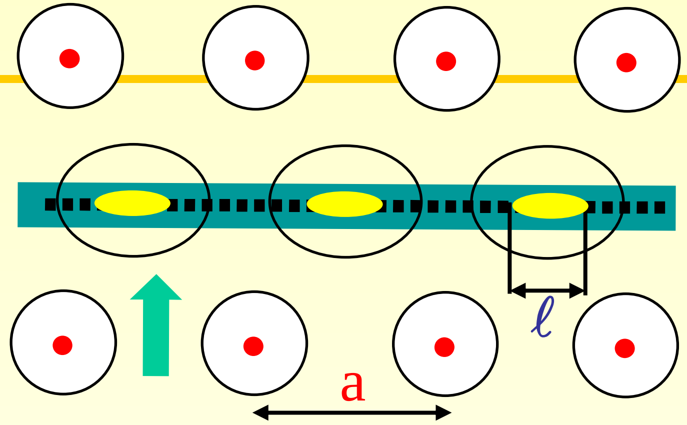


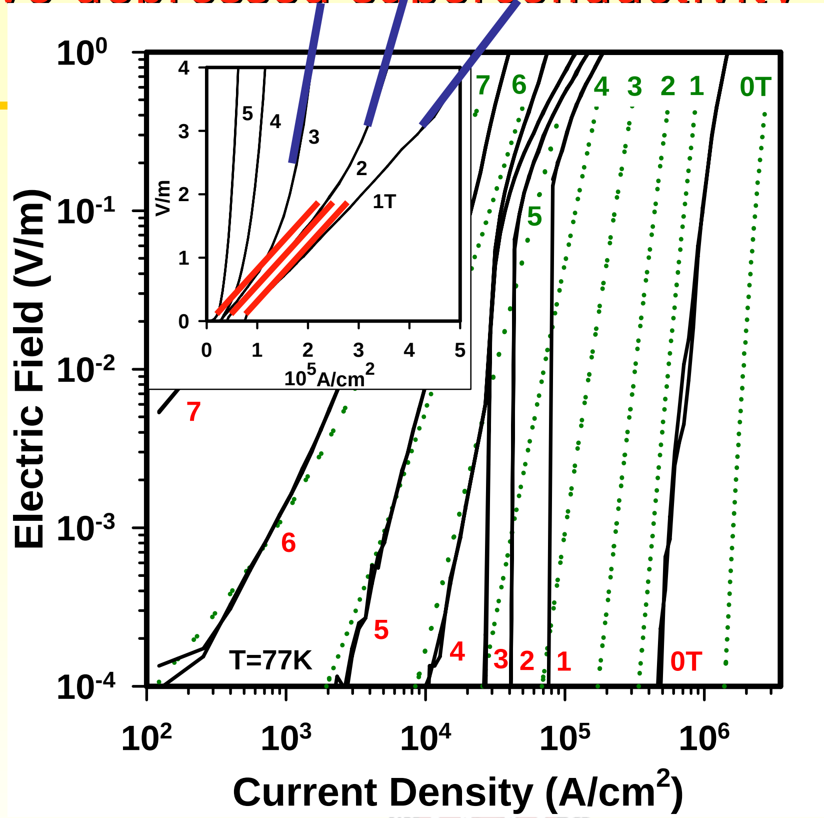


Feldmann et al. APL 2000, 2001

Also LTLSM mapping of E field by Abraimov, Kiss

Dissipation at a straight YBCO GB occurs by slippage of the vortex chain in the GB – HTS GBs easily have depressed superconductivity





$\ell = \lambda_J^2/\lambda = \xi J_d/J_b$

- A-J core size:
- The A-J cores overlap if $\ell > a$, or

(Gurevich, PRB48, 12857 (1993); PRB46, R3187 (1992)):

$$H > (J_b/J_d)^2 H_{c2}$$

- Viscous flux motion $V = (I - I_b)R$
- R(B) is independent of B, if a single vortex chain moves along GB, while $\ell > a$

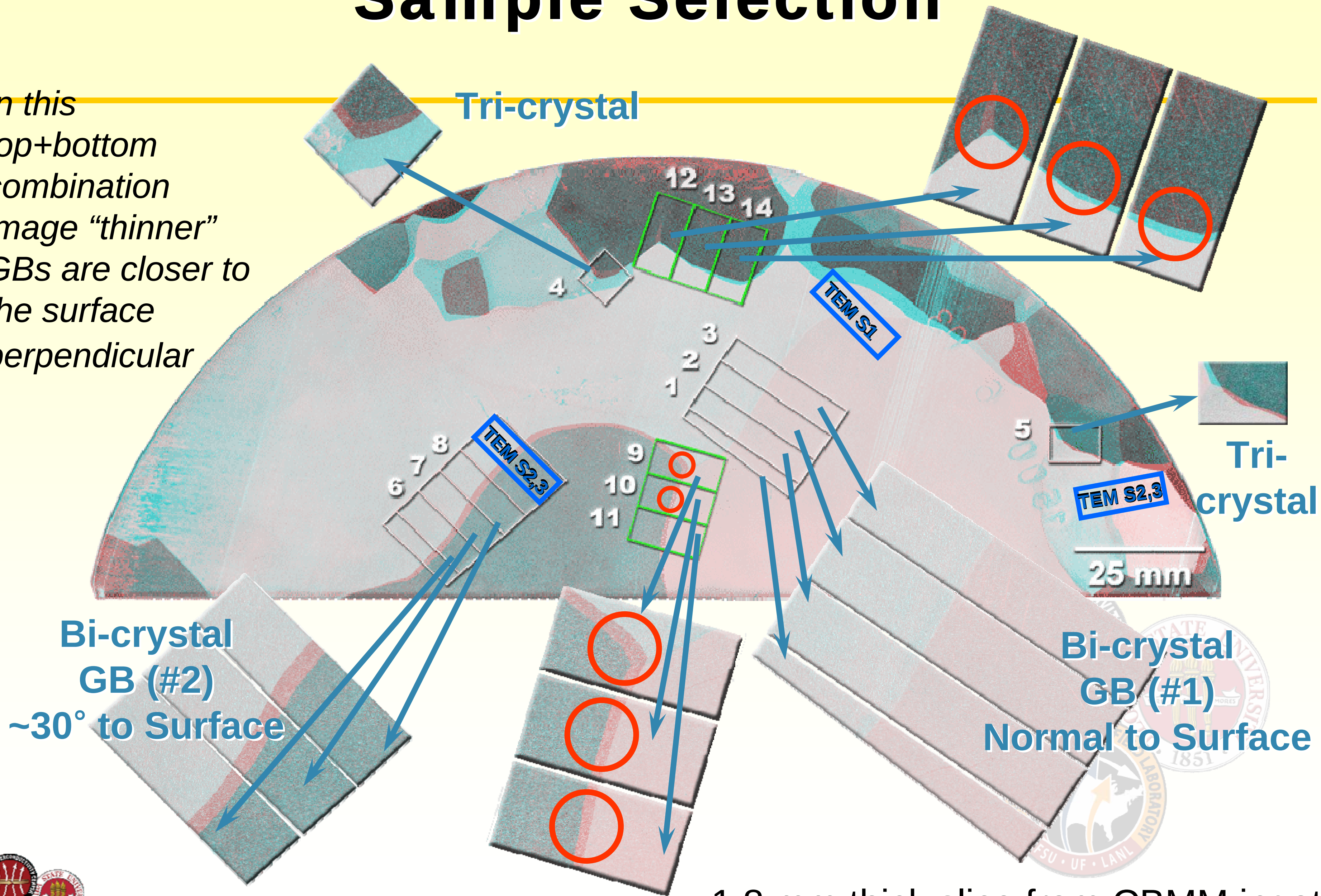
Collective depinning of multiple vortex rows along GB:

$$R(B) = w(B)\rho_n B/B_{c2}$$

Gurevich et al. PRL 88, 097001 (2002)

Sample Selection

In this top+bottom combination image "thinner" GBs are closer to the surface perpendicular



1.8 mm thick slice from CBMM ingot

Scheme of magnet system for Transport Exp.