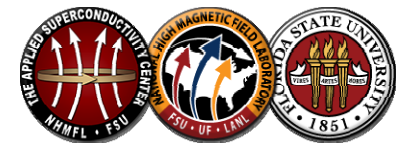


Evidence for suppressed superconductivity across buffer chemically polished grain boundaries of SRF quality niobium

Z.H. Sung, A.A. Polyanskii, P.J. Lee, A. Gurevich, and
D.C. Larbalestier

Applied Superconductivity Center
National High Magnetic Field Laboratory
Florida State University



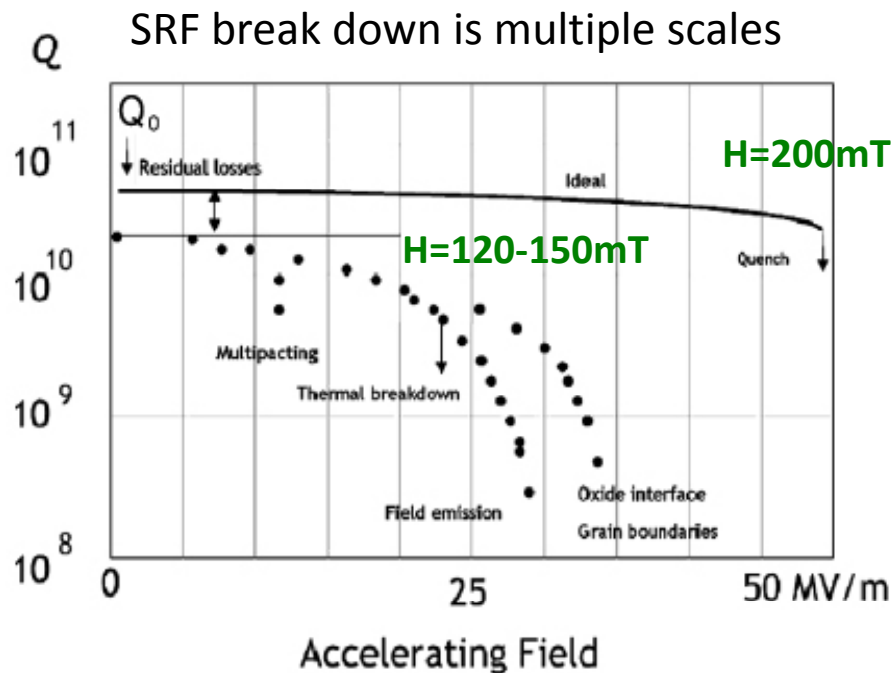
Outline

- **Issues – Why do we need to investigate the GB effects on SRF Nb?**
- **Localized premature flux penetration at the GB**
- **Microstructure at the vicinity of the GB**
- **Field dependent flux flow resistivity of the GB and its angular dependence.**
- **Summary**
- **Current investigation using micro Hall probe**



Why do we investigate the GBs effects on SRF Nb cavity?

GBs may be defects responsible for the degradation of the SRF cavity performance



Courtesy of H. Padamsee

Figure of merit - Quality factor (Q_0)

$$Q_0 = \frac{wU}{P_{diss}} = \frac{G}{R_s}$$

$$R_s = R_{BCS}(T, H) + R_0$$

$$R_{BCS}(T, H) = A \frac{f^2}{T} \exp\left(\frac{-\Delta(0)}{K_B T}\right)$$

GBs can locally reduce superconducting gap (Δ) and the depairing current density, $J_b \rightarrow$ suppress the onset of vortex penetration \rightarrow increase $R_s \rightarrow$ increase power dissipation



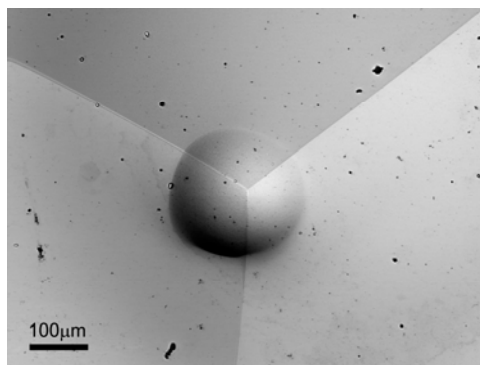


Courtesy of W. Singer. ILC Cavity Group 9th Meeting. January 27th. 2009

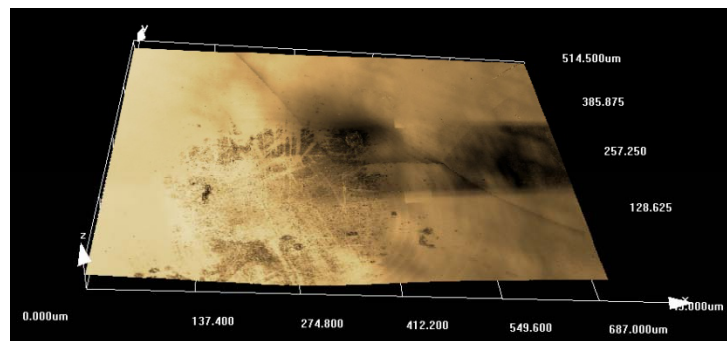
PIT on Cold spot



PIT-BSD image



PIT : 3D topology

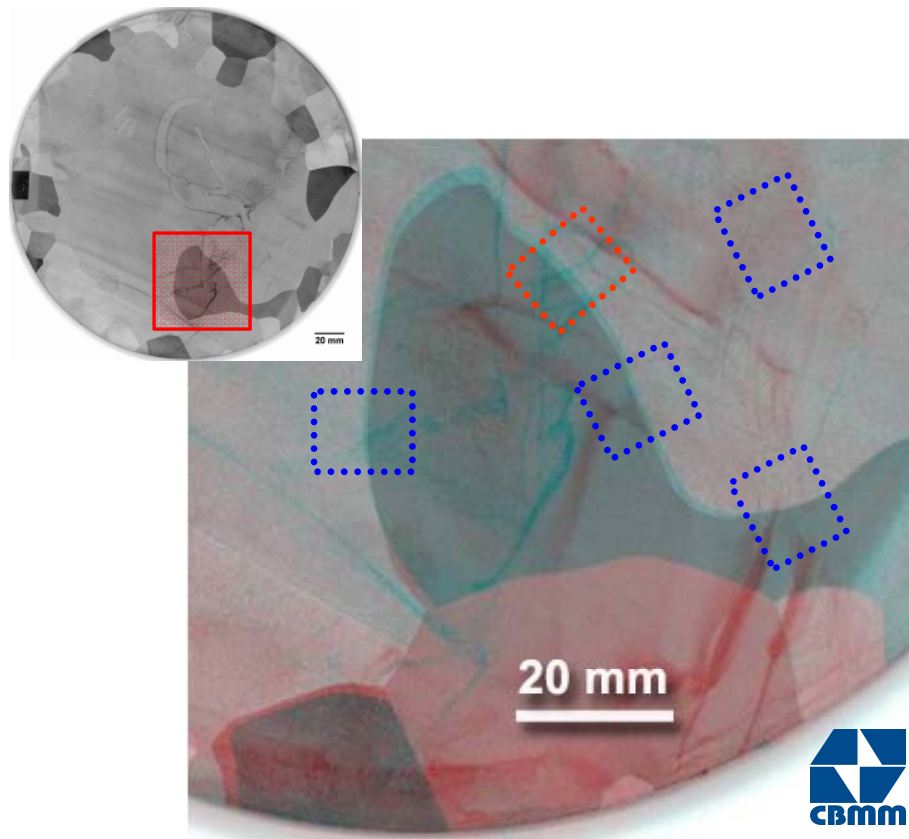


“Cold” spot in the high field Q-slope limited (large grain BCP’ed) cavity
 - provided by A. Romaneko @ FNAL)



Description of specific grains and grain boundaries

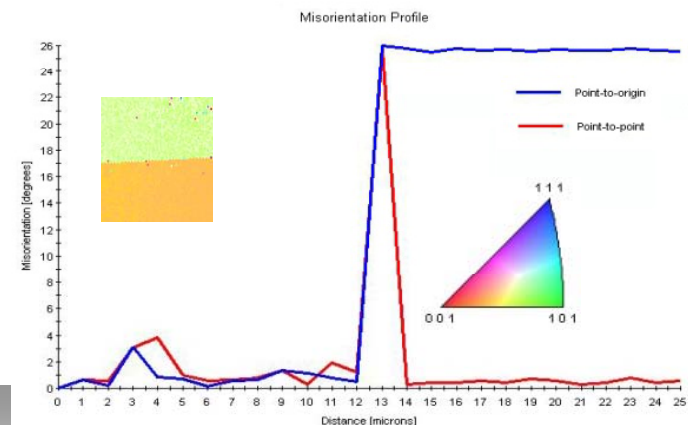
Large grain Nb sheet from JLAB (P. Kneisel and Co-workers)



Overview of the as-received niobium slice

- “**Thinner**” GBs have planes which are closer to the surface perpendicular. “**Thicker**” GBs are inclined $\sim 20\text{-}30^\circ$ from the perpendicular.
- This as-received slice (RRR ~ 280 , 3.1mm thick) has a very large grain size ($\sim 50\text{-}100\text{mm}$), which allowed us to isolate multiple bi-crystals.

Grain misorientation $\sim 25.7^\circ$

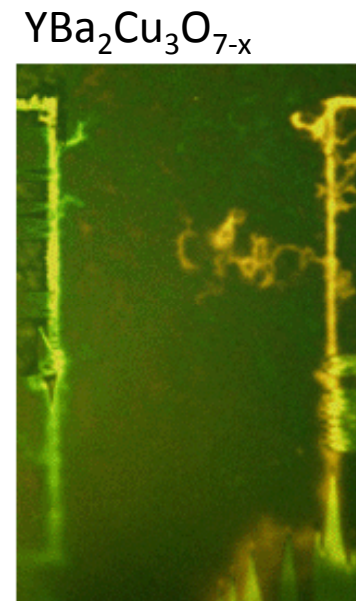
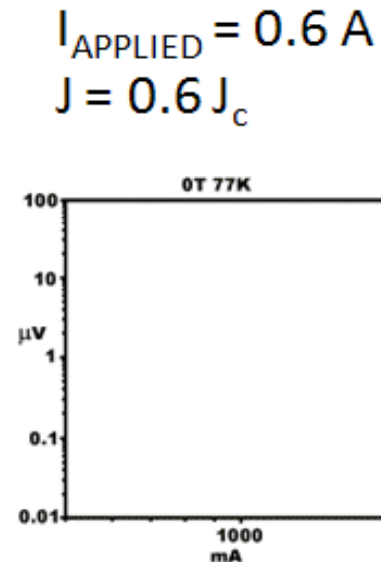


Localized and premature flux penetration at the GB

Magneto-Optical Imaging & Surface topology by 3D-SLCM (Scanning Laser confocal microscope)

– *MO imaging by Dr. A.A. Polyanskii (ASC-NHMFL)*

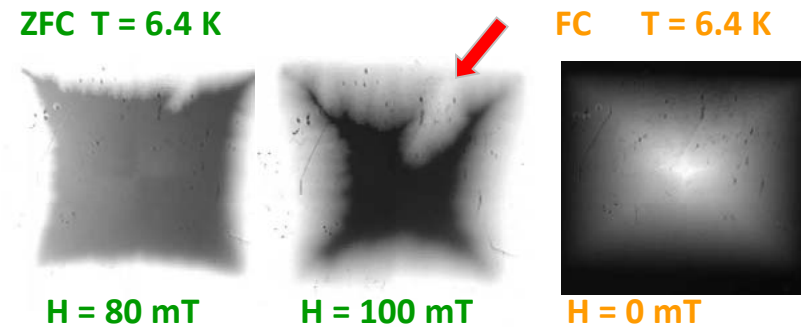
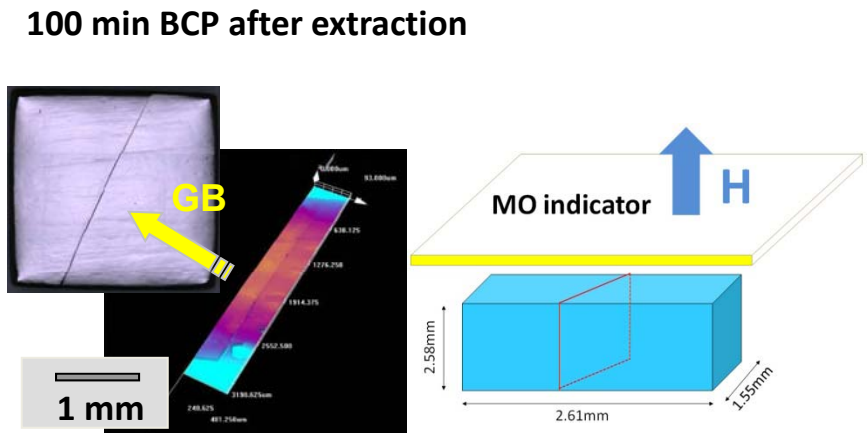
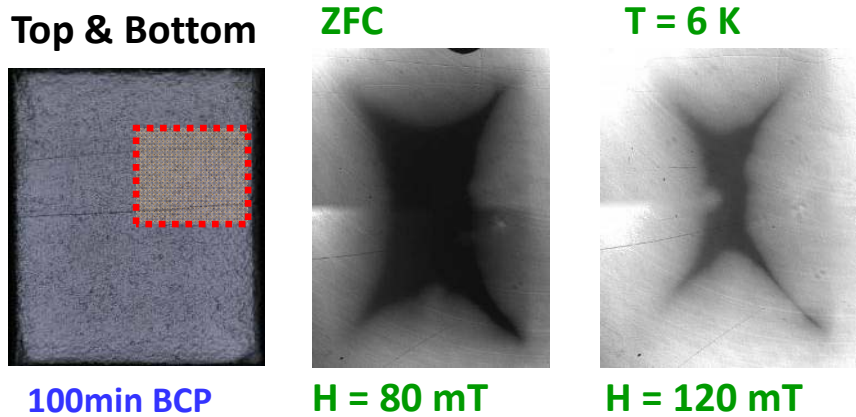
- Current percolates from before onset of dissipation to well after
- J_c of link limited by only a small number of existing GBs



Flux penetration and its correlation to direction of H_{ext}

Lack of flux penetration at tilted GB

Asymmetric preferential flux penetration ($H_{ext} // GB$)



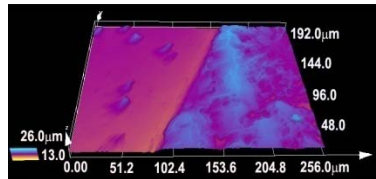
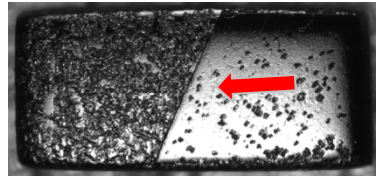
Flux did not preferentially penetrate along the GB, in spite of the large mis-orientation of the H_{ext} vector and the bulk pinning of magnetic flux is symmetric and only the flux penetration is asymmetric.

Do BCP and EP have different effects on flux penetration?

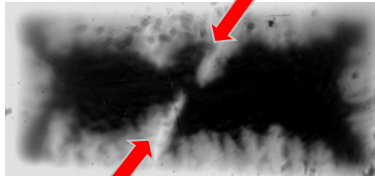
Both: GB// H_{ext}

Reduced thickness, and then compared with the EP'ed bi-crystal cut from same GB

BCP'ed

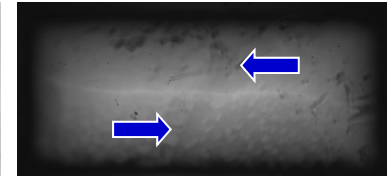


ZFC T = 6 K

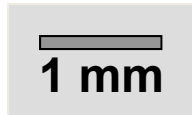


H = 58 mT

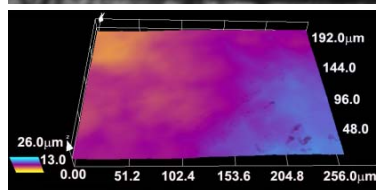
FC T = 6 K



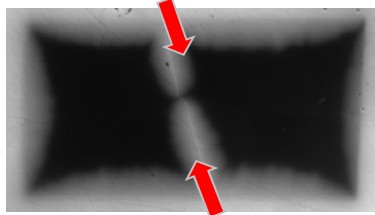
H = 0 mT



EP'ed

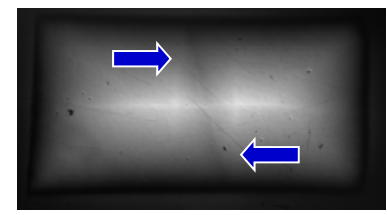


ZFC T = 6.5 K



H = 72 mT

FC T = 6.5 K



H = 0 mT

GB is a weak link only when H_{ext} is aligned parallel with the GB plane
The GB groove may not be the cause of the preferential flux penetration



Microstructure at the vicinity of GB

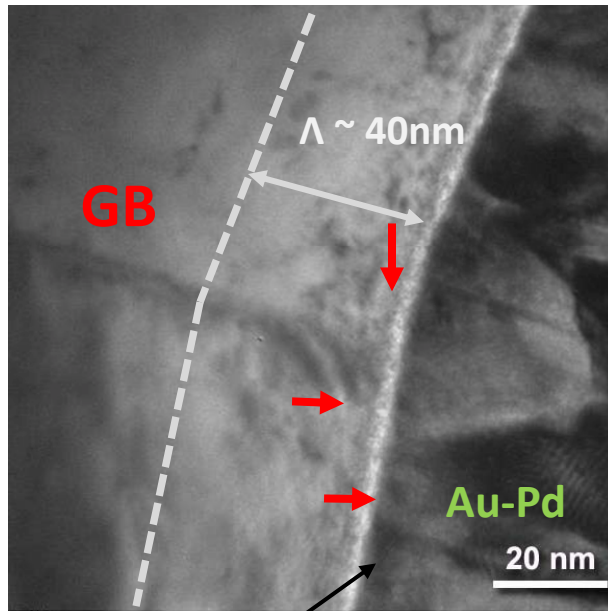
Transmission Electron microscopy using *state-of-art* FIB technique and conventional sample prep. (by BCP)



What is the real microstructure at the GB after BCP?

HRTEM used to observe the vicinity of the GB

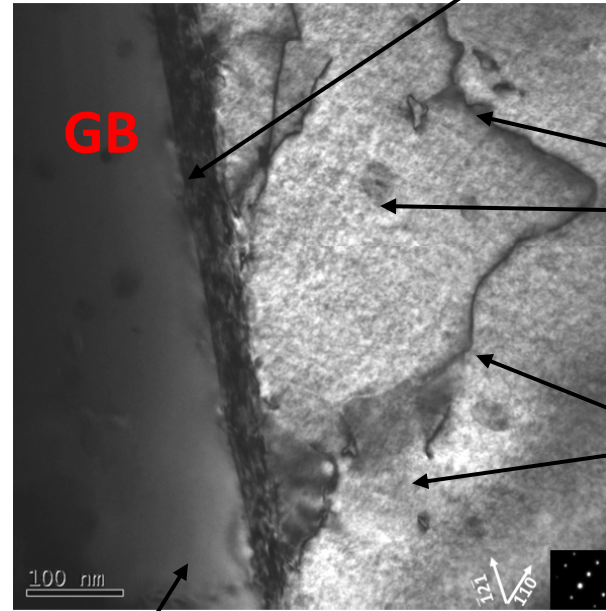
Cross sectional view of GB



Prepared by FIB

Nb oxides layers

Plane-view of GB



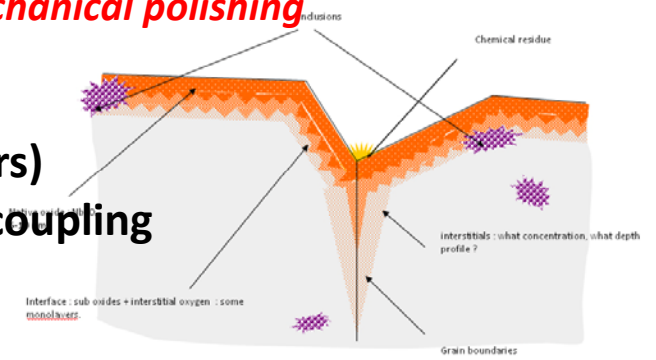
Prepared by BCP & mechanical polishing

Vs

Dark contrast due to high mis-orientation

Coherence length of Nb ($\approx 40\text{nm}$) \gg HTS (\approx few nanometers)

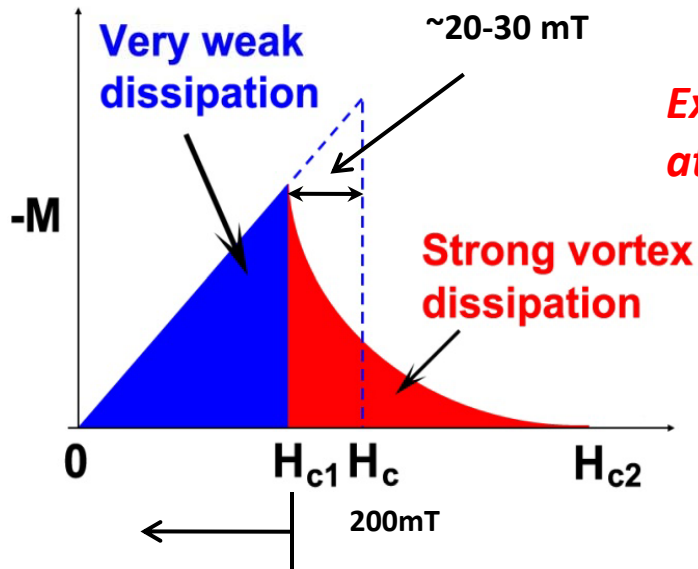
Dislocation pile-ups at GB suggests pinning rather than decoupling



Field dependent flux flow resistivity of the GB and its angular dependence.

The depairing critical current density & suppression of
superconducting order parameter at the GB: DC transport
 $V-I$ characterization with 1T Electro-magnet



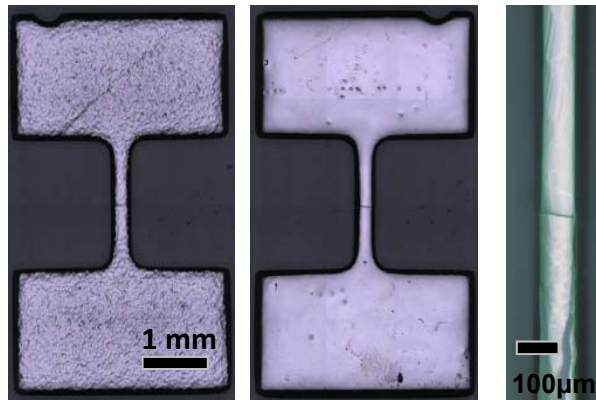


Expand the gap between H_{c1} (170mT) and H_{c2} (200mT) at 4.2 K \rightarrow Make vortex penetration at lower H_{ext}

The procedures

1. Cut samples into I-shape with wire-EDM
2. Mechanically grind down the bottom of the sample surface to $\sim 150\text{-}250\mu\text{m}$, so the top surface remain as-received condition
3. Ultra fine polish with vibratory polisher (Vibromet[®] Buehler)
4. Finalize all surfaces with either BCP or EP
 - Make surfaces representative of real cavity surface
5. Further reduce the bridges of some I-shape single- & bi- crystals with extra BCP
6. Artificially groove with FIB and mechanically smear away the grooved produced by the chemical treatments

As-received Mechanically ground



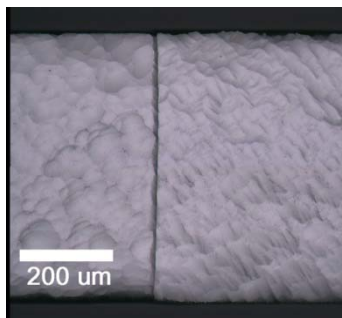
Surface image of I-shape sample after BCP treatment



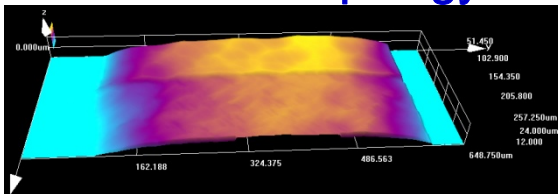
Surface topology of the variously treated samples

BCP'ed Bi xtal

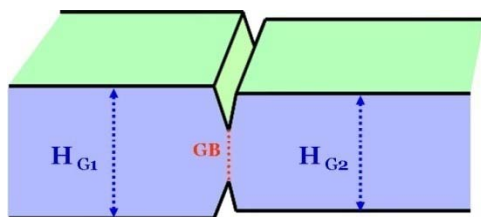
Deep & sharp groove effect



3D surface topology



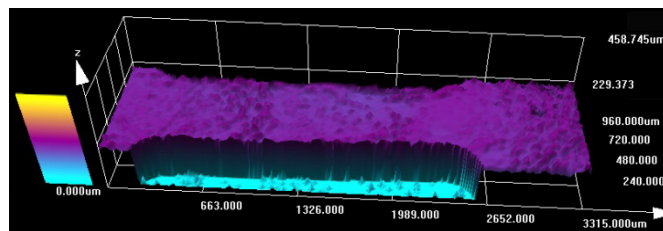
~ 5 to 8 μm depth groove



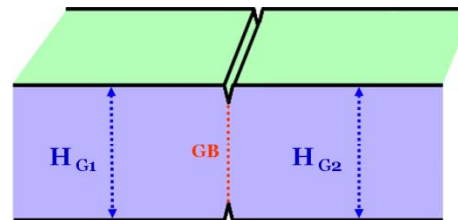
$$H_{G1} > H_{G2}$$

EP'ed Bi xtal

Uniform surface & less groove effect



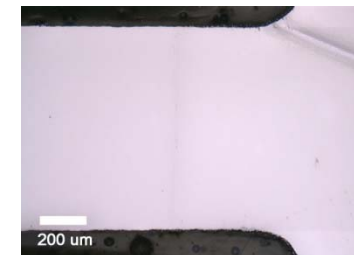
~ 0.3 to 0.5 μm depth groove



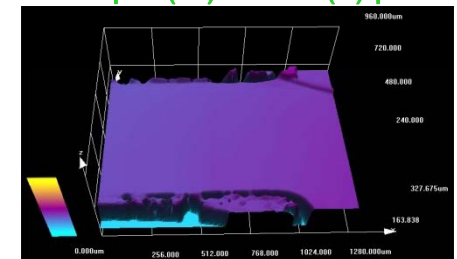
$$H_{G1} \approx H_{G2}$$

Flattened Bi xtal

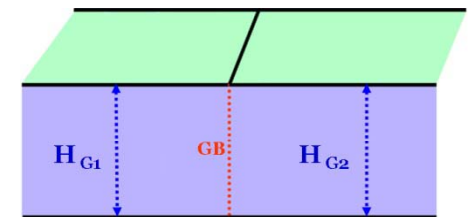
No groove effect



742 μm (W) × 46.5 (T) μm



Flat surface

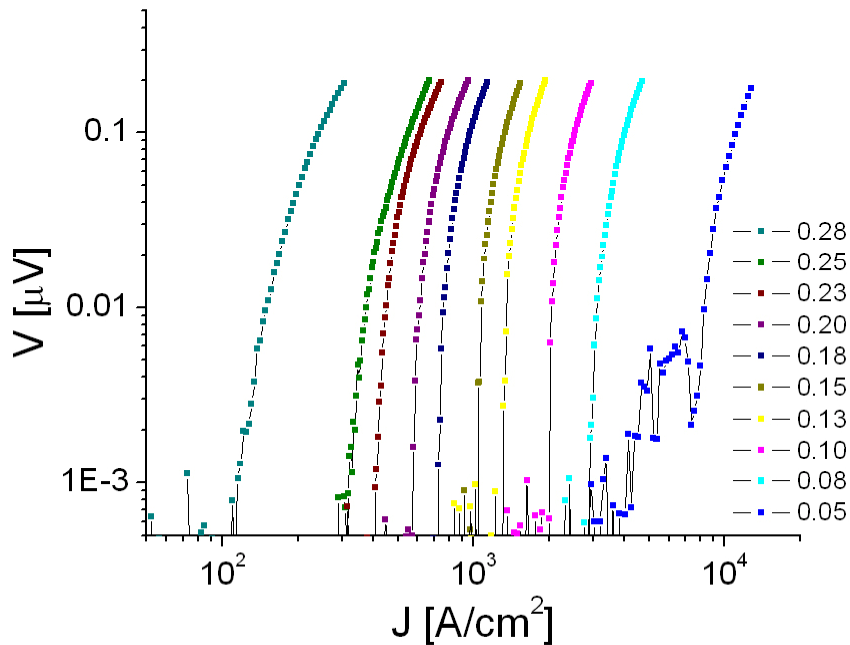


$$H_{G1} = H_{G2}$$

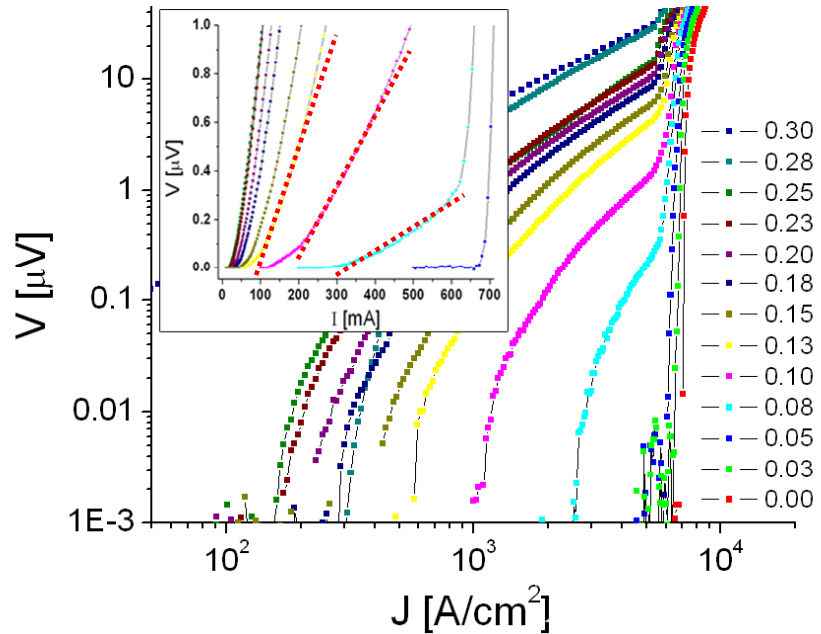


Preferential flux flow on the deep GB groove

No groove ($\sim 0.5\text{--}2.0\mu\text{m}$ roughness)



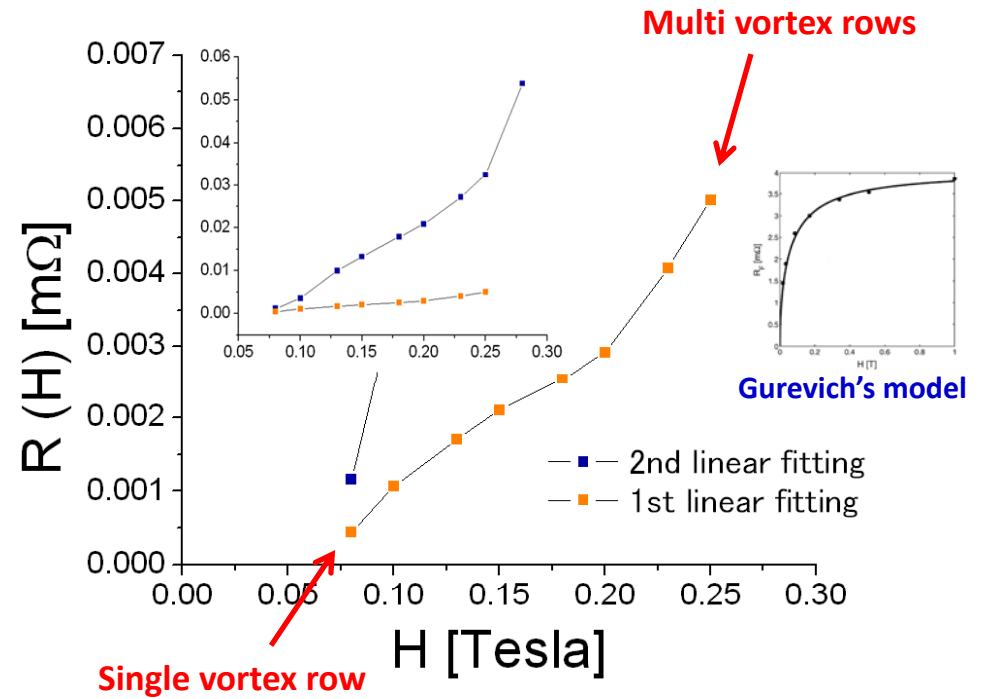
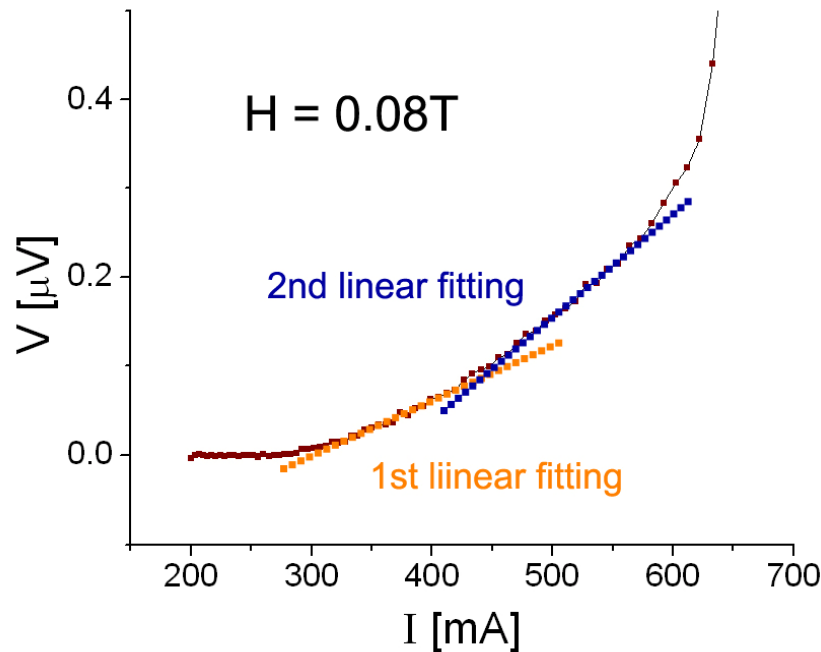
A deep ($3\text{--}5\mu\text{m}$) and highly inclined groove



• Flux flow evidence from $H = 0.08\text{ T}$ to 0.28 T

- The V - J characteristics show that the grain boundary is a channel of preferential flux flow (FF) by weakly pinned vortices.
- However, the slightly non-ohmic V - I response suggests that flux flow is not just confined to a single vortex row flowing along the grain boundary

Field dependent GB resistivity : $R_f(H)$



Collective depinning of multiple vortex rows along GB

$$R_f(H) = \frac{\rho_n \times W(H, J) \times H}{H_{c2} \times A}$$

$$dV_{row} = \frac{W}{a} = W \sqrt{\frac{B}{\Phi_0}}$$

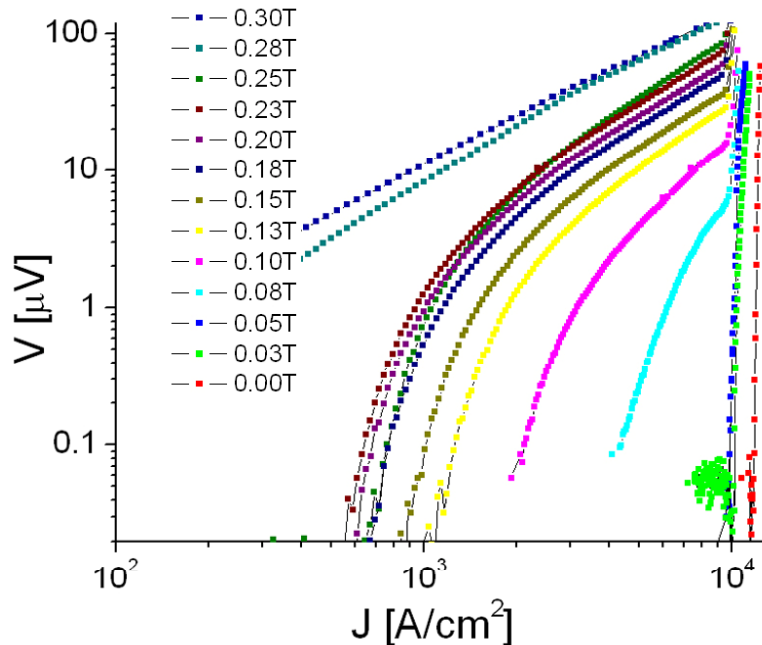
The width of the FF channel (W) & The number of vortices flowing on the channel (dV_{row}) at $H = 0.08\text{T}$ are $0.185 \mu\text{m}$ and $0.489 \mu\text{m}$, representing 1.15 and 3.04 rows, respectively from the low and high V portions of the V - I curve



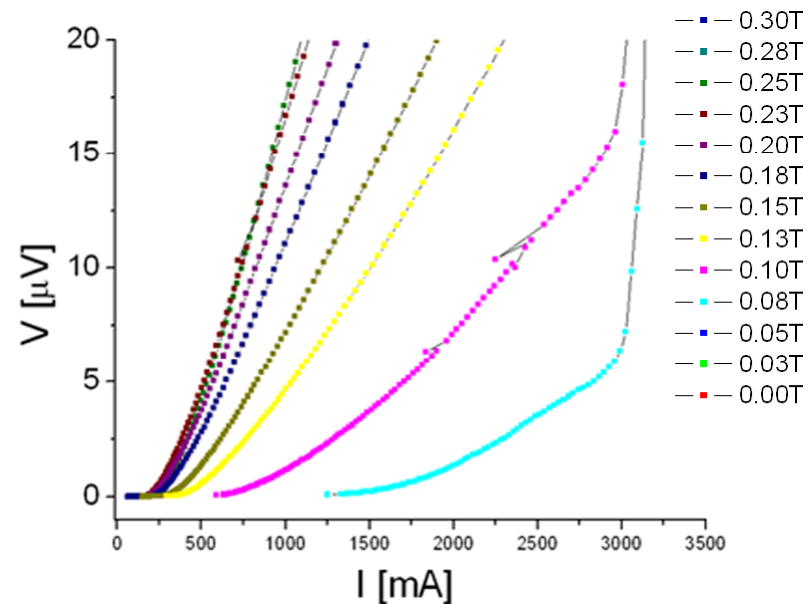
Preferential GB flux flow is not associated with the GB groove – still present after smooth mechanical polishing

Very flat surface by ultra-fine mechanical polishing

V-J response [log-log]



V-J response [Linear]

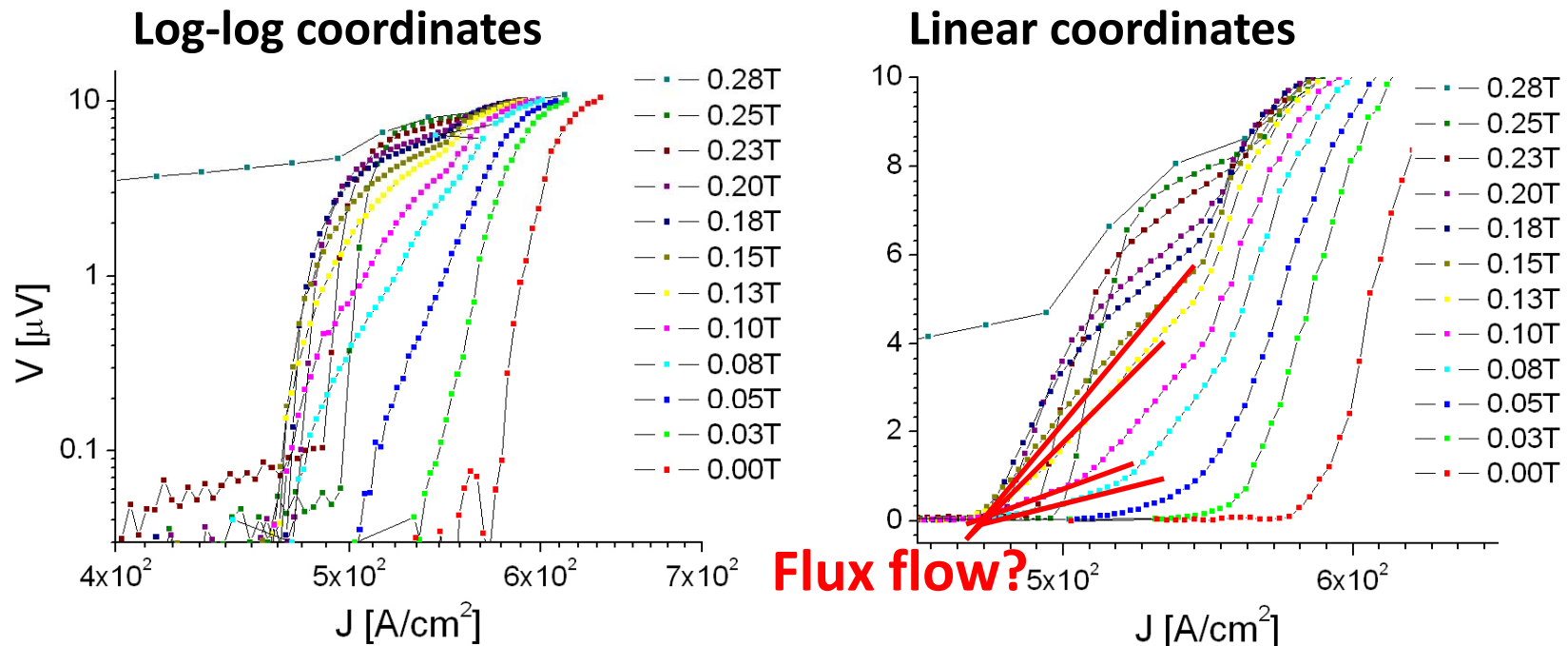


- Linear GB flux flow of vortices starts at $H_{\text{ext}} \approx 0.08\text{T}$
- Very similar behavior to BCP'ed bi-crystal
- At $H_{\text{ext}} = 0.08\text{T}$, the # of vortices rows flowing at the GB are ~ 4.90 , then the # rapidly increases as applied current increased (finally ~ 33.51) – Multi flux flow rows



Little evidence for GB flux flow for EP bi-crystal

Shallow ($\sim 0.5\text{--}2.0\mu\text{m}$) groove at the electropolished GB

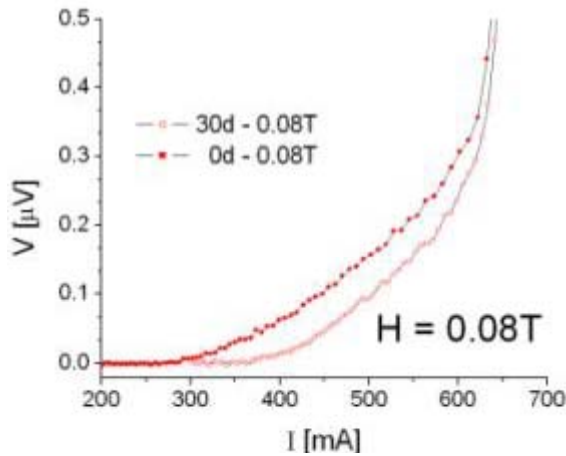


- However, traces of flux flow along the GB are visible

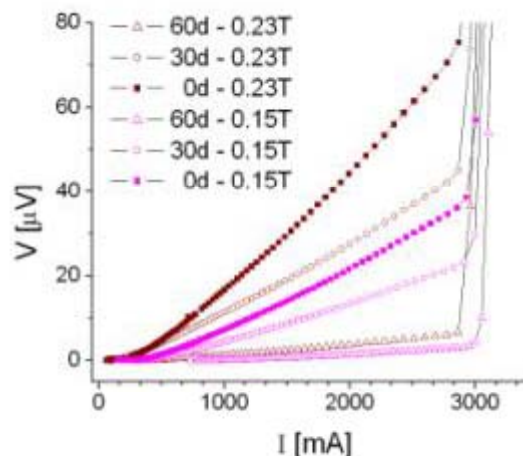
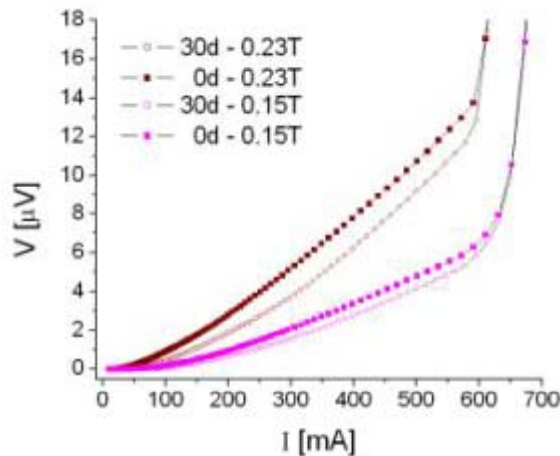
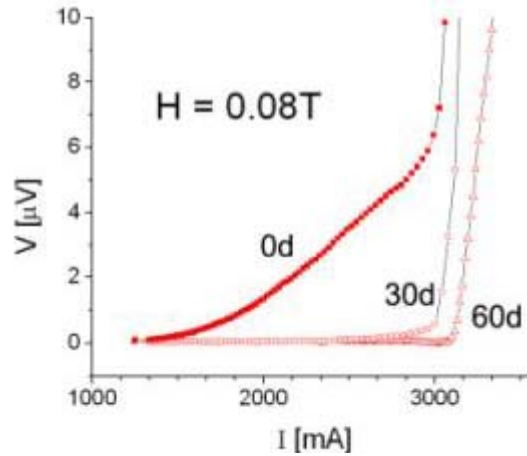


Dissipation is enhanced when the angle between H_{ext} and GB is minimized

**BCP'ed Bi crystal
(a deep groove)**



Mechanically flattened Bi crystal (No groove)

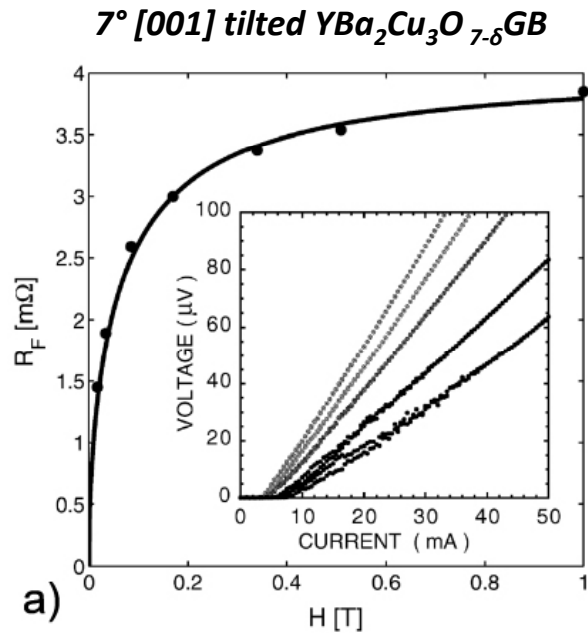


The number of degrees indicate the angle between the plane of GB and H_{ext}

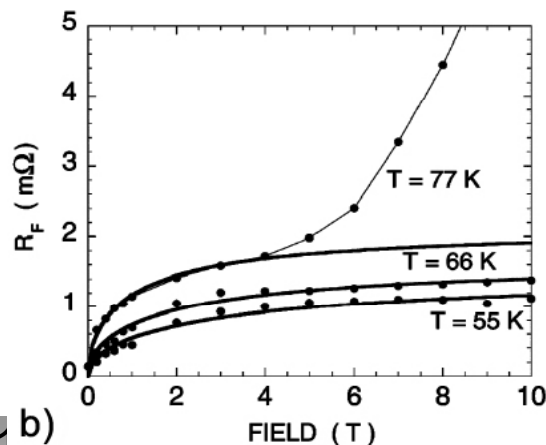
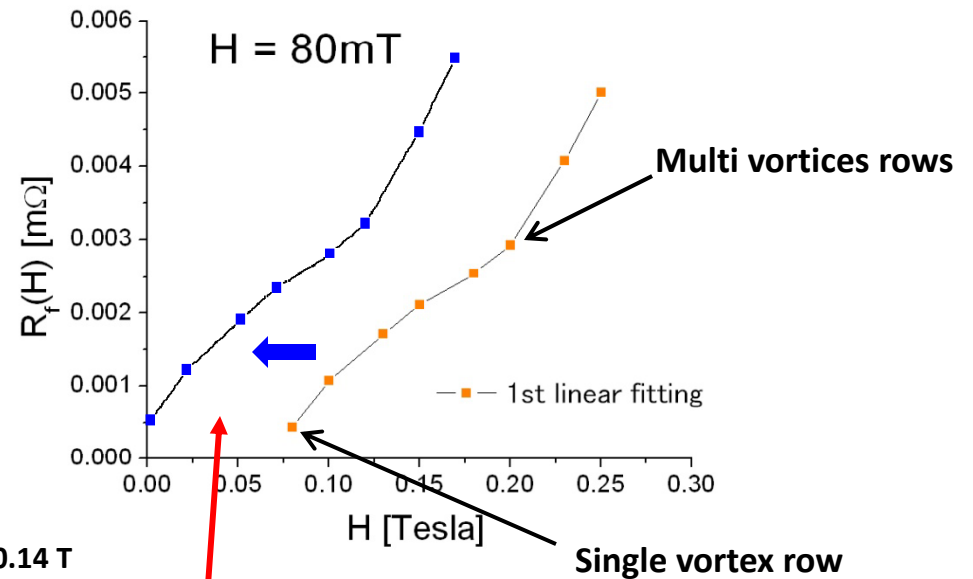
- The GB flux flow dissipation decreases as the angle increases
- GB flux flow is more prominent on non-grooved bi-crystal, compared to BCP'ed bi-crystal with prominent GB groove.



The proposed work: the depairing current density at GB



BCP'ed Nb Bi-crystal (26° misoriented GB)



$R = 4.05\text{ m}\Omega$ and $H_0 = 0.14\text{ T}$

$$R_f(H) = \frac{R\sqrt{H}}{\sqrt{H + H_0}}$$

$$J_b = \frac{c\sqrt{\phi_0 H_0}}{8\pi\lambda^2}$$

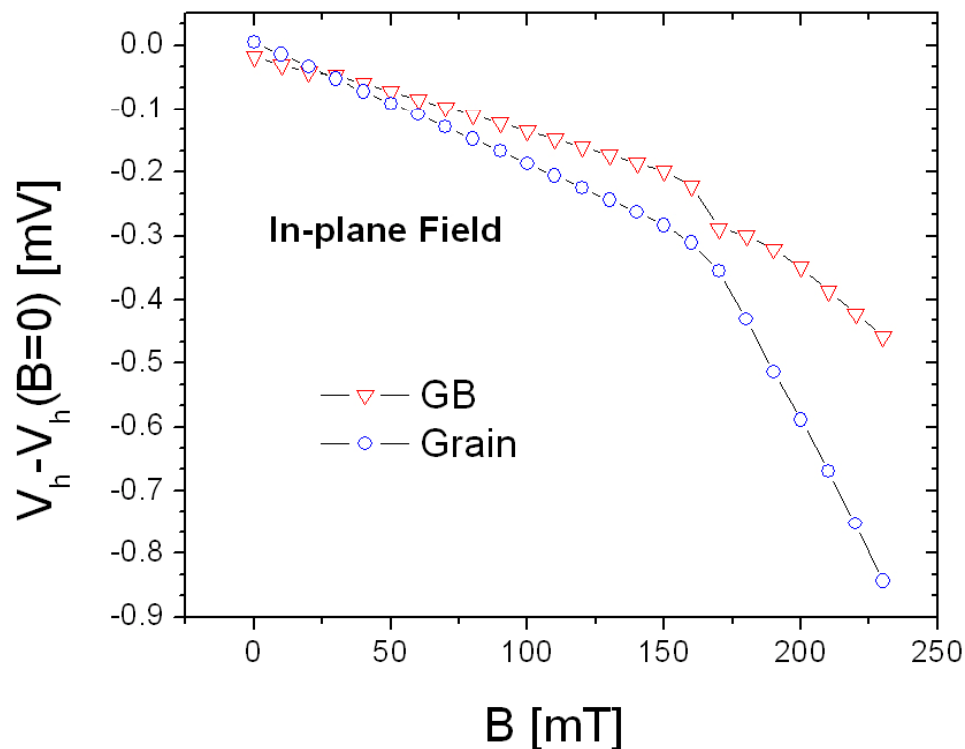
$$B = H - \eta \cdot M$$

η = demagnetization factor
 M = magnetization

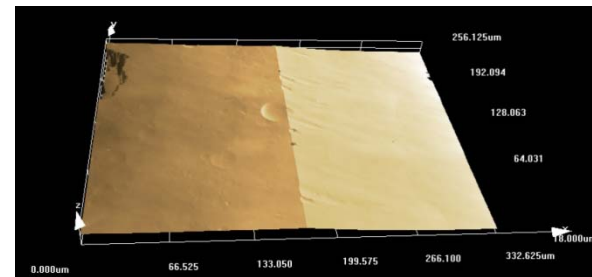


Measurement of field enhancement at GB using micro Hall probe (50 μm by 50 μm)

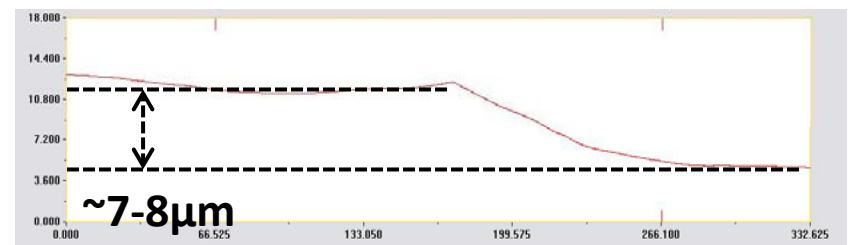
Response voltage on Hall probe



Surface Topology at the GB (LSCM)



Roughness profile at the GB



Summary

- MO imaging showed that GBs can preferentially admit magnetic flux before it is admitted to grains when GB is aligned close to \mathbf{H}_{ext} .
- Combining MO with LSCM, we were able to show that topological features introduced by BCP or EP were not the cause of the preferential nucleation of magnetic flux.
- TEM showed 5-7nm thick niobium-oxide layers with no evident GB penetration, in strong contrast to Halbritter's model.
- Transport measurements on BCP'ed samples showed clear evidence of preferential GB flux flow enhanced when plane of GB is parallel to the \mathbf{H}_{ext} vector.
- Direct correlation of local \mathbf{H} at GB shows that flux flow corresponds to GB flux entry



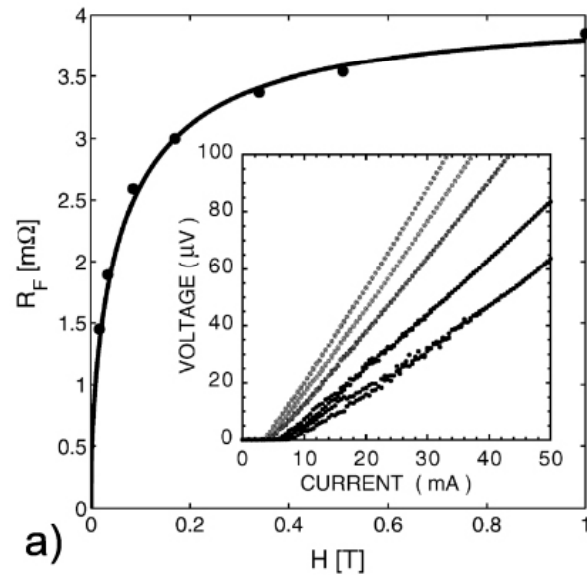
Acknowledgement

- We would like to thank Peter Kneisel and Ganapati Rao Myneni and their colleagues at TJNL for providing the Nb slice.
- Pierre Bauer, formerly at FNAL, now at ITER, for his support and valuable discussions in the early stages of this work.
- Lance Cooley, SRF Materials Group Leader, at FNAL.
- Special thanks to Ian Winger (Physic department, FSU) for wire-EDM
- This work was supported by the US-DOE under grants DE-FG02-05ER41392 and DE-FG02-07ER41451 and by the State of Florida support for the National High Magnetic Field Laboratory

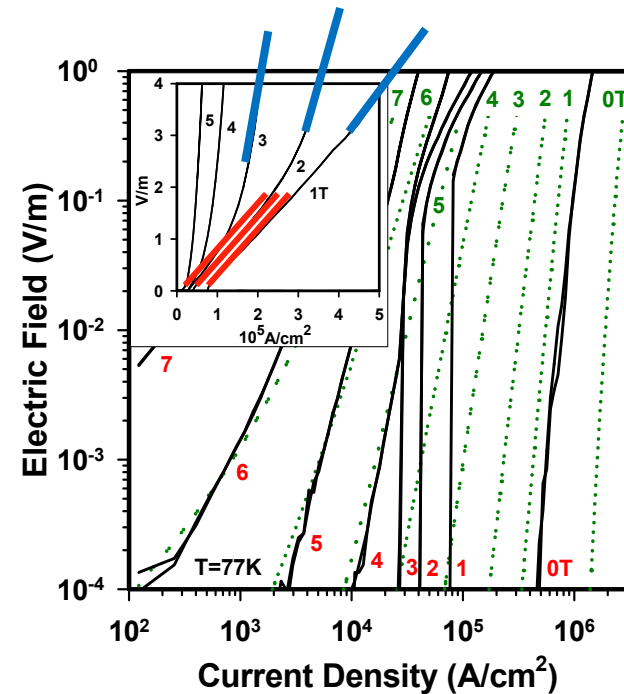


Field dependent flux flow resistivity of the GB

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



Signature of AJ vortices at 7° [001] tilted $YBa_2Cu_3O_{7-\delta}$ GB



- A-J core size: $\ell = \lambda_j^2 / \lambda = \xi J_d / J_b$
- The A-J cores overlap if $\ell > a$, or
(Gurevich, PRB48, 12857 (1993); PRB46, R3187 (1992)):

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

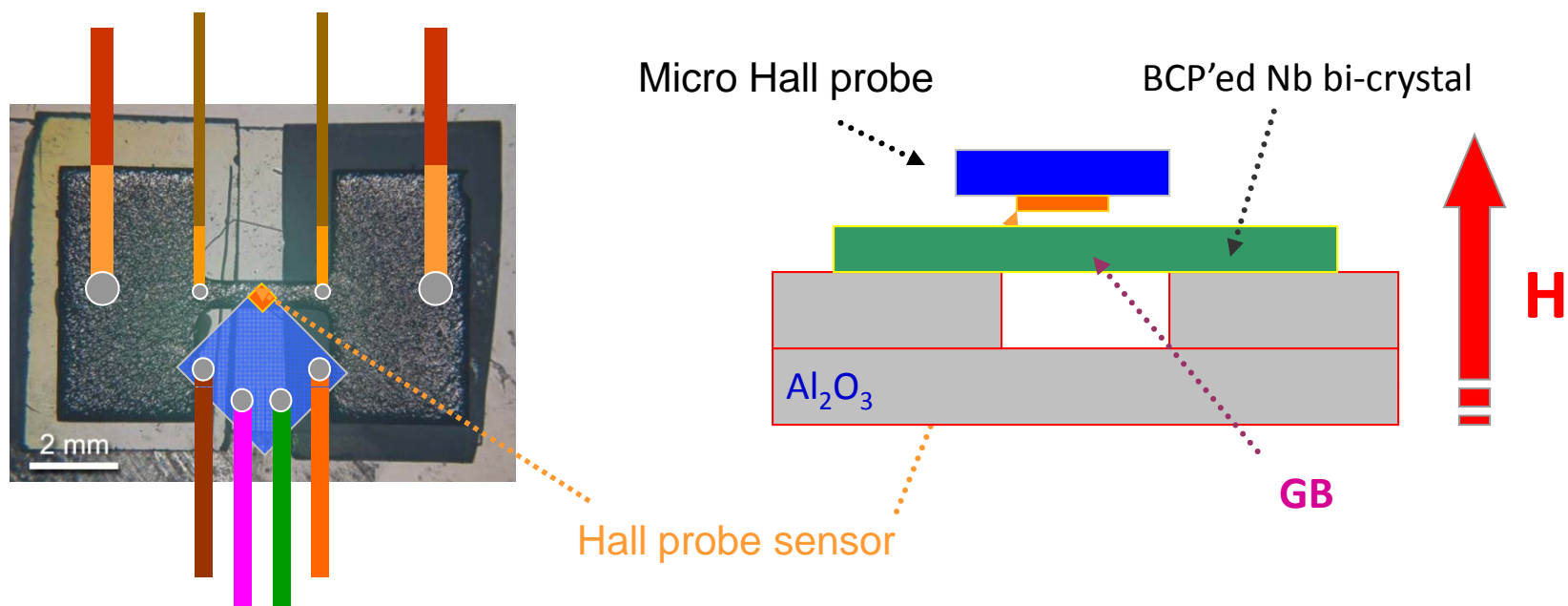
- Viscous flux motion $V = (I - I_b)R$
- $R(B)$ is independent of B , if a single vortex chain moves along GB, while $\ell > a$

$$R_f(H) = \frac{R\sqrt{H}}{\sqrt{H + H_0}} \quad J_b = \frac{c\sqrt{\phi_0 H_0}}{8\pi\lambda^2}$$

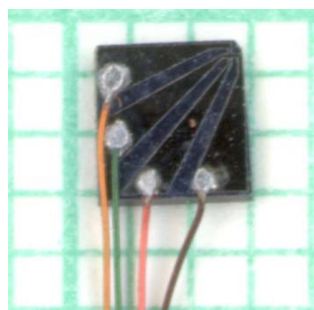
Gurevich et al. PRL 88, 097001 (2002)



Obtaining both B & H on the bi-crystal using a Hall sensor

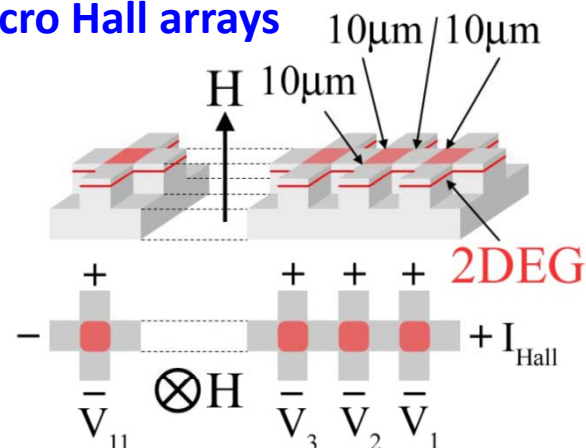


Micro Hall probe



- Spec: ~ 2mm by 2mm
- Total thickness: ~0.4mm
 - 0.4mm thick GaAs substrate
 - 5 μ m thin InSb layer
 - Activation area: 50 μ m by 50 μ m
 - Minimum distance between the sample surface and the Hall probe is ~0.2mm

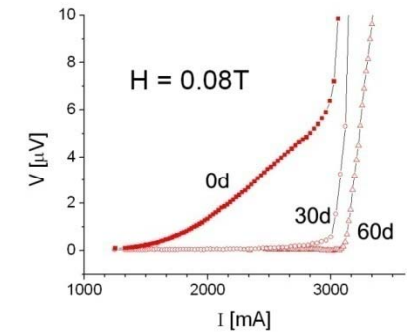
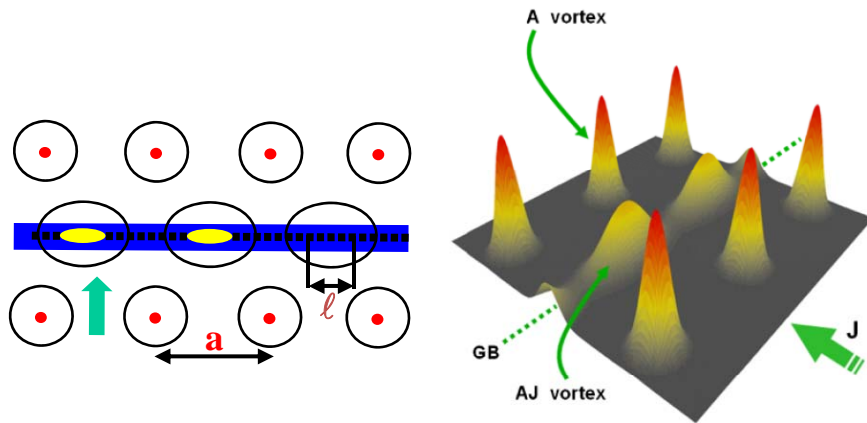
Micro Hall arrays



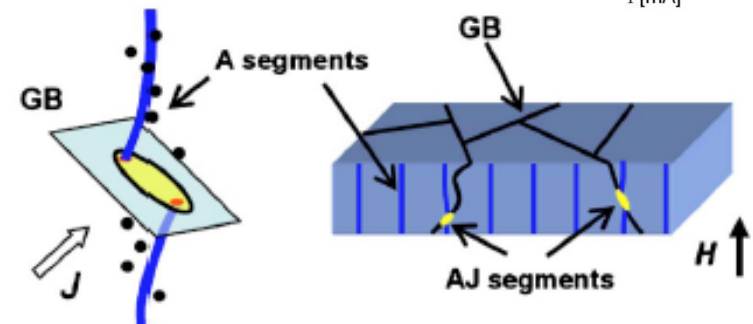
GaAs/AlGaAs heterostructure *Courtesy of Dr. Eli Zeldov*
 The Applied Superconductivity Center
 The National High Magnetic Field Laboratory
 Florida State University



Mechanisms of suppression of flux flow on the GB



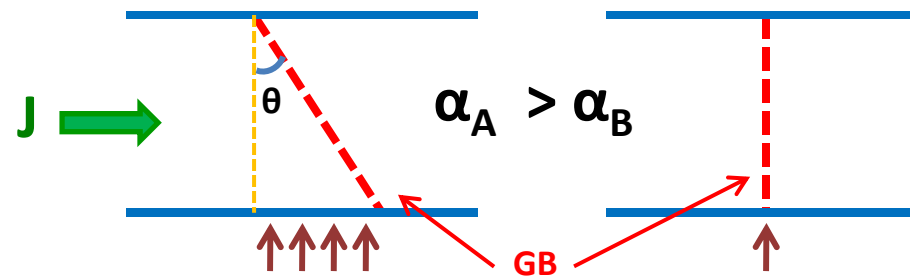
1. Split A vortex



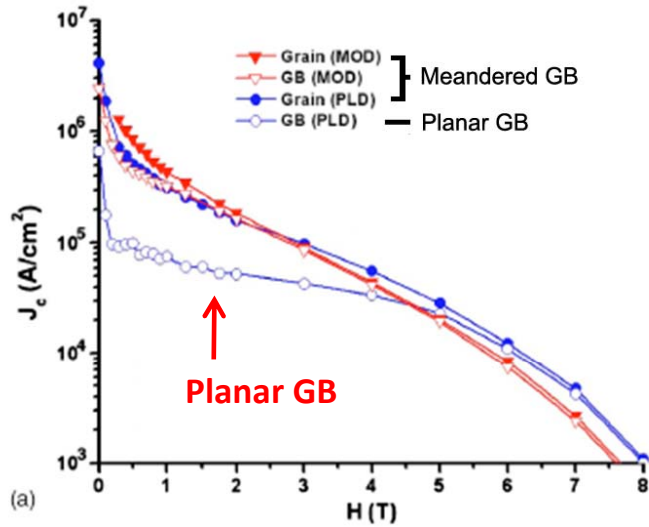
- A-J core size: $l = \lambda_j^2 / \lambda = \xi J_d / J_b$
 - The A-J cores overlap if $l > a$, or
- $$H > (J_b / J_d)^2 H_{c2}$$
- Viscous flux motion $V = (I - I_b)R$
 - $R(B)$ is **independent of B**, if a single vortex chain moves along GB, while $l > a$

Gurevich et al. PRL 88, 097001 (2002)

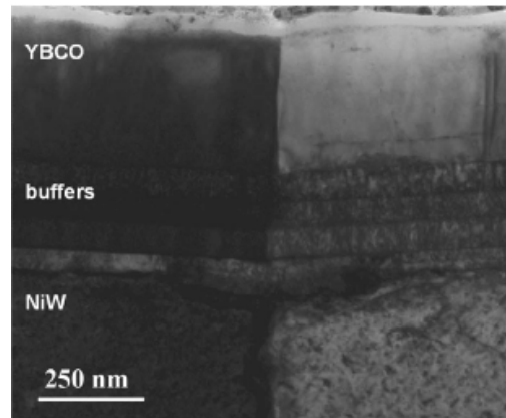
2. Increase of GB area



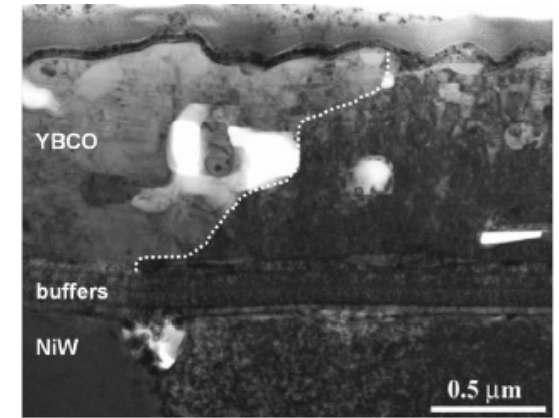
J_c enhancements on meandered GBs of YBCO



Planar GB (PLD)

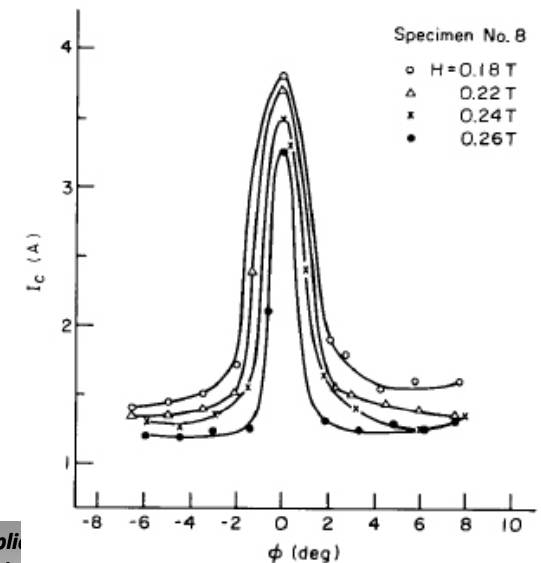
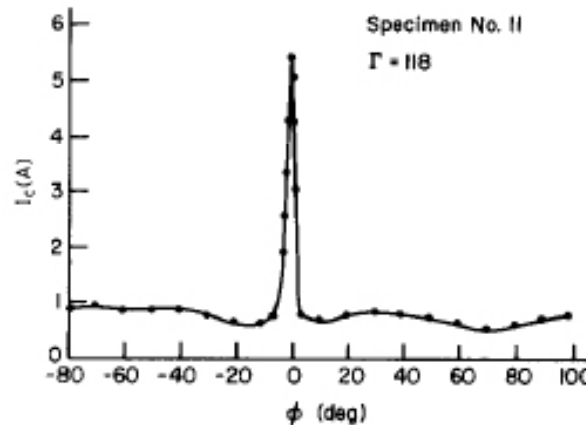
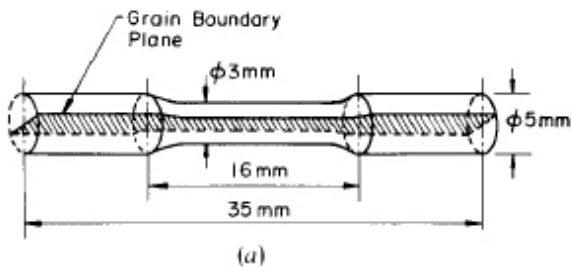


Meandered GB (PLD & MOD)



Feldmann et al., JAP 102 (2007); J.Am.Ceram.Soc (2008)

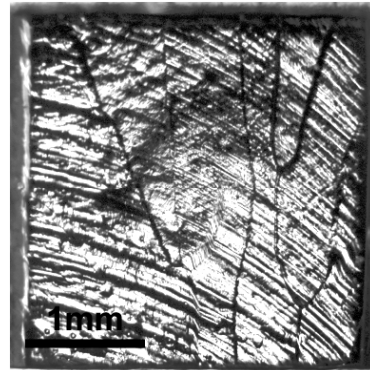
I_c enhancement when $H_{ext} //$ the GB plane of Nb



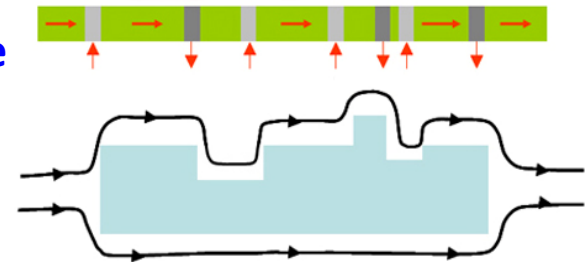
B.C. Cai et al., Phil. Mag. B. (1987); A. Dasgupta et al, Phil. Mag. B. (1978)



Field enhancement by severe surface topology @GB

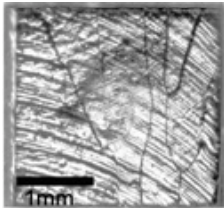


Mo contrast due to topological defects



WELDED AREA: Machine marks (like grooves), large grains and height steps at GBs are well visible

As-received



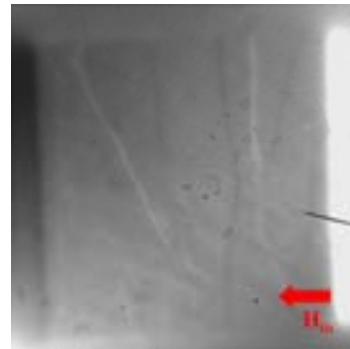
Flattened



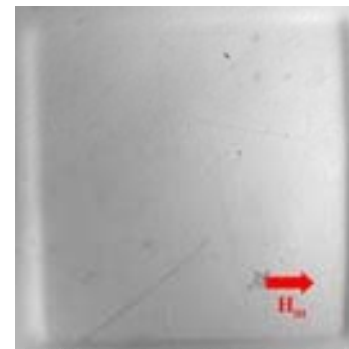
Post-BCP



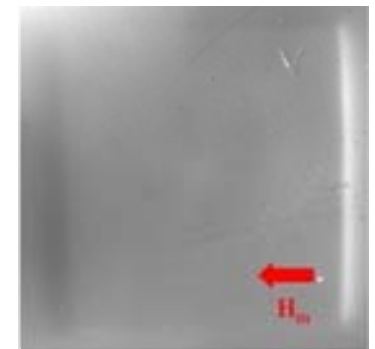
As-received



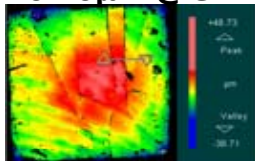
Flattened



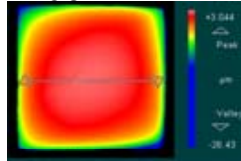
Post-BCP



10-20μm @GB



Flat



3-7μm @GB

