An Investigation of the Properties of Grain Boundaries in BCP Nb for SRF Cavities

Presented by Peter J. Lee (UW-Madison)
On behalf of David Larbalestier, Alex Squitieri, Anatolii Polyanskii, Matt Jewell, Bill Starch (UW-Madison) and Pierre Bauer, Cristian Boffo and Leo Bellantoni, Helen Edwards (FNAL)

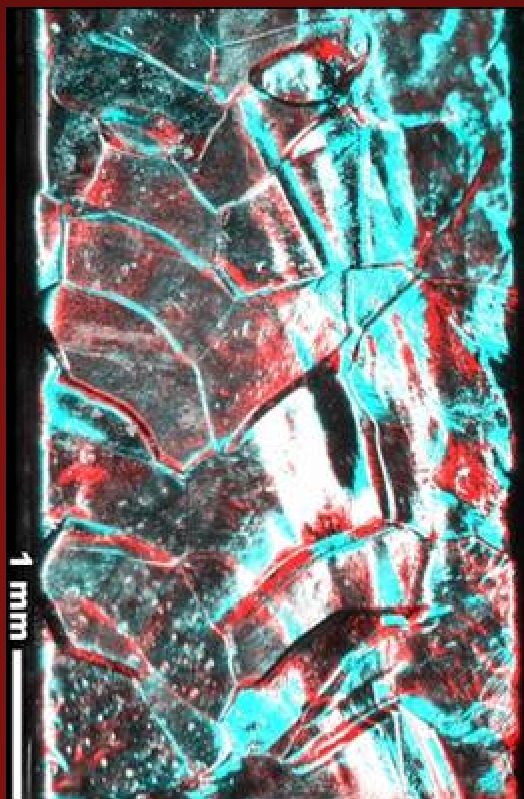
FNAL–UW-Madison Collaboration

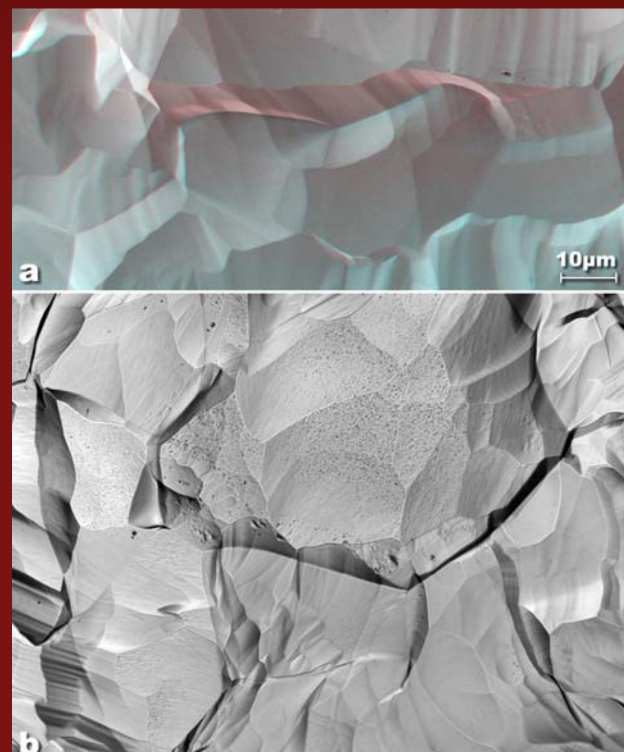
- Primary thrust:
  - Better understanding of the effect of grain boundaries on the RF surface resistance using existing tools available at the Applied Superconductivity Center and FNAL
    - **Magneto-optics (shown here)**
    - Surface analysis – XPS and SAM
    - **Microscopy (shown here)**
    - Transport measurements.
    - Specific Heat and magnetization
- Small scale samples fabricated from sheet at FNAL

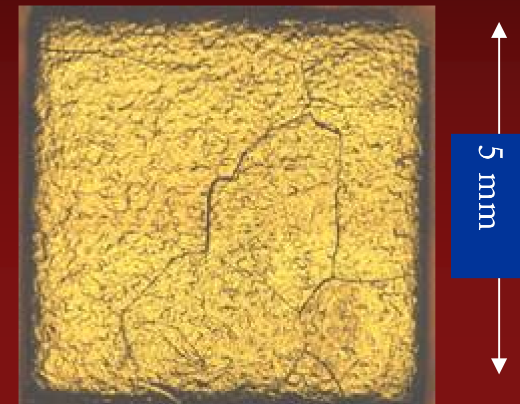
From your background material: P. Kneisel

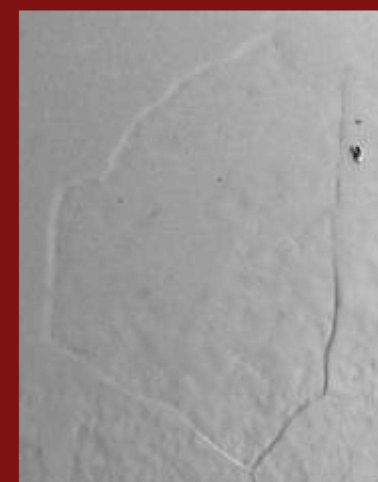
"Investigations on samples using "classical" surface analytical tools are useful, but it seems to be a "dream" for now to correlate the findings from such sample tests to cavity performance. After all, these methods use "outer" (valence) electrons, whereas the superconducting properties are determined by conduction electrons. **Therefore, "superconducting" methods such as penetration depth, magnetization, pinning and susceptibility seem to be better suited to correlate sample features to cavity performance."**

High Purity Nb Samples









Weld-sample ("BCP1")

BCP100,HT8005hr,BCP20

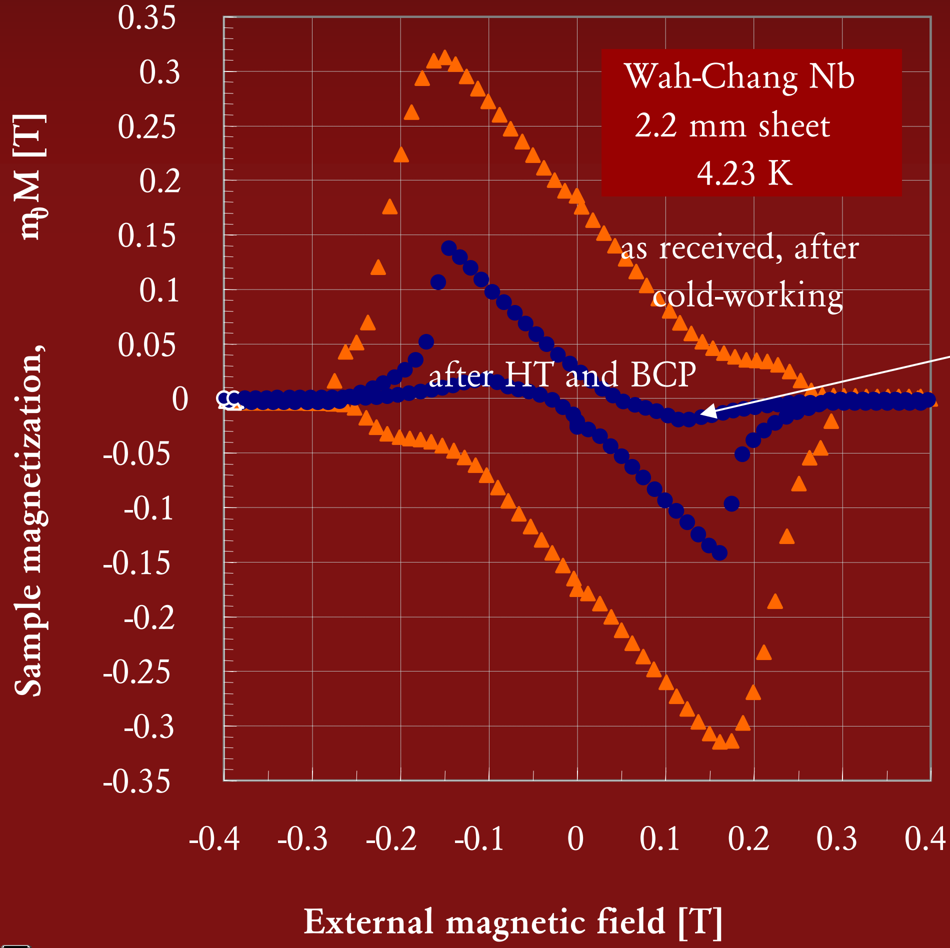
Regular sample (50m) ("BCP2")

BCP80,HT8005hr,BCP20

Large Grain sample ("Texas")

(BCP2 + 1250C24hr+50mBCP)

MAGNETIZATION: AS RECEIVED AND AFTER BCP (100mm) & HT (800C/5hr)



Flux pinning due to cold working → strongly reduced after etching and heat treatment

Result consistent with recent data for BCP & HT samples (RRR300) from Casalbuoni:

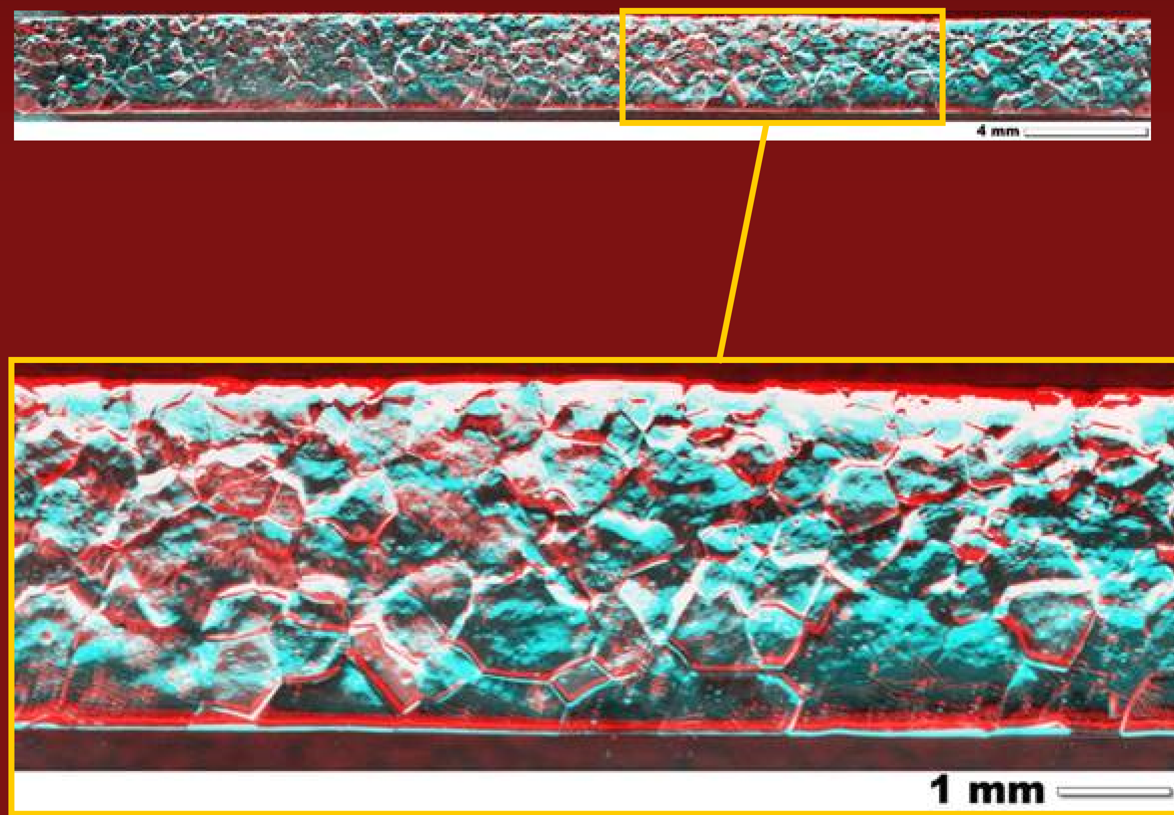
$B_{c,therm}$(4.2 K)=142 mT (→ 147 here)

B_{c2}(4.2 K)=267 mT (→ 290 here)

C. Boffo - FNAL

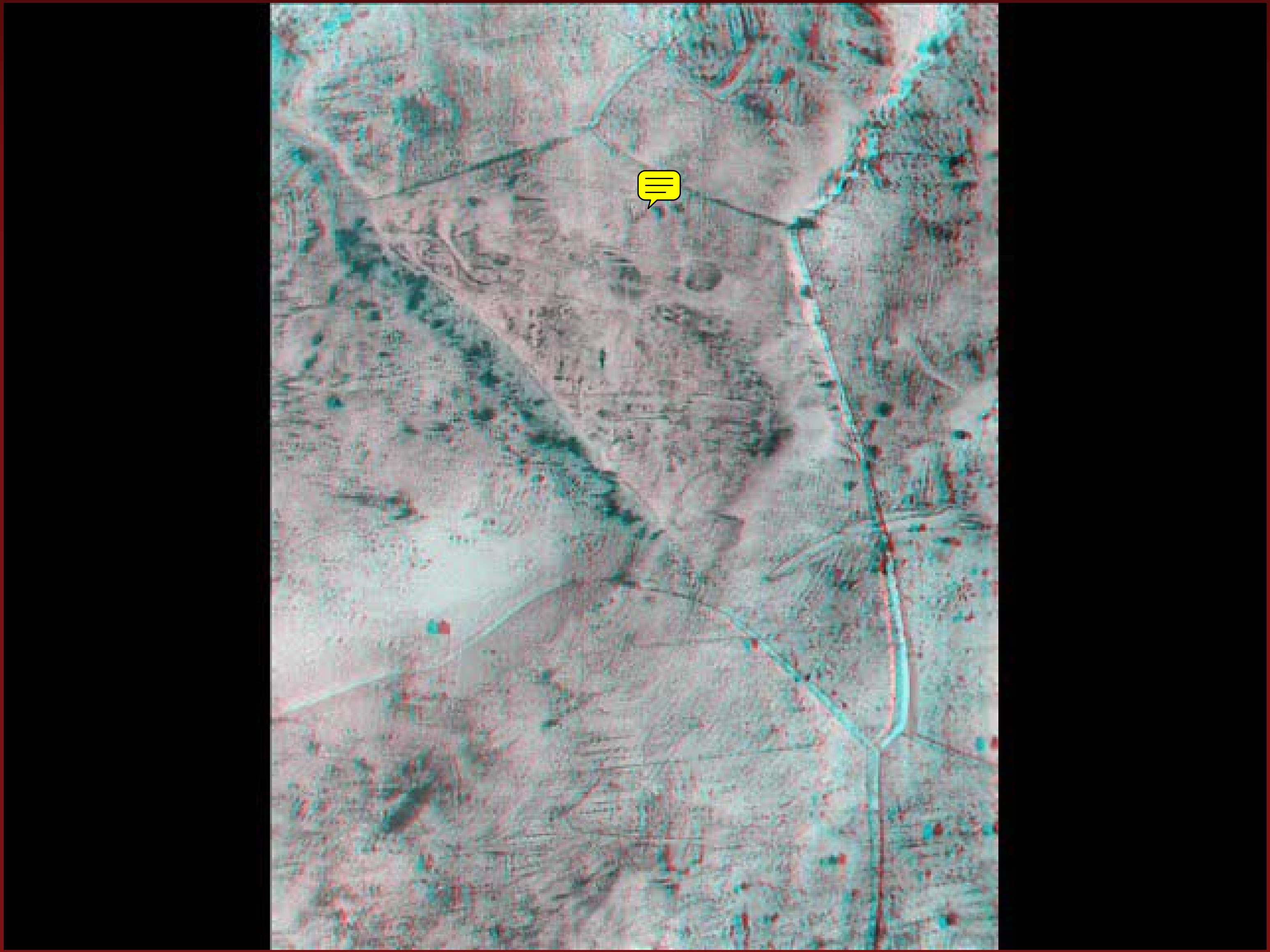
Weld Region: Grain Boundary Grooving

- Flatbed Scanner Images

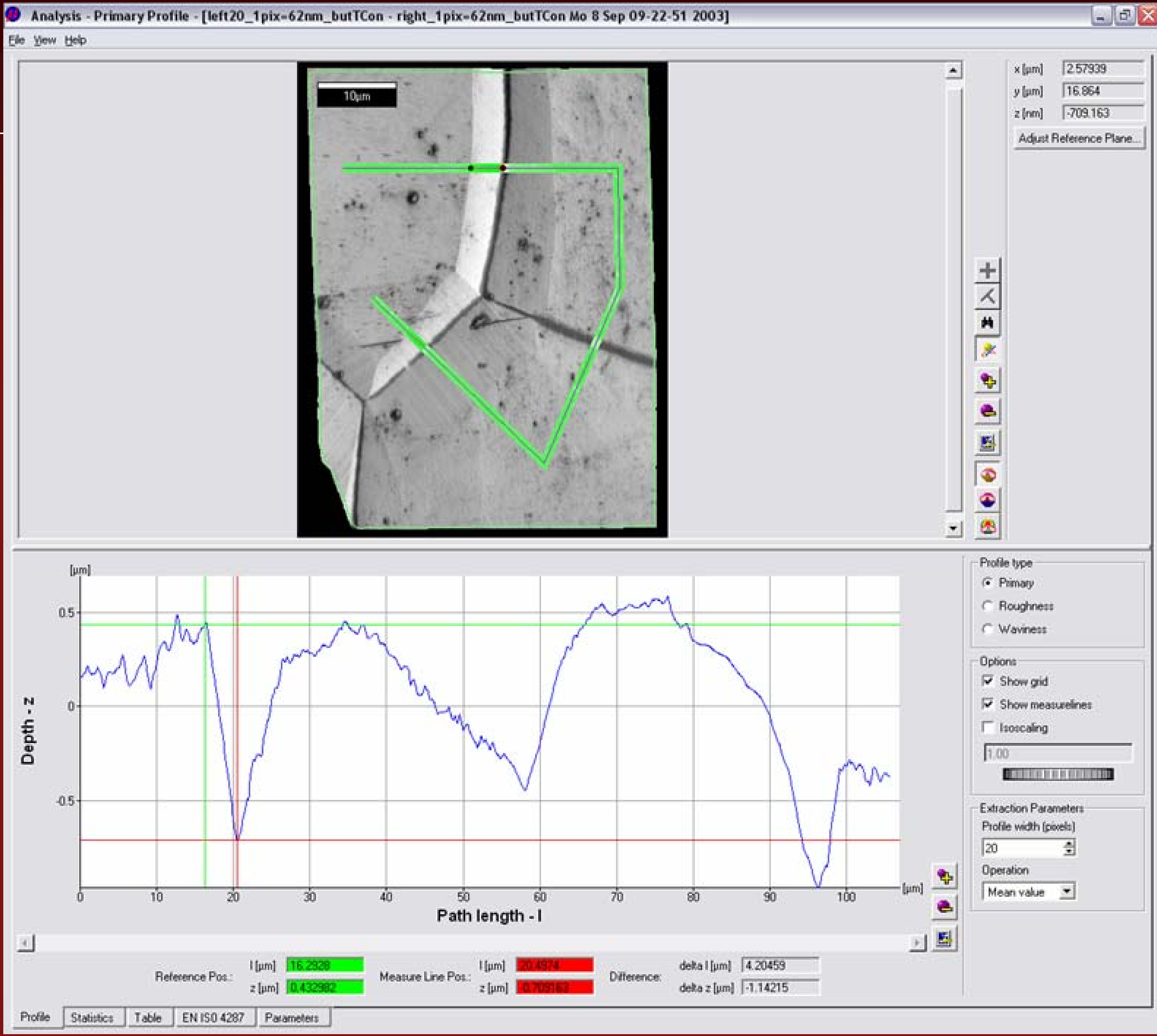


Grain Boundary Groove in Weld Region (Weld)

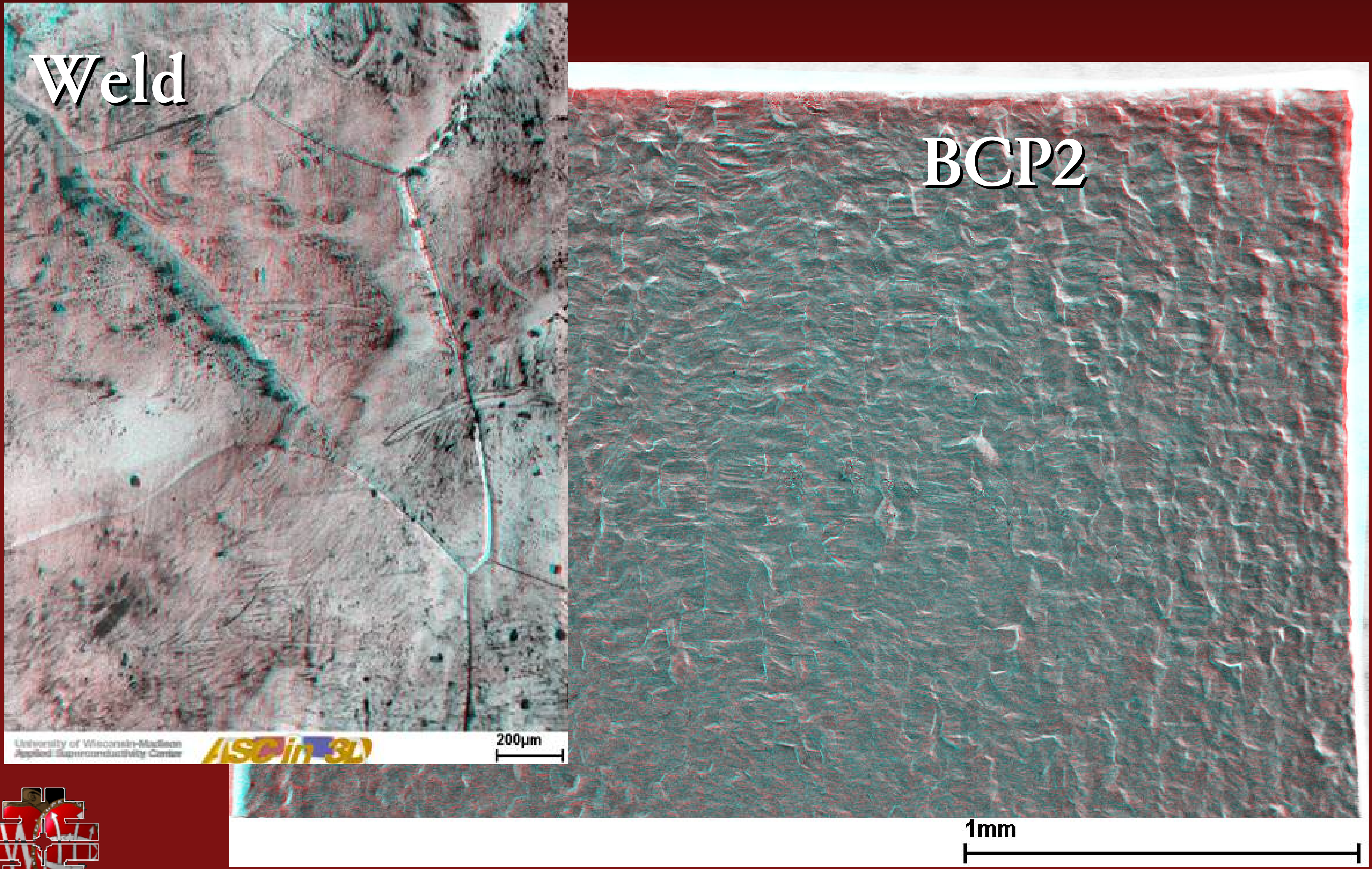
3D Movie



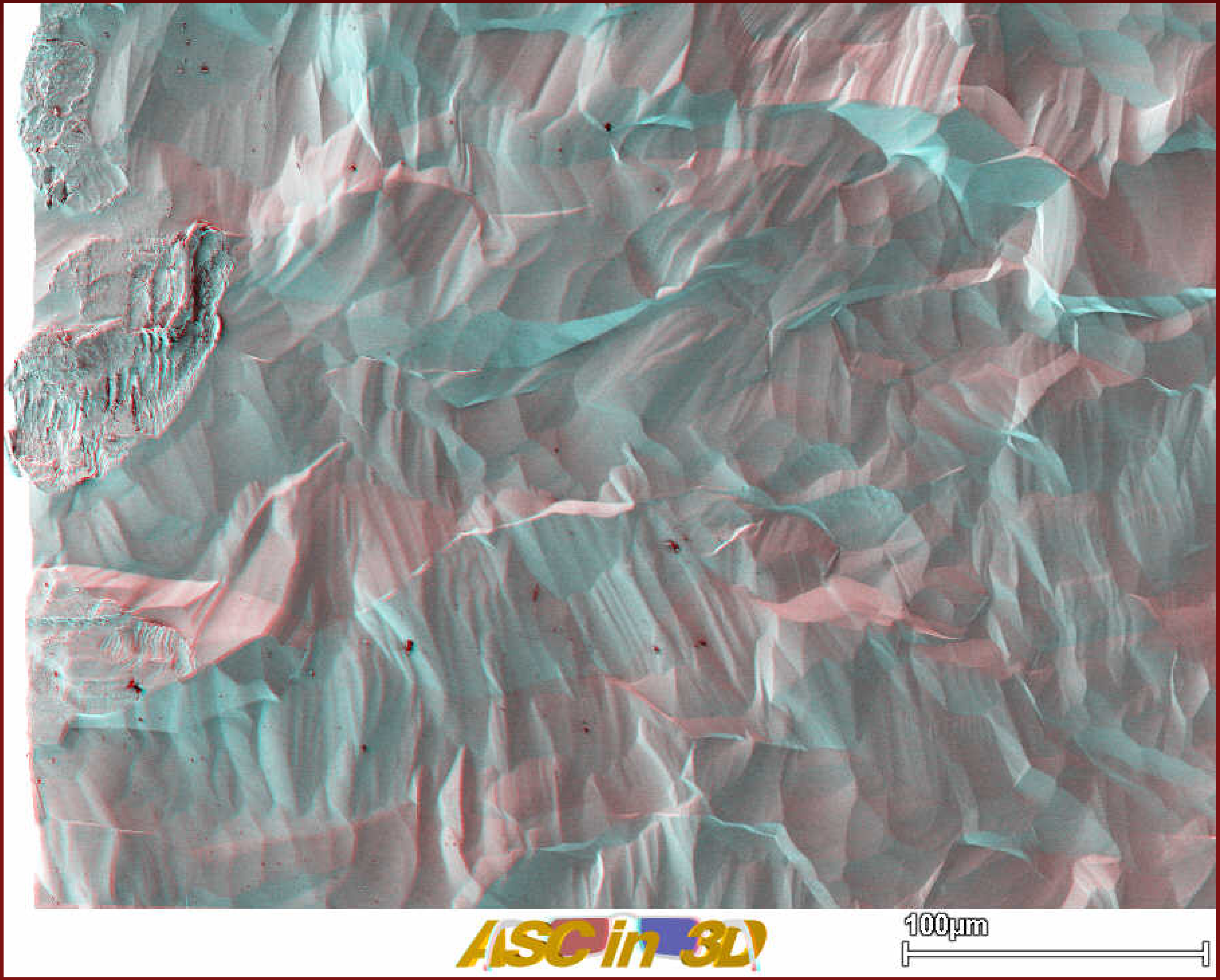
Depth map from grain boundary



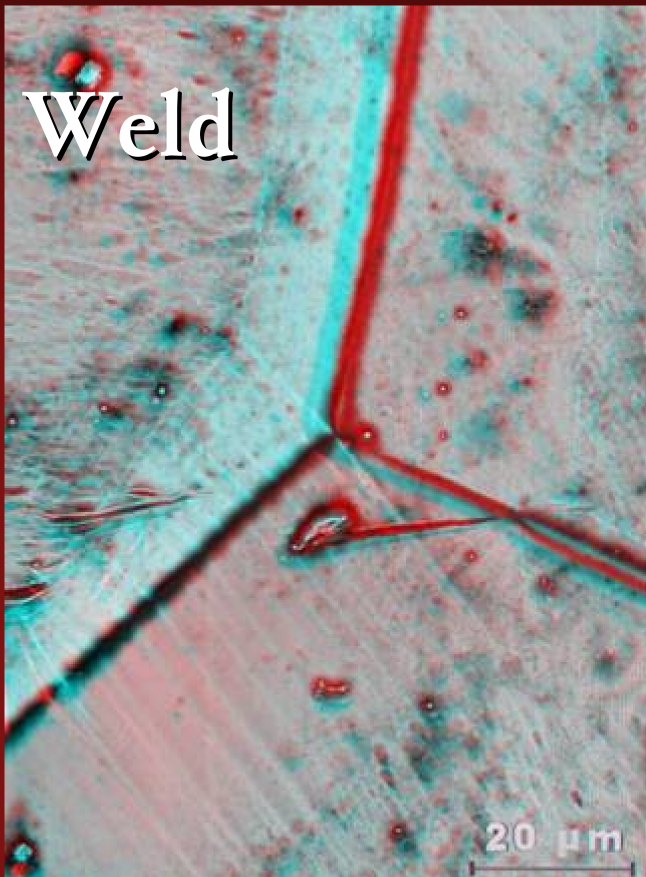
Weld vs BCP2 at same Magnification



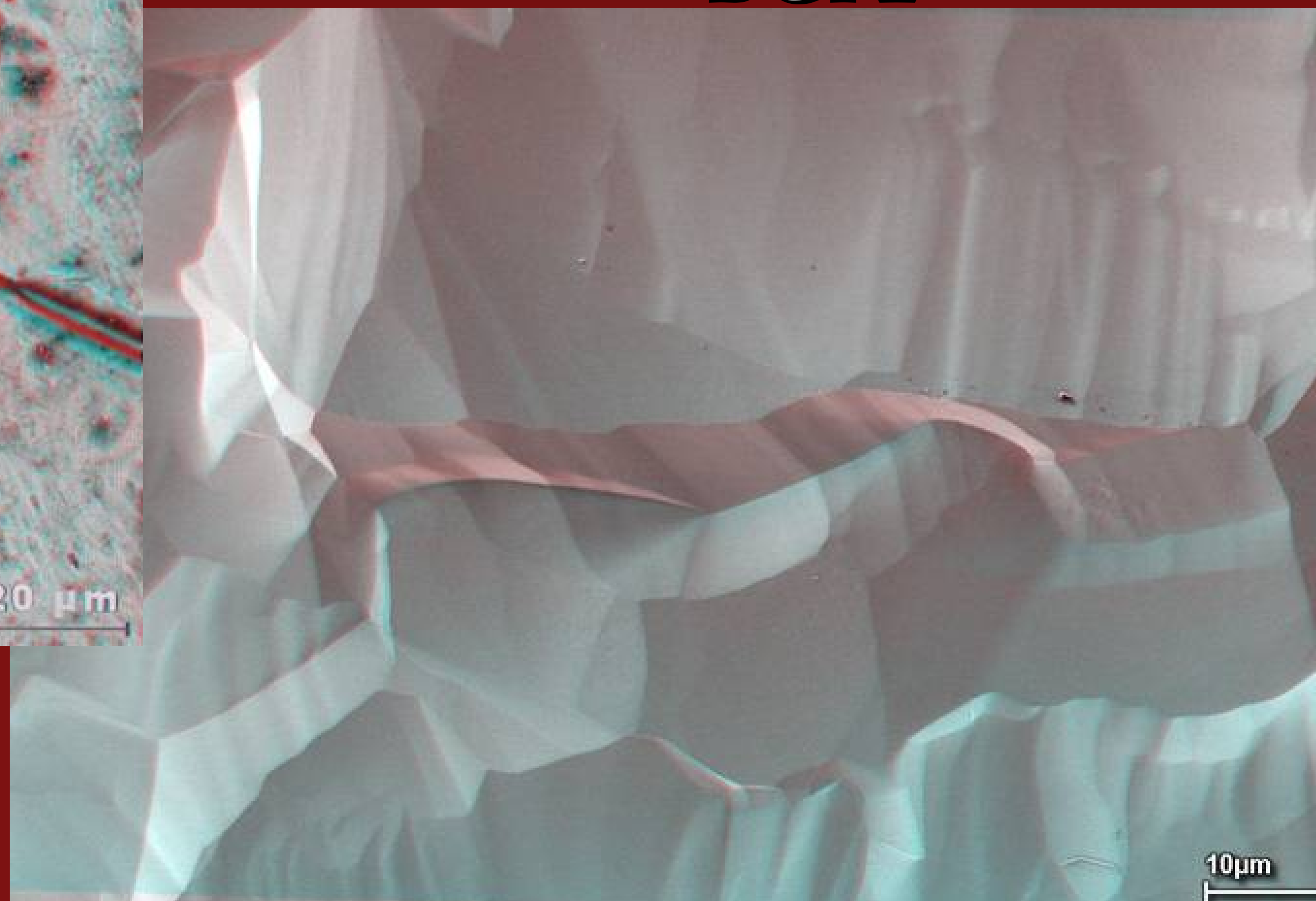
BCP2 x 250



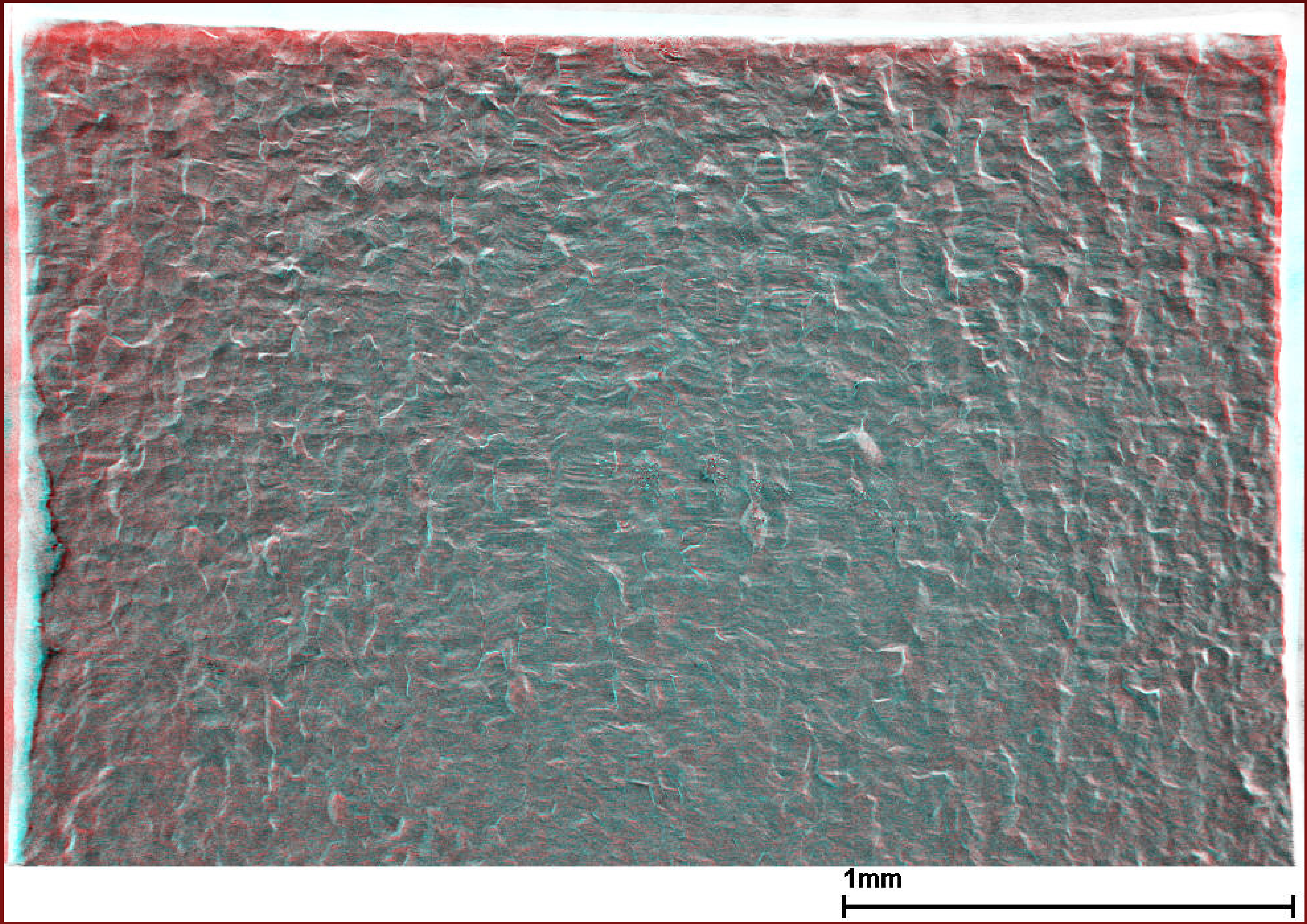
Weld vs BCP2 at × 1,000 magnification



BCP2

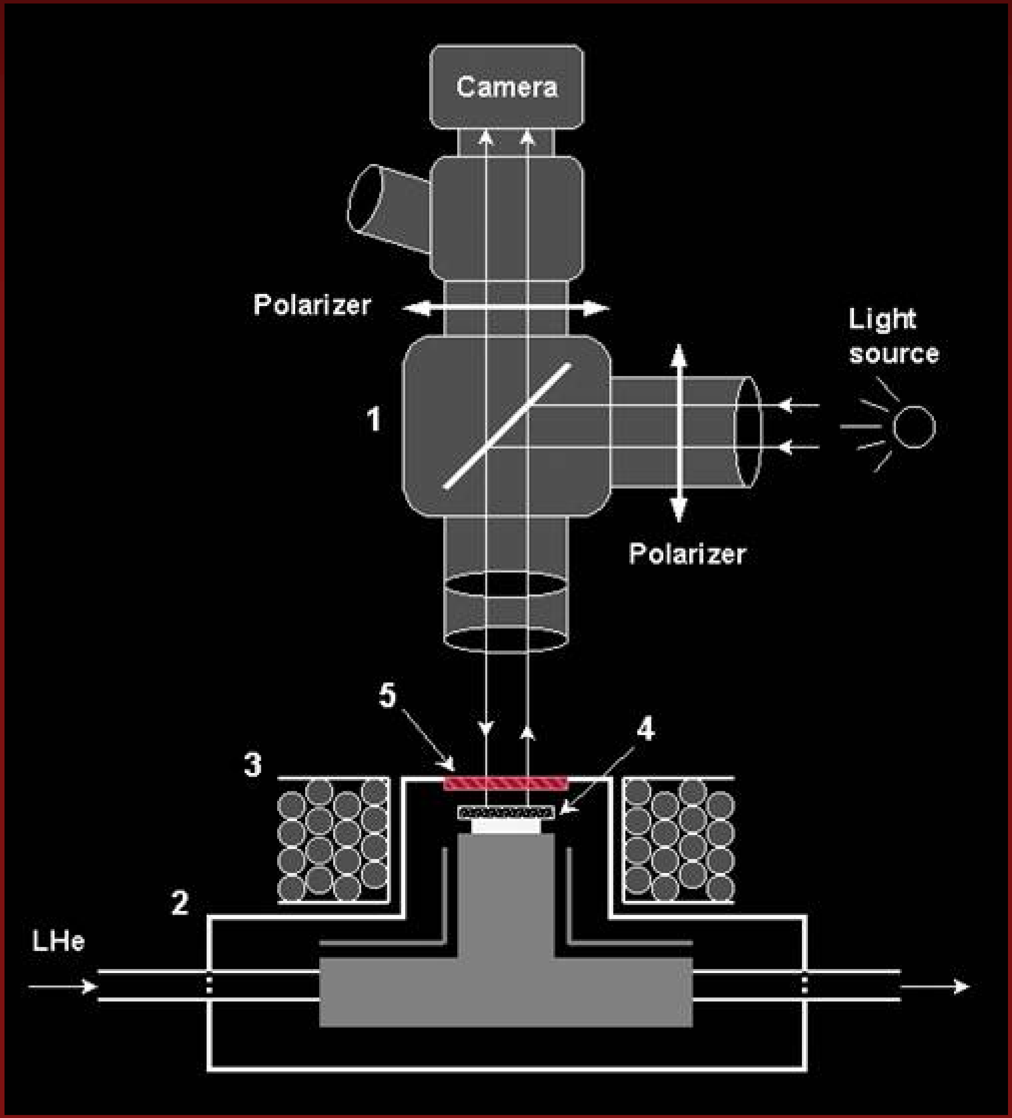


BCP2: Reg. sheet, EDM cut, RRR=400



Magneto-Optical Imaging

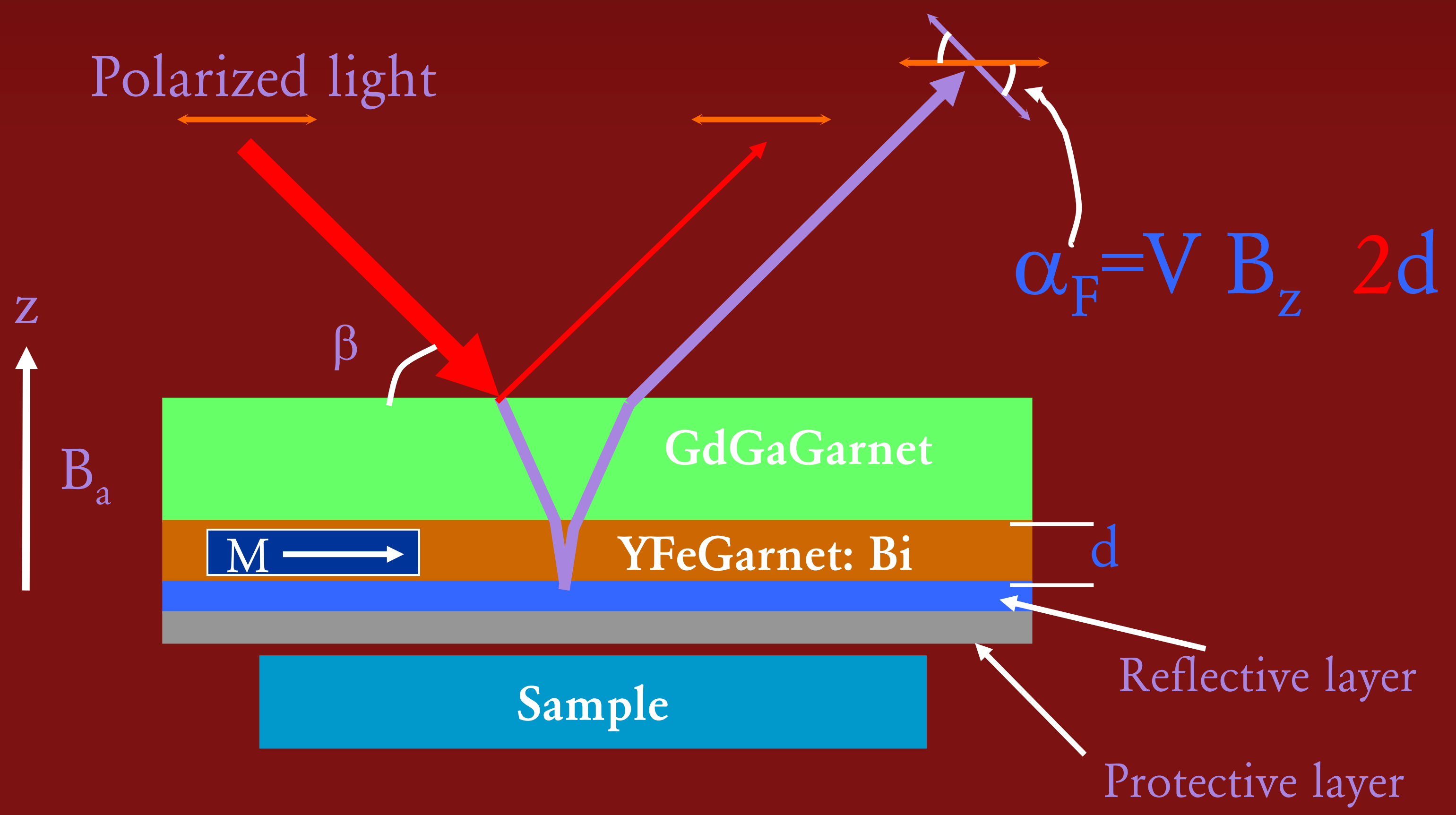
1. Microscope
2. Cryostat
3. Solenoid (Magnet)
4. Sample and Indicator Film
5. Optical window



Slide by Matt Feldmann

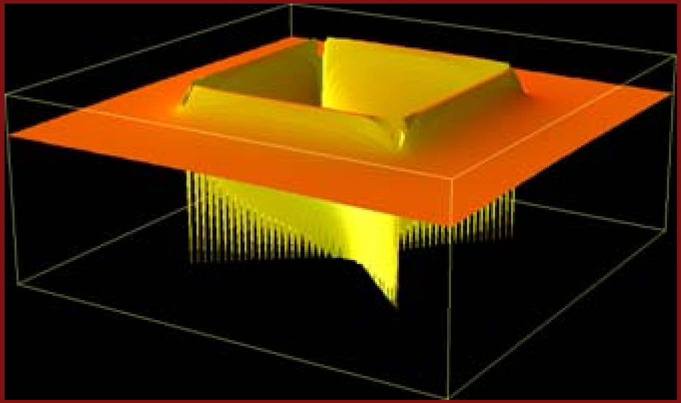
Principle of Magneto optical (MO) measurement of B_z

Double Faraday rotation using in-plane magnetized YIG

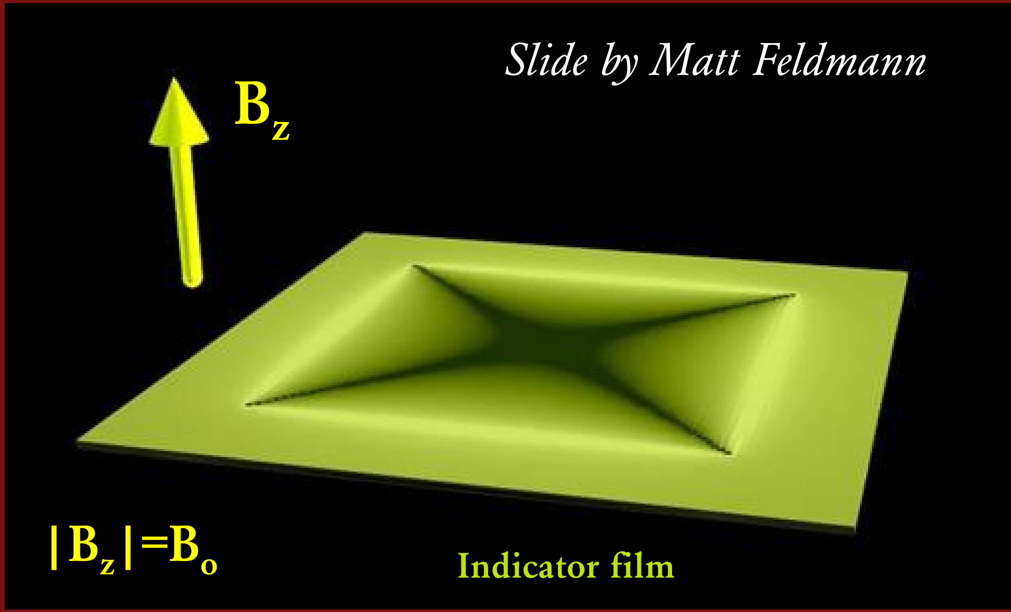


Magneto-Optical Imaging

- Method of visualization of Magnetic Flux
  - Visualizes B_z in a plane above a supercondcutor
- Faraday Effect
  - Rotation of the plane of polarization of light by a magnetic field



3D plot of $B_z(x,y)$ above a superconductor sheet



Intergranular flux penetration in YBCO is common

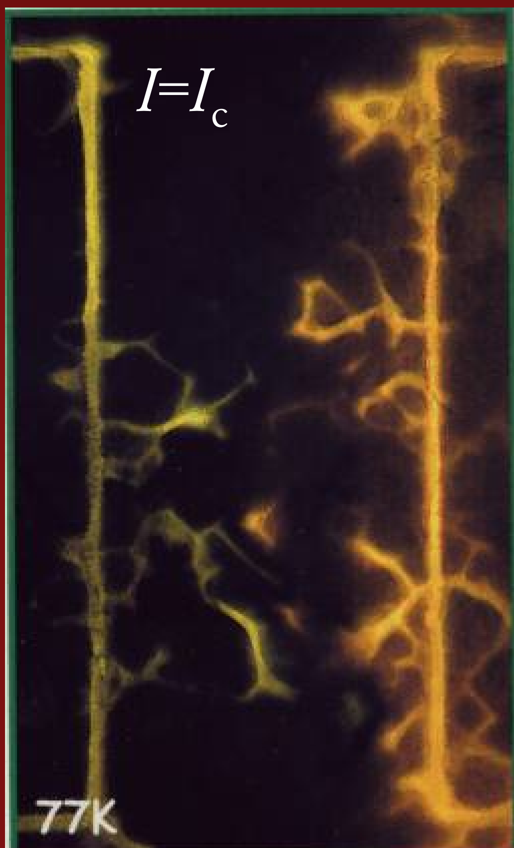
Transport Current

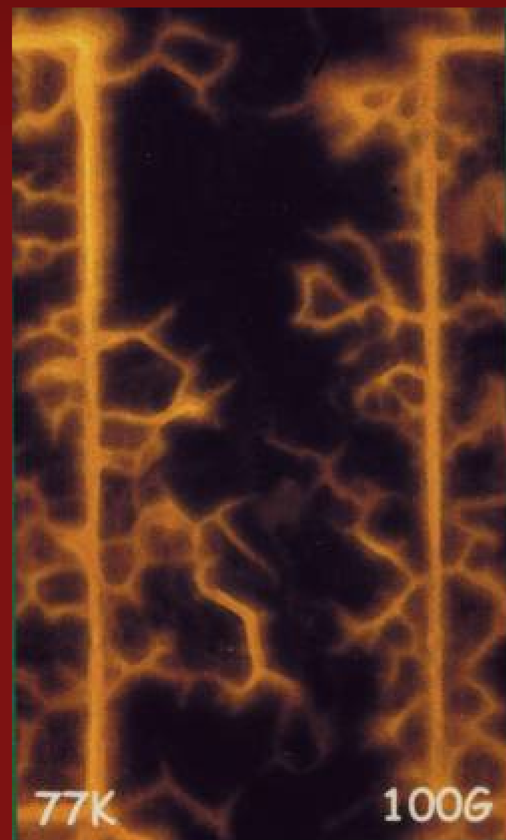
J_c=1.3 MA/cm^2

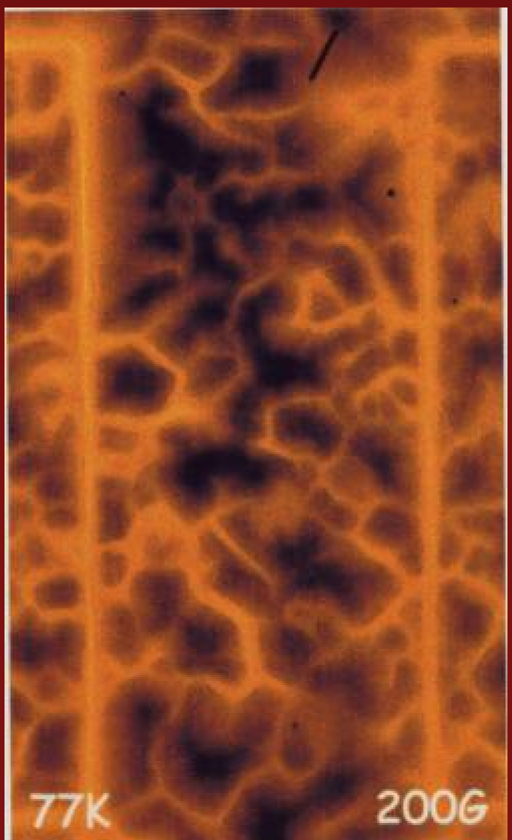
Induced Magnetization Currents

Partial critical state

Full Critical State



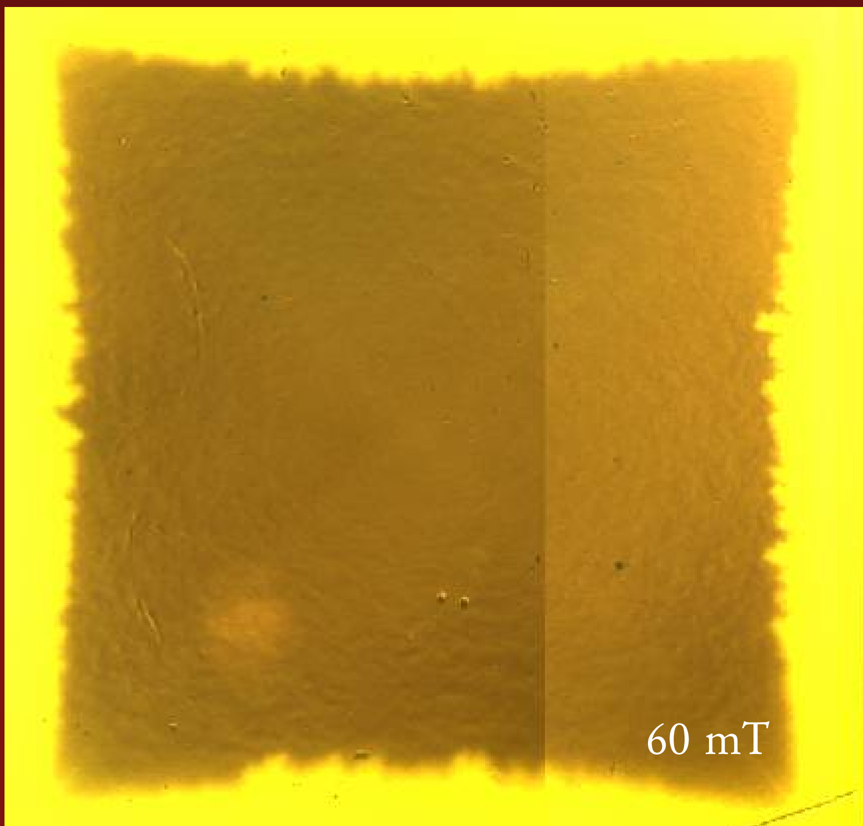


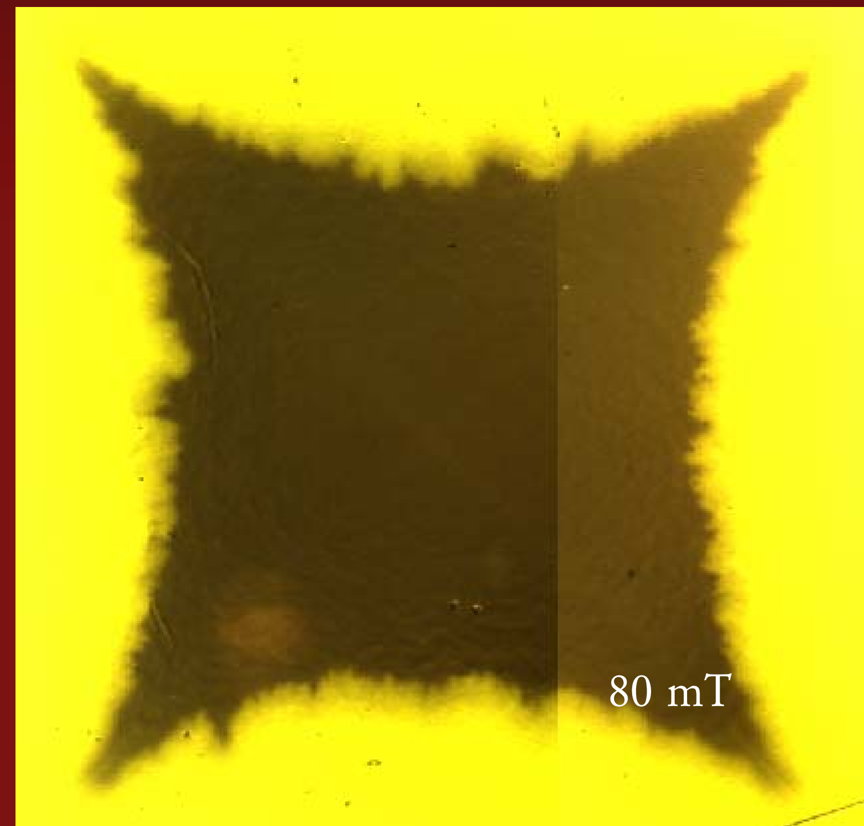


GBs are visible in Magnetizaton MO when: J_c(GB) < J_c(grains)

Even at I_c sample is not yet in the critical state!

Progressive flux penetration above H_{c1} in fine-grain Nb



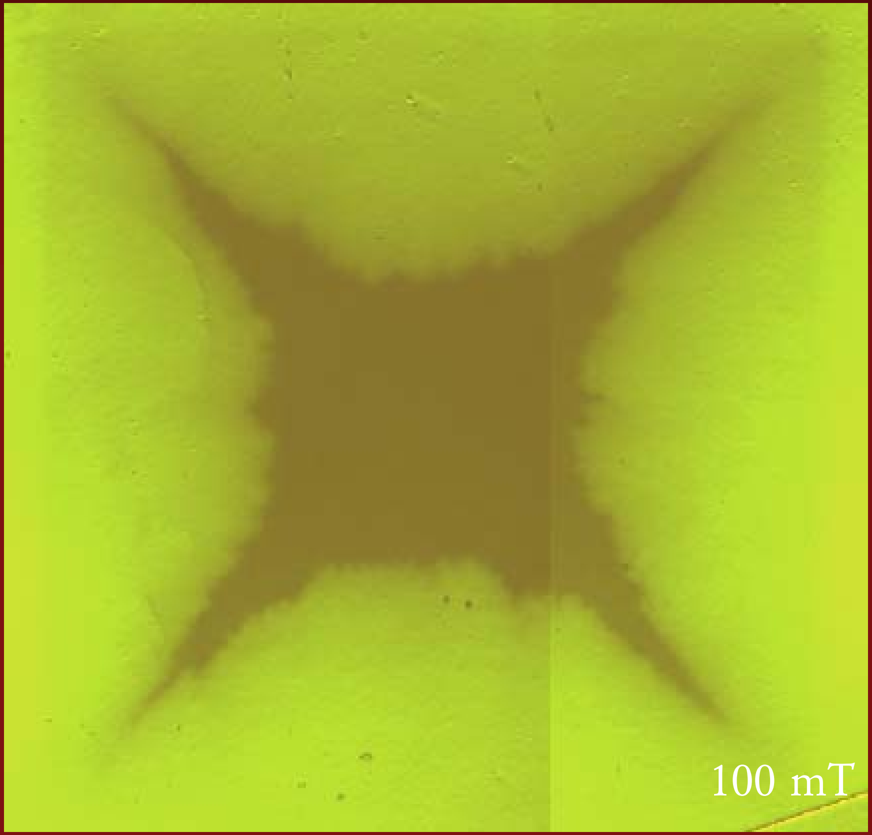


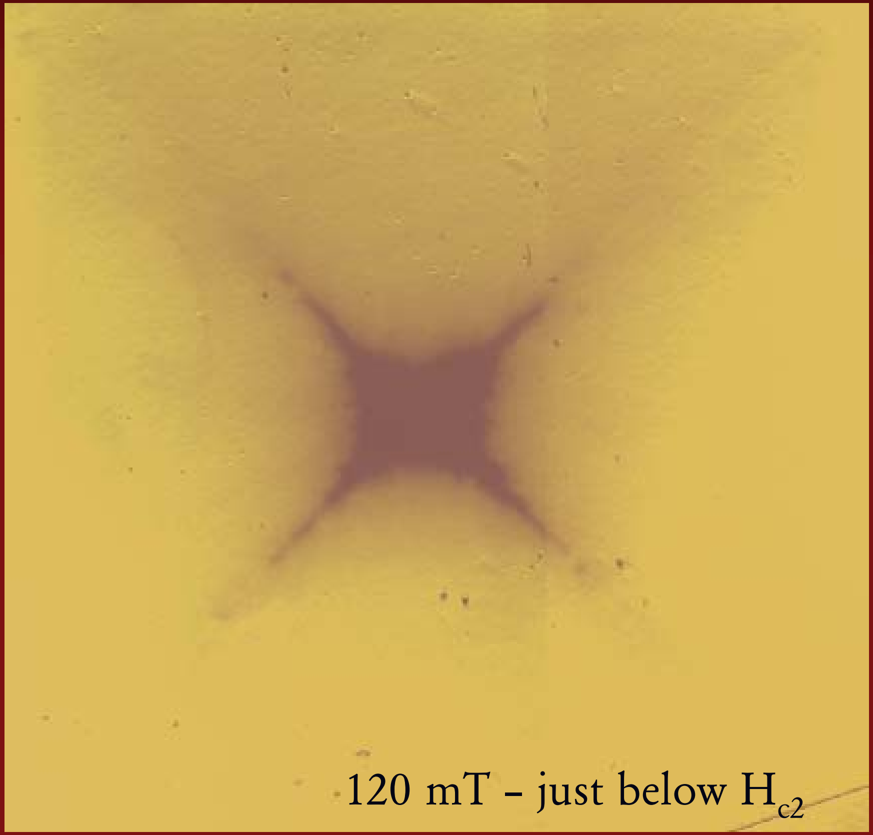
BCP2: Reg. sheet,
EDM cut, RRR=400

H_{c1} ~60 mT, ~38 mT with demagnetization

1 mm

Progressive flux penetration above Hc1 in fine-grain Nb – higher fields

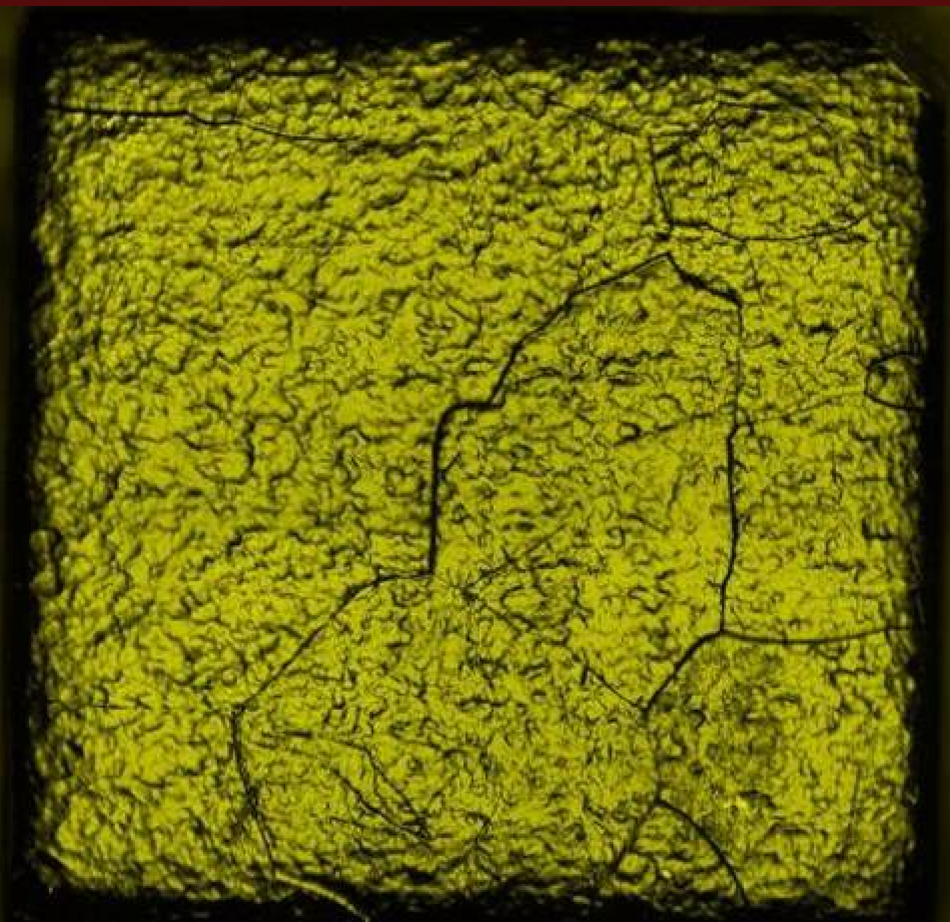




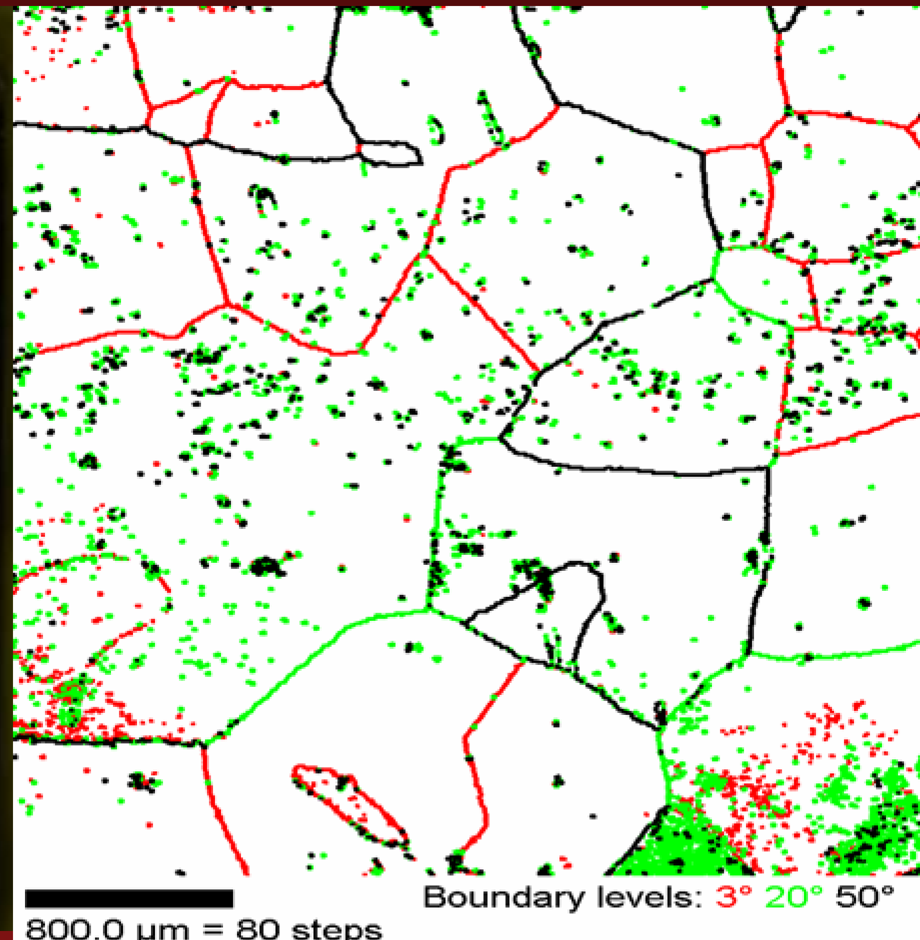
Not quite full flux penetration to the center

Macroscopic B_z flux pattern rather uniform

Large grain sample - some GBs appear after etching



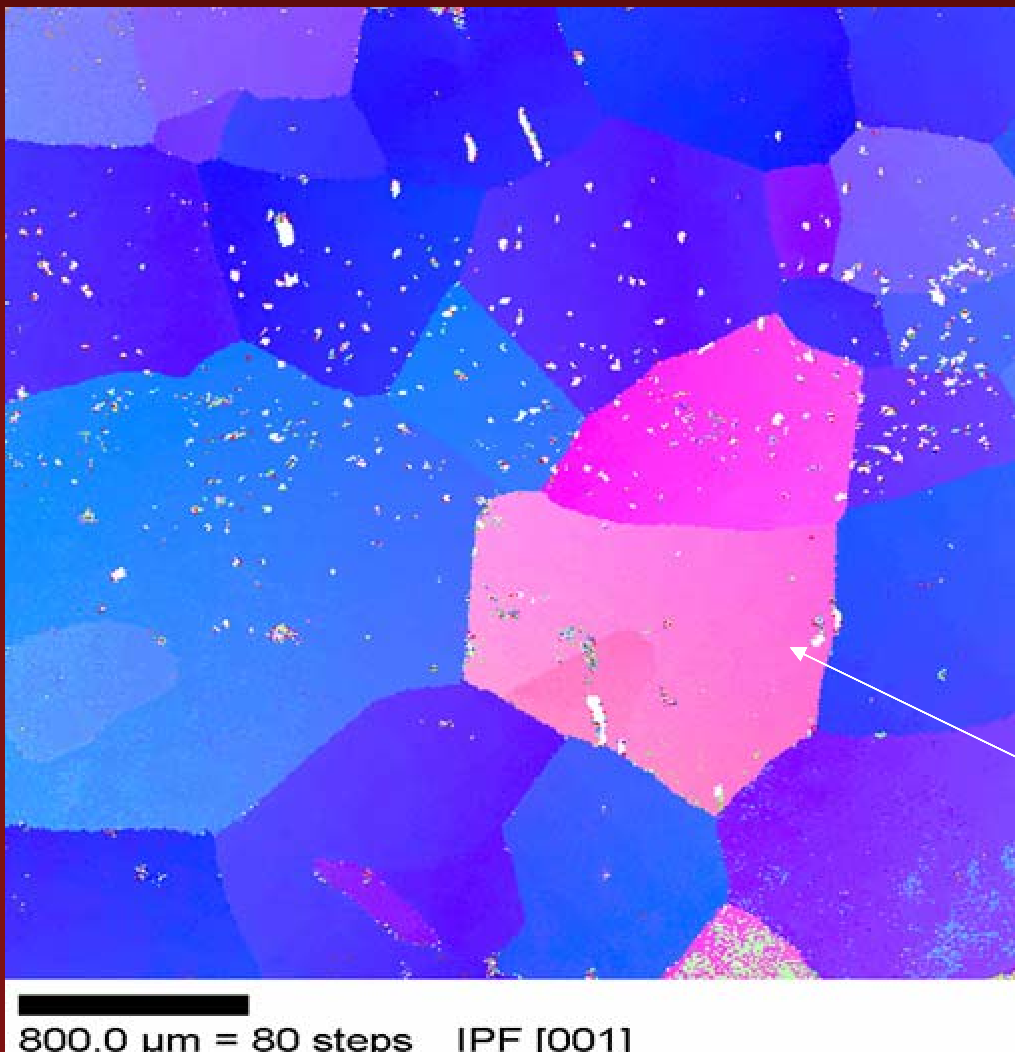
RRR dropped from ~400 to 260

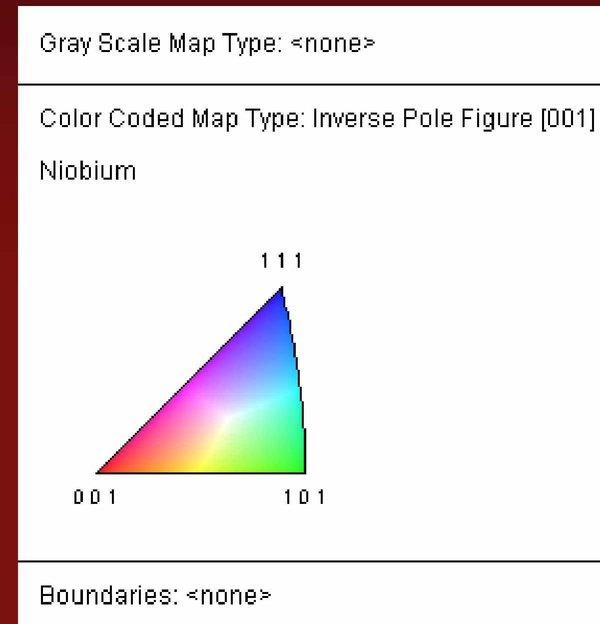


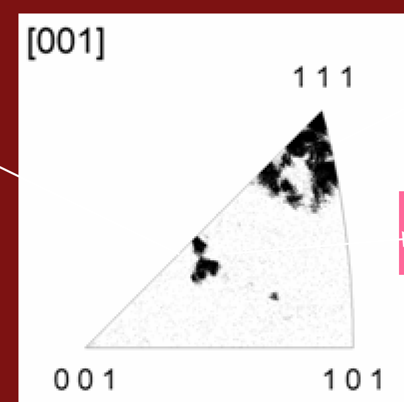
FESEM Orientation imaging (Sang Il Kim)

Large grains produced by 1250 °C, 24hr, 10^{-7} T HT at JLAB

Inverse Pole Figure indicates some texture







Blue

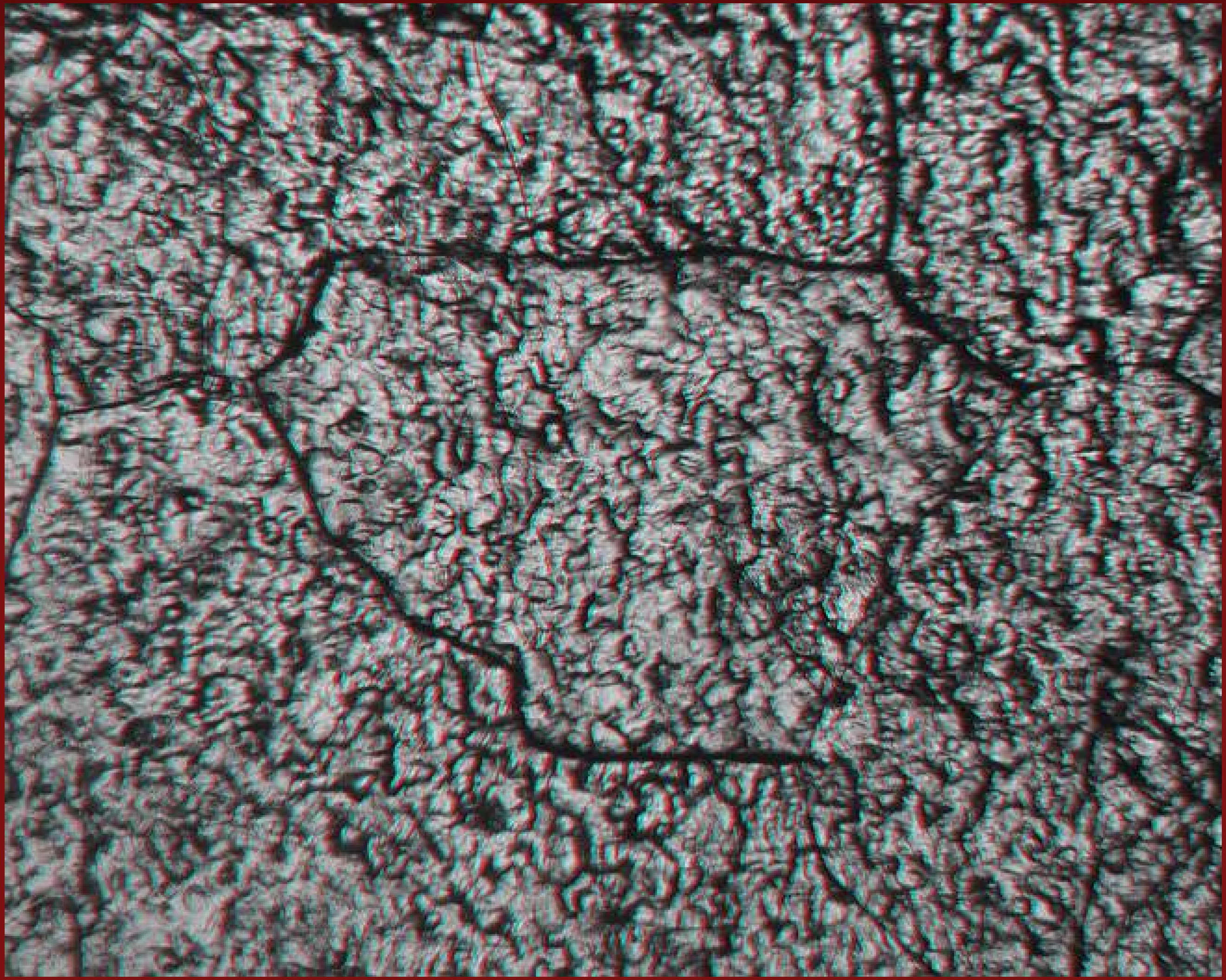
Reddish

Light Microscope Depth Reconstruction

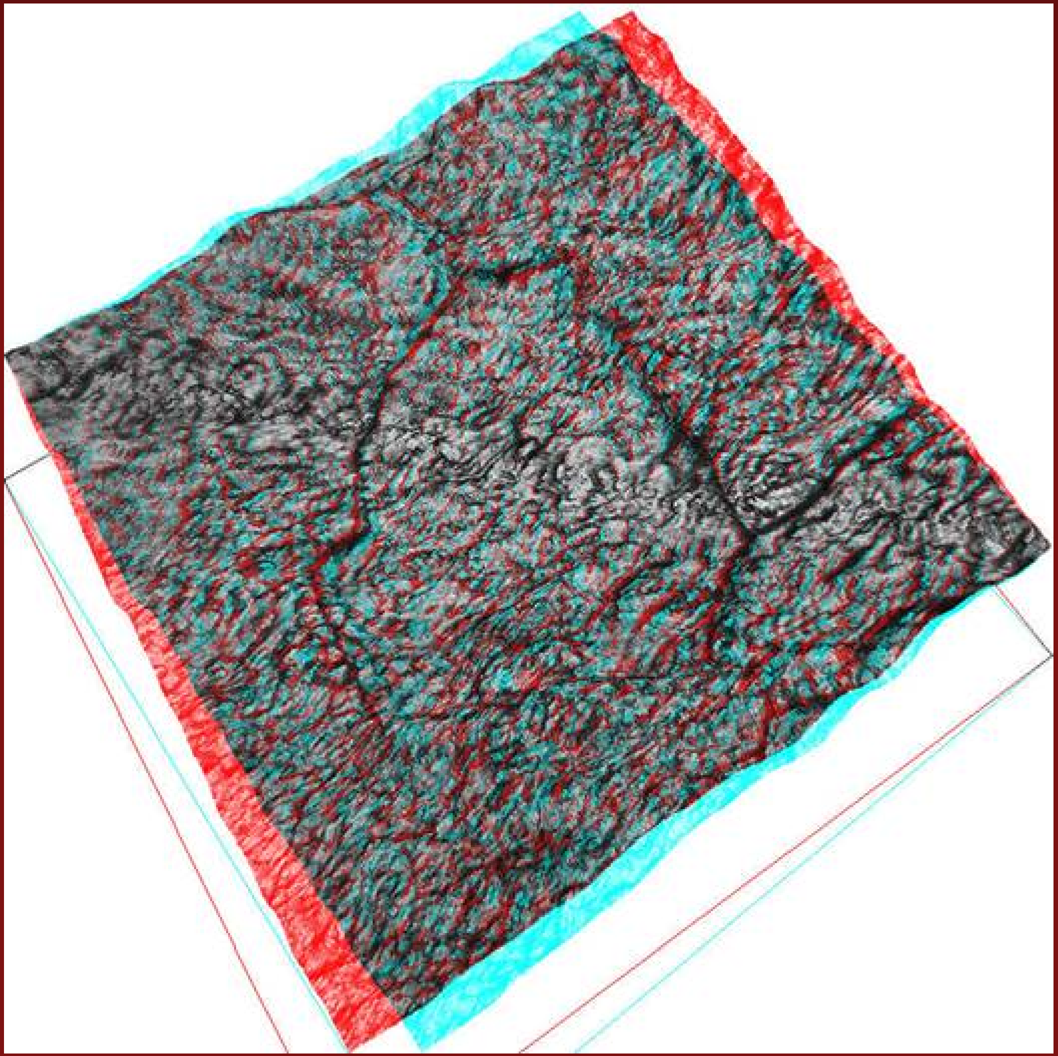
120 µm range at
10 µm focus intervals

ImageJ with Extended Depth of Field plugin by Daniel Sage @ epfl.ch

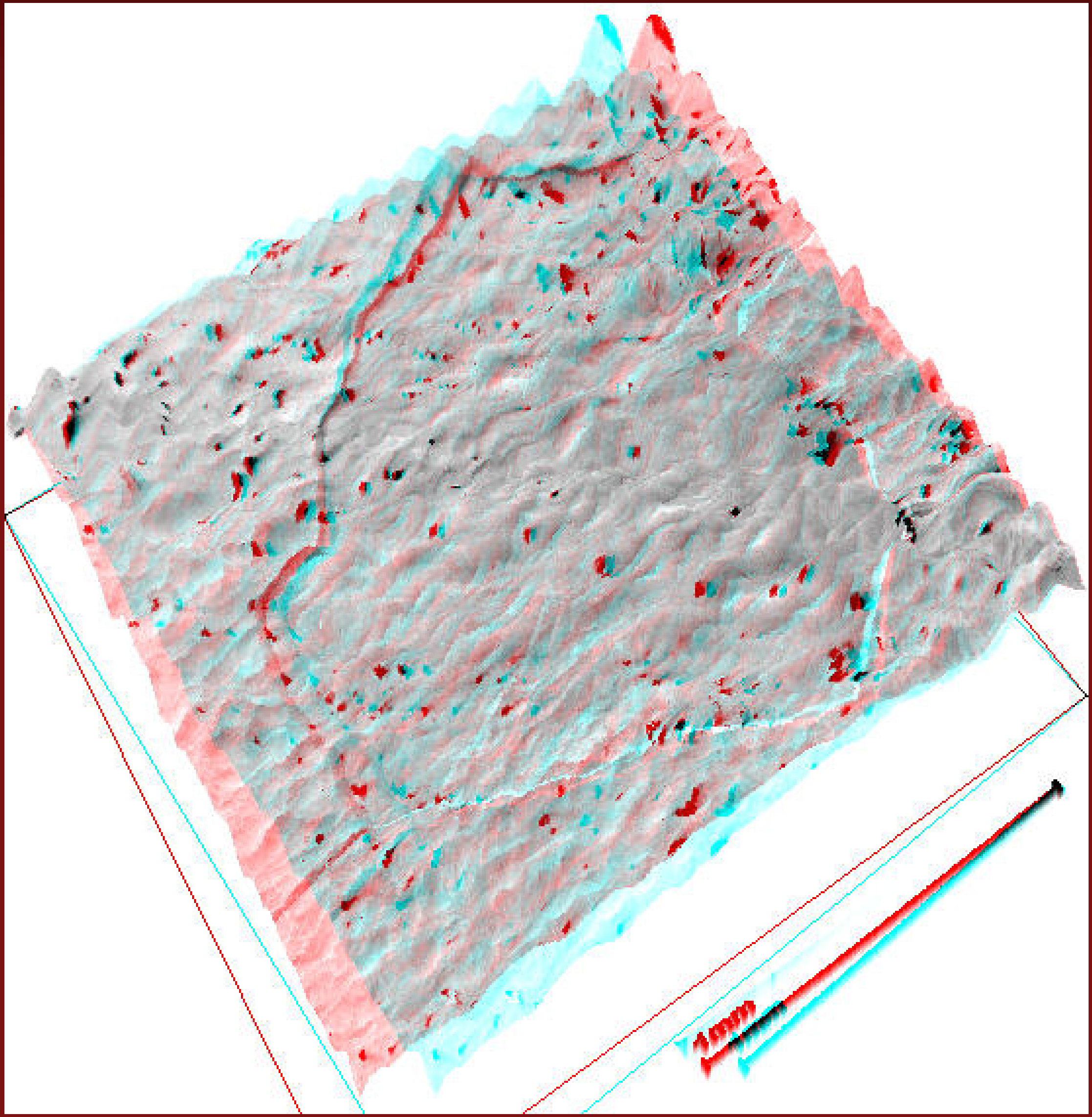
B. Forster, D. Van De Ville, J. Berent, D. Sage, M. Unser, "Extended Depth-of-Focus for Multi-Channel Microscopy Images: A Complex Wavelet Approach," Proceedings of the Second 2004 IEEE International Symposium on Biomedical Imaging: From Nano to Macro (ISBI'04), Arlington VA, USA, April 15-18, 2004, pp. 660-663.



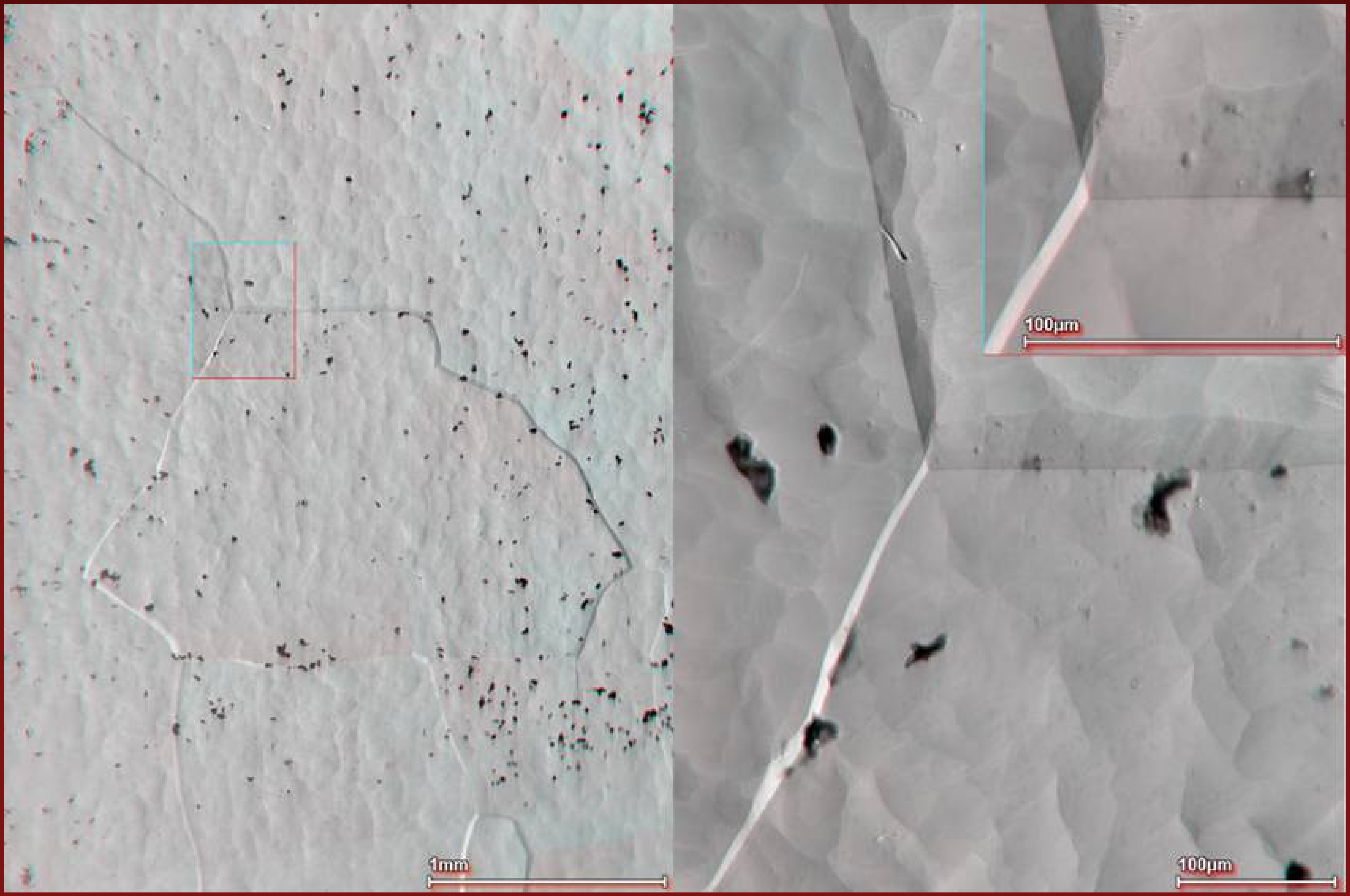
Projection from light microscope height map



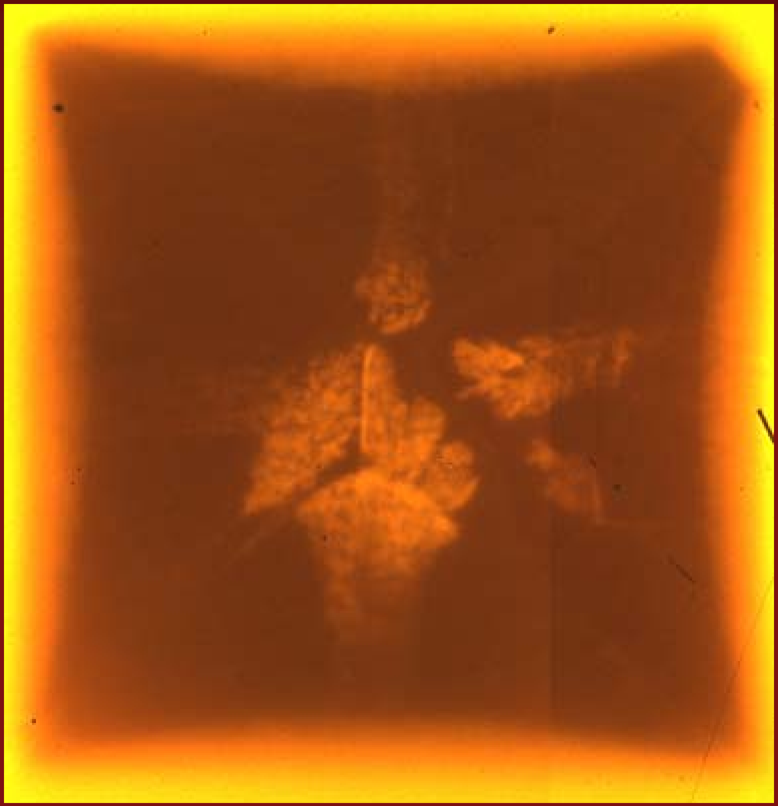
FESEM Image: Height Reconstruction from tilt pair



FESEM: detail of GB facets



Inhomogeneous flux penetration after ZFC to 7K, then 57 mT, well above Hc1 ~ 48 mT



Flux has leaked to center, even as major flux fronts are only partially penetrated

#9-10 H=572 Oe, 27 Apr

Sequential flux maps in increasing H and t, then decrease H to zero

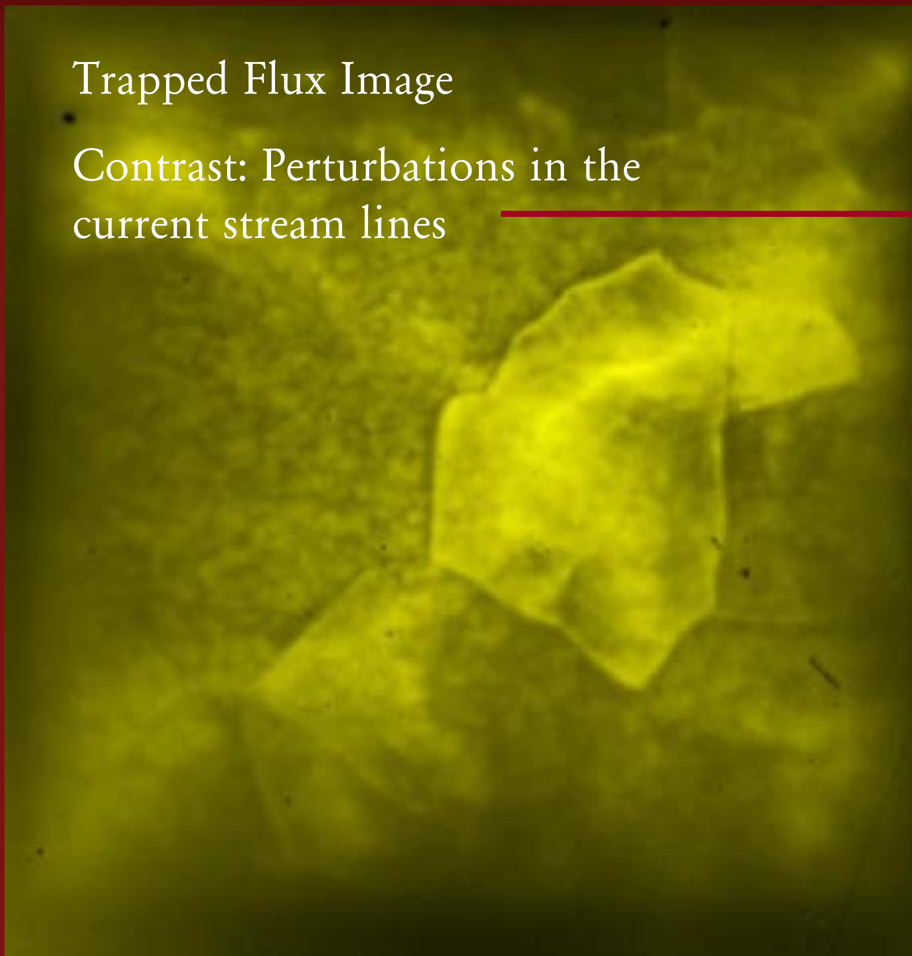
H=0 mT

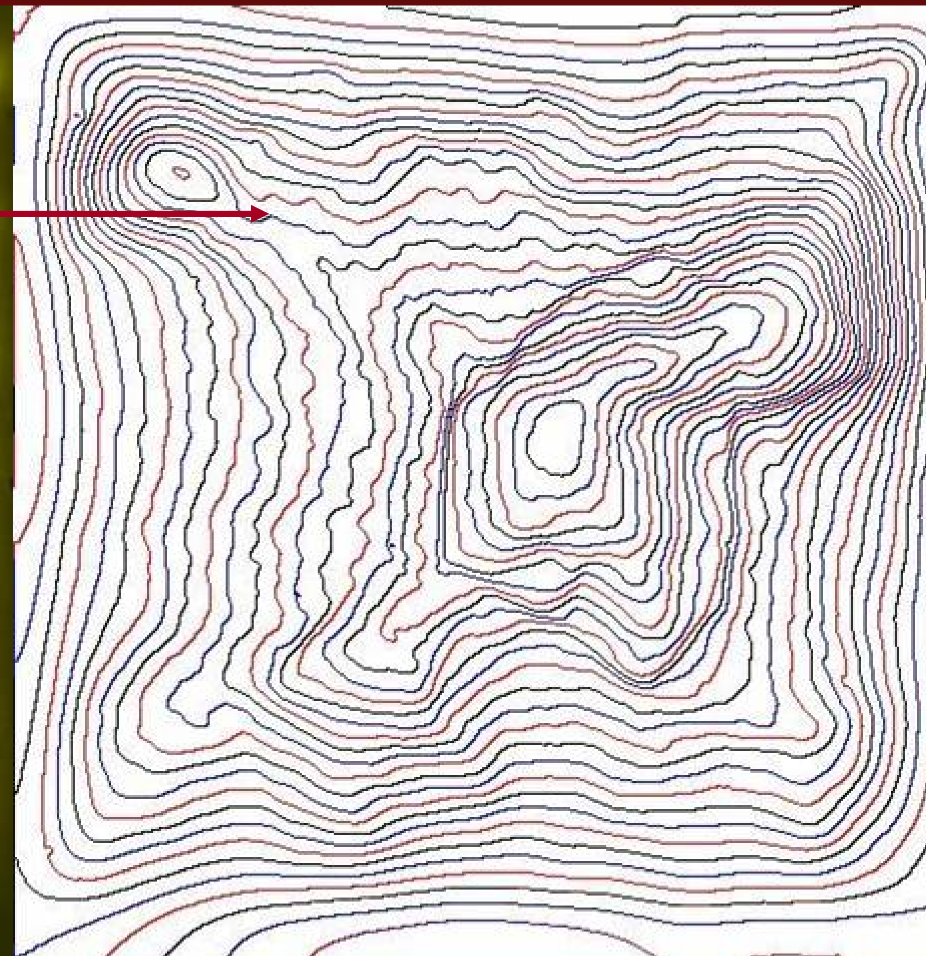
MOVIE

H_{c1} (7K) ~ 48mT (1.6 demagnetization factor ~30 mT)

H_{c2} ~120 mT

MO image shows partial grain structure after magnetizing above H_{c2}





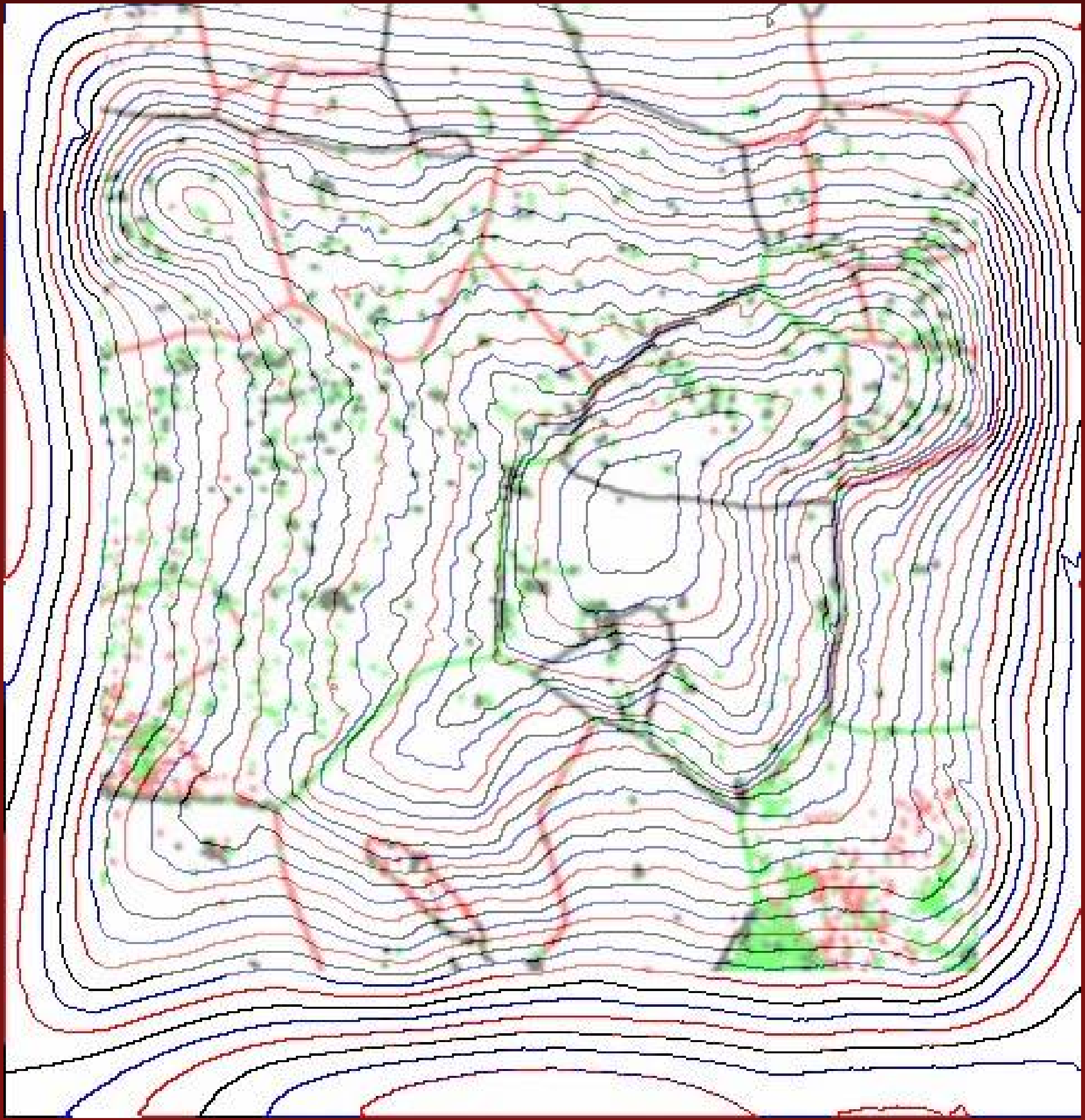
6K remanent MO image after 120 mT

Approximate 2D Current Streamlines

Current streamlines over GB map

Some GBs partially obstruct

Others benign

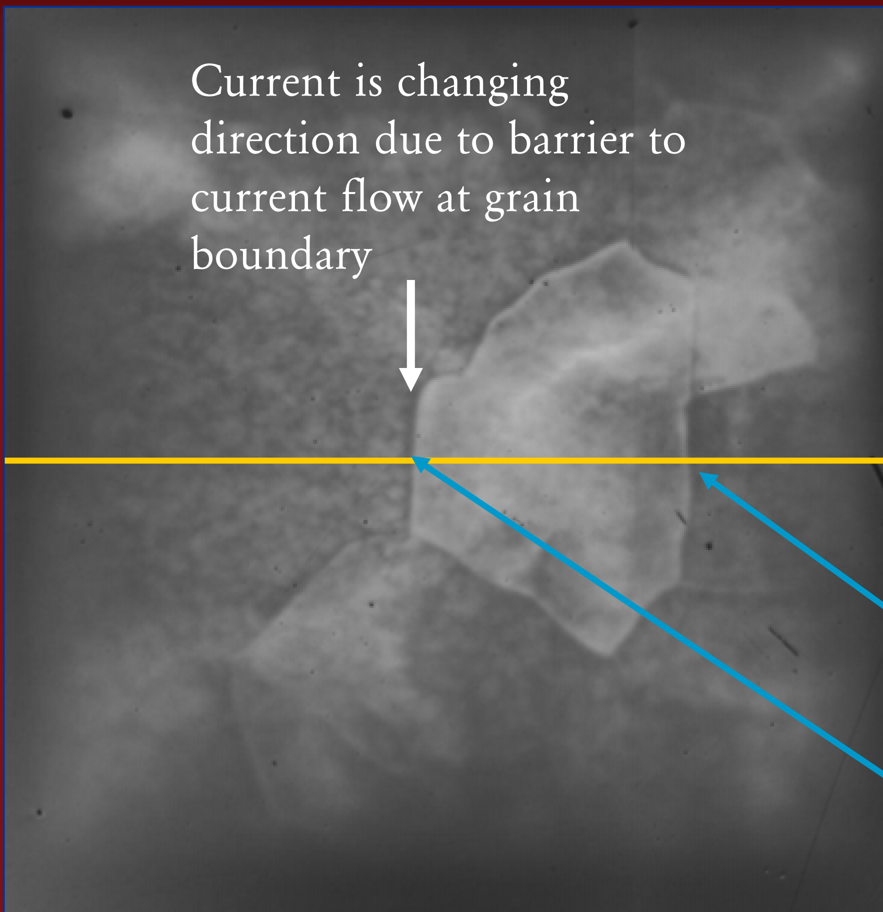


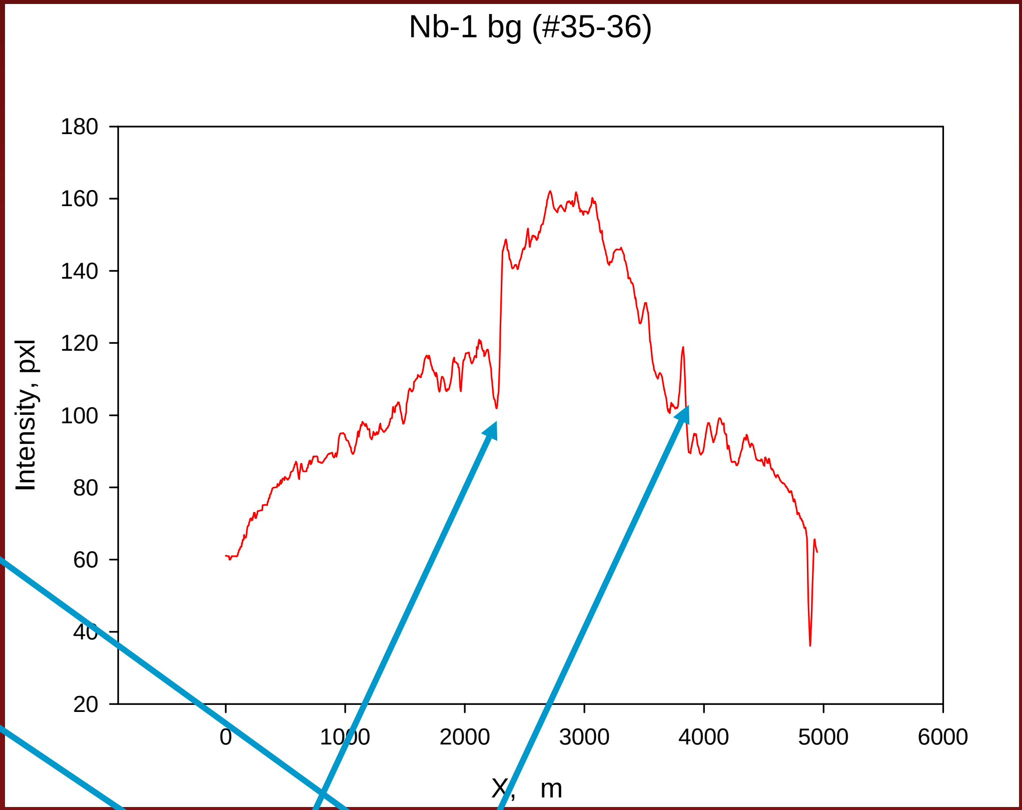
Green 3-20°

Red 20-50°

Black >50°

Flux profile of remanent image after magnetizing to 120 mT

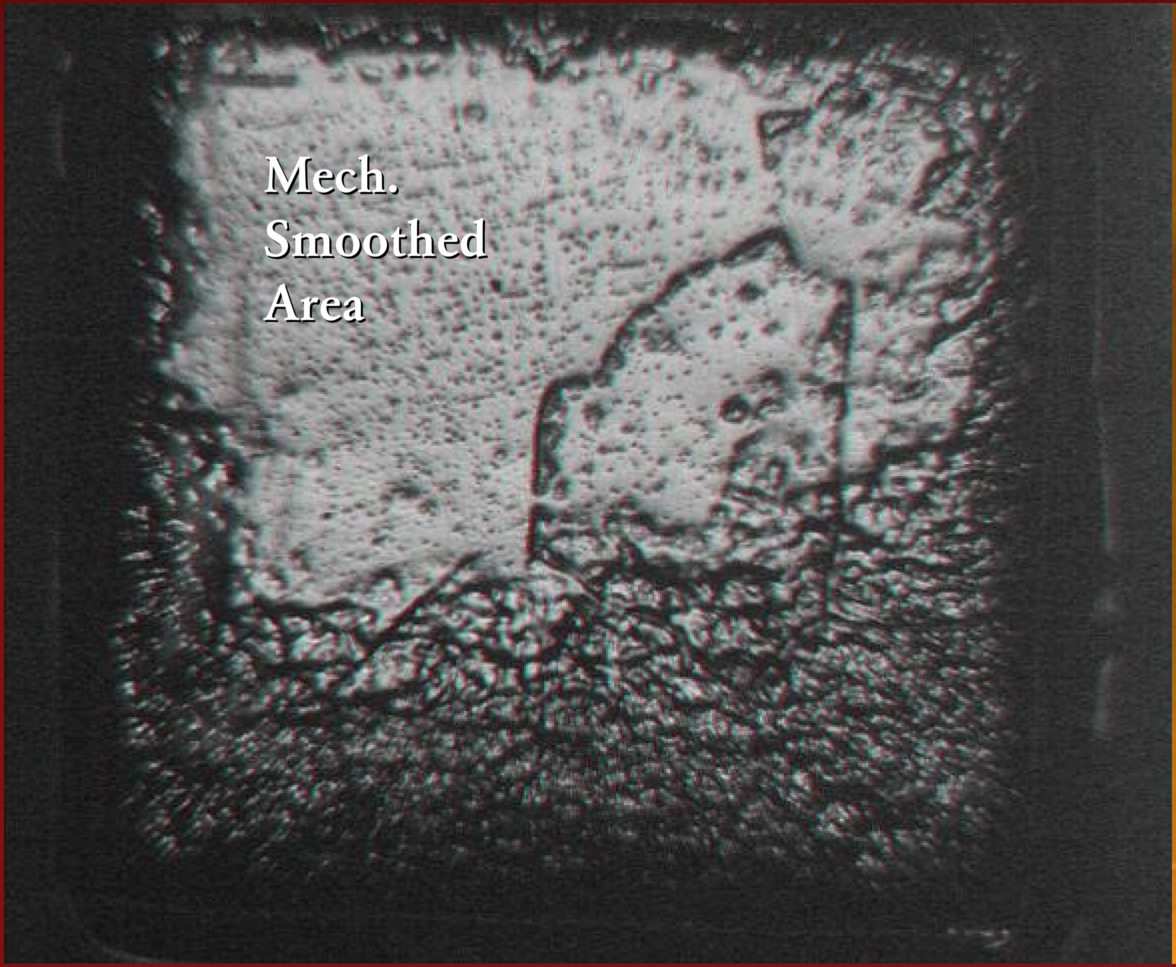




H=0 (FC in H=1200 Oe)

Reverse Gradient

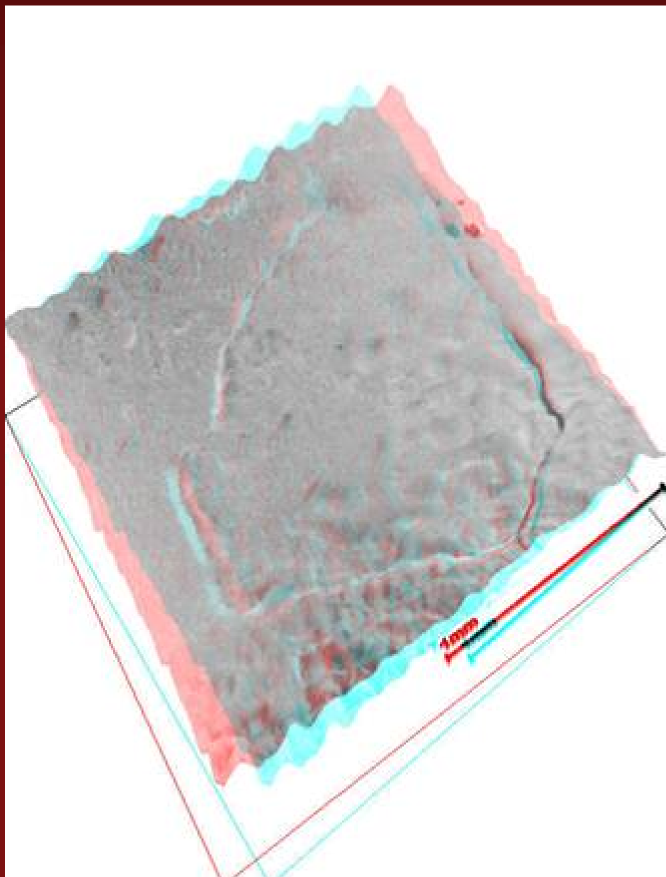
Now mechanically alter surface: Partial

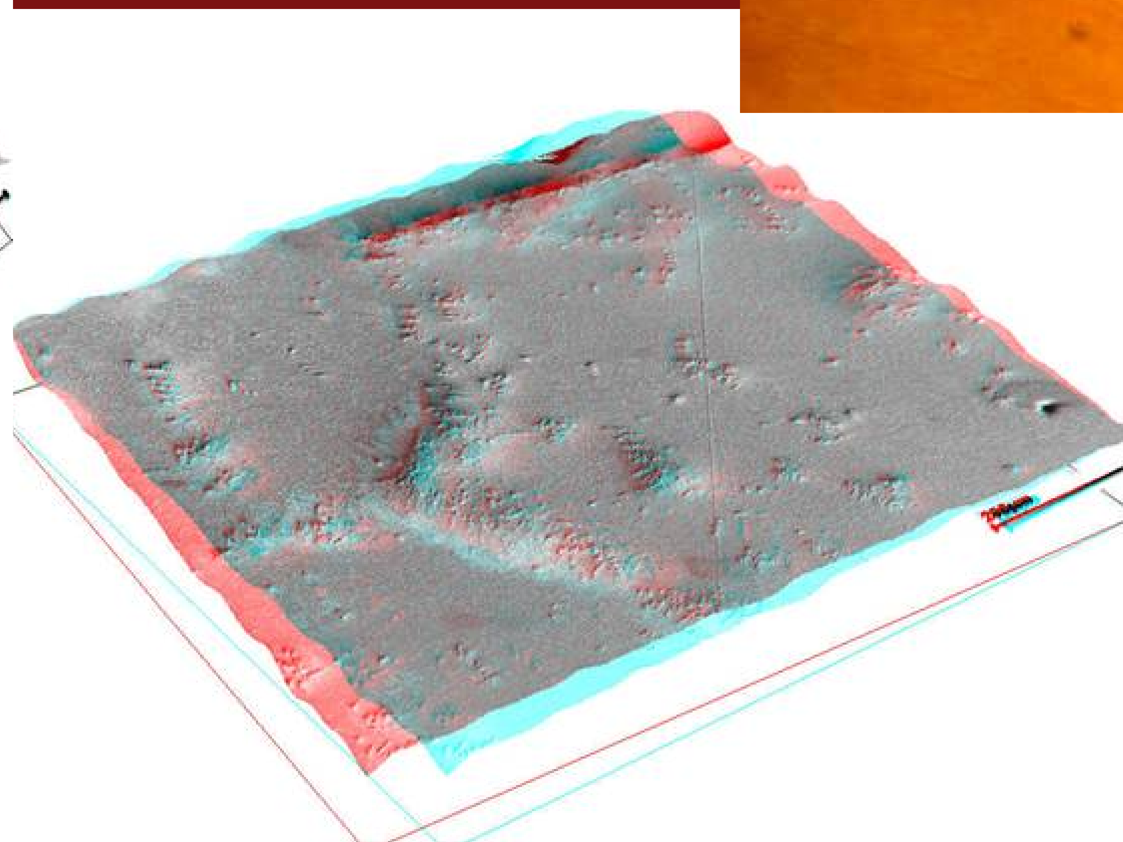


Light Microscope: EDF 3D reconstruction

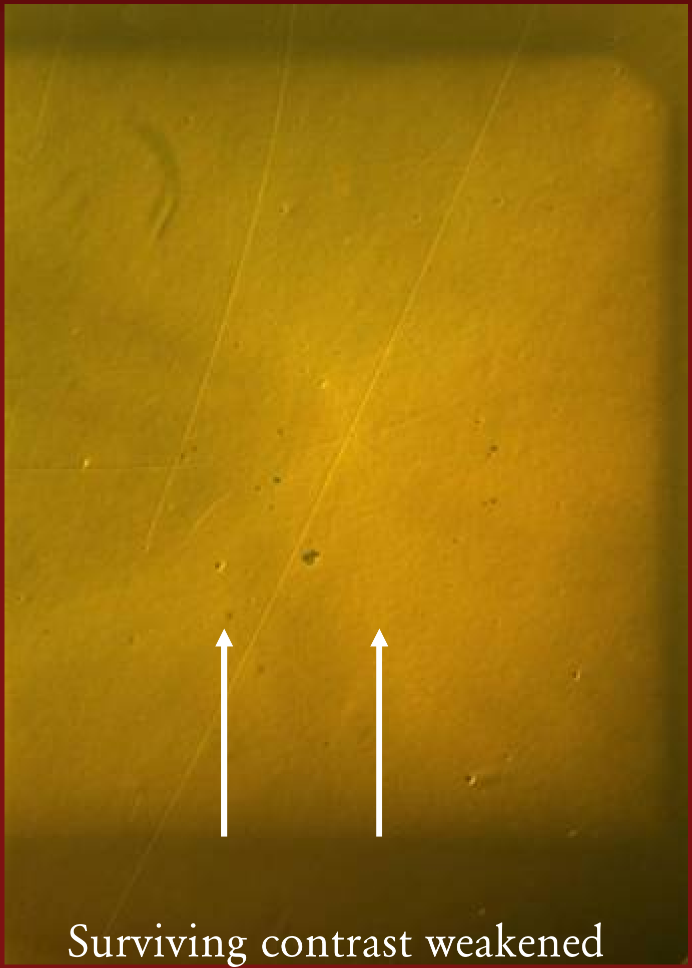
Remanent Field after 120 mT at 7K

½ Polish: FESEM reconstruction



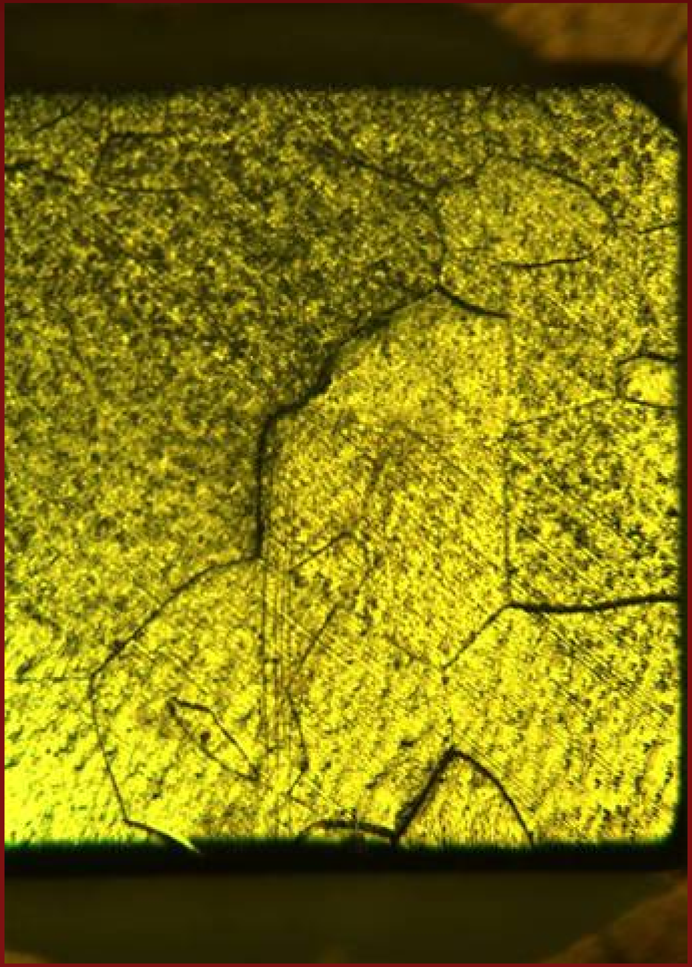


Complete Mech. Smoothed Surface



Remanent Field after 120 mT at 7K

Mechanically Smoothed Surface removed by Etch

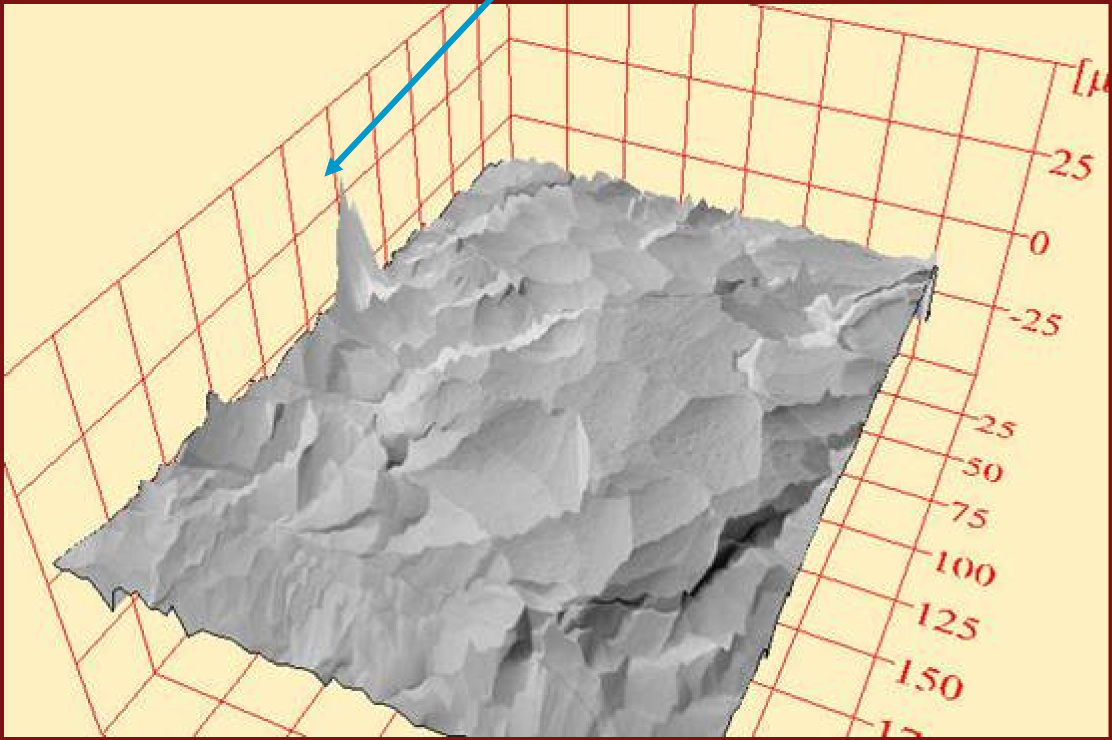


Remanent Field after 120 mT at 7K

Alternative Profilometry II:

- STEREO module of "AnalySIS" image analysis

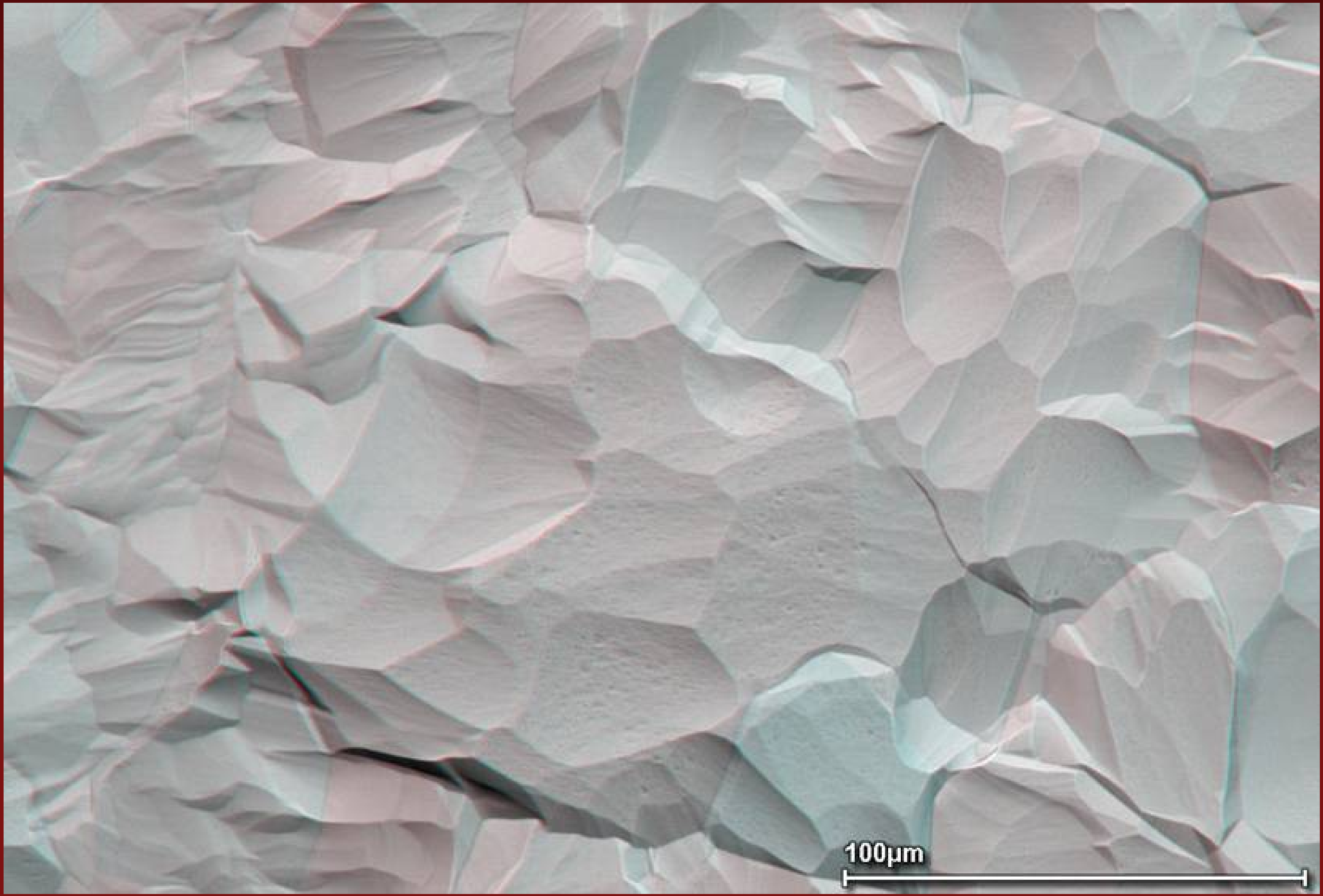
Edge Artefact



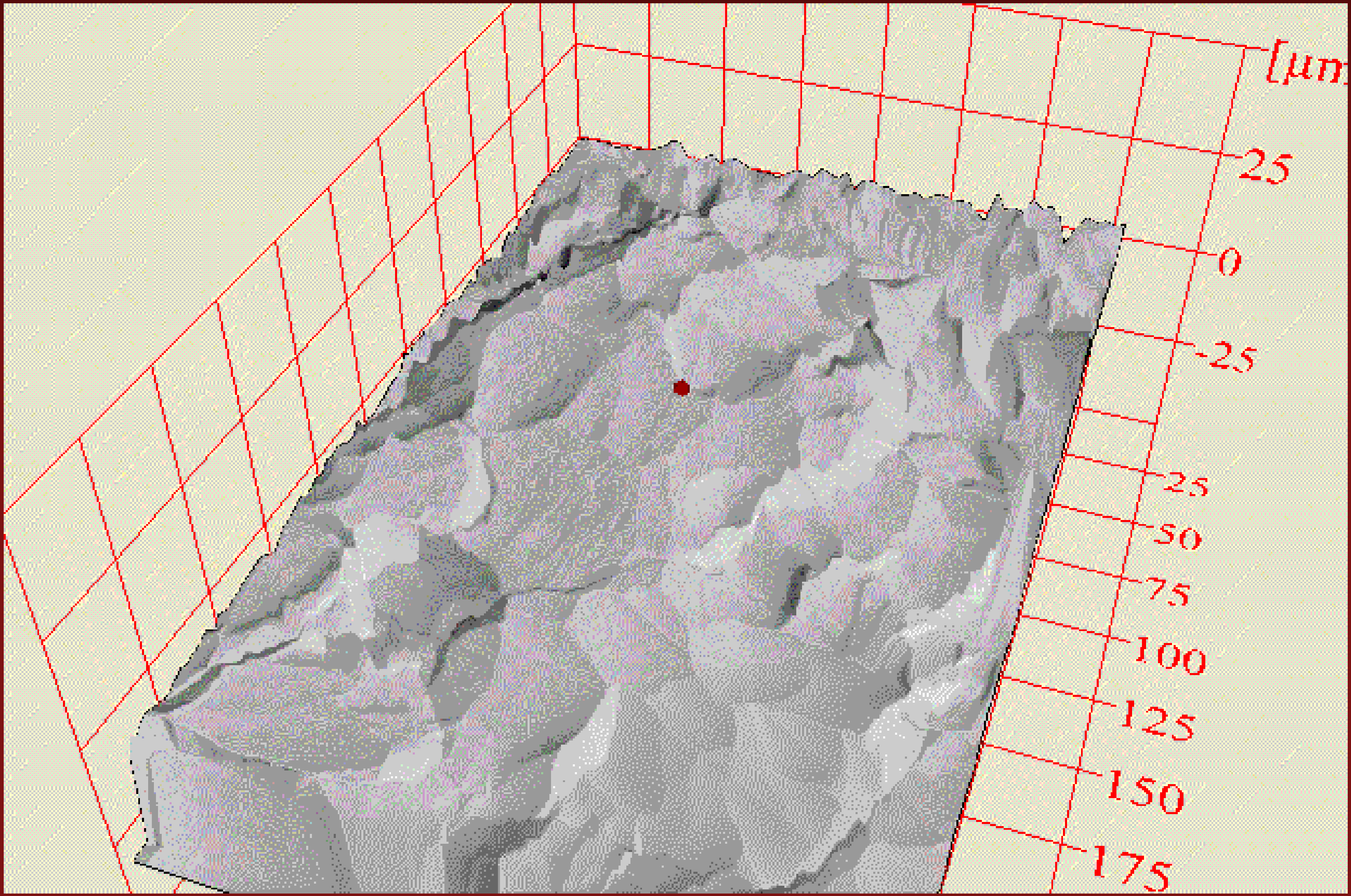
Demo analysis performed by Andrew Cahill

Soft Imaging System Corp.
12596 W. Bayaud Ave.
Suite 300
Lakewood, CO 80228
web: www.soft-imaging.com
email: aca@soft-imaging.com

Original Stereo Anaglyph used for analysis



"AnalysSIS" analysis



Summary

- Magneto-Optical Imaging shows that non-uniform flux penetration can occur along some grain boundaries
  - Is this topological or chemical or both?
- Considerable variation in surface topology observed
  - Local inhomogeneity: Orientation of facet surfaces
  - Grain size variation across weld region
  - Grooved grain boundaries in weld region and grain-growth heat treatment.
- Software tools enable quantification of surface topology over large areas.

Appendix I: Sample Summary

Sample	Clean	Etch 1	HT	Etch 2	RRR
RFNB_BULK1_**Weld**_W_HT1 _**RRR**_1,2,3 Taken from **WELD**	2x(US-M90+UPW rinse until 12MΩ) dried in lam N2	BCP 1:1:2, 100 min, rinse in UPW ~15-18 C	800 C 5 hr 10^{-6} T	BCP 1:1:2, 20 min, rinse in UPW T=17.5C	~350
RFNB_BULK2_**BCP2**_S_HT1 _**RRR**_1,..,10 Reg. sheet, EDM cut	2x(US-M90+UPW rinse until >10MΩ) dried in lam N2	BCP 1:1:2, 80 min, rinse in UPW ~15 C	800C 5hr 10^{-6} T plateau: 24hr @ 400C	BCP 1:1:2, 20 min, rinse in UPW ~15C	~400
RFNB_BULK2_**BCP2**_S_HT1 _**MO**_1,..,5 RFNB_BULK2_**BCP2**_S_HT1 _**MAG**_1,..,3 RFNB_BULK2_**BCP2**_S_HT1 _**BC**_1,..,4 RFNB_BULK2_**BCP2**_S_HT1 _**M**_1,..,3 Reg. sheet, EDM cut	—||—	—||—	—||—	—||—	—||—
RFNB_BULK2_**BCP2**_S_HT1 _**RRR**_? RFNB_BULK2_**BCP2**_S_HT1 _**MO**_?	Same as above + HT and etch to grow extra large grains		1250C,24hr,10^{-7} T (JLAB)	BCP 1:1:2, 50 min, rinse in UPW ~15C	260

Appendix II: Storage and Transport

All BCP2 samples are held in sterile PE tubes with dry, filtered N_2