Dislocation substructures, and low temperature N doping impact on flux penetration and trapping in SRF Nb

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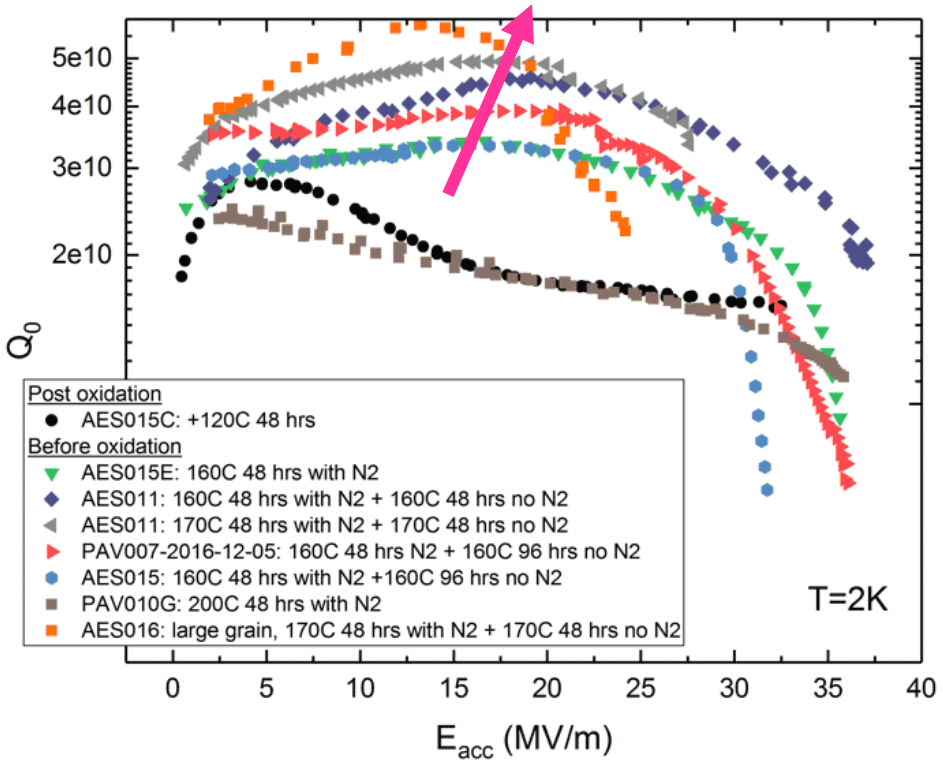
N2 Treatments have advanced SRF performance but at the expense of flux trapping



Grassellino et al., arXiv:1701.06077

- High Temperature N doped cavities: **High Q_0 at low accelerating gradients**
- **No 120°C bake needed**
- **But residual flux trapping on cool-down is an issue**
- Recently (2016): FNAL has reported both increase in Q and E_{acc} in cavities infused with Nitrogen at 140°C - 170°C. **No final EP required**
- Doping is here . . . and here to stay:
- Is it possible to obtain high Q_0 and high E_{acc}?
- What happens during the doping process? Surface composition…
- Which is better LG/FG? Role of boundaries and defects

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Our Goals

- **Characterization/Analysis of current cavity technology:**
  - Correlate metallurgical state and magnetic properties with strategically characterized samples
  - SRF relevant properties- Surface superconductivity(H_{c3}) , Surface pinning, defect classification
  - Comprehensive microstructural, chemical, and property evaluation of SRF cavity coupons/ SRF grade coupons
- **Develop coupon measurement techniques to:**
  - Explain and predict SRF cavity performance
  - Enable faster/cheaper testing of new materials, processing strategies, and techniques

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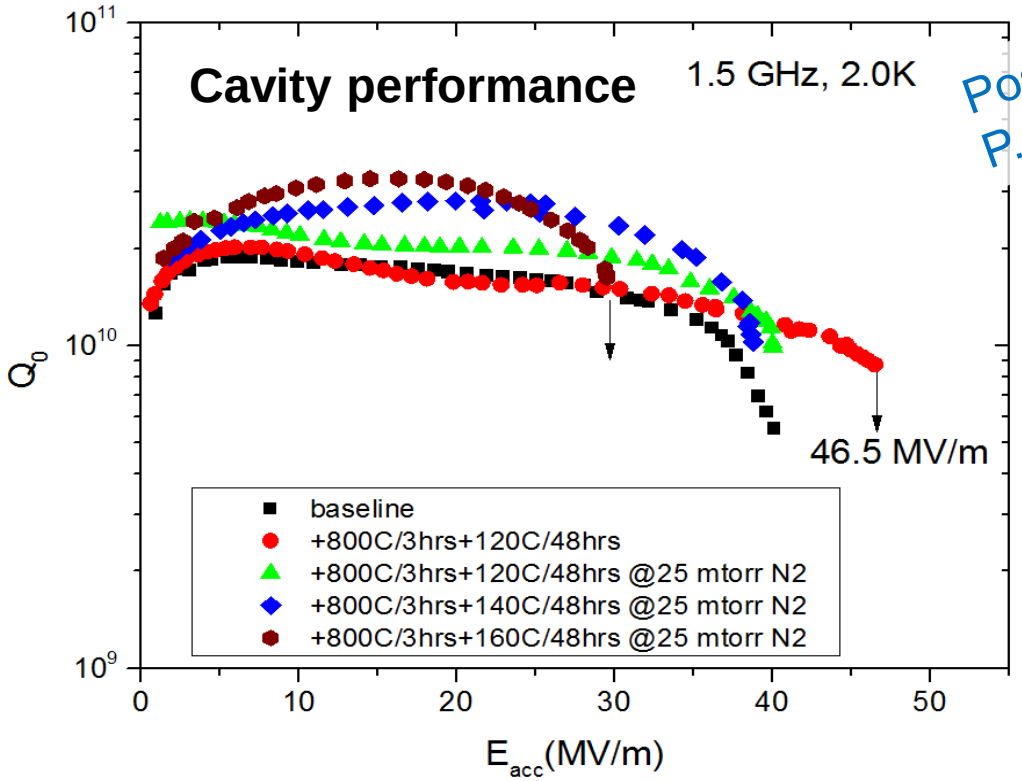
RDL-02 Cavity test results from JLAB



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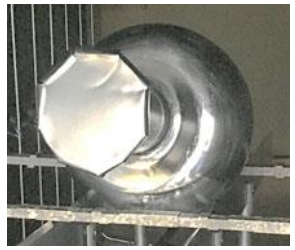

After HPR, cavity HT'ed in furnace with endcaps. ILC Recipe: 150 µm EP + 800°C/2hrs + 20 mm EP

Values estimated from fit to average $R_s(T)$

Test	Δ/k_BT_c	mfp (nm)	R_{res} (nΩ)
Baseline (ILC)	1.82 ± 0.02	164 ± 27	2.2 ± 0.1
+120 °C/48h*	1.86 ± 0.02	26 ± 7	4.6 ± 0.2
+800 °C/3h+ 120 °C/48h@ 25 mTorr N2	1.84 ± 0.02	28 ± 5	1.9 ± 0.1
+10 µm EP	1.81 ± 0.02	164 ± 23	1.2 ± 0.1
+800 °C/3h+ 160 °C/48h@ 25 mTorr N2	1.80 ± 0.02	54 ± 14	1.0 ± 0.1
+10 µm EP	1.79 ± 0.02	122 ± 16	0.5 ± 0.1
+800 °C/3h+ 140 °C/48h@ 25 mTorr N2	1.80 ± 0.02	26 ± 87	0.9 ± 0.1

No post chemistry after each furnace treatment.

Coupon samples also underwent the recipes shown and the same color-scheme will identify the HT

Cylindrical sample for magnetization

Flat sample for XPS and SEM

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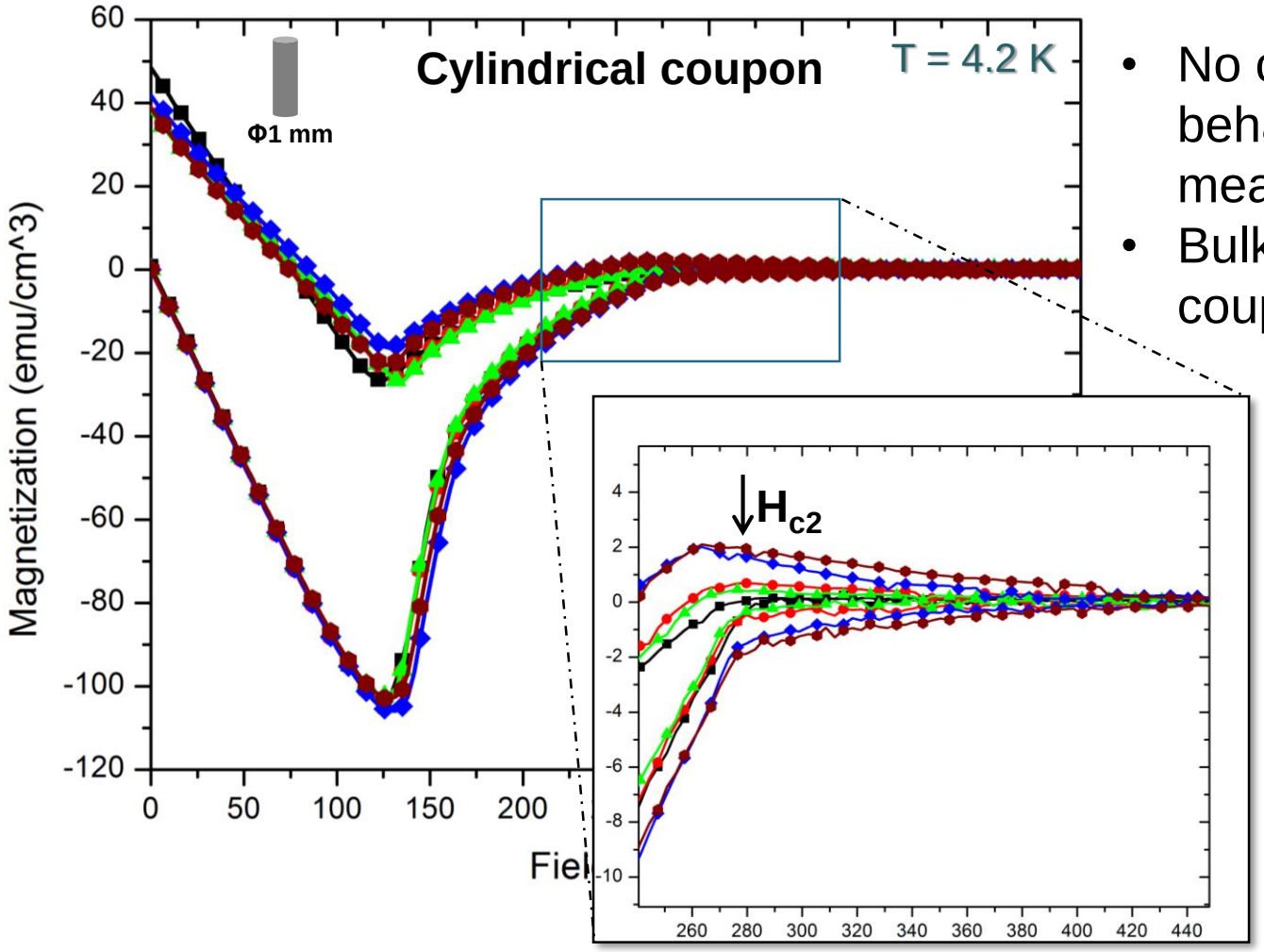
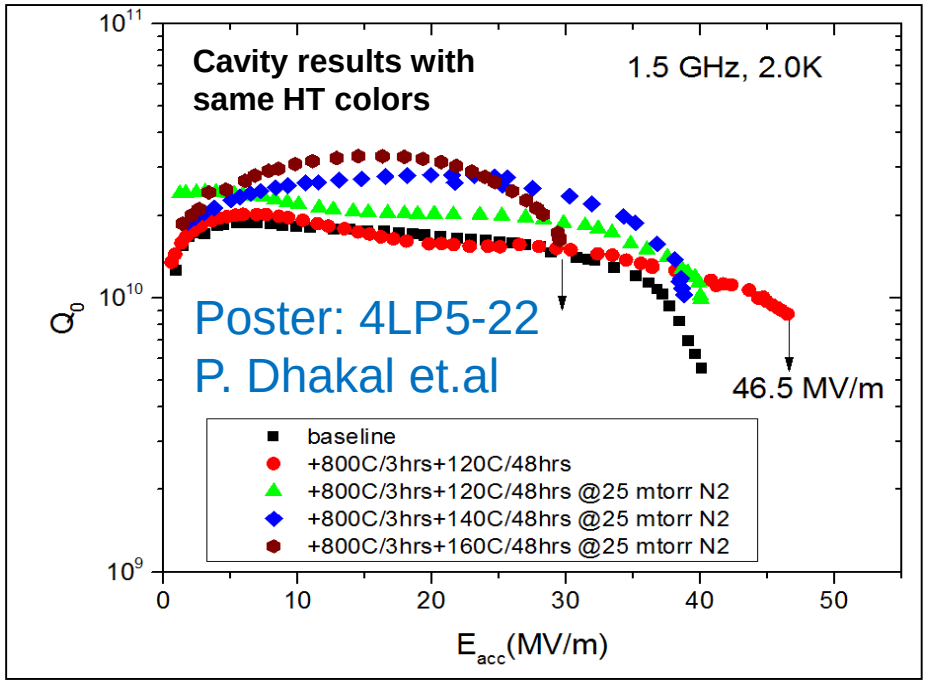
Coupon DC magnetization Result: No changes in bulk behavior observed with low temperature N infusion



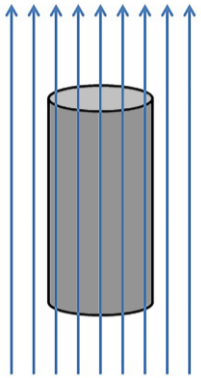
- No clear variations in early flux penetration behavior observed by DC magnetization measurements
- Bulk pinning characteristics are similar in all the coupon cylinders analyzed

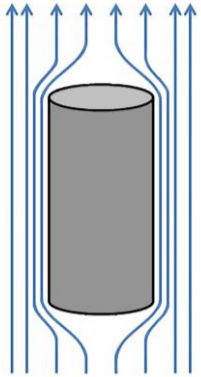


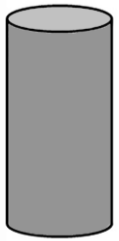
Measured in 5T QD SQUID

Coupon Flux Trapping Method: Magnetization measurements

1 mm diam.
3 mm long
Nb cylinders







The samples were field cooled (1 mT) to below T_c (8.5 K) of Nb. Cooling rate: 5 K/min. Then the field was removed

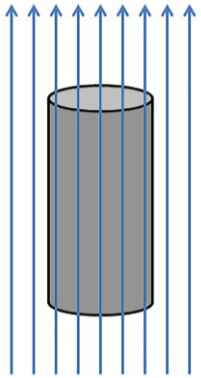
M = 0

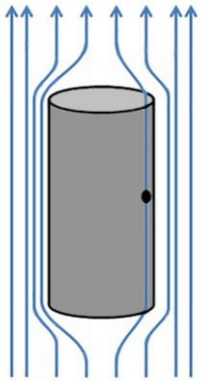
T = 12 K >9.2 K(T_c)
B_{ext}= 1 mT

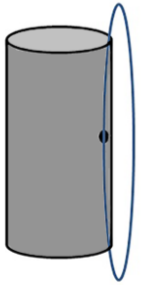
T=8.5 K< 9.2 K(T_c)
B_{ext} = 1 mT

T=8.5 K< 9.2 K(T_c)
B_{ext} = 0 mT

Flux Trapping



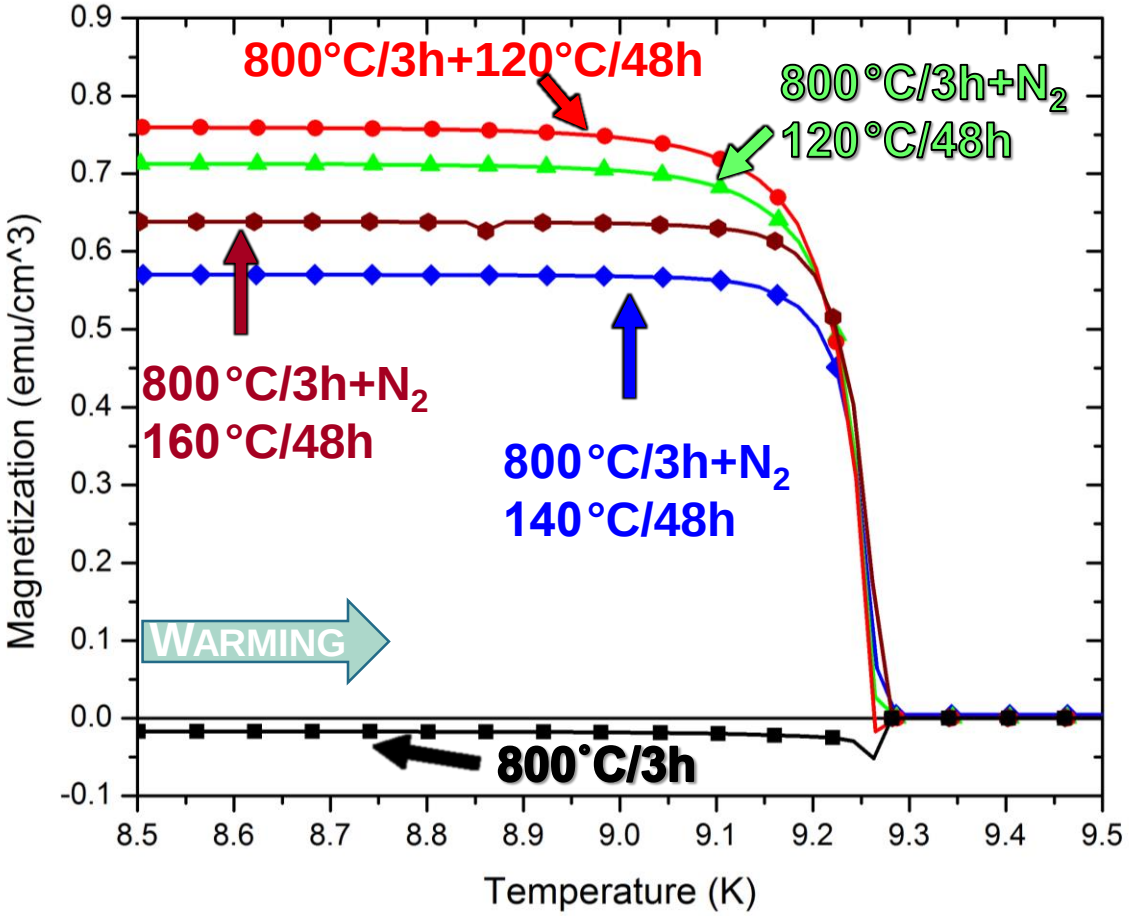




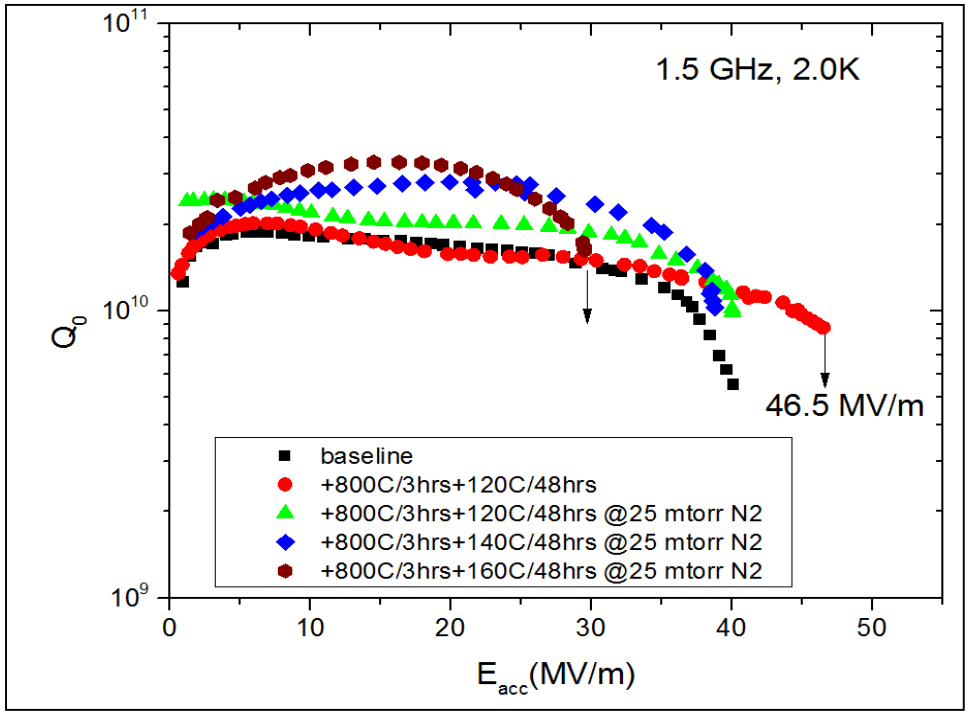
Any magnetization observed under this condition is due to the trapped flux

M ≠ 0

Result: Baking causes incomplete flux expulsion with or without low temperature N infusion during bake



- **No** DC flux trapping in **800°C/3h** coupon cylinder
- Lowest flux trapping and best overall Q in 140°C/48h+N infused sample



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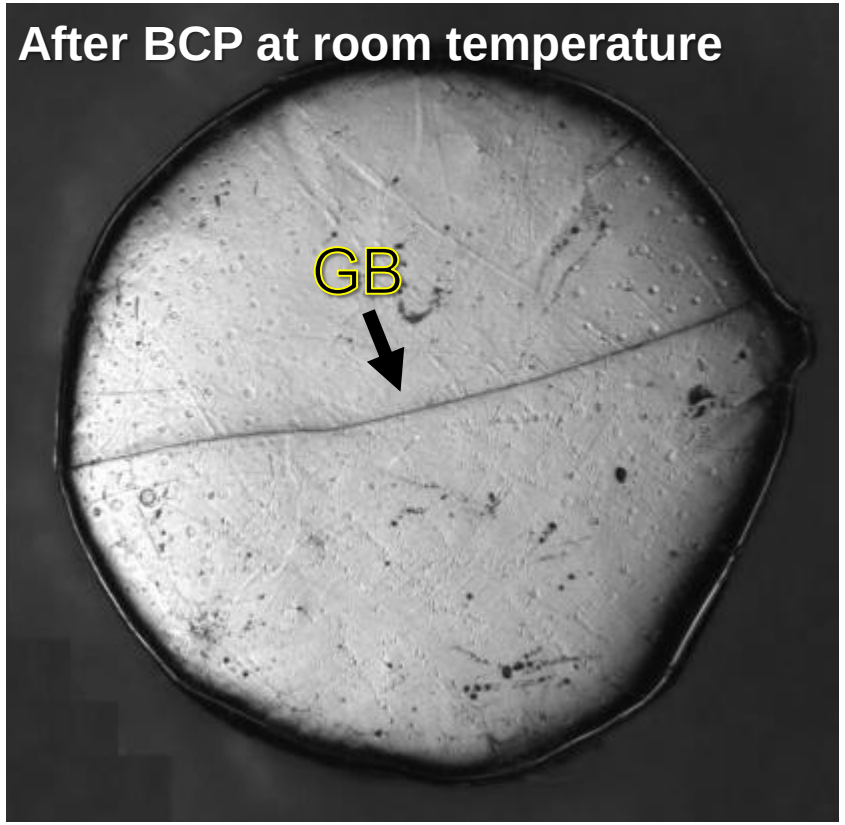
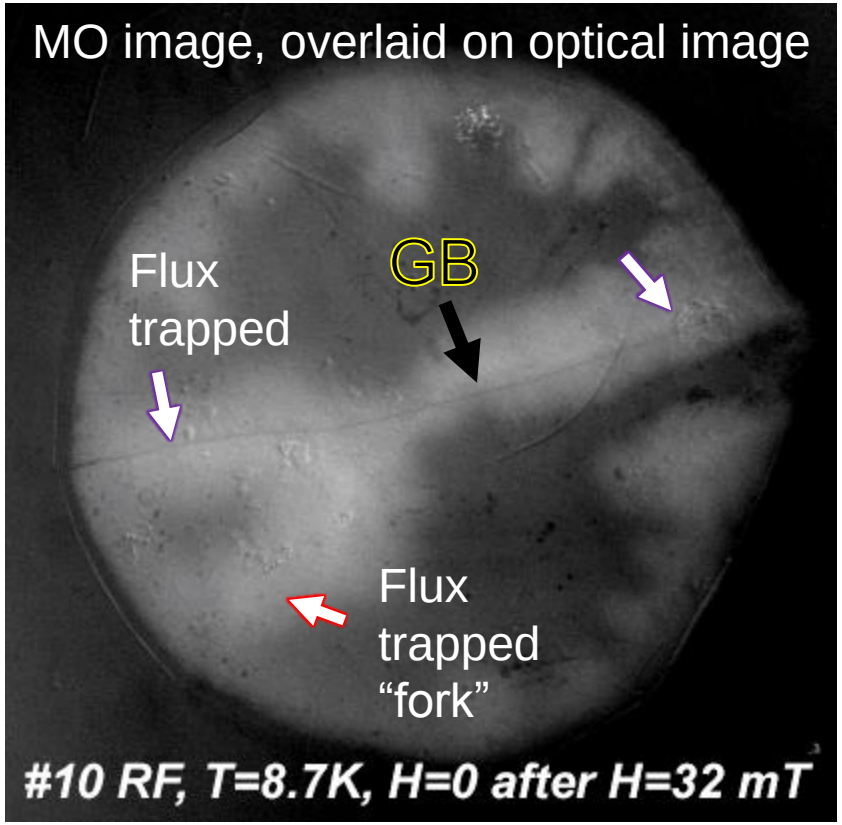
Magneto optical (MO) imaging of disk: Flux trapping is a bulk effect with microstructural correlations



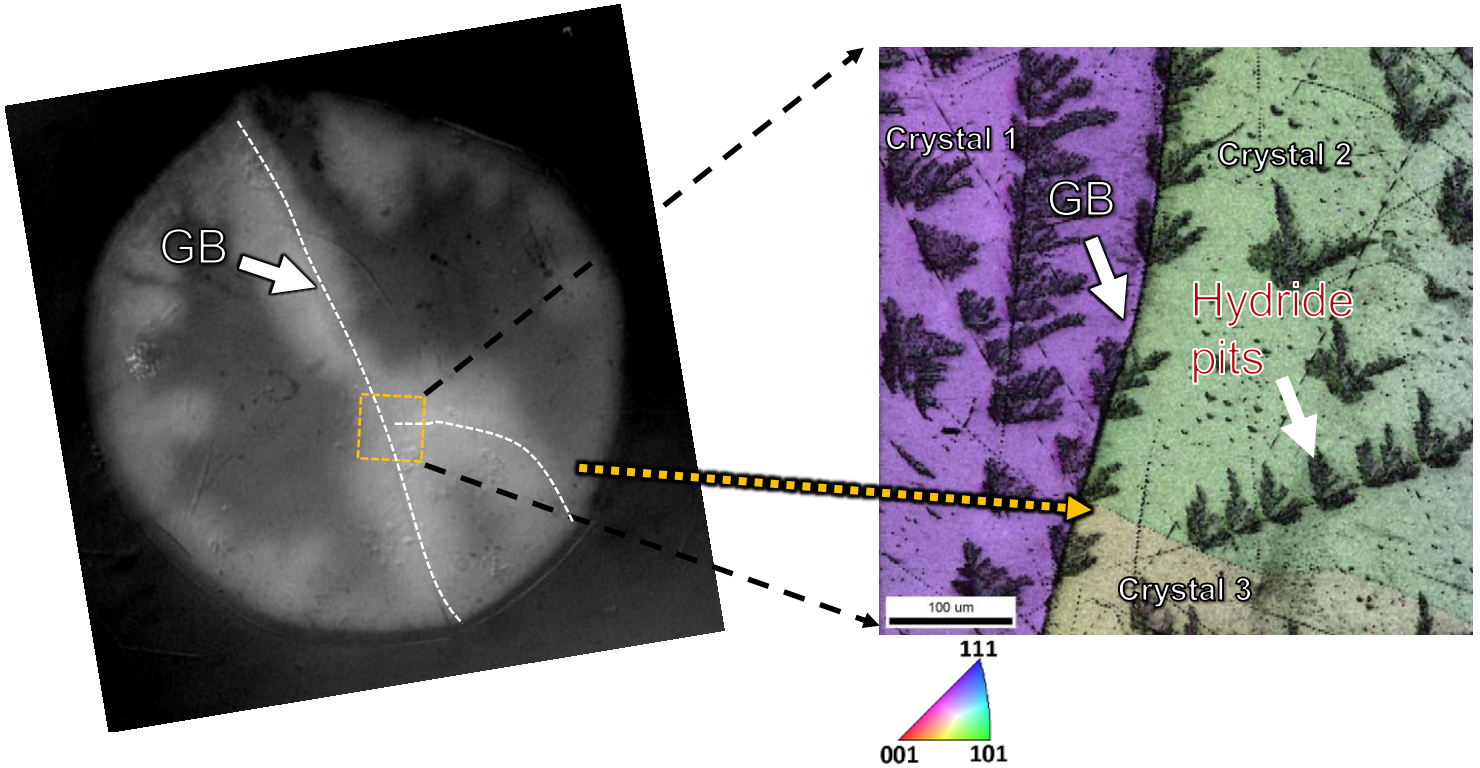
3 mm diameter: EDM + BCP of **as-received Ningxia sample**



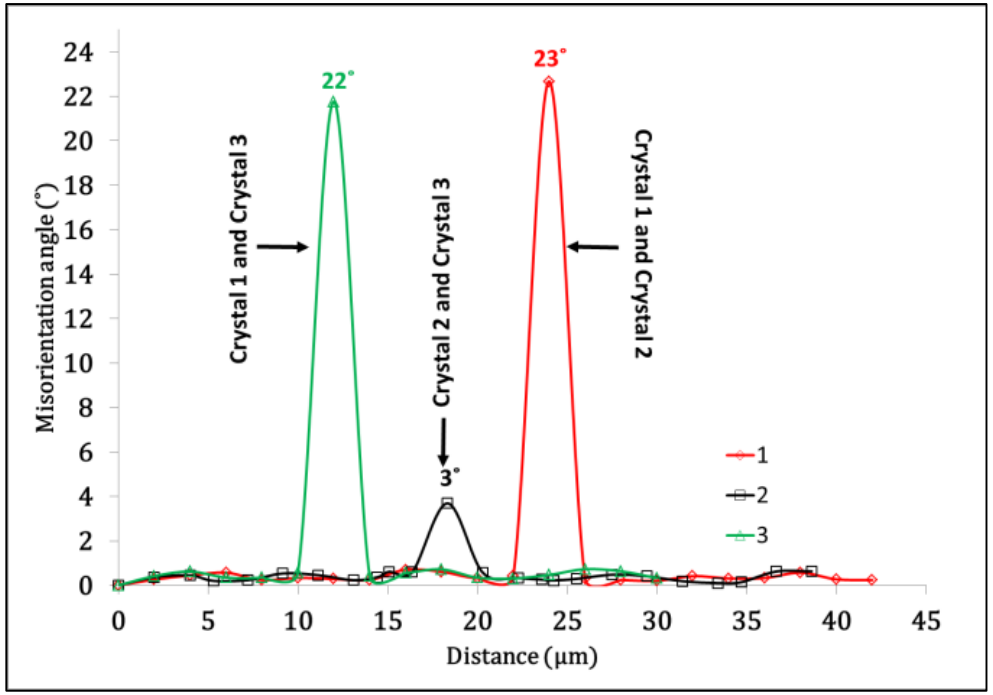
Applied field H points out of plane

- GB oriented normal to the applied field causes flux penetration (known)
- The fork in the sample was unexpected

Magneto optical: DC flux trapping can occur due to low angle boundaries in SRF Nb

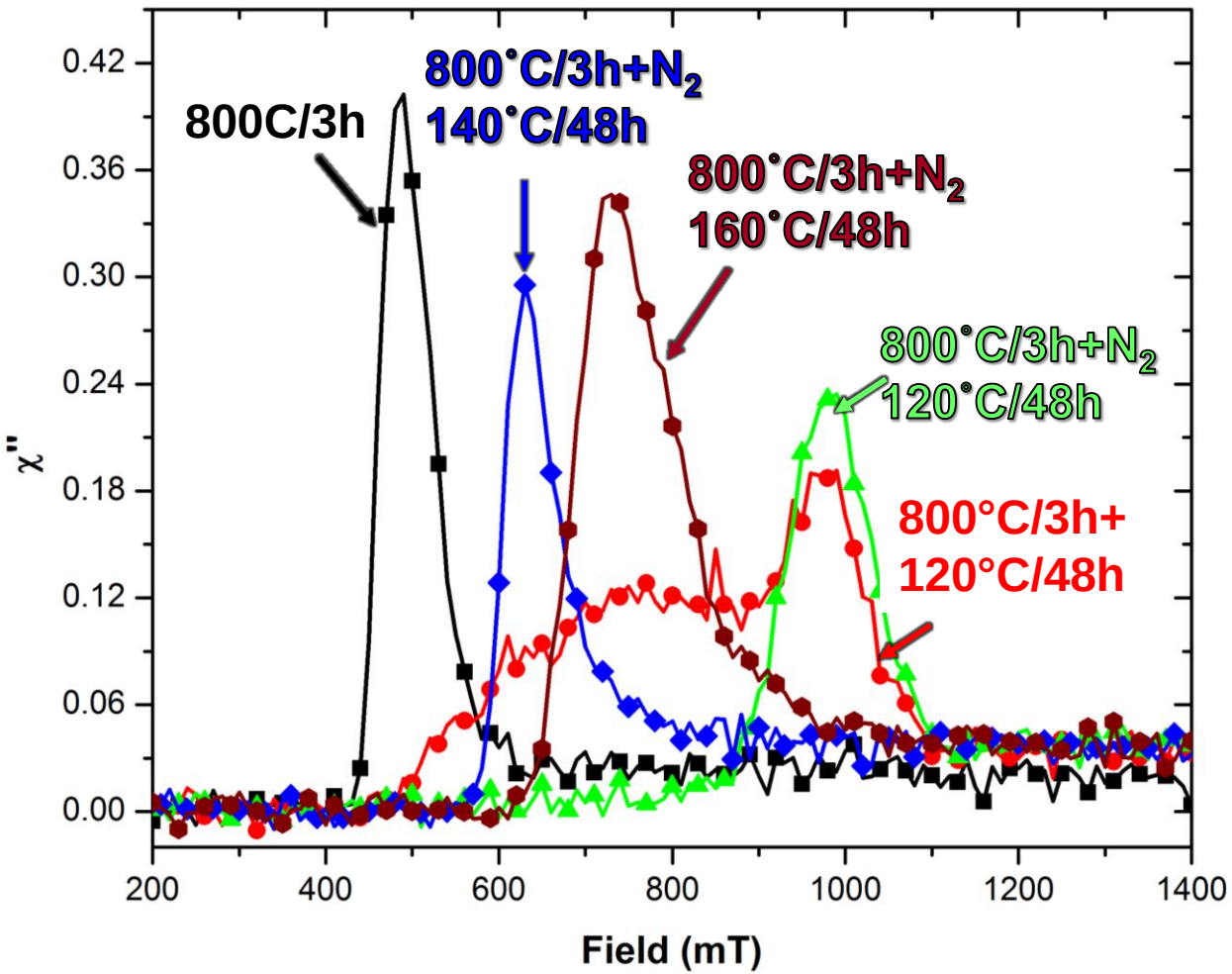


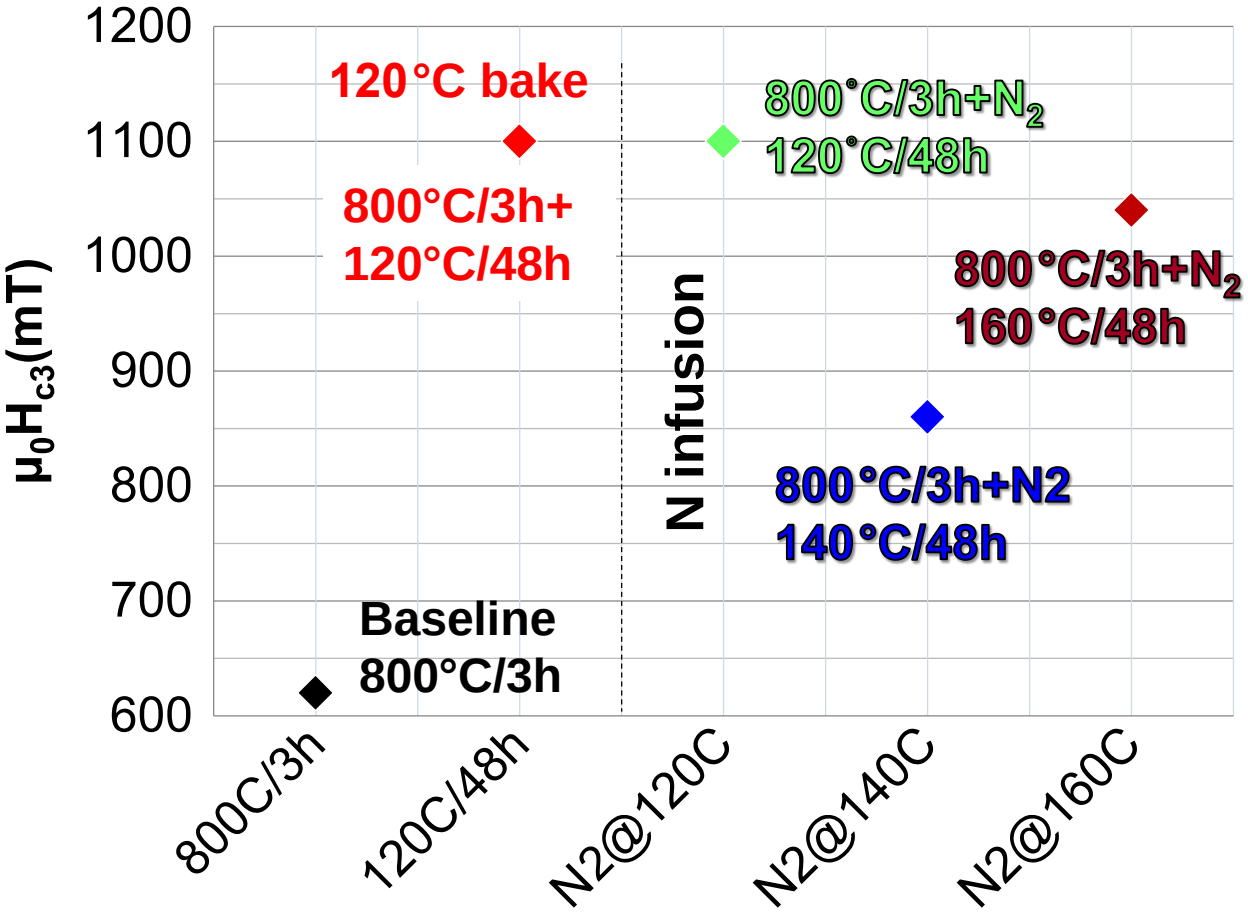
RF, T = 8.7 K, H = 0, after H =32 mT



- Misorientation measurements across boundaries indicates a low-angle boundary with a misorientation of 3°
- Identifying these boundaries are a challenge

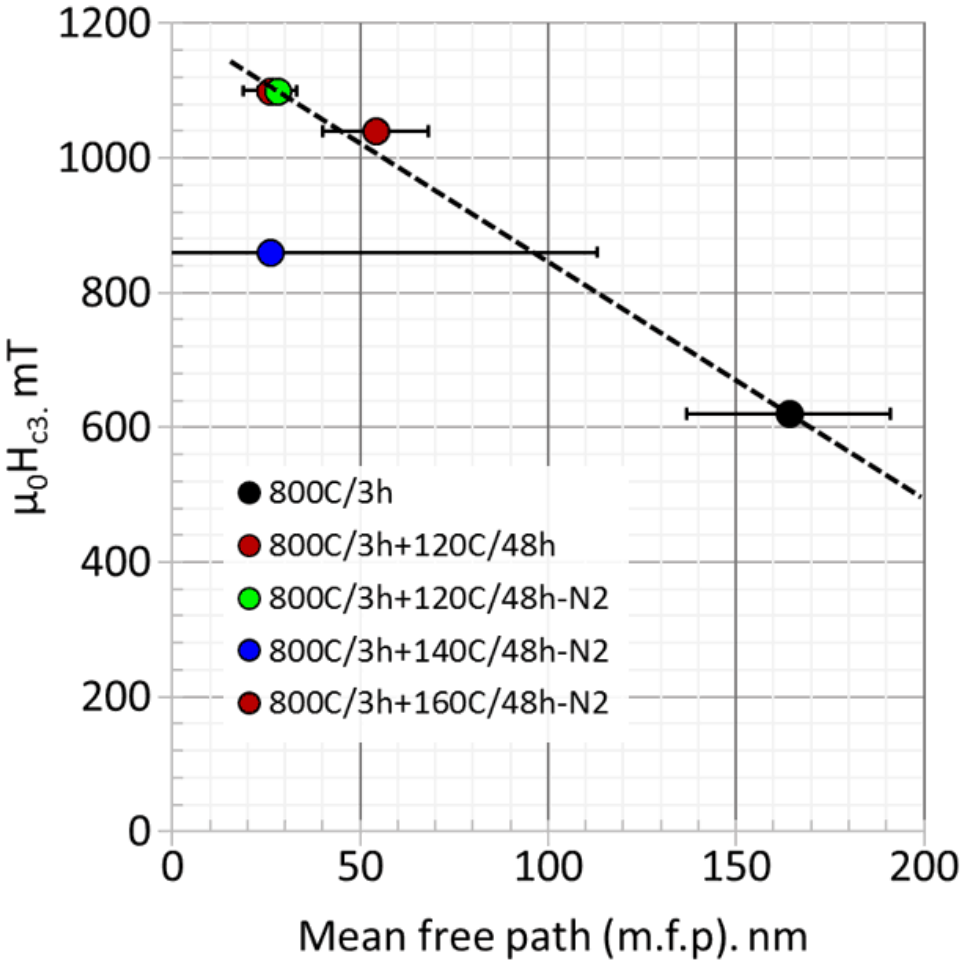
AC Susceptibility: Higher surface superconductivity (H_{c3}) in low temperature baked Nb samples with and without N infusion





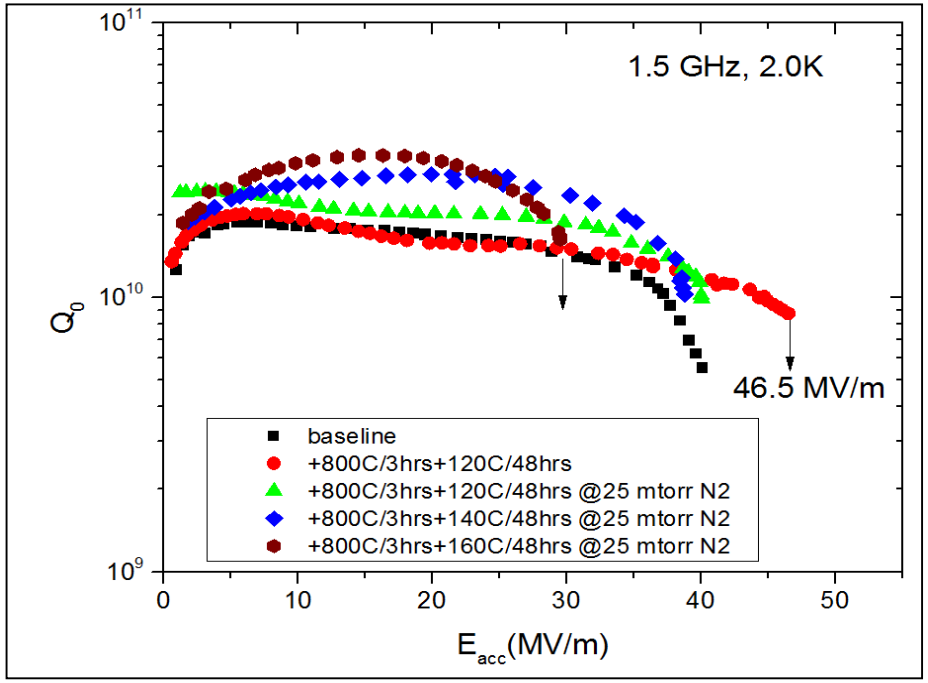
AC amplitude= 0.1 Oe, frequency = 100 Hz, T = 4.2 K

H_{c3} coupon tests correlate with mean free path measured in cavity tests



mfp estimated from cavity $R_s(T)$ fit

- H_{c3} value increases linearly with decrease in surface mean free path
- First direct correlation between coupon test and cavity test



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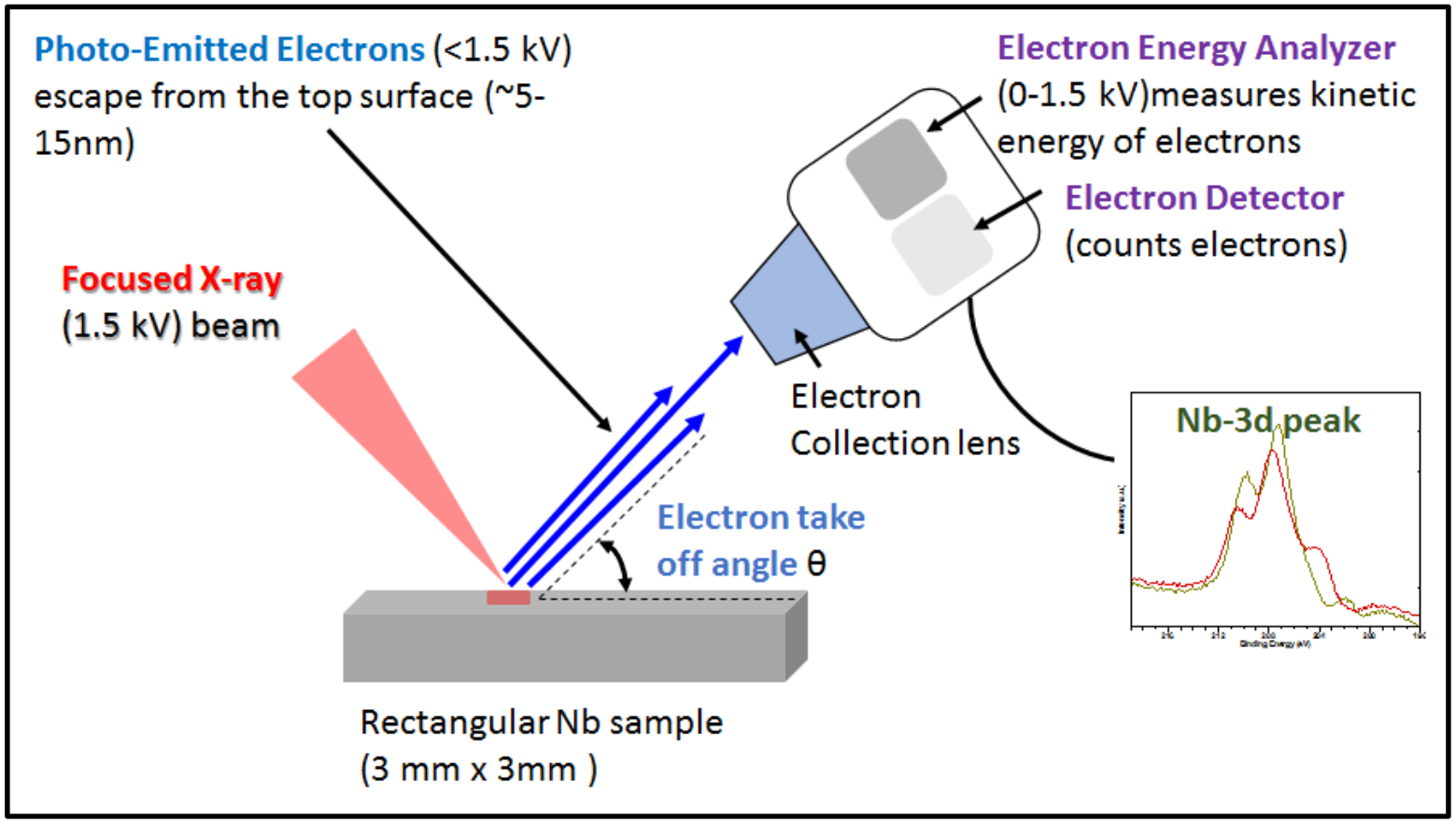
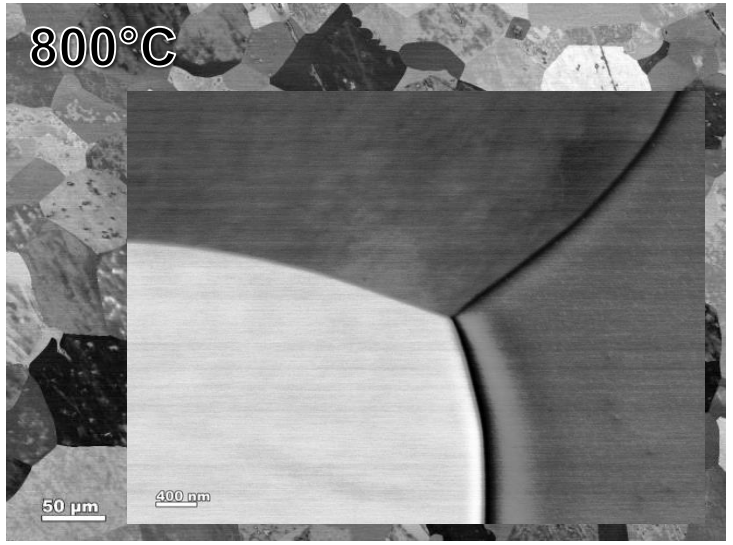
Surface Chemistry method: Angle resolved XPS

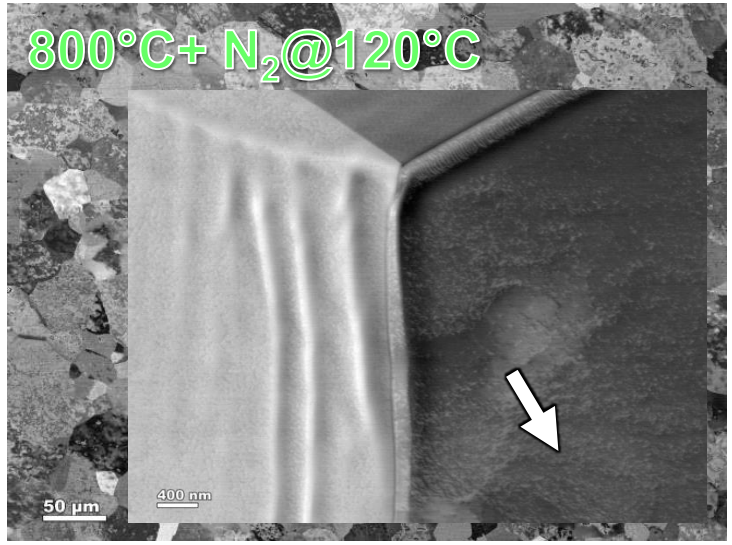


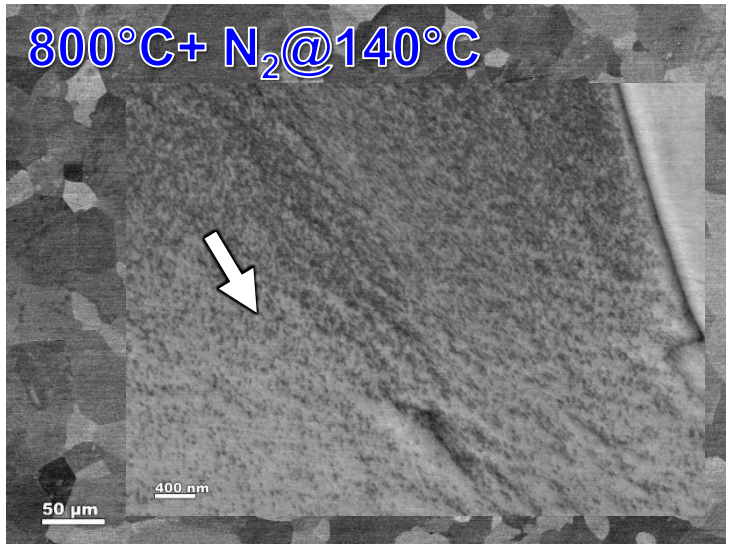
- Identification of surface chemical composition possible
- Angle resolved XPS (θ) is able to provide depth profiling
- Lower take off angles (θ↓) graze the surface- Majority signal from near surface
- Higher take off angles (θ↑) more averaging possible

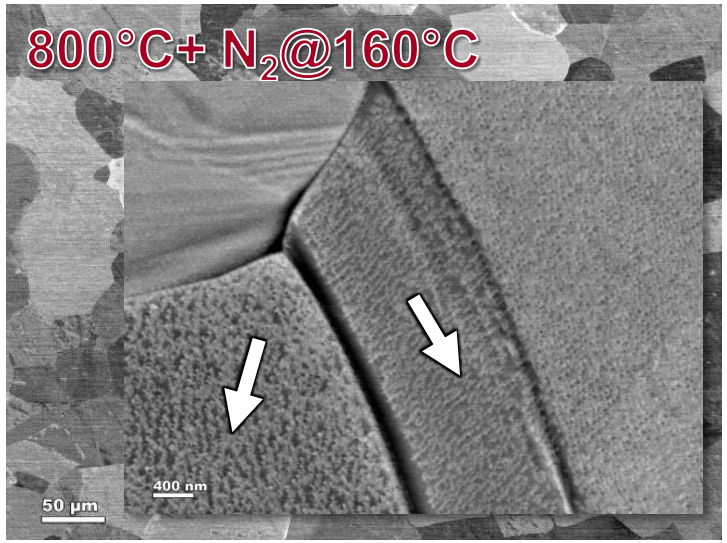
FESEM: Backscattered electron images indicate granular features in N infused samples

- Back scatter imaging reveals surface sensitive strain and phase contrast
- Granular features appear in low temperature N infused samples









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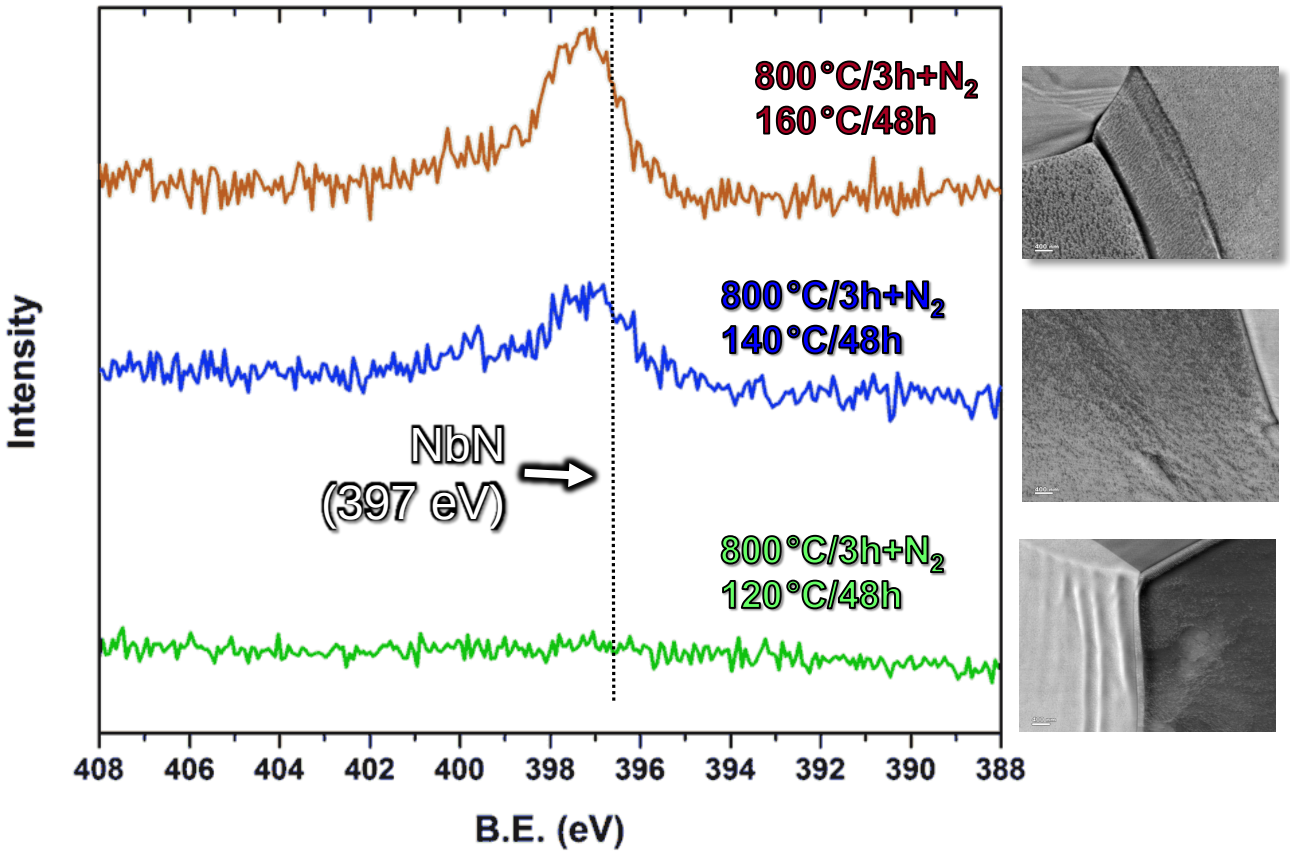
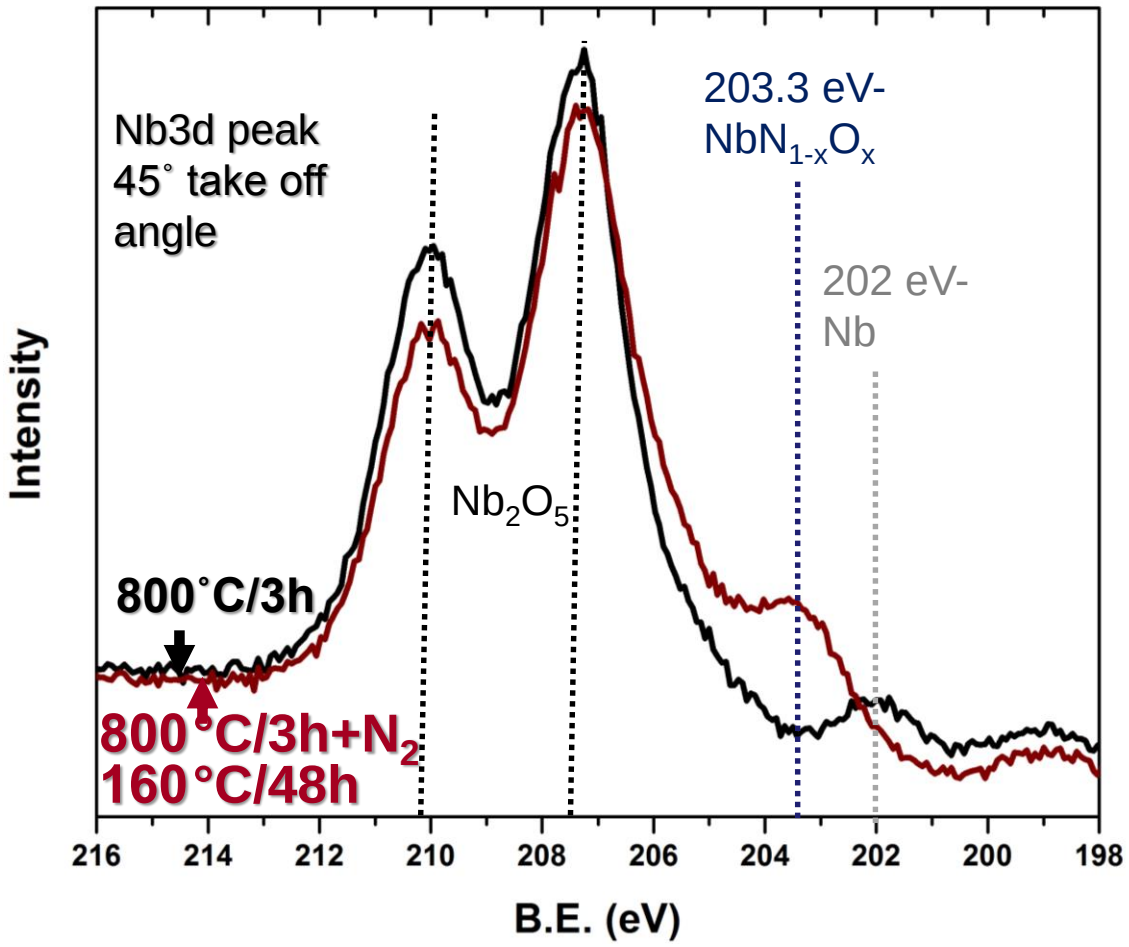
Surface analysis by XPS: Presence of nitrogen in the form of nitride detected



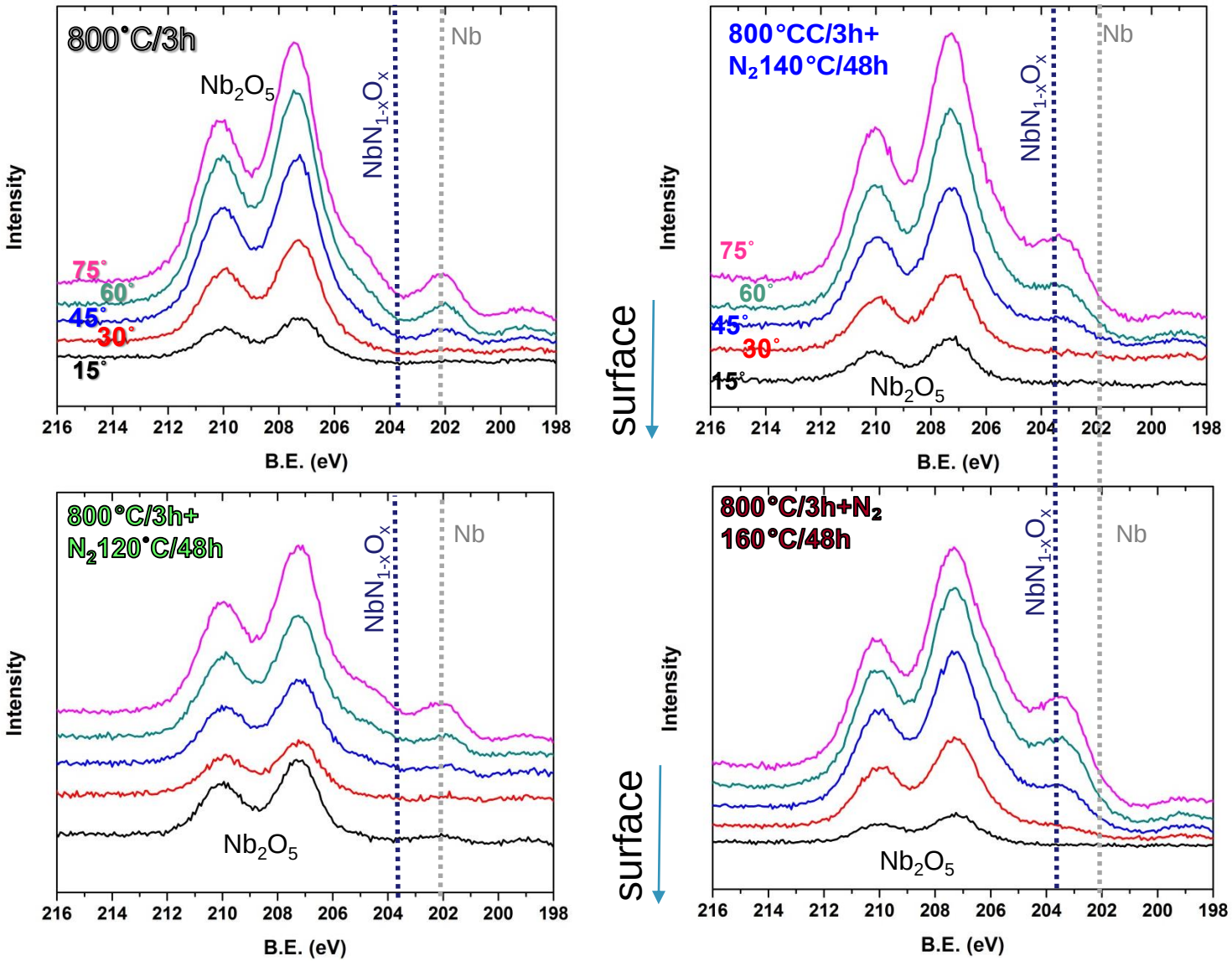
- Nitrogen peaks detected in samples after 140°C/48h, 160°C/48h N infusion treatments but **not** for the 120°C/48h N
- The granular phase contrast could be indicative of a Nb nitride phase

XPS Result 2: presence of NbN1-xOx in N infused samples



- Surface composition differences between samples exists after N infusion
- N infused Nb has a layer of $NbN_{1-x}O_x$ (Nb oxy-nitride)
- Nb signal appears in sample with no N infusion, but is absent in the N infused sample, indicating thickness of the oxy-nitride layer

Angle resolved XPS: Nb-3d peaks indicate significant Nb oxy-nitride after 140°C, 160°C, N infusion



- Increase in take off angle indicates more signal from the bulk than surface
- $NbN_{1-x}O_x$ forms below Nb_2O_5 for N_2/140°C and N_2/160°C
- But no $NbN_{1-x}O_x$ signal for the N_2/120°C sample

Summary and Conclusions

- **Systematic examination of coupon samples provides methodology to relate surface superconducting behavior with cavity performance**
  - First direct comparisons between cavity test and coupon samples presented
  - H_{c3} from coupon measurements correlates well with mfp calculations from cavity tests
  - High fidelity technique- Sample surface is largely unmodified during these investigations
- **Flux trapping observed in low temperature N infused coupon samples**
  - Bulk magnetization measurements show no significant difference in early flux entry in N infused samples compared to baseline measurements
  - N infusion could possibly enhance flux penetration, and trapping
- **Nb oxy-nitride ($NbN_{1-x}O_x$) present in N infused coupon samples**
  - Nitride presence in the form of oxy-nitride is detected in 140°C, and 160°C coupon samples
  - Nb-NbO interface is not detected in the probed volume, suggesting $NbN_{1-x}O_x$ is thicker than ~10 nm
  - Magnetic properties of oxy-nitrides are unknown…

Thank you!

Acknowledgements

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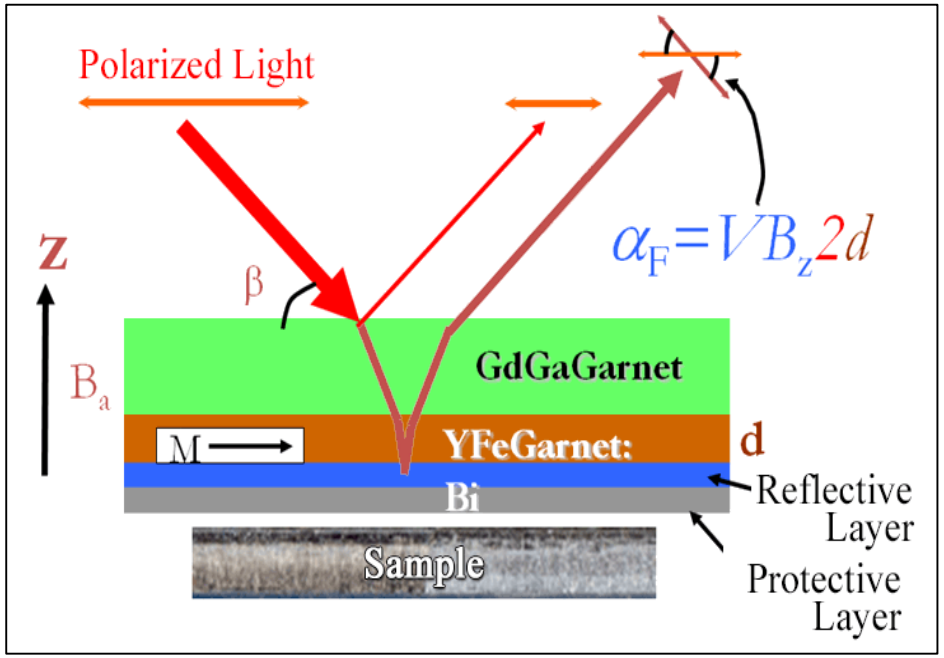
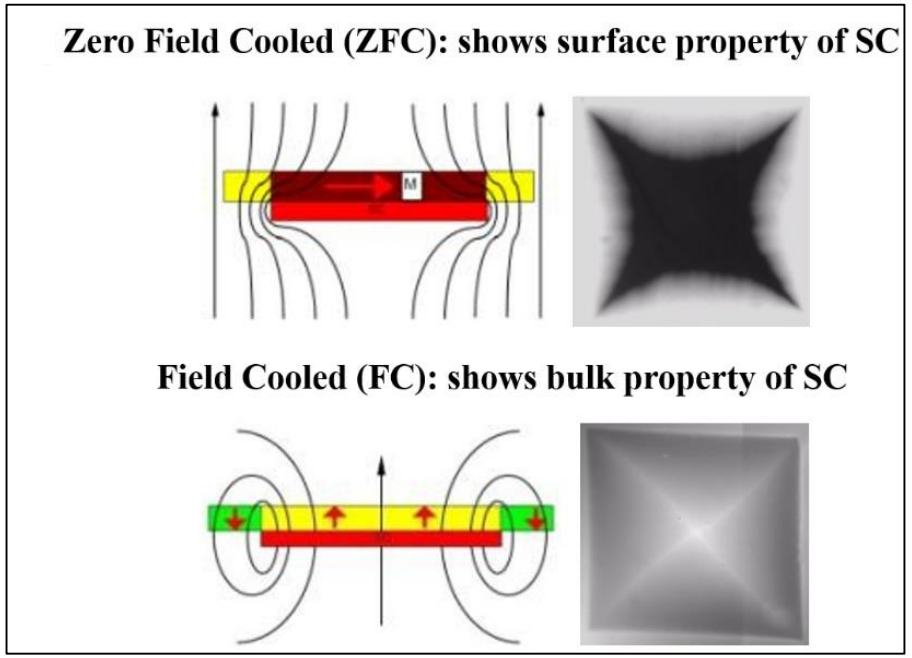
Bonus slides

Flux penetration observations in SRF Nb by Magneto Optical Imaging



MOI Schematic and principle

- Allows direct Imaging of Bz in plane above superconductor
- Double Faraday effect occurs in reflective mode using Bi-doped YFe indicator film with in-plane magnetization



ZFC: Cooled in the absence of external fields to below T_c, at $T < T_c$ visualized in an external magnetic field.

FC: Sample cooled below Tc in the presence of a magnetic field, and visualized after removing the external field