Improved understanding of the role of strained structure in degradation of surface superconductivity of superconducting RF quality niobium

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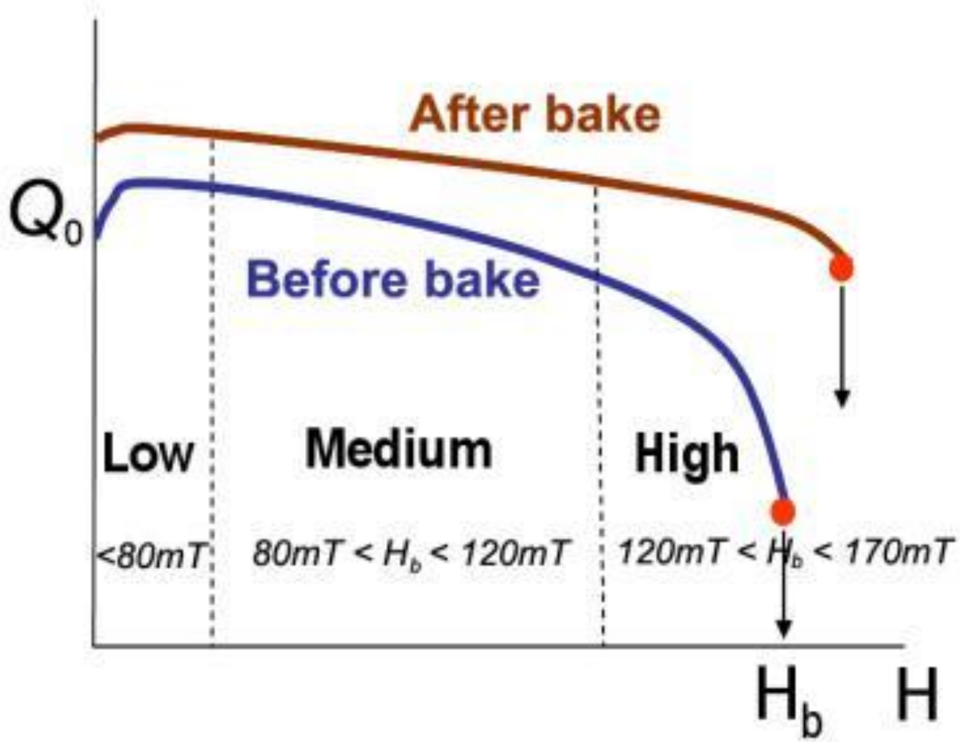
Motivations

Three distinct regions in Q vs E



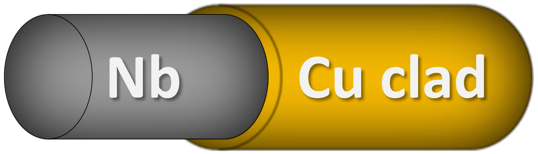
Courtesy of A. Gurevich

- The mechanism of HFQS (high field Q-slope) is not clear yet.
- Residual surface defects produced by cold-work deformation during cavity fabrication.
  - Grain boundaries, oxides, hydride, and dislocations
- The role of these defect in surface superconductivity.

Methodology

- Heavily deformed Nb wire from high purity SRF Nb rod by deep wire-drawing to introduce high cold work effect.
- For comparison, unstrained single and bi crytal sample from high purity (RRR >250) large grain Nb sheet.
- Optimized SRF Nb cavity surface treatment was applied to change surface properties.
- Surface roughness and strain microstructure by confocal microscopy and EBSD-OIM.
- AC susceptibility measurement for quantification of variation of surface superconductivity.

Nb wire fabrication by deep-drawing

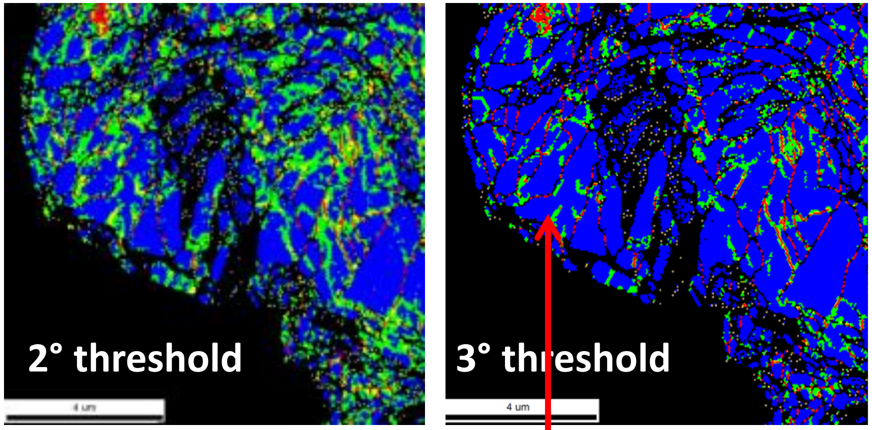


Annealed Nb rod starting Ø~0.428”(10.9 mm) & 6 ”in length (152.4 mm): starting with $\varepsilon_0 = 0$

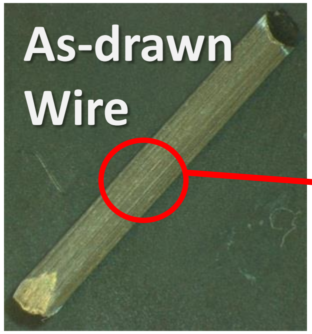
Final Nb size .044 (1.1 mm)

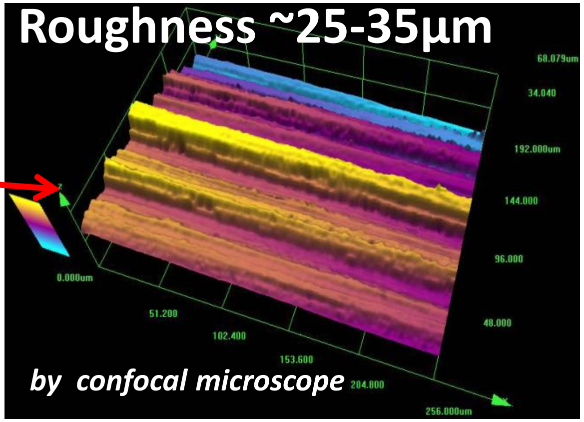
~ 90% reduction (ε_0~ 4.17)

Local misorientation map of cross section of an as-drawn wire by EBSD-OIM



High possibility of lower misorientation strained structures at GBs





Cu removed by HNO_3

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A series of surface treatments to change the level of surface defects

The recipe of "optimized" SRF cavity treatments

by A. Dzyuba @ FNAL

As-drawn (No EP)	50 min EP	2 hrs EP	3 hrs EP	50 m EP +120°C/ 48hrs	2 hrs EP +120°C/ 48hrs	3 hrs EP +120°C/ 48hrs	50 m EP +800°C/ 2hrs	2 hrs EP +800°C/ 2hrs	3 hrs EP +800°C/ 2hrs
As-drawn (No BCP)	30 min BCP	1 hr BCP	1.5 hrs BCP	30 m BCP +125°C/ 48hrs	1 hr BCP +125°C/ 48hrs	1.5 hrs BCP +125°C/ 48hrs	800°C HT is ongoing		

Eliminating surface defects

Reducing surface oxygen effect

Eliminating surface Hydrogen effect

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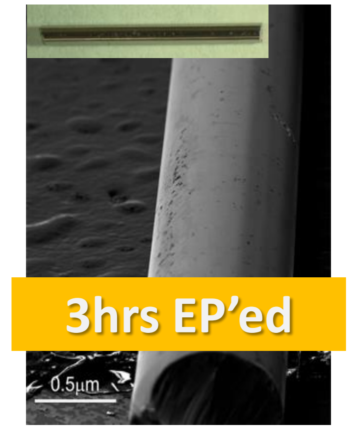
EP and BCP improved surface uniformity

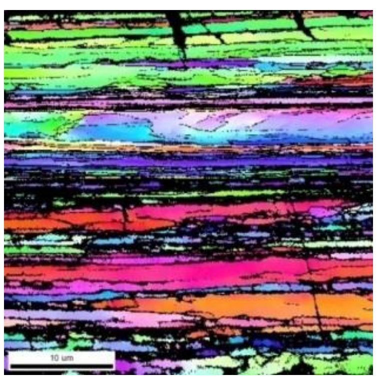


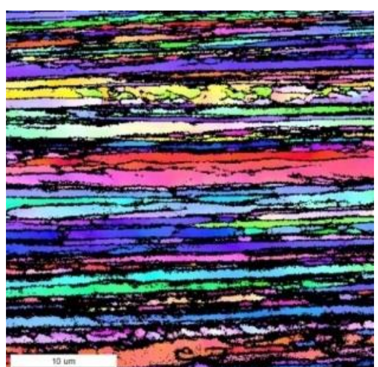
Roughness evaluated by SLCM (Scanning Laser Confocal Microscopy)

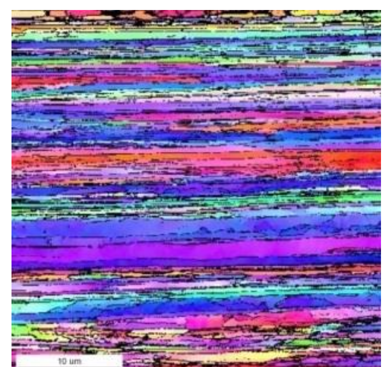
50 min EP’ed: 18.07 ± 0.19 μm

2 hrs EP’ed: 2.21 ± 0.14 μm

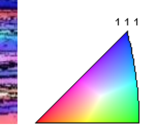
3 hrs EP’ed: 1.67 ± 0.08 μm





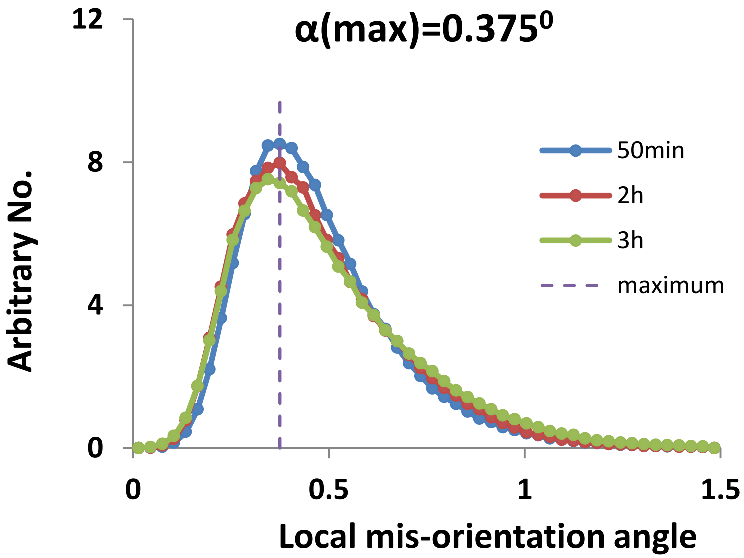


IPF map of the surface of EP’ed wires by EBSD-OIM

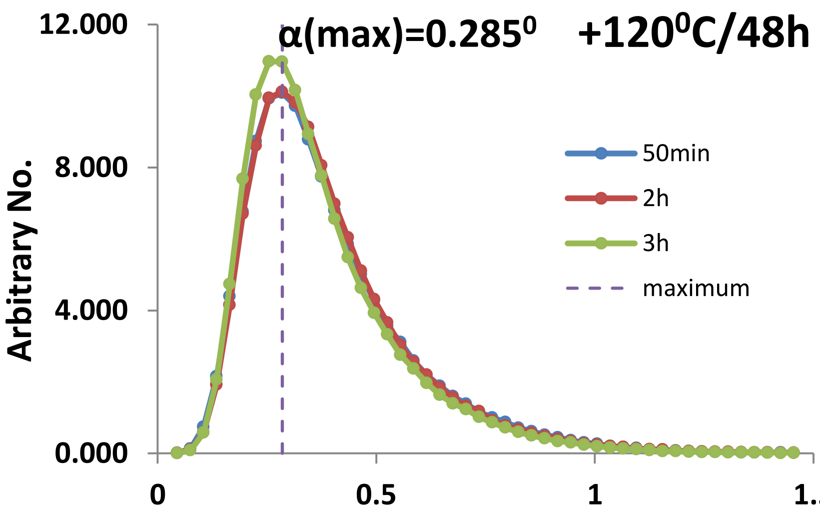


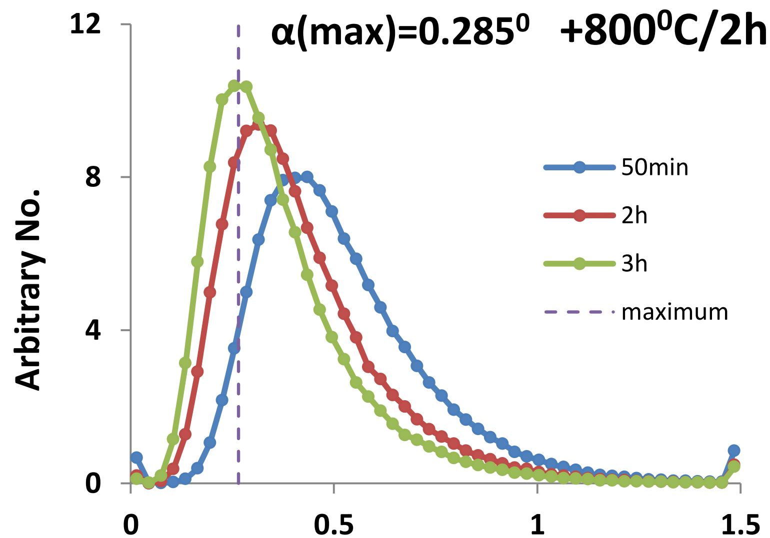
IPF map

Reduction of local surface strain effect after 120°C/48h, but Redistribution by 800°C/2h HT



Local misorientation map shows the density of dislocations on the surface





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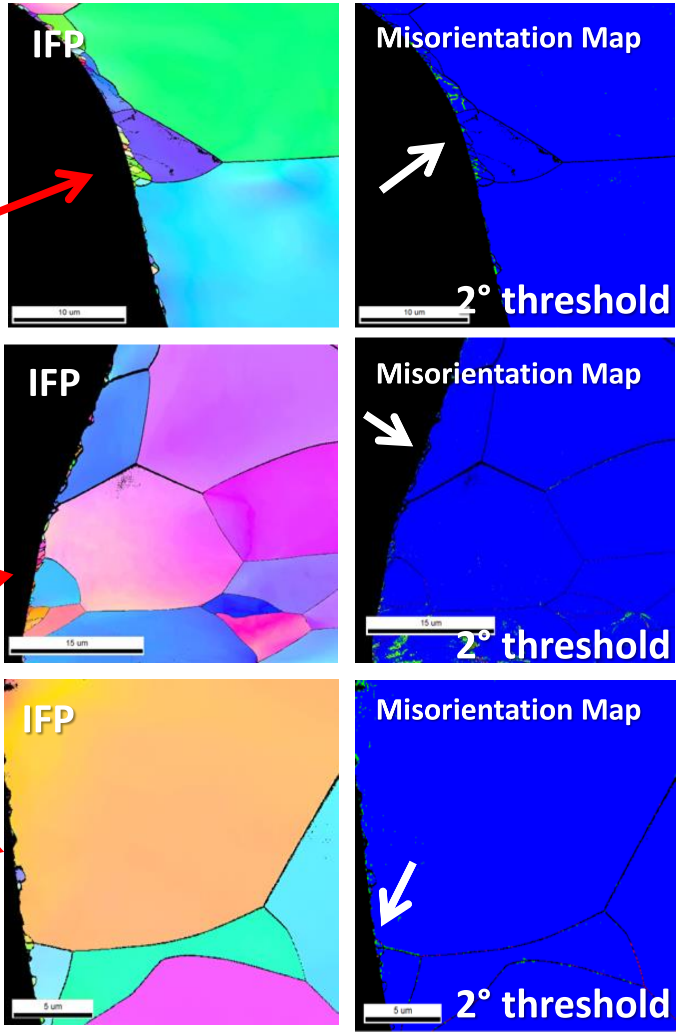
Cross sectional EBSDOIM for strained gradient of 800°C/2h HT'ed wires

800°C/2h HT almost recovers strained structure on the bulk, but strained effect still remain on the surface

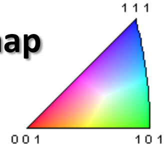
50 min EP'ed + 800°C/2hrs HT

2 hrs EP'ed + 800°C/2hrs HT

3 hrs EP'ed + 800°C/2hrs HT



IPF map



Z-H. Sung June 19th, 2013

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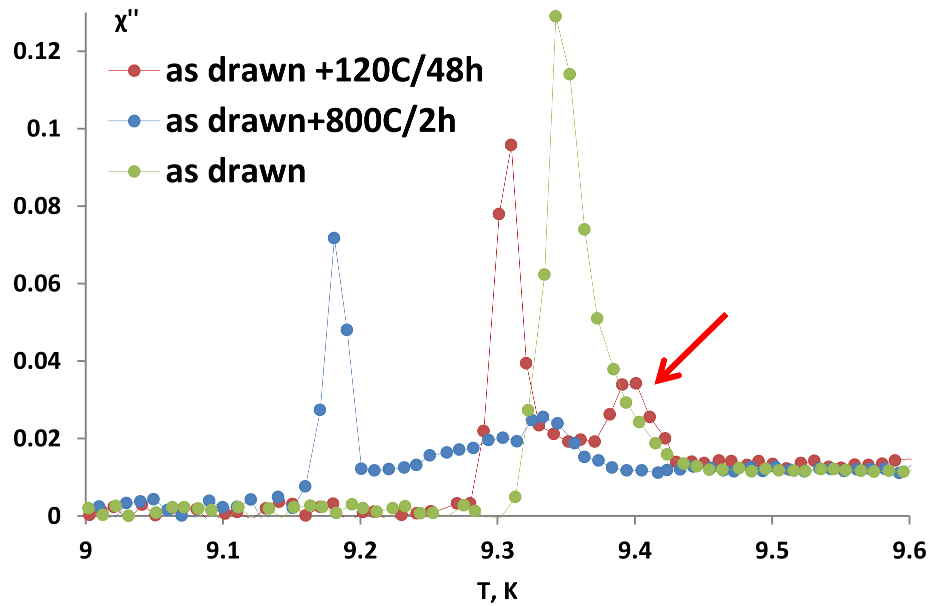
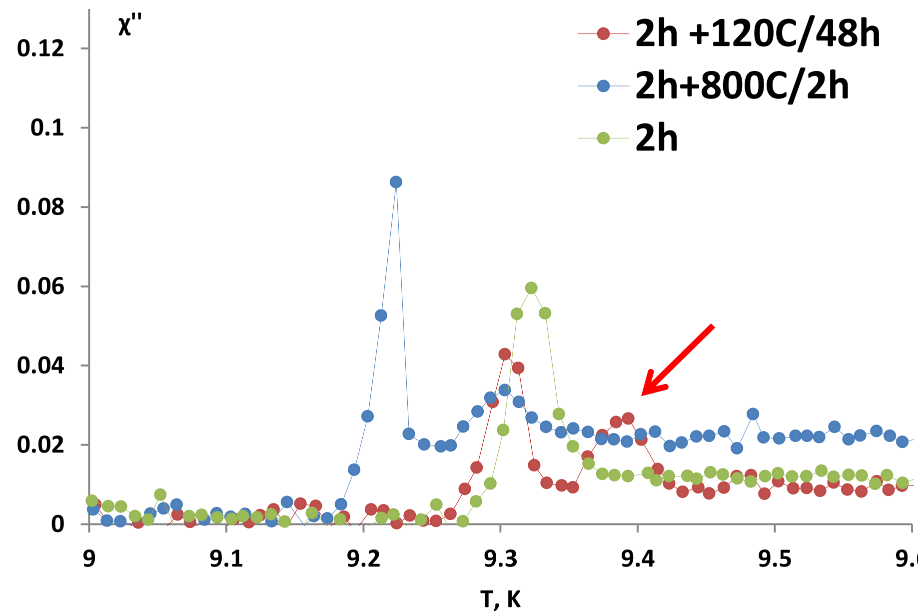
The onset of T_c varies by surface effect on AC susceptibility measurement

Another bump appears after 120°C/48hrs HT

***As-drawn wire* with AC = 100 Hz, 0.1 Oe – Imaginary part**



***2hrs EP'ed wire* with AC = 100 Hz, 0.1 Oe – Imaginary part**



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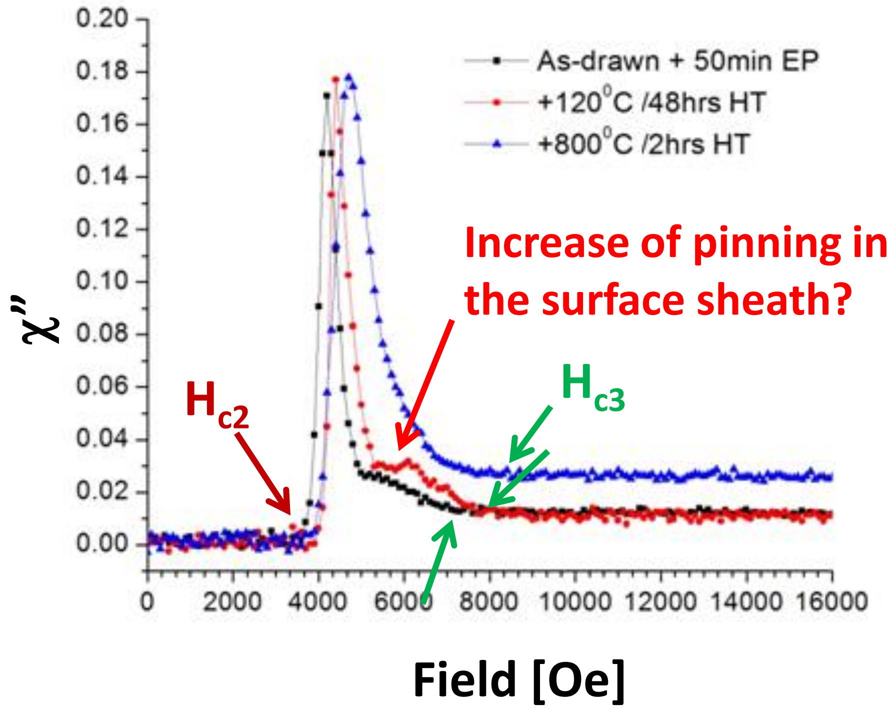
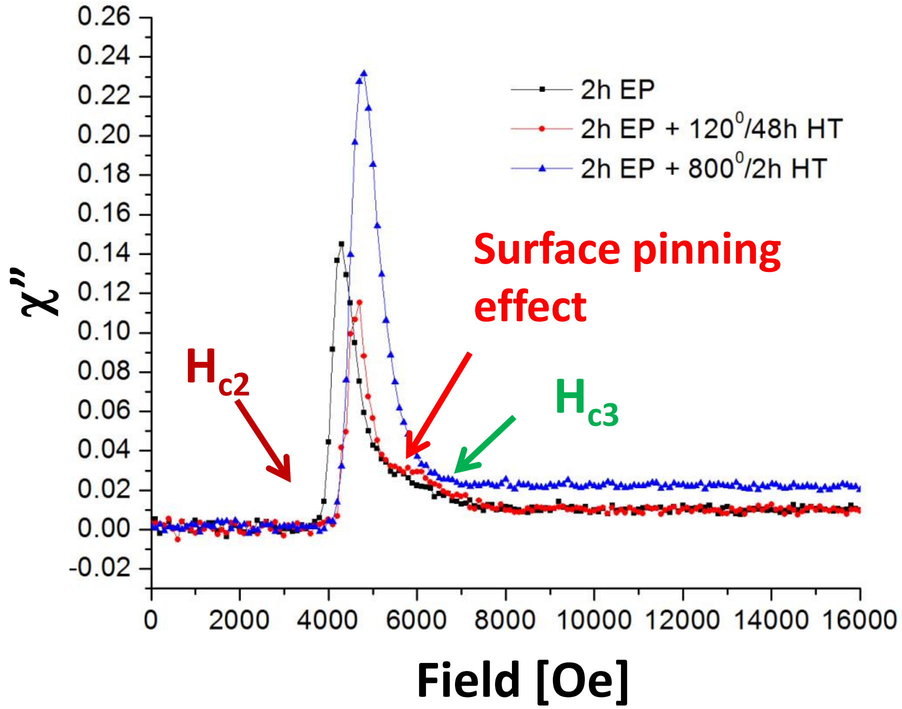
The onset of H_{c3} also changed by surface pinning effect

50m EP'ed wire with 100 Hz, 0.1 Oe @5K – Imaginary part



2h EP'ed wire with 100 Hz, 0.1 Oe @5K – Imaginary part



Surface effect disappears after 800°C/2h HT

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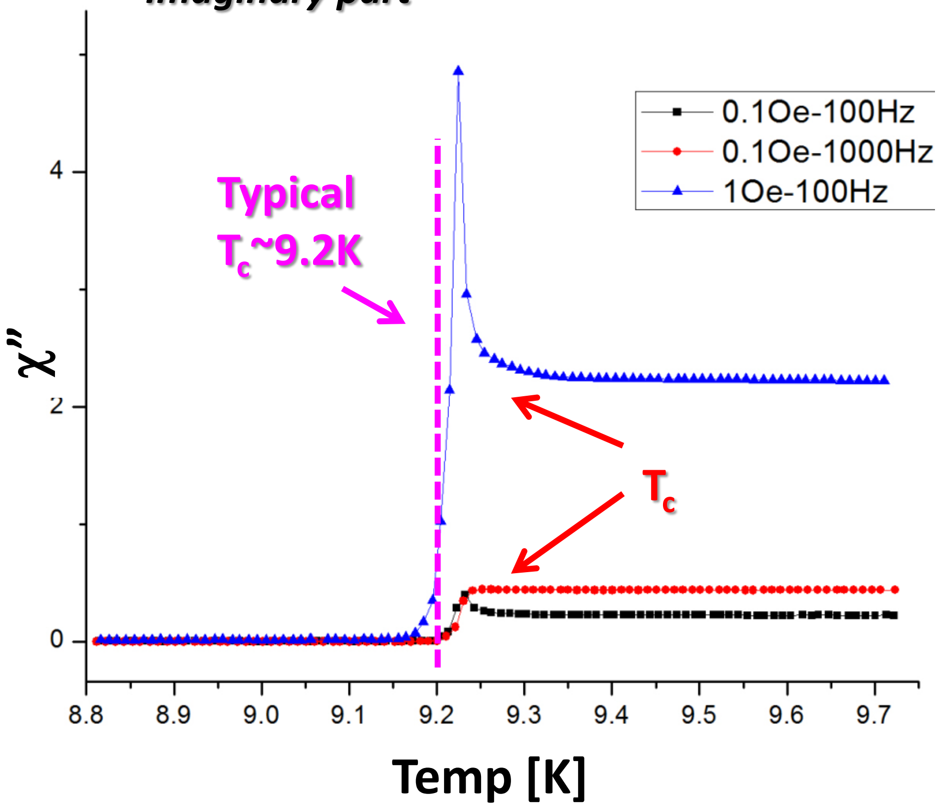
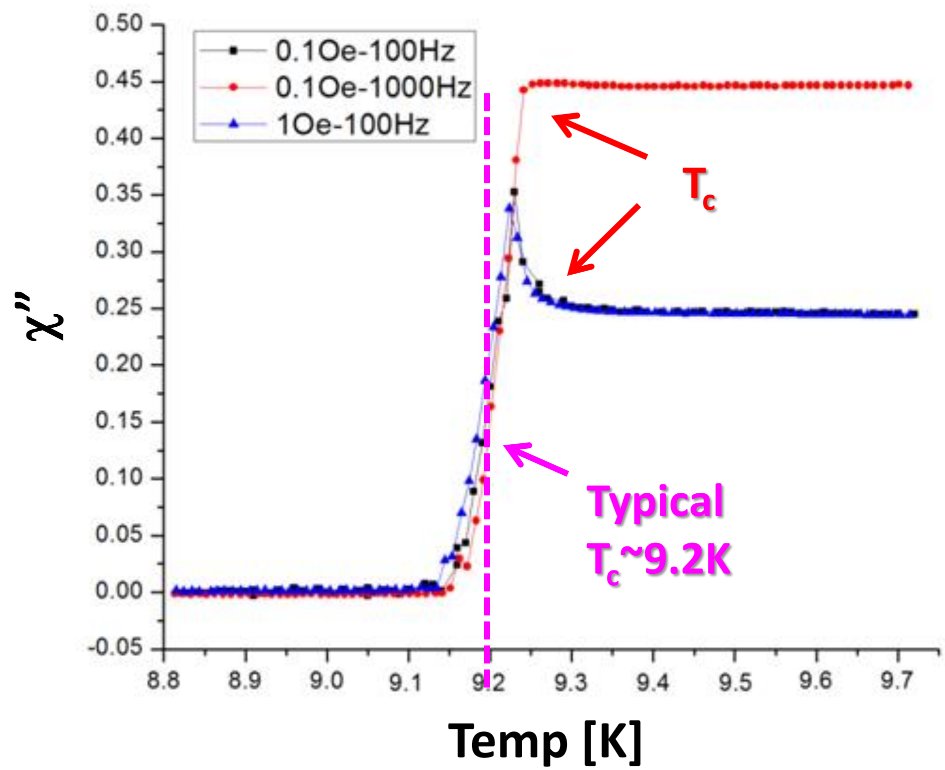
No extra surface layer effect on T_c transition of BCP'ed single and bi-Xtal after 125°C/48hr HT

30m BCP'ed Single Xtal + 125°C/48hrs
– Imaginary part



30m BCP'ed Bi Xtal + 125°C/48hrs
– Imaginary part



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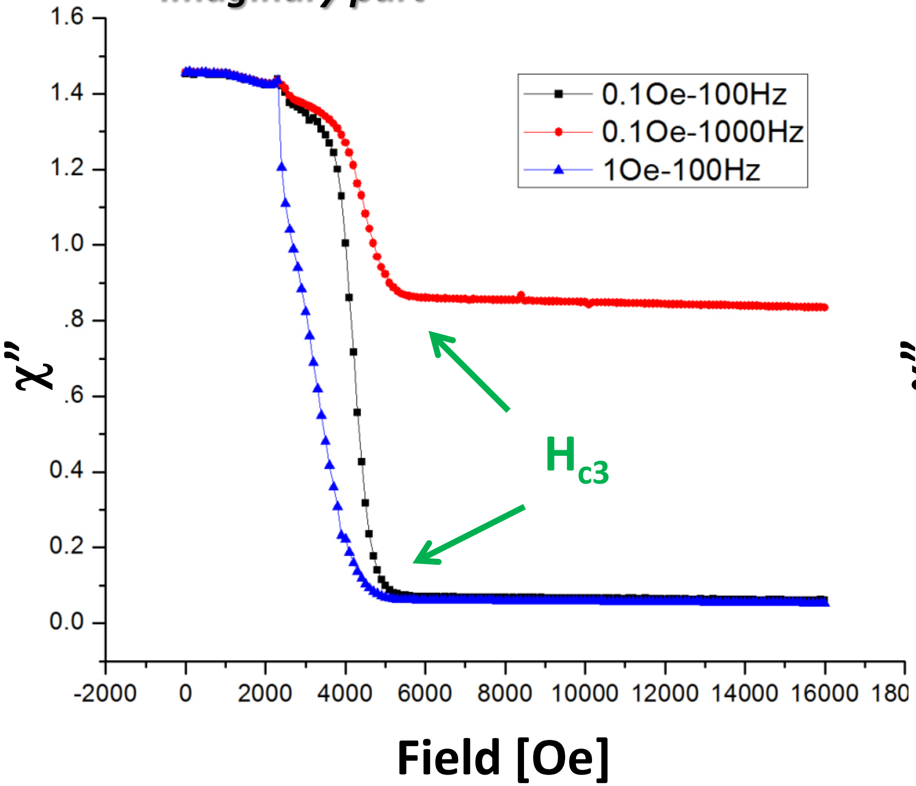
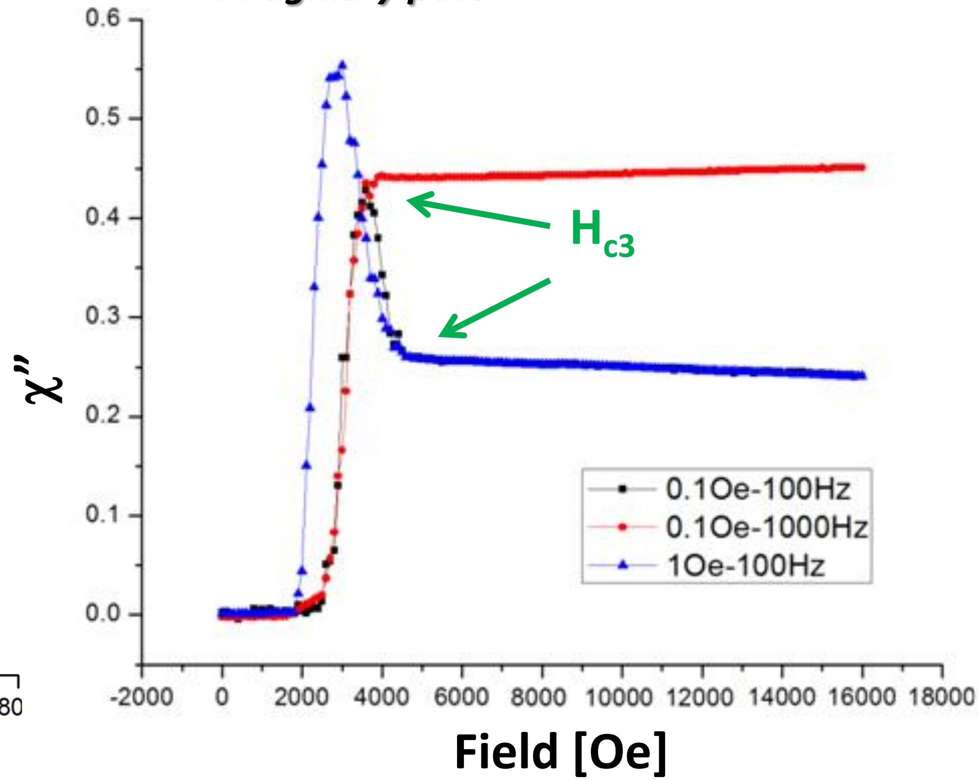
No extra surface layer effect on the onset of H_{c3} of BCP'ed single and bi-Xtal after 125°C/48hr HT

30m BCP'ed Single Xtal +120°C/48hrs – Imaginary part



30m BCP'ed Bi Xtal +120°C/48hrs – Imaginary part



	T_c [K]	ΔT [K]	H_{c2} at 5K [Oe]	H_{c3} at 5K [Oe]	r_{32} at 5K [H_{c3}/H_{c2}]
50 min EP	9.32	0.10	3598	6990	1.943
2 hrs EP	9.32	0.09	3825	7359	1.924
3 hrs EP	**9.33**	**0.08**	3986	6719	1.686
50 min EP + 120°C HT	9.29	0.14	4314	**7631**	1.769
2 hrs EP + 120°C HT	9.30	0.15	4175	7576	1.814
3 hrs EP + 120°C HT	9.30	0.12	**3553**	7281	**2.049**
50 min EP + 800°C HT	9.22	**0.17**	3931	**7631**	1.941
2 hrs EP + 800°C HT	9.22	0.15	4092	6931	1.694
3 hrs EP + 800°C HT	**9.22**	0.16	**4470**	**6903**	**1.544**

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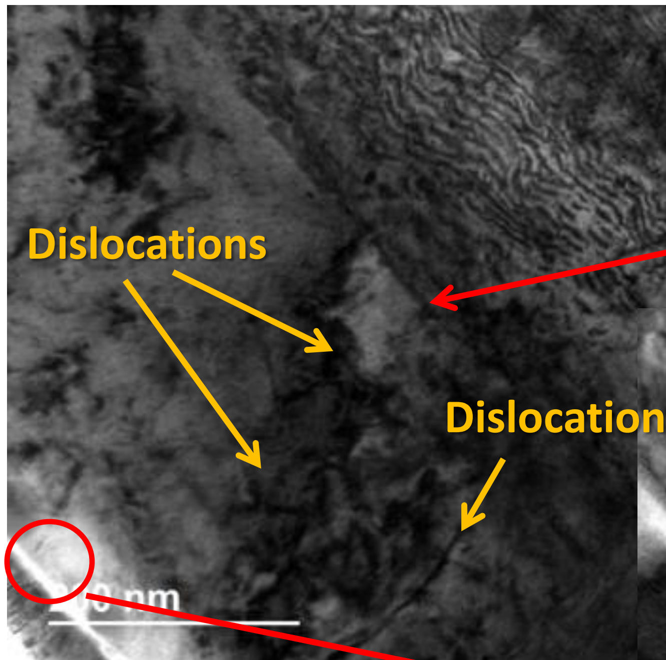
Conclusion

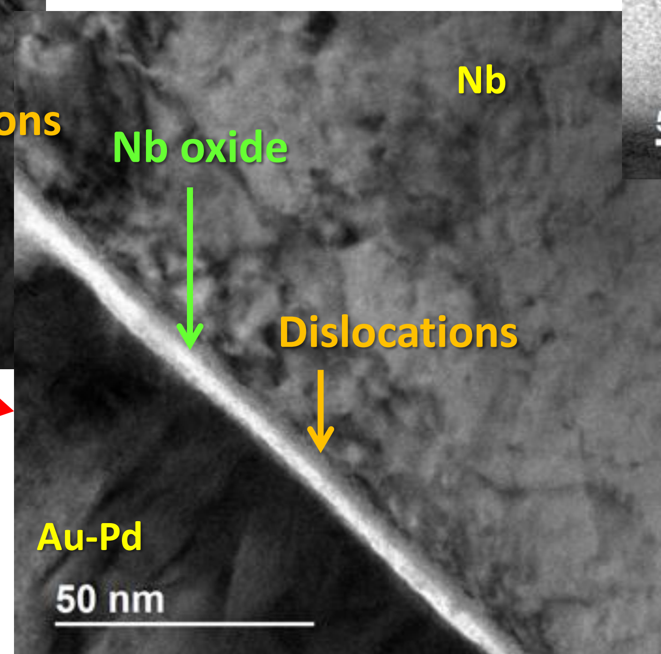
- **Strained grain boundaries or GNB, or strained layers highly enhance impurities migration to the surface layer.**
- **Longer EP and high temp baking returns r_{32} to GL value (≈1.695) or even lower.**
- **Migration of impurities (H or O) to the surface layer may increase by mild baking then reduce H_{c3} values.**
- **800°C/2hrs HT is enough to clean the surface. However, it may not be enough to get rid of all impurities in the wire.**
- **Is H_{c3} increase by surface pinning good or bad to SRF?**
  - **Higher H_{c3} is desirable for better surface superconductivity.**
  - **But more pinning sites may increase surface residual resistivity.**

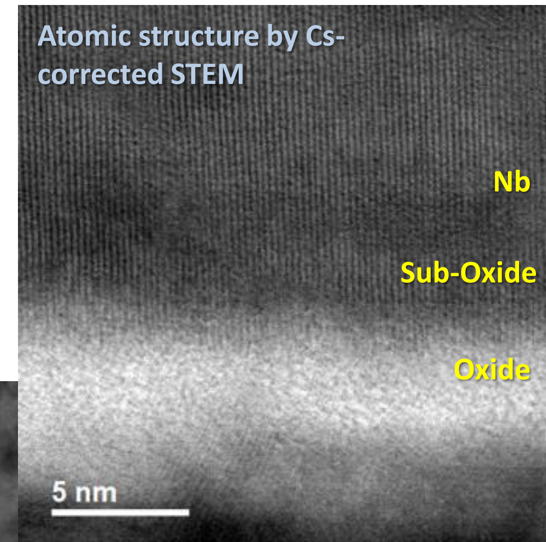
Dislocation structures as interstitial super highways versus the passive Nb oxide

Only GBs? Or Dislocations at GBs?, but surface Oxide layers?

HR TEM image of as-drawn Nb wire



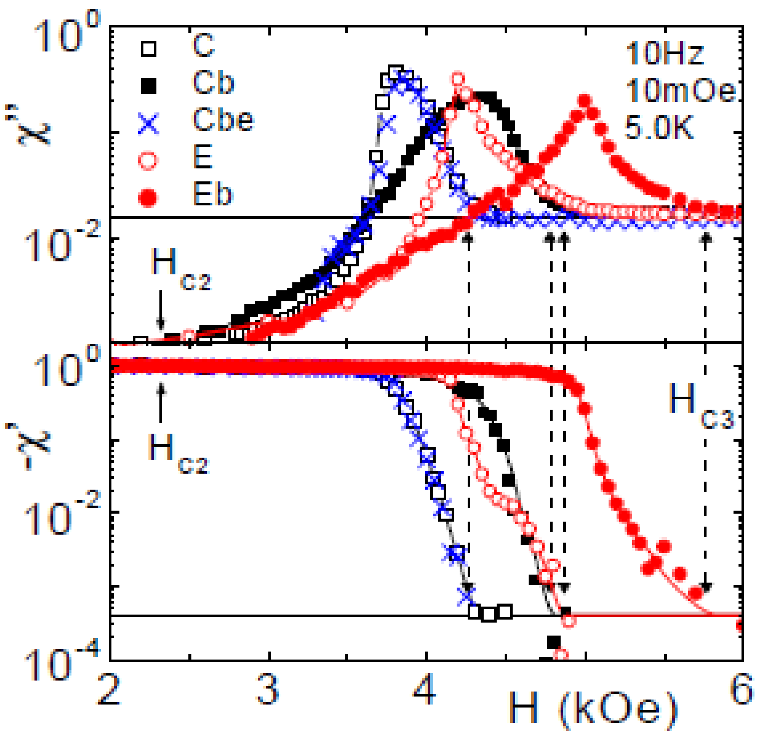




Oxide layer firmly connected to Nb matrix

AC Susceptibility measurement enables measurement of H_{c3} (Surface superconductivity)

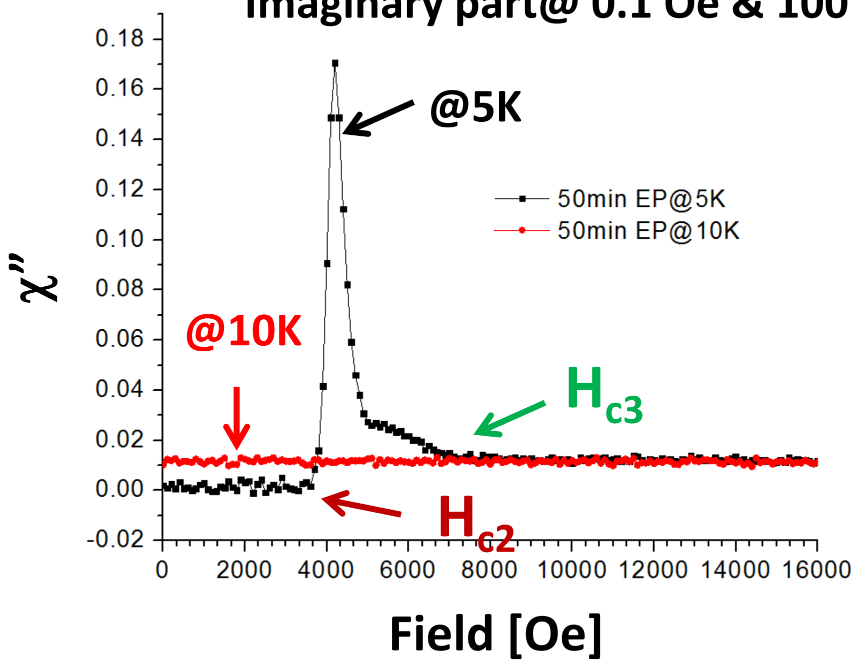
10 Hz, 10m Oe, 5K by S. Casalbuoni (2005)



2.5mm by 2.8mm disk-shape by MPMS2

H_{c3}: deviation from the linear fitting of normal state (@10K)

50 min EP'ed wire-
Imaginary part@ 0.1 Oe & 100 Hz

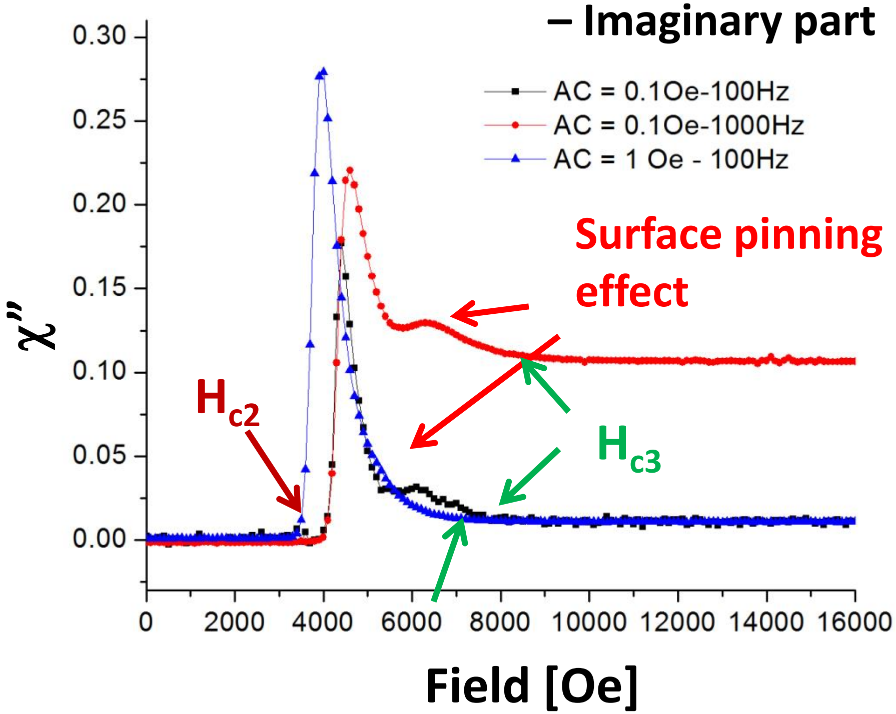


Ø~0.8mm l~8mm wire-shape by PPMS

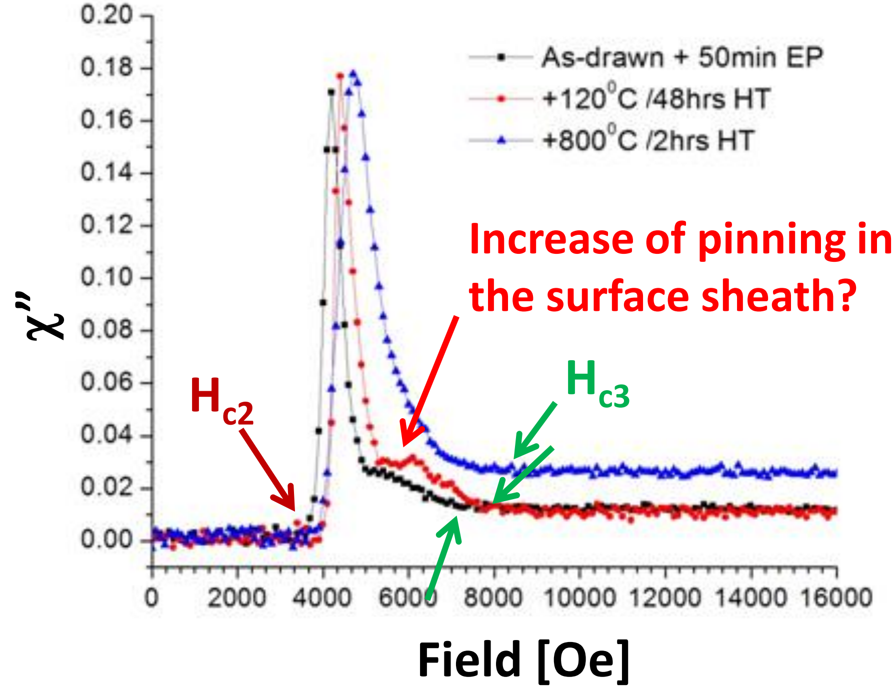
Surface pinning effect on Hc3 also revealed by varying AC amplitude and frequency

Onset of H_{c3} vary by surface pinning effects

50 m EP'ed + 120°C/48hrs HT @5K – Imaginary part

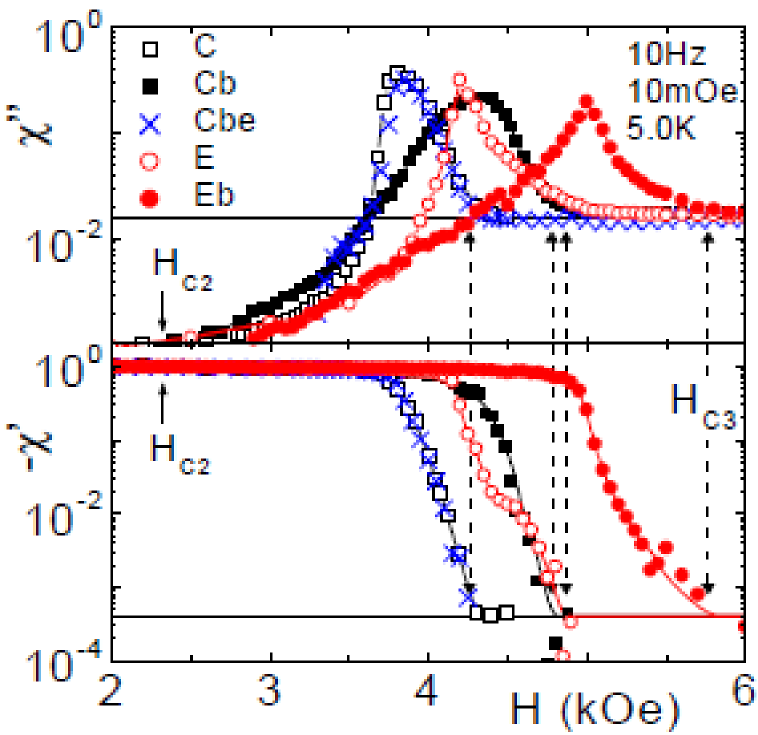


100 Hz, 0.1 Oe @5K – Imaginary part



The ratio (r_{32}) of H_{c3} to H_{c2} of SRF-processed Nb deviate markedly from the GL values of 1.695

10 Hz, 10m Oe, 5K by S. Casalbuoni (2005)

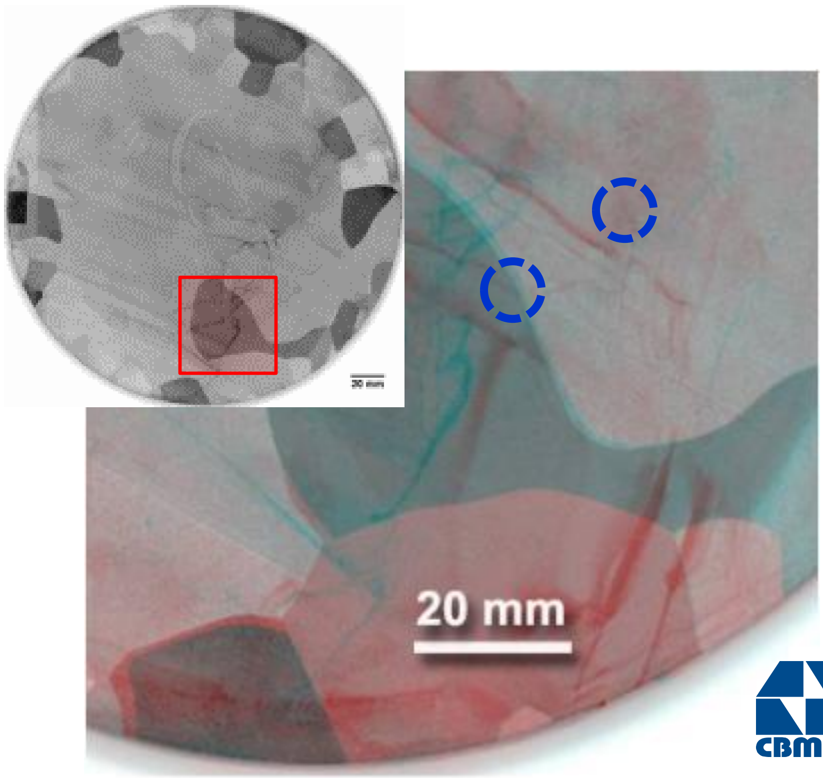


2.5mm by 2.8mm disk-shape by MPMS2

Table 3
The ratio $r_{32} = B_{c3}/B_{c2}$ for all samples

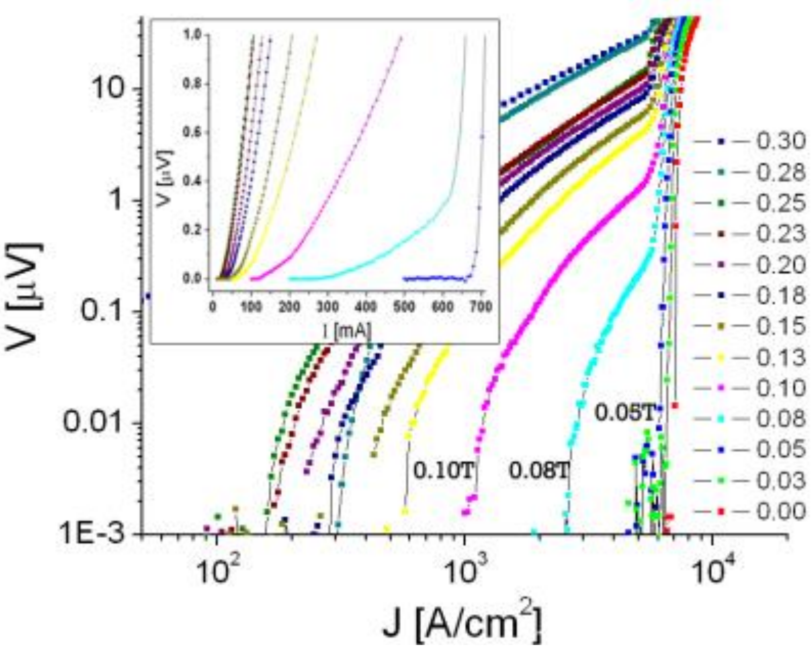
Sample	$r_{32} = B_{c3}/B_{c2}$
BCP only	1.86 ± 0.03
BCP + baking	
BCP + 24 h 100 °C	1.93 ± 0.05
BCP + 48 h 100 °C	1.93 ± 0.05
BCP + 96 h 100 °C	1.93 ± 0.05
BCP + 48 h 120 °C	2.16 ± 0.03
BCP + 24 h 123 °C	2.17 ± 0.05
BCP + 48 h 123 °C	2.33 ± 0.05
BCP + 96 h 123 °C	2.32 ± 0.05
BCP + 24 h 144 °C	2.59 ± 0.05
BCP + 48 h 144 °C	2.59 ± 0.05
BCP + 96 h 144 °C	2.59 ± 0.05
BCP + baking + BCP	
BCP + 48 h 120 °C + 1 μmBCP	1.90 ± 0.03
BCP + 48 h 120 °C + 5 μm BCP	1.87 ± 0.03
BCP + 48 h 120 °C + 10 μm BCP	1.86 ± 0.03
BCP + EP	
BCP + 40 μm EP	1.92 ± 0.05
BCP + 80 μm EP	2.10 ± 0.03
BCP + 145 μm EP	1.99 ± 0.05
BCP + 165 μm EP	1.99 ± 0.05
BCP + EP + baking	
BCP + 40 μm EP + 48 h 123 °C	2.64 ± 0.05
BCP + 80 μm EP + 48 h 123 °C	2.57 ± 0.05
BCP + 145 μm EP + 48 h 123 °C	2.40 ± 0.05
BCP + 165 μm EP + 48 h 123 °C	2.40 ± 0.05

Does grain boundary make this layer on surface superconductivity?



Overview of the as-received niobium slice

Preferential flux flow on the deep GB groove by transport characterization



- ***Flux flow evidence from H = 0.08 T to 0.28 T***

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