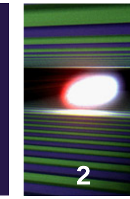
CONSIDERATION OF NB MATERIALS SPECIFICATION - FEEDBACK FROM EXPERIENCE AND REVISITING THE DRIVING REQUIREMENTS

WG2 Summary Part I

Waldemar Singer

TTC Meeting, DESY, March 27, 2014

Specification for European XFEL high purity niobium.

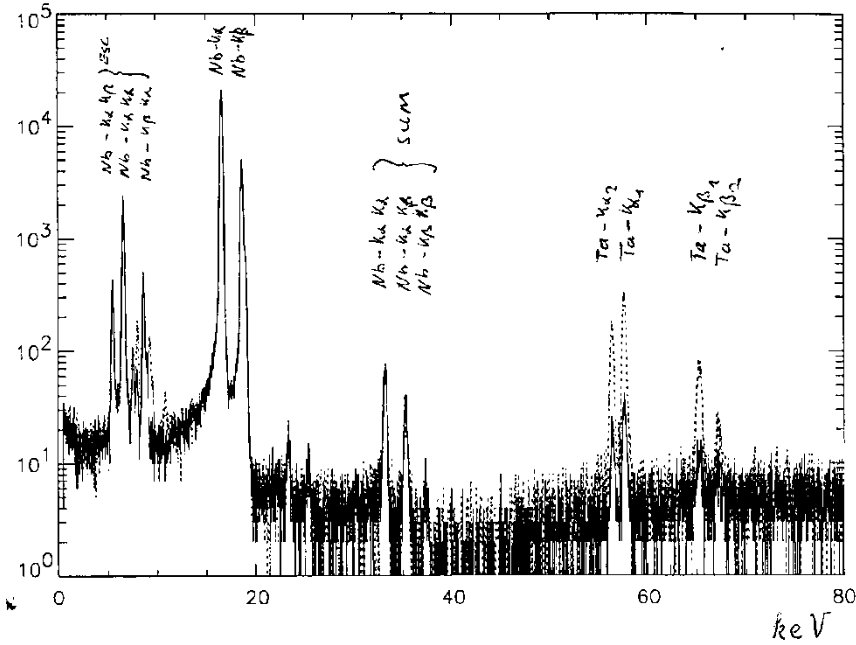


Concentration of impurities in µg/g				Mechanical properties	
Ta	≤ 500	H	≤ 2	Yield strength $\sigma_{0,2}$	$50<\sigma_{0,2}<100$ N/mm^2 (Mpa)
W	≤ 70	N	≤ 10	Tensile strength	> 100 N/mm^2 (Mpa)
Ti	≤ 50	O	≤ 10	Elongation at break	30 %
Fe	≤ 30	C	≤ 10	Vickers hardness HV	≤ 60
Mo	≤ 50	RRR	≥ 300	Absence of foreign material inclusions	Proven by Eddy Current scanning
Ni	≤ 30	Grain size	50-100 µm		

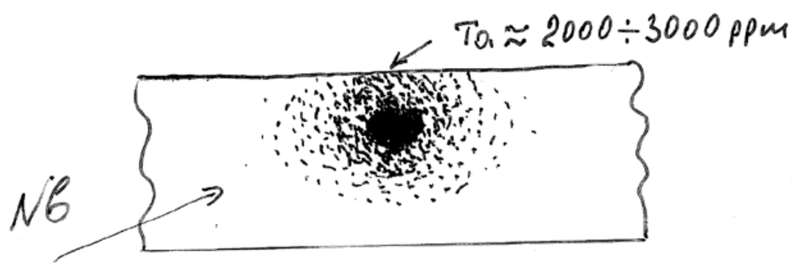
Revisiting: Ta content, RRR, Scanning, use LG

Effect of Ta in Cavity Performance

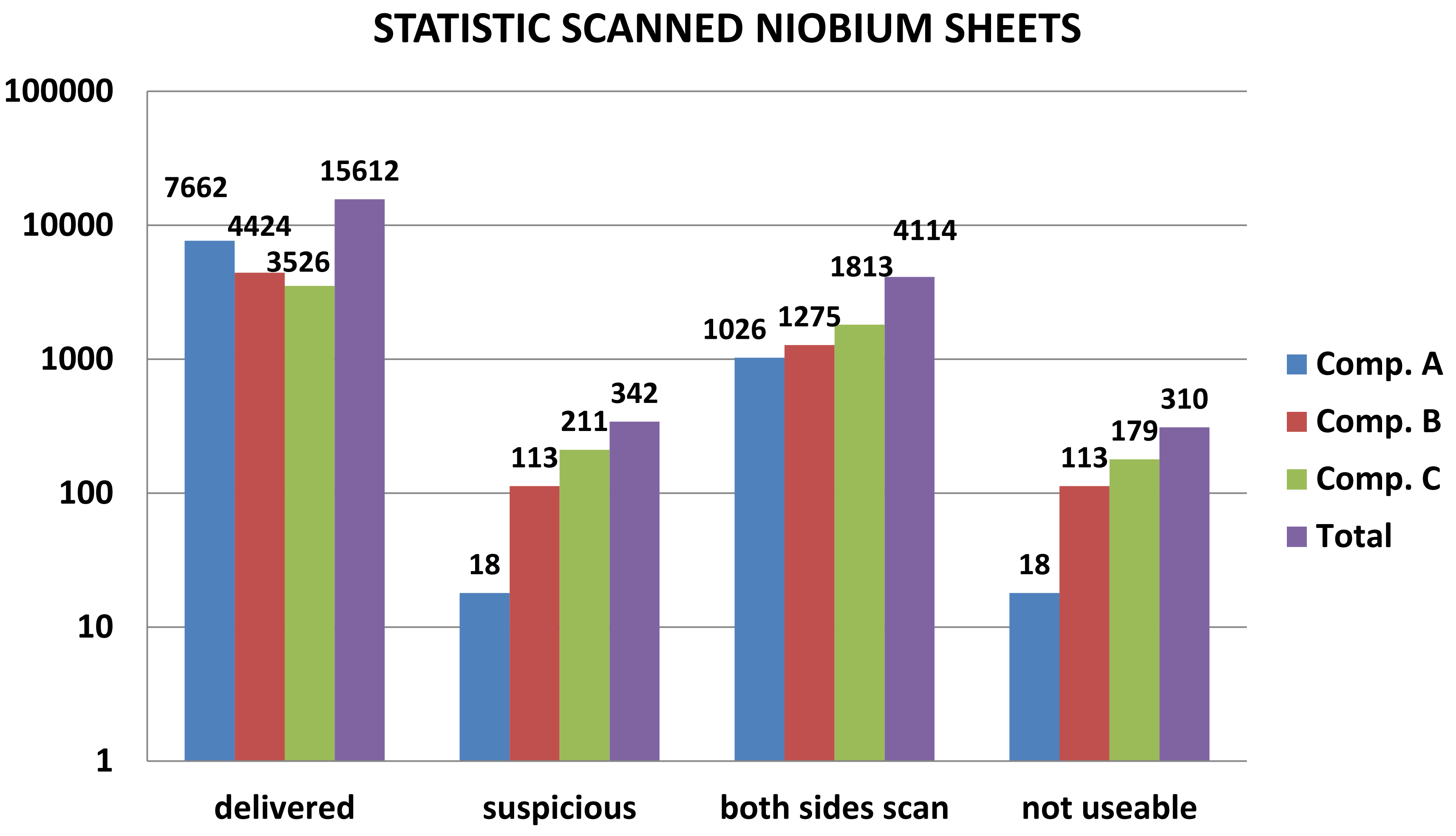
- In 1995 we conducted a study of the influence of Ta concentration in the Nb on cavity performance for concentration from 130 ppm to 1300 ppm (P.Kneisel et al, PAC '95, p.3955)
- No significant dependence of quench field on Ta contents was found; in an ILC shape cavity a gradient of E = 30.5 MV/m was measured corresponding to a $B_{p,quench}$ ~ 130 mT
- In a 9-cell ILC cavity (JLab-LG1) made from CBMM Nb with 1300 wtppm of Ta a gradient of 31 MV/m ($B_{p, quench}$ = 131 mT) was measured (G. Ciovati et al., SSTIN'10, p. 25)



Spectrum in the Ta spot of cav D6 (Eacc=13 MV/m) area (dotted line) and far away of the spot (full line)



To scan or not to scan the sheets. Statistic to Eddy Current scanning of Nb sheets for EXFEL



Fe (Iron) inclusion before EP

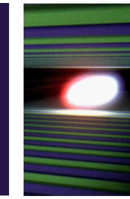
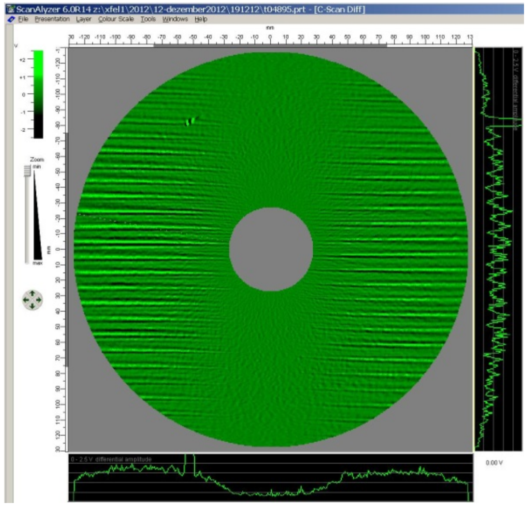
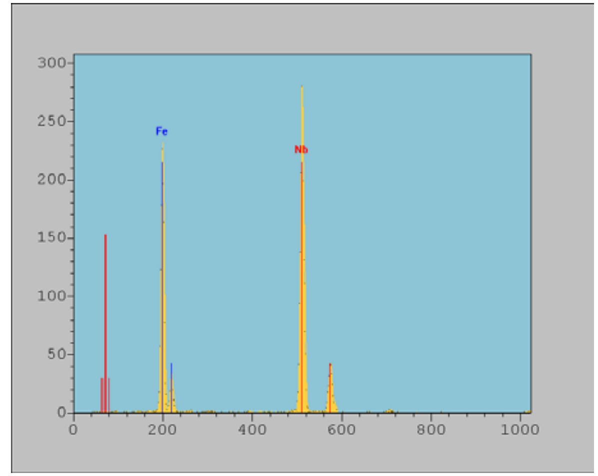
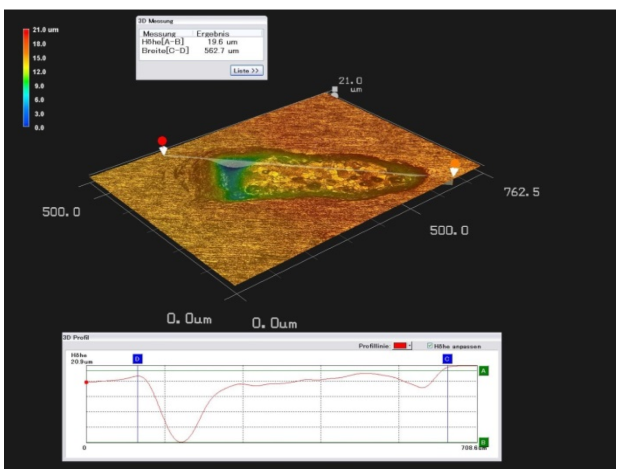


Image of eddy-current scanning on Nb-sheet No. T04895. S.1.

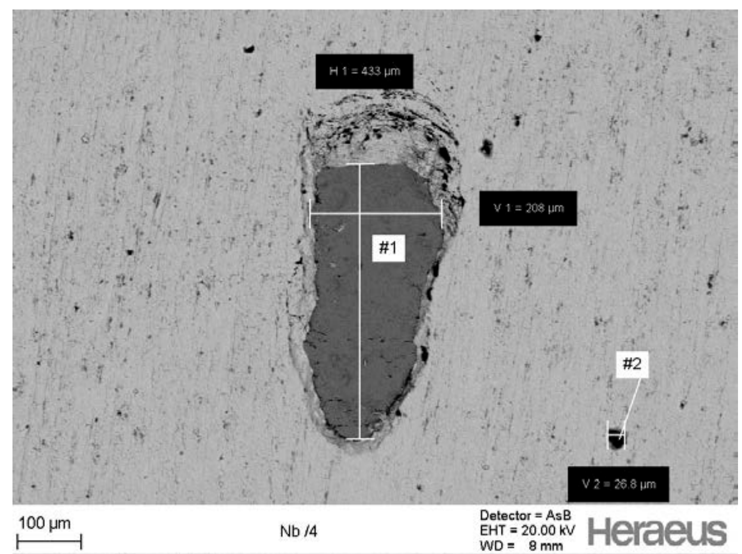




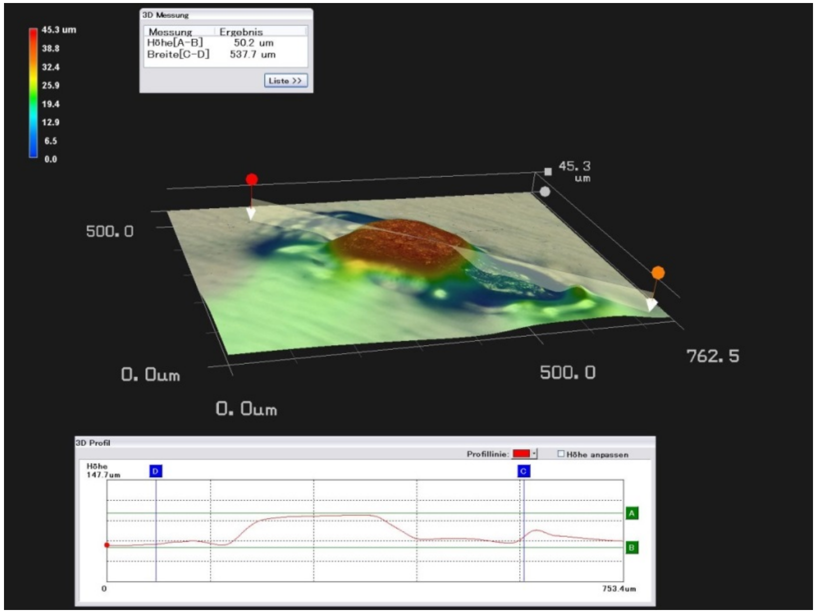
Non-destructive element analysis on sheet No. T04895. S.1



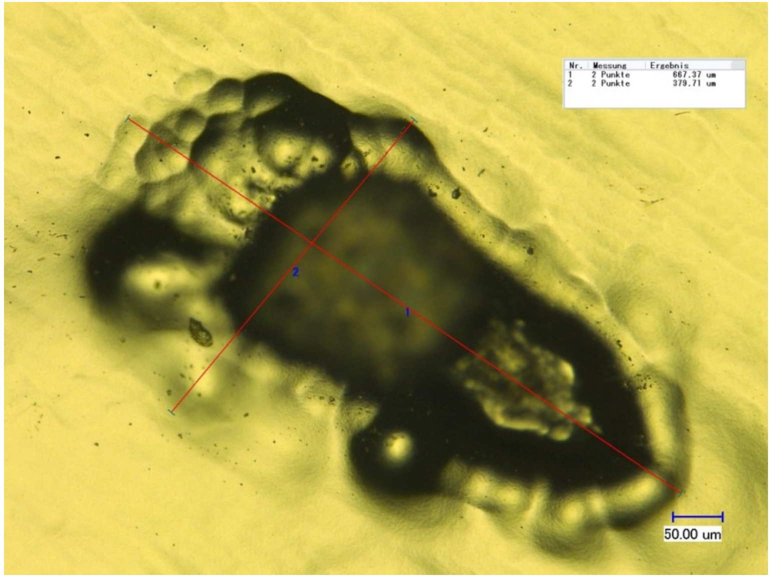
Digital microscope (left) and SEM image (right) . Sample of Nb-sheet No. T04895. S.1.



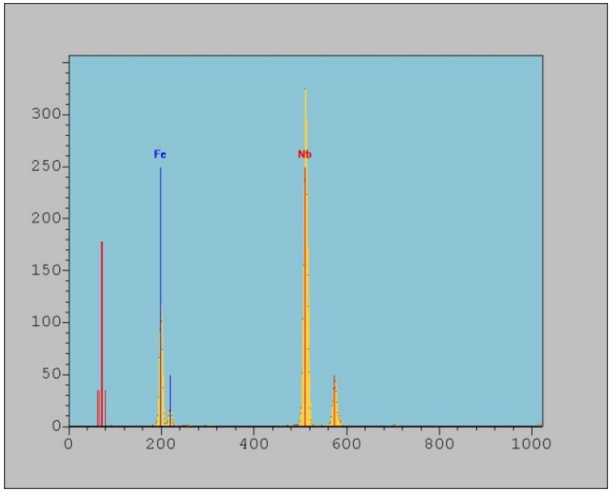
Fe (Iron) inclusion after ca. 150µm EP



Digital microscope images. Sample from Nb- sheet No. T04895. S.1.

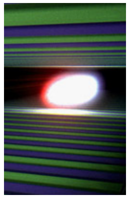


Non-destructive element analysis. Sample from Nb-sheet No. T04895. S.1



Rests of iron remains after EP

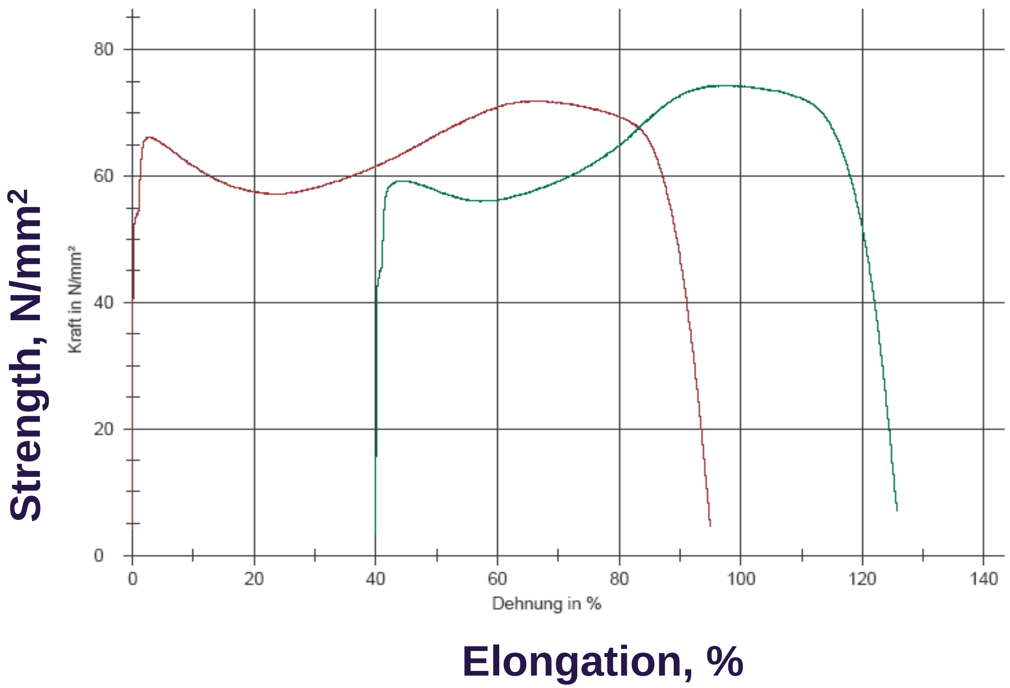
Is LG Nb applicable for SC Linacs?? LG is softer as FG. Fulfill the PED (PMA), stand crash test



Ergebnisse:

Nr	Probe	b0 mm	a0 mm	Rp 0.2 N/mm²	Rm N/mm²	A 30mm %
1	Mitte Ingotscheibe	7,95	2,56	52,6	71,9	95,0
2	radial am Rand Ingotscheibe	8,15	2,88	43,0	74,3	85,7

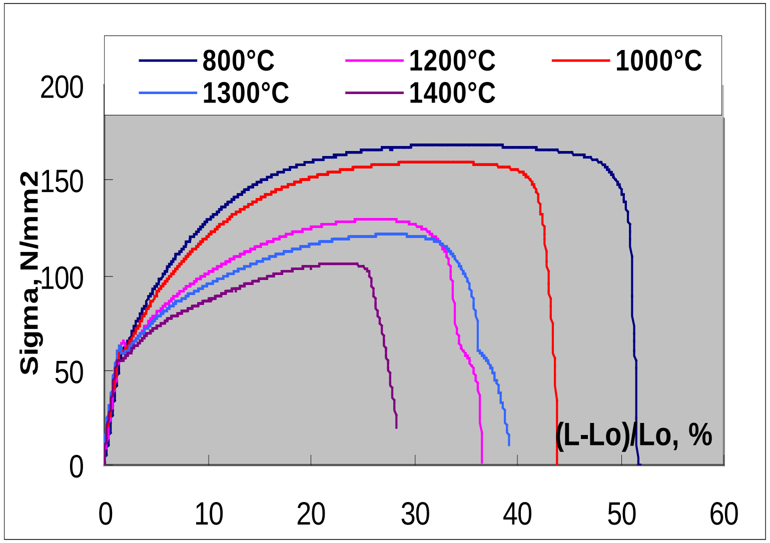
Seriengrafik:



Strain-stress curves of large grain Nb (data of B. Spaniol, Fa. Heraeus)

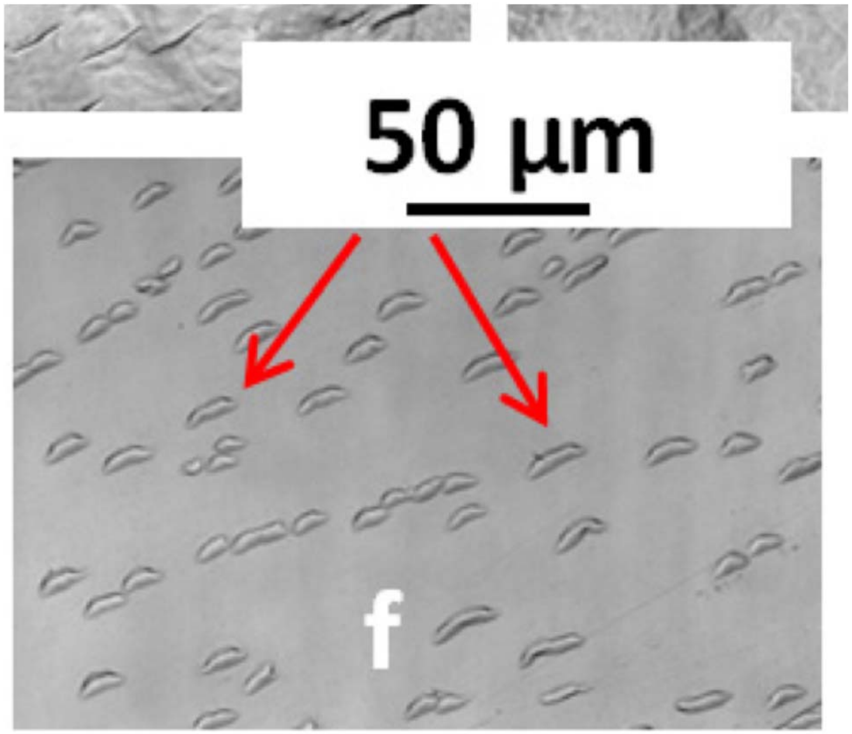
Remark to LG:

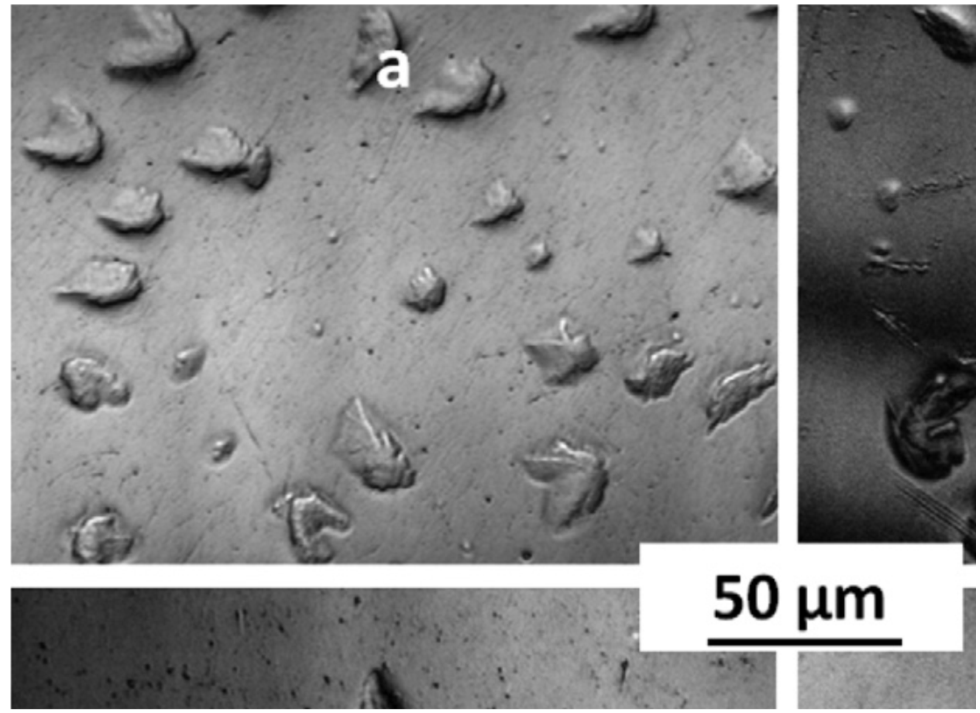
- Better to use terminology Ingot Nb.
- Ingot Nb (LG) has to be defined more precisely (grain size, crystallographic orientation, etc).



Strain-stress curves of FG Nb after different annealing

LG - FG: Hydrides – do not form preferentially at GB

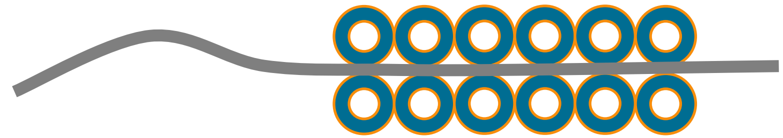




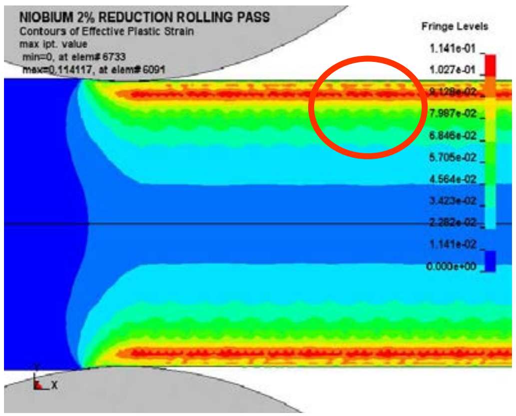
Insensitivity of LG to furnace contamination (carbon, Ti?)
Flux trapping/expulsion – fast/slow- no difference FG/LG
Flux pinning strength- LG very low pinning

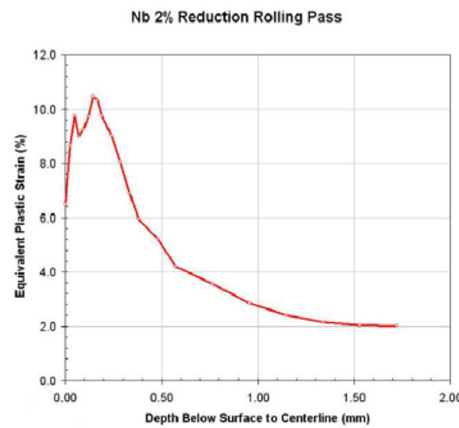
DAMAGED LAYER

After rolling sheets undergo a skin pass for planarity



[R. Crook et al, Black Laboratory]





Finite element simulation of 2% reduction of 3.5 mm sheet with 1 cm diameter rolls (Courtesy Non-Linear Engineering, L.L.C.). Stress is concentrated in the near-surface region (~300 µm). Localized strain exceeds the average by a factor of 5

- Damage layer = deformed grains + high density of dislocations + (foreign atoms)
- Rolling leaves a damage layer ~2-300 µm with a texture resistant to recrystallization, i.e. same order of magnitude than the necessary etching of material.

wire sawing of L G discs at W.C.Heraeus

LG seems not to have damage layer caused by rolling

Main conclusion:

- It might be of advantage / cost saving to use material with different specifications based on the required project needs : costs are e.g. RRR/RG~2.3, RRR/Ingot ~ 6
- For high Q and moderate gradients (Bp $\leq$ 90 mT), material with lower RRR might be sufficient

TESLA

Ta $\leq$ 500 wt ppm
RRR > 300
Grain size 50 micron
Yield strength > 50 N/mm^2
Tensile strength > 100 N/mm^2
Elongation $\geq$30%
Vickers Hardness < 50

CW SRF

Ta $\leq$ 1300 wt ppm
RRR 75 – 200
Grain size > 1 cm
Yield strength > 50 N/mm^2
Tensile strength > 100 N/mm^2
Elongation $\geq$ 30 %
Vickers Hardness < 50

WG 2b
Coupling of materials and processes: How do processes change materials in beneficial ways?

C. Reece

WG2b

Theme:

Improving understanding of material features which impact rf losses.

- "Stanford" Nb spec. extended to high RRR = "Standard" RRR spec
  - <500 ppm Ta, grain size ASTM 5
- "Ingot Nb" = sheet stock derived from slicing from ingot = grain size "large"
- "High Ta Nb" = < 1500 ppm Ta content

- New data present **fresh challenges for the parameterization and interpretation** of the field dependence of the SRF surface resistance, R_s. ($Q_0 = G/\,R_{s\ avg}$)

$$R_s = \mathrm{R}_{bcs}(T,B) + \underbrace{R_{flux} + R_{norm} + \mathrm{R}_{pe}(T,B)}_{R_{residual}} + \mathrm{R}_{eff\text{-topo}}(B) + R(E)_{eff\text{-FE}} + \dots$$

BCS	ambient flux pinned	normal metal	proximity effect	roughness field enhancement	field emission

***R**residual*

WG2b

A. Other factors controlled, **Ingot Nb appears to have somewhat lower RF losses**. (Although the difference can be overwhelmed by other factors if not controlled)

B. Fine-scale roughness (< ~5 micron) inducing locally enhanced $H > H_c$, appears to **explain high-field Q drop delta between BCP & EP** finished "standard" FG Nb.

C. The phenomenon of remarkably **reduced rf losses with surface diffusion of select impurities** has been recently identified and confirmed.

D. These reduced losses lower low-field BCS resistance and **extend the "low-field Q rise" to Bpk ~100 mT**.

E. A convenient implementation of such surface diffusion is 800 C low-pressure nitrogen doping "**HT-N**" followed by shallow EP discovered at FNAL.

F. R_s reduction is **not from unusual Nb** or Nb compound phase.

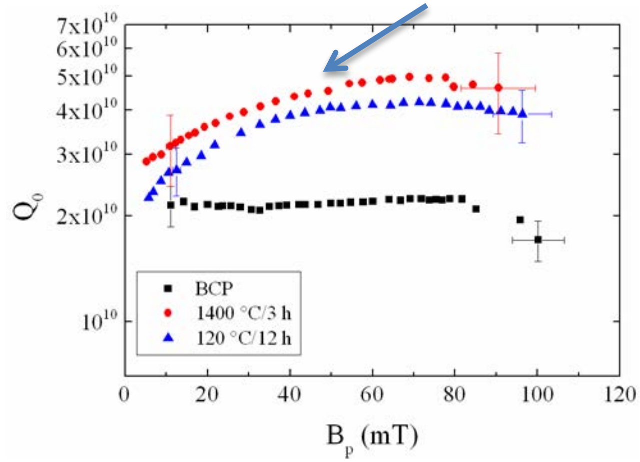
G. 1400 C weak diffusion of Ti into Nb produces similar R_s reduction.

H. Development of robust protocol to exploit lowered losses is underway.

Summary of results presented at SRF'13

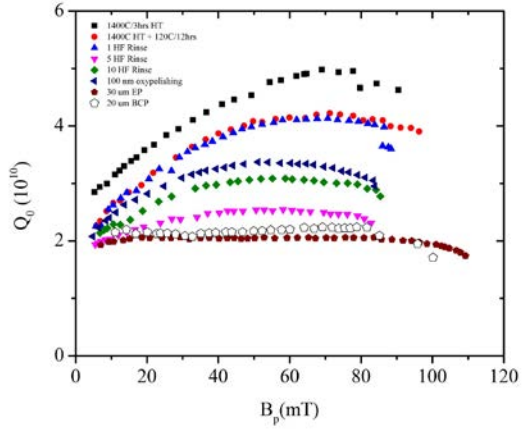
- Investigation of the cause for the extended Q-increase:

  ✓ Series of tests on "all Nb" LG and FG cavities support the hypothesis that this phenomenon is related to the diffusion of Ti (~1 at.%) in a µm-thick layer, as initially measured by SIMS, sublimating from Ti45Nb flanges



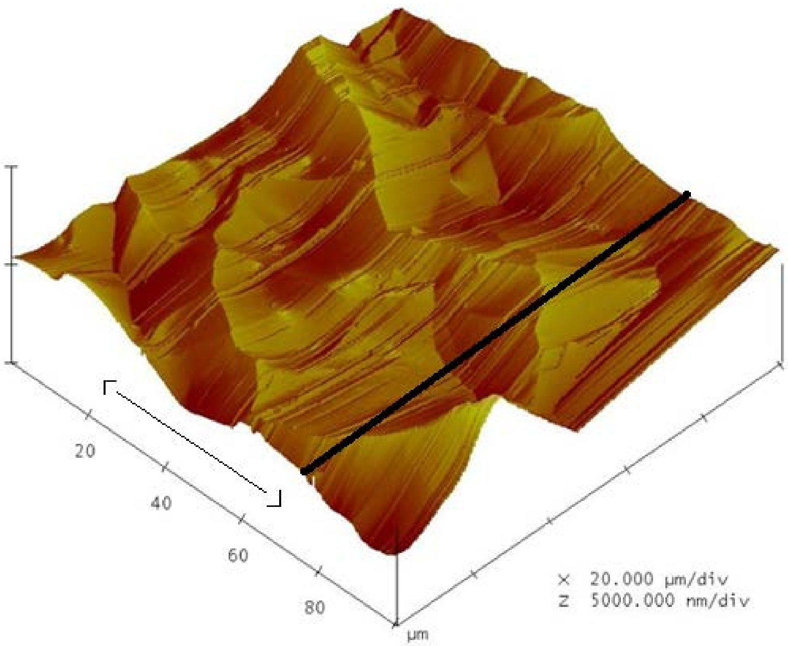
- Depth of Nb exhibiting extended Q-increase:

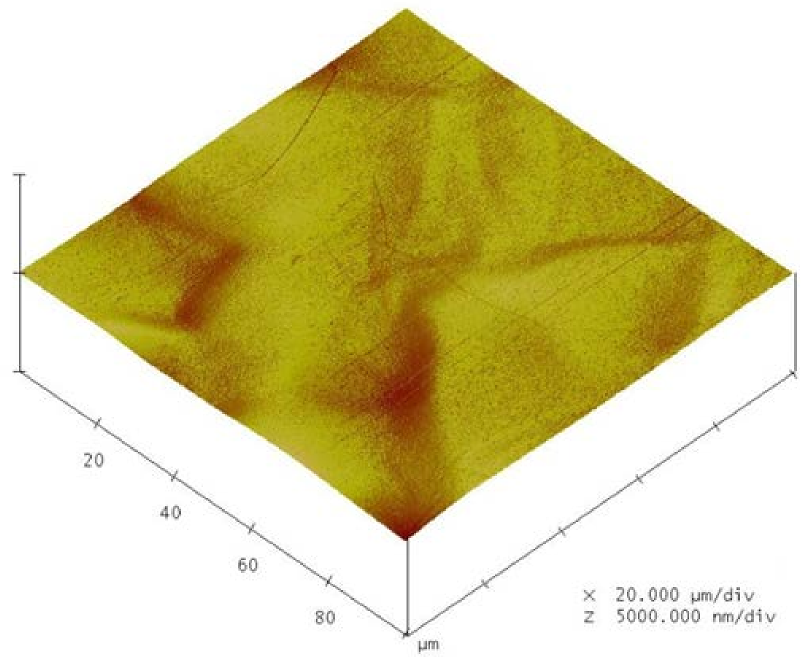
  ✓ Multiple nanoremoval by HF and oxypolishing indicate that the thickness, *t*, of Nb affected by the HT is 120 nm < *t* < 1 um



P. Dhakal et al, TUIOC04, SRF 13

BCP vs EP Topography

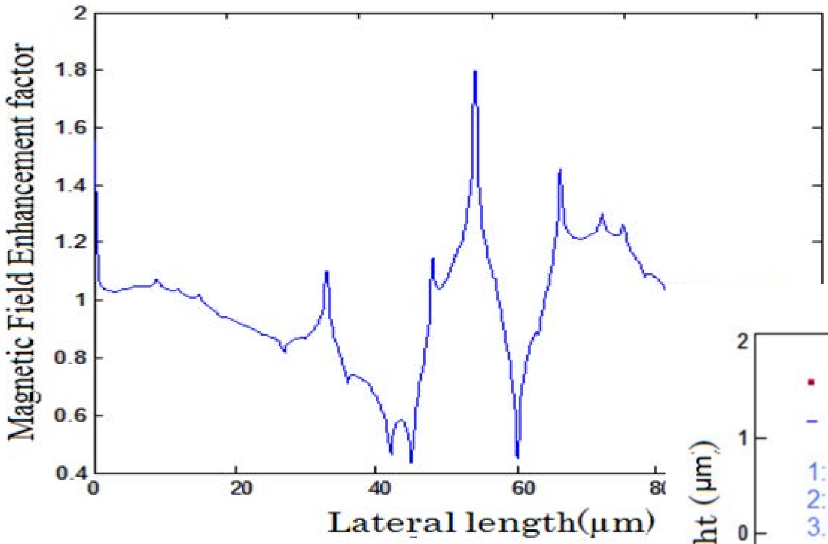


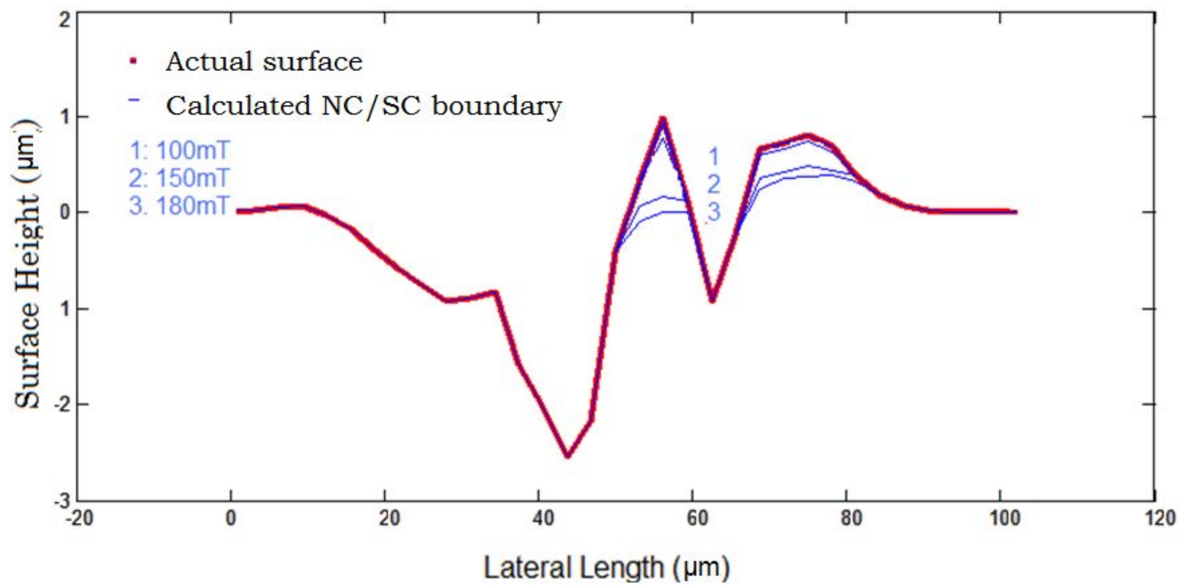


AFM images from a fine grain niobium sample with a) ~ 100μm removal by BCP, b) after EP to remove 48 μm. Horizontal scale is 20 μm per division and vertical scale is 5 μm per division.

PRST-AB 14, (2011) p.123501

FG BCP Topography



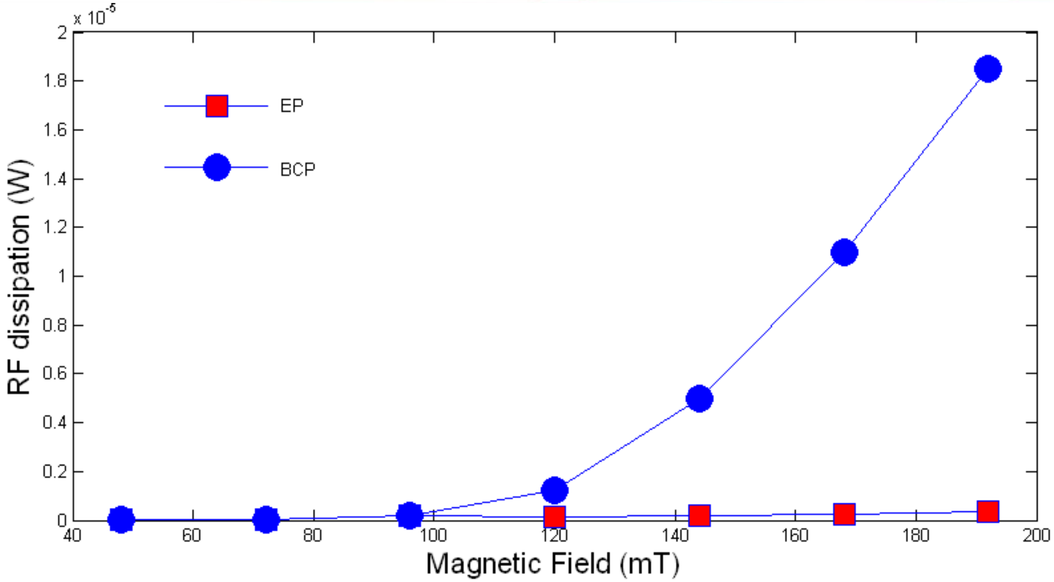


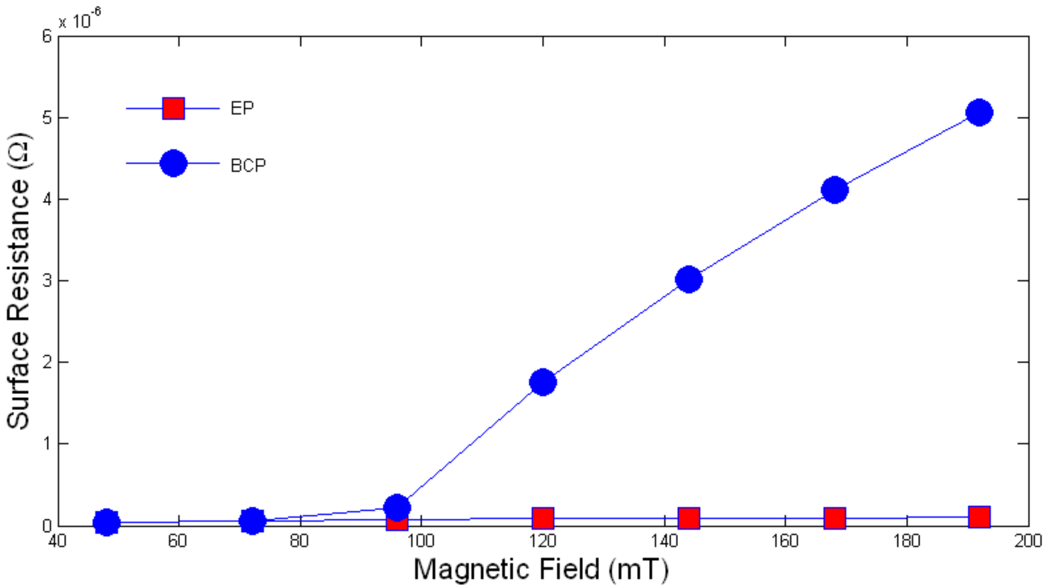
Treat the volume where $H > H_c$ as normal material, but $T < T_c$.
Depth is less than normal skin depth.
Thermally stable because very small local volumes.

IPAC12 Chen Xu *et al*.
SRF2013 Chen Xu *et al*.

Effective R_s due to BCP and EP Topography

- Average the dissipated power over representative 100 × 100 µm surface
- Convert to effective surface resistance with applied H
- Repeat with EP-derived topography
- Model neglects increased R_s of SC material with increasing *T*

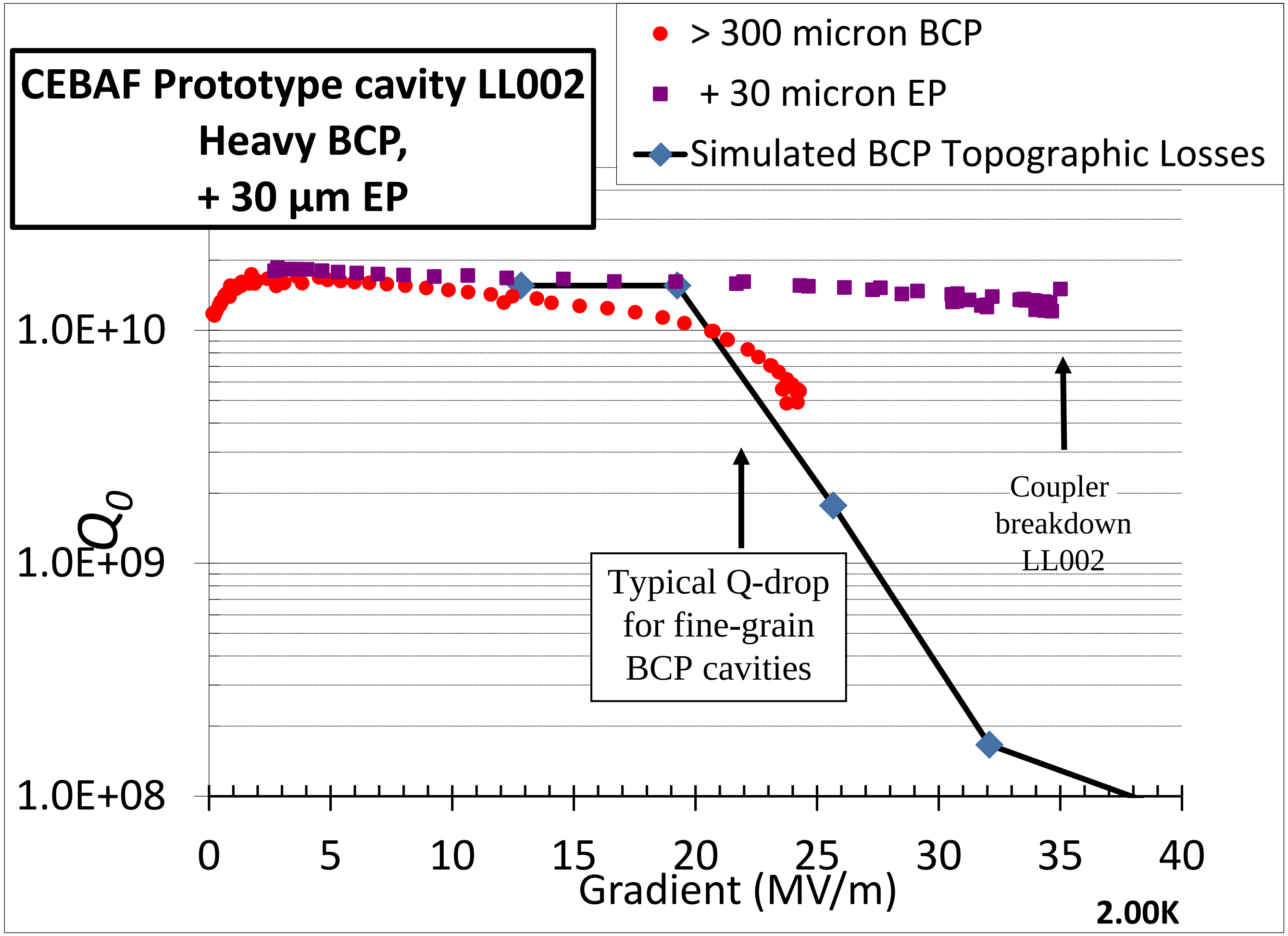




Chen Xu, to be published

Effective R_s due to BCP Topography

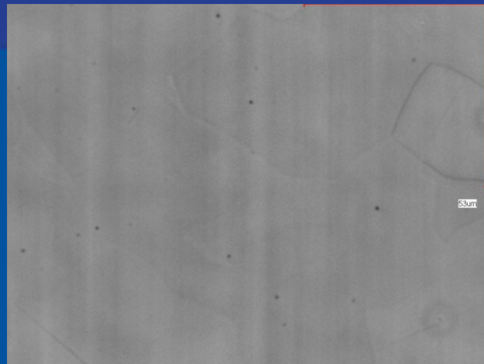
- Compare with BCP vs EP performance of a JLab cavity
- Analysis can be improved by using a finer mesh.
- In qualitative agreement with experimental experience.



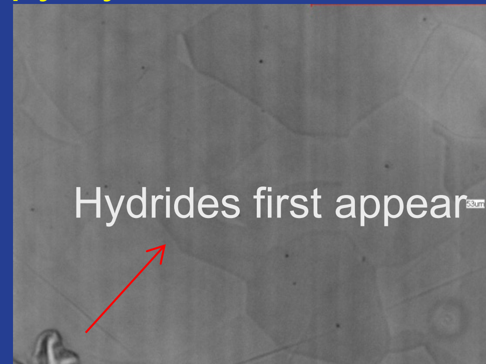
Jefferson Lab

Cryogenic Laser Confocal Microscopy by A. Romanenko et al: First cooldown

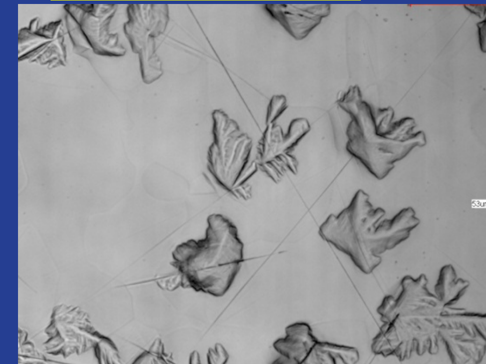
50 um



T=300K

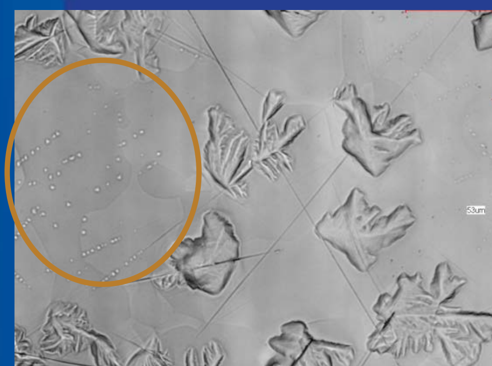


T=160K

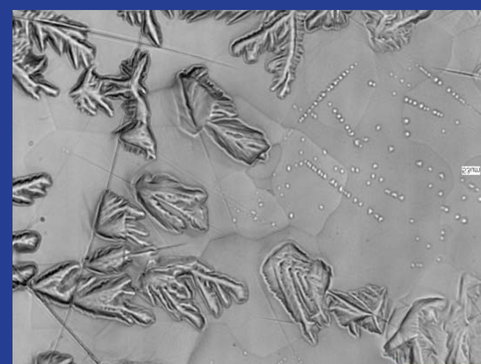


T=140K

Second (smaller) phase of hydride forms



T=100K

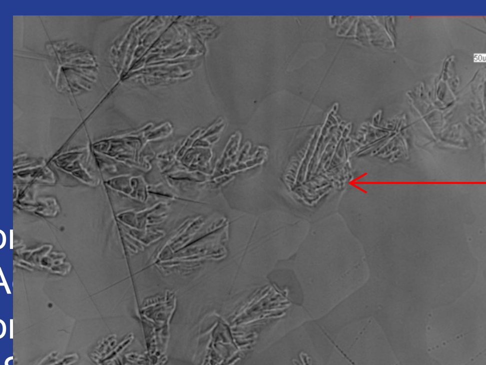


T=6K

T=180K

Large phase starts to dissolve

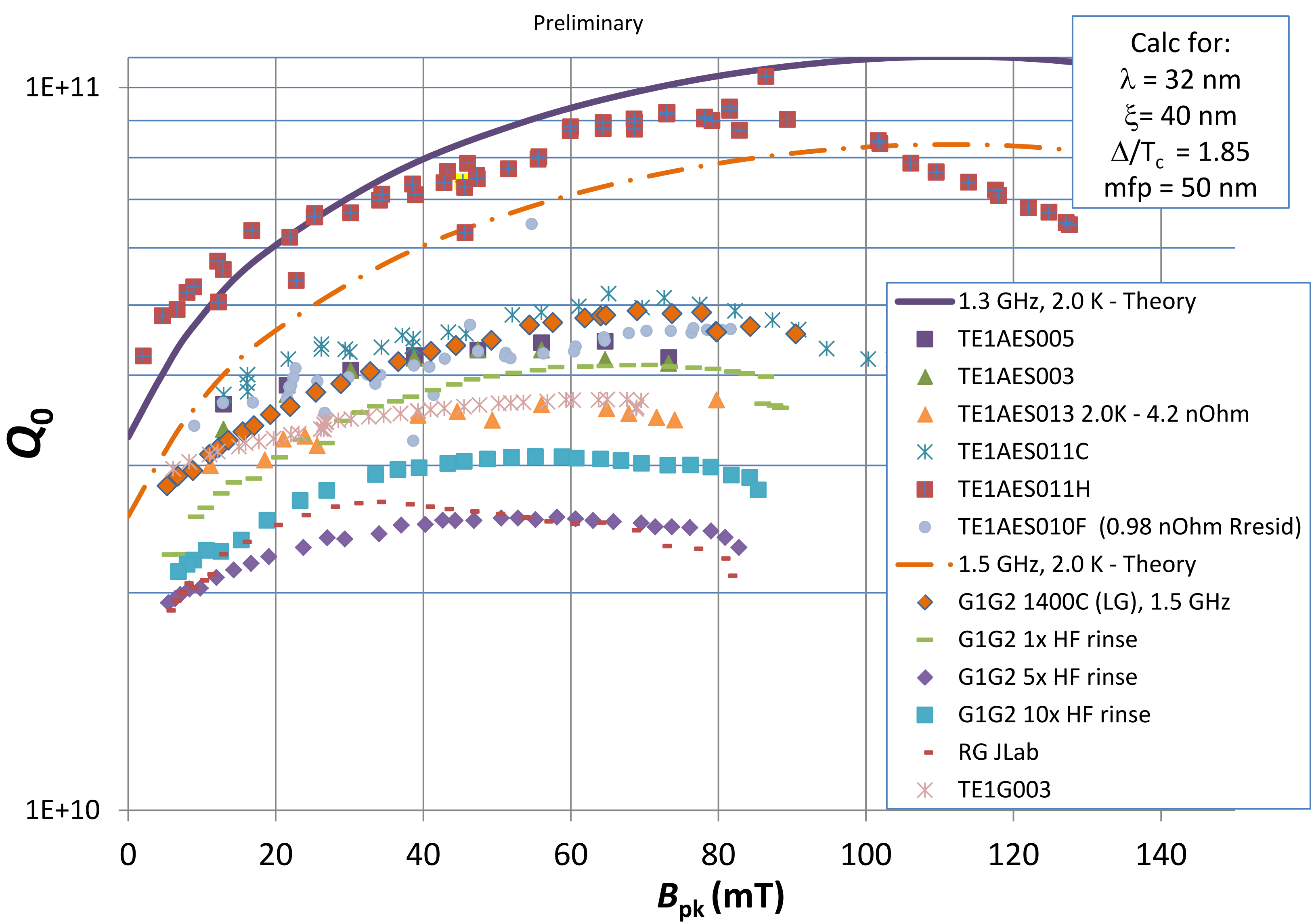
T=210K



T=260K

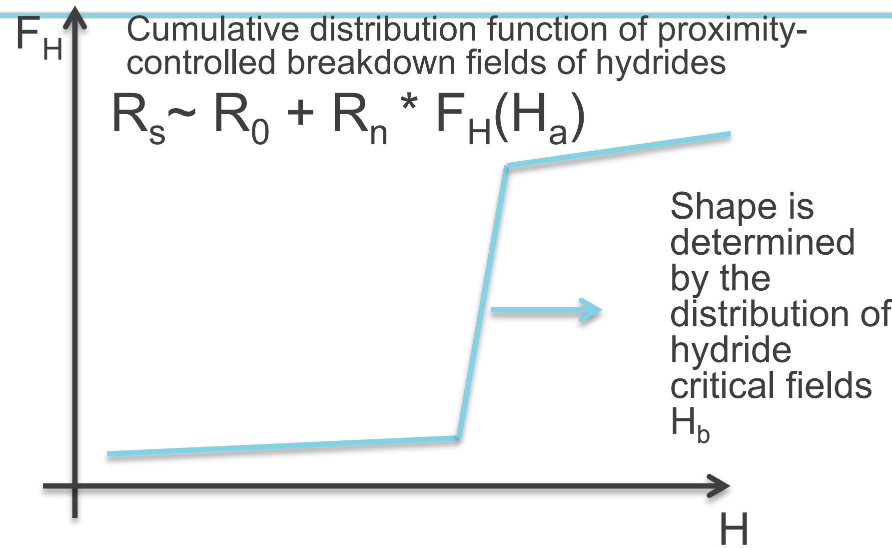
Hydrides gone, dislocation skeleton (deformation) remains on the surface

Pursuit of High Q_0 via HT-N

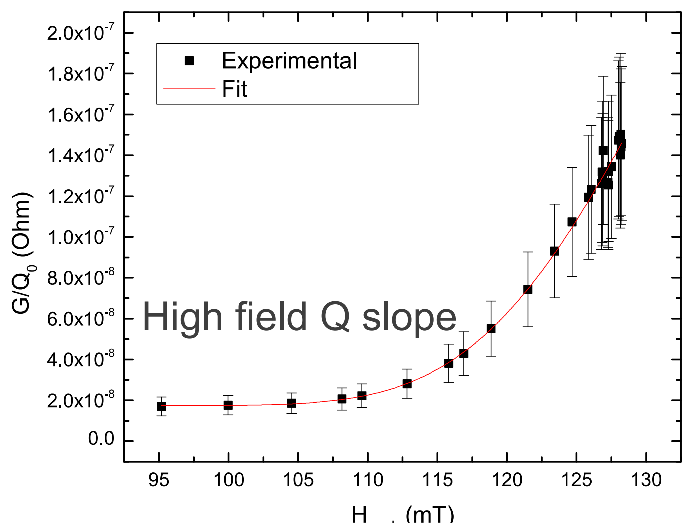


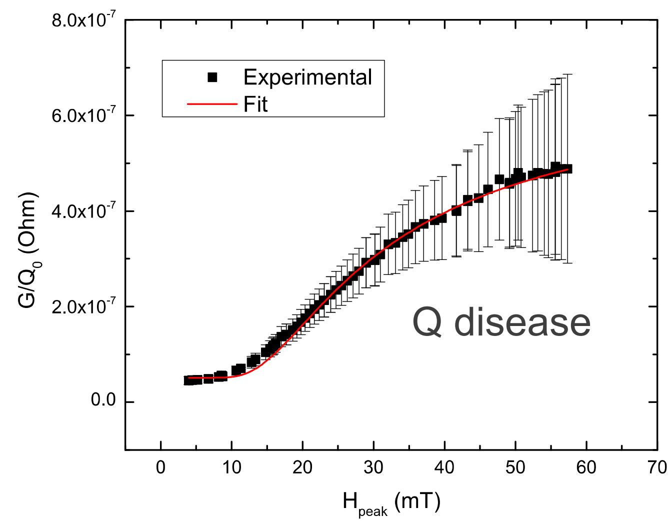
Proximity effect model

- Normal conducting hydrides of size *d* are superconducting by proximity effect up to the field $H_b \sim 1/d$



- Excellent fits



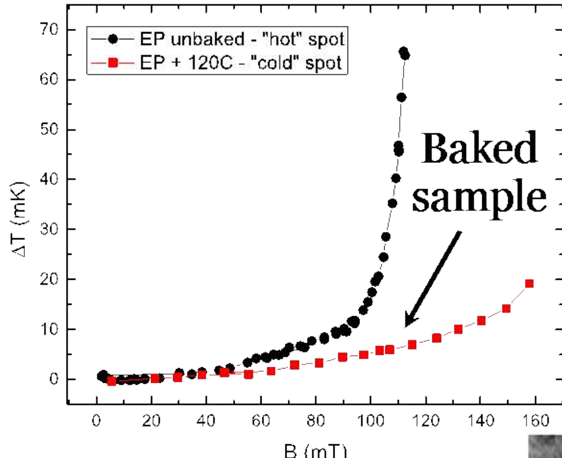


A. Romanenko, F. Barkov, L. D. Cooley, A. Grassellino, Supercond. Sci. Technol. 26 (2013) 035003

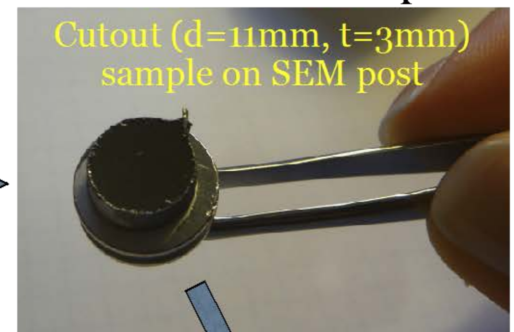
TEM evidence for nanohydrides

- Direct imaging of the cross-sections of cavity cutouts in cryo-TEM [see Y. Trenikhina et al, TUP043]

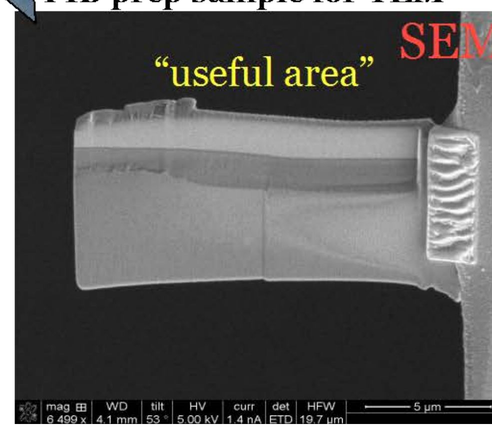
Cold: 120C in situ bake for 48hours
Hot: no such bake

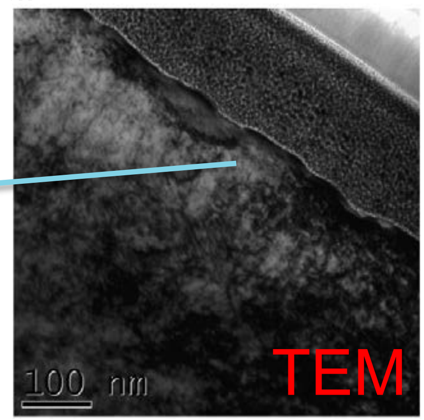


Heating: comparison of "cold" and "hot" spots



FIB prep sample for TEM





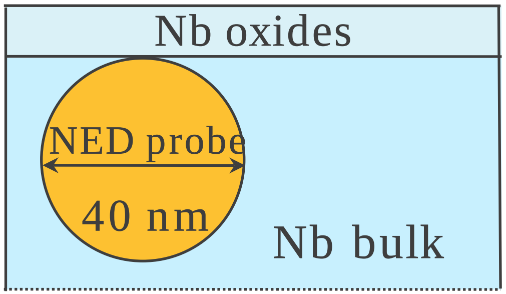
Look at this area with subnanometer resolution in TEM at room AND T<100K temperatures

See also R. Tao et al, J. Appl. Phys. **114**, 044306 (2013) and TUP042 for cryoimaging of H-reach Nb samples

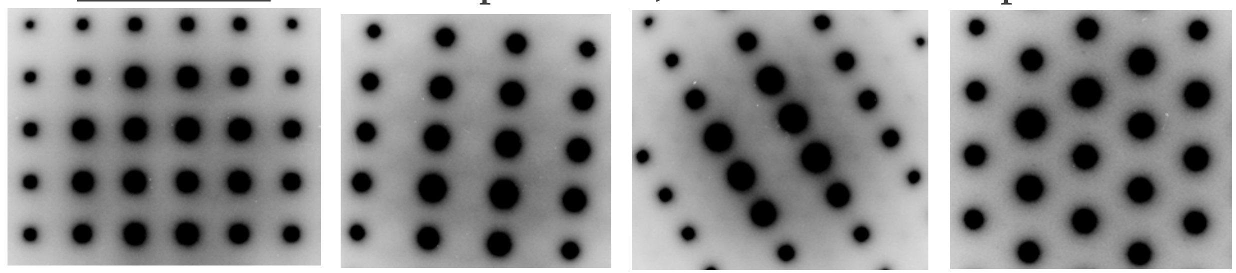
Investigation of HFQS mechanism.

1. T-dependent TEM studies of hot and cold spots: NED

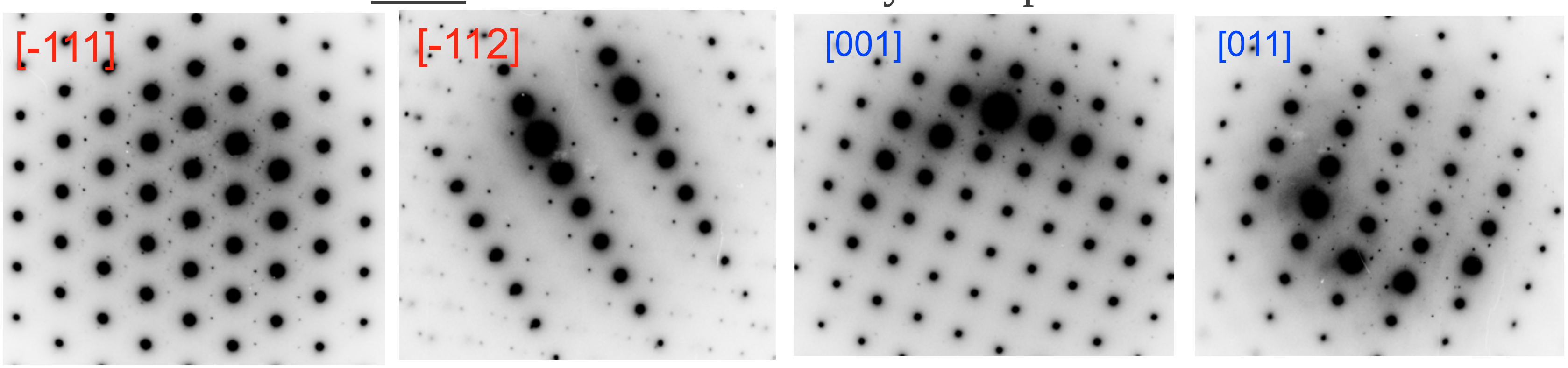
Direct **nano-area electron diffraction (NED)** phase characterization of the surface of the SRF cavity cutouts before and after in situ mild vacuum bake at room temperature and at 94K.



Room T: BCC Nb patterns, **NO** additional phases



94K: stoichiometric Nb hydride phases!

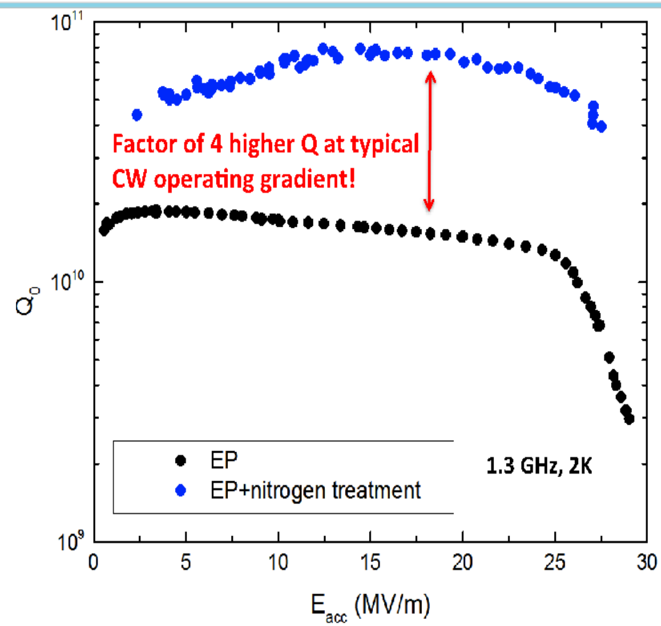


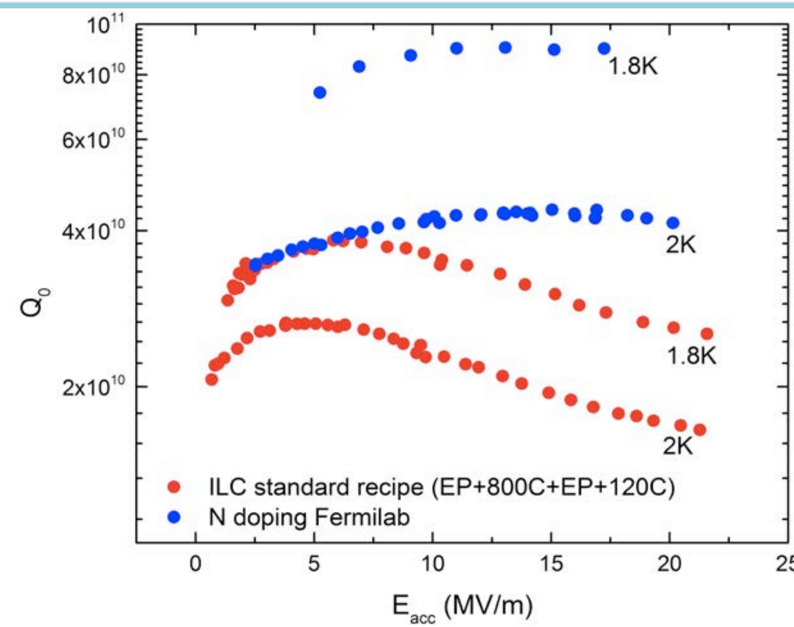
Hot spot: 44-68% of probed spots

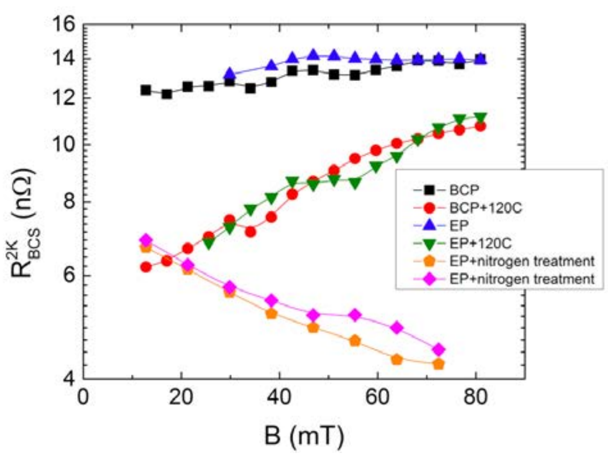
Baked spot: 26-29% of probed spots

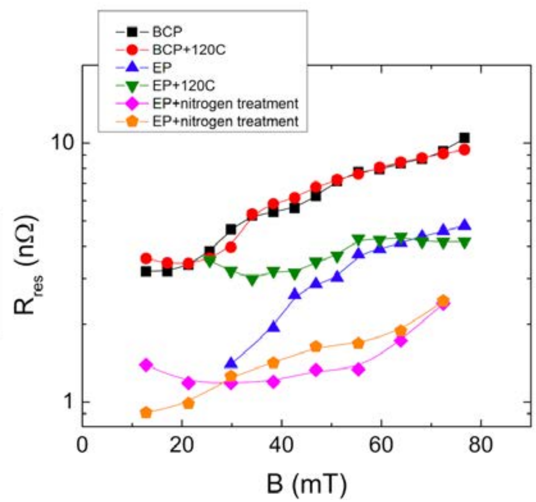
1. cryo-TEM demonstrate formation of **nanohydrides in SRF cavities**;
2. 120C bake appears to affect the number/size of the nanohydrides.

Nitrogen doping



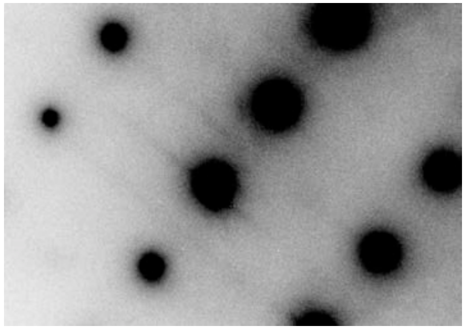




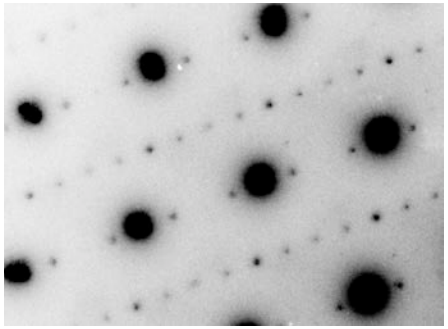


No nanohydrides found in nitrogen doped cutouts

2. T-dependent TEM structural characterization (NED) at room T and at 94K.



Room T and 94K: **NO** additional Nb hydride phases right underneath an oxide (within ~50 nm)!!!



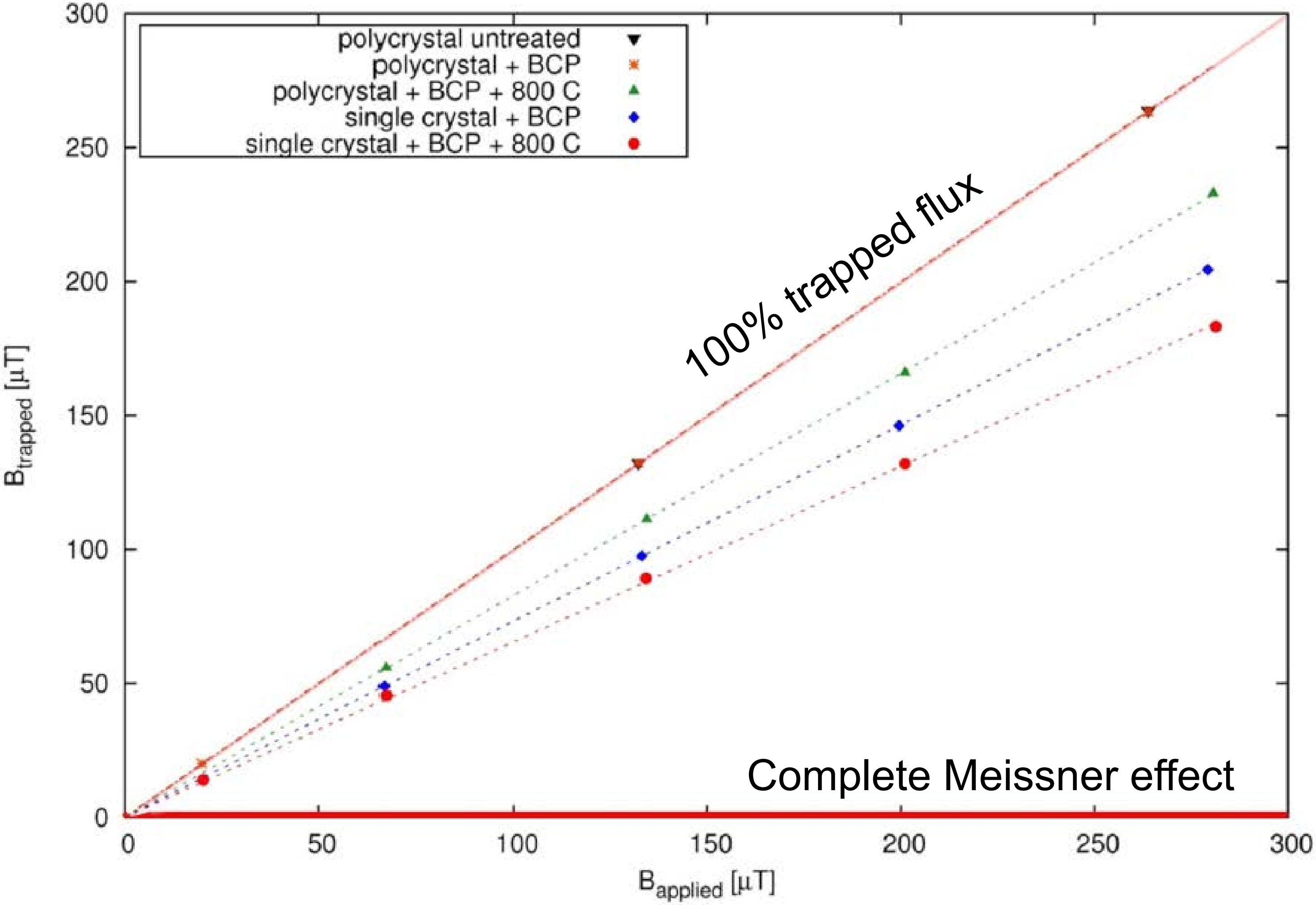
94K: stoichiometric Nb hydrides deeper into the bulk Nb! Further investigation is needed...

Influence of surface treatment on flux pinning

Oliver Kugeler (Diploma thesis Sarah Aull)

TTC meeting at DESY 24.3.2014 - 27.3.2014

RESULTS



polycrystal untreated
polycrystal + BCP
polycrystal + BCP + 800 C
single crystal + BCP
single crystal + BCP + 800 C
100% trapped flux
Complete Meissner effect
$B_{trapped}$ [μT]
$B_{applied}$ [μT]
0
50
100
150
200
250
300

RESULTS

Crystal structure	Treatment	Maximum applied field	Fraction of trapped flux
Polycrystalline		2.3 mT	100%
Polycrystalline	BCP	2.3 mT	100%
Polycrystalline	BCP+800°C	300 µT	83.1 ± 0.8 %
Single crystal	BCP	300 µT	73.3 ± 0.8 %
Single crystal	BCP+800°C	300 µT	66.9 ± 0.8 %

Interpretation of measurements:

- BCP has no influence on flux trapping
- 800°C bakeout reduces trapped field
- crystallinity has largest impact on flux trapping tendency

From a flux trapping perspective cavities should be made from single crystal material.

Pursuit of High Q_0 via HT-N

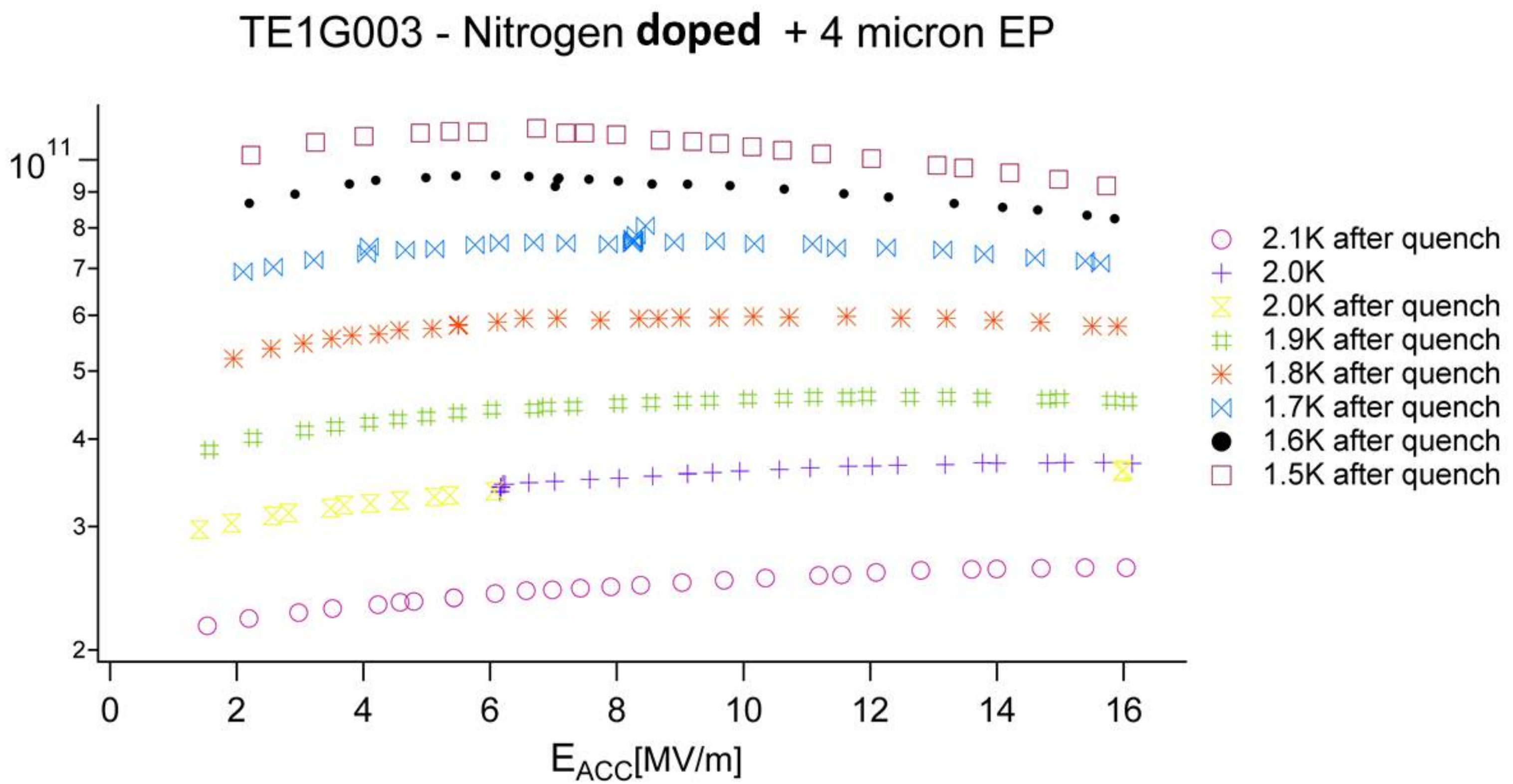
- JLab has launched a program to
  - **Establish the infrastructure** to process and test high Q_0 cavities – furnace upgrade, police test stand mag field
  - Perform a **structured N doping and EP removal study** in pursuit of a robust protocol – using 12 single-cell cavities
  - Help **debug extension of protocol to 9-cell cavities**
  - **Assess "robustness"** of candidate protocol to off-normal occurrences
  - With partners establish candidate LCLS-II protocol
  - Apply candidate protocol (frozen on July 1) **"production" style** to a set of 6 new 9-cell cavities
  - Complete by October 2014 !
  - Evaluate any **FG/LG differences** in high Q_0
  - Analyze and interpret the $R_s(T,B)$ from the data set

Pursuit of High Q_0 via HT-N

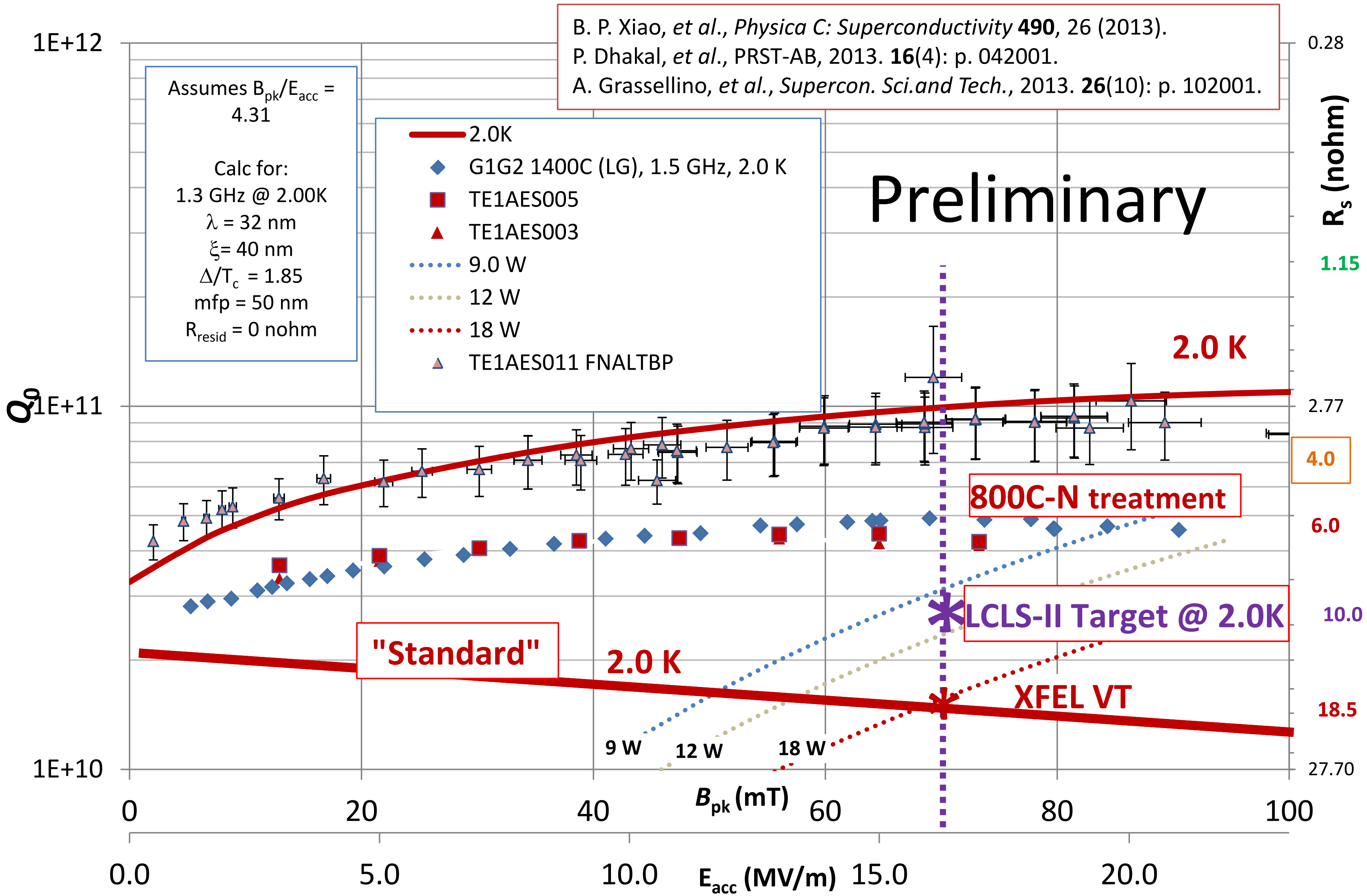
- As part of system commissioning, two of these cavities were HT-N treated in FNAL furnace and EP'd 4 µm at Jlab.
- One tested last week (FG), one to test this week (LG)
- First furnace commissioning run this week.

FG 1.3 GHz
CBP + BCP + HT-N + EP

Not bad for a "shake-down" test



Theoretical, Previous Standard, and Recent - Q_0 Limit @ 1.3 GHz, Tesla cell Shape



WG2b

By next TTC meeting:

A. Expect more (small) projects to adopt use of "ingot niobium" in order to obtain peak performance.
   A. E.g. compact accelerators and 3.9 GHz systems

B. Expect Q_0 of 3-4e10 @ 2K for 1.3 GHz @ 20 MV/m to be routine in labs and start of development at lower freq.

C. Expect new tougher constraints on test hardware and cooling conditions through T_c in order to minimize cavity residual resistance.