Cavity Heat Treatments

Ari Palczewski

USPAS Course:

SRF Technology: Practices and Hands-On Measurements

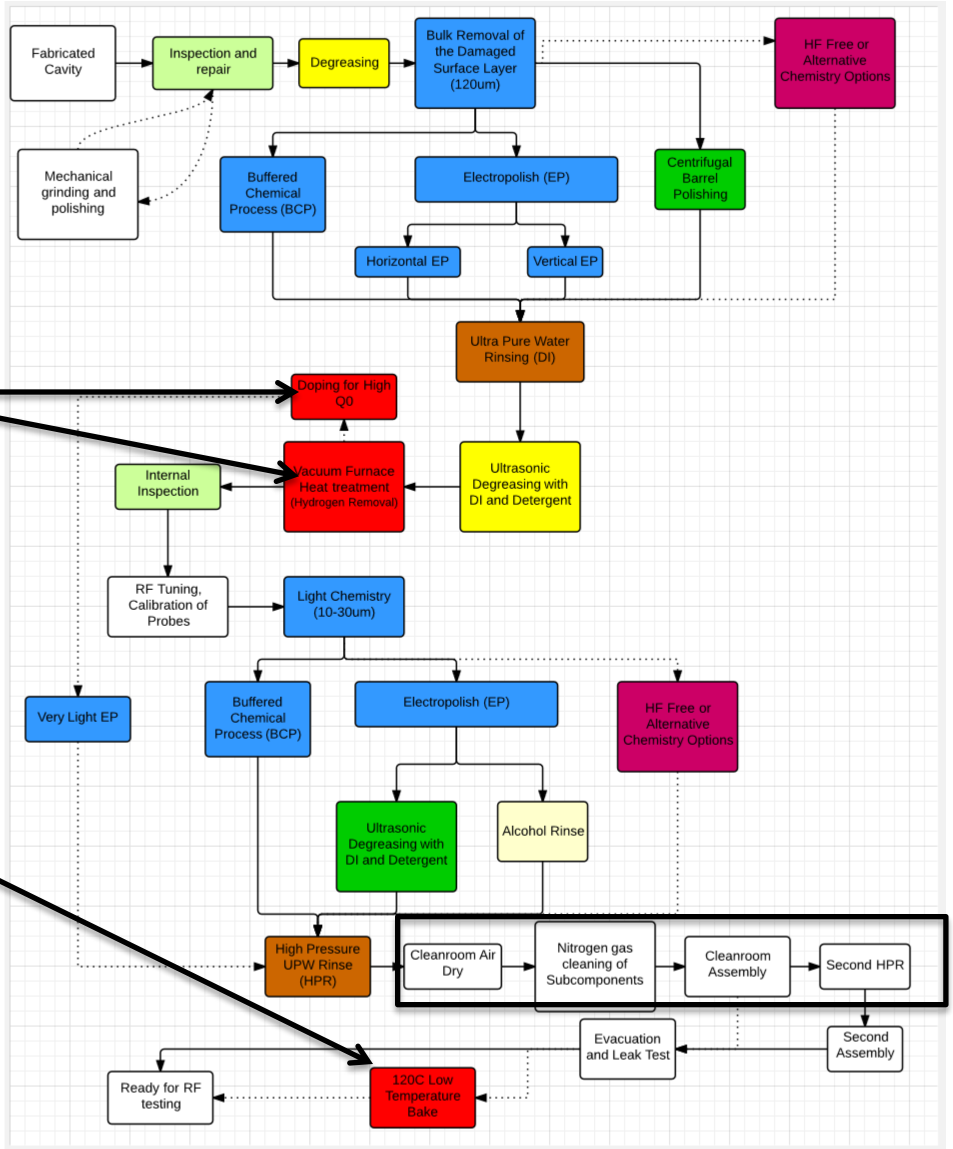
January 2015

Cavity preparation – heat treatments

Back to John's Talk – 9T SRF cavity preparation

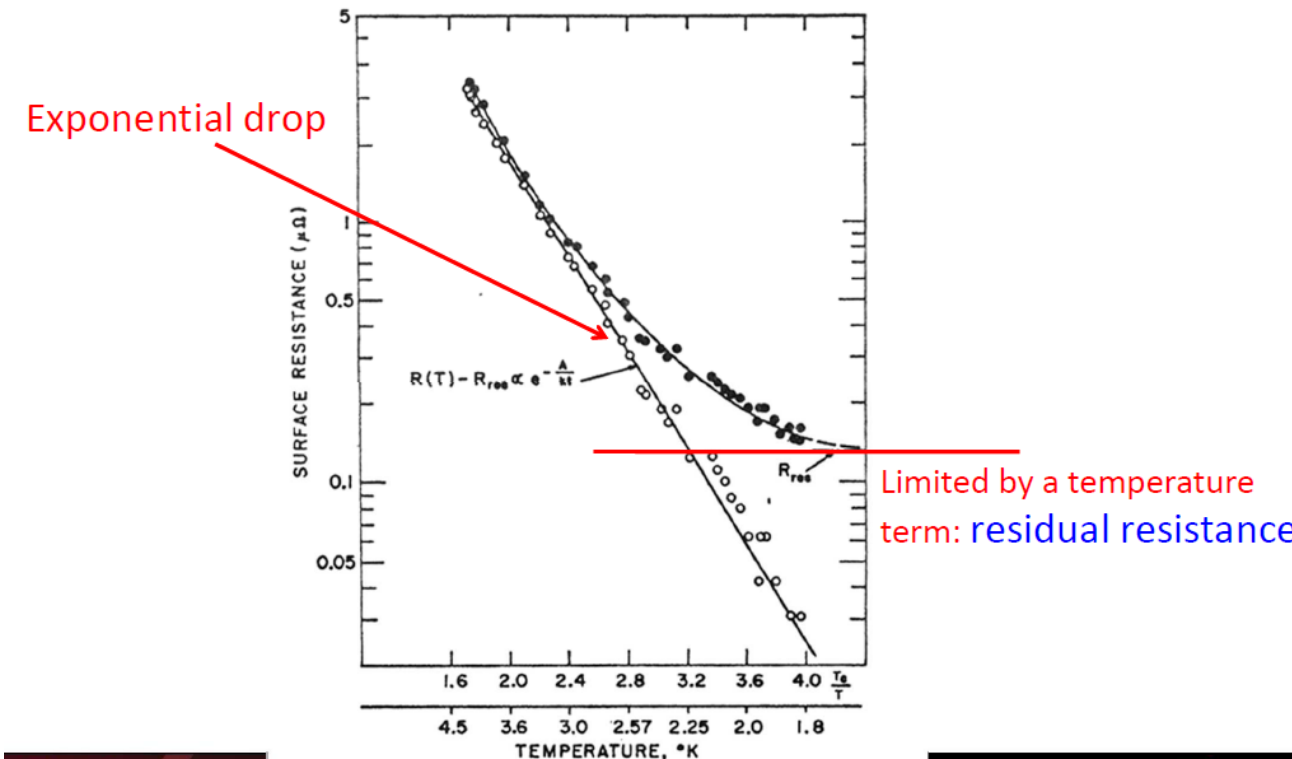
Furnace treatment either with nitrogen doping or without

120C bake



Reminder from Geng T1 and T3 - losses

Losses in Superconductor: measurements



We will see how the BCS as well as residual terms are effected by different heat treatments

Exponential drop part is what we call "BCS" resistance – it is the temperature dependant part

Residual term is not temperature dependant.

Heat treatments

Cavity Bakeing

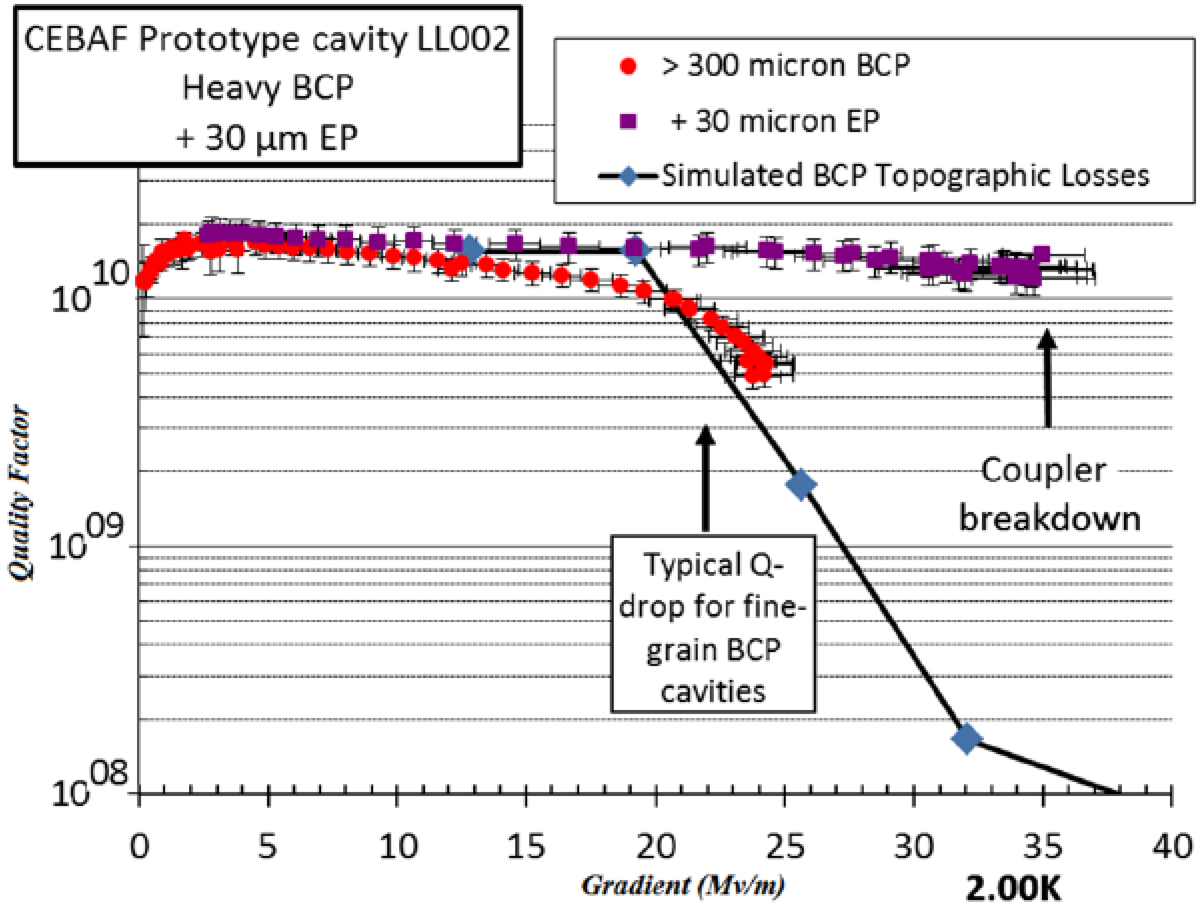
- Temperatures from 50C to 300C – usually 120C
- Usually performed after final chemistry
- Done on test stand while the cavity is the vacuum vessel
- Used primarily to remove high field Q-slope – and enhance Q0 @ 2k in certain cavities
- Used to removing residual water in cavity
- Used to reduce multi-packing by changing secondary yield coefficient

Cavity furnace/heat treatment

- Temperatures between 400C to 1800C (600C to 1400C modern)
- Usually done before final chemistry
- Usually with cavity open is large vacuum furnaces
- Primarily to remove hydrogen from manufacturing (welding and bulk chemistry)
- Sometimes used to purify niobium (T>1000C)
- Sometimes used to "Soften" niobium (large grain stress from stamping)
- Used to dope cavities with Nitrogen and Titanium for high Q0

“Bakeing”

From Reece 13T – topography not "Q-slope"



C. Xu, C. E. Reece and M. J. Kelley, "Simulation of non-linear SRF losses derived from characteristic Nb topography: comparison of etched and electropolished surfaces," *http://arxiv.org/abs/1406.7276*, 2014.

The BCP surface 600C for 10H, but still shows a Q-slope – this if from topography (mostly)

The Ep'ed surface was also baked @ 120C for 24 hours, removing the high field Q-slope

Q-slope and bake BCP - LG cavity

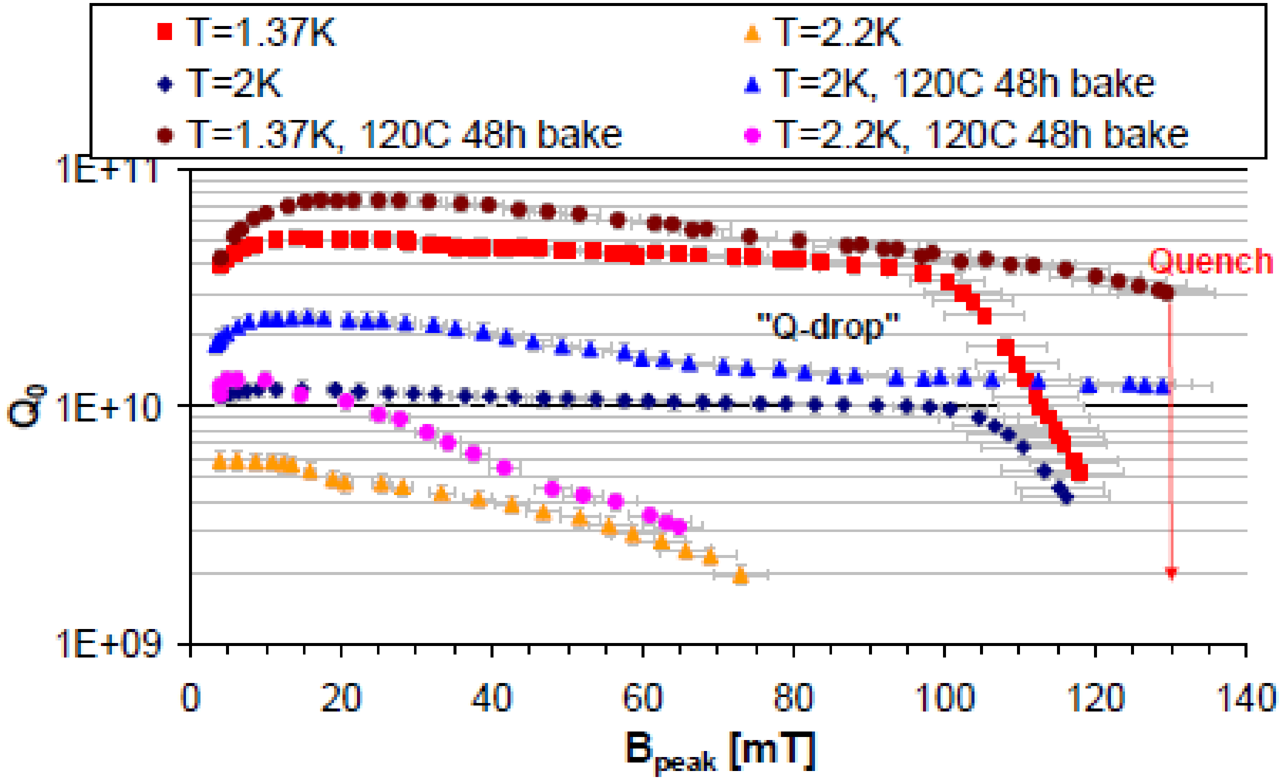


Figure 14: Q_0 vs. B_{peak} before and after 120°C, 48h baking.

Q-slope from BCP'ed cavity is because cavity was not baked, not a surface roughness like the fine grain cavity on previous slide

G. Ciovati et al. Effects of low temperature baking on niobium cavities
http://srf2003.desy.de/fap/paper/WeO14.pdf

“Residual” vs. “BCS” before and after bake

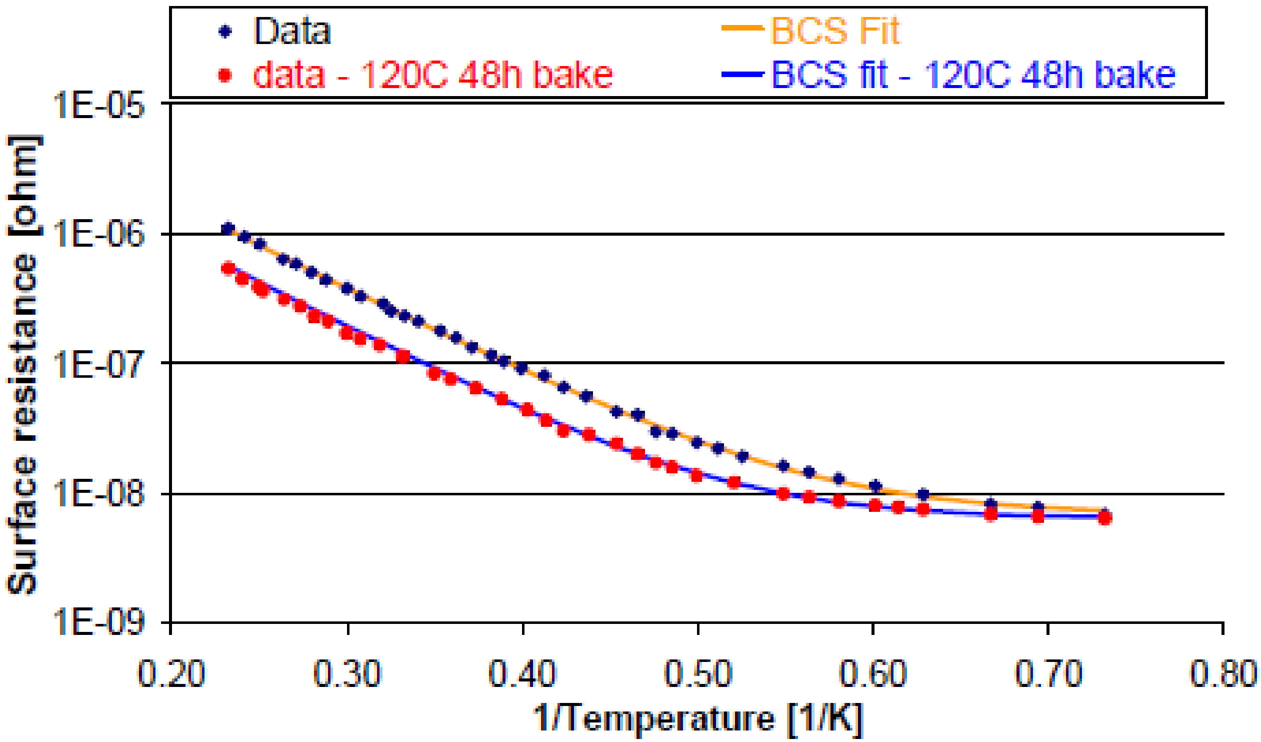


Figure 15: Surface resistance vs. 1/temperature before and after 120°C, 48h baking.

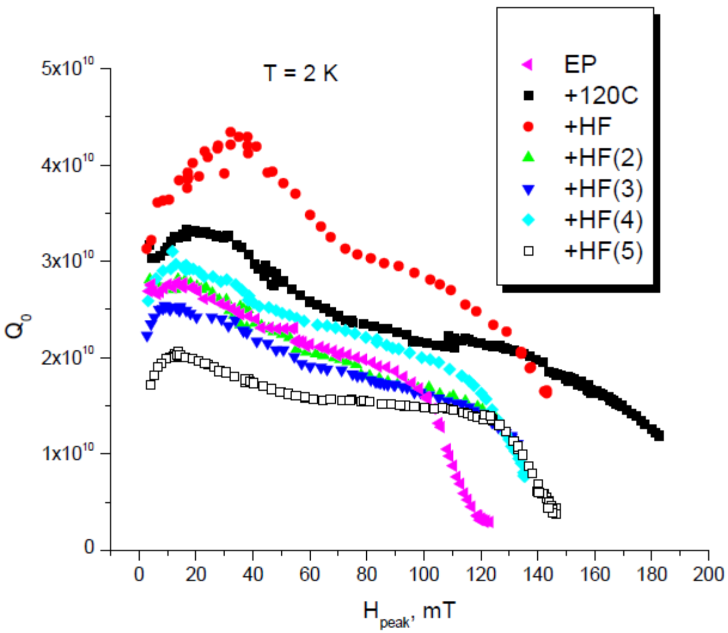
Cavity shown is Large grain from previous slide, but effect is the same for fine grain cavities

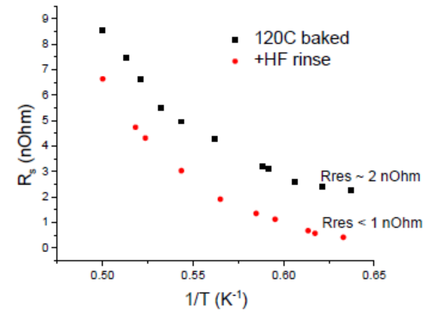
- BCS resistance goes down with bake
- Residual resistance goes up
- @ 2.0K and 1.3 to 1.5 Ghz Q0 goes up

G. Ciovati et al. Effects of low temperature baking on niobium cavities
http://srf2003.desy.de/fap/paper/WeO14.pdf

Side note – HF rinsing on baked cavities

Results on EP fine grain (tumbled)





- ✓ Single HF rinse after mild baking significantly improves medium field Q0
- ✓ Multiple HF rinse cycles do bring the high field Q-slope back
- ✓ Onset field is still higher than before baking by ~25 mT after total 5 HF rinse cycles
  - ✓ Further rinses in queue

HF rinsing does not change BCS term from 120C bake, but lowers residual term – Q0 in mid field goes up

2/6/12 A. Romanenko - All Experimenters Meeting 13

https://www.fnal.gov/directorate/program_planning/all_experimenters_meetings/special_reports/Romanenko_SCRF%20Cavities_02_06_12.pdf

Optimal temperature for bake – Large grain

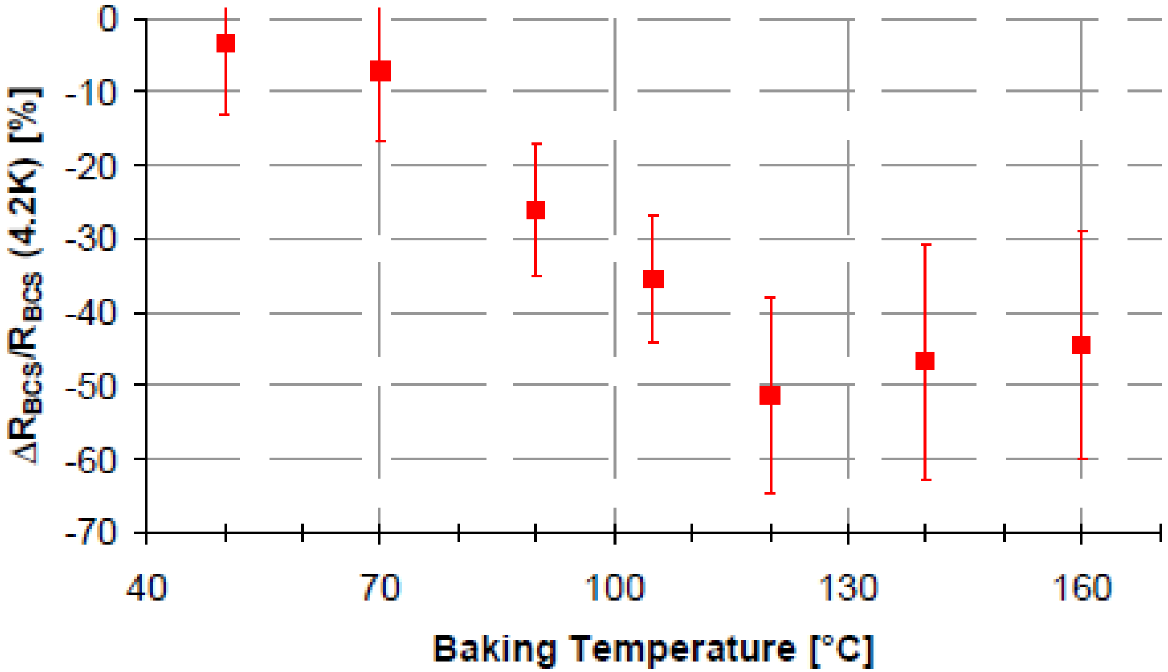


Figure 8: Variation of BCS surface resistance at 4.2K as a function of the baking temperature.

From coupons, BCS change is because mean free path changes, coupled to cavity data

120C to slightly above is the sweet spot for best Q0 @2K @~1.3 to 1.5

G. Ciovati et al. Effects of low temperature baking on niobium cavities
http://srf2003.desy.de/fap/paper/WeO14.pdf

Optimal temperature for bake – Large grain

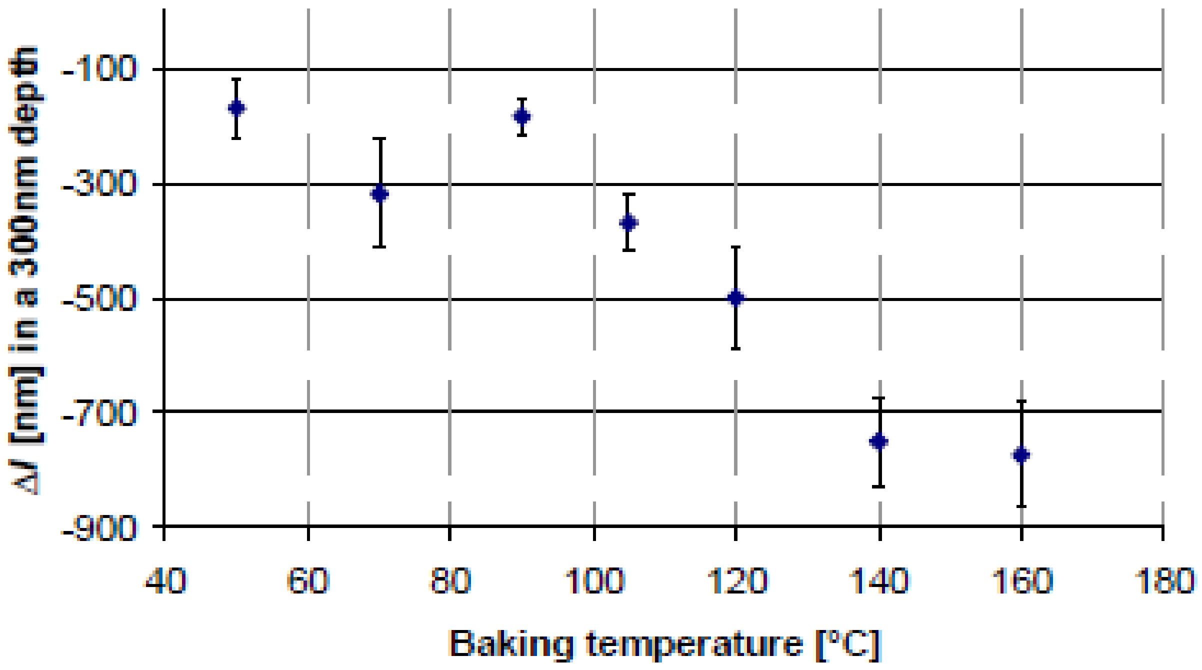


Figure 9: Variation of mean free path as a function of the baking temperature.

Baking changes the mean free path at the syrface

G. Ciovati et al. Effects of low temperature baking on niobium cavities
http://srf2003.desy.de/fap/paper/WeO14.pdf

Optimal temperature for bake – Large grain

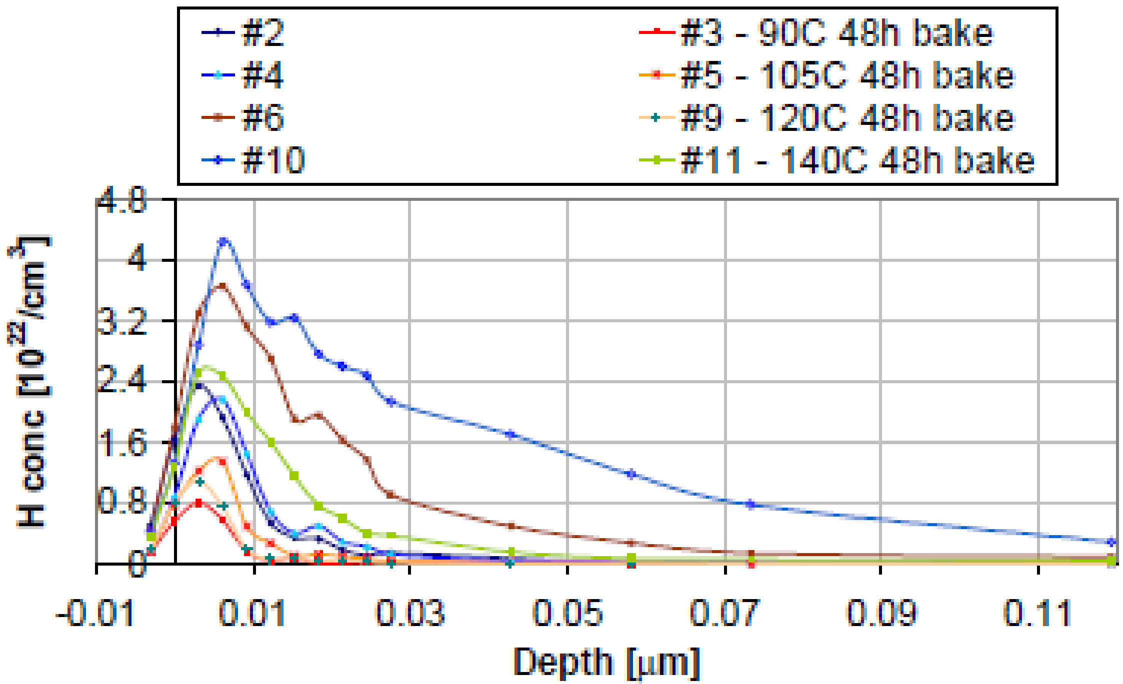


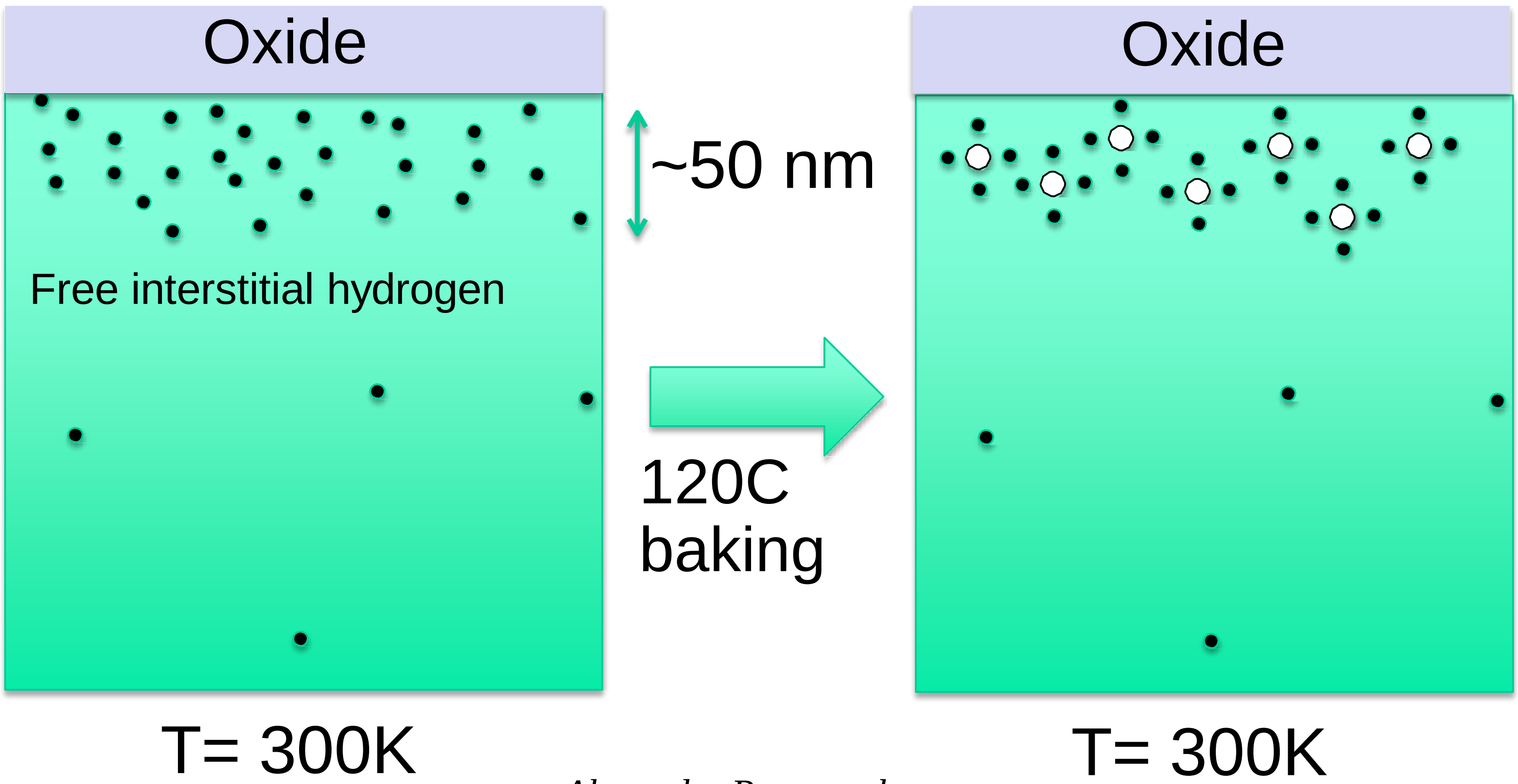
Figure 13: Hydrogen concentration vs. depth for samples baked and not baked.

Hydrogen content at surface is greatly reduced by bake, room temperature!

G. Ciovati et al. Effects of low temperature baking on niobium cavities
http://srf2003.desy.de/fap/paper/WeO14.pdf

120 C Baking Effect

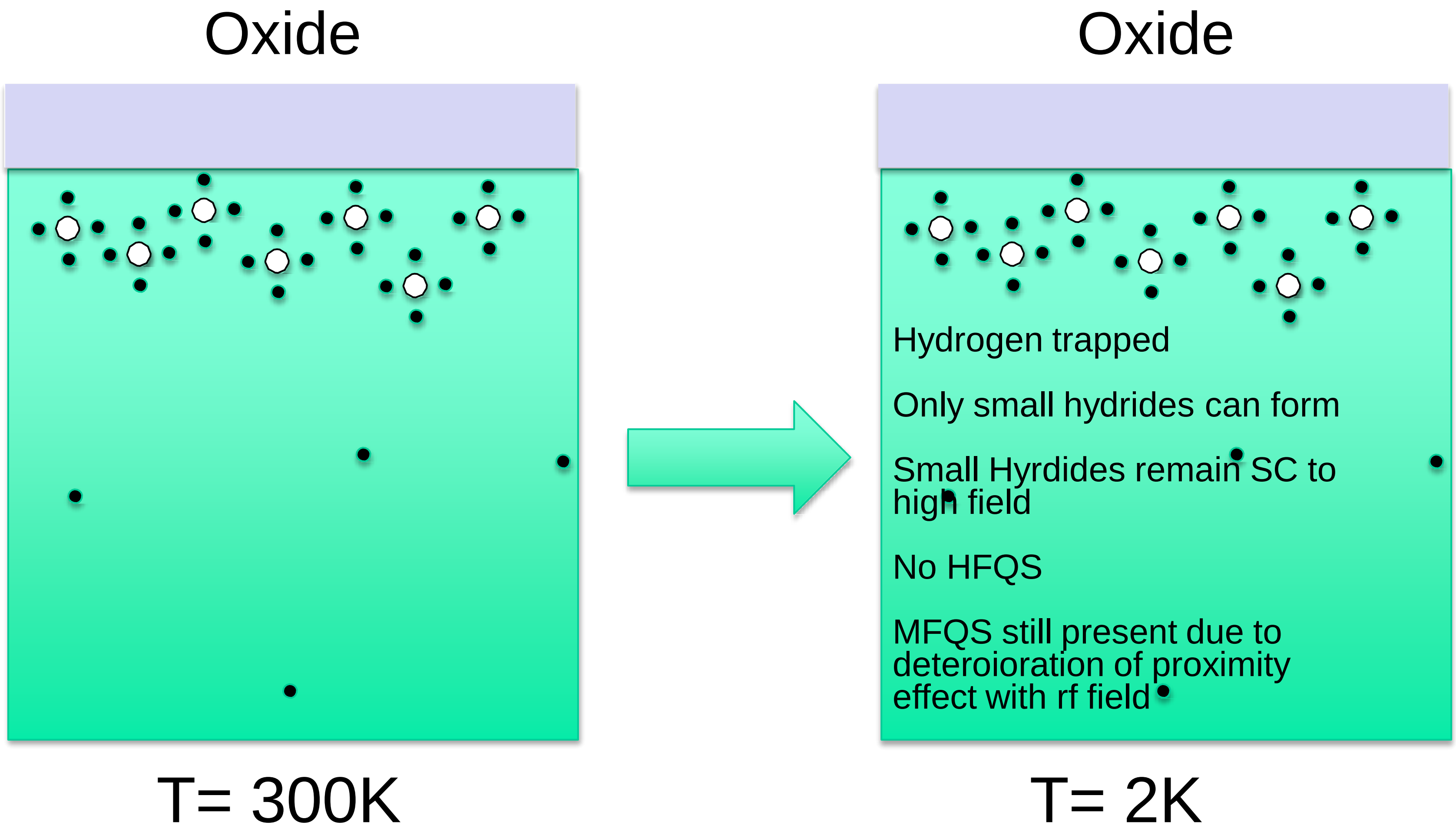
Vacancies trap H, Prevent Nb-H formation



Alexander Romanenko

A. Romanenko, C. J. Edwardson, P. G. Coleman, P. J. Simpson, Appl. Phys. Lett. **102**, 232601 (2013)

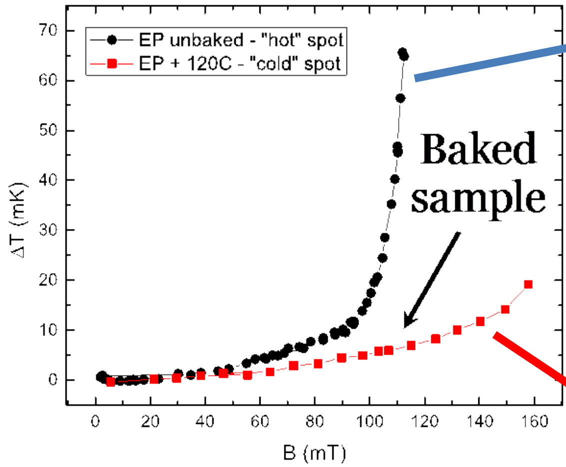
Cool down of 120C baked niobium

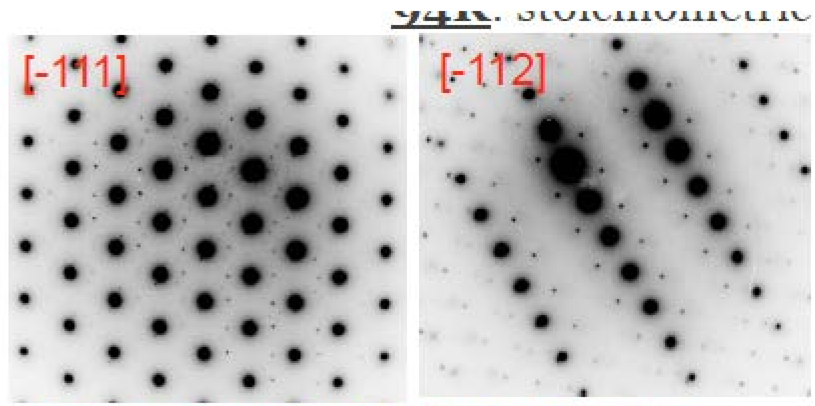


Alexander Romanenko

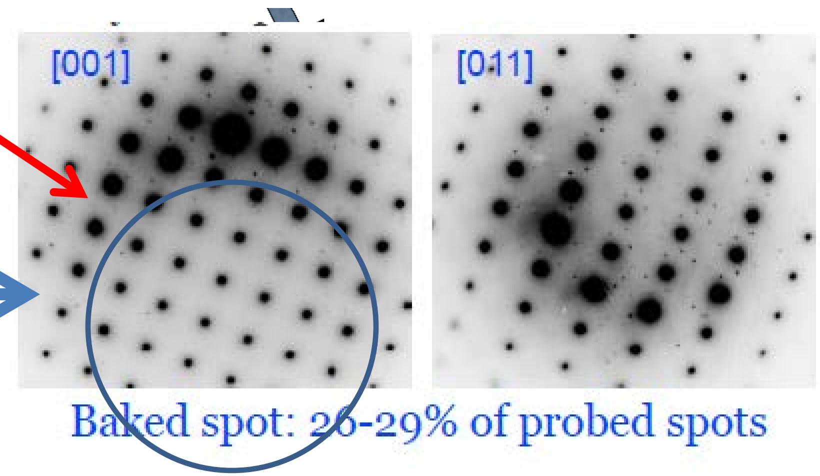
120 C Bake Inhibits Nb-H formation Romanenko (SRF 13)

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





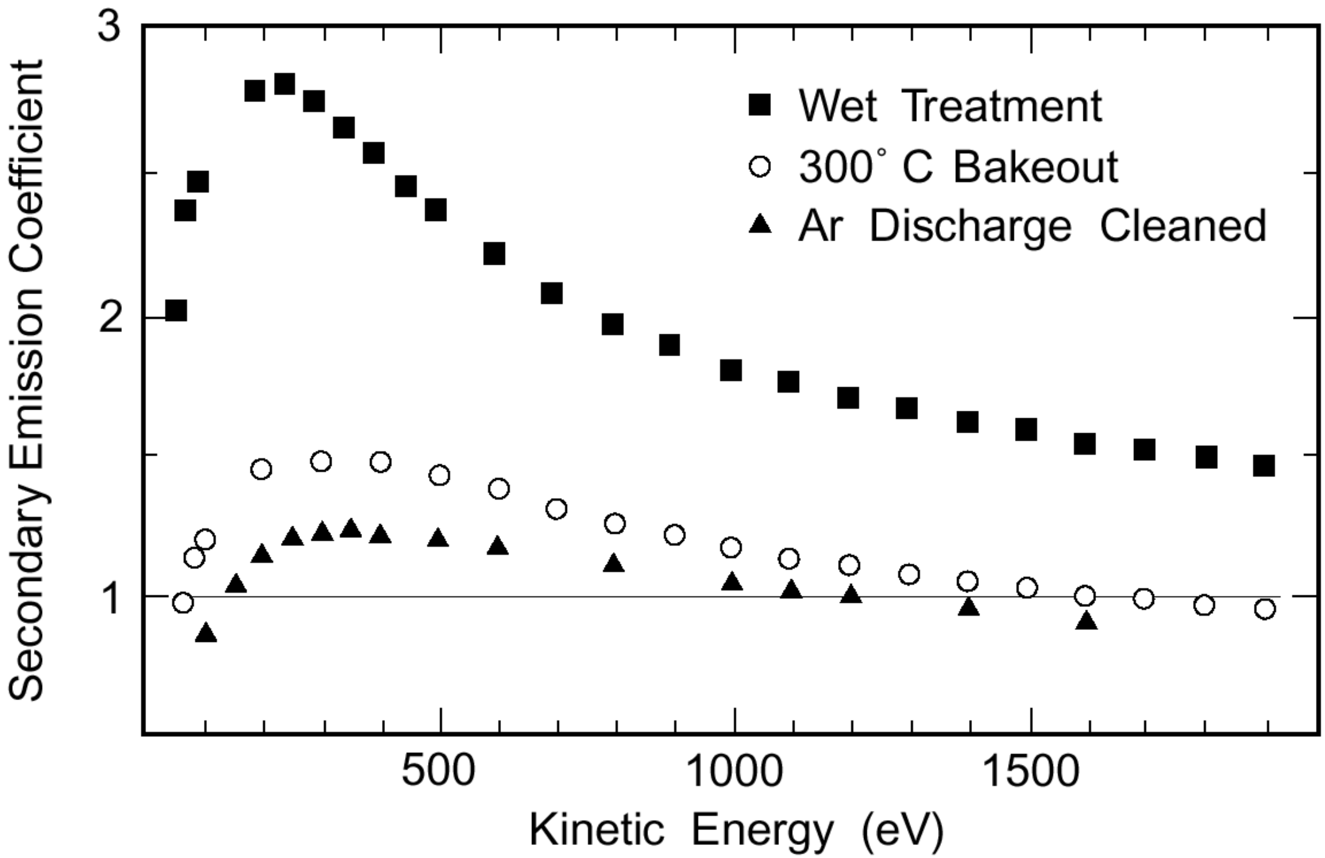
Hot spot: 44-68% of probed spots



Baked spot: 26-29% of probed spots

- Substantial reduction of Hydride formation after 120 C Bake

Multipacting reduction by bake



Baking can reduce secondary emission coefficient so that Multipacting is less prevalent – also has been shown to work for 120C

http://uspas.fnal.gov/materials/08UMD/SRF_Limitations.pdf

High Temperature heat treatments

1970s → ~1800 ^{o}C UHV HT for ~10 hrs.

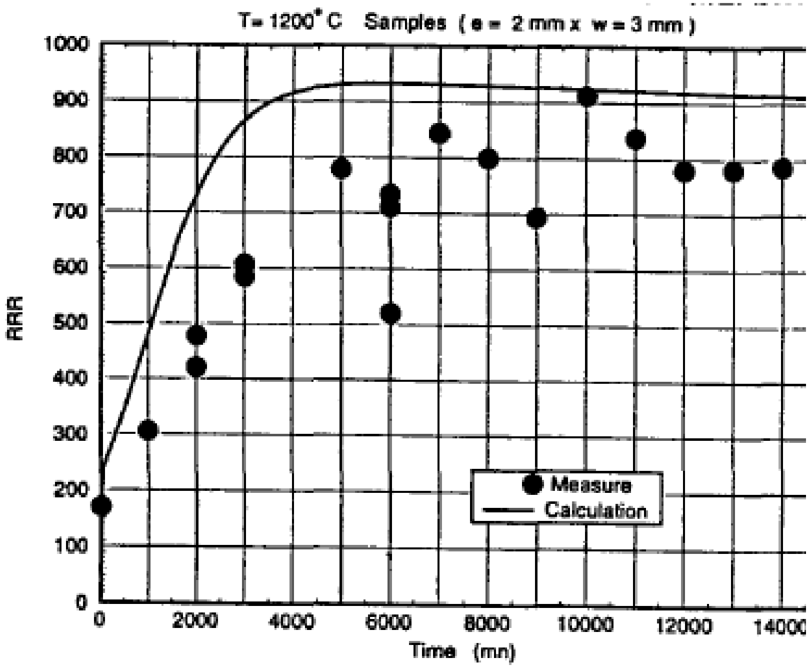
1980s → ~1300 ^{o}C solid state getter, such as Titanium,
was used in-side the furnace to "post-purify”.

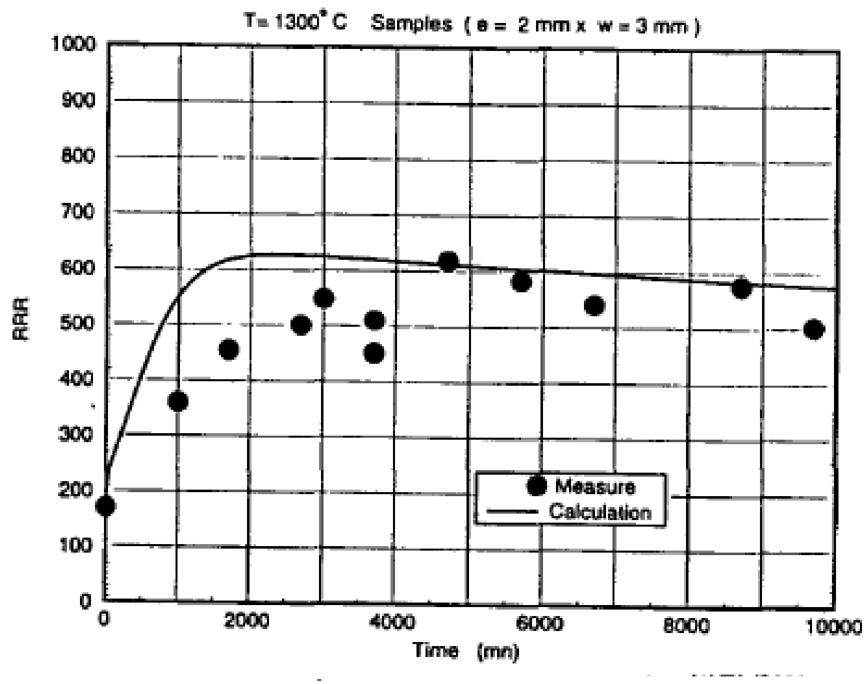
2000s → **600 10h -800 ^{o}C 2-3h**, mainly just to degas hydrogen
absorbed
by the Nb during cavity fabrication and surface
treatments.

2010’s →clean furnace studies from 600 to 1400C to reduce need for final chemistry

2012 → “doping” “polluting” “contaminating” cavity @ 800 to 1400C with titanium and Nitrogen – Extended Q-rise

Nb PURIFICATION by Ti GETTERING





Titanium gettering to improve RRR of cavities , remove impurities

H. Safa, Proceedings of the 1995Workshop on RF Superconductivity

Nb PURIFICATION by Ti GETTERING

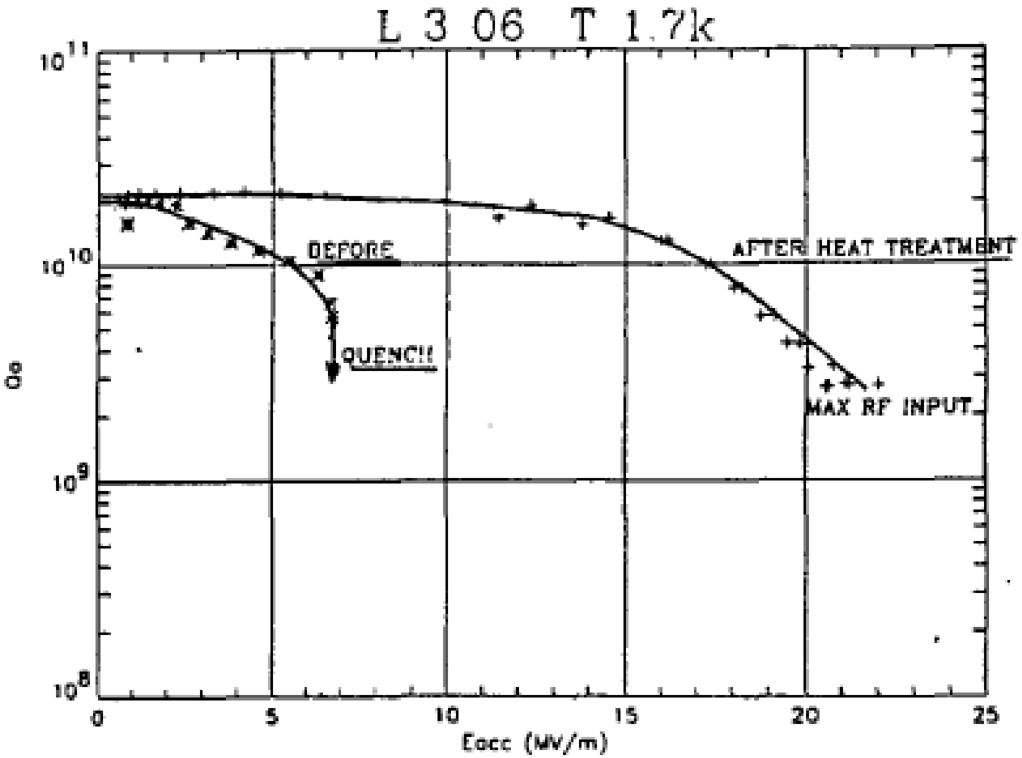


Figure 5 – A 3-cell 1.5GHz cavity having a quench at an accelerating field of 6.5MV/m due to an identified defect. After heat treatment, its quench level was pushed up higher than 22MV/m.

Gettering increases RR of cavity with also increases thermal conductivity y at low temperatures.

Improves quench field from localized defects

H. Safa, Proceedings of the 1995Workshop on RF Superconductivity

From Geng T1 and T3 – Q-Disease

Symptom of Residual Losses

- Q-disease
  - Q_0 at low field degrades when cavity parked at a temperature 70-150 K for extended period of time
  - Similar effect when cavity cool down rate is slower than 1K/min in passing 70-150 K

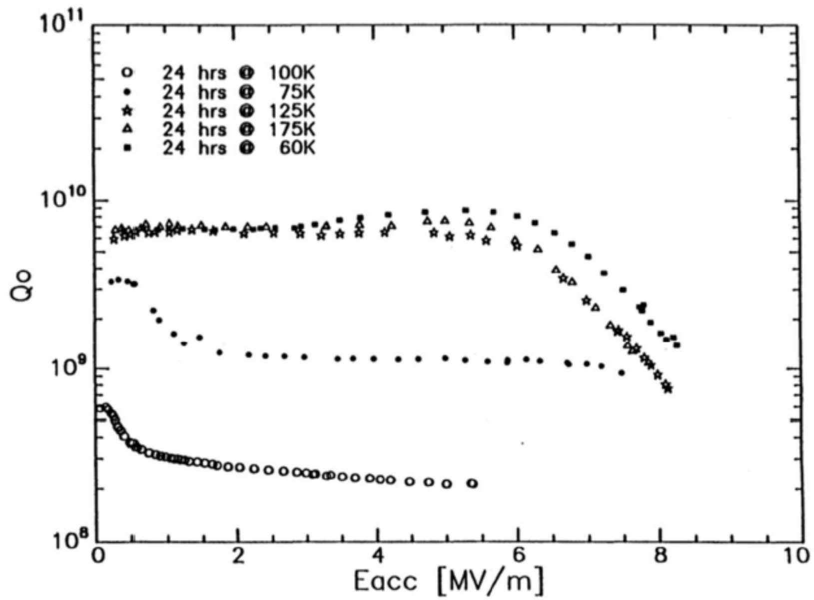


Figure 1 : Eacc – Dependence of Q – Degradation on "Holding" Temperature

J. Halbritter, P. Kneisel, K, Saito, SRF1993

Cavities which are not high temperature heat treated after heavy weld manufacturing, after bulk chemistry or mechanical polishing All show Q-Disease

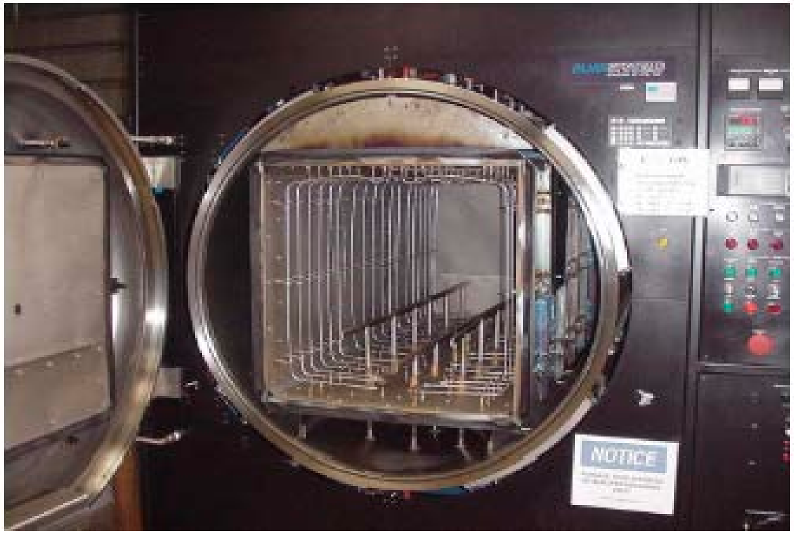
From Geng talk – minimize Q-disease

Overcoming Residual Losses

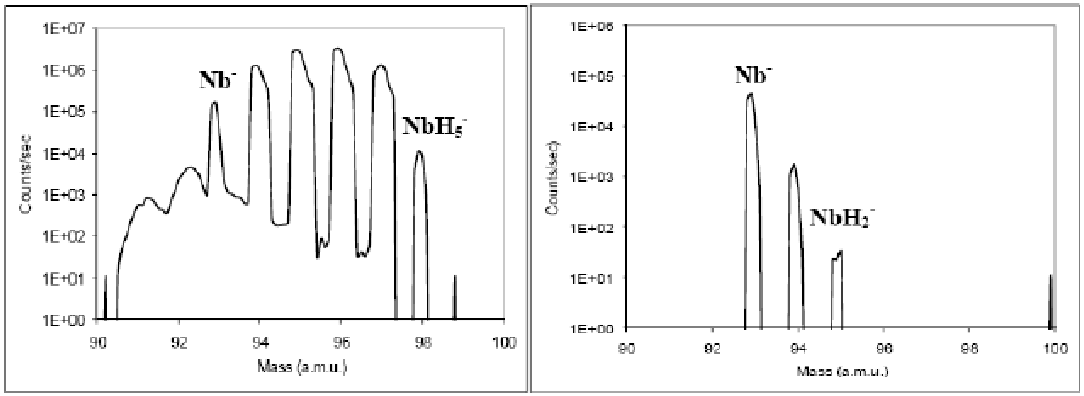
- Minimize H uptake from processing
  - BCP etching at < 15 ℃
  - “H free” EP
- Hydrogen out-gassing in vacuum furnace
  - 800 ℃ x 2hr
  - Or at lower temperature for longer time
- Minimum or no chemistry after out-gassing

R.L. Geng USPAS SRF Course Jan. 2015 120 Jefferson Lab

Standard (600-800 °C) Furnace Treatment



The standard furnace used for the high-temperature heat treatment of SRF cavities is an ultra-high-vacuum furnace with molybdenum hot-zone; molybdenum (or tungsten) resistive heating   elements and cavities are heated by radiation from the heating elements.



(a) (b)

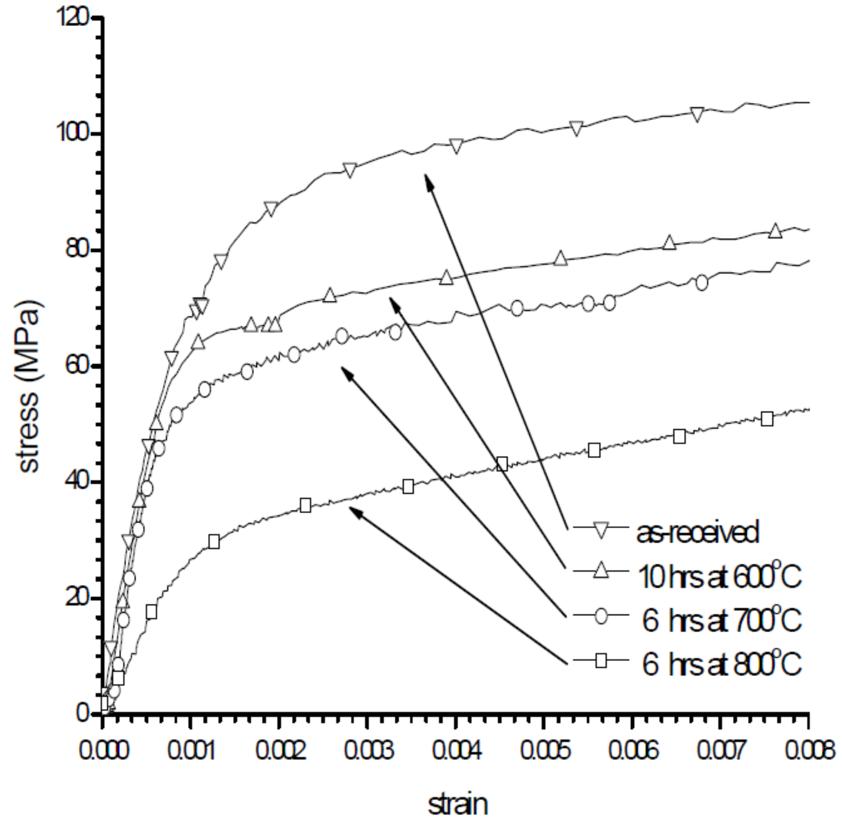
FIGURE 1. SIMS mass spectra showing difference in H between (a) non-heat treated and (b) heat treated sample.

High temperature annealing removes gross hydrogen

*Ciovati et al, PRSTAB **13**, 022002 (2010)*

Physical properties with heat treatment – FG 4K

Apparent YM Issue



Jefferson Lab

Thomas Jefferson National Accelerator Facility
Institute for SRF Science and Technology

SRF 2003 3 September '03

Operated by the Southeastern Universities Research Association for the U.S. Department of Energy

Talk G.R. Myneni - WEO11

Jefferson Lab

Physical properties with heat treatment – FG 4K

TD data summary

Summary of the TD niobium mechanical properties

Niobium	Yield Strength (KSI)		Tensile Strength (KSI)		% Elongation		RRR	Hv
	SSR	FSR	SSR	FSR	SSR	FSR		
ASR	7.4	7.9	21	24	44	48	260	52
600 C	7.0	7.5	21	22	48	49	300	47
800 C	5.7	--	19	--	47	--	350	43
1250 C	4.5	6.3	15	19	32	33	375	36

SSR ~ 5.5E-5

FSR ~ 2.0e-4 up to Yield point and 1.0e-3 until break

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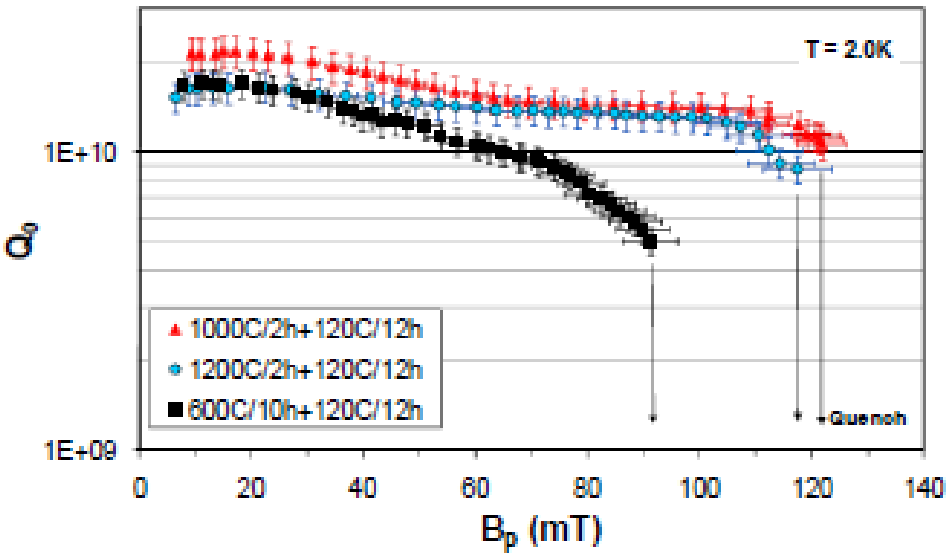
Talk G.R. Myneni - WEO11

No wet chemistry after heat treatment - JLab



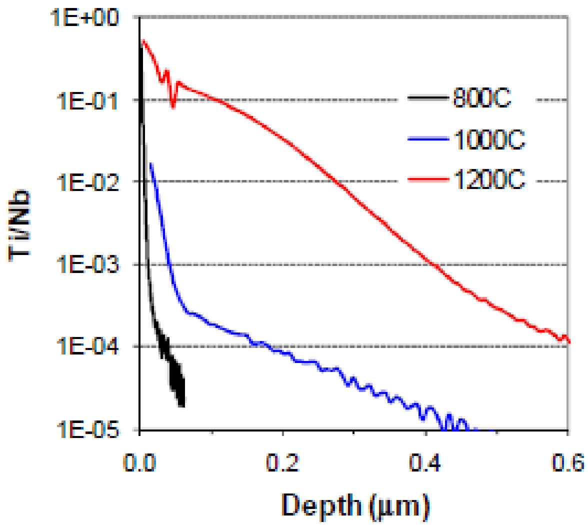
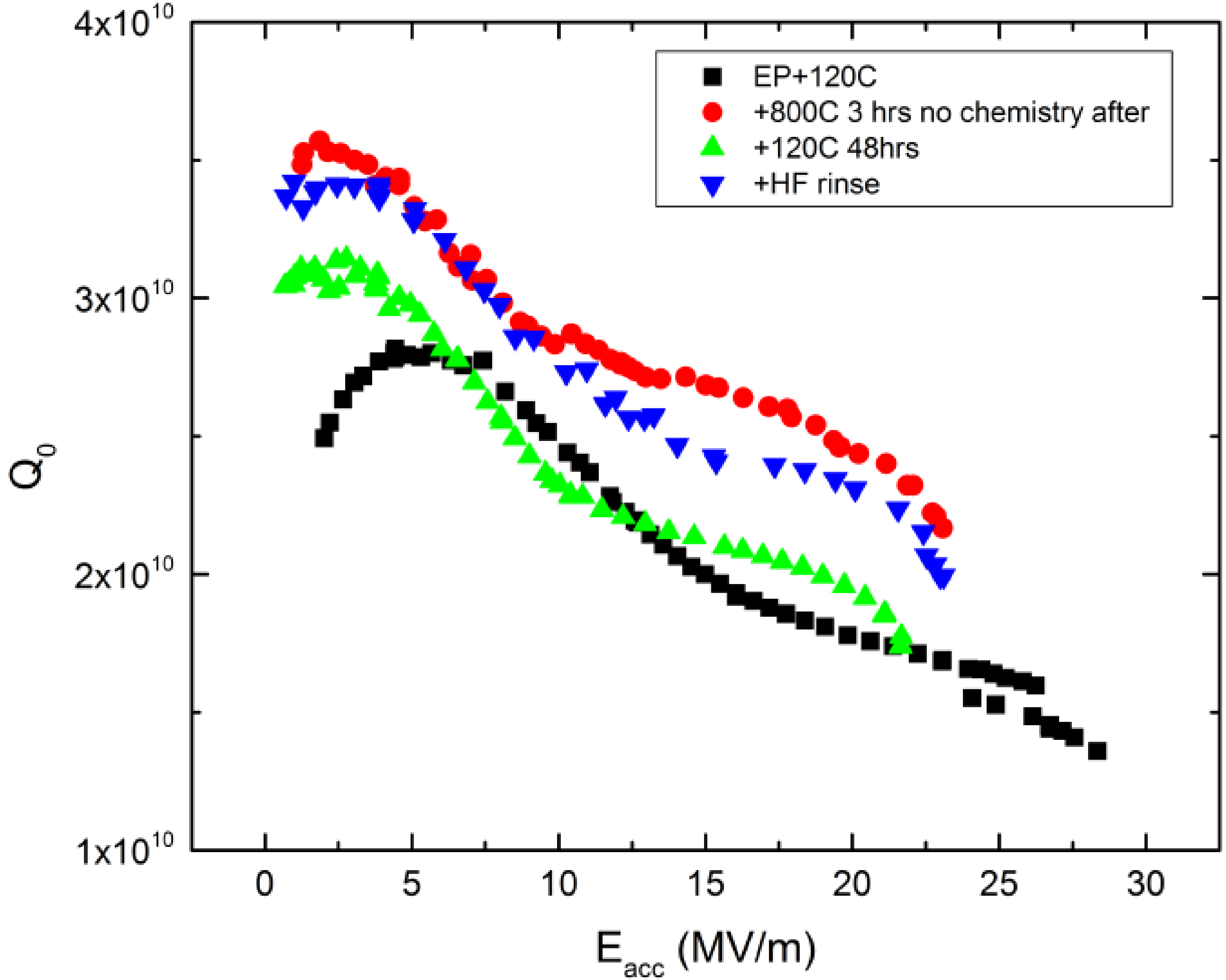


Figure 8: Depth profile of Ti in Nb samples heat treated at different temperature.

- Titanium contamination from furnace on surface no matter what heat treatment
- Large grain cavities are not limited by contamination except for Q slope

G. Ciovati et al. SRF2013 Chicago TUPO051

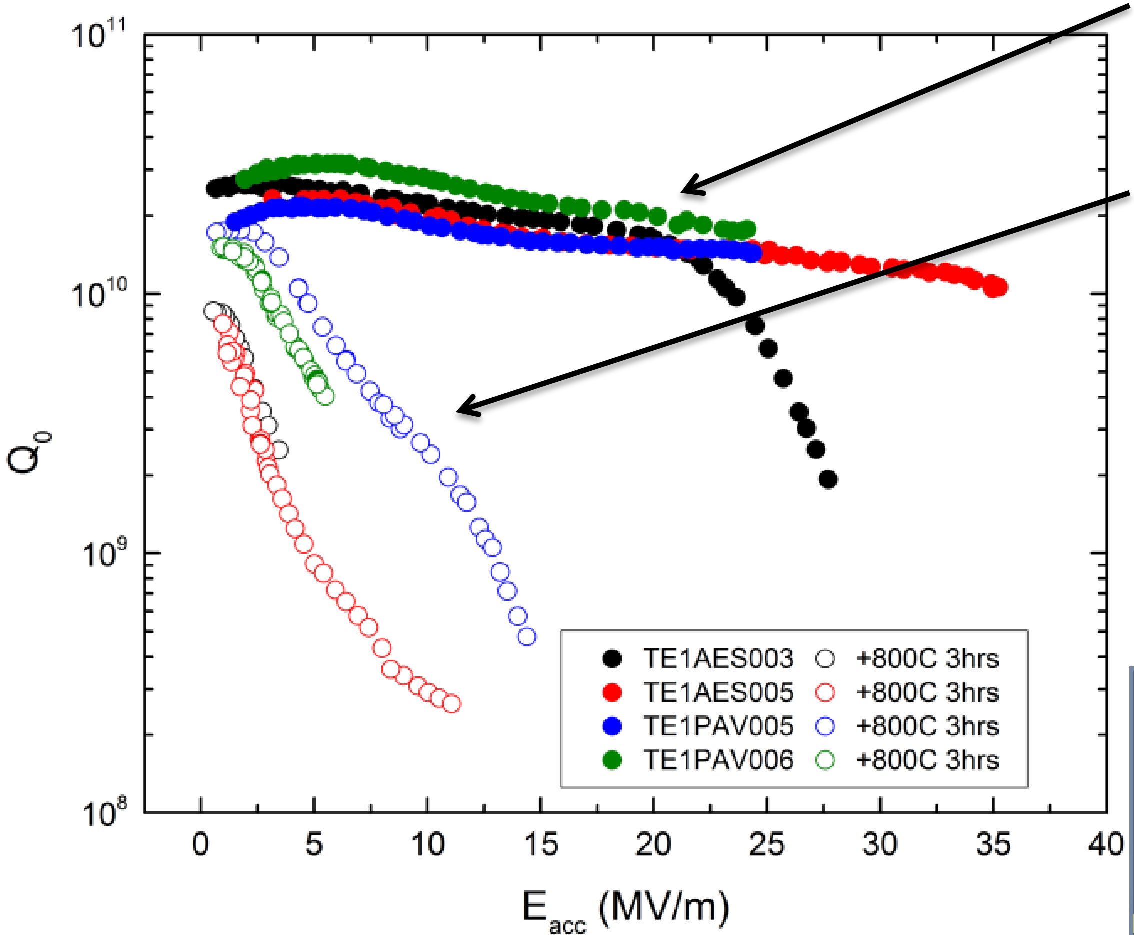
Heat treatment on Large grain material - FANL



Small ~ 30% improvements to Qo removing final chemistry on fine grain cavities.

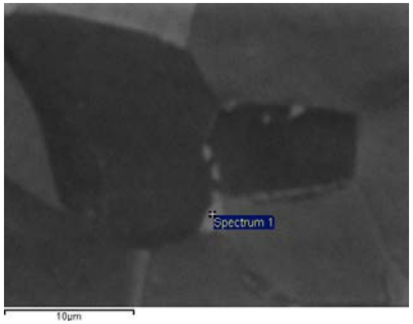
A. Grassellino - http://arxiv.org/ftp/arxiv/papers/1305/1305.2182.pdf

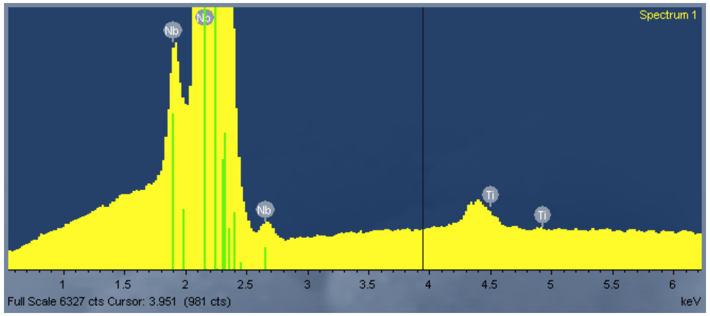
Heat treatment on Fine grain material - FNAL



Before furnace treatment

After furnace treatment
- Titanium contamination from furnace on gain boundaries (many boundaries on FG cavity)



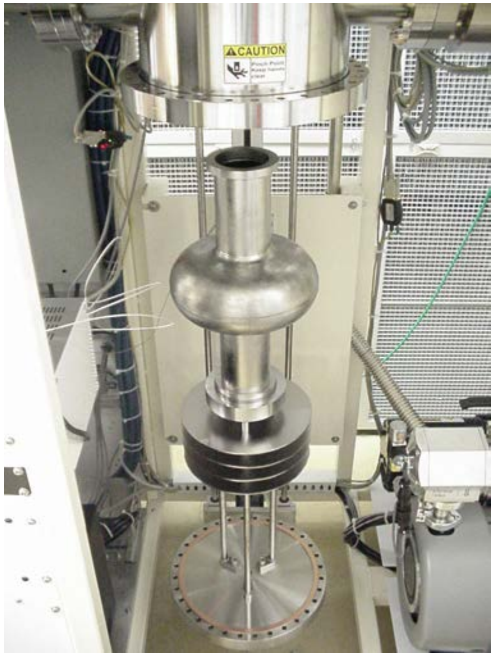


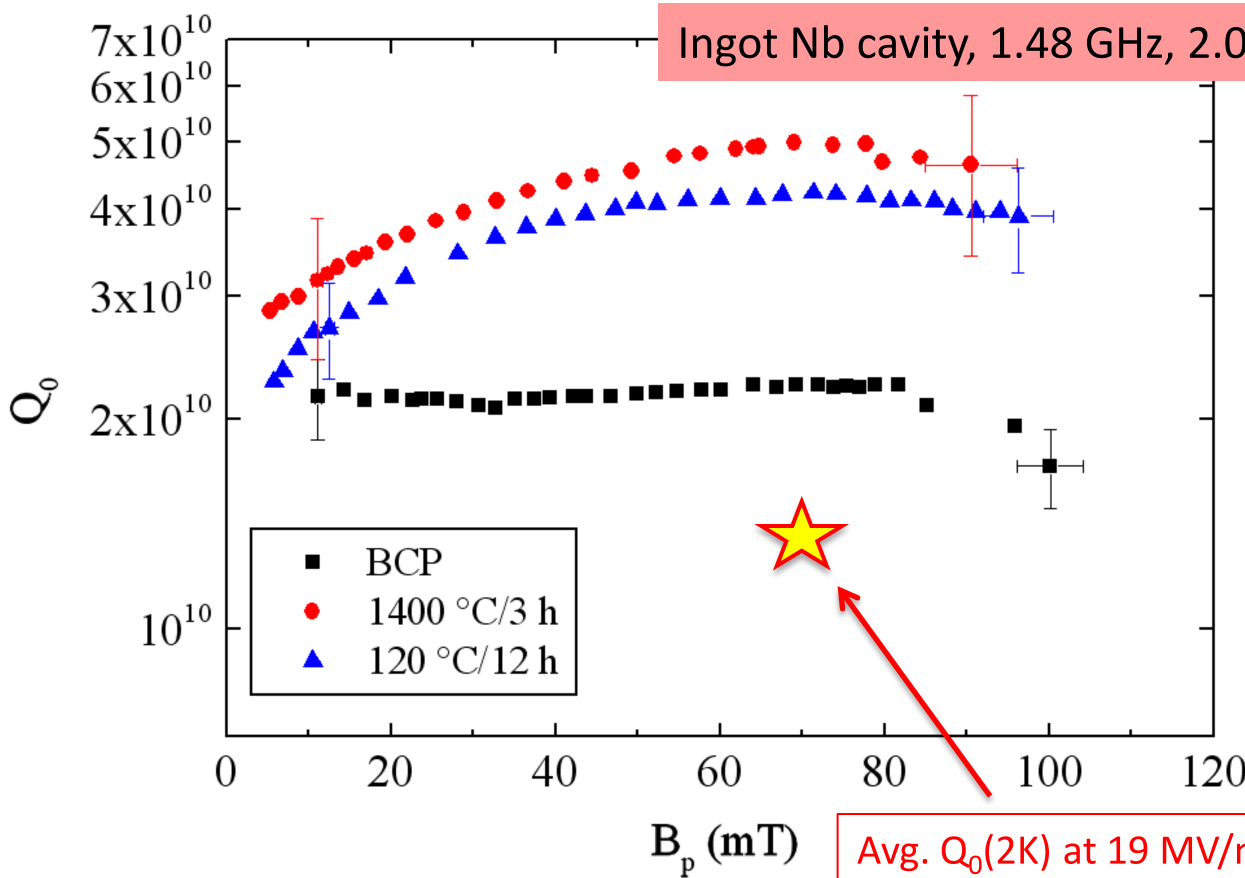
A. Grassellino - http://arxiv.org/ftp/arxiv/papers/1305/1305.2182.pdf

High-Q_0 by Ti doping during furnace treatment

- A new induction furnace was designed and installed at JLab to continue the high-temperature annealing study above 800°C in a “clean” environment and without subsequent chemistry.
- In 2012, heat treatment at 1400°C/3h of an ingot Nb cavity with NbTi flanges at JLab resulted in doping of the surface with Ti (~1 at./%, ~1 mm deep) producing an unprecedented high $Q_0 \cong 4.5\times10^{10}$ at 2 K, 90 mT

High-Q_0 by Ti doping





Avg. Q_0(2K) at 19 MV/m measured in VT of C100 cavities

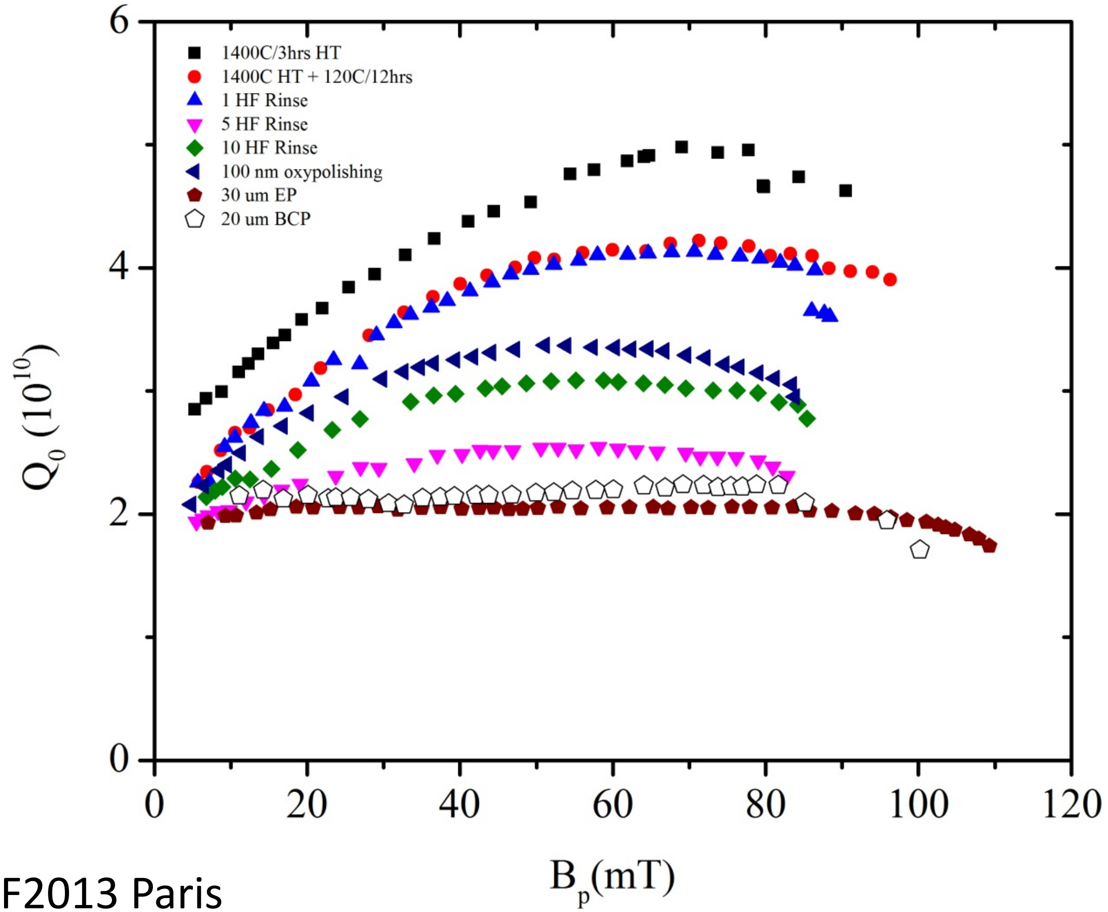
P. Dhakal, Rev. Sci. Inst. **83**, 065105 (2012)
P. Dhakal et al., *Phys. Rev. ST Accel. Beams* **16**, 042001 (2013)
P. Dhakal et al., IPAC'14, p. 2651

Ciovati

Ti-doping and nano-removal

Multiple nano-removal, oxypolishing and EP was done

- No performance degradation while keeping in cabinet for a year
- Extended Q-rise present even after the removal of ~120 nm inner surface
- EP after 30 μm reproduce the baseline performance
- Sims measurements



TUIOC04_talk pashupati SRF2013 Paris

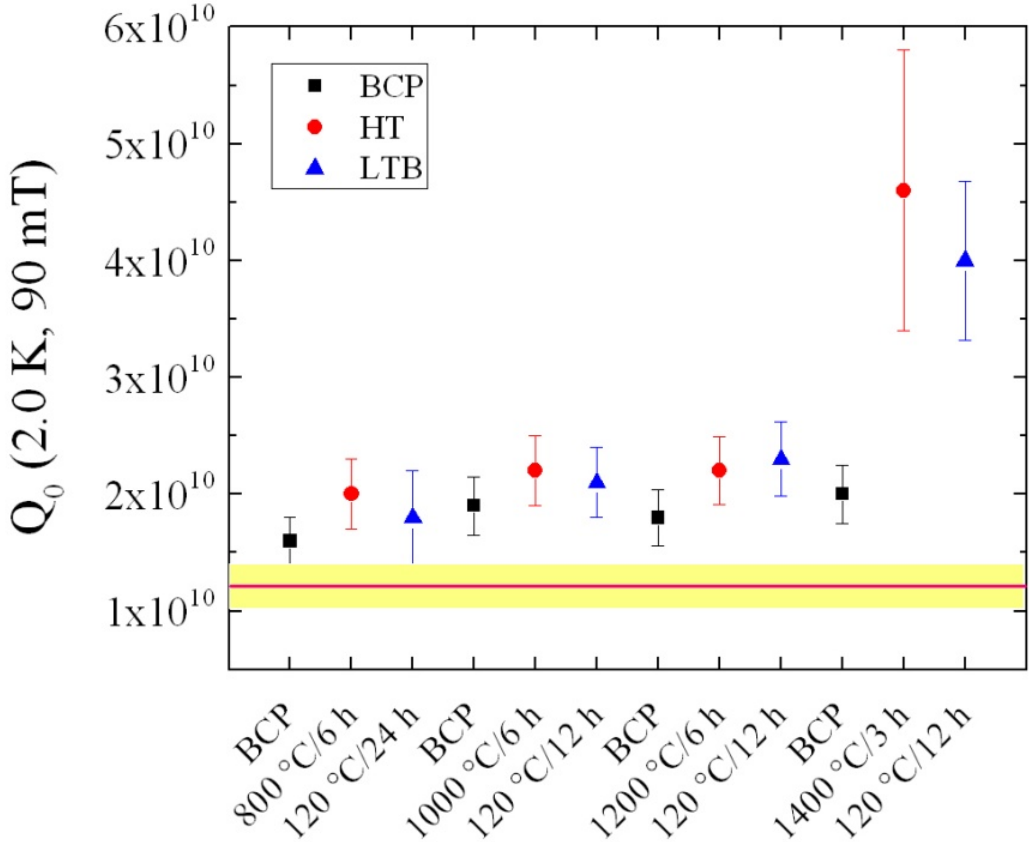
HT extended up to 1400°C with new furnace

- Ingot Nb cavity from CBMM (RRR~200, Ta~1375 wt.ppm), treatment sequence after fabrication: CBP, BCP, HT, HPR

Samples' analysis after 1400°C show:

Reduced H content and ~1 at.% Ti content

Higher energy gap and reduced broadening parameter

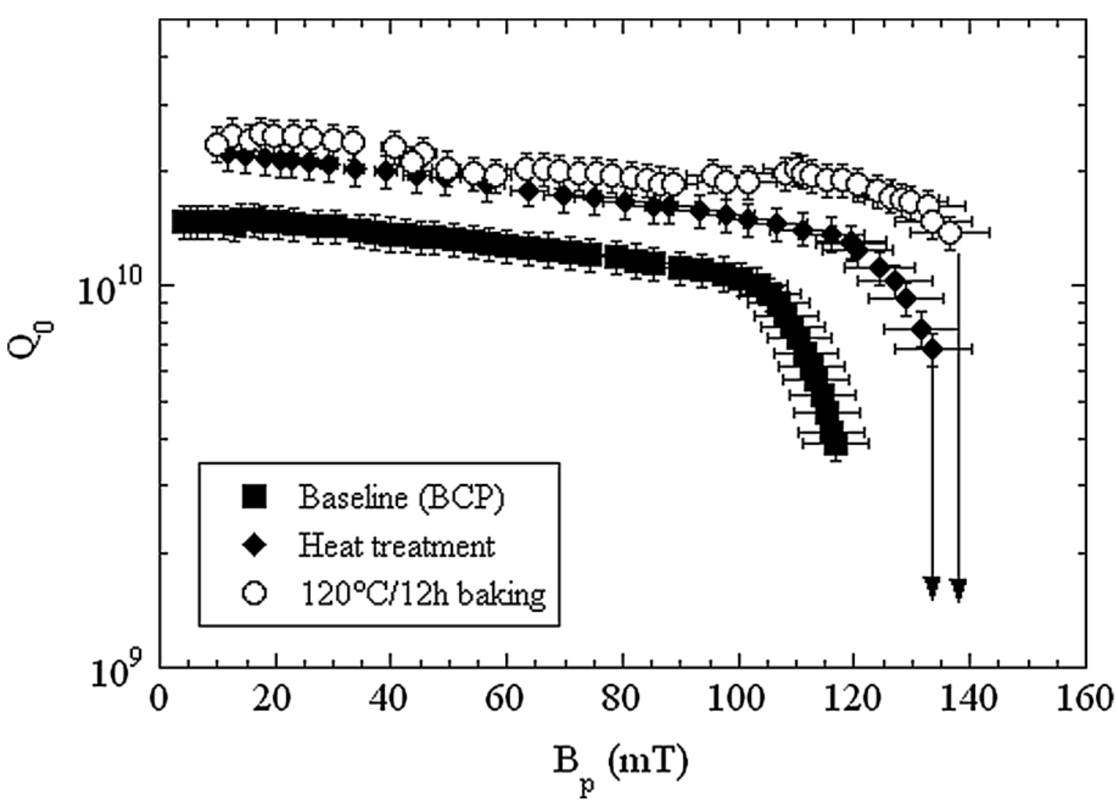


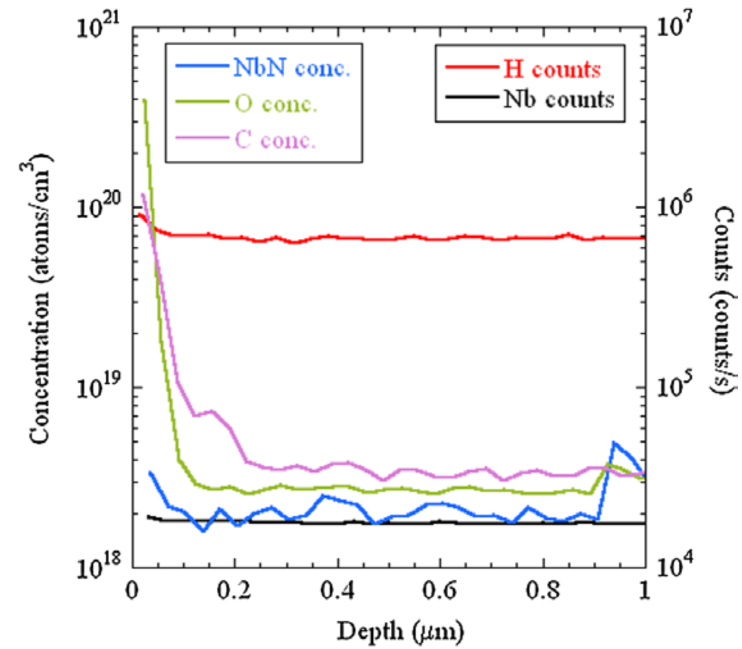
Phys. Rev. ST Accel. Beams **16**, 042001 (2013)

Nitrogen doping during furnace treatment

- 2009 JLab attempted to make a Hydrogen blocking niobium nitride layer on the surface of a cavity (purposed in the 1970’s), with no post heat treatment chemistry. Limited to 1e^-4 torr because of interlock so higher pressures never used. ~30% gain in Q0 (not doping)
- 2013 an attempt was made to create niobium nitride (Tc=NbN) on the surface of the SRF cavity with nitrogen @ ~20mtorr and 800C. The experiment failed at FNAL. Q = 1e7. But after random removal choice cavity showed new Q-rise not seem before (except with Ti doping the year before)

Niobium nitride study JLab

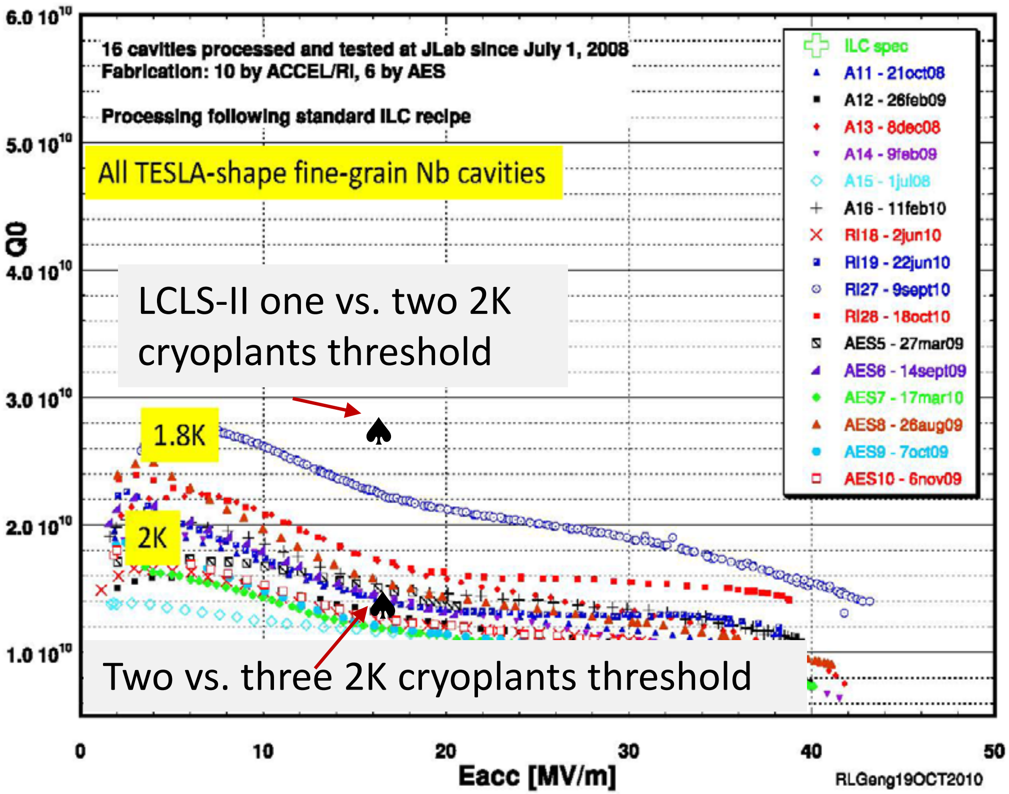




Clean furnace, so Q0 gain was the same as no doping, study canceled because pressures could not go high enough Because of safety interlocks on furnace!

G. Coivati et al., PRST - ACCELERATORS AND BEAMS 13, 022002 (2010)

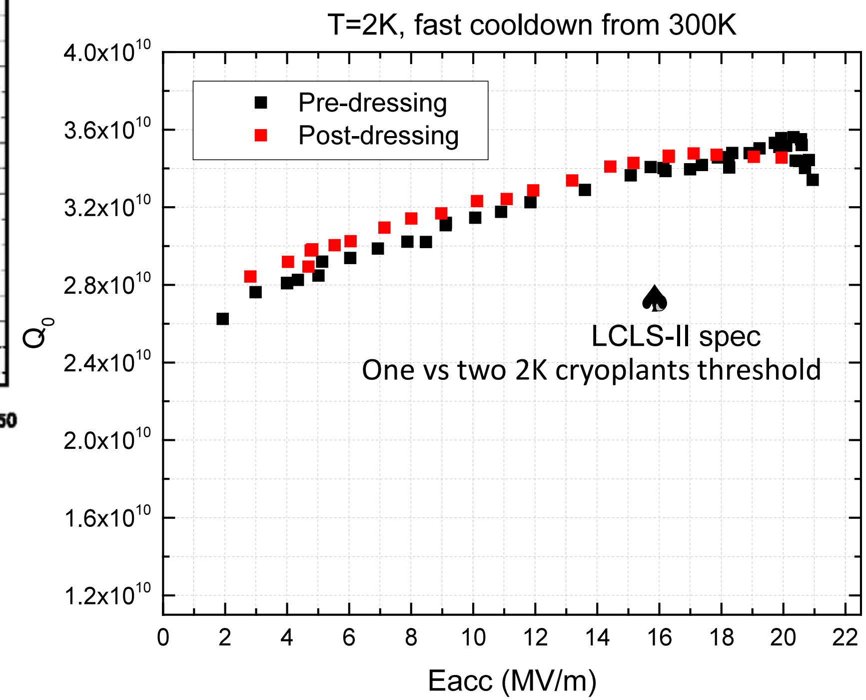
XFEL/ILC recipe vs. N doping



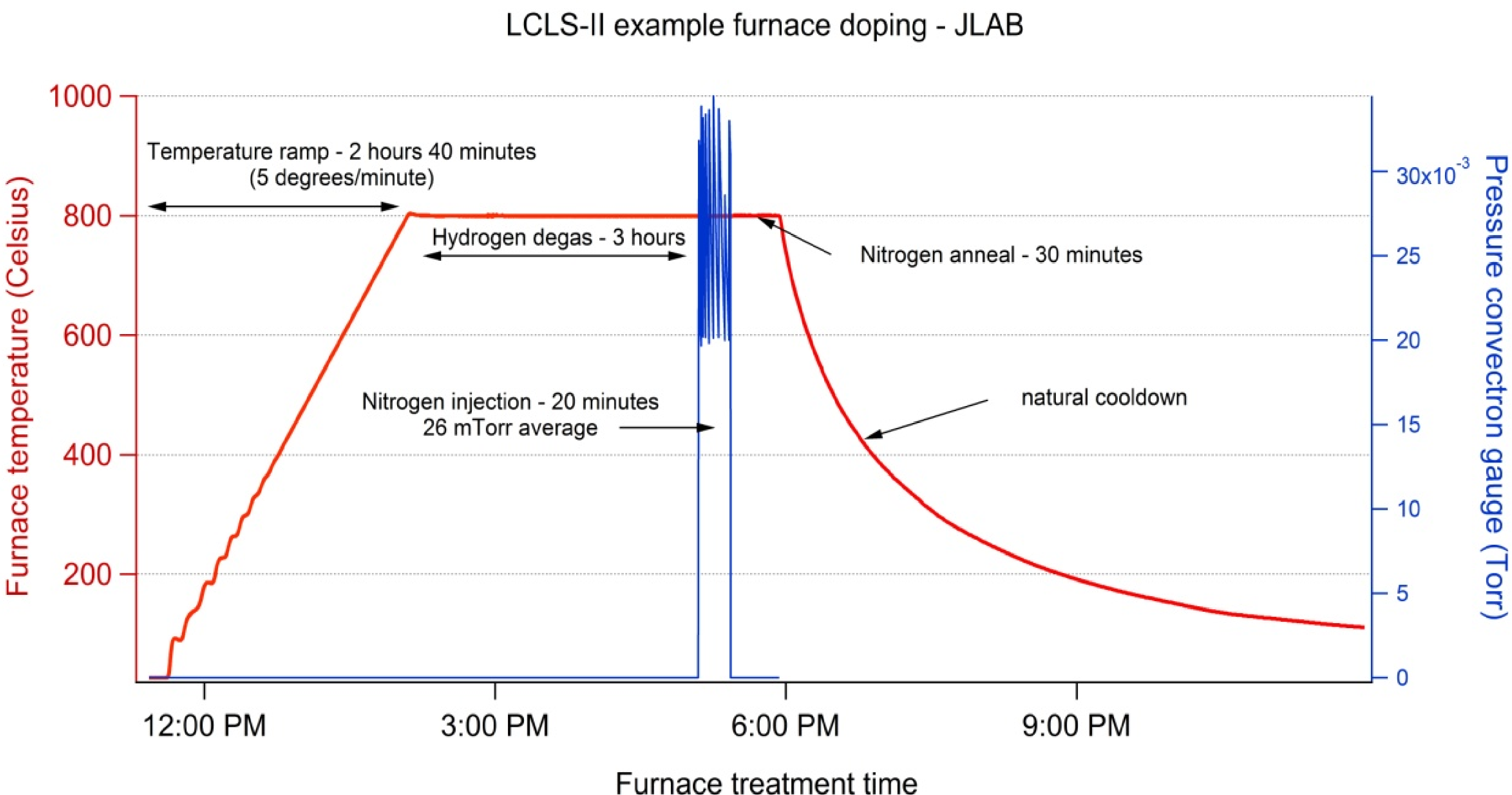
"The best cavities of 2010" (120C bake)
→ Could be marginal for 2 cryoplants (likely to require 3 with slow cooling)

The SRF world is changing

"The best dressed cavity of 2014"
(N doped)
TB9AES011

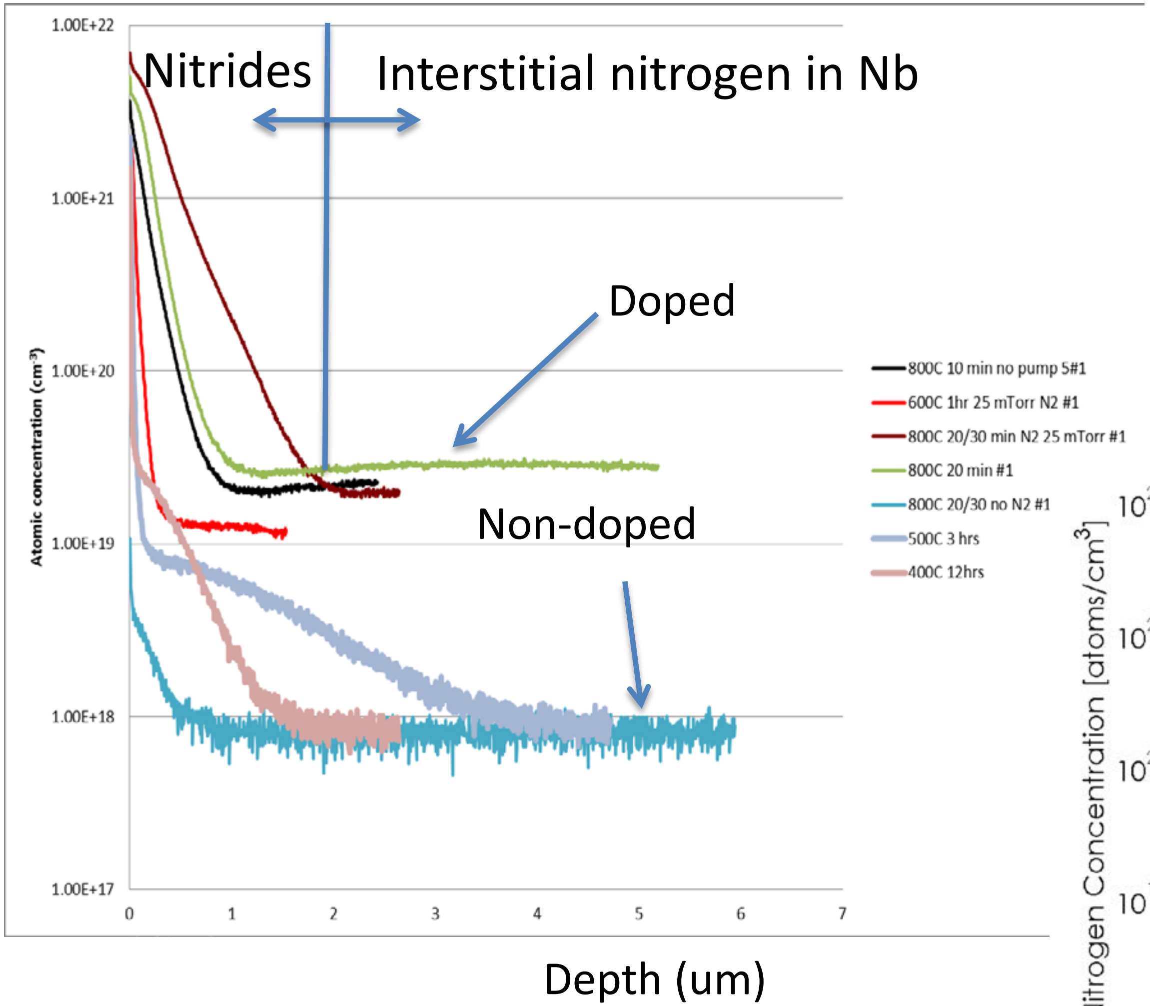


Nitrogen Doping Process

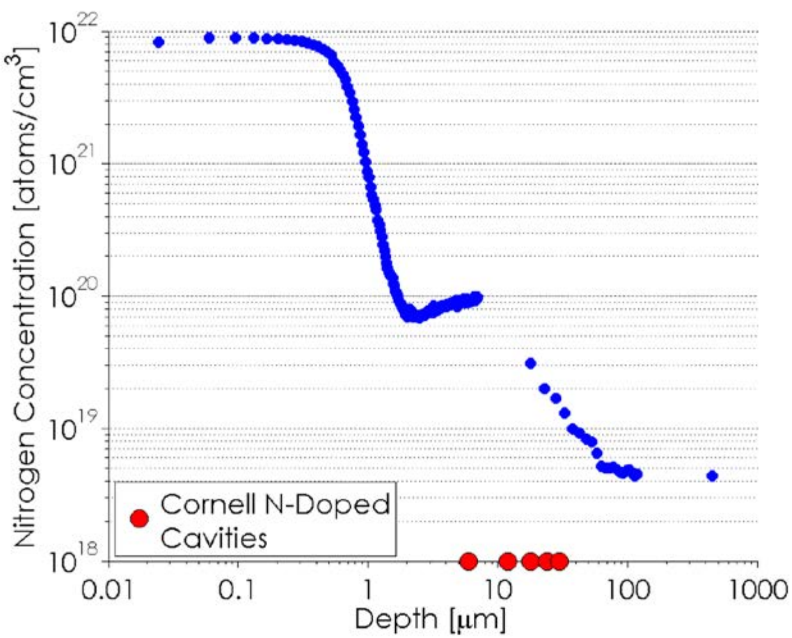


Palczewski

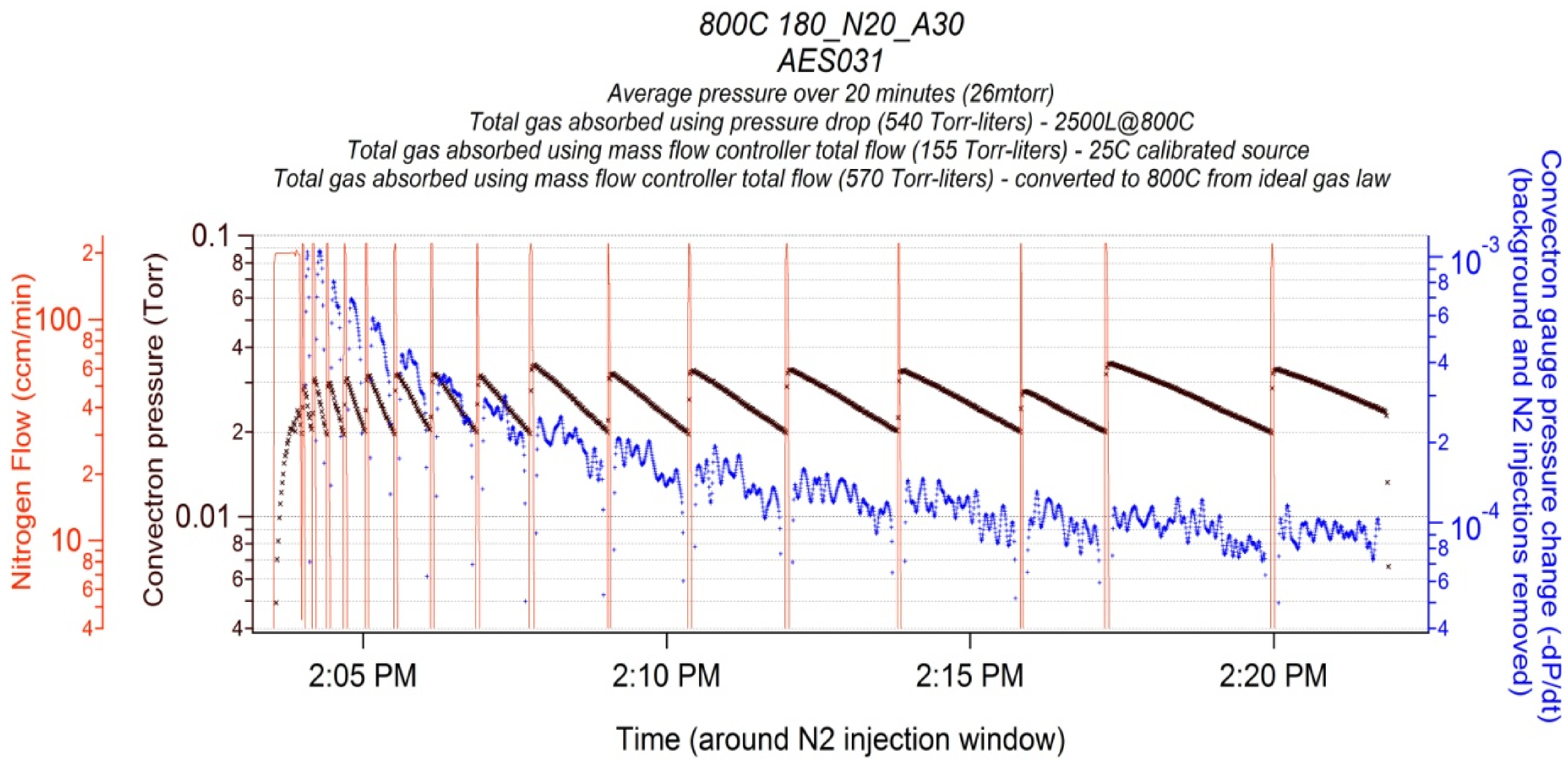
What does N treatment do? N depth profiles by SIMS



See A. Romanenko, talk at LINAC 2014, Geneva
And D. Gonnella et al, LINAC 2014, Geneva

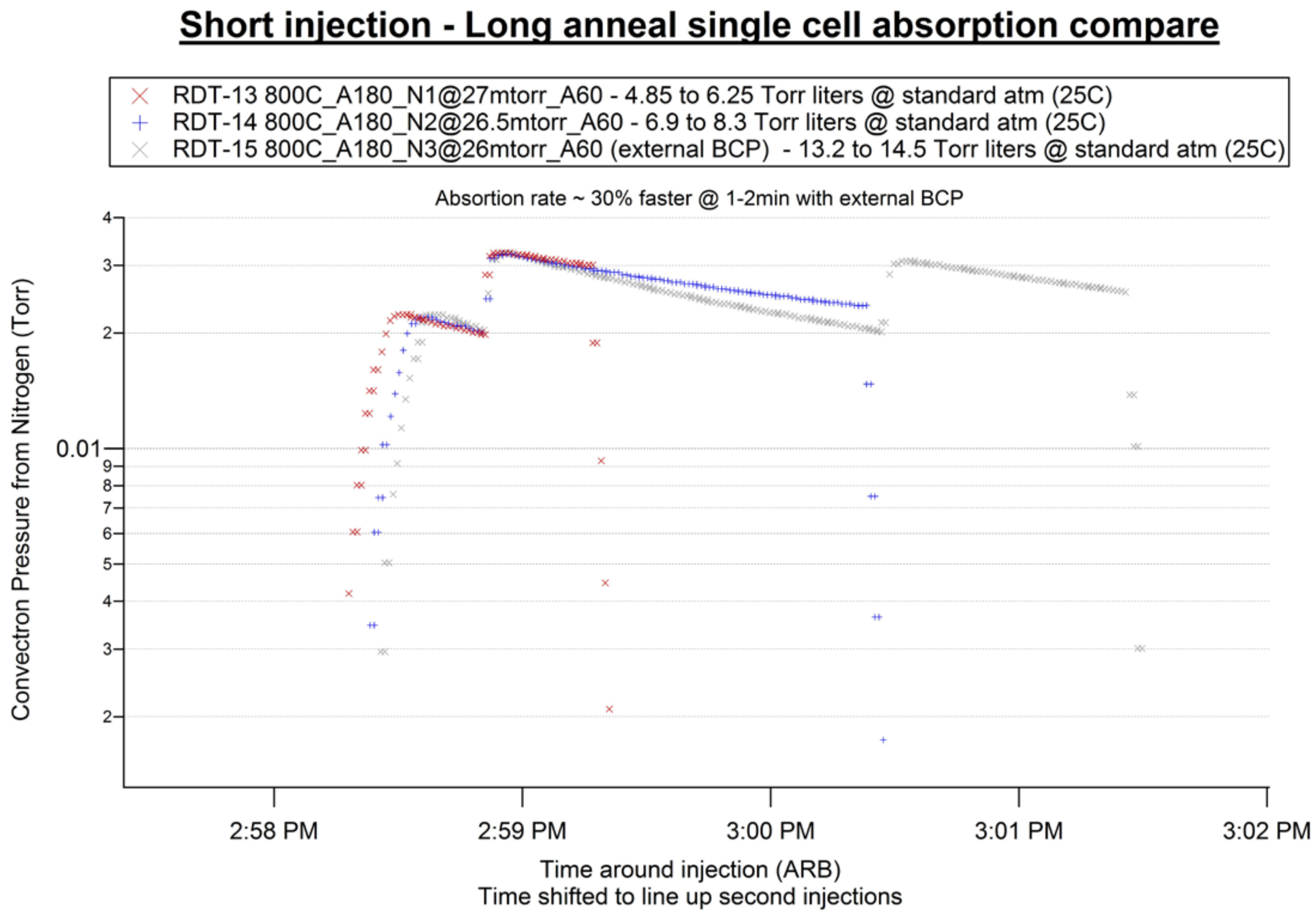


Amount of nitrogen absorbed



Jefferson Lab

Re-doped external BCP - % nitrides



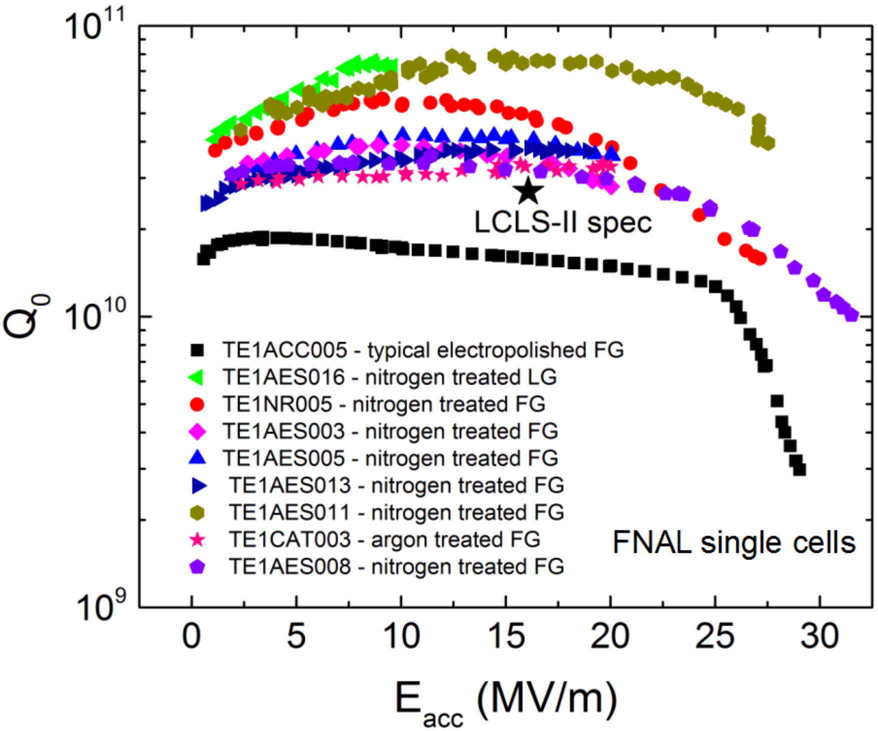
Minimum 60 to 70% of nitrogen goes into nitrides on surface, the rest goes into the cavity

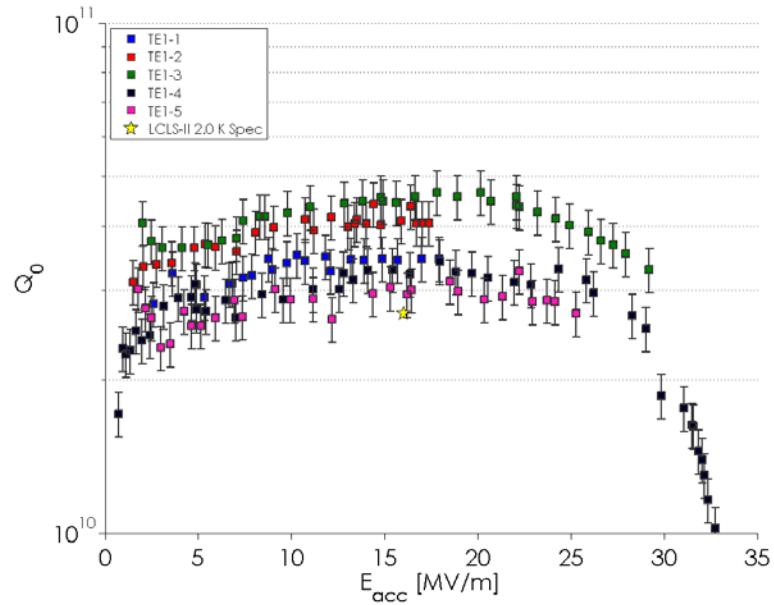
Current doping/EP recipes tried that “worked” – rising Q0

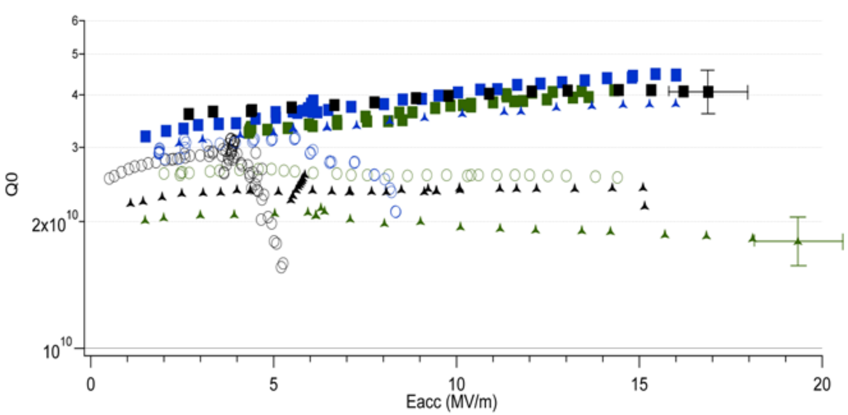
LAB	pressure	Time N	Anneal time	EP microns
JLAB	~26mtorr	1	40,60	5,10,15
JLAB	~40mtorrr	2	10,20,30	5,10,15
JLAB	~26motorr	20	10,30,60	10,15,17,20
JLAB	~26motorr	2	6	5
Cornell	~40mtorr	20	30	5,12,18,24,30
FNAL	~20mtorr	10	0	10
FNAL	~20mtorr	2	6,20	5-30
FNAL	~20mtorr	20	30	10,20,30
FNAL	~10mtorr		?	?
FNAL	~20mtorr	60	0	10,40,80

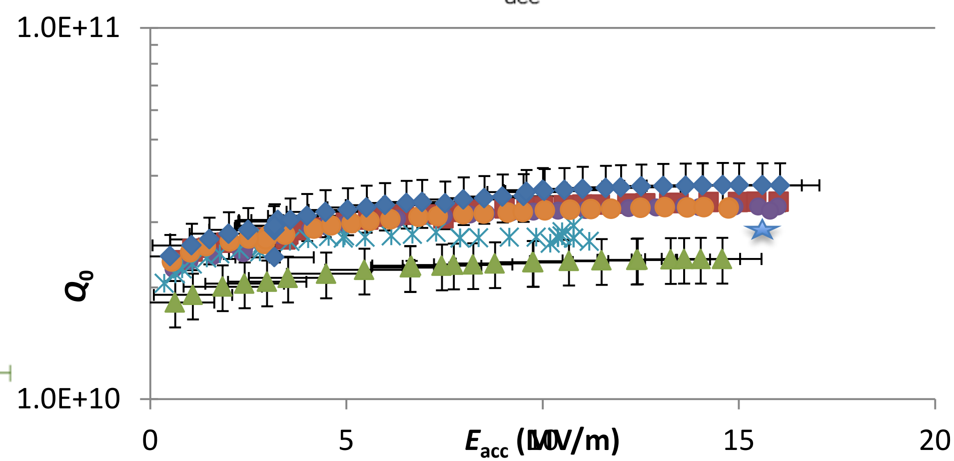
Incomplete list – ones I have verified with my notes, I know there are others especially at FNAL (Sorry)

Multiple cavity tests – N doping



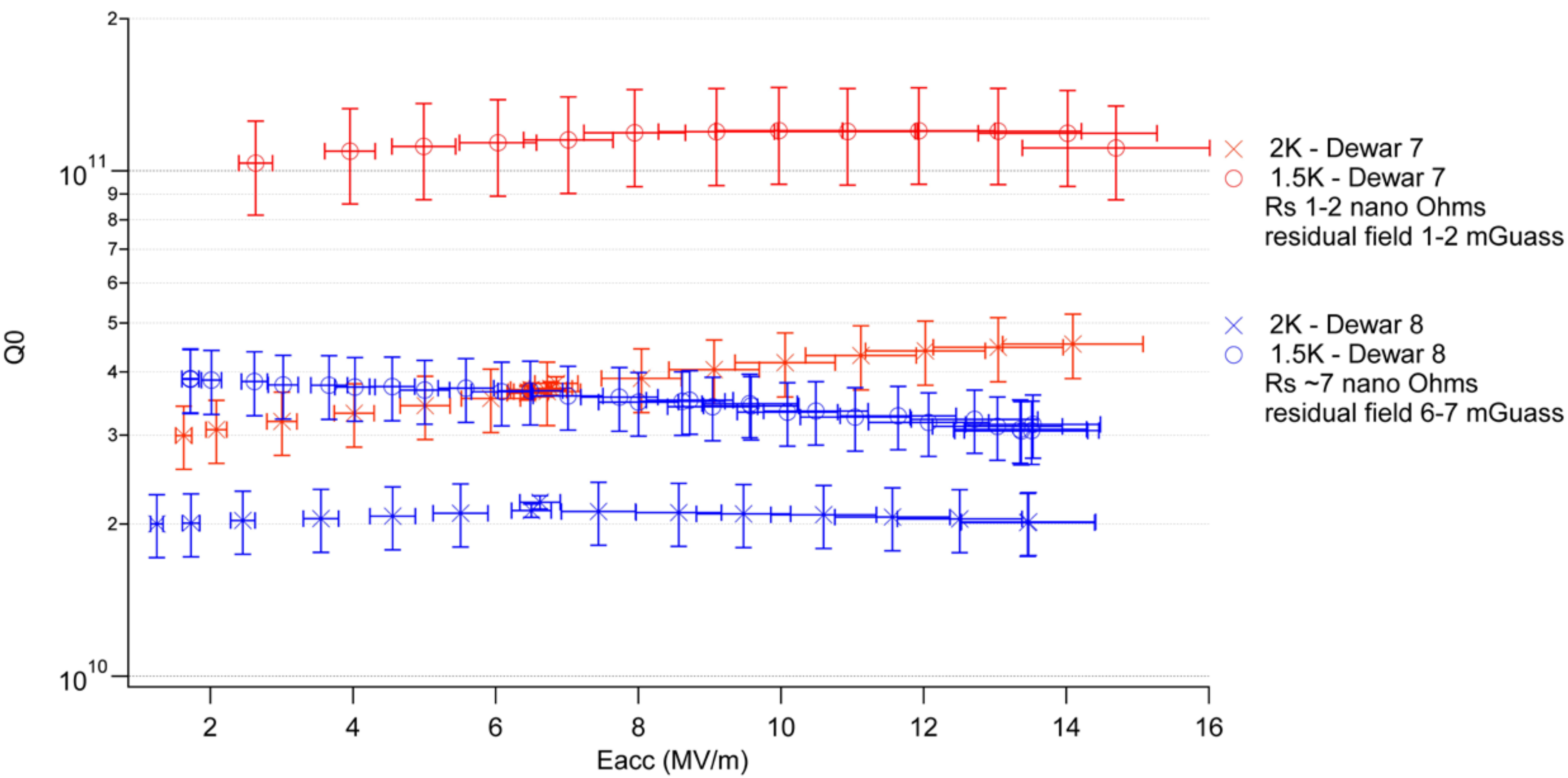






Problem with N-doping- enviroment

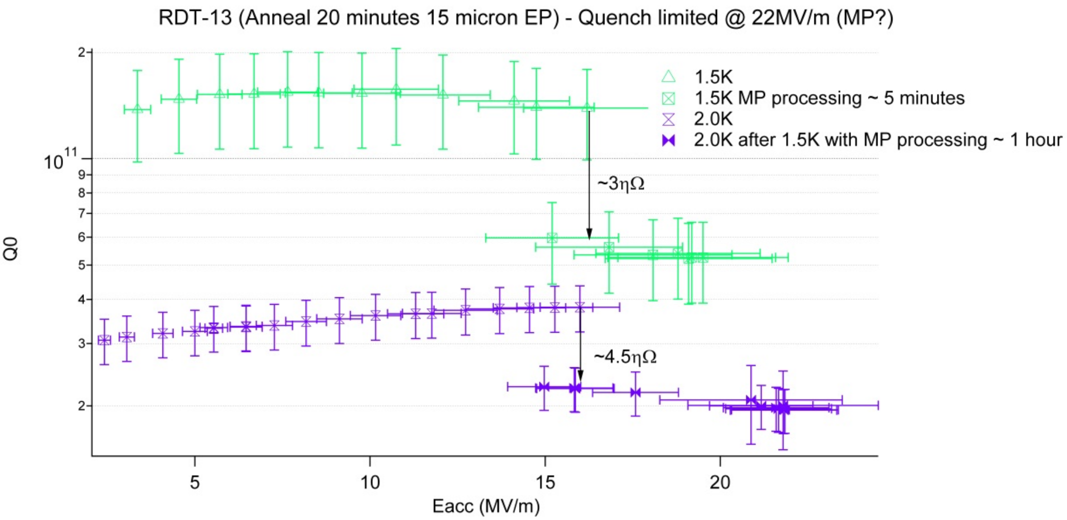
RDT-15 800C 3h_N20_A_50 + 15micron EP - quench @ 15MV/m

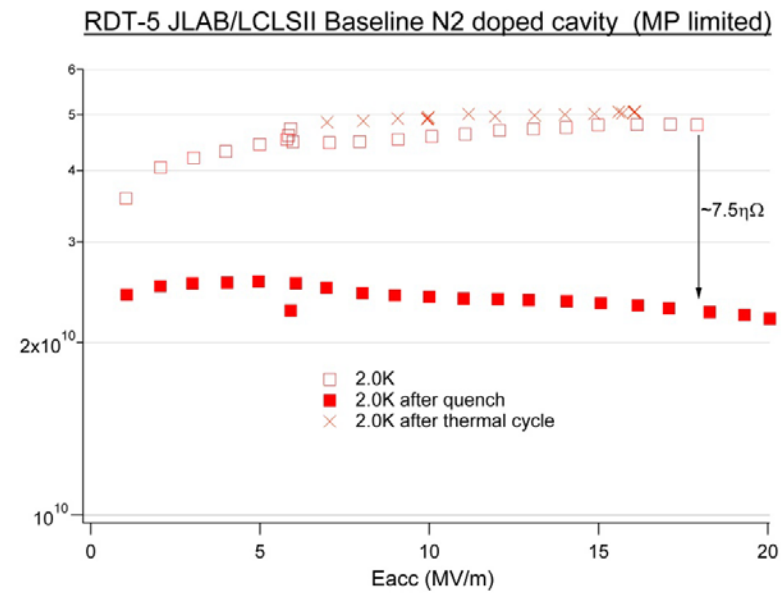


Cavities are highly supposable to environmental factors, where the remnant magnetic field can dramatically change Q performance, for standard cooldowns

Problem with N-doping- temporary quench degradation

Amount of flux captured during a quench and the resulting drop in Q_0 is quite variable.





Example of multipacting-induced quenching Q_0 degradation and recovery with thermal cycle

Residual Resistance vs Trapped Flux

- N-doped cavities appear to be more sensitive to trapped flux.
- Higher R_{res} for same flux
- Due to higher R_{NC} from lower mfp?

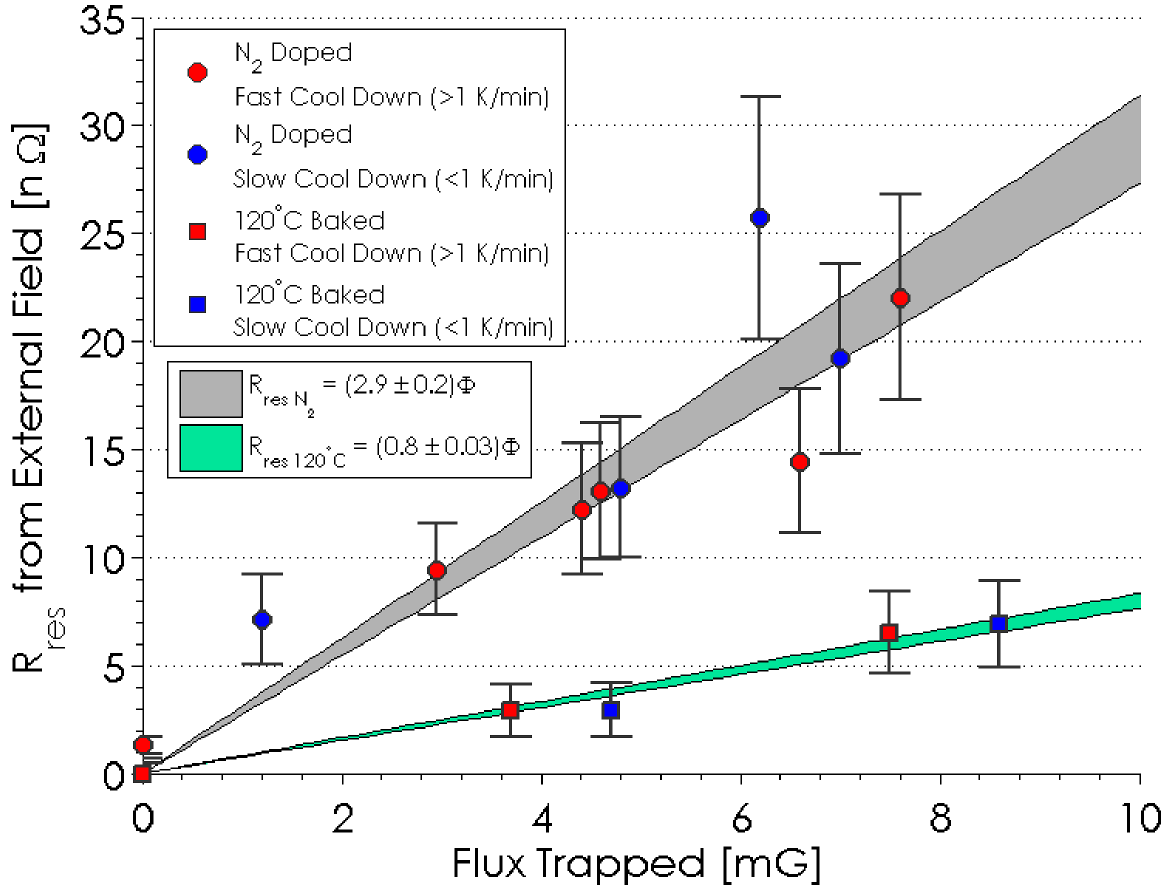
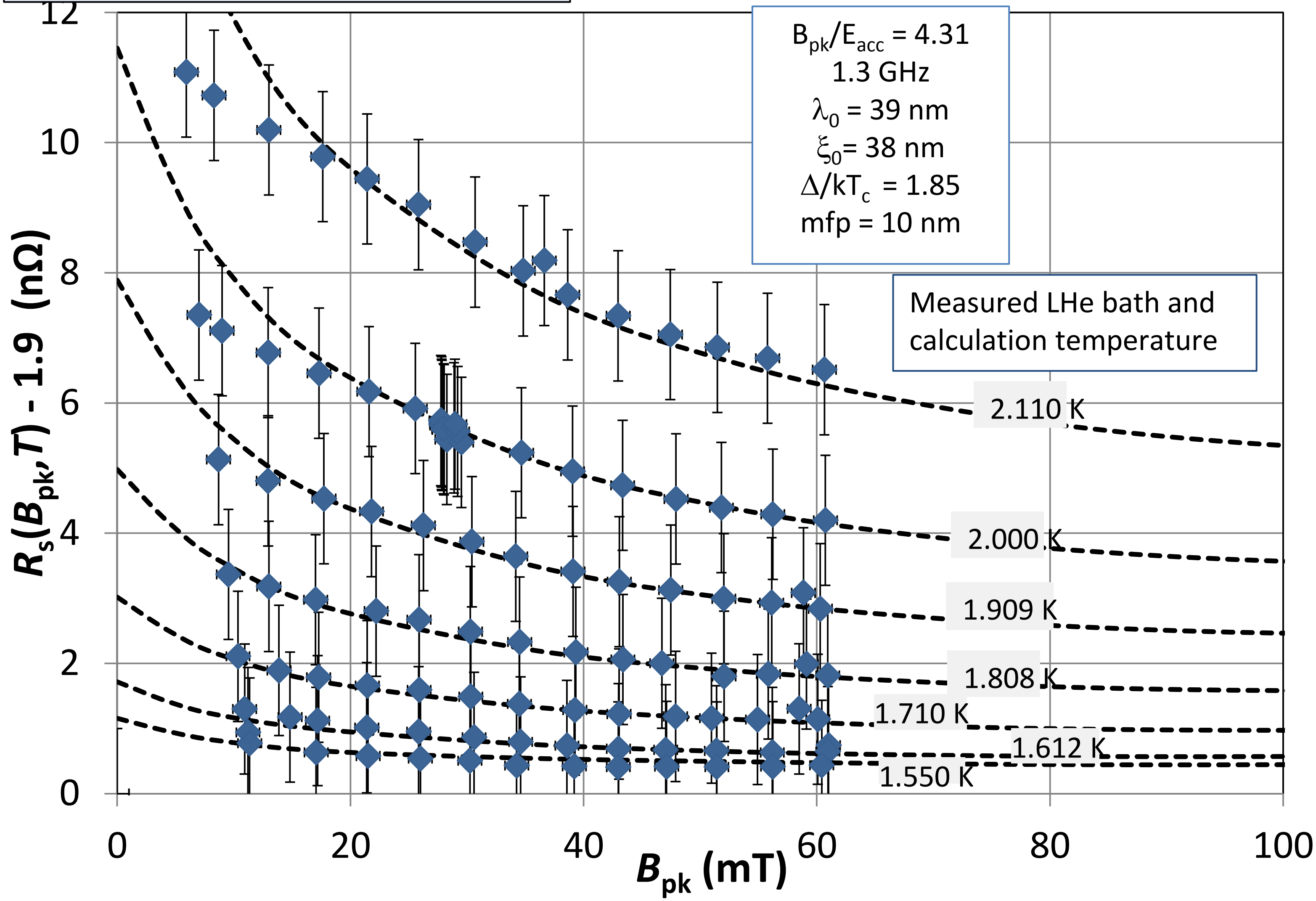


Image Courtesy of Dan Gonnella
Cornell University
See IPAC13 WEPRI063

The Best Doped Cavities Match the new R_s Theory

RDT-15 180/20N/50 + 15 micron EP

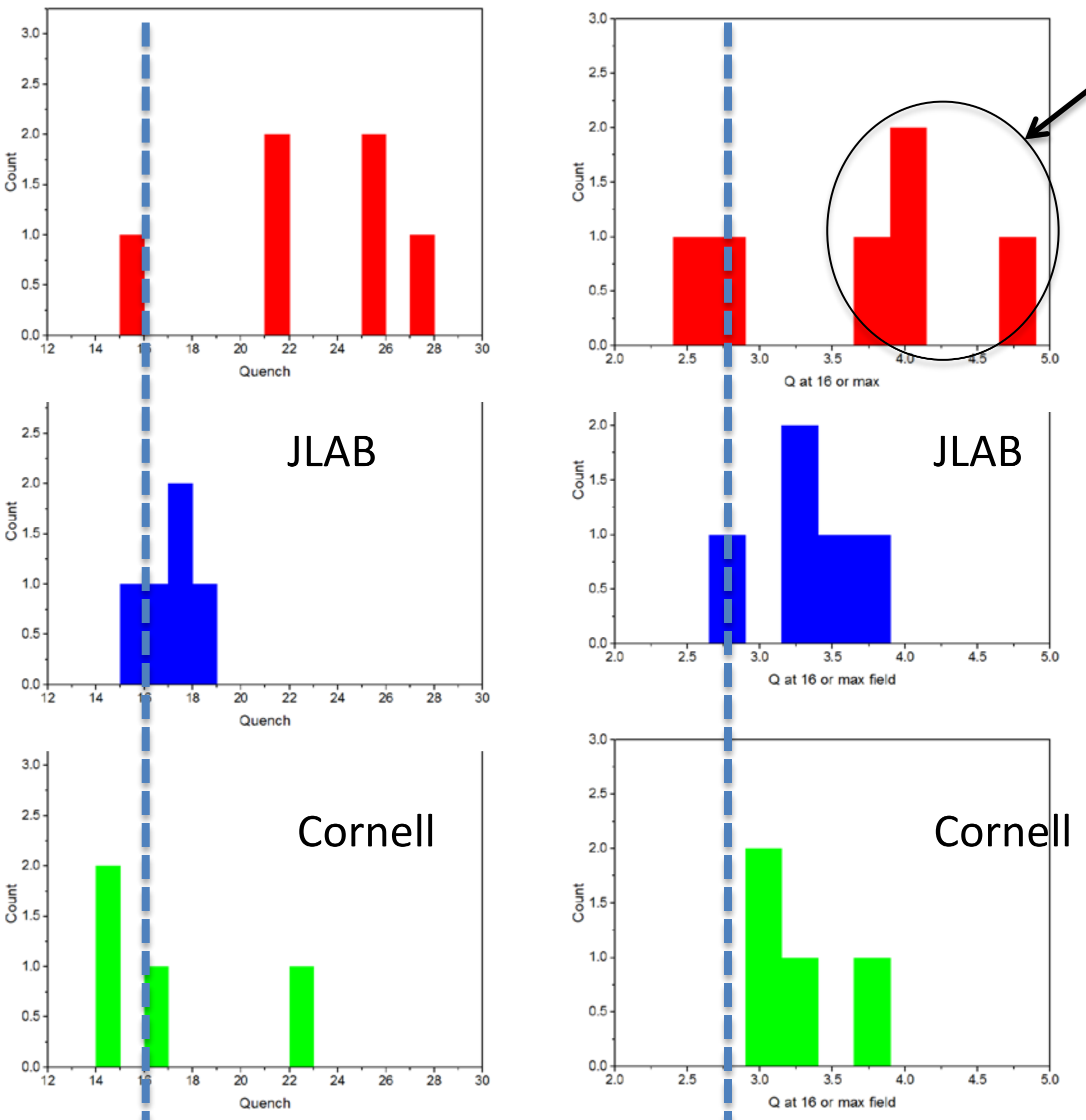
Not yet sure how to interpret this agreement



C. Reece

9 cell studies LCLS-II baseline Q0 and quench field – Nitrogen doping

16 MV/m **2.7e10**



NbTi flanges

FNAL N2 A6 EP 5

Quench field definitely dependant on doping, where lower doping is better! From single cell higher doping appears to produce better Q0

N20 A30 EP~15-25

Nine cell frozen recipes results

	Gas bake details	Average Q	Average quench field	First pass yield	Second pass yield
FNAL **"recipe 1"** N=6	• 800C 3 hours in HV • 2 min at 800C with N ~ 20 mTorr • 6 min at 800C in HV	**3.7e10**	**~23 MV/m** (2nd pass) ~21 MV/m (1st pass)	**67% @18 MV/m**	**83% @18 MV/m**
Jlab/Cornell **"recipe 2"** N=10	• 800C 3 hours in HV • 20 min at 800C with N ~ 40 mTorr • 30 min at 800C in HV	**3.5e10 (Jlab)** **3e10 (Cornell)**	**~16.6 MV/m (Jlab)** **17 MV/m (Cornell)**	**60% @16 MV/m** **20% @18 MV/m**	

Anna Grassellino, LCLS-II DOE Status Review, June 30th, 2014

Question?