

ANALYSIS OF POST-WET-CHEMISTRY HEAT TREATMENT EFFECTS ON NIOBIUM SRF SURFACE RESISTANCE

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R&D on SRF

- Driven by Project Need
 - * CW applications
 - * Pulse applications
 - * High current
 - * 4.2 K applications
- Conventional R&D
 - * Maximize E_{acc} and Q_0
 - * Search for alternative materials
 - * Process improvement

Overall goal : Minimize construction and operation cost with reliable and efficient SRF cavities

Quality Factor and Surface Resistance

Quality Factor (Q_0) = G / R_s

G = Geometry factor (shape dependent)

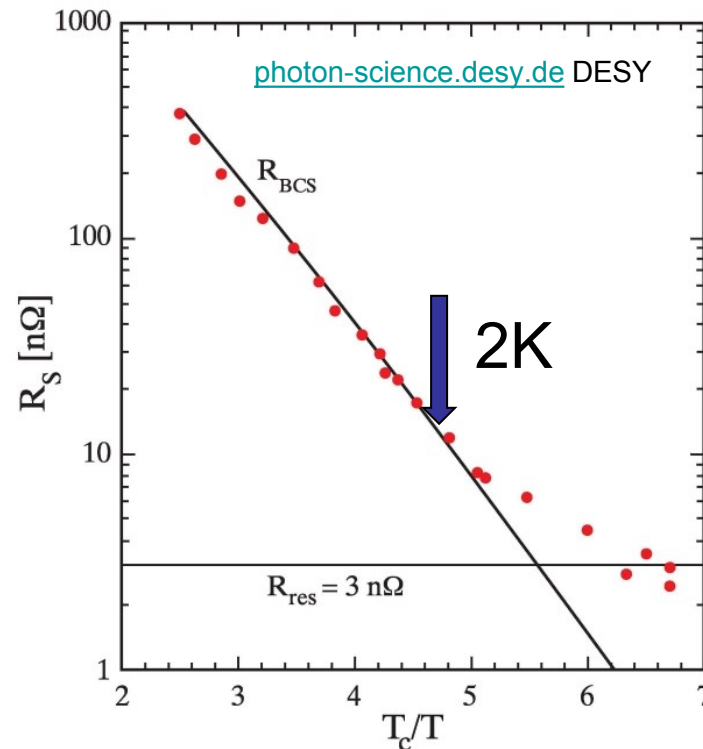
$$R_s = R_{\text{res}} + R_{\text{BCS}}(f, T, \Delta, \lambda_L, \xi_0, I)$$

Possible sources of R_{res}

- Trapped magnetic field
- Normal conducting precipitates
- Grain boundaries, dislocations
- Interface losses
- Subgap states

Remedies:

- High treatment heat treatments
- Magnetic shielding
- ---

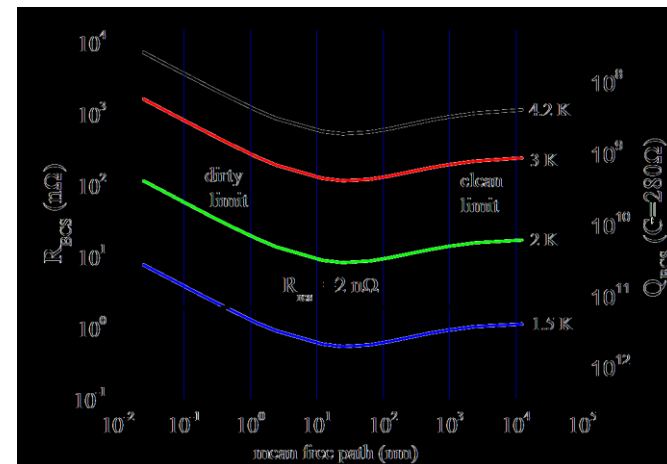


At temperature below 2K,
 R_s is dominated by R_{res}

Quality Factor and Surface Resistance

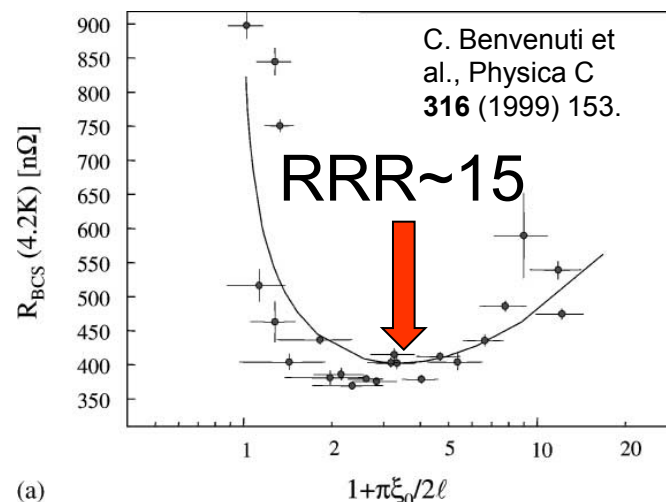
BCS surface resistance results from the interaction between the RF electric field within the penetration depth and thermally activated electrons in a superconductor.

$$R_{\text{BCS}}(f, T, \Delta, \lambda_L, \xi_0, l) = (A f^2 / T) e^{-\Delta / k_B T} c$$



Minimizing BCS Resistance

- Lower frequency
- Higher T_c superconductors
- Higher energy gap
- Optimal electronic mean free path



(a)

Typical SRF cavity Processes

- ~150 μm heavy BCP/ CBP
- Heat treatment 600-800 $^{\circ}\text{C}$
-
- High Pressure Rinse with DI water
- Low temperature baking (100-140 $^{\circ}\text{C}$ for 12-48 hours)
- RF Test

Note: Surface chemistry of ~20 μm is like making new cavity surface

High Temperature Heat Treatment

1970s → ~1800 °C UHV HT for ~10 hrs.

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

2000s → 600-800 °C, mainly just to degas hydrogen absorbed
by the Nb during cavity fabrication and surface
treatments.

- **BCP/ EP needed to remove “polluted” layer after HT**
- **Reintroduces hydrogen**
- **May be the cause of strong RF losses**

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.

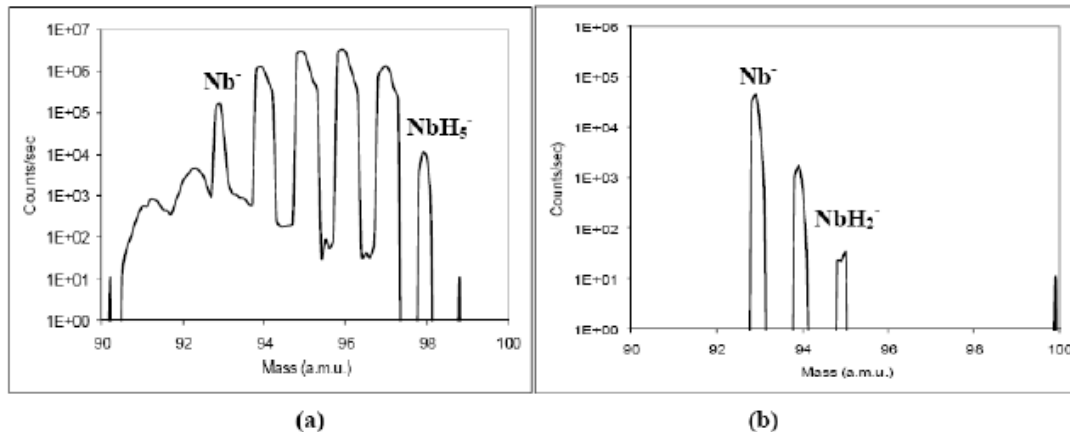


FIGURE 1. SIMS mass spectra showing difference in H between (a) non-heat treated and (b) heat treated sample.

**High temperature
annealing
removes gross hydrogen**

Ciovati et al, PRSTAB 13, 022002 (2010)

Earlier Results (No post chemistry)

TABLE III. Values of $\Delta/k_B T_c$, R_{res} , R_{BCS} (4.3 K, ≈ 10 mT), Q_0 (100 mT)^a, $B_{p,\text{max}}$, and improvement factors of Q_0 (100 mT), $B_{p,\text{max}}$ over the baseline test for all the cavities and rf tests described in Secs. VA1–VA4.

Cavity	Treatment	$\Delta/k_B T_c$	R_{res} (n Ω)	R_{BCS} (n Ω)	Q_0 (100 mT) ($\times 10^{10}$)	$B_{p,\text{max}}$ (mT)	Q_0 improvement	$B_{p,\text{max}}$ improvement
Yellow	Baseline 1 (20 μm BCP)	1.75	11.1	1068	1.05	118	Yellow	Yellow
	Heat treatment 1 (800°C/3 h, 400°C/20 min N ₂)	1.87	10.3	825	1.52	134		
	Baking (120°C/12 h)	1.97	9.7	614	1.88	136		
	Baseline 2 (5 μm BCP)	1.79	5.6	971	0.91	108		
	Heat treatment 2 (800°C/3 h, 400°C/20 min N ₂ , 120°C/6 h)	1.90	8.4	675	1.13	118		
	Baseline 3 (2 μm BCP)	1.80	7.9	933	1.07	112		
	Heat treatment 3 (800°C/3 h, 400°C/20 min)	1.92	3.2	697	1.89	112		
	Baseline (10 μm BCP, 600°C/10 h, 13 μm BCP)	1.75	4.7	782	0.75	109		
	Heat treatment (800°C/3 h, 400°C/20 min N ₂ , 120°C/6 h)	1.87	4.8	576	1.05	117		
	Baking (120°C/48 h)	1.98	8.2	414	0.94	115		
Orange	Baseline (122 μm VEP)	1.80	5.7	724	0.92	122	Orange	Orange
	Heat treatment (800°C/3 h, 400°C/20 min)	1.85	4.5	656	1.46	137		
	Baking (120°C/24 h)	2.00	7.9	437	1.40	179		
	Baseline 3 (1 μm BCP)	1.83	4.9	831	1.16	119		
	Heat treatment (800°C/3 h, 120°C/12 h)	2.00	4.2	412	1.44	128		
Blue							Blue	Blue

^aThe values of Q_0 (100 mT) were measured at 2.0 K, except for the first tests of the large-grain CEBAF cavity when they were measured at 1.7 K.

G. Ciovati et al., *Phys. Rev. ST Accel. Beams* **13**, 022002 (2010)

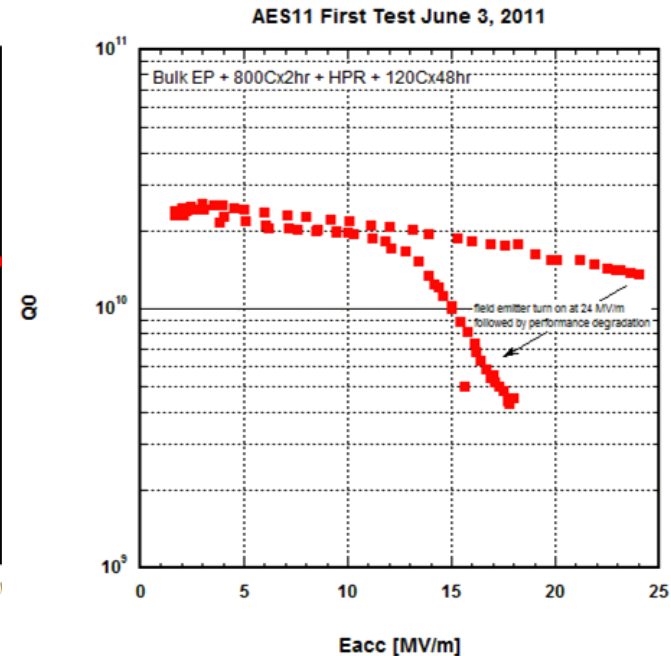
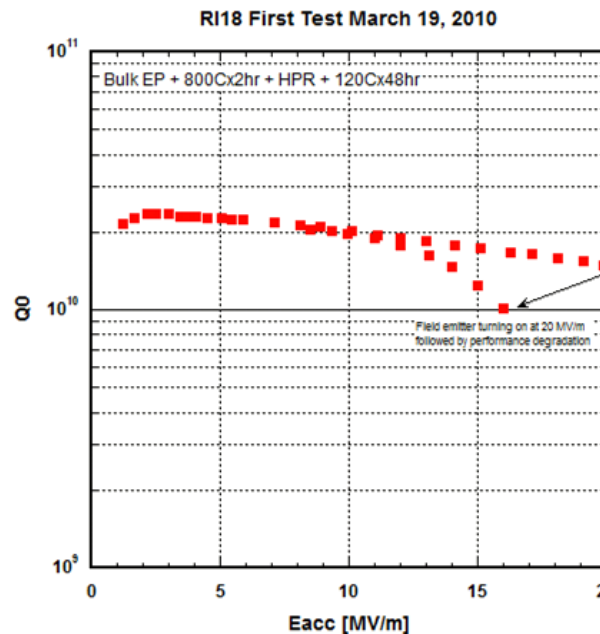
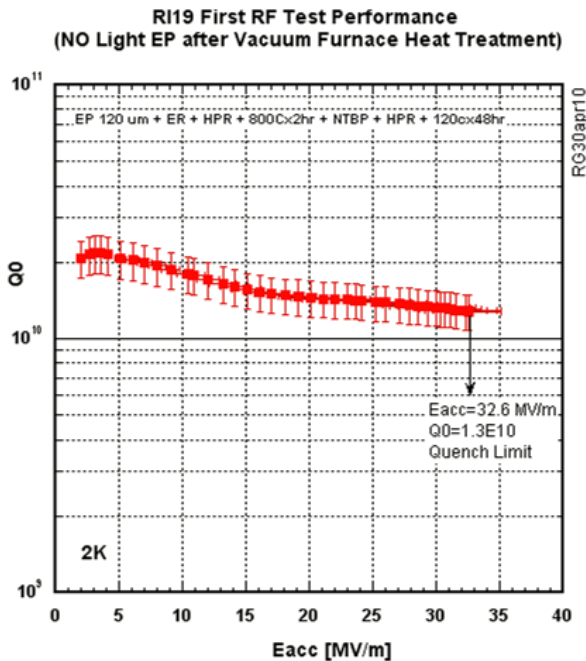


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Fine-Grain 9-cell cavity results

- 9- cell cavity results with no chemistry after 800 C HT

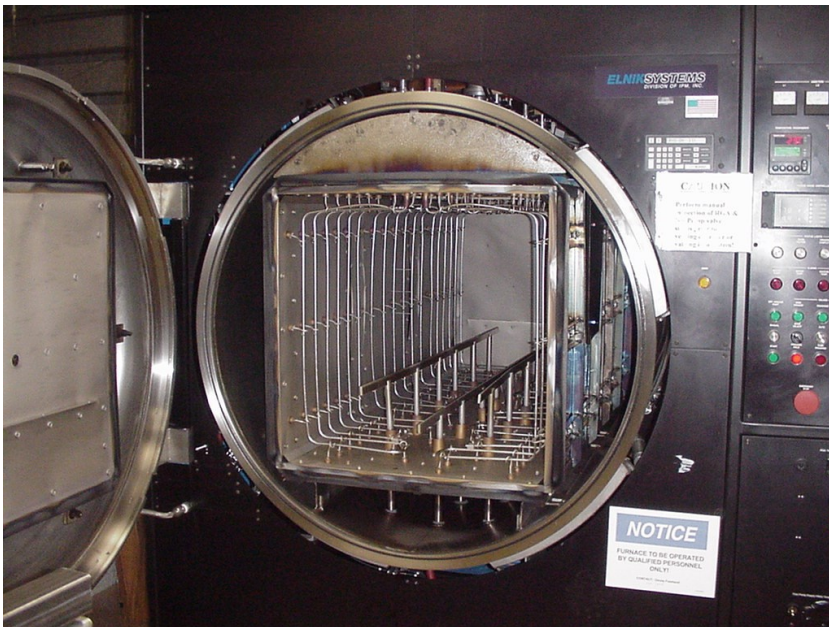


No chemistry after high temperature heat treatment
Only requirement in clean furnace

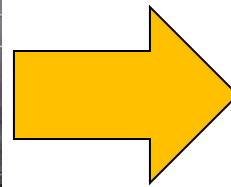
R. Geng, 2010

Induction Heating

New “**All Nb**” furnace is designed with induction heating.



Production Furnace

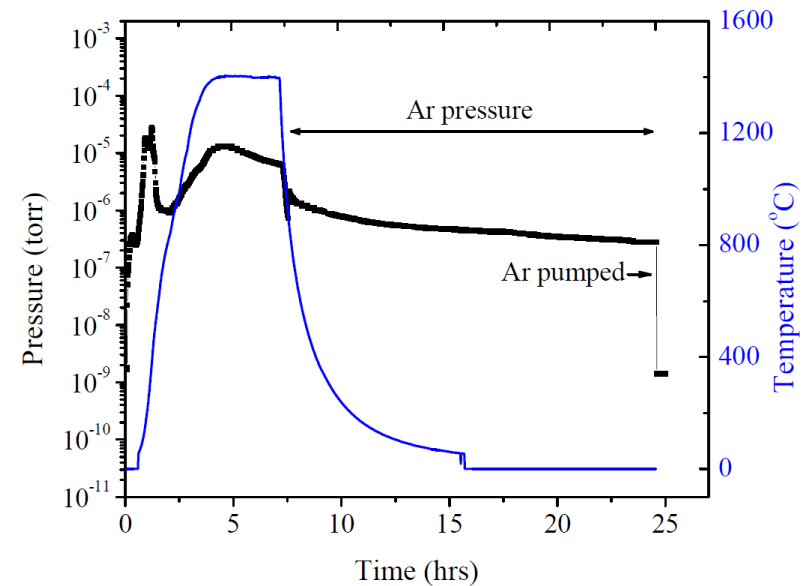


Induction Furnace

Dhakal et al, Rev. Sci. Instrum. 83, 065105 (2012)

Induction Heating

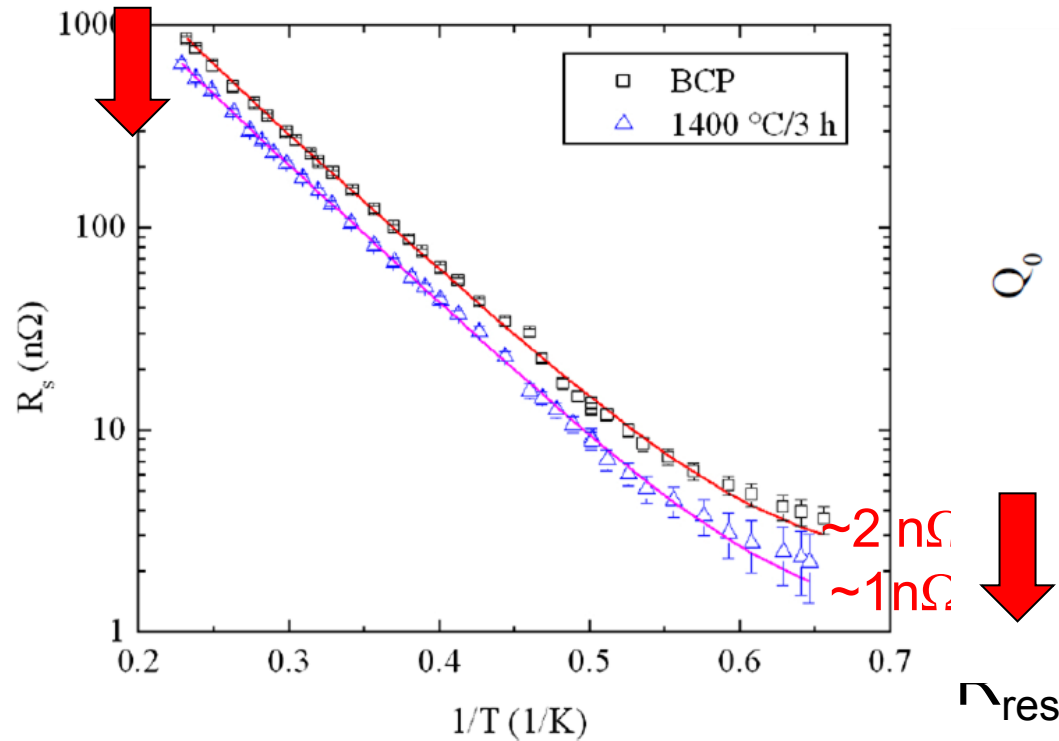
- Heat the cavity in all Nb environment to the target temperature
- Introduce pure Ar while cooling down (this will dilute H_2 and minimize reabsorption)
- Vent the furnace with dry oxygen for better oxidation of the surface of cavity (Surface Passivation)



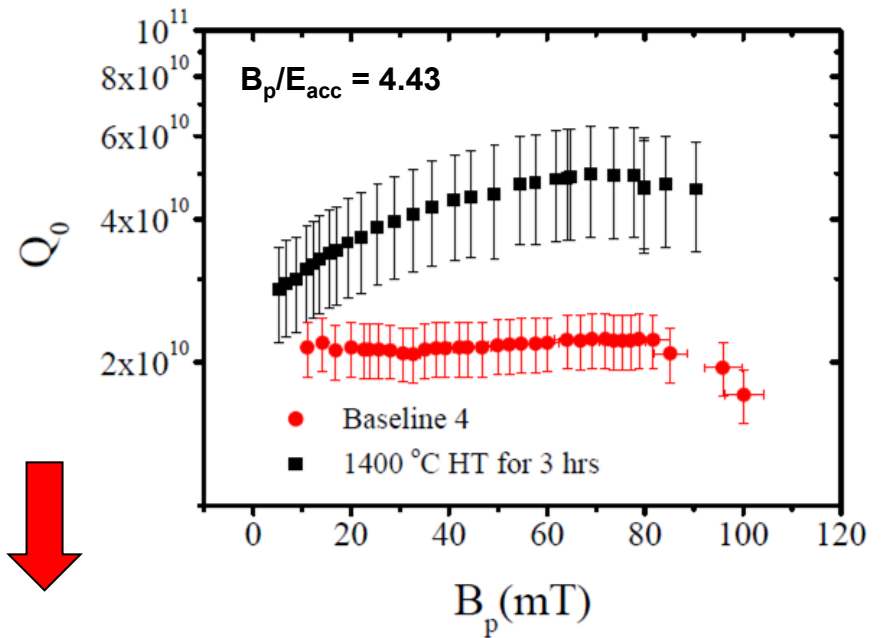
Typical heating curve

R_s vs T data

Reduction in R_{BCS}



4.6×10^{10} @ 20 MV/m



P. Dhakal et al., Phys. Rev. ST Accel. Beams **16**, 042001 (2013)



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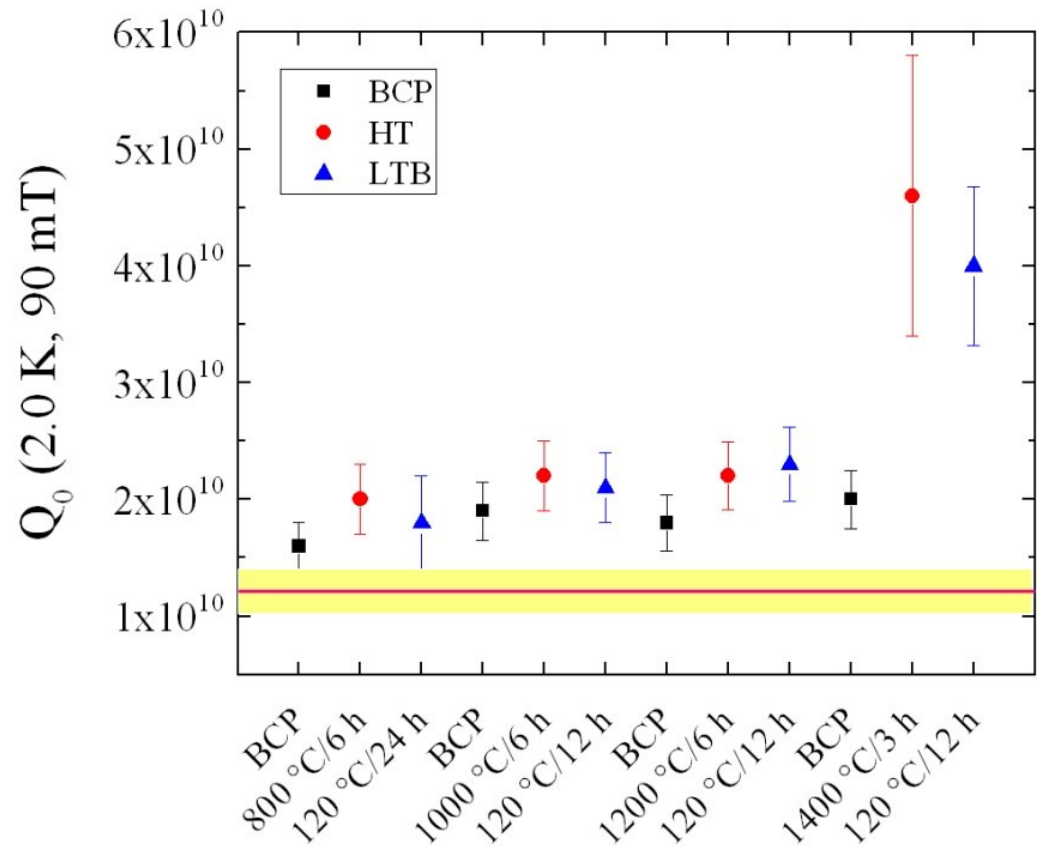
HT extended up to 1400°C with new furnace

- 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

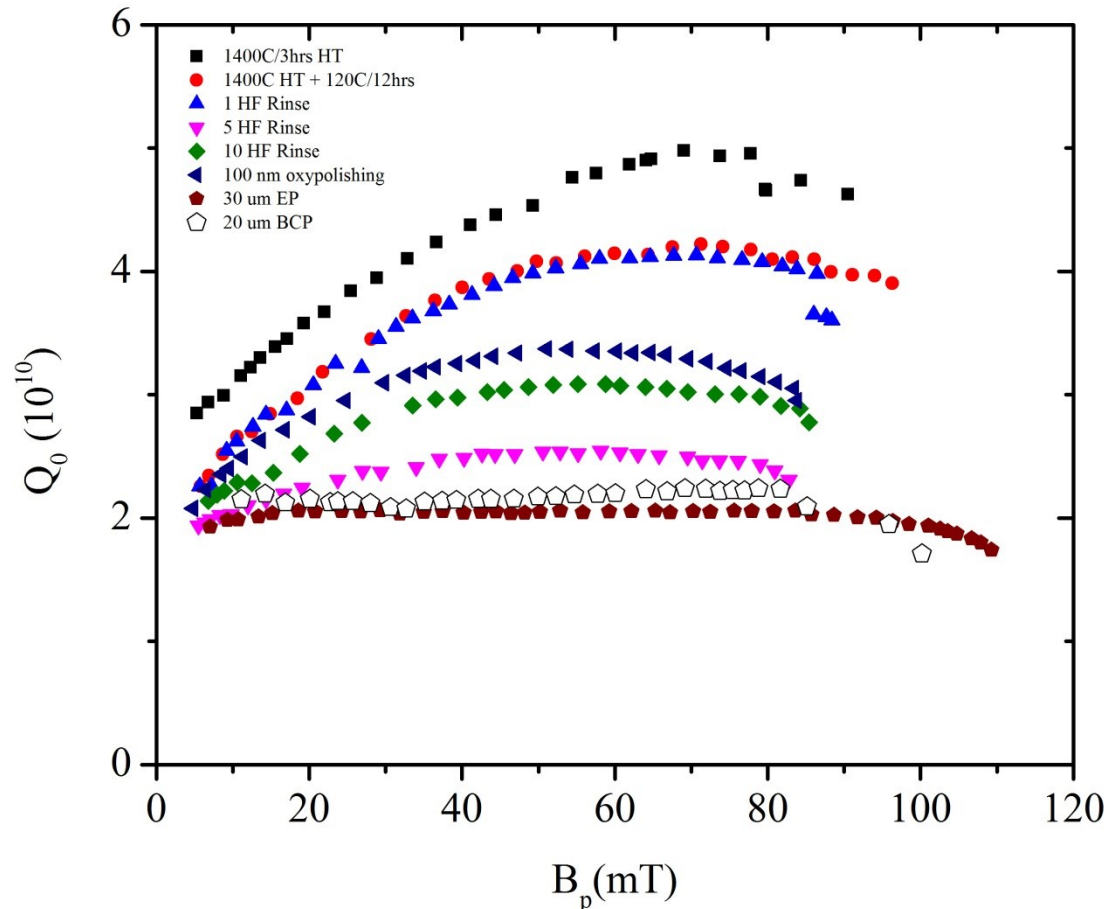
Higher energy gap and reduced broadening parameter



Extended Q-rise

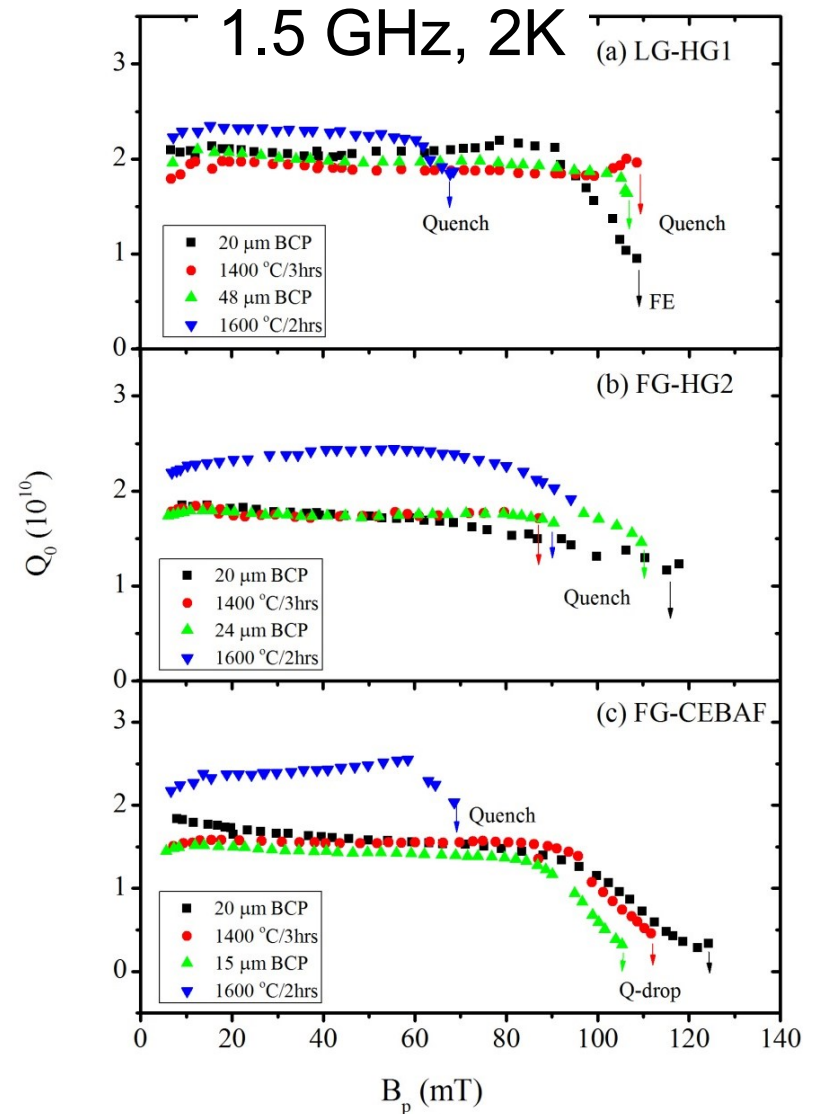
Multiple nano-removal, oxypolishing and EP was done

- 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



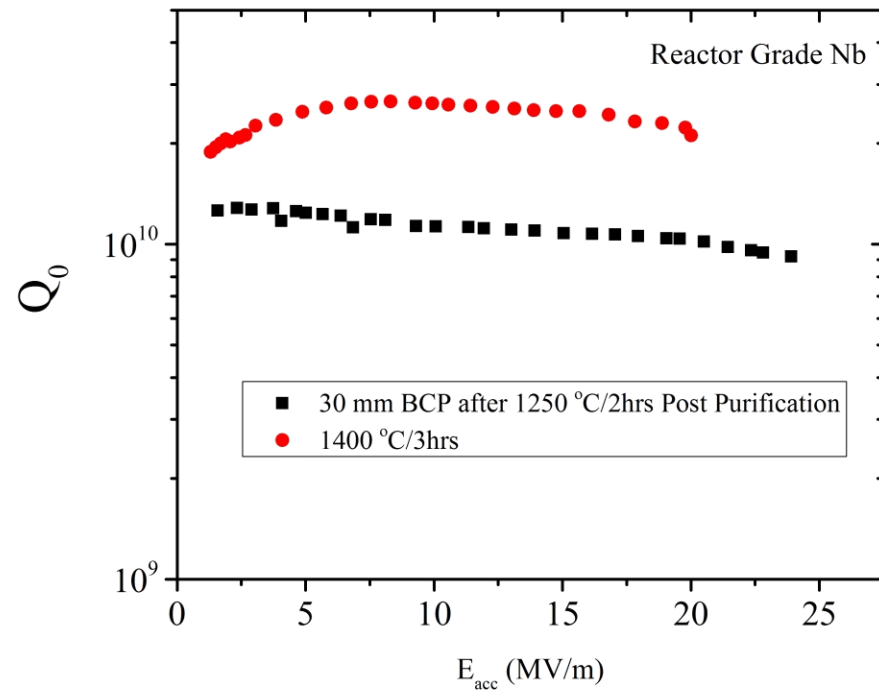
HT results for “All Nb Cavity”

- 3 “all Nb” cavities (2 LG and 1 FG) are heat treated up to 1600 C
- Improvement in Q in medium field range up to ~70%
- Small Q-rise



HT results for Reactor Grade Nb

- RRR ~ 40
- Extended Q- rise up to 35 mT with **factor of 2** improvement in Q at **20 MV/m**
- Cavity was purified in the presence of Ti and surface removal of ~30 μm before the baseline test



Note: In early 80's those high Q cavities are made from reactor grade and heat treated at very high temperatures.

Some Theoretical Models on R_s vs B_p

- Halbritter model based on NbO_x clusters on the surface causes the low field Q-rise
- Two layer superconductors model on Q-rise
- Non-linear BCS and thermal feedback model
- Weingarten model based on the surface defects
- Numerical calculation based on the Mattis and Bardeen theory modified to account for moving Cooper pairs under the action of the rf field

MOIOC02, TUP011



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Future Works

- The reliability of the proposed process to obtain cavities with $Q_0(2.0\text{K}, 90\text{ mT}, 1.5\text{ GHz})$ of $\sim 4 \times 10^{10}$
- Investigation on the impact of surface impurities on the $Q_0(B_p)$ curve through cavity rf tests and studies on small samples cutout from the cavities.



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Conclusions

- High temperature heat treatment *without subsequent chemistry* improves the Q_0 in medium field range, compared to that obtained after BCP and low-temperature (120 C/48h) baking
- $Q_0(2\text{ K}, 90\text{mT})$ as high as 5×10^{10} at 1.5 GHz have been achieved because of an “extended” rise of Q_0 with increasing field
 - Related to the presence of impurities (Ti) near the surface
 - Understanding the origin of this effect is a new challenge in the R&D of bulk Nb