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

Pashupati Dhakal, Giangulai Ciovati,
Peter Kneisel and Ganapati Rao Myneni
Jefferson Lab, USA
09/24/2013

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)

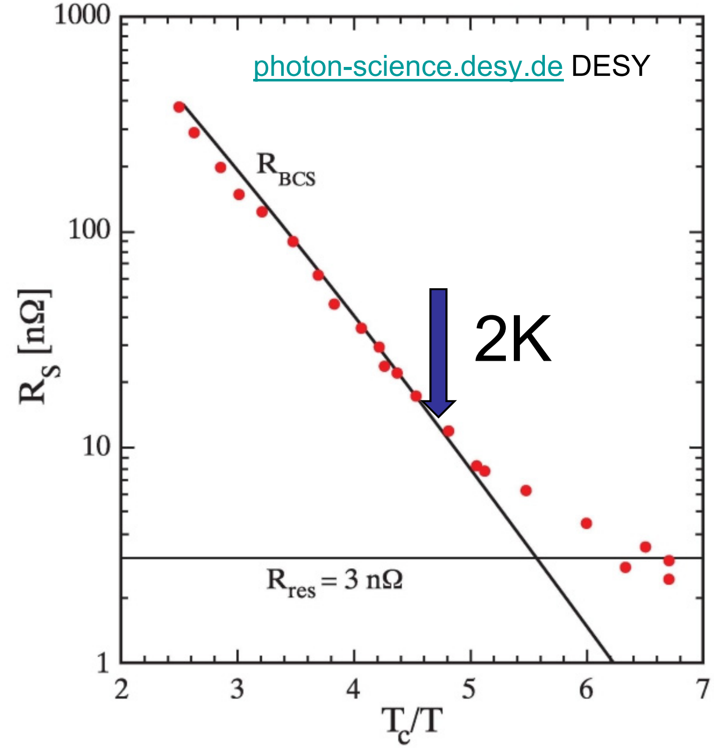
$R_s = R_{res} + R_{BCS}(f, T, \Delta, \lambda_L, \xi_0, l)$

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
- ---

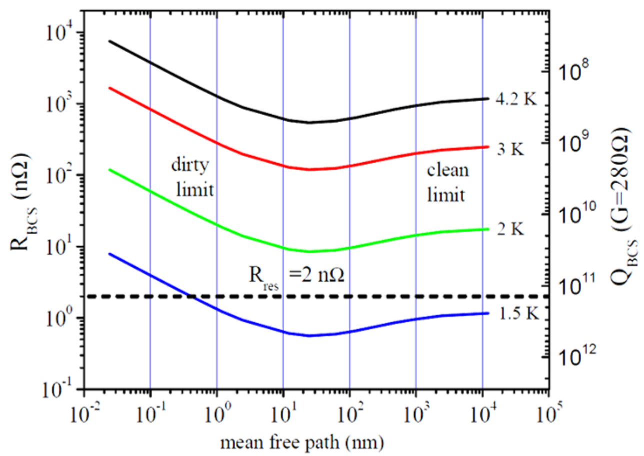


At temperature below 2K, R_s is dominated by R_{res}

Quality Factor and Surface Resistance

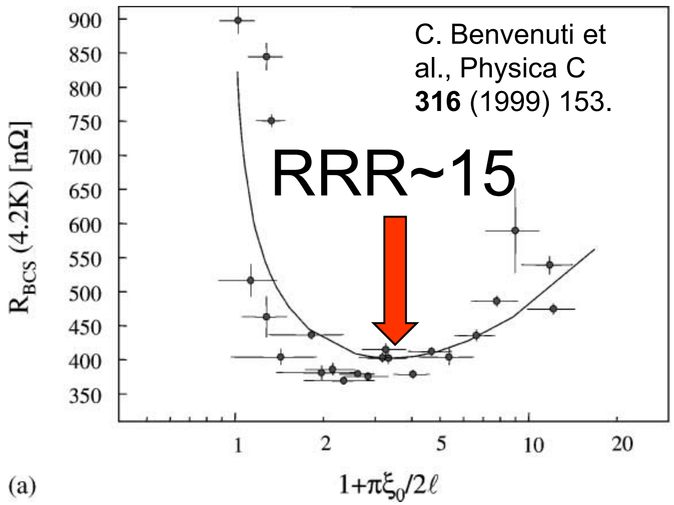
BCS surface resistance results from the interaction between the RF electric field within the penetration depth and thermally activated electrons in a superconductor.

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



Minimizing BCS Resistance

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



Typical SRF cavity Processes

- ~150 μm heavy BCP/ CBP
- Heat treatment 600-800 ºC
- Light BCP/EP (~20μm)
- High Pressure Rinse with DI water
- Low temperature baking (100-140 ºC for 12-48 hours)
- RF Test

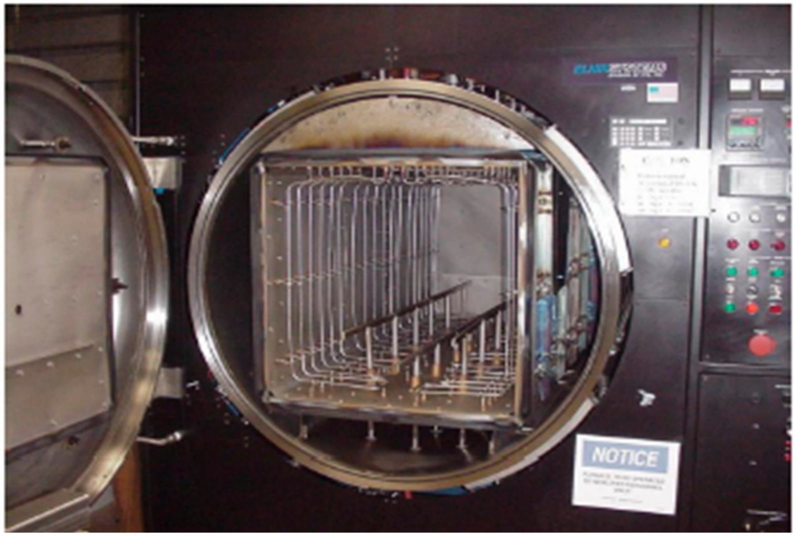
Note: Surface chemistry of ~20 μm is like making new cavity surface

High Temperature Heat Treatment

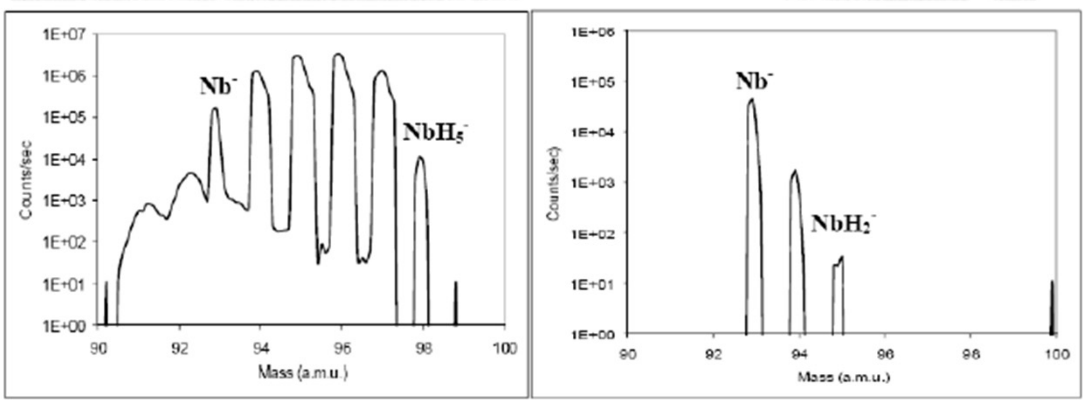
1970s → ~1800 °C UHV HT for ~10 hrs.
1980s → ~1300 °C solid state getter, such as Titanium, was used in-side the furnace to "post-purify".
2000s → 600-800 °C, mainly just to degas hydrogen absorbed by the Nb during cavity fabrication and surface treatments.

- 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.



(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)*

Earlier Results (No post chemistry)

TABLE III. Values of Δ/k_BT_c, R_{res}, R_{BCS}(4.3 K, ≈ 10 mT), Q_0(100 mT)[a], $B_{p,max}$, and improvement factors of Q_0(100 mT), $B_{p,max}$ over the baseline test for all the cavities and rf tests described in Secs. VA 1–VA 4.

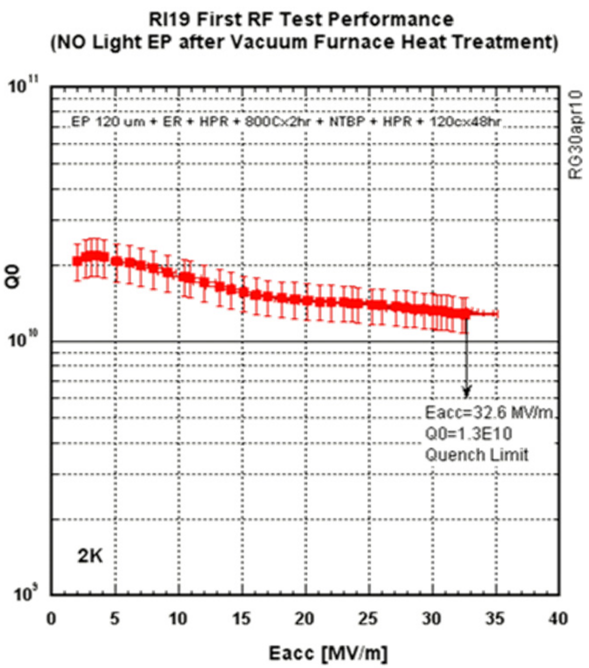
Cavity	Treatment	Δ/k_BT_c	R_{res} (nΩ)	R_{BCS} (nΩ)	Q_0(100 mT) ($\times 10^{10}$)	$B_{p,max}$ (mT)	Q_0 improvement	$B_{p,max}$ improvement
LG CEBAF	Baseline 1 (20 μm BCP)	1.75	11.1	1068	1.05	118		
LG CEBAF	Heat treatment 1 (800°C/3 h, 400°C/20 min N_2)	1.87	10.3	825	1.52	134	45%	14%
LG CEBAF	Baking (120°C/12 h)	1.97	9.7	614	1.88	136	79%	15%
LG CEBAF	Baseline 2 (5 μm BCP)	1.79	5.6	971	0.91	108		
LG CEBAF	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	24%	9%
LG CEBAF	Baseline 3 (2 μm BCP)	1.80	7.9	933	1.07	112		
LG CEBAF	Heat treatment 3 (800°C/3 h, 400°C/20 min)	1.92	3.2	697	1.89	112	77%	0%
SC ILC	Baseline (10 μm BCP, 600°C/10 h, 13 μm BCP)	1.75	4.7	782	0.75	109		
SC ILC	Heat treatment (800°C/3 h, 400°C/20 min N_2, 120°C/6 h)	1.87	4.8	576	1.05	117	40%	7%
SC ILC	Baking (120°C/48 h)	1.98	8.2	414	0.94	115	25%	6%
FG ILC	Baseline (122 μm VEP)	1.80	5.7	724	0.92	122		
FG ILC	Heat treatment (800°C/3 h, 400°C/20 min)	1.85	4.5	656	1.46	137	59%	12%
FG ILC	Baking (120°C/24 h)	2.00	7.9	437	1.40	179	52%	47%
LG ILC	Baseline 3 (1 μm BCP)	1.83	4.9	831	1.16	119		
LG ILC	Heat treatment (800°C/3 h, 120°C/12 h)	2.00	4.2	412	1.44	128	24%	8%

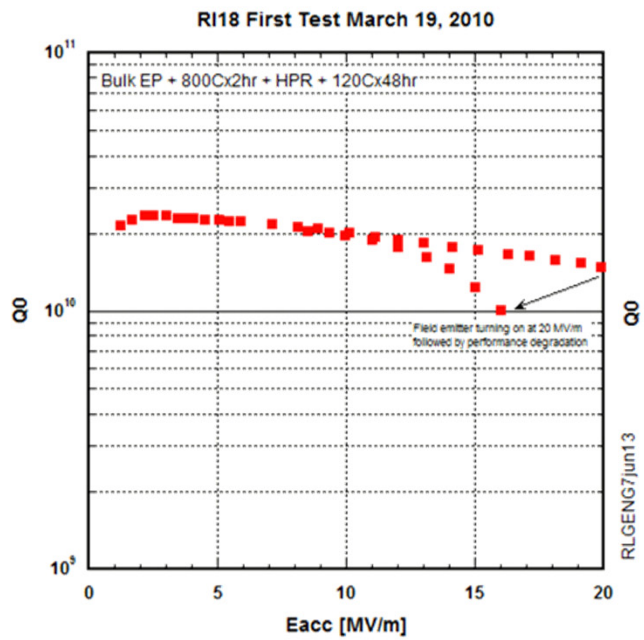
[a]The values of Q_0(100 mT) were measured at 2.0 K, except for the first tests of the large-grain CEBAF cavity when they were measured at 1.7 K.

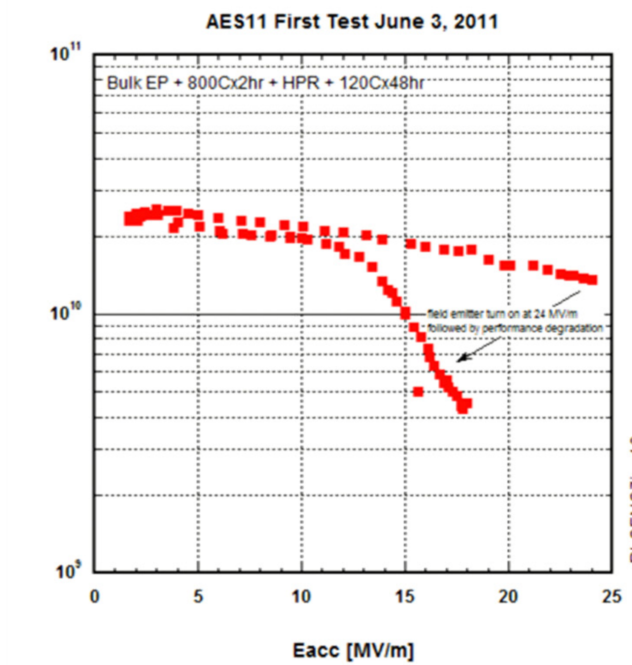
G. Ciovati et al., Phys. Rev. ST Accel. Beams ***13****, 022002 (2010)*

Fine-Grain 9-cell cavity results

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





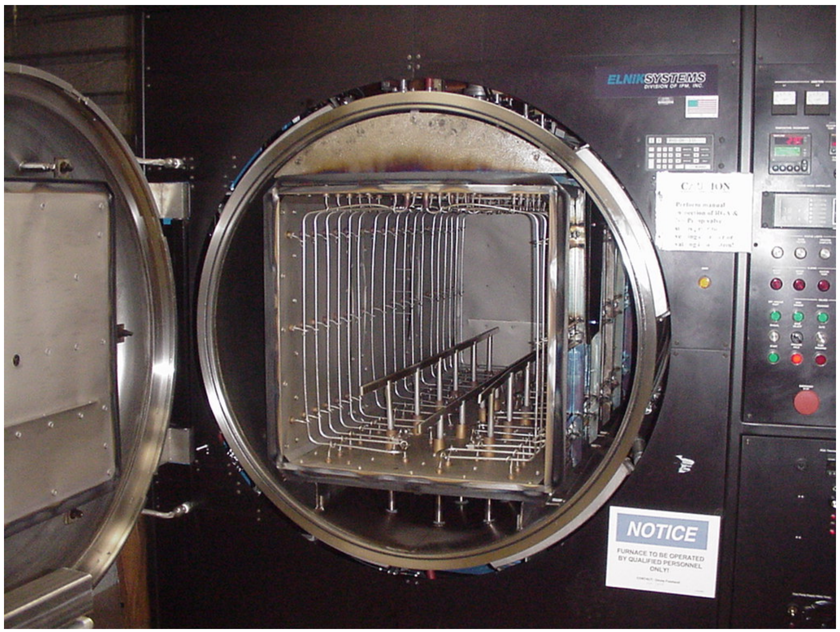


No chemistry after high temperature heat treatment
Only requirement in clean furnace

R. Geng, 2010

Induction Heating

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



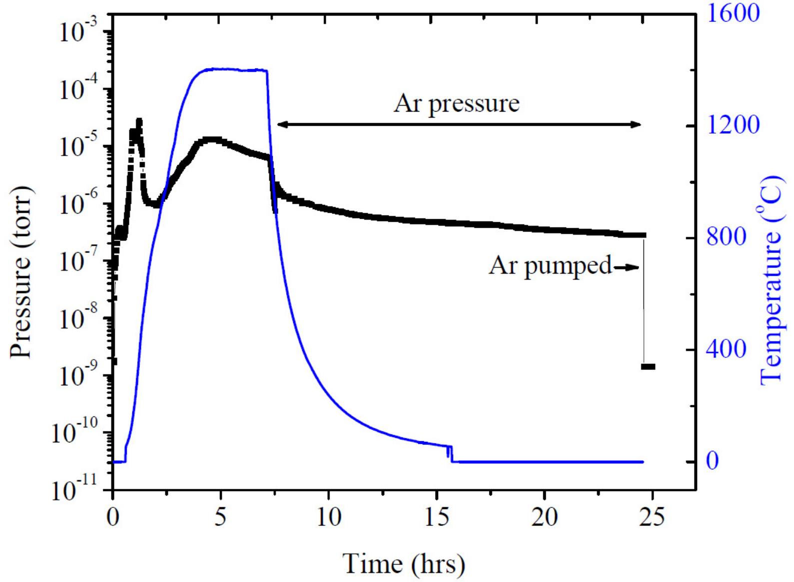
Production Furnace

Induction Furnace

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

Induction Heating

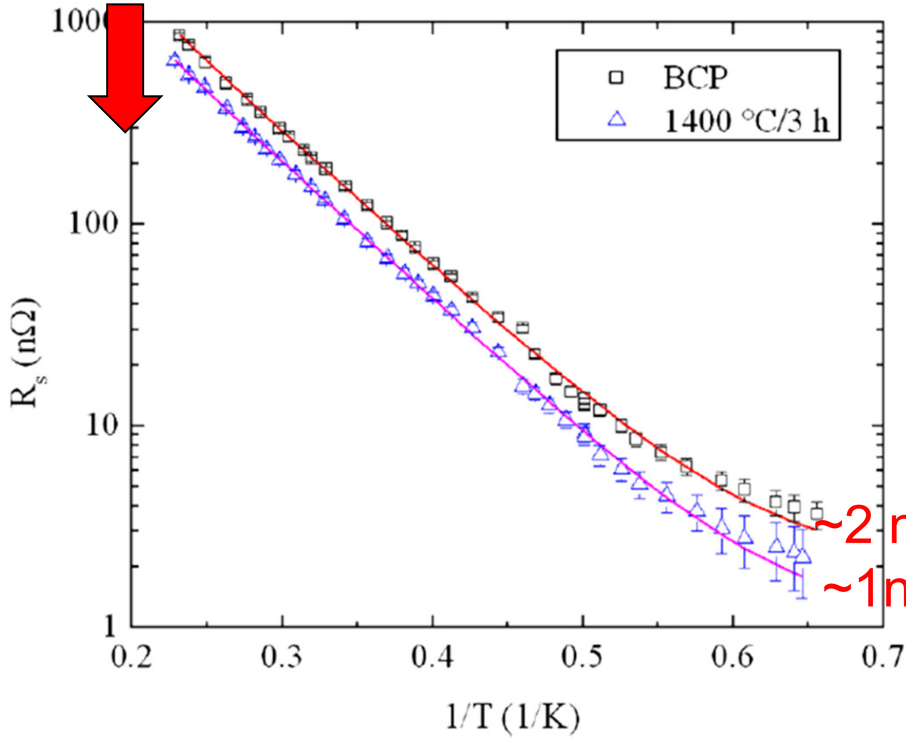
- **Heat the cavity in all Nb environment to the target temperature**
- **Introduce pure Ar while cooling down (this will dilute H_2 and minimize reabsorption)**
- **Vent the furnace with dry oxygen for better oxidation of the surface of cavity (Surface Passivation)**



Typical heating curve

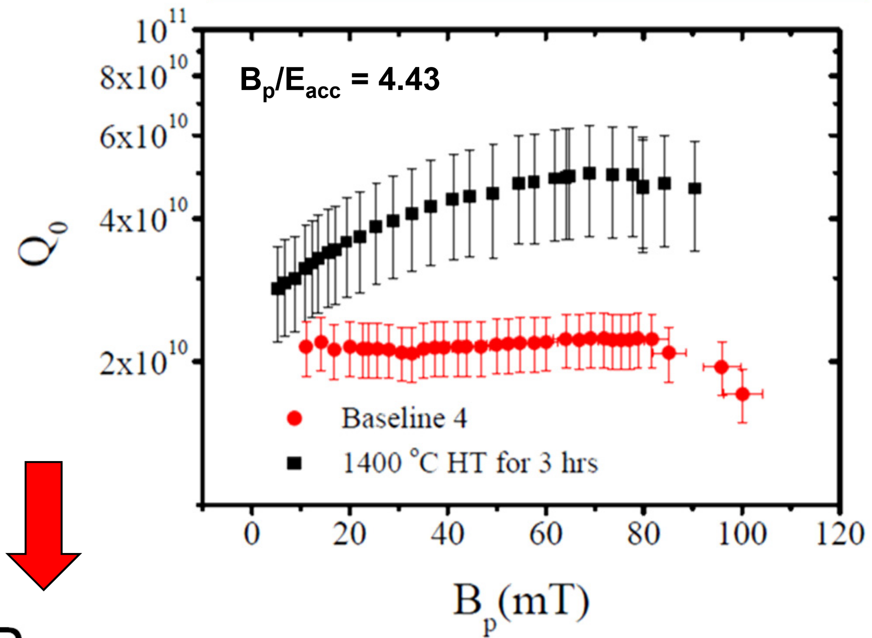
R_s vs T data

Reduction in R_{BCS}



R_{res}

4.6x10[10] @ 20 MV/m



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

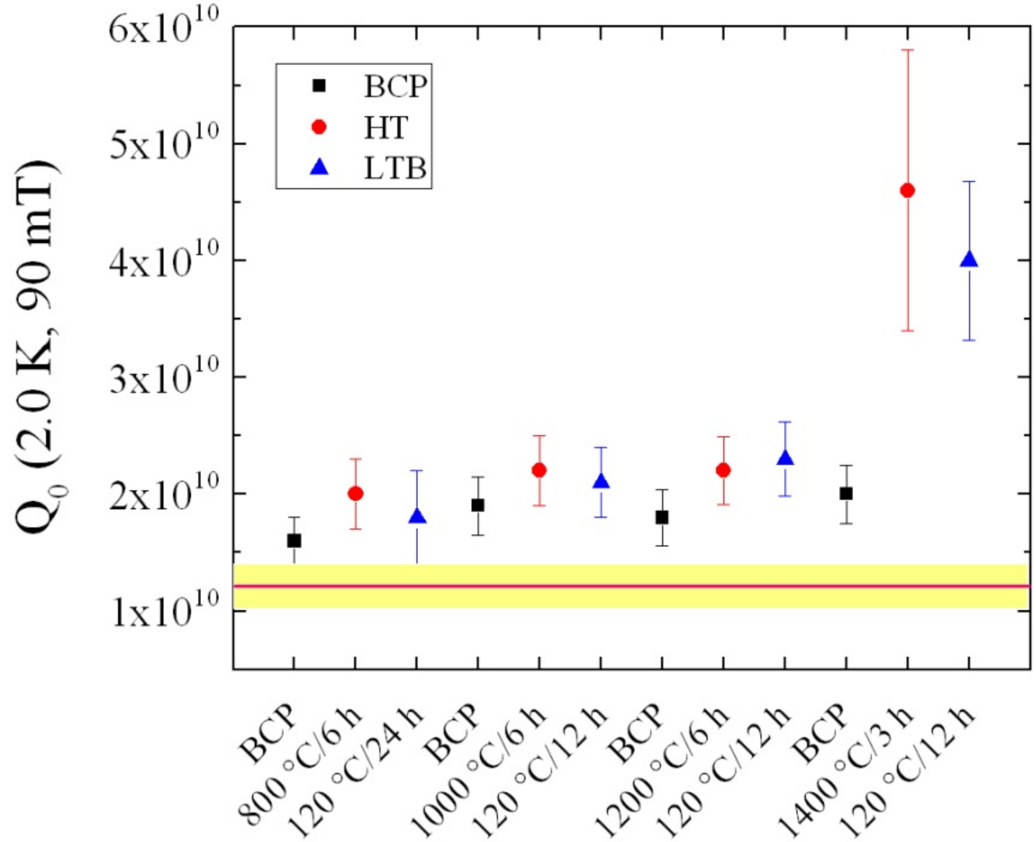
HT extended up to 1400°C with new furnace

- 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

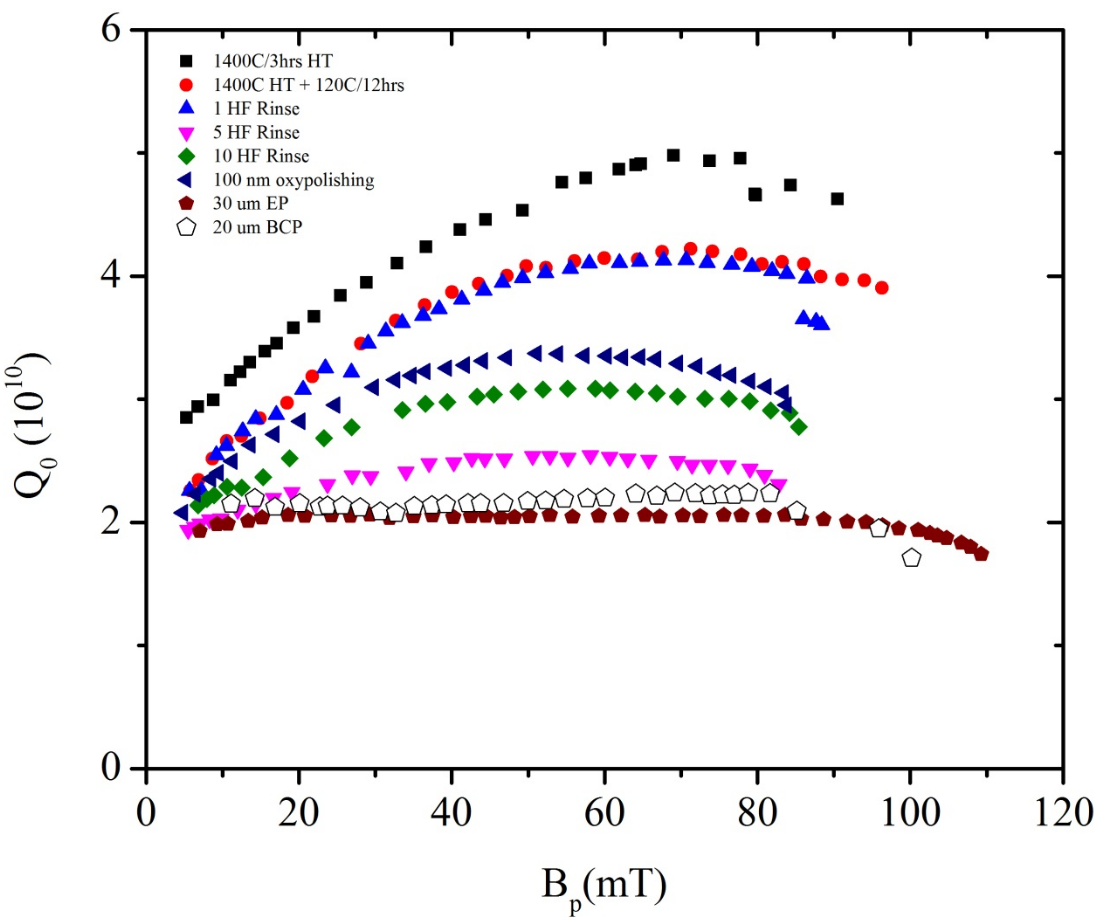


Phys. Rev. ST Accel. Beams **16**, 042001 (2013)

Extended Q-rise

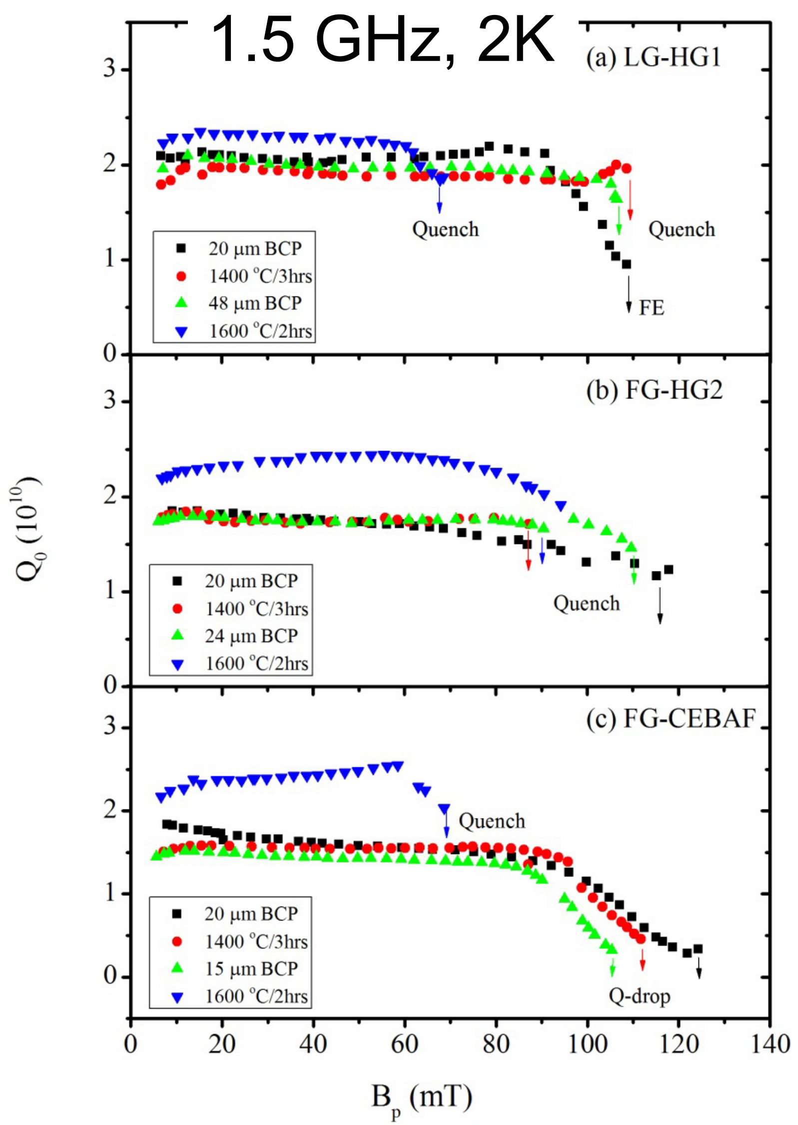
Multiple nano-removal, oxypolishing and EP was done

- 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



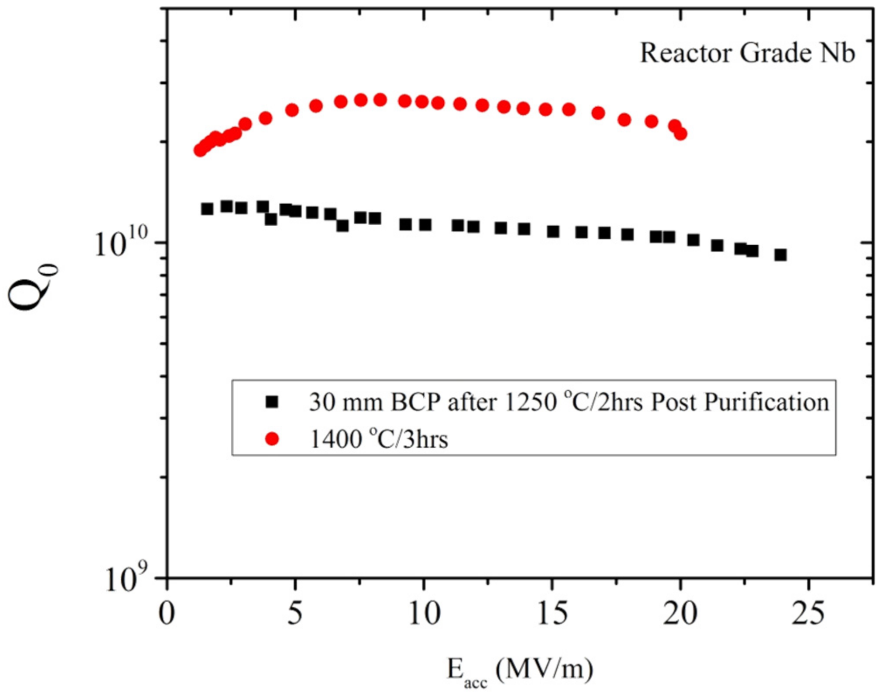
HT results for "All Nb Cavity"

- **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**

Future Works

- The reliability of the proposed process to obtain cavities with Q_0(2.0K, 90 mT, 1.5 GHz) of ~ 4×10^{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.

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 K, 90mT) 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