

COMPARING THE EFFECTIVENESS OF LOW TEMPERATURE BAKE IN EP AND BCP CAVITIES*

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Abstract

Electropolishing (EP) and buffered chemical polishing (BCP) are conventional surface preparation techniques for superconducting radiofrequency (SRF) cavities. Both EP and BCP treated SRF cavities display high field Q-slope (HFQS) which degrades performance at high gradients. While high gradient performance in EP cavities can be improved by introducing oxygen via a low temperature bake (LTB) of 120°C by 48 hours, LTB does not consistently remove HFQS in BCP cavities. There is no consensus as to why LTB is not effective on BCP prepared cavities. We examine quench in EP, BCP, EP+LTB, and BCP+LTB treated 1.3 GHz single-cell Nb cavities by studying the heating behavior with field using a temperature mapping system. Cavity performance is correlated to characterizations of surface impurity profile obtained via time of flight secondary ion mass spectrometry studies. We observe a difference in near surface hydrogen concentration following BCP compared to EP that may suggest that the causes of quench in EP and BCP cavities are different.

INTRODUCTION

Superconducting radiofrequency (SRF) cavities are resonators with extremely low resistivity that enable high performance accelerators. For the realization of the next generation SRF accelerator, we need to push the limits of achievable quality factor (Q_0) and quench field. Recent work has demonstrated the importance of the profile of impurities in the first 100 nm of the surface in achieving high Q_0 and high accelerating gradients (E_{acc}). Introducing a uniform concentration of nitrogen via nitrogen doping has yielded high Q_0 of $> 4 \times 10^{10}$ [1]. Introducing oxygen via *in-situ* baking has been shown to achieve similar effects as nitrogen in the high Q_0 regime [2, 3]. However, the path to reliably reaching high E_{acc} is less clear. Nitrogen infusion, which introduces a sharp inhomogeneous surface disorder, has repeatedly displayed E_{acc} of 45 MV/m, but other cavity treatments have not had much success with consistently reaching such high quench fields [4–7]. The 75/120°C modified LTB has achieved record high quench fields of 50 MV/m, but this treatment has displayed a bifurcation in performance that is not fully understood [7].

There are two main types of limiting factors to achieving high quench fields. First, the thermal breakdown of

superconductivity may occur when inclusions or defects in the cavity drive significant heating and raise the local temperature above the transition temperature. An example of this is the precipitation of dissolved hydrogen within the niobium surface as non-superconducting niobium hydrides [8]. These hydrides cause the proximity breakdown of superconductivity, leading to the phenomena of high field Q-slope (HFQS) [8, 9]. Secondly, quenches of magnetic origin occur when the intrinsic superheating field of Nb is reached [10]. Surface defects and impurities may drive local magnetic field enhancement above the superheating field and cause premature breakdown of superconductivity [10, 11]. Increased surface roughness and the precipitation of impurities at grain boundaries have also been shown to lower quench fields through local field enhancements [4, 11, 12].

In this work, we will be exploring these limiting factors by studying the two conventional surface preparation techniques: electropolishing (EP) and buffered chemical polishing (BCP). In the late 1990s, EP replaced BCP for its superiority at reaching high gradients > 30 MV/m [12–14]. More recent studies have proven that introducing oxygen via low temperature baking (LTB) reliably cures HFQS by suppressing the precipitation of hydrides in EP cavities [15, 16]. However, LTB is not effective at curing HFQS for BCP treated cavities [12, 13]. We study the role of impurities and surface roughness in the performance of EP, BCP, EP+LTB and BCP+LTB treated SRF cavities to better understand the origins of and limiting factors to quench for these treatments.

EXPERIMENTAL METHOD

Two single-cell TESLA shaped Nb cavities with resonant frequency of 1.3 GHz were first degassed at 800°C for 3 hours. The first cavity received 40 μm EP removal while the second cavity received 40 μm BCP removal with the parameters described in Ref. [17]. Both cavities were then low temperature baked at 120°C for 48 hours. The BCP cavity underwent an additional 200°C × 1 hour *in-situ* bake to further diffuse oxygen into the surface. After the initial EP and BCP, the cavities were fully assembled and never reopened to maintain vacuum in between and during treatments.

Following each treatment set, the two cavities were tested at the Fermilab vertical test stand (VTS) to find Q_0 vs. E_{acc} at both 2 K and low T (< 1.5 K) in continuous wave (CW) operation for the decomposition of surface resistance into BCS and residual resistances [18]. Cooling followed the fast cool down protocol to minimize the possibility of trapping magnetic flux [2]. We also investigated how the cavity heated with increasing fields with temperature mapping (TMAP) [7]. 576 carbon resistance temperature detectors (RTDs) were

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installed on the outside of the cavity during assembly, and the temperature at each RTD was recorded during CW testing. The heating profile for each sensor displays the change in temperature as a function of field.

To associate cavity performance with surface composition, we conducted sample studies. Two 1 cm × 1 cm samples made from Nb cavity fabrication drop off were subject to the same treatments as the single-cell cavities as described above. The samples were analyzed with time of flight secondary ion mass spectrometry (SIMS) and laser confocal scanning microscopy (LCSM) [19]. SIMS uses a Cs ion beam to sputter away material and produce secondary ions that are then identified with time-of-flight spectrometry. The concentration of each impurity is normalized to the niobium concentration at each depth. Each reported impurity profile is the average of 3 different 500 μm × 500 μm spots on each sample. Regions with high contamination are excluded from the analysis to better represent the sample. LCSM is used to measure the surface roughness of a surface by varying the objective length distance to determine focal distance and create a 3D profile of the surface. Surface roughness measurements were taken for 6 different scan length from 200 – 1400 μm and are each reported as the average of 6 different scans over 3 locations on each sample.

RESULTS AND DISCUSSION

There are many similarities between the performance of EP and BCP cavities. Figure 1 shows that both EP and BCP display HFQS onset at around 20 – 25 MV/m that arises from an increase in residual resistance at the same fields (Fig. 2). Cavity performance diverges after LTB. EP+LTB no longer displays HFQS; BCP+LTB still exhibits HFQS, but the onset increases from 20 MV/m to 23 MV/m. The bottom plot of Fig. 2 shows that BCS resistance (R_{BCS}) both with and without LTB are consistent up until the onset of HFQS, confirming that the diffusion of oxygen via LTB is

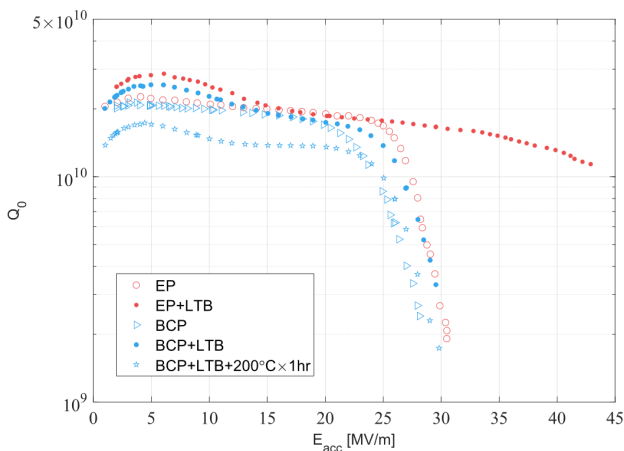


Figure 1: Q_0 vs. E_{acc} data taken at 2 K acquired at Fermilab VTS system.

comparable. There appears to be a knee in R_{BCS} at 20 MV/m that suggests an evolution towards anti-Q slope behavior [2].

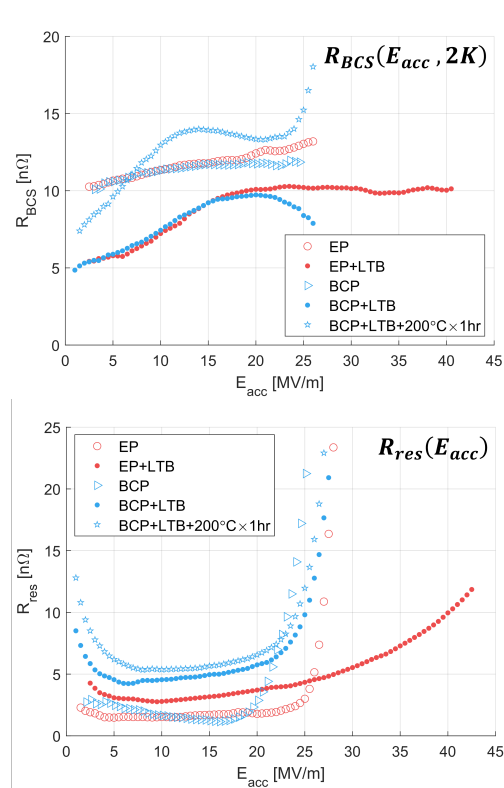


Figure 2: (top) BCS resistance at 2 K and (bottom) residual resistance with field without trapped flux contributions.

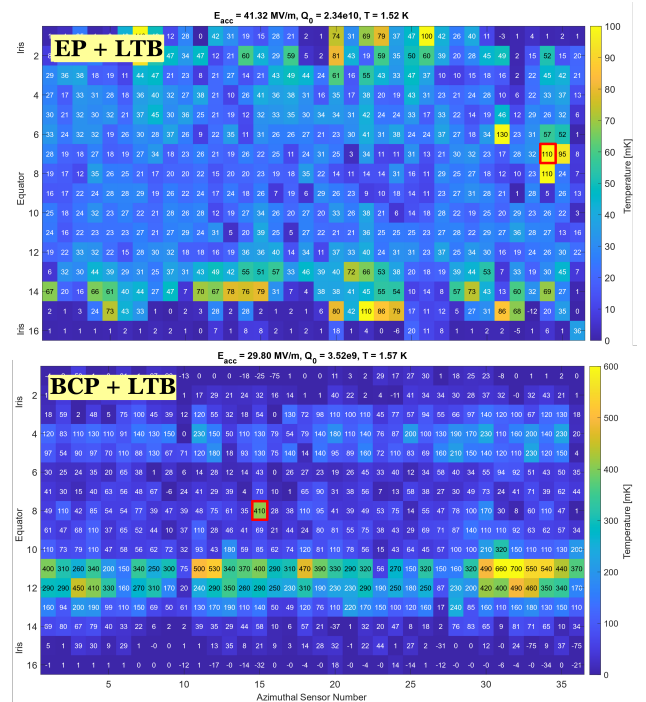


Figure 3: Maps of the temperature within the (top) EP+LTB cavity and (bottom) BCP+LTB cavity just before quench. Respective quench locations are labeled in red.

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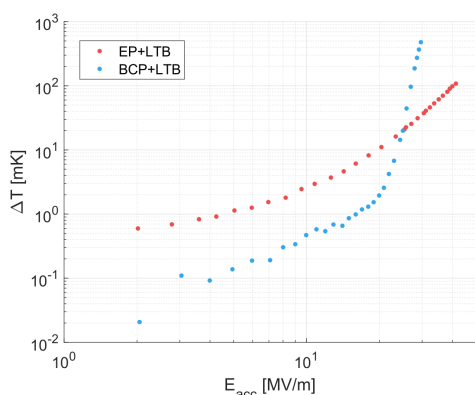


Figure 4: Heating profile at quench RTD for each cavity.

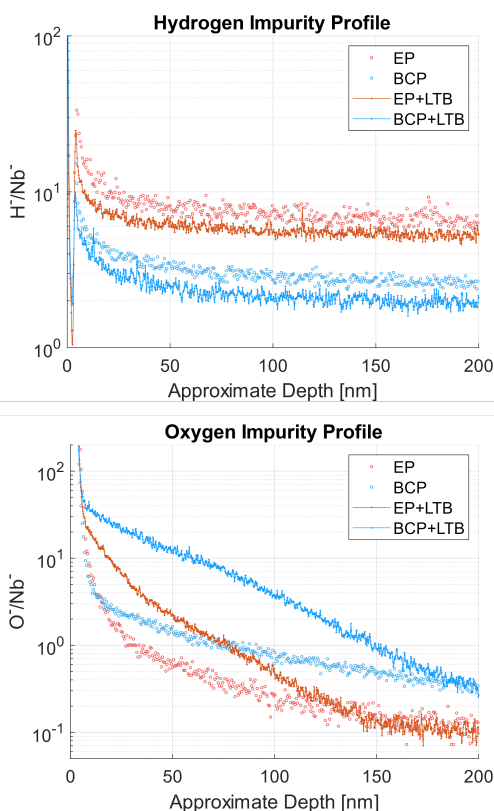


Figure 5: SIMS data showing impurity depth profiles of (top) hydrogen and (bottom) oxygen. H^-/O^- signal is normalized to the detected Nb^- signal.

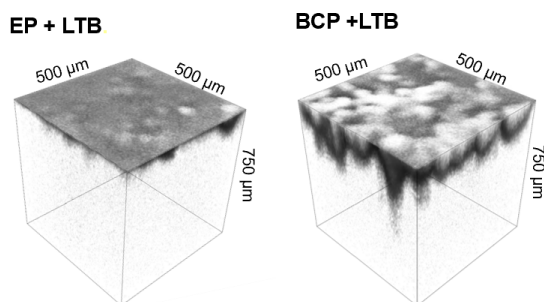


Figure 6: 3D rendering of O concentration with depth from SIMS for (left) EP+LTB and (right) BCP+LTB.

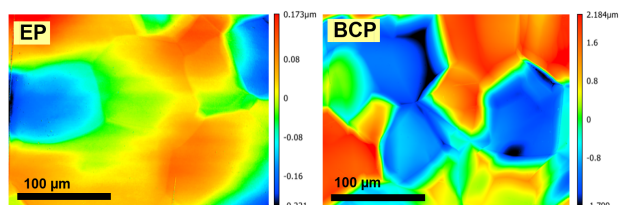


Figure 7: 3D profile of sample surface acquired with LCSM for (left) EP and (right) BCP.

Figure 3 contains two TMAPs of the temperature within the cavities just before quench. The sensors at which quench occurred are labeled in red. The presence of bands of heating in the TMAP for the BCP+LTB cavity is typically an indication of HFQS in EP cavities [15]. TMAP results at the location of quench are summarized in Fig. 4. The heating profile for BCP+LTB displays a sharp change in slope reminiscent of HFQS. Prior to HFQS onset, there is less heating in BCP+LTB compared to EP+LTB. TMAPs and heating profiles confirm that while EP+LTB successfully cured HFQS, BCP+LTB had little to no effect in improving performance, suggesting that the hydrides precipitating in BCP cavities are not fully suppressed by LTb.

To introduce more oxygen to suppress hydrides, we baked the cavity for an additional 200 °C for 1 hour. Typically, 200 °C × 1 hour on EP cavities diffuses enough oxygen to observe a knee in R_{BCS} at 12 MV/m [2]. We observe the same knee with BCP as the base treatment but still observe HFQS. Our hypothesis of BCP having more hydrides was also disproved by the SIMS results shown in Fig. 5. The BCP sample contained only half the concentration of H as the EP sample. After LTb, the concentration of H decreases for both EP and BCP. There also appears to be a higher concentration of O in the BCP sample. Figure 6 is a 3D rendering of the concentration of O in two 500 μm × 500 μm regions, one for EP+LTb and one for BCP+LTb. Compared to the relatively uniform EP+LTb surface, there are regions of significant O concentration in the BCP+LTb surface. These regions of high O are likely from the increased surface area of some Nb grains compared to other grains. The variability across grains makes it difficult to get a good representative sample of a BCP surface without sampling a larger area. The increase in HFQS onset from 20 MV/m to 23 MV/m may be the hydride trapping effect of oxygen diffused from LTb. No additional increase in HFQS onset is observed with 200 °C × 1 hour. Hydrogen does not appear to be the main driver of degradation in Q for BCP cavities.

We therefore turned to surface roughness, another limiting factor of quench. Figure 7 shows that an EP surface is much smoother than a BCP surface, which displays distinct grain structure and grain boundaries. This suggests the BCP preferentially etches the grain boundaries. The grain boundary structures are of the same size as the regions of increased O concentration in Fig. 6. No distinct differences in non-H or O containing impurities were observed in SIMS, but a more careful examination of the grain boundaries is necessary to fully rule out the precipitation of impurities as the cause

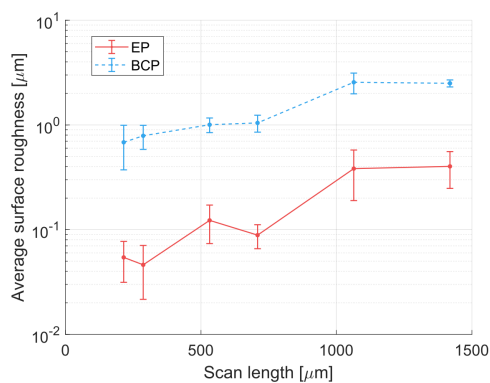


Figure 8: Average surface roughness as a function of scan length acquired from LCSM.

of quench. The surface roughness in the BCP sample is an order of magnitude larger than that of EP for all scan lengths (Fig. 8). Surface roughness may also drive Q degradation and local field enhancements at the grain boundaries in BCP cavities.

CONCLUSION

Despite the similarities in Q_0 vs. E_{acc} , residual resistance, BCS resistance and heating patterns of EP and BCP cavities, SIMS data suggests that Q degradation in BCP treated cavities is not entirely caused by hydrides. There appears to be less free hydrogen in a BCP treated surface than an EP treated surface, but hydrogen concentration drops for both following LTB. The onset of HFQS increases for BCP+LTB, but the degradation in Q is still present. From the 3D profiles of samples, BCP appears to preferentially etch the grain boundaries, increasing surface roughness by an order of magnitude compared to EP. Q degradation in BCP may be from the preferential etching of grain boundaries instead of from hydrides, which makes LTB an ineffective treatment for eliminating HFQS-like performance in BCP cavities. Further sample studies are necessary to determine whether the local field enhancements are from surface roughness, impurities precipitating at the grain boundaries, or a combination of both.

REFERENCES

- [1] A. Grassellino *et al.*, “Nitrogen and argon doping of niobium for superconducting radio frequency cavities: a pathway to highly efficient accelerating structures”, *Supercond. Sci. Tech.*, vol. 26, p. 102001, 2013. doi: 10.1088/0953-2048/26/10/102001
- [2] D. Bafia *et al.*, “The role of oxygen concentration in enabling high gradients in niobium SRF cavities”, in *Proc. SRF’21*, East Lansing, MI, USA, Jun.-Jul. 2021. 10.18429/JACoW-SRF2021-THPTEV016
- [3] E. M. Lechner *et al.*, “RF surface resistance tuning of superconducting niobium via thermal diffusion of native oxide”, *Appl. Phys. Lett.*, vol. 119, p. 082601, 2021. doi: 10.1063/5.0059464
- [4] A. Grassellino *et al.*, “Unprecedented quality factors at accelerating gradients up to 45 MV/m in niobium superconducting

resonators via low temperature nitrogen infusion”, *Supercond. Sci. Tech.*, vol. 30, 2017.

doi: 10.1088/1361-6668/aa7afe

- [5] V. Ngampruetikorn and J. A. Sauls, “Effect of inhomogeneous surface disorder on the superheating field of superconducting RF cavities”, *Phys. Rev. Res.*, vol. 1, p. 012015, 2019. doi: 10.1103/PhysRevResearch.1.012015
- [6] S. Posen *et al.*, “Ultralow surface resistance via vacuum heat treatment of superconducting radiofrequency cavities”, *Phys. Rev. Applied*, vol. 13, p. 014024, 2020. doi: 10.1103/PhysRevApplied.13.014024
- [7] D. Bafia, “Exploring and Understand the Limitations of Nb SRF Cavity Performance”, Ph.D. thesis, Phys. Dept., Illinois Institute of Technology, Chicago, IL, 2020.
- [8] P. G. De Gennes and J. P. Hurault, “Proximity Effects Under Magnetic Fields: II - Interpretation of ‘Breakdown’”, *Phys. Lett.*, vol. 17, p. 03181, 1965. doi: 10.1016/0031-9163(65)90468-3
- [9] F. Barkov *et al.*, “Precipitation of Hydrides in High Purity Niobium after Different Treatments”, *J. Appl. Phys.*, vol. 114, p. 164904, 2013. doi: 10.1063/1.4826901
- [10] H. Padamsee, J. Knobloch, and T. Hays, *RF Superconductivity for Accelerators*. Weinheim, Germany: Wiley-VCH GmbH, 1998.
- [11] T. Kubo, “Magnetic Field Enhancement at a Pit on the Surface of a Superconducting Accelerating Cavity”, *Prog. Theor. Exp. Phys.*, vol. 2015, p. 073G01, 2015. doi: 10.1093/ptep/ptv088
- [12] K. Saito *et al.*, “Superiority of electropolishing over chemical polishing on high gradients”, in *Proc. SRF’97*, Padova, Italy, Oct. 1997, paper SRF97D02, pp. 795-813.
- [13] L. Lilje *et al.*, “Improved Surface Treatment of the Superconducting TESLA Cavities”, *Nucl. Instrum. Methods Phys. Res., Sect. A* vol. 516, pp. 213-227, 2004. doi: 10.1016/j.nima.2003.08.116
- [14] P. Kneisel, “Preliminary Experience with in-Situ Baking of Niobium Cavities”, in *Proc. SRF’99*, Santa Fe, NM, USA, Nov. 1999, paper TUP044, pp. 328-335.
- [15] A. Romanenko, “Surface characterization of Nb cavity sections - understanding the high field Q-slope”, Ph.D. thesis, Cornell University, Ithaca, NY, 2009.
- [16] A. Romanenko *et al.*, “Effect of mild baking on superconducting niobium cavities investigated by sequential nanoremoval”, *Phys. Rev. ST Accel. Beams* vol. 16, pp. 012001, 2013. doi: 10.1103/PhysRevSTAB.16.012001
- [17] M. P. Kelly and T. Reid, “Surface processing for bulk niobium superconducting radio frequency cavities”, *Supercond. Sci. Technol.*, vol. 30, p. 043001, 2017. doi: 10.1088/1361-6668/aa569a
- [18] M. Martinello *et al.*, “Effect of interstitial impurities on the field dependent microwave surface resistance of niobium”, *Appl. Phys. Lett.*, vol. 109, p. 062601, 2016. doi: 10.1063/1.4960801
- [19] A. S. Romanenko *et al.*, “First Direct Imaging and Profiling TOF-SIMS Studies on Cutouts from Cavities Prepared by State-of-the-Art Treatments”, in *Proc. SRF’19*, Dresden, Germany, Jun.-Jul. 2019, pp. 866-870. doi: 10.18429/JACoW-SRF2019-THP014