ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP061

GRADIENTS OF 50 MV/m IN TESLA SHAPED CAVITIES VIA MODIFIED LOW TEMPERATURE BAKE*

D. Bafia[1]†, A. Grassellino, Z. Sung, A. Romanenko, O. S. Melnychuk,
Fermi National AcceleratorLaboratory, Batavia, USA
J. F. Zasadzinski, [1]Illinois Institute of Technology, Chicago, USA

Abstract

This paper will discuss the 75/120 C modified low temperature bake capable of giving unprecedented accelerating gradients of above 50 MV/m for 1.3 GHz TESLA-shaped niobium SRF cavities in CW operation. A bifurcation in the Q_0 vs E_{acc} curve is observed after retesting cavities without disassembly in between, yielding performance that ranges from exceptional to above state-of-the-art. Atomic Force Microscopy studies on cavity cutouts gives a possible mechanism responsible for this branching in performance, namely, the dissociation and growth of room temperature niobium nano-hydrides that exist near the RF surface, which are made superconducting only through the proximity effect. In-situ low temperature baking of cavity cutouts reveals a dissociation of these room temperature nano-hydrides, which could explain the higher performance of cavities subject to similar in-situ heating in the dewar.

INTRODUCTION

SRF cavities are a key technology for efficient particle accelerating. To help realize future accelerators such as the International Linear Collider (ILC), higher accelerating gradients must be consistently achieved. The current ILC surface treatment, the 120 C bake, regularly produces single cell cavities that give quench fields of ~45 MV/m [1,2]. However, to lower the cost of future accelerators, this quench field must be further improved. This paper discusses a new high gradient surface treatment for SRF cavities, the 75/120 C modified low temperature, developed at Fermi National Accelerator Laboratory (FNAL) [3,4]. This surface treatment can produce single cell cavities with quench fields of above 50 MV/m. The performance of cavities subject to this surface treatment is presented along with possible microscopic origins for this dramatic increase in the quench field.

HIGH GRADIENT SURFACE TREATMENTS

All niobium SRF cavities used in this study are TESLA-shaped with a resonant frequency of 1.3 GHz. The cavities are subject to the 75/120 C bake outlined in Table 1. As described in [3,4], the cavities are first heated to 800 C for 3 hours in ultra-high vacuum to degas the cavity surface of any impurities, leaving the RF surface clean. Second, a mild bake is applied, in which the cavities are baked at 75 C for 4 hours. Finally, the cavities are baked for 48 hours at 120 C in UHV. This differs from the standard 120 C bake in that there is an additional mild bake applied before the final treatment. Figure 1 displays a plot of the furnace residual gas analyser (RGA) while the cavities underwent this surface treatment. To obtain measures of the resulting performance, cavities were RF tested at FNAL's vertical test stand (VTS).

Table 1: Comparison of High Gradient Treatments

120 C Bake	75/120 C Bake
800 C x 3 hr in UHV	800 C x 3 hr in UHV
N/A	75 C x 4 hrs in UHV
120 C x 48 hr in UHV	120 C x 48 hr in UHV

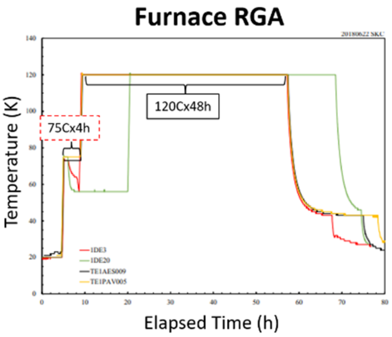

Figure1: Furnace RGA plot of a 75/120 C bake cycle.

HIGH GRADIENT SURFACE TREATMENTS

Unprecedented Performance

Figure 2 shows a comparison of Q_0 vs E_{acc} curves for a typical 120 C baked cavity and a 75/120 C baked cavity. The former has a quench field of ~41 MV/m while the latter quenches at ~50 MV/m, one of the highest obtained for this cavity shape. Many high gradient single cell cavities have been created using this 75/120 C bake; a histogram depicting the quench fields of these cavities is shown in Figure 3. Note that the 3 cavities that quench below 28 MV/m were found to have physical defects that likely limited the performance. Figure 4 shows Q_0 vs E_{acc} curves for 7 cavities subject to this 75/120 C bake that achieve quench fields at or above 48 MV/m, showing that these high gradients are indeed reproducible. In addition to very high

---

* This work was supported by the United States Department of Energy, Offices of High Energy Physics under Contracts No. DE-AC05- 06OR23177
† dbafia@fnal.gov

quench fields, the 75/120 C bake also gives a lower sensitivity to trapped magnetic flux when compared to a standard 120 C bake cavity, as shown in Figure 5.

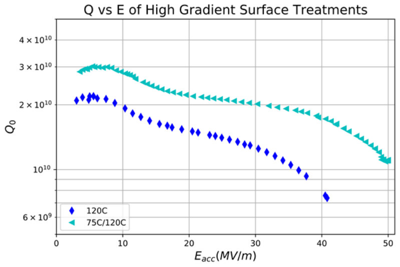

Figure 2: Quality factor vs accelerating gradient curves taken at a temperature of 2K of a standard 120 C bake cavity and a cavity post 75/120 C bake.

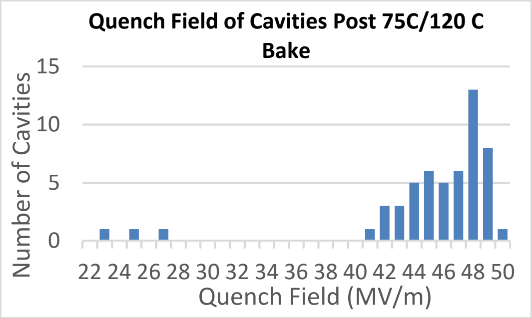

Figure 3: Histogram depicting quench fields of cavities subject to the 75/120 C bake.

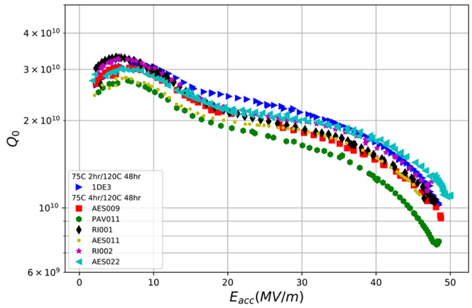

Figure 4: Q_0 vs E_{acc} curves of cavities post 75/120 C bake with quench fields of ~49 MV/m.

Cavity AES022 post 120 C bake has a sensitivity to trapped magnetic flux of 1.0 nΩ/mG at 30 MV/m. However, after resetting the cavity with a 40 µm removal of the surface via electropolishing (EP) and treating with the 75/120 C bake, this sensitivity decreases to ~0.8 nΩ/mG. This difference in sensitivity could hint at a difference in the pinning strength of vortices within the cavity, giving insight on possible mechanisms responsible for the larger increase in performance.

Bifurcation in Performance

A puzzling effect is observed for cavities subject to this 75/120 C bake; a bifurcation in the Q_0 vs E_{acc} curves. This bifurcation is outlined in Figure 6. Cavity 1DE3 was treated with the 75/120 C bake and tested on 05/08, where it achieved a quench field of ~49 MV/m, an exceptionally high gradient. However, the cavity was retested on 05/31 without disassembly (or loss of vacuum) and quenched at 41 MV/m; the Q_0 was also significantly lower. Cavity AES009 post 75/120 C bake also quenched at ~49 MV/m the first time it was tested. Again, a retest of the cavity without disassembly after the first test gave a quench field of ~43 MV/m with a Q_0 lower than what was previously measured. Cavity AES022, on the other hand, first quenched at ~43 MV/m and later experienced an increase in quench by +3 MV/m, up to ~46 MV/m, again without disassembly in between tests.

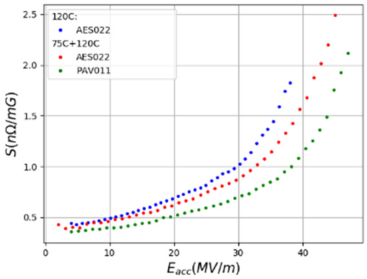

Figure 5: Sensitivity to trapped magnetic flux plotted against the accelerating gradient for cavities subject to surface treatments outlined in Table 1.

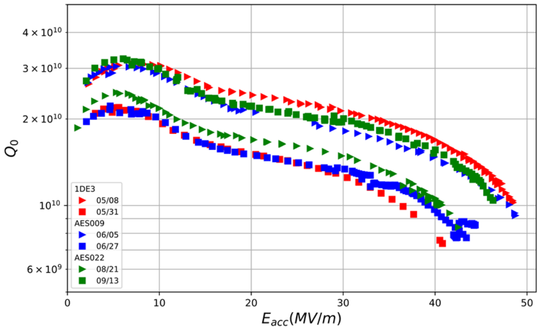

Figure 6: Bifurcation in performance of cavities post 75/120 C bake retested without disassembly in between.

This branching in performance is independent of experimental setup. This bifurcation in performance has been observed in two separate dewars and for various top plates and cables. Calibration of the cables have been investigated and measured Q_{ext2} values show no correlation with the branching in performance. In addition, the possibility of trapped magnetic flux causing this bifurcation in performance is not possible because the cavities are generally cooled in zero field using Helmholtz coils. Nonetheless, it is useful to get an estimate of the trapped field necessary to cause this bifurcation. Taking the bifurcation of 1DE3 shown in Figure 6, an estimate of the surface resistance at 40 MV/m for the upper and lower branches are 16 nΩ and 36 nΩ, respectively. This means that trapped flux must introduce an additional 20 nΩ of surface resistance to account for the difference in Q_0. An estimate of the sensitivity to trapped magnetic flux may be obtained from Figure 5. Assuming the case of higher sensitivity, the curve for AES022 post 75/120 C bake in Figure 5 gives a value of

ISBN: 978-3-95450-211-0 doi:10.18429/JACoW-SRF2019-TUP061

~1.6 nΩ/mG. Dividing 20 nΩ by this value gives 12.5 mG; that is, the cavity must trap 12.5 mG of field to cause this bifurcation, which is not possible in the test dewars as they only allow 5 mG of longitudinal field and less than 0.5 mG of transverse field. To ensure that this is true, Figure 7 shows the field at the equator of 1DE3 for both tests shown in Figure 6 as measured by a single axis flux gate. In addition to the fact that the cavity never saw 12 mG of field through transition between these two tests, Figure 7 also shows that when the cavity was *not* equipped with field compensation coils (and hence, sat in a field of 5 mG through transition), the cavity achieved the upper branch performance. From this, it is concluded that the bifurcation in cavity performance is not due to trapped magnetic flux.

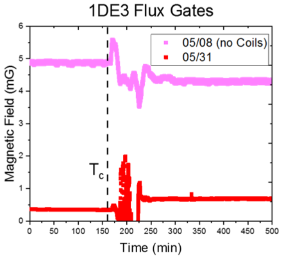

Figure 7: Longitudinal magnetic field measured at the equator of 1DE3. T_c denotes the transition into the superconducting state.

One possible cause for this bifurcation in performance is now discussed: the suppression of niobium nano-hydride formation.

Hydride Formation

It is well known that niobium has a high affinity for hydrogen [5]. At room temperature, hydrogen exists as a gas within the niobium lattice. However, upon cooling, the hydrogen diffuses toward nucleation sites where poorly superconducting niobium hydrides form. The formation of the niobium hydrides is described by the NbH phase diagram shown in Figure 8 [6]. These niobium hydrides are made superconducting only through the proximity effect, lowering the superconducting gap of the surrounding material. This diminishing in superconducting properties has been shown to affect SRF cavity performance and cause high field Q-slope [5].

Treatments have been developed to combat the formation of these hydrides [2]. It is understood that different surface impurity structures that result from these treatments produce different amounts of hydrogen capture sites such as vacancies, nitrogen interstitial, dislocations, etc. This varying amount of hydrogen capture sites will vary the concentration of free hydrogen present near the RF surface of the cavity. Figure 8 shows that for very low concentrations of hydrogen (< 8 %), small changes in H concentration with strongly affect the temperature at which niobium hydrides form. As such, it is expected that different surface preparations of cavities will produce a varying amount of niobium hydrides as they are cooled to cryogenic temperatures.

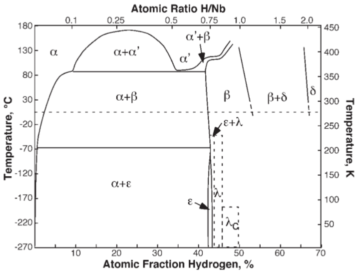

Figure 8: Nb-H phase diagram.

The formation of these niobium hydrides for various cavity surface treatments has been investigated using cryogenic atomic force microscopy (AFM). Scans of the surface were taken at various temperatures and the average number of hydrides that formed at that temperature was recorded. The results are summarized in Figure 9.

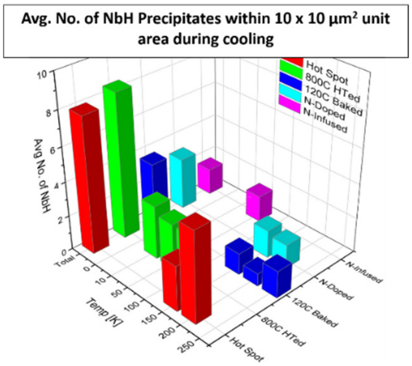

Figure 9: Histogram describing the average number of niobium hydrides formed as a function of cooling to 2 K within a 10 x 10-micron area for cavity cutouts of various surface treatments. Measurements were taken with a cryogenic AFM.

Upon cooling, it is found that the sample cut from the hot spot of a cavity forms many niobium hydrides from 200 K to 150 K. A total average of 8 niobium hydrides formed within a 10 x 10-micron unit. The 800 C high temperature bake cutout instead shows that most of the hydride formation occurs at temperatures from 50 K to 10 K; the total average number of resulting hydrides is the same as for the hot spot case. Nitrogen doped cutouts show the formation of only a few hydrides in the range of 200 K to 150 K. Nitrogen infused cutouts show even fewer hydrides formed at even lower temperatures. It is interesting to see, however, that although the total average number of hydrides formed for the 120 C baked cavity cutout is low, they form very close to room temperature. A 75/120 C bake cavity cutout has not yet been analysed but plans to do so in the immediate future exist

The size of these hydrides was also studied as a function of warming. The 120 C baked cavity cutout was warmed up to a temperature of 320 K. Two AFM scans performed on the same region of the sample taken at 300 K and 320 K are shown in Figure 10.

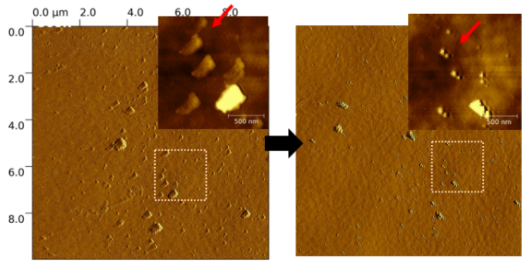

Figure 10: (Left) AFM scan of a 120 C baked cavity cutout taken at 300 K. The yellow dotted square shows the area of the blown up inset located on the upper right-hand corner. Nano-hydrides are observed. (Right) AFM scan of the same area of the 120 C baked cavity cutout taken at 320 K. The red arrow on in the inset tracks the location of a single hydride.

Niobium nano-hydrides were observed at 300 K, with sizes on the order of 300 nm. However, after warming the sample to 320 K, the size of these hydrides decreased considerably. Line profiles of a single hydride taken at 300 K and 320 K are shown in Figure 11.

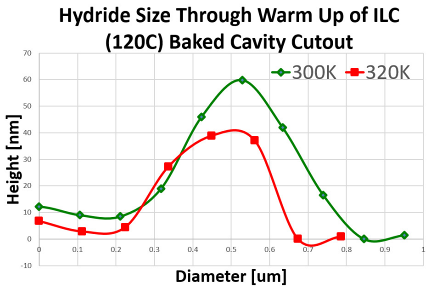

Figure 11: AFM line profiles of a single hydride taken at 300 K and at 320 K on a 120 C baked cavity cutout.

This shows a clear decrease in the size of the nano-hydrides after warming to 320 K. This idea of warming the cavity to 320 K to dissolve any possible room temperature hydrides was applied in cavity RF tests, which will now be discussed.

In-situ Cavity Heating

Four 1.3 GHz TESLA-shaped Nb cavities, RI001, RI002, AES011, and AES022 were treated with the 75/120 C bake and tested in the Fermilab VTS dewar using standard cavity testing protocol. This involved starting a "fast" cool down (~10 K/min) from a dewar temperature of ~295 K. The resulting Q_0 vs E_{acc} curves of the four cavities are summarized in Figure 12 in green. All four cavities quenched around 43 MV/m, giving the characteristic lower branch of performance discussed in Figure 6. Three of the cavities, RI001, RI002, and AES011 were taken out of the dewar and reinserted without disassembly or loss of vacuum. These cavities were subject to an in-situ heating up to 320 K in the dewar, which was then followed by a fast cool down. The Q_0 and E_{acc} of all three cavities improved and gave upper branch performance. The quench of RI001 increased from 43.6 MV/m to 46.8 MV/m and the crossing of Q_0 =2E10 moved from 15 MV/m to 23 MV/m. The quench of RI002 improved from 42.8 MV/m up to 45.6 MV/m, with the 2E10 crossing moving from 15 MV/m to 27 MV/m. Cavity AES011 experienced a similar increase in performance, with the quench moving from 42.5 MV/m up to 45 MV/m with a the 2E10 crossing increasing from 12 MV/m to 15 MV/m.

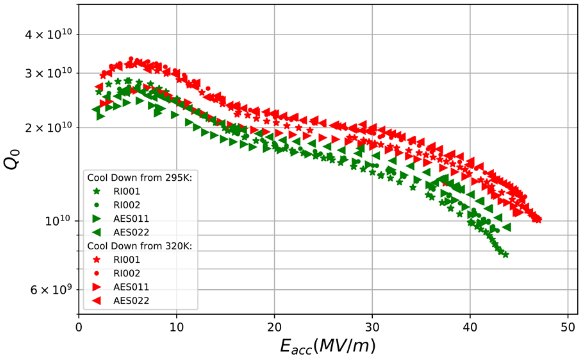

Figure 12: Results of RF tests of four cavities post 75/120 C bake after fast cooling from an initial dewar temperature of 295 K and from 320 K. Note that a full cable calibration was conducted before every test.

Cavity AES022 was also retested after an in-situ dewar heating to 320 K; however, this *cavity was never taken out of the dewar* after the first test from 295 K. The quench field of this cavity improved from 43.8 MV/m to 46.5 MV/m, with the 2E10 crossing moving from 16 MV/m to 30 MV/m. In short, the performance of these four cavities was improved from the lower branch of the bifurcation to the upper branch after in-situ 320 K heating in the dewar. Lastly, note the shapes of the Q_0 vs E_{acc} curves of the cavities post 320 K in-situ heating is slightly elongated in the horizontal direction, giving a slightly different curve shape. This gives further implication that there could be a fundamental difference in the surfaces of the cavities between these two tests, reinforcing again that this bifurcation is not due to trapped magnetic flux.

A decomposition of the surface resistance into its BCS and residual components is shown in Figure 13. There is a clear branching in both the residual as well as the BCS resistance of the cavities, which once again reinforces the fact that this bifurcation is a result of a physical difference and not due to trapped magnetic flux. The BCS resistance after the initial test from 295 K is larger than the test from 320 K by about 2 nΩ at 20 MV/m. The residual resistance between the two initial dewar temperatures agrees well at low fields but tends to diverge above 20 MV/m

The cause of this bifurcation in both BCS and residual resistances is attributed to the suppression of niobium nano-hydride formation. The above AFM studies show that the heating of 120 C baked cavity cutouts to 320 K causes room temperature niobium nano-hydrides to dissolve. This observation agrees well with the results of the cavity RF tests before and after the in-situ heating to 320 K. This elevated initial dewar temperature could be

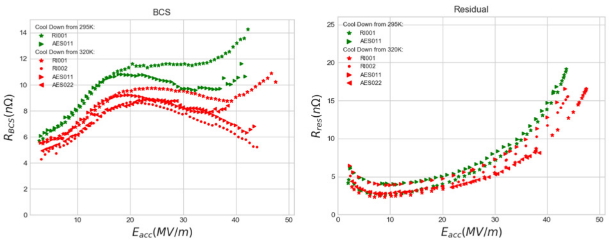

Figure 13: Decomposition of the surface resistance into (Left) BCS at T = 2 K and (Right) residual at T < 1.5 K components for the cavities discussed in Figure 12. Low temperature data of RI002 and AES022 was not taken after the initial test from 295 K.

causing the room temperature nano-hydrides to dissociate, preventing the further formation of nano-hydrides. The additional 75 C mild bake that precedes the standard 120 C bake could be introducing more vacancies that would in turn capture more hydrogen, preventing the formation of these poorly superconducting niobium nano-hydrides, thereby correcting for the proximity effect. This could be responsible for the upper branch in performance of cavities subject to the 75/120 C bake, giving quench fields that can reach and exceed 50 MV/m. However, different cavities come from various vendors and materials. As such, the starting substrate could have differing amounts of hydrogen capture sites and free hydrogen. For these cavities, it is believable that an additional 320 K bake is necessary to dissolve any pre-existing room temperature niobium nano-hydrides. This would explain why the cavities discussed in Figure 12 and Figure 13 achieved upper branch performance only after in-situ heating. The suppression of niobium nano-hydride formation is also consistent with the fact that cavities subject to the 75/120 C bake show a lower sensitivity to trapped magnetic flux when compared to the standard 120 C bake, hinting at differences in pinning strengths of vortices caused by difference in dislocations [7].

In addition to the effect of elevated initial dewar temperature, the effect of the cool down rate has also been studied to some extent. The initial motivation of this stems from the idea that cavities that dwell in the “dangerous regions” of hydride formation described in Figure 9 could form larger hydrides, thereby lowering the performance of the cavity, giving the characteristic lower branch of the bifurcation. However, after extensive study, no obvious relationship between cool down rate and cavity performance was established. This investigation is still ongoing.

The upper branch performance of SRF cavities subject to this 75/120 C bake is some of the best ever obtained for this shape and frequency. A short comparison of measurements performed by other laboratories on cavities subject to this surface treatment is now presented.

High Gradient Reproducibility by other Laboratories

Two 1.3 GHz TESLA-shaped SRF cavities, AES009 and AES022, were treated to the 75/120 C bake and tested extensively at FNAL. After testing was complete, the two cavities were sent to laboratories across the world for further investigation. The results obtained by other laboratories are summarized in Figure 14. The Q_0 vs E_{acc} curves measured by other laboratories are in good agreement with the ones measured at FNAL. Further comparison of interlaboratory performance of these cavities can be found in [8].

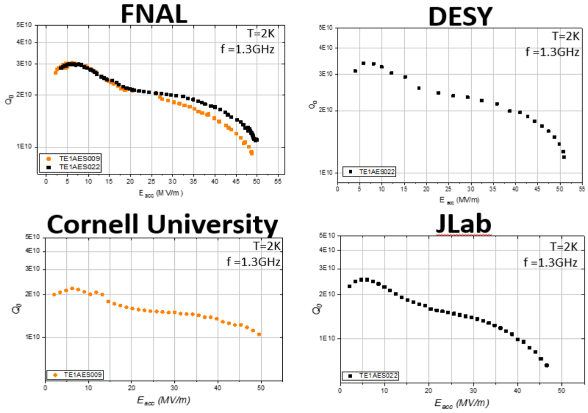

Figure 14: (Upper Left) FNAL data of TE1AES009 and TE1AES022. (Upper Right) Data taken of TE1AES022 taken at DESY. (Lower Left) Curve of TE1AES009 taken at Cornell University. (Lower Right) Curve of TE1AES022 taken at JLab. All curves taken at a temperature of 2 K.

CONCLUSIONS

Extraordinarily high quench fields of 1.3 GHz niobium TESLA-shaped SRF cavities above 50 MV/m have been achieved with the 75/120 C bake surface treatment developed at FNAL. In addition to very high gradients, the sensitivity to trapped magnetic flux is also decreased. The bifurcation in the Q_0 vs E_{acc} curves as well as the BCS and residual resistances that arises from this treatment is well explained by the suppression of niobium nano-hydride formation. This conclusion is reinforced with the decrease in nano-hydride size upon warming to 320 K, as observed in atomic force microscopy images. However, to fully understand the origins of this bifurcation, further material studies are necessary. Insights on the microscopic origins of these very high quench fields could help in the tailoring of future surface treatment to push the current limitations of SRF cavity performance even further.

REFERENCES

[1] A. Romanenko, A. Grassellino, F. Barkov, and J. P. Ozelis, “Effect of mild baking on superconducting niobium cavities investigated by sequential nanoremoval,” *Phys. Rev. Spec. Top. Accel. Beams*, vol 16, p. 012001, Jan. 2013. doi:10.1103/PhysRevSTAB.16.012001

[2] A. Grassellino *et al.*, "Unprecedented quality factors at accelerating gradients up to 45 MVm^{-1} in niobium superconducting resonators via low temperature nitrogen infusion," *Supercond. Sci. Technol.*, vol 20, p. 094004, Aug. (2017). `doi:10.1088/1361-6668/aa7afe`

[3] A. Grassellino *et al.*, "Accelerating fields up to 49 MV/m in TESLA-shape superconducting RF niobium cavities via 75C vacuum bake", arXiv preprint arXiv:1806.09824, (2018).

[4] D. Bafia *et al.,* "Low T Bake – Cool Down Studies", presented TESLA Technology Collaboration Workshop at TRIUMF (TTC'19), Vancouver, Canada, February 5-8, 2019.

[5] A. Romanenko, F. Barkov, L. D. Cooley, A. Grassellino, "Proximity Breakdown of Hydrides in Superconducting Niobium Cavities", arXive preprint arXiv: 1212.3384v3, (2013).

[6] R. E. Ricker, G. Myneni, "Evaluation of the Propensity of Niobium to Absorb Hydrogen During Fabrication of Superconducting Radio Frequency Cavities for Particle Accelerators," *J. Res. Nat. Inst. Stand. Technol.*, vol 115, p. 353-371, Sep-Oct 2010.

[7] M. Checchin *et al.*, "Frequency dependence of trapped flux sensitivity in SRF cavities," *App. Phys. Lett.*, vol 112, p. 072601, Feb. 2018. `doi:10.1063/1.5016525`

[8] M. Wenskat *et al.*, "A Cross-Lab Qualification of Modified 120 C Baked Cavities", presented at the 19th International Conference on RF Superconductivity (SRF'19), Dresden, Germany, July 2019, paper MOP026, this conference.