

# NITROGEN DOPING STUDY IN INGOT NIOBIUM CAVITIES\*

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## Abstract

Thermal diffusion of nitrogen in niobium superconducting radio frequency cavities at temperature  $\sim 800^\circ\text{C}$  has resulted in the increase in quality factor with a low-field  $Q$ -rise extending to  $B_p > 90$  mT. However, the maximum accelerating gradient of these doped cavities often deteriorates below the values achieved by standard treatments prior to doping. Here, we present the results of the measurements on ingot niobium cavities doped with nitrogen at  $800^\circ\text{C}$ . The rf measurements were carried out after the successive electropolishing to remove small amount of material from the inner surface layer. The result showed higher breakdown field with lower quality factor as material removal increases.

## INTRODUCTION

In the past few years much progress has been made in the development of the high quality factor superconducting radio frequency (SRF) cavities via the material diffusion in the thin layer of inner surface of the cavities. The motivation behind this process development is to reduce the cryogenic operating cost of current and future accelerators with reliable operation. The possibilities of higher quality factor in SRF cavities were first realized by the titanium doping [1, 2, 3] during annealing ( $\sim 1400^\circ\text{C}$ ) without any post-annealing chemistry and later by nitrogen doping at  $800^\circ\text{C}$  [4], followed by electropolishing (EP). The diffusion process not only showed the increase in quality factor at low field levels, but also an increase in quality factor with increasing accelerating gradient, contrary to the previously observed  $Q$ -slope; except some anodized cavities showed the extended  $Q$ -rise in the past [5,6]. Possible explanation for the high quality factors are trapping of hydrogen due to the diffused materials [7,8] at interstitial sites, prohibiting the formation of lossy hydrides; the  $Q$ -rise phenomenon is a result of the broadening of the peaks at the gap edges in the electronic density of states by the rf current within the rf penetration depth [9,10].

A project-driven collaboration between Jefferson Lab, Fermi Lab and Cornell University began to investigate the robustness of the nitrogen doping process to meet the specifications for cavities for the LCLS-II, requiring a  $Q_0$  value of at least  $2.7 \times 10^{10}$  at 2.0 K and a gradient of  $E_{acc} = 16.0$  MV/m in 9-cell, 1.3 GHz cavities [11]. The

work of this collaboration was focused on developing the process on fine-grain (ASTM  $> 5$ ), high purity (RRR $>300$ ) Nb cavities. In this contribution, we present the results of the rf measurements on high-purity single cell ingot niobium SRF cavities doped with nitrogen at  $800^\circ\text{C}$  followed by the successive EP steps in small increments to understand the effects of nitrogen diffusion on ingot niobium and its correlation to the cavity performances.

## CAVITY PREPERATION AND TEST RESULTS

Ingot Nb is an alternative material for the fabrication of SRF cavities having grain size of few  $\mu\text{m}^2$ . Cavities made from ingot have potential for material cost reduction and tend to have higher quality factor [12] even after the standard treatments [13]. Two single-cell 1.3 GHz cavities (labeled TD #3 and #4) with  $B_p/E_{acc} = 4.12$  mT/(MV/m) made from ingot niobium with RRR $\sim 300$  supplied by Tokyo-Denkai are used in this present study. The cavities have been processed thru several steps of typical cavity processing technique including the high temperature heat treatment ( $800$ - $1250^\circ\text{C}$ ) and buffer chemical processing. The baseline rf measurements were done after  $\sim 40$   $\mu\text{m}$  surface removal by buffer chemical polishing (BCP). The breakdown field at 2.0K was measured  $>150$  mT during the baseline rf measurements. After the baseline rf measurements, cavities were heat treated at  $800^\circ\text{C}$  for 3 hours followed by 20 minutes of exposure to nitrogen at pressure of  $\sim 25$  mTorr at this temperature. The nitrogen is then evacuated and the cavities were further annealed at  $800^\circ\text{C}$  for 30 min. The duration of the nitrogen exposure and subsequent annealing time were explored on several single and multi-cell fine grain cavities [14]. The cavities' inner surface was electropolished to remove the inner surface layer, followed by high pressure rinse with ultra-pure water. The rf test consisted of the measurements of  $Q_0(T)$  at different constant  $B_p$  from 4.3–1.6 K and  $Q_0(B_p)$  at different temperature between 2.1–1.6K. In some measurements the temperature maps of the outer cavity surface were also taken during the rf tests [15].

Cavity TD#3 was measured after the successive (10, 20, 30 and 35  $\mu\text{m}$ ) EP of the inner surface. In all cases the temperature dependence of surface resistance is measured at  $B_p \sim 10$  mT of peak rf field and material parameters such as the energy gap, electronic mean free path and residual resistance were extracted using the measured surface resistance to the calculation of surface resistance using the BCS theory [16] as shown in Table 1. No significant change in material parameters are observed regardless of

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the amount of material removal from the inner surface consistent with the recent SIMS measurements on samples which showed high concentration of the nitrogen on the surface and a very flat profile over the length of  $\sim 50 \mu\text{m}$  deep into the bulk. The breakdown field and quality factor just before the breakdown is also

summarized in Table 1. The dependence of the  $Q(B_p)$  is changing from increasing  $Q$  to decreasing as we remove more material from the surface, but the quality factor is still higher than the baseline. The temperature mapping during the rf test are also measured [15].

Table 1: Material parameters extracted for cavity TD#3 from the fits of  $Q(T)$  curves using the BCS theory after the several subsequent material removal steps by EP. Also summarized are the maximum breakdown field and quality factor.

Average material removal via EP ( $\mu\text{m}$ )	$\Delta/K_B T_c$	mfp (nm)	$R_{\text{res}}$ (n $\Omega$ )	$B_{p,\text{max}}$ (mT)	$Q_{Bp,\text{max}}$
10	$1.84 \pm 0.01$	$26 \pm 34$	$3.0 \pm 0.1$	$80 \pm 5$	$(3.4 \pm 0.5) 10^{10}$
20	$1.89 \pm 0.02$	$62 \pm 24$	$2.8 \pm 0.1$	$105 \pm 6$	$(3.9 \pm 0.4) 10^{10}$
30	$1.85 \pm 0.01$	$40 \pm 19$	$3.2 \pm 0.1$	$94 \pm 4$	$(3.5 \pm 0.3) 10^{10}$
35	$1.85 \pm 0.01$	$28 \pm 45$	$2.3 \pm 0.1$	$129 \pm 6$	$(1.9 \pm 0.2) 10^{10}$

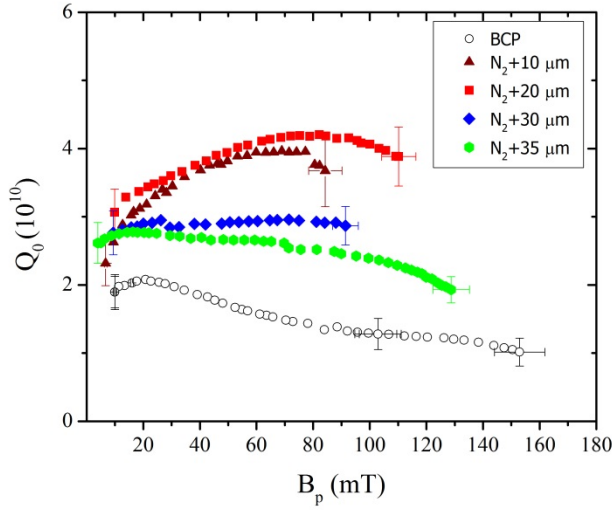


Figure 1:  $Q_0(2K, B_p)$  measured during the rf test after the cumulative EP on cavity TD#3. The test was limited by quench.

The  $Q(B_p)$  dependence was also measured up to the break down field at 2.0K and is shown in Fig. 1. The breakdown field increased after the cavity was subjected to the more surface removal, however the quality factor decreased. A High quality factor  $\sim 2 \times 10^{10}$  at breakdown field of  $\sim 129 \pm 6$  mT ( $E_{\text{acc}} \sim 32$  MV/m) is measured even after  $\sim 35 \mu\text{m}$  inner surface removal, confirming an even deeper diffusion of nitrogen into the bulk.

To further understand the  $Q(B_p)$  dependence on nitrogen doped cavities, the  $Q(T)$  dependence was measured from 4.3-1.6K for different values of  $B_p \sim 5$ -20 mT for cavity TD#4 after 10  $\mu\text{m}$  EP. The data were analyzed using the generic form of thermally activated surface resistance at  $T \ll T_c$  as  $R_s(T_s) = Ae^{-(U/KT_s)} + R_i$ , where  $U$  is quasi-particle activation energy,  $K$  is Boltzmann constant and  $T_s$  is temperature of inner cavity surface. In the limit of weak rf field  $Ae^{-(U/KT_s)}$  is a good approximation of Mattis-Bardeen expression for the BCS surface resistance [17]. The parameters  $A(B_p)$ ,  $U(B_p)$  and

$R_i(B_p)$  are extracted from the fit as described in Ref. [9], taking into account of the heat balance equation between the rf power and the power transferred into the helium bath. The results of these fits for cavity TD#4 are shown in Fig. 2. There is a small field dependence on  $R_i$  as opposed to previously observed in Ti-doped cavities [9], however the quasi-particle excitation energy remains field independent.  $A(B_p)$  shows the logarithmic field dependence consistency with the doped cavities [9, 18].

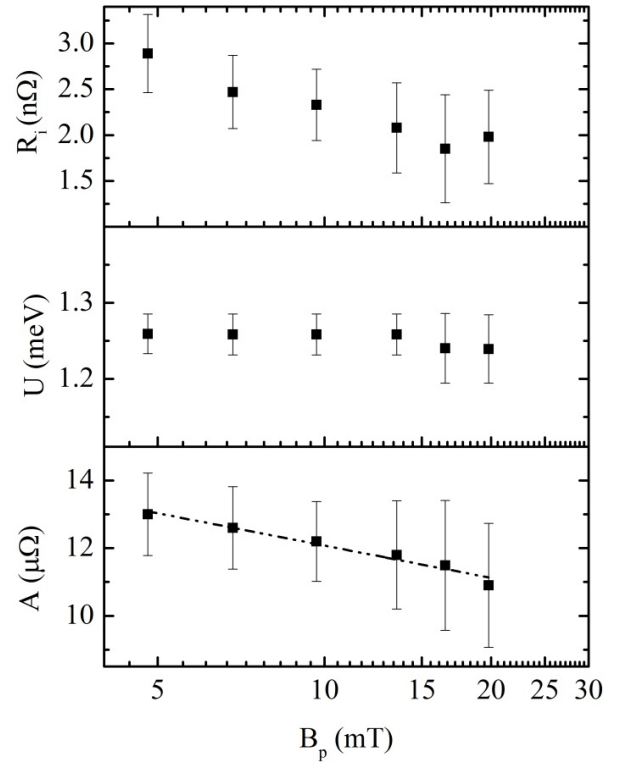


Figure 2: Dependencies of  $R_i$ ,  $U$  and  $A$  on  $B_p$  from the fitting of Eq. (1) with the method described in Ref. [9] on the amplitude of the peak magnetic field for cavity TD#4 after 10  $\mu\text{m}$  EP. The dashed line in the plot is a linear fit of  $A[\ln(B_p)]$ .

Several  $Q(T, B_p)$  measurements were taken in the temperature range of 2.1-1.6K as shown in Fig. 3. The increase in quality factor as a peak magnetic field increases has been observed for all temperatures. The curvature in  $Q(B_p)$  at low temperature may be due to the inefficient heat transfer to the helium bath due to the higher Kapitza resistance. The quality factor of  $> 4 \times 10^{10}$  is measured at 2.0K with peak magnetic field  $B_p \sim 90$  mT ( $E_{acc} \sim 22$  MV/m) compared to  $\sim 1.5 \times 10^{10}$  at the same field after the standard BCP treatment.

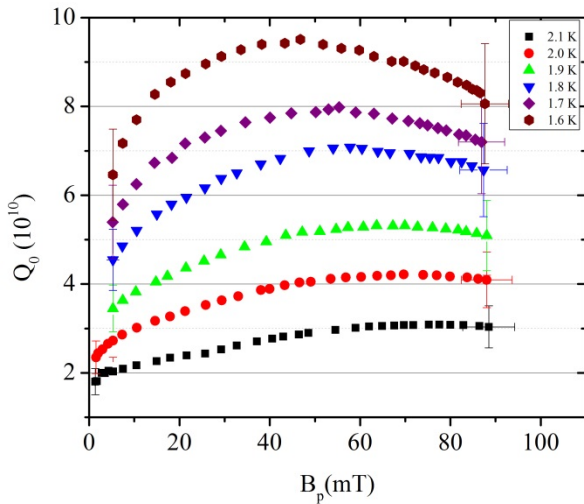


Figure 3:  $Q_0(T, B_p)$  measured during the rf test after the  $\sim 10$   $\mu\text{m}$  EP on cavity TD#4. The test was limited by quench.

## CONCLUSIONS

The results presented here show that the nitrogen diffusion followed by surface removal by EP, results in an improvement of the  $Q$ -value of ingot Nb cavities, compared to that after standard treatments, which is similar to the improvement obtained for fine grain cavities. The  $Q$ -rise phenomenon of both nitrogen doped and Ti-doped cavities is consistent with a qualitative explanation based on the broadening of the peaks at the gap edges in the electronic density of states of “dirty” Nb by the rf current [9]. A theory explaining the non-linearity of the field dependence of the surface resistance consistent with the  $Q$ -rise of doped cavities has been recently published [10]. The breakdown field has been increased after further EP the inner surface of the cavity but the quality factor is reduced by additional EP. The role of the nitrogen on degrading the breakdown field hasn’t been understood yet. Recent experiments where the amount of nitrogen introduced during annealing was reduced showed an increase of the breakdown field while maintaining the higher  $Q_0$  in ingot niobium cavity [19].

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