

INSIGHTS INTO THE ROLE OF C, N, AND O INTRODUCED BY LOW TEMPERATURE BAKING ON NIOBIUM CAVITY PERFORMANCE*

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Abstract

Previous experiments have shown that introducing N₂ gas during low temperature bakes (120 – 160 °C) of niobium resonator cavities introduces C, N, and O impurities to the first 10 – 100 nm of the surface. This new treatment results in higher quality factors, Q₀, and, in some cases, ‘Q-rise’. However, it is not entirely clear the role that each of these impurities plays in the performance enhancement of the cavities. It has been suggested that interstitial N within the first ~5 nm of the surface is solely responsible for the observed improvement in performance, but little work has been done to understand the role of C and O. Because both C and O are abundant in much higher quantities than N near the surface, it is important to understand whether they are beneficial or detrimental to cavity performance. We provide further insight into the effects of C and O on cavity performance by baking in an ambient atmosphere rich in CO₂ as opposed to N₂.

INTRODUCTION

Low-temperature nitrogen ‘infusion’ was first discovered at Fermilab and involved baking clean bulk niobium superconducting cavities at low temperature (120 – 160 °C) in an ambient partial-atmosphere of nitrogen gas [1, 2]. This process resulted in higher Q₀ and quench fields when baked at 120 °C and ‘Q-rise’ when baked at 160 °C [1, 2]. Initially, it was believed that nitrogen ‘infusing’ into the first ~5 – 10 nm of the surface resulted in the observed improved cavity performance. Secondary ion mass spectroscopy (SIMS) analysis of single-crystal niobium sample baked with one of Fermilab’s recipes revealed high concentrations of C and O in the RF penetration layer (~100 nm) [3]. This prompted further investigation into the role these two impurities played in low-temperature nitrogen ‘infusion’. To isolate these impurities from nitrogen, the low temperature bakes in this letter used an Ar/CO₂ gas mixture instead of nitrogen gas.

SURFACE TREATMENT

We used two 1.3 GHz TESLA-shaped niobium cavities, labeled LTE1-1 and LTE1-2, throughout this experiment. Both cavities received heavy vertical electropolishing (VEP) to remove any trace of previous surface treatments. Both cavities received an 800 °C de-gas bake for 5 hr immediately followed by a bake at 160 °C for 48 hr in an Ar/CO₂ gas mixture. The mixture consisted of Airgas Research Plus Ar gas (99.99999% purity) mixed with 10 ppm CO₂. A

single-crystal niobium sample witness was baked along side the cavities for the purpose of performing SIMS analysis to reveal the concentration profiles of the interstitial impurities. The concentration profiles of these impurities as a function of depth into the sample are shown in Fig. 1

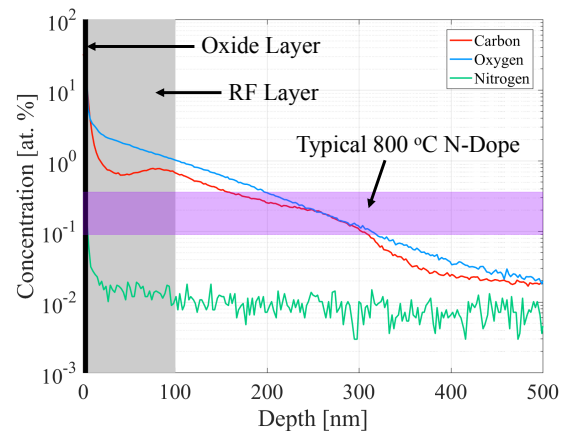


Figure 1: Concentration profiles for C, N, and O as a function of depth into the single-crystal niobium sample baked alongside LTE1-1 and LTE1-2 in the Ar/CO₂ gas mixture.

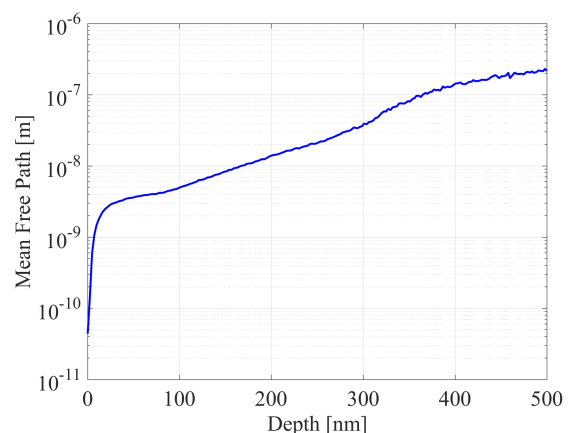


Figure 2: Mean free path calculated from SIMS data as a function of depth into the niobium single-crystal sample.

SIMS analysis of the witness sample revealed very high concentrations of C and O – especially within the first 100 nm (i.e. the RF layer). The N concentration was significantly lower at all depths and non-negligible only within the first ~10 nm. Etching any more than 10 nm should eliminate any contribution to cavity performance due to N near the surface. For reference, the purple band in Fig. 1 corresponds to the typical range of nitrogen concentration in high temperature (e.g. 800 °C) N-doped cavities [4].

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From the SIMS data, the electron mean free path was calculated using the relationship between concentration of impurity and the resulting change in resistivity

$$\Delta\rho = a \cdot c \quad (1)$$

where $a = 4.3 \times 10^{-8} \Omega \cdot \text{m}$ for C and N and $4.5 \times 10^{-8} \Omega \cdot \text{m}$ for O and c is the concentration of the impurity in at. %. The mean free path is related to the change in resistivity by:

$$\ell = \frac{\sigma}{\Delta\rho_C + \Delta\rho_N + \Delta\rho_O}. \quad (2)$$

Since the N concentration is order of magnitudes lower than that of C and O, therefore resulting in a negligible change in resistivity due to N, Eq. 2 reduces to:

$$\ell \approx \frac{\sigma}{\Delta\rho_C + \Delta\rho_O}. \quad (3)$$

The calculated mean free path is shown in Fig. 2 as a function of depth into the niobium. Within the first ~ 10 nm ℓ is sub-nanometer, but deeper into the niobium ℓ quickly levels off to $\sim 3 - 5$ nm in the range from $\sim 10 - 100$ nm.

CAVITY PREPARATION

The sequence of tests and post-bake etching is summarized in Table 1. To clarify any misunderstanding, LTE1-1 received HF rinse then was used in Test 2. After Test 2, it was rinsed with de-ionized water in a high pressure rinsing (HPR) system, received on oxypolish (OP), and was HPR one last time before Test 3. Thus, the final etching amount that LTE1-1 received before its Test 3 is ~ 32 nm. Cavity LTE1-2 was taken directly out of the furnace and HPR before Test 1. It then received another HPR, oxypolishing, and one last HPR before Test 4.

Table 1: Cavity Surface Treatments and Tests in Chronological Order

Test	Cavity	Post-Bake Etching
1	LTE1-2	–
2	LTE1-1	HF rinse (~ 5 nm)
3	LTE1-1	HF rinse (~ 5 nm) + OP (~ 27 nm)
4	LTE1-2	OP (~ 54 nm)

RF PERFORMANCE

The cavity performance for Tests 1 – 4 is shown in Fig. 3. Test 1 showed poor performance – very low Q_0 and strong Q -slope. The maximum Q_0 reached was only $\sim 5 \times 10^9$. Test 2 revealed the post-bake HF rinsing was very beneficial with greatly increased Q_0 and the appearance of ‘ Q -rise’. The HF rinse removed what was an apparently very lossy oxide layer. Test 3, after ~ 27 nm of OP, showed similar Q_0 at low field but a strong ‘ Q -slope’ reappeared. Test 4 revealed better Q_0 than Test 3, but Q -slope was still apparent.

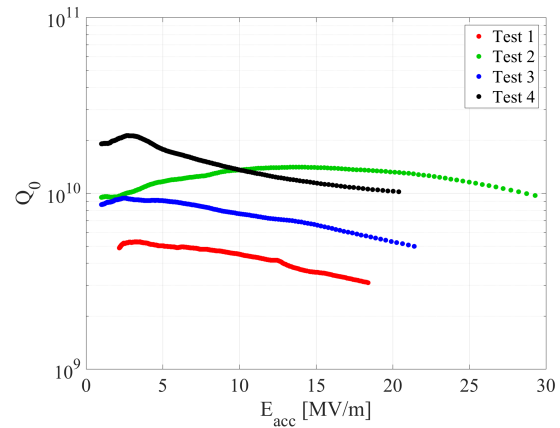


Figure 3: The $Q_0(E_{\text{acc}})$ performance of cavities LTE1-1 and LTE1-2 for Tests 1 – 4.

To further understand behavior of Q_0 the surface resistance was decomposed into its temperature-dependent, R_{BCS} , and independent, R_{res} components. R_{BCS} is well described by

$$R_{\text{BCS}} = A \left(\frac{1}{T} \right) \omega^2 \exp(-\Delta(0)/k_B T) \quad (4)$$

where the energy gap $\Delta(T) \approx \Delta(0)$ for $T < 0.5T_c$, A is a constant that depends on material properties such as the coherence length, ξ_0 , the London penetration depth, $\lambda_L(0)$, and the mean free path, ℓ , and ω is the frequency of the RF field [5]. The field dependence of these two components of the resistance are shown in Fig. 4. The cavity in Test 2 had a sharp decrease in R_{BCS} with increasing field. This behavior was previously most commonly seen in high-temperature nitrogen-doped cavities [4, 6]. The R_{res} stayed relatively flat throughout the field range compared to Test 3 and 4. Test 3 revealed the same characteristic decrease in R_{BCS} with field but with lower overall magnitude. However, the R_{res} was significantly higher at low fields and increased sharply as the field increased.

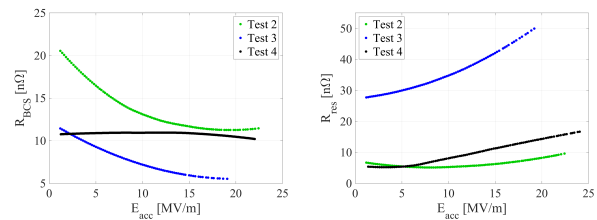


Figure 4: The field dependence of R_{BCS} and R_{res} for the second, third, and fourth test.

Normalizing R_{BCS} and R_{res} to their low field values allows a better comparison between the tests. The relative rate of reduction in R_{BCS} between Test 2 and 3 remains the same up until ~ 10 MV/m. Above this field, they begin to diverge slightly with test 3 reducing to 50% of its low field value. Again, it is seen that R_{BCS} in Test 4 remains relatively con-

stant over the entire range. Apparently, the optimal reduction in R_{BCS} occurs when ~ 32 nm of etching (HF + OP) is done. The reduction disappears after ~ 54 nm of etching.

In Test 2, the residual resistance decreases, reaches a minimum at ~ 7 MV/m, and then begins to increase. The residual resistance in Test 3 rises immediately and steadily increases over the entire range of E_{acc} . Finally, in Test 4, R_{res} dips ever so slightly before steadily increasing linearly at a very fast relative rate reaching 3 times its low field value at ~ 25 MV/m. This data suggests a correlation between the amount of etching and the strength of the increase in the residual resistance.

Although the reduction of R_{BCS} is greatest in Test 3, it also has the highest overall R_{res} . Test 2 yields a similar reduction in R_{BCS} but has a higher overall value throughout compared to Test 3. On average, however, it possesses the lowest average R_{res} resulting in the most desirable $Q_0(E_{acc})$. See also Figure 5.

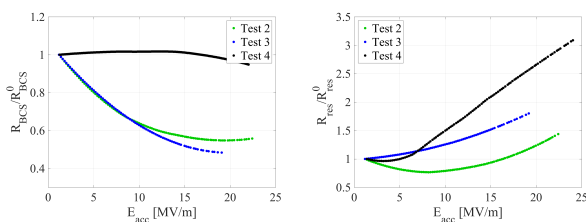


Figure 5: The field dependence of the normalized R_{BCS} and R_{res} for the second, third, and fourth test. The resistances are normalized to their low field value (~ 1 MV/m.)

DISCUSSION

The observed relative drop in R_{BCS} seen in Test 2 and 3 is very similar to that seen in high-temperature N-doped cavities [4, 6]. Gurevich provides a theory that helps to describe the reduction in R_{BCS} by suggesting that the density of states at zero applied field is sharpened by the presence of high concentrations of interstitials and that the effective energy gap increases since the density of states ‘smears’ out as the field strength increases making the effective energy gap larger [7]. The strength of the drop is explained by quasi-particle overheating – the difference in temperature between the quasiparticles and the lattice. The greater the doping level (i.e. higher concentration of interstitials), the lower the quasiparticle overheating. Maniscalco takes experimental results from high-temperature N-doping and fits the data to the Gurevich theory, demonstrating its usefulness at describing the drop in R_{BCS} [8]. Maniscalco shows that the Gurevich theory also fits well to the cavity data obtained from the Ar/CO₂ bakes in this article [9]. Further testing is required to understand why cavity Test 4 did not have a drop in the R_{BCS} as was the case in Test 2 and 3.

CONCLUSION

We showed that low temperature (160 °C) baking with an ambient Ar/CO₂ gas mixture resulted in high concentrations of C and O throughout the RF penetration layer with orders of magnitude less N. Etching several to tens of nm resulted in a very strong reduction of R_{BCS} . However, the R_{res} increased more strongly with field as the amount of etching increased. The most important conclusion we can draw is that C and O present in high concentrations in the RF layer lead to a field-dependent reduction of R_{BCS} even when the concentration of N is negligible. This demonstrates that impurities other than N can be used to achieve the same effect.

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