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EFFECT OF LOW TEMPERATURE INFUSION HEAT TREATMENTS AND "2/0" DOPING ON SUPERCONDUCTING CAVITY PERFORMANCE*

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Abstract

Under specific circumstances, low temperature infusion heat treatments of niobium cavities have resulted in the ubiquitous "Q-rise". This is an increase in quality factor with increasing field strength or equivalently a decrease in the temperature-dependent component of the surface resistance. We investigate the results of various infusion conditions with infusion bake time as a free parameter. To study the very near surface effects of infusion, we employ HF rinsing, light VEP, and oxypolishing to remove several or tens of nm at a time. We present results from RF performance tests of low temperature infusion heat treated niobium cavities, and correlate these with SIMS impurity depth profiles obtained from witness samples. We also present results of a cavity doped at 800 °C with the "2/0" recipe.

INTRODUCTION

Cornell has been studying the effects of nitrogen doping and nitrogen infusion as part of the LCLS-II HE R&D project [1]. These are recent advances in fundamental SRF technology that can dramatically improve the RF performance of niobium cavities: doping (typically performed at 800 to 900 °C in a 40 mTorr N_2 atmosphere for 2 to 60 minutes) and infusion (typically performed at 120 to 180 °C in a 40 mTorr N_2 atmosphere for 1 day or more) can give rise to a high intrinsic quality factor that increases further with increasing field strength in the "Q rise" effect [2–5].

Our studies reported here include three cavities given nitrogen infusion runs at 160 °C for 24, 48, and 192 hours, respectively, as well as some light surface removal by HF rinsing, light VEP (vertical electropolishing), and OP (oxypolishing). Two of these cavities, SC-06 and RDTTD-4, featured niobium-titanium "DESY seal" flanges, while the third, LTE1-1, featured niobium indium seal flanges. Further results reported include "2/0" doping (800 °C degas step followed by 2 minutes at 800 °C in 40 mTorr of N_2 with no vacuum anneal, followed later by 5 μm VEP) on three cavities, RDTTD-4, SC-06, and EZ-002.

Cavities that received RF testing had Q_0 vs. E_{acc} measurements taken at temperatures ranging from 1.6 to 4.2 K; using this data we performed field-dependent BCS fitting to yield $R_0(E_{acc})$ and $R_{BCS}(E_{acc}, T)$ curves. Here we present data from 2.0 K. These cavities were 1.3 GHz TESLA single-cells with B_{pk}/E_{acc} = 4.28 mT/(MV/m).

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Figure 1: Close-up photo of RDTTD-4 NbTi flange, showing black speckling after initial infusion treatment.

NITROGEN INFUSION RESULTS

The cavities RDTTD-4, SC-06, and LTE1-1 all received variations of the nitrogen infusion treatment, beginning with a VEP reset, followed by a 3-hour degas step in vacuum at 800 or 900 °C, a temperature ramp down to 160 °C and an equilibration step at 160 °C for 3 hours, followed by a 160 °C infusion run in 40 mTorr of N_2 gas. Table 1 summarizes these preparations.

Cavity SC-06 began with a 192-hour infusion run. Initial RF test results revealed an R_{BCS} that was quite high, near 20 to 25 nΩ, but with a mild field-dependent decrease at low fields reminiscent of Q-rise behavior. The cavity also showed high residual resistance in the 10 to 15 nΩ range. The cavity quenched at 26 MV/m. Visual inspection of the cavity showed discolored spots on the NbTi flanges, possibly indicating chemical activity of the NbTi flanges during the baking process. Figure 1 shows this speckling. The discoloration was mostly removed by scrubbing the flanges azimuthally with a white 3M pad.

SIMS (secondary ion mass spectrometry) analysis of a single crystal sample baked alongside SC-06 showed that the treatment introduced interstitial titanium impurities into the RF surface; Figure 2 shows these results. Under the suspicion that the speckling and high resistance values were related to this titanium contamination on the cavity surface, we performed an HF rinse on the cavity, effectively removing the oxide layer, converting the first ~ 2.5 nm of Nb into the new oxide layer, and revealing a new metal RF surface at a depth of ~ 2.5 nm into the original metal surface. This greatly improved R_{BCS}, causing a more dramatic Q-rise-

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Table 1: Summary of Nitrogen Infusion Cavity Preparations

Cavity	VEP (μm)	Degas temp. (°C)	Degas time (hr)	Infusion time (hr)	Test #2 prep.	Test #3 prep.
SC-06	50	900	3	192	HF rinse	2nd HF rinse
RDTTD-4	80	800	3	48	2x HF rinse	100 nm VEP
LTE1-1	10	800	3	24	54 nm OP	—

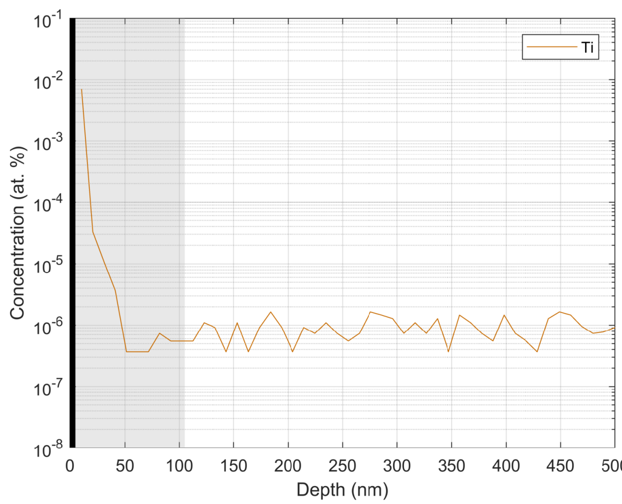

Figure 2: SIMS measurement of atomic concentration of titanium impurities on a witness sample baked alongside SC-06 during the nitrogen infusion run. Black bar indicates surface oxide; gray bar approximates RF penetration layer.

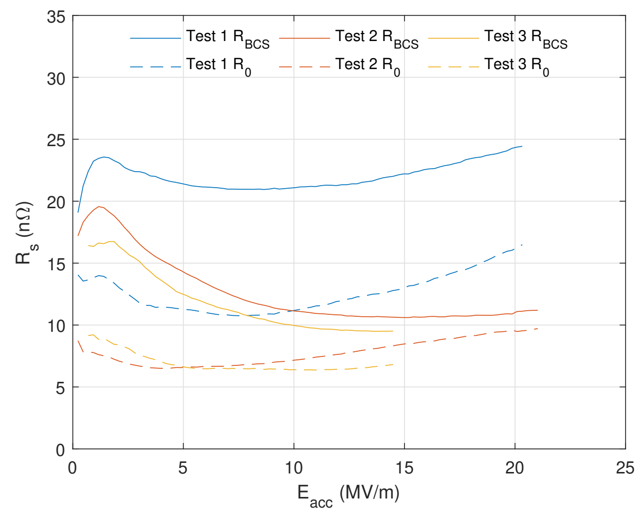

Figure 3: RF test results of the three infusion tests of cavity SC-06. R_{BCS} taken at 2 K. Peak field shown here does not indicate quench field.

related field-dependent decrease and lowering the minimum R_{BCS} to 10 nΩ; this also improved R_0, lowering it to the range of 7 to 10 nΩ. The quench field was 24 MV/m. We performed a second HF rinse on SC-06 as an attempt to further improve performance. After this final treatment, R_{BCS} was reduced by a further 2 nΩ, though R_0 stayed in the same range. The cavity quenched near 32 MV/m. Figure 3 shows the combined surface resistance results for the three infusion tests of cavity SC-06 at a temperature of 2 K.

Figure 4 shows SIMS results of carbon, nitrogen, and oxygen impurities for a single-crystal Nb witness sample baked with SC-06 during its initial infusion run. Nitrogen is only present above background levels in the first 10 to 20 nm of the surface; oxygen and carbon are present in much higher levels.

Cavity RDTTD-4 received a 48-hour nitrogen infusion run. Similar to cavity SC-06, RDTTD-4 showed black speckling on the NbTi flanges; this discoloration was removed by azimuthal scrubbing with a 3M pad. Like SC-06, this cavity showed high R_{BCS} of around 20 nΩ at 2 K with only a very mild field-dependent decrease in the medium-field range. R_0 was quite high as well, in the range of 15 to 20 nΩ. Our hypothesis is that these high resistances were again related to titanium contamination of the RF surface. The cavity quenched at 20 MV/m. We performed two HF rinses on this cavity in an attempt to improve RF performance as with cavity SC-06. HF rinsing lowered R_{BCS} to a minimum near

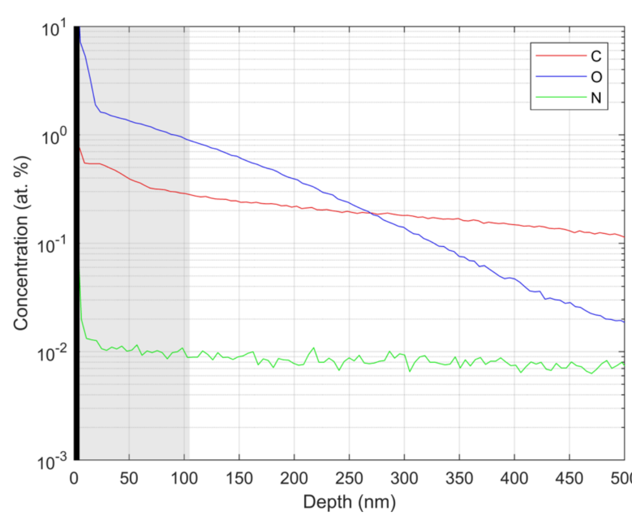

Figure 4: SIMS measurement of atomic concentration of impurities in a witness sample baked with cavity SC-06 in its infusion run. Black bar indicates surface oxide; gray bar approximates RF penetration layer.

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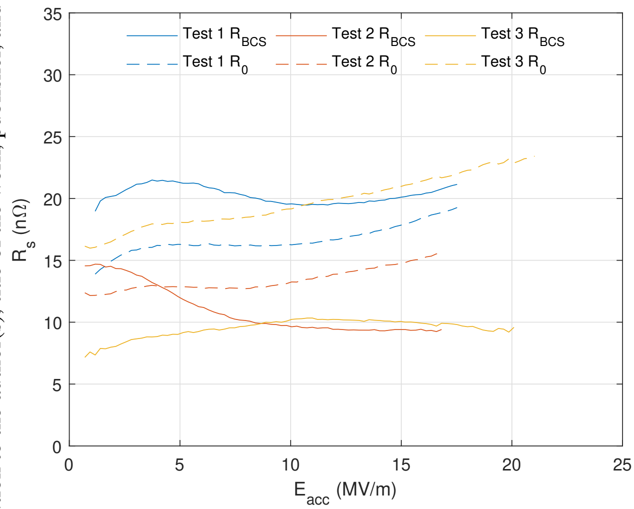

Figure 5: RF test results of the three infusion tests of cavity RDTTD-4. R_{BCS} taken at 2 K. Peak field shown here does not indicate quench field.

10 nΩ and revealed a Q-rise-related field-dependent decrease. R_0 was lowered as well to the range of 10 to 15 nΩ. The quench field was again near 20 MV/m. We performed more light surface removal, this time polishing away 100 nm by cold VEP. After this procedure, R_{BCS} was greatly improved at low fields but the Q-rise-related field-dependent decrease in R_{BCS} was removed. R_0 was higher during this RF test, though we believe that this was caused by high trapped flux due to an error in the cooldown procedure. The cavity's quench field after the cold VEP was 31 MV/m. Figure 5 shows the combined surface resistance results for these three tests of RDTTD-4.

Figure 6 shows SIMS results for a single-crystal Nb sample treated alongside RDTTD-4 during its initial infusion run. Like the results for SC-06, these show a high concentration of nitrogen in a spike in the first 10 nm or so but little nitrogen deeper into the sample. Carbon and Oxygen levels are much higher out to a depth of 200 to 300 nm.

Cavity LTE1-1 was given a nitrogen infusion run of 24 hours. Unlike SC-06 and RDTTD-4, this cavity showed strong Q-rise and low surface resistances. This cavity had Nb indium-seal flanges, serving as evidence for our titanium contamination hypothesis. R_{BCS} exhibited a Q-rise-related field-dependent decrease with a minimum near 7 nΩ, while R_0 was in the range of 5 to 8 nΩ. The cavity had a quench field of 25 MV/m, with light field emission beginning at 20 MV/m.

Continuing with our surface removal study, we removed 54 nm of the RF surface by oxypolishing. After this light removal, R_0 was reduced to a minimum of 2 nΩ, but the field-dependent decrease in R_{BCS} was removed, with the BCS resistance staying in the same overall range. The quench field was increased to 29 MV/m. Figure 7 shows the surface resistance results for these two tests of LTE1-1.

Figure 8 shows SIMS results for the witness sample treated alongside LTE1-1 during its infusion run. Like the others,

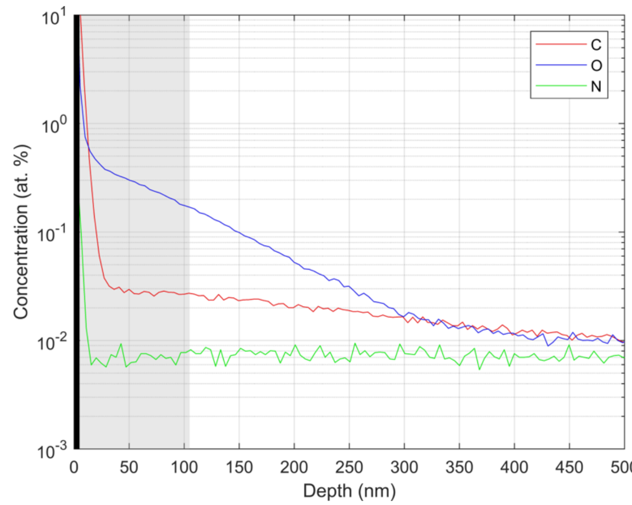

Figure 6: SIMS measurement of atomic concentration of impurities in a witness sample baked with cavity RDTTD-4 in its infusion run. Black bar indicates surface oxide; gray bar approximates RF penetration layer.

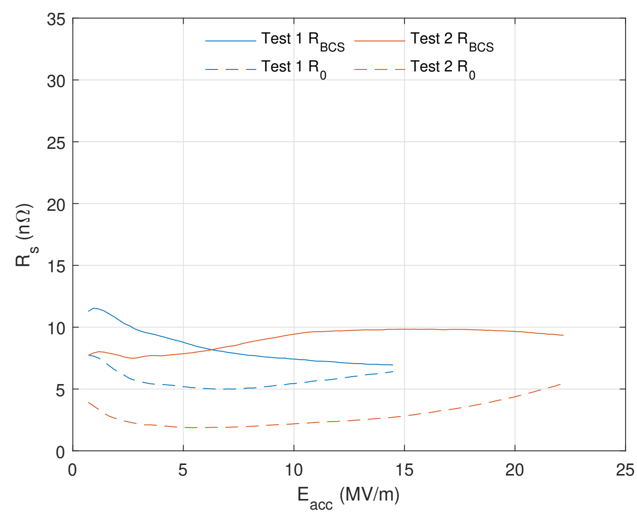

Figure 7: RF test results of the two infusion tests of cavity LTE1-1. R_{BCS} taken at 2 K. Peak field shown here does not indicate quench field.

these results show a spike of nitrogen near the surface with concentrations at background level elsewhere. Carbon and oxygen are present in significantly higher concentrations for the first 200 nm or so.

Comparing the best Q-rise-related field-dependent decrease in R_{BCS} for the three different infusion step lengths, we find a marginal difference between the 192-hour and 48-hour treatments. The 24-hour treatment showed lower overall R_{BCS}, but this may be related to the titanium contamination or to increasing the electron mean free path closer to the BCS minimum [6]. Table 2 summarizes the R_{BCS} performance of these cavity tests.

In all, these nitrogen infusion results suggest several conclusions. First, these treatments may be extremely sensitive to surface contamination. One of the main benefits of ni-

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Table 2: Summary of Nitrogen Infusion Cavity RF Performance

Cavity	Test number (see Table 1)	Relative field-dependent decrease in R_{BCS}	B_{pk} at minimum in R_{BCS} (mT)
SC-06	1	9%	35
SC-06	2	45%	60
SC-06	3	45%	60
RDTTD-4	1	11%	50
RDTTD-4	2	11%	60
RDTTD-4	3	N/A[1]	N/A[1]
LTE1-1	1	42%	60
LTE1-1	2	N/A[1]	N/A[1]

[1] No Q-rise or field-dependent decrease in R_{BCS} observed for this test.

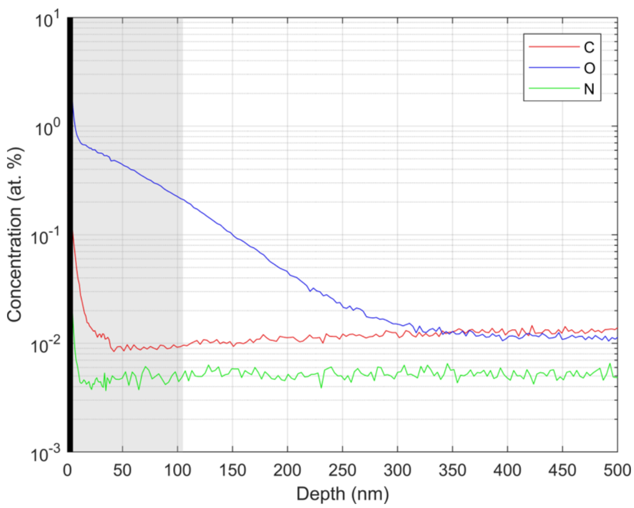

Figure 8: SIMS measurement of atomic concentration of impurities in a witness sample baked with cavity LTE1-1 in its infusion run. Black bar indicates surface oxide; gray bar approximates RF penetration layer.

trogen infusion compared to high-temperature doping is the lack of post-bake chemistry; the need to cure surface contamination negates that benefit. Surface contamination issues will need to be addressed for nitrogen infusion to be a successful cavity preparation technique.

Second, the surface removal studies point towards nitrogen as the culprit responsible for Q-rise in doped and infused cavities, though this is far from conclusive. Although the tests with surface removal of more than 20 nm had no field-dependent decrease in R_{BCS}, this characteristic change in RF performance might also be attributed to the removal of the surface concentration spikes in C and O, not only N. Interesting as well is the quite similar R_{BCS} for the 192-hour and 48-hour infusion runs, which feature *quantitatively* different impurity concentrations in the bulk but *qualitatively* similar impurity spikes near the RF surface, especially similar in the case of nitrogen. Further, a very low amount of titanium contamination may be enough to negate the Q-rise effect. At the very least, these results indicate that Q-rise and the field-dependent decrease in R_{BCS} are strongly dependent on the properties of the very near surface. More studies will be necessary to elucidate the role of these these properties in Q-rise.

Further analysis of these results is presented elsewhere at this conference [7].

2/0 DOPING RESULTS

After chemical reset by VEP, we performed “2/0” doping on cavities RDTTD-4, SC-06, and EZ-002. For each of the three cavities, the 5 µm VEP step after the bake behaved unusually. After electropolishing, the electrolyte solution was discolored from its normal clear state to a dark gray. Moreover, the cavities were marked with an unusually high amount of pits and scratches. We speculate that these were caused by interactions between the acid and a rich nitride layer present on the cavity surface after the bake, a layer which would normally be allowed to diffuse more into the cavity bulk during the annealing step in *e.g.* a 2/6 dope; this reaction may have produced a large number of hydrogen bubbles, marking the surface, and also may have left a byproduct in solution in the acid, causing the discoloration.

We tested SC-06 and EZ-002 under RF after the 2/0 doping treatment. Test results for these cavities were mixed. Both exhibited extremely similar R_{BCS}, with a Q-rise-related field-dependent decrease down to a minimum of 6 nΩ. The residual resistance R_0 was in the range of 3 to 5 nΩ for cavity SC-06; this cavity showed Q-rise up to 10 to 15 MV/m. For EZ-002, on the other hand, R_0 was much higher, in the range of 15 to 20 nΩ. This may have been caused by accidental flux trapping during the cooldown before RF testing. SC-06 quenched at 21 MV/m and EZ-002 quenched at 22 MV/m with field emission setting in near 16 MV/m. Figure 9 shows the surface resistance results for the RF tests of these two 2/0-doped cavities.

These results indicate that 2/0 doping can give RF performance similar to that of 2/6 doping, in terms of surface resistance and quench field (if indeed the high R_0 of EZ-002 was due to flux trapping) [8].

CONCLUSIONS

We tested several cavities prepared with nitrogen infusion for varying infusion step times, finding potential sensitivity to surface contamination requiring light surface removal;

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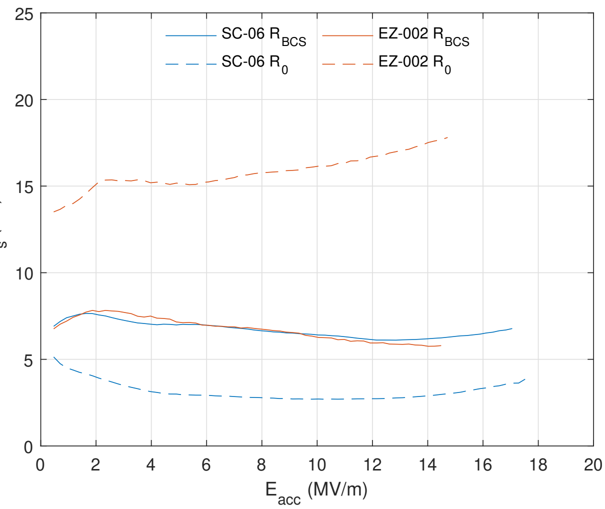

Figure 9: RF test results of the two cavities treated with 2/0 doping. R_{BCS} taken at 2 K. Peak field shown here does not indicate quench field.

after light chemical treatment of the cavities possibly contaminated with titanium, these cavities exhibited similar Q-rise-related field-dependent decreases in R_{BCS} and moderate-to-high R_0. After chemically removing 50 to 100 nm of the surface these cavities no longer exhibited Q-rise or field-dependent decreases in R_{BCS}, suggesting that the Q-rise of infused cavities is strongly dependent on the properties of the near-surface material.

We also tested several cavities with the 2/0 doping treatment. These cavities had quite similar behavior to 2/6-doped cavities in terms of their field-dependent R_{BCS} and quench fields. The 5 µm post-bake VEP for these cavities behaved somewhat unusually, with the acid turning dark gray over the course of the VEP and the cavities developing a high number of pits and scratches; these may be related to a heavy reaction between the acid and the rich nitride layer present on the surface after the doping with no anneal.

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