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NEW INSIGHT ON NITROGEN INFUSION REVEALED BY SUCCESSIVE NANOMETRIC MATERIAL REMOVAL*

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Abstract

In this study we present new insight on low temperature nitrogen infusion on bulk niobium superconducting radio-frequency (SRF) cavities. Nitrogen infusion is a thermal treatment recently discovered at Fermilab that allows to reach high accelerating gradients, of the order of 45 MV/m, with high Q-factors, of the order of $2 \cdot 10^{10}$. Detailed depth dependent RF studies (by means of subsequent HF rinses) and comparison with SIMS results pinpointed interstitial nitrogen as the responsible for the improved performance and uncovered the extension of its profile inside the material.

INTRODUCTION

Future SRF-based accelerators call for higher gradient operation with affordable values of cryogenic dissipation in order to decrease the capital cost of their construction and to fully exploit the potential of SRF technology to achieve high duty cycles. State-of-the-art high-gradient SRF cavities show high field quality factors of $9 \cdot 10^9$ and are limited by localized quench to accelerating fields of the order of 40 MV/m [1]. The novel surface process discovered at Fermilab, so-called Nitrogen infusion (N-infusion) [2], allows to reach high accelerating gradients, of the order of 45 MV/m, with high Q-factors, of the order of $2 \cdot 10^{10}$—basically doubling the cryogenic efficiency at high gradients with respect to the state-of-the-art treatments.

The cost of high-energy linear accelerators (LINACs) is driven by the number of cryomodules required to reach the designed beam energy, which is set by the operational gradient of cavities. The operational gradient is then a major cost driver in the construction of a large machine. By increasing the gradient by a factor of 2, the LINAC length decreases accordingly by a factor of 2, resulting in an estimated saving of about $ 3.7 million per cryomodule (based on Fermilab experience of LCLS-II cryomodule production).

Nitrogen infusion is therefore extremely important in the framework of ILC cost reduction. The smart engineering of the concentration profile at the near-surface can in principle improve the accelerating gradient in SRF cavities and allow for higher duty factors as well. In this paper, we present a systematic study aimed to unveil the origin of the improved performance of N-infused cavities through depth-dependent RF measurements enabled by successive material nanoremovals by means of hydrofluoric acid (HF) rinses. With this study, we are able to corroborate the thesis that interstitial nitrogen is the sole responsible for the improved performance in N-infused cavities [2] and rule out other interstitial elements (oxygen and carbon), as instead suggested in other studies [3].

EXPERIMENTAL PROCEDURE

Two superconducting radio-frequency (SRF) single-cell TESLA-type [4] bulk niobium cavities (te1aes010 and

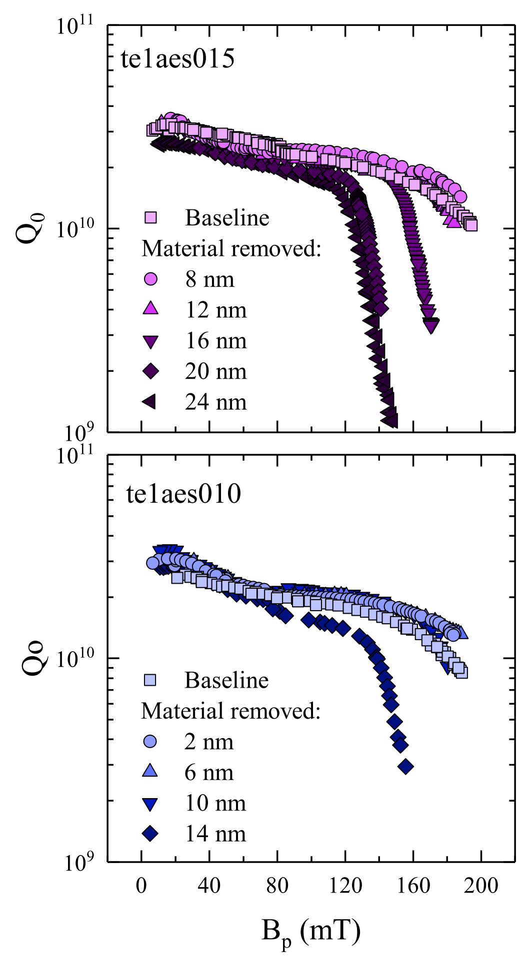

Figure 1: Q-factor versus peak magnetic field of the two cavities studied (te1aes010 and te1aes015) after successive material removals via HF rinsing.

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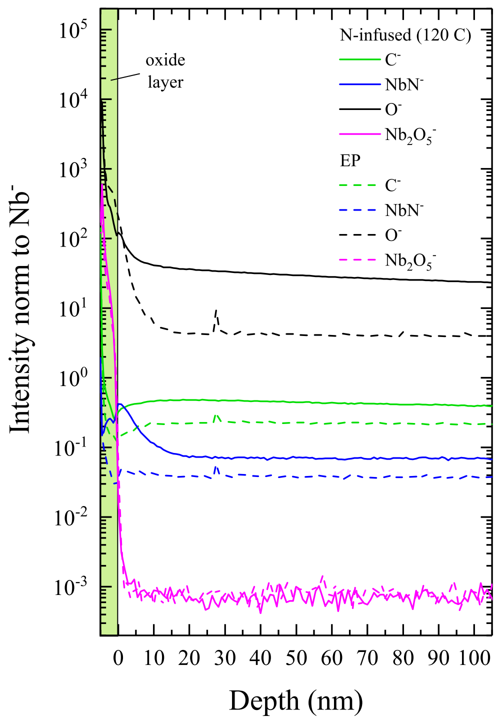

Figure 2: Comparison between TOF-SIMS data for the 120 °C N-infused and EP cavity cut-outs.

te1aes015) where N-infused at 120 °C. The cavities received bulk electropolishing (EP) and were then baked at 800 °C for 3 hours to degas hydrogen. After this step the temperature in the furnace was lowered at 120 °C and kept constant for 48 hours while nitrogen, with partial pressure of 25 mTorr, was inlet in the furnace. The cavities so prepared where high water pressure rinsed and RF-tested at the vertical test facility of Fermilab. The performance of the two cavities are reported in Fig. 1 and labeled with baseline. Both the cavities reached fields of the order of 45 MV/m (∼190 mT).

Subsequent tests of the cavities were performed after removing material from the inner surface by means of HF rinsing. Whenever HF is in contact with the cavity surface, it dissolves the native oxide (∼5 nm), and upon subsequent water rinse, a new oxide layer is grown, consuming about 2 nm of niobium [5]. After every HF rinsing step, the two cavities were RF tested and the Q-factor versus accelerating field data recorded. As shown in Fig. 1, the performance changes gradually any time material is removed from the cavity inner surface, eventually reverting back to the typical HFQS behavior found for EP cavities (∼100 mT [1]) after 20 nm of material are removed. Such a finding proves that the N-infusion treatment affects the first tens of nanometers into the RF surface and not the material bulk.

In order to pinpoint the origin of such effect in terms of material properties, a third cavity was prepared in the same way and RF-tested in order to guarantee that the performance where the same. Interesting spots of the cavity, identified by means of temperature mapping [6] during the RF test, were cut out and characterized with a time-of-flight secondary ion mass spectroscopy (TOF-SIMS) system. Depth profiles of oxygen, carbon, and nitrogen bearing fragments were acquired and compared to the depth profiles for the same fragments acquired from an EP cavity cut-out, as reported in Fig. 2. The detailed study of such a cavity is discussed in Ref. [7].

DISCUSSION

As highlighted by the RF data in Fig. 1, only impurity variations in the first tens of nanometers can be responsible for the improved performance: carbon signal is basically constant within 20 nm (as shown in Fig. 2) from the surface and can then be ruled out. Oxygen can be ruled out as well since an oxygen-rich layer is observed in both N-infused and EP cases, meaning that such an element is not involved in the improovement of the performance.

On the contrary, the nitrogen signal seems to be in perfect agreement with the RF data. In Fig. 3, the HFQS onset—the magnetic field value for which the Q-factor versus gradient curve starts to bend as a function of material removal—is plotted in comparison with the TOF-SIMS data for the nitrogen and oxide layer. The HFQS onset data shows that for the first ∼10 nm of removed material, the cavity is still limited by quench at about 190 mT, but no HFQS reappearance is recorded. By removing more material, the HFQS onset starts to become visible, until around 20 nm of material removal,

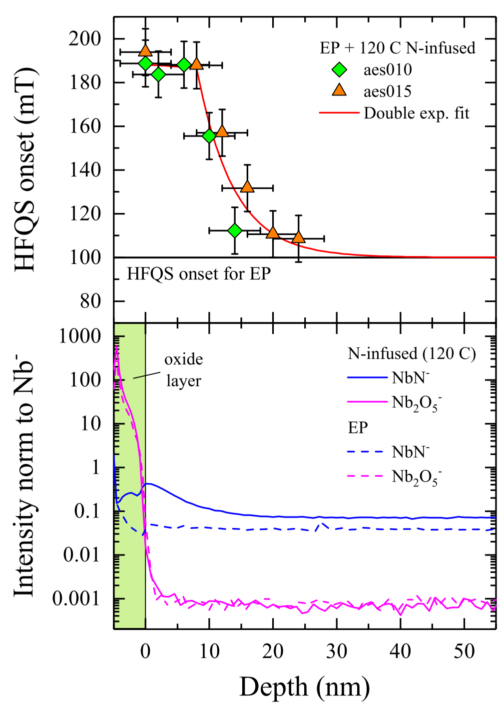

Figure 3: The HFQS onset data as a function of material removed compared to the nitrogen concentration profile measured with TOF-SIMS on cavity cut-outs. The RF data reported from zero up to ∼10 nm correspond to the maximum field reached by the cavity (quench field) since HFQS was not observed.

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it starts approaching the typical value of ~100 mT for EP cavities. The nitrogen concentration profile is clearly related to the HFQS reappearance data, which as well changes in the first 20 nm and becomes constant at larger depths.

Such a result unequivocally shows that the origin of the N-infusion performance is due to this nitrogen-enriched layer just below the oxide, which is not found in EP cavities. Other studies suggests that the origin of the improved performance observed in N-infused cavities may arise from interstitial carbon and oxygen [3]. However, our RF and SIMS data shows that in N-infused cavities carbon and oxygen do not play any role in removing the HFQS nor enhancing the accelerating gradient.

Such a finding is also corroborated by the fact that whenever the thermal treatment here discussed is performed without nitrogen the HFQS is eliminated, meaning that a near surface nitrogen profile is acting against the HFQS, as also pointed out in Ref. [2].

CONCLUSION

In this work, we have demonstrated that the HFQS in niobium cavities is removed by the interstitial nitrogen profile generated by the low temperature diffusion treatment and that nitrogen is the only interstitial impurity playing a role in enhancing the cavity performance in N-infused cavities. Such a finding is corroborated by the RF data on HFQS onset reappearance together with the SIMS data on a cavity cutout. Indeed, the HFQS onset is observed at ~100 mT only after the nitrogen profile in the material is totally removed.

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