UPDATE ON NITROGEN-DOPING: QUENCH STUDIES AND SAMPLE ANALYSIS*

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Abstract

author(s), title of the work, publisher, and DOI. Recently, nitrogen-doping of niobium has emerged as a promising preparation method for SRF cavities to reach higher intrinsic quality factors than can be reached with typical cavity preparation. Nitrogen-doped cavities prepared at Cornell have shown quality factors higher than 4×10^{10} at Cornell have shown quality factors higher than 4×10^{10} at 2.0 K and 16 MV/m. While Q results have been very exciting, a reduced quench field currently limits nitrogen-doped $\frac{1}{3}$ cavities with quench typically occurring between 15 and ain 25 MV/m. Here we report on recent results from Cornell on single-cell and 9-cell cavities, focusing on new preparations and maximum and critical fields. First we discuss results from over-doping niobium with nitrgoen, baking nitrogendoped cavities at 120°C, and doping with Argon. For a work subset of these cavities we show results from quench studies : that have been completed using temperature mapping. Fianally, we present the first measurements of the higher critical

in the field, H_{c2} , for nitrogen-doped niobium samples. **INTRODUCTION** Nitrogen-doping has been shown to increase the quality factor, Q_0 , of niobium SRF cavities to be Nitrogen-doping has been shown to increase the intrinsic quality factor, Q_0 , of niobium SRF cavities to levels previ- $\widehat{\mathfrak{D}}$ ously unreachable. Doping consists of giving cavities a heat $\stackrel{\text{O}}{\sim}$ treatment at high temperatures in a gaseous atmosphere [1]. 0 An ongoing effort has been undertaken at Cornell, Jefferson Lab, and Fermilab to understand the benefits of nitrogen-doping and to create an optimal recipe for cavity preparation. Q: Unfortunately, many cavities prepared with nitrogen-doping have shown a lower quench field than cavities prepared by other means. Cornell has recently been focusing on studying the cause of this lower quench field by systematically preparing cavities by different methods. Furthermore, we have employed the use of both single and 9-cell temperature mapping E systems for quench detection along with OSTs. We have also measured the higher critical field, H_{c2} of nitrogen-doped niobium samples using Physical Property Measurement System (PPMS). Additionally, we have continued studying different preparation techniques such as doping with argon instead of g nitrogen and subjecting a nitrogen-doped cavity to a 48 hours $\stackrel{\circ}{\simeq}$ 120°C bake. These latest measurements push us closer to $\frac{2}{3}$ understanding the mechanisms behind higher Q_0 's, lower $\frac{1}{2}$ quench fields, and other unique properties of nitrogen-doped $\frac{1}{2}$ cavities.

Content from this **WEPTY073** 3450



Figure 1: 2.0 K Q_0 vs E_{acc} performance for the over-doped cavity. Quench field increases with more material removal.

SINGLE-CELL STUDIES

Over-Doping

A single-cell 1.3 GHz ILC shaped cavity was given an "overdoping" of nitrogen. This consisted of a bulk electropolish (EP), followed by a heat treatment in vacuum at 900° for 3 hours, followed by 900°C heat treatment in 60 mTorr of nitrogen gas for 20 minutes, followed by an additional 900°C heat treatment in vacuum for 30 minutes. Finally, the cavity was given a series of 3 additional EPs in steps of 6 μ m with tests in between. Typically, cavities have been treated at 800°C [1,2]. By increasing the temperature by 100° C, we saw an increase in nitrogen uptake of ~ $1.75 \times$ that for previous cavities. The 2.0 K Q_0 vs E_{acc} results are shown in Fig. 1. We can see that initially (after 6 μ m EP), the cavity quenched at 8.3 MV/m with a maximum Q_0 of 2.7×10^{10} . After an additional 6 μ m EP (total of 12 μ m) the quench field increases to 11.5 MV/m with a maximum Q_0 of 3.7×10^{10} . Finally, after another additional 6 μ m (total of 18 μ m), the quench field increases to 15 MV/m with a maximum Q_0 of 4.6×10^{10} .

The increase in Q_0 with additional material removal is fairly well understood. Nitrogen-doping has been shown to optimize the BCS material properties and thus minimize the BCS resistance of SRF cavities [3]. By removing more material, we are reaching a more optimial place on the BCS curve. Nitrogen-doping has also been shown to form a lossy nitride layer on the surface. It is possible that this layer (which is now significantly thicker due to the higher temperature doping) causes the quench field to be even lower in this cavity than in previous cavities. This is consistent with the quench field increasing as more material is removed. A discussion of the quench location will be presented in a later section.

> 7: Accelerator Technology **T07 - Superconducting RF**

Work supported by the US DOE and the LCLS-II High Q0 Program and NSF Grants NSF-PHY-1305500 and NSF-PHY-1416318. This work made use of the Cornell Center for Materials Research Shared Facilities which are supported through the NSF MRSEC program (DMR-1120296). dg433@cornell.edu

6th International Particle Accelerator Conference ISBN: 978-3-95450-168-7



Figure 2: 2.0 K Q_0 vs E_{acc} performance for a nitrogendoped cavity before and after 48 hour 120°C bake.

N-Doping + $120^{\circ}C$ Bake

Standard niobium cavities treated with a 48 hour 120°C bake have been shown to have strong benefits in terms of BCS material properties and high field O slope reduction. In order to test if a 120°C bake would have a similar benefit on nitrogen-doped cavities, a single-cell cavity was prepared with nitrogen-doping: a bulk EP, heat treatment in 800°C in vacuum for 3 hours, heat treatment in 60 mTorr of nitrogen gas for 20 minutes, in vacuum again for 30 minutes, and finally a 24 μ m EP. Following this preparation, the cavity was tested and then given a 48 hour 120°C bake and tested again. The 2.0 K Q_0 vs E_{acc} results are shown in Fig. 2. We can see that the Q_0 remained unchanged from the 120°C bake however the quench field dramatically decreased from 35 MV/m to 17 MV/m. Clearly, the baking did not have a beneficial effect on the cavity. The lack of change in Q_0 is unsurprising since nitrogen-doping has been shown to "dirty" niobium in such a way to reach optimal BCS material properties. The change in the quench field however is quite surprising. More studies will need to be conducted in order to understand how the quench field was affected by the bake.

Argon Doping

The benefits of nitrogen-doping have been thoroughly discussed in the literature. It had initially been suggested by Grassellino et. al. that doping with argon may also give a similar benefit without forming a lossy nitride layer on the surface [1]. In order to further explore this, a single-cell ILC shaped cavity was prepared with argon-doping: a bulk EP, heat treatment in 800°C in vacuum for 3 hours, 800°C heat treatment in 60 mTorr of argon gas for 20 minutes, and in vacuum again for 30 minutes. The cavity was then tested followed by a bulk BCP and tested again to compare as a baseline test. The Q_0 vs E_{acc} performance at 2.0 K for these two tests is shown in Fig. 3. We can see that argon doping had virtually no effect on the quench field or the Q_0 below 20 MV/m of the cavity. Above 20 MV/m, the argon doping reduced the high field Q slope seen in the BCP-prepared surface. This could be caused by low levels of argon doping in the RF penetration layer, sufficient to reduce high field Q slope, e.g. by suppressing formation of lossy hydrides in cool down [4]. It is certainly clear that argon does not produce the same benefits as nitrogen at medium fields.



doi:10.18429/JACoW-IPAC2015-WEPTY073

IPAC2015, Richmond, VA, USA

JACoW Publishing



Figure 3: 2.0 K Q_0 vs E_{acc} performance for a cavity before (bulk BCP treatment) and after argon doping.



Figure 4: 2.0 K Q_0 vs E_{acc} performance for a 9-cell cavity before and after nitrogen-doping.

9-CELL STUDY

In order to more closely track quench field degradation from nitrogen-doping, a 1.3 GHz 9-cell ILC shaped cavity was prepared. First, it was prepared with standard preparations: bulk EP (50 μ m), hydrogen degassing in vacuum furnace, field flatness tuning, light EP (5 μ m), and 48 hour 120°C bake. The cavity was cooled down from room temperature to 4.2K quickly with the cool down rate of >5 K/min (fast cool down). The 2.0 K results from this baseline test are shown in Fig. 4. The achieved Q_0 during VT was 2.0×10^{10} at 16 MV/m, 2K. The cavity performance was limited by quench at E_{acc} of 21 MV/m with Q_0 of 1.6×10^{10} . A small amount of radiation of 1mR/h was detected at quench field.

After the baseline test, nitrogen-doping was performed. The cavity received a light EP (10 μ m) to remove low temperature bake effect, heat treatment at 800°C in vacuum for 3 hours, in 20 mTorr of nitrogen gas for 2 minutes, in vacuum again for 6 minutes, field flatness tuning, and light EP (5 μ m). The cavity received the same fast cool down as in the baseline test, and was tested at 2.0 K. The cavity displayed a strong Q-slope above 10 MV/m. The achieved Q_0 was 2.0×10^{10} at 15 MV/m and limited by quench, but no detectable radiation during the test. This curve can also be seen in Fig. 4. A thorough discussion of the quench source will be presented in the following section.

QUENCH STUDIES

Single-Cells

Using the Cornell single-cell temperature mapping system (T-Map), the quench location of cavities can be detected (the

and details of this method are described in [5]). This method publisher, quickly measures the time given resistors in the T-Map are "warm" after a quench. This technique was carried out on the over-doped single-cell cavity discussed above after 6 μ m and 18 μ m (total) EP. After 6 μ m removal, the quench was centered one location (see Fig. 5(a)). After 18 μ m total eremoval, the quench moved but was still dominated by a 5 single location near the equator but now centered on board $\frac{b}{H}$ 14 (see Fig. 5(b)). A localized quench location suggests that the quench was caused by a defect region. Any distribution of this work must maintain attribution to the author(s).



Figure 5: Quench detection with single-cell temperature mapping system.

9-Cells

BY 3.0 licence (© 2015). A similar quench detection system was used on the 9-cell cavity, using the Cornell multi-cell T-Map system. The Tmap covered the middle 7 cells of the 9-cell cavity. In both tests, the strongest quench signal occurred in cell 8, near he $\frac{1}{2}$ the equator. Additionally, quench location was found using OSTs. 16 OSTs were installed with the cavity and successfully detected the quench signal during the two RF tests. 2 The OSTs predicted the same quench location as the T-Map $\frac{1}{2}$ during the two tests, confirming that the quench location did not change due to nitrogen doping though the quench field sed was strongly reduced. Upon optical inspection, a protrusion was found at the quench location. This protrusion persisted þ after nitrogen-doping. We theorize that the nitrogen-doping E lowered the lower critical field, H_{C1} . This together with magnetic field enhancement at the protrusion caused the Content from this work quench field to drop dramatically.

H_{c2} MEASUREMENTS

PPMS allows one to measure the upper critical field, H_{c2} , for a given samples. By measuring the change in critical tem-



Figure 6: Critical temperature vs applied field strength for the sample that had 24 μ m of removal after doping.

perature as a function of applied external field strength and excitation current through the sample, H_{c2} can be extracted. The exact details of these measurements are discussed in [6]. Two samples were prepared with nitrogen-doping: heat treatment in vacuum at 800° for 3 hours, followed by heat treatment in 60 mTorr of nitrogen gas for 20 minutes, followed by an additional heat treatment in vacuum for 30 minutes. The two samples were then given a different amount of BCP: 12 μ m and 24 μ m, respectively. The data from the 24 μ m samples is shown in Fig.6. The extracted H_{c2} for the two samples was 520 \pm 120 mT (12 $\mu m)$ and 550 \pm 110 μm (24 μ m). This data suggests that the nitrogen-doped samples are indeed dirty, with a lower mean free path than clean niobium.

CONCLUSIONS

A series of experiments have been carried out at Cornell in search of the cause of quench degradation due to nitrogendoping. In both single and 9-cells quench was found to be located at localized areas on the cavities. The quench spot was associated with a protrusion in the 9-cell cavity. Over-doping of a single-cell cavity with nitrogen produced excellent Q_0 's but low quench fields that increased with more material removal. 120°C baking was shown to have no effect on the Q_0 of a nitrogen-doped cavity but severely reduced the quench field. Argon doping was also shown to have no effect on the medium field Q_0 , however high field Q slope was eliminated, possibly due to light doping of the RF penetration layer. Finally, we presented the first measurements of H_{c2} for nitrogen-doped niobium samples. These samples showed a significantly higher H_{c2} than clean niobium.

Future work will continue H_{c2} measurements with PPMS on more samples of different treatments. Also we will study the superheating field of nitrogen-doped niobium using a high power klystron at Cornell. All together this work represents a critical step towards fundamentally understanding the mechanism behind nitrogen-doped SRF cavities.

REFERENCES

[1] A. Grassellino, A. Romanenko, D. Sergatskov, O. Melnychuk, Y. Trenikhina, A. Crawford, A. Rowe amd M. Wong, T. Khabiboulline, and F. Barkov. Nitrogen and argon doping of niobium

> 7: Accelerator Technology **T07 - Superconducting RF**

for superconducting radio frequency cavities: a pathway to highly efficient accelerating structures. *Superconductor Science and Technology*, 26(102001), 2013.

- [2] Dan Gonnella, Mingqi Ge, Fumio Furuta, and Matthias Liepe. Nitrogen Treated Cavity Testing at Cornell. In *Proceedings of LINAC 2014, Geneva, Switzerland*, number THPP016, Geneva, Switzerland, September 2014. JaCoW.
- [3] Dan Gonnella and Matthias Liepe. New Insights into Heat Treatment of SRF Cavities in a Low-Pressure Nitrogen Atmosphere. In *Proceedings of IPAC 2014, Dresden, Germany,* number WEPRI064, pages 2634–2637, Dresden, Germany, June 2014. JaCoW.
- [4] A. Romanenko, F. Barkov, L. D. Cooley, and A. Grassellino. Proximity breakdown of hydrides in superconducting niobium cavities. *Superconductor Science Technology*, 26:5003, 2013.
- [5] Dan Gonnella, Sam Posen, and Matthias Liepe. Quench Studies of a Superconducting RF Cavity. In *Proceedings of IPAC* 2012, New Orleans, LA, USA, number WEPPC071, pages 2375–2377, New Orleans, LA, USA, May 2012. JaCoW.
- [6] J.T. Maniscalco, D. Gonnella, D.L. Hall, S. Posen, and M. Liepe. HC2 Measurements of Nb3Sn and Nitrogen-Doped Niobium Using Physical Property Measurement System. In *Proceedings of IPAC 15, Richmond, VA, USA*, number WEPTY075, Richmond, VA, USA. JaCoW.