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INDUSTRIAL CAVITY PRODUCTION: LESSONS LEARNED TO PUSH THE BOUNDARIES OF NITROGEN-DOPING*

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Abstract

Nitrogen doping has been proven now in several labs to enhance Q_0 values of 1.3 GHz cavities in the gradient domain favored by CW operation. The choice of doping for the LCLS-II project has given the community a wealth of statistics and experience on the challenge of transferring the doping technology to industry. Overall, industry-produced nitrogen-doped cavities have shown excellent performance, however some technical issues have arisen. This talk focuses on lessons learned from the production of over 300 nitrogen-doped cavities for LCLS-II and how issues were mitigated to further improve performance. Finally, I will discuss pushing the boundaries of nitrogen-doping further by exploring different doping regimes in order to maintain excellent Q_0 performance, while reaching higher quench fields.

INTRODUCTION

Nitrogen-doping has been successfully demonstrated in the lab setting to produce SRF cavities capable of reaching previously unprecedented Q_0 values at 2 K and in the medium field regime [1]. The Linac Coherent Light Source II (LCLS-II), currently being constructed at SLAC National Accelerator Laboratory, is the first large-scale accelerator project to employ nitrogen-doping technology in order to reach new levels of cryogenic efficiency in a CW machine. Early results from the production of nitrogen-doped cavities for LCLS-II demonstrated the transfer of the technology from the lab setting to SRF industry [2]. Production of the cavities for LCLS-II is now complete and has been overall successful, however, not without challenges. Here we provide a summary of the major lessons learned from the production of 373 nitrogen-doped cavities for LCLS-II. In light of the success of LCLS-II cavity performance, we also look to the future in how doping can be pushed further in order to enable high Q_0 operation at higher gradient values for the LCLS-II High Energy (HE) project.

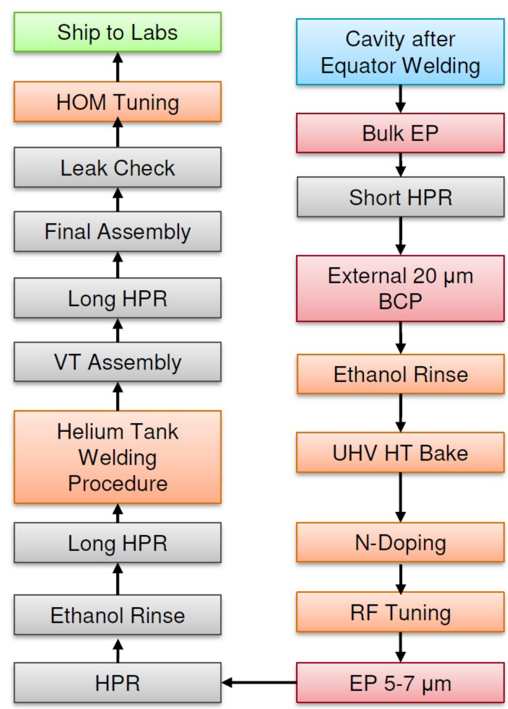

Figure 1: Production sequence for the nitrogen-doped cavities for LCLS-II.

LCLS-II CAVITY PRODUCTION AND REQUIREMENTS

The LCLS-II linac consists of 35 cryomodules, each with 8 1.3 GHz 9-cell cavities. An additional 5 cryomodules are being constructed as production spares. In order to reach an electron energy of 4 GeV, the cavities must operate at an average gradient of 16 MV/m in CW. These cavities must meet an average Q_0 of $2.7{\times}10^{10}$ at the operating gradient in order to enable operation of the linac on a single cryoplant. In order to meet this difficult specification, the cavities are prepared with nitrogen-doping. A snapshot of the production sequence for the cavities is given in Fig. 1. In the beginning

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of production, the bulk EP was 140 μm nominal removal and an 800°C UHV bake for hydrogen degassing. These parameters were changed mid-production, which will be discussed in the next section. The nitrogen-doping protocol was the so-called "2/6 nitrogen-doping" - consisting of two minutes at 800°C in a low pressure (~25 mTorr) of nitrogen, followed by 6 minutes in vacuum. The 2/6 doping has been routinely shown to produce cavities that meet the Q_0 and gradient requirements for LCLS-II.

To enable the construction of the 40 cryomodules (including two prototypes), and to account for yield losses from unqualified cavities, a total of 373 1.3 GHz 9-cell cavities were ordered. The cavities were produced by Ettore Zanon S.p.a. (EZ) and RI Research Instruments (RI). The niobium for the cavities was produced by Tokyo Denkai (TD) and OTIC Ningxia (NX). Additionally, two prototype cryomodules were built using cavities from ILC R&D, produced by AES and made up of ATI Wah-Chang (WC) niobium. The cavities shipped from the vendors under vacuum and fully equipped for vertical test. In order to qualify for cryomodule string assembly, a given cavity must, in vertical test, meet a Q_0 of 2.5×10^{10} at 16 MV/m and a quench field ≥19 MV/m with no detectable field emission up to the maximum field. The lowering of the Q_0 spec from 2.7×10^{10} to 2.5×10^{10} is due to the presence of a stainless steel blank on the short side of the cavity, causing an additional residual resistance (R_{res}) of 0.6 nΩ [2]. These requirements are summarized in Table 1.

LCLS-II HE will increase the electron energy of the LCLS-II linac from 4 to 8 GeV by adding an additional 20 cryomodules, operating at an average gradient of 20.8 MV/m and increasing the average gradient in the LCLS-II cryomodules to 18 MV/m. In order to keep the cryoload below the capacity of the two 2 K 4 kW cryoplants at SLAC, the HE cavities must maintain a Q_0 of 2.7×10^{10} at 21 MV/m. These stricter specifications require further development of the nitrogen-doping protocol.

LESSONS LEARNED FROM CAVITY PRODUCTION

Overall, cavity performance of the nitrogen-doped cavities for LCLS-II has been very good. Figures 2 and 3 show histograms of maximum gradient and Q_0 for all of the cavities tested. They are colored to highlight the main issues that were encountered during production, which will be discussed in the succeeding sections. As can be seen, most cavities exceed the requirements of LCLS-II, but there are a handful of low performers.

In terms of gradient, there were two main factors which limited performance:

- Poor fabrication techniques at EZ
- Furnace contamination entering the cavities at temperatures above 950°C.

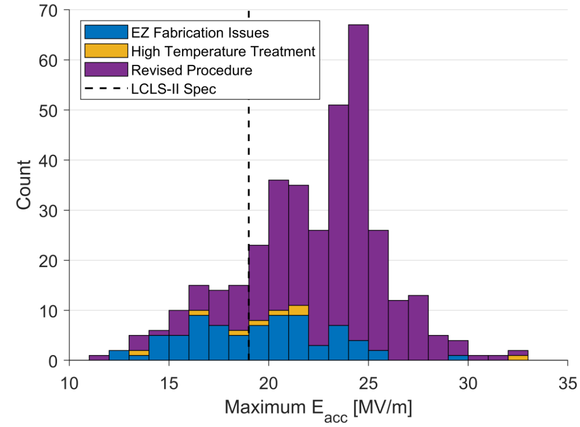

Figure 2: Maximum E_{acc} for the cavities tested for LCLS-II. Also shown is the vertical test specification of 19 MV/m. Bars are stacked.

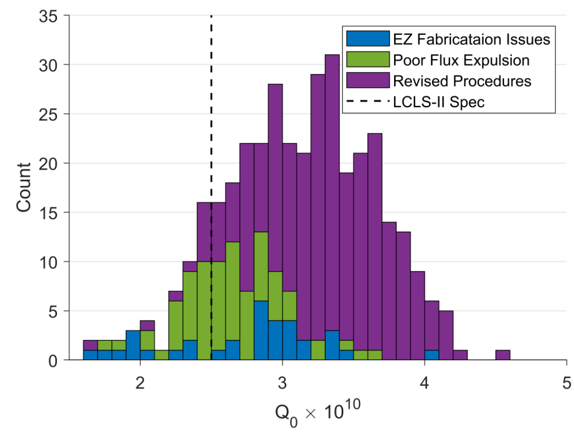

Figure 3: Q_0 at 16 MV/m (or highest attainable field) for the cavities tested for LCLS-II. Also shown in the vertical test specification of 2.5×10^{10}. Bars are stacked.

These two issues highlight major lessons learned from the nitrogen-doping production. For Q_0 there were also two main factors which limited performance:

- Poor flux expulsion of material annealed at too low temperatures.
- Poor fabrication techniques at EZ leading to strong Q-slope.

These items leave three lessons learned from the cavity production, which were not learned by the previous large-scale accelerator project, the European XFEL. The use of nitrogen-doping for LCLS-II uncovered these issues - not necessarily because nitrogen-doped cavities are more sensitive to them but because great care must be taken to achieve high Q_0 cavities.

Flux Expulsion

The first production LCLS-II cavities were produced with TD material and given a hydrogen degas at 800°C prior to the

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Table 1: LCLS-II and LCLS-II HE Cavity Operating Parameters

Parameter	LCLS-II	LCLS-II HE
# 1.3 GHz CMs	35	20
# Cavities Purchased	373	160[1]
Operating Gradient	16 MV/m	20.8 MV/m[2]
Q_0 at Operating Gradient	2.7×10^{10}	2.7×10^{10}
Q_0 at Operating Gradient in VT	2.5×10^{10}	2.5×10^{10}
Quench field in VT	≥19 MV/m	≥23 MV/m

[1] Nominal amount, cavities not yet ordered.
[2] LCLS-II cryomodules will operate at an average gradient of 18 MV/m for LCLS-II HE.

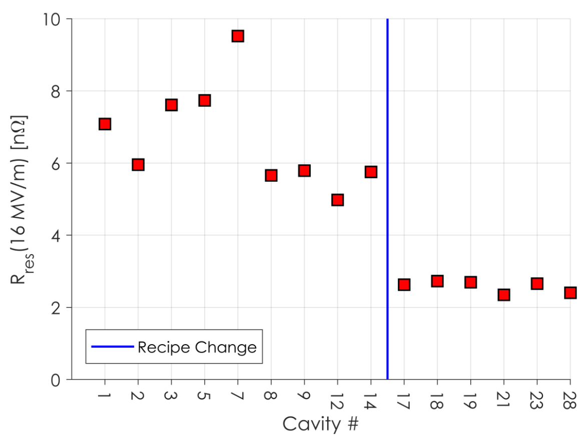

Figure 4: R_{res} of the first production cavities from RI, separated by the point in production where the degas temperature was increased from 800°C.

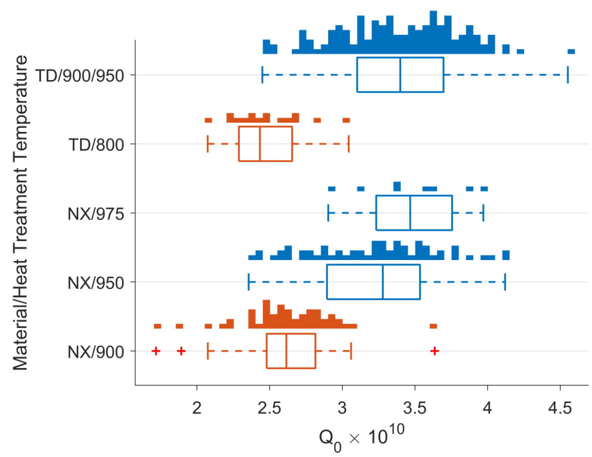

Figure 5: Boxplots and histograms showing the Q_0 performance of cavities in vertical test, separated by material vendor and heat treatment temperature. TD/800 and NX/900 cavities performed poorly while treating at higher temperatures produced cavities with high Q_0.

nitrogen-doping phase, as discussed above. However, these cavities did not reach the expected high levels of Q_0 that had been demonstrated on lab-produced nitrogen-doped cavities. Further investigation unveiled that this was due to excessive R_{res}, as can be see in Fig. 4. It was expected to receive cavities with R_{res} of 2-3 nΩ, but more than double this was observed. Concurrent to this discovery, work by Posen et. al. demonstrated that heat treatment temperature has a strong effect on the flux expulsion of niobium cavities [3]. Subsequent studies found that indeed the first production cavities did not expel flux as efficiently as was expected. Therefore an increase in the furnace degas temperature was required in order to improve flux expulsion.

The next batch of cavities (still produced with TD material) were treated at 900°C instead of 800 and performance was significantly better. In Fig. 4, one can see that after the recipe change, R_{res} was in line with expectations of 2-3 nΩ.

Increasing the degas temperature however did not improve flux expulsion on all cavities. Mid-production it was found that NX material did not respond to the same temperature as TD material. In fact, increasing the heat treatment temperature to 950 and 975°C in some cases was required to achieve the same level of flux expulsion as the early cavities produced with TD material and treated at 800°C. This was verified on single-cell cavities constructed out of the same material as the 9-cells and using the method described in [3]. At the end of production, with a new batch of TD material being used, it was found that 950°C was required for some cavities.

A summary of Q_0 performance separated by material and heat treatment temperature is shown in the combined boxplot and histogram in Fig. 5. An important and costly lesson was thus learned from this - material flux expulsion needs to be checked and verified prior to the construction of 9-cell cavities. For LCLS-II and the future LCLS-II HE cavity order, this means sorting of niobium for cavity fabrication by heat treatment lot (at the niobium vendor). Each lot can then have a single-cell constructed from it which can be used to determine the minimum temperature at which good flux expulsion is achieved.

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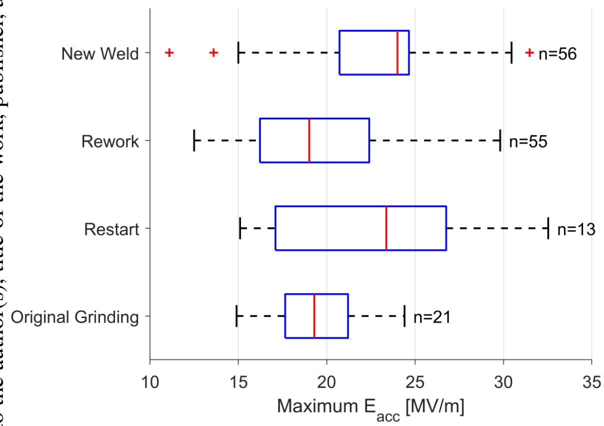

Figure 6: Gradient performance of the different groups of EZ cavities. Cavities produced with the original grinding and weld-stackup procedures had poor gradient reach. Improvement of grinding procedures led to an increase in quench fields on average but still many low performers. Rework of these cavities was marginally successfull. Improvement of all procedures led to good gradient reach.

Vendor Oversight and Process Control

The first production nitrogen-doped cavities produced by EZ showed significantly worse gradient performance than expected. Cavities on average quenched at ~17 MV/m and many showed a strong Q-slope starting at ~13 MV/m. This Q-slope also dragged the Q_0 of the cavities down, and combined with poor flux expulsion (as discussed in the previous section) resulted in many cavities that were below the Q_0 specification at 16 MV/m. The distribution of first production unites from EZ is shown in Fig. 6.

In light of the poor performance, all production at EZ was placed on hold and a thorough audit of their procedures was completed by JLab and SLAC staff. Three main issues were found:

1. Etching rather than polishing during the bulk and light EP's.
2. Grinding of dumbbells with aggressive tooling which caused the embedding of normal conducting media into the cavity surface.
3. Dirty weld stackup procedures which led to excessive weld splatter leading to cat-eye defects.

EP Issues The electropolishing technology was transferred from JLab to EZ without extensive on-site project oversight. Changing of the EP parameters from those EZ had used before, combined with using the incorrect cathode resulted in the EP's being in the etching rather than the polishing regime. This ultimately led to extremely rough surfaces. It is well-known that rough surfaces, such as those produced by BCP, cannot be used with nitrogen-doping due to gradient limitations. This issue was ultimately fixed through vetting of the EP parameters and re-training of the EZ staff by JLab EP experts.

Aggressive Grinding It was also found that dumbbells left the machine shop at EZ with extensive defects such as pitting. This is typically not an issue for high performing SRF cavities if the defects are handled properly. Unfortunately EZ staff were grinding the entirety of the dumbbell surface to a uniform smoothness with an aggressive paddle-wheel style grinder. This ultimately led to the embedding of normal conducting media, such as aluminum in the bulk of the niobium. After the 200μm bulk EP, this media could be revealed, however it would not be etched away by the EP due to the lack of nitric acid in the acid mixture. Improvement of grinding procedures, requiring significantly less aggressive tooling and disallowing of whole dumbbell grinding ultimately fixed this issue. However, this required vigilant project staff to evaluate procedures and train EZ staff.

Weld Defects Improvement of the previous two issues improved gradient performance significantly to an average quench field of ~22 MV/m, however there were still a handful of low performers with quenches below 15 MV/m being produced. Further investigation by project staff at EZ found that dirty weld stackup procedures were resulting in weld splatter on some cavities which ultimately led to cat-eye defects inside the cavities. These defects ultimately would lead to lower quench fields. Weld splatter was not explicitly forbidden in the cavity contract, however it was required to be ground away. Improvement of these stackup procedures significantly improved cavity performance and ultimately brought EZ cavities in line with those produced by RI.

Rework of Rejected Cavities Ultimately, approximately 70 cavities were effected by the issues at EZ discussed above. A fraction of these cavities (~50) were reworked to improve performance. The exact rework for each cavity depended on the state of the cavity (some were already in helium vessels, some welded but not yet in helium vessels, and some not yet welded). Typically this rework involved BCP to attack the normal conducting media that had been embedded in the surface from the aggressive grinding. Typically this rework had a success rate of ~50%, an improvement of 20% compared with the original cavity performance. Cavities which were not yet welded (in the dumbell stage) had significantly better success with rework than those already welded. This is attributed to the fact that they were welded after improvement of the weld stackup procedures. The distributions of gradient reach for the rework cavities is given in Fig. 7.

Issues at EZ were ultimately resolved through thorough vetting of vendor procedures and on-site presence of project staff. This highlights that oversight is still required for cavity production, especially when dealing with nitrogen-doped cavities which have proven to be more susceptible to issues that arise during production.

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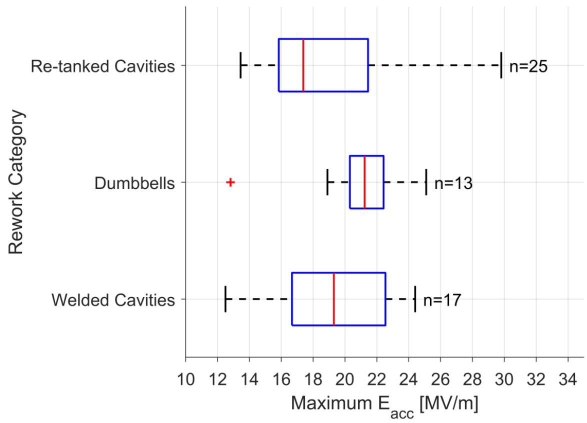

Figure 7: Gradient performance of the EZ rework cavities. Cavities that were constructed of reworked dumbbells showed the highest rates of rework success. In total ~50% of cavities were successfully reworked.

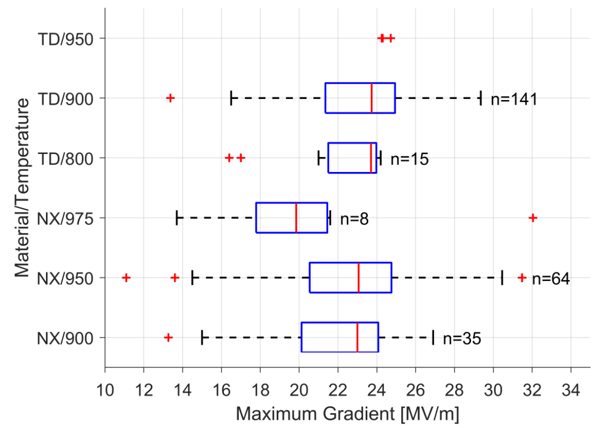

Figure 8: Quench field versus material and heat treatment temperature. Cavities treated at 975°C showed a drop in quench field consistent with furnace contamination, however statistics are limited.

High Temperature Heat Treatment and Quench Field

In general, there has been no difference in gradient reach for cavities produced with material from different vendors. Figure 8 shows the quench field distributions for cavities separated by material vendor and heat treatment temperature. Typically there is also no correlation between quench field and heat treatment temperature either, however, there is a small drop of ~3 MV/m in quench field for cavities treated at 975°C in RI's furnace. At 975°C a small pressure rise was observed after about an hour, suggesting that part of the furnace warmed up and started outgassing. This likely caused contamination of the cavities which resulted in a lower quench field. This is unfortunate since high temperatures were required to improve Q_0, but could in turn cause a lowering of E_{acc}. Therefore great care must be taken to ensure that furnaces stay clean. When contamination is known to be present, a two stage electropolish and furnace treatment could be used where the cavity is first treated at high temperatures, the receives an EP to clean contamination, and then a second furnace run including the doping. This has been theorized to cure the contamination issue.

Applying Lessons Learned

Applying the three lessons learned described in the previous section results in excellent performance of nitrogen-doped cavities. Figures 9 and 10 show E_{acc} and Q_0 performance of the cavities prepared with good procedures and sufficient heat treatment to achieve good flux expulsion.

Following implementation of the lessons learned, cavities have demonstrated an average E_{acc} of 23.1 MV/m and an average Q_0 of 3.3×10^{10}. This performance is significantly better than what is required for LCLS-II and enables pushing the boundaries further to construct an accelerator with even more ambitious requirements than that of LCLS-II.

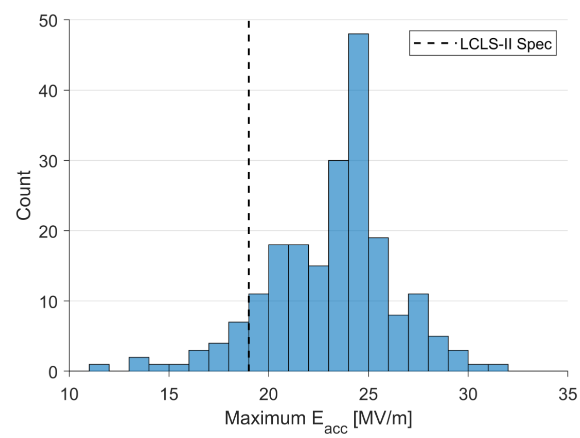

Figure 9: E_{acc} performance of the cavities prepared with improved procedures for dumbbell grinding and weld stackup.

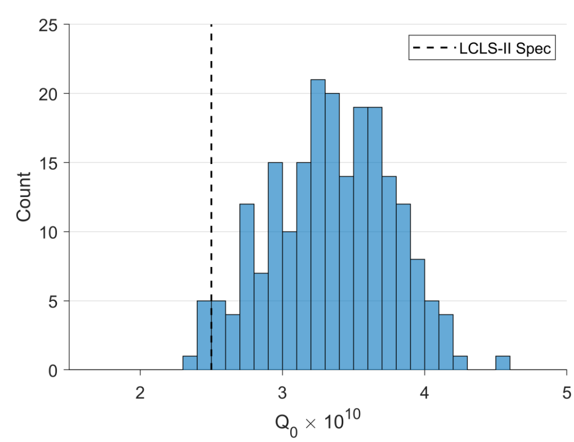

Figure 10: Q_0 performance of the cavities prepared with improved fabrication procedures and heat treated at high enough temperatures for sufficient flux expulsion.

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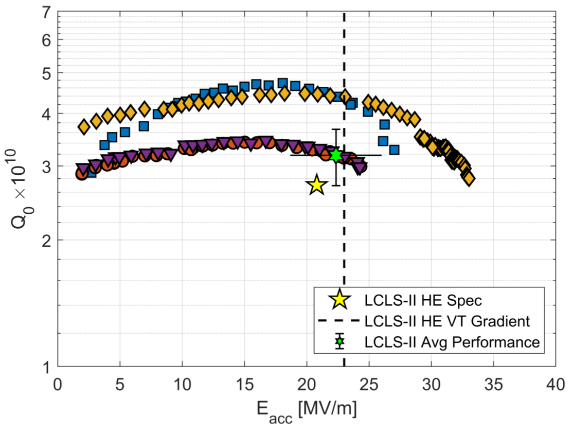

Figure 11: Q_0 vs E_{acc} performance at 2 K of single-cell cavities prepared with the 2/0 nitrogen-doping. All cavities tested demonstrated performance in excess of the LCLS-II HE requirements.

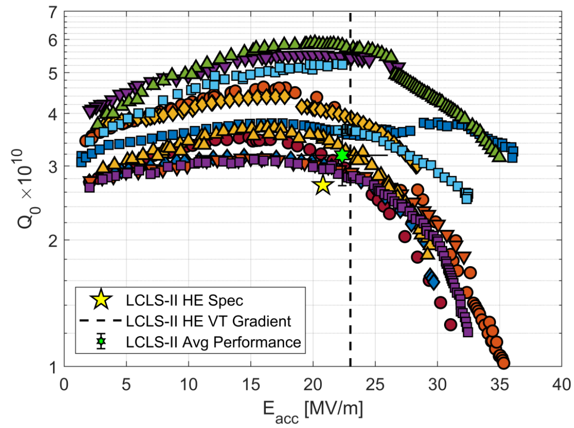

Figure 12: Q_0 vs E_{acc} performance at 2 K of single-cell cavities prepared with the 3/60 nitrogen-doping. Nearly all cavities tested demonstrated performance in excess of the LCLS-II HE requirements.

NITROGEN-DOPING IN THE FUTURE: LCLS-II HE

As discussed in the previous section, SRF cavities can be produced, through the use of nitrogen-doping and great care, which achieve an average quench field of 23 MV/m. For LCLS-II HE however this is not good enough. HE has a qualifying vertical test gradient of 23 MV/m, therefore if the LCLS-II cavities were used, ~40% of the cavities would not qualify. Therefore, a change to the cavity doping protocol must be done to push the gradient reach higher, but with the same high Q_0. In order to achieve this lofty goal, an R&D program was developed and has been thoroughly described in [4]. Two candidate recipes have been pursued, the 2/0 (two minutes with nitrogen, no anneal time), and the 3/60 (three minutes with nitrogen, followed by a 60 minute anneal). Single-cell results have been excellent thus far and are shown in Figs. 11 and 12. Nearly all the cavities tested exceed the LCLS-II HE requirements.

Success of the single-cell program motivated preparing 9-cell cavities with one or both of the new doping recipes. This endeavor encountered significant issues as described in [4]. However, improvement of the furnace caps and electropolish system has proved successful in producing an HE qualified 9-cell with exceptional gradient performance. This is shown in Fig. 13. A cavity from LCLS-II, prepared by RI with the 2/6 recipe quenched at 23 MV/m. After reset and doping with the 2/0 recipe at FNAL, it quenched at 32 MV/m, a 30% increase in quench field. This result paves the way for the production of more 9-cell cavities prepared with the new recipe in order to reach higher gradients while still maintaining high Q_0.

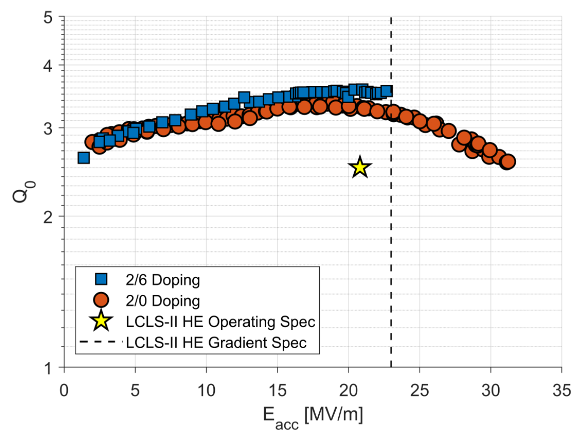

Figure 13: Q_0 vs E_{acc} performance at 2 K of a 9-cell cavity produced with the 2/6 nitrogen-doping and then again with the 2/0 doping. Increase of the quench field by 30% was observed.

CONCLUSIONS

The production of nitrogen-doped cavities for LCLS-II has largely been successful. Three important lessons were learned throughout production which have proven to be necessary to implement in order to achieve excellent cavity performance.

Good flux expulsion is crucial in order to maintain high Q_0. At 16 MV/m and 2 K, a 2/6 nitrogen-doped cavity has ~10 nΩ of surface resistance. At that level, every additional nOhm of surface resistance is important and trapped flux will contribute significantly to the total surface resistance. In order to guarantee good flux expelling cavities, single-cell cavities must be fabricated from each niobium heat treatment batch. Moreover, niobium must be sorted by required heat treatment temperature for production cavities.

Vendor oversight and thorough vetting of procedures is necessary to produce excellent performing cavities. While the cavity vendors can produce good cavities, on-site presence is important to maintain a high level of quality. This ultimately led to the improvement of cavities produced by

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EZ and brought their routine cavity performance to be in line with that of RI.

Due to the flux expulsion requirements, some material needed to be treated at temperatures as high as 975°C. It was found that for these cavities, there was a significant drop in quench field compared with cavities treated at lower temperatures. This has been attributed to furnace contamination. Great care must be taken to ensure the cleanliness of vendor furnaces and using a two stage EP and furnace cycle may be necessary to achieve both high Q_0 and high gradient.

With improvement to procedures, LCLS-II cavities performed well in excess of their expectations. Nitrogen-doping is well on its way to industrialization, but it is not fully industrialized yet. Project oversight and vigilance is still required to maintain good cavity performance. Moreover as LCLS-II HE further pushes the boundaries of nitrogen-doping, further care will need to be taken in order to reach even better performance.

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