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TRANSFER OF EP AND DOPING TECHNOLOGY FOR PIP-II HB650 CAVITIES FROM FERMILAB TO INDUSTRY*

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Abstract

Fermilab has optimized the surface processing conditions for PIP-II high beta 650 MHz cavities. This encompasses conditions for bulk electropolishing, heat treatment, nitrogen doping, post-doping final electropolishing, and post-processing surface rinsing. The technology has been effectively transitioned to industry. This paper highlights the efforts made to fine-tune the process and to smoothly share them with the partner labs and an associated vendor.

INTRODUCTION

The Proton Improvement Plan II (PIP-II) is an upgrade to the Fermilab accelerator complex, designed to deliver an 800 MeV proton beam to create the world's most intense neutrino beam for the Deep Underground Neutrino Experiment (DUNE). The PIP-II linear accelerator (LINAC) will be developed with support from international partners and industry, focusing on the integration of various types of niobium (Nb) superconducting RF (SRF) cavities into the LINAC. The international partner laboratories responsible for delivering the high-beta (0.92) 650 MHz (HB650) cavities are Raja Ramanna Centre for Advanced Technology (India) and Science and Technology Facilities Council, UK Research and Innovation (UKRI, UK), with additional support from industry. For UKRI, Zanon Research and Innovation Srl (Italy) is tasked with fabricating these cavities and conducting the necessary surface processing.

Previous projects like LCLS-II and LCLS-II HE, led by the United States with industrial collaboration, have set a successful precedent. The achievements of these projects were made possible through effective technology sharing with industry partners [1, 2]. Surface processing of the Nb cavities is a critical step in ensuring the cavities meet the performance specifications required for the cryomodules. The knowledge gained from these projects is invaluable for future endeavors and is currently being applied to the ongoing PIP-II project.

This paper reports on the surface processing technology and the lessons learned, which have been shared with partner labs and the vendor working on the HB650 cavities.

LESSONS LEARNED ON EP OF HB650

Fermilab and other SRF facilities have extensive experience in electropolishing (EP) 1.3 GHz cavities and other surface processing techniques [1, 3]. The HB650 cavity is significantly larger, with nearly twice the surface area of a 1.3 GHz cavity, making the same standard EP conditions unsuitable for achieving a smooth surface, as shown in our latest EP study conducted with LB650 and HB650 cavities [3, 4]. Based on the studies performed, key findings and lessons learned were identified as summarized below.

The standard 18 V was found to be insufficient for the EP of HB650 cavities, resulting in a rough equator surface and premature quench of the cavities [3]. This conclusion is supported by I-V curves measured for 650 MHz cavities under various conditions. The I-V curve with two major regions, linear and plateau regions, provides insight into the chemistry of the cavity surface. A smooth surface is achieved when EP is conducted at a voltage within the plateau region.

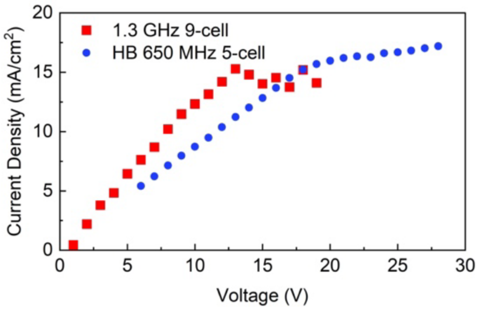

Figure 1: I-V curves measured for 1.3 GHz and HB 650 MHz cavities. The I-V curve for HB650 was obtained with new cathode structure.

Unoptimized cathode structure and surface area fail to yield the current plateau even up to 25 V [3]. A new cathode was designed to enhance the surface area and acid flow conductance, and to reduce cathode polarization. The plateau onset (V_{onset}) was obtained at relatively lower voltage as compared to the initial cathodes. A lower EP voltage is also important to mitigate sulfur generation. However, HB650 cavities still requires an EP voltage higher than the standard voltage. The I-V curves for a 1.3 GHz 9-cell cavity and an HB650 cavity are compared in Fig. 1.V_{onset} varied with the type of cathode, cavity temperature, and acid conditions.

A higher EP current either due to a higher cavity temperature or a stronger acid (higher HF or F$^-$ ions) increases V_{onset}. The acid resistance and cathode polarization might be responsible for the higher V_{onset} at a higher EP current. The V_{onset} shows linear relation with EP current [4].

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Acid conditions can vary significantly at different laboratories. Different batches of acid from the same supplier have shown variations, as observed at Fermilab. Due to these inconsistencies, the exact same EP parameters may not be suitable for implementation at other laboratories. To address this, the selection of EP voltage has been standardized to prevent incorrect voltage choices that could arise from variations in acid condition and temperature.

We have defined EP voltage V_{EP} as $V_{EP} = V_{onset} + 5$. Setting V_{EP} at least 5 V higher than the V_{onset} ensures that the entire cavity surface, including the distant equator from the cathode, remains in a diffusion-limited condition. Typical maximum voltages for warm and cold EP were estimated to be 25 V and 20 V, respectively. Here, warm and cold EP refer to EP conducted at maximum cavity temperatures of 25°C and 12°C, respectively. The cavity temperature must be maintained uniformly within ±1°C.

Achieving an accurate V_{onset} requires proper I-V curve measurements under controlled cavity temperature conditions and a proper voltage ramp up rate. To minimize inaccuracies in determining V_{onset}, the I-V curve measurement protocol has been standardized. A voltage increase of 1 V every 12 seconds was found to be appropriate. For I-V measurements, data logging should be performed with a small timestamp, typically 250 milliseconds, to capture detailed information, especially current oscillations.

Current oscillations, considered an indicator of optimal EP, were found to be synchronized with the onset voltage V_{onset}. However, the current oscillations were absent when using aged acid, particularly during cold EP, even when the EP conditions were adequate to produce a smooth surface. Additionally, low-amplitude current oscillations were detected during unoptimized EP. Therefore, it is essential to correctly identify the nature of current oscillations if they are to be relied upon as a sign of optimal EP.

The HB650 production cavities have been considered to receive nitrogen-doping treatment, which is discussed in the following section. The N-doped cavity surface requires only light removal of 7-10 µm removal in the final EP process. Recent EP R&D on N-doped surfaces at Fermilab revealed that high voltage and elevated EP surface temperatures can cause pitting on the N-doped surface [5]. To address this issue, we developed a two-step EP method to prevent pitting on N-doped surfaces. In the first step, the top thin nitride layer is removed by etching at a low voltage, outside the plateau region. This is achieved through an I-V scan that includes the low-voltage region. The second step involves performing optimal EP within the plateau region. Given the high EP voltage required for 650 MHz cavities, this two-step EP method could be important to avoid pitting and prevent performance degradation caused by surface pits.

HEAT TREATMENT AND N-DOPING

N-doped cavities are sensitive to trapped flux, which enhances surface resistance and lowers Q_0. The R&D work [6] suggests that a high temperature (900 °C) heat treatment (HT), compared to 800 °C, improves flux expulsion. HT at 900 °C for 3 h has been implemented on LCLS-II and HE project cavities, which are N-doped, to reap the benefit. Lessons learned from the R&D work, as well as from the LCLS-II and LCLS-II HE projects, led to the adoption of 900 °C for PIP-II HB650 cavities as well.

N-doped studies were mainly performed with 1.3 GHz cavities. Different doping recipes including 2/6, 3/60, and 2/0 were studied. The 2/0 recipe was applied to cavities in R&D with an expectation of benefiting from N-doping on Q_0 and achieving a higher quench field due to the comparatively mild doping. Indeed, 2/0 doping showed an increased quench field compared to other doping recipes in 9-cell cavities [7].

Given 650 MHz cavities have lower surface BCS resistance, N-doping effect might not be as significant. To confirm this, Fermilab conducted 2/6 and 3/60 N-doping studies with HB650 single-cell cavities [8]. The results confirmed the benefit of applying N-doping to HB650 cavities in terms of improved Q_0 value. The quench field with the 2/6 N-doping recipe appeared similar as in LCLS-II 9-cell production cavities processed with the same N-doping recipe. 5-cell HB650 cavities assembled in the prototype cryomodule also received N-doping and satisfied the cryomodule specification ($Q_0 = 3.3\text{x}10^{10}$ at 18.8 MV/m) [9]. The study confirmed that N-doping has the potential to enhance Q_0 at medium fields in HB650 cavities.

The promising performance of 2/0 N-doped 9-cell cavities for LCLS-II HE, this doping method was also considered for HB650 cavities. To verify its effect, a pre-production HB650 cavity (B92F-RI-203) underwent 900 °C HT, 2/0 N-doping, and a two-step final EP for 10 µm removal. The cavity performance, shown in later section, met the cryomodule specification. Also, mechanical stiffness was checked by the drop tests on other 650 MHz cavities [10]. The test results confirmed that 900 °C HT can be applied to HB650 cavities. Based on those results, final parameters were confirmed and communicated to the partners and vendor.

SURFACE PROCESSING IN PRODUCTION AND TECHNOLOGY SHARING

The major surface processes applied to the cavities during the production phase include bulk EP (160 µm), HPR, 900 °C HT, 2/0 N-doping, final two-step cold EP (7-10 µm), ultrasonic cleaning or ethanol rinsing, HPR, vertical test, jacketing, and a further vertical test.

The lessons learned, as mentioned in the earlier sections, have been shared with the partner labs and Zanon. The following important key aspects of EP were considered. (1) EP voltage (2) cathode structure (3) I-V measurement, (4) cavity temperature for warm and cold EP, and (5) two-step final EP after N-doping. The patented cathode design [11] has also been shared. The partner labs and vendor have flexibility to use the same design or develop their own cathodes compatible with their EP tools. However, the I-V curve must display the EP plateau within the specified voltage range at the desired cavity temperature in warm and cold EP cases. While the two-step final EP can be performed using the same setup and EP acid once everything is in place, implementing this process did involve some upfront costs, although relatively modest in the overall scope of the contract. Specifically, Zanon had to modify their automated EP software, conduct testing, and train staff, as well as rewrite protocols and procedures. Additionally, a new power supply was purchased to allow higher bulk EP voltage requirement. The partner labs and vendor also visited FNAL/ANL to observe the EP facility and the EP run of HB650. In person and remote support was provided during the EP of the UKRI-owned first pre-series cavity B92M-ZRI-301 at Zanon to fine-tune the EP process.

Since the vendor had experience from past projects, sharing the HT and N-doping process was relatively smooth. However, the set protocols for HT conditions, residual gas analysis, and N-doping were still shared.

Other essential conditions for surface cleaning through ultrasonic cleaning and high pressure water rinsing (HPR) were also shared with the partner labs and the vendor. All documents related to processing were reviewed by subject matter experts at the partner labs and Fermilab.

PRE-SERIES PRODUCTION CAVITY RESULTS

The surface processing of the first UKRI cavity, B92M-ZRI-301, has been completed at Zanon. I-V curves for B92M-ZRI-301 before EP were measured at 9 and 16 °C. The curves in Fig. 4 shows apparent EP plateaus with V_{onset} values of 10 and 13 V, respectively. These onset voltages align with the linear relation between onset voltage and EP current density obtained at Fermilab (see Fig. 4), validating that the Zanon facility can perform optimal EP within the plateau region. EP was conducted using the recommended V_{EP} that was at least 5 V higher than V_{onset}.

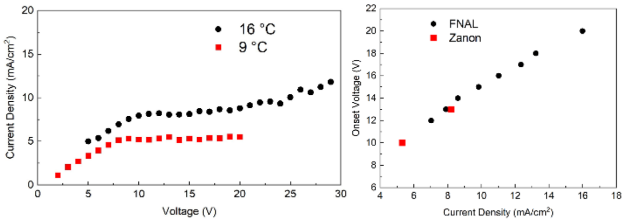

Figure 4: I-V curves measured with B92M-ZRI-301 cavity at Zanon (left). V_{onset} comparison with FNAL data (right).

The cavity B92M-ZRI-301 underwent bulk EP, N-doping, final EP, post-EP processing, and preparation for vertical test at Zanon, following the established protocols for each step. The cavity was shipped to Fermilab for a vertical test.

The cavity was tested at 2 K in the vertical cryostat at Fermilab. The test result (Q_0 vs E_{acc} curve) is shown in Fig. 5. The cavity exhibited excellent performance and met the cryomodule specification. The Q_0-E_{acc} curve of the jacketed B92F-RI-203 cavity processed at Fermilab is also shown in Fig. 5 for comparison. The cavity B92M-ZRI-301 has been jacketed at Zanon and will be tested at UKRI.

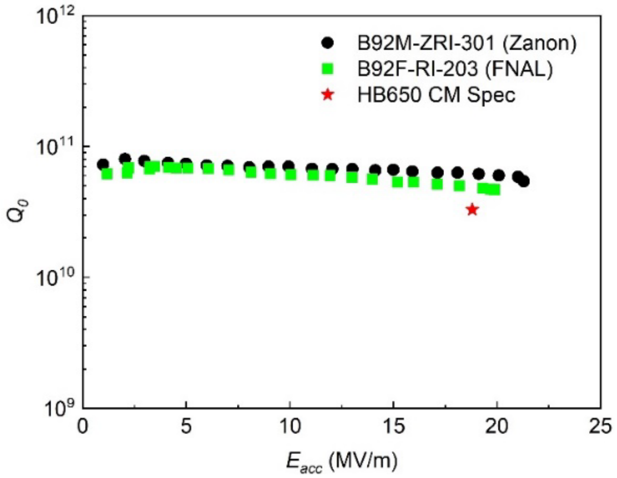

Figure 5: Q_0 versus E_{acc} curve measured for the UKRI's pre-series production cavity processed at Zanon. The result compares with performance of B92F-RI-203 jacketed cavity processed at Fermilab.

SUMMARY

This paper summarizes the lessons learned from EP R&D and N-doping studies and protocol set for surface processing of HB650 cavities. EP conditions, which differ from the standard EP conditions, were shared with the partner labs and successfully transferred to the vendor. The performance of the first UKRI-owned pre-series production cavity, processed by Zanon, was met the cryomodule specifications satisfactorily.

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REFERENCES

[1] C. E. Reece *et al.*, SRF2015, MOPB110, 2015, pp. 418.

[2] D. Gonnella *et al.*, SRF2019, FRCAA3, 2019, pp.1199.

[3] V. Chouhan *et al*., *Nucl. Instrum. Methods Phys. Res., Sect. A* 1051, 2023, pp. 168234.

[4] V. Chouhan *et al.*, SRF2023, TUPTB042, 2023, pp. 512.

[5] V. Chouhan *et al.*, SRF2023, WEIXA05, 2023, pp. 641.

[6] S. Posen *et al.*, *J. Appl. Phys.* 119, 2016, pp. 213903.

[7] D. Gonnella *et al.*, The LCLS-II HE high Q and gradient R&D program, SRF2019, Dresden, Germany, 2019, pp. 154.

[8] M. Martinello *et al.*, *J. Appl. Phys.* 130, 2021, pp. 174501.

[9] M. Martinello *et al.*, SRF2021, TUPCAV005, 2021, pp. 448.

[10] J. Holzbauer *et al.*, SRF2023, TUPTB047, 2023, pp. 536.

[11] V. Chouhan *et al.*, "Cathode structure for electropolishing of niobium elliptical superconducting cavities", U.S. Patent, Application no. 63/450,439, USA.