

# COMMISSIONING OF JLAB VERTICAL CAVITY PROCESSING SYSTEM FOR SRF Nb SINGLE CELL AND MULTI-CELL CAVITY WITH HF-FREE PULSE-REVERSED ELECTROPOLISHING\*

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## Abstract

Pulse reversed electropolishing of niobium SRF cavities, using a dilute aqueous H<sub>2</sub>SO<sub>4</sub> electrolyte without HF yields equivalent RF performance with traditional EP. Comparing with present EP process for Nb SRF cavity which uses 1:10 volume ratio of HF (49%) and H<sub>2</sub>SO<sub>4</sub> (98%), pulse reverse EP (also known as bipolar EP (BPEP)) is ecologically friendly and uses relatively benign electrolyte options for cavity processing. In this study, we report the commissioning of a new vertical cavity processing system for SRF Nb single cell and multi-cell cavities with HF-free pulse-reverse electropolishing at Jefferson Lab, together with RF test of cavities being processed. We report the scale-up challenges and interpretations from process R&D to implementation.

## INTRODUCTION

Chemical etching or electropolishing of niobium requires the removal of the passive Nb<sub>2</sub>O<sub>5</sub> film. Typically, an aggressive chemical, such as hydrofluoric acid (HF) is added to the chemical solution or electrolyte to accomplish this. The state-of-the-art in preparation of high gradient Nb SRF cavity surface for accelerator uses electropolishing [1]. In this process, an electrolyte of concentrated sulfuric acid (98%) and hydrofluoric acid (49%) by volume ratio 10:1 typically is used in combination with a constant voltage. The disadvantages for this process is not only the use of the hazardous concentrated acids, especially the presence of hydrofluoric acids presents considerable challenge in terms of worker safety and waste control, but also it is associated with the cost of the EP process system and associated labor burden which contribute a large budget cost for large accelerator projects [2].

As an alternative, in order to depassivate the surface, cathodic pulses may be interspersed within anodic pulses, in place of or in conjunction with off-times. Use of such cathodic pulses eliminates the need for HF and/or fluoride salts or other chemicals to remove the surface oxide [3–6]. As part of our mission is to advance the technology of producing SRF-based particle accelerators, in collaboration with Faraday Technology, Inc. and KEK, JLab has been developing HF-free EP of Nb cavities via pulse-reversed electropolishing using several different HF-free electrolytes. Faraday Technology, Inc. has

patented the process under the name FARDAYIC™ Pulse-Reverse ElectroPolishing [4]. In the SRF community it has more casually come to be known as bipolar EP (BPEP). Bench-scale process analytics at JLab have yielded a systematic way of characterizing the processes and are guiding application to single and multi-cell cavities [7,8]. Jefferson Lab has developed a low-cost pulse control technique and implemented it in a vertical EP processing system integrated into the JLab cleanroom suite. The polishing process dynamics learned from different electrolytes, the implementation in the vertical processing system and pulse controller, and the effectiveness in application to single cell SRF cavity will be reviewed.

## VERTICAL CAVITY PROCESSING SYSTEM WITH PULSE REVERSED ELECTROPOLISHING AT JLAB

The HF-free BPEP process system at Jefferson lab uses an inherited chemistry cabinet which was previously used for buffered chemical polishing (BCP). This closed chemistry cabinet provides not only the controllable electrolyte flow but also a big capacity reservoir (~70 gallons) with temperature control. See Figure 1.

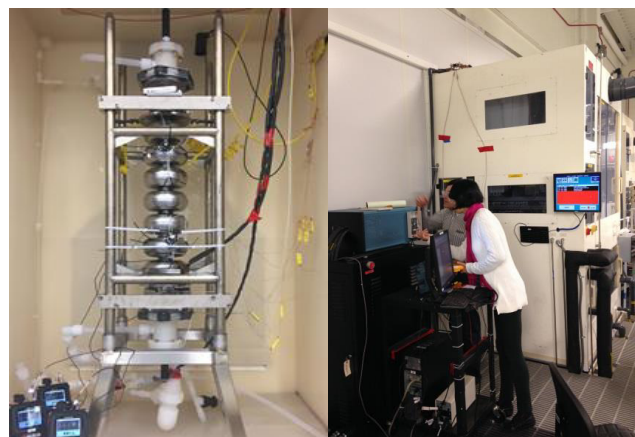


Figure 1: Jefferson Lab BPEP system.

The BPEP process system uses a Jefferson Lab custom-designed IGBT driver pulse controller and capacitor banks to enable two DC power supplies (AMETEK 40V, 250A) to provide adjustable and controllable positive and negative pulses, this technology solution is the subject of a recent patent application. Because of the large current transients, care is required to minimize lead inductance. A

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500 F capacitor bank is used to drive the cathodic pulse current. For safety, the cavity is held at ground potential while the counter electrode is alternately connected to differently polarized capacitor banks.

At present, this system has demonstrated the capability to process single cell and multi-cell Nb cavities. The Nb cavity is mounted on a vertical stand with a mixed-metal-oxide (MMO) coated Cu bar as the counter electrode inserted coaxially through the cavity. The in-situ removal of Nb was measured by Olympus 38DL PLUS ultrasonic thickness gages, which were mounted on beam tubes and equator of cavity. The temperature of the cavity at multiple positions was continually monitored.

### PROCESS PARAMETER STUDY FROM SAMPLES AND SINGLE CELL CAVITIES

The previous mechanistic studies leads to the understanding that BPEP works by cyclic oxide growth and removal by successive positive and negative pulses. For a given set of parameters, lengthening the negative (cathodic) pulse yields no additional removal [7, 8]. In addition, lengthening the positive pulse duration does not help the uniformity of the oxide layer when the anodization process finishes. Our studies revealed that higher anodic voltage simply helps the oxide layer grow thicker and more quickly; however, sustaining the cathodic pulse condition too long, after removing all of the oxide, risks the cathodic generation of hydrogen at the niobium surface driving stress corrosion cracking and hydrogen loading of the bulk [8]. Figure 2 shows that the removal rate increases with cathodic voltage, but it seems to reach to a maximum when the oxide is totally removed by each cathodic pulse. From this figure, we learned that the removal rate of Nb by BPEP increases with concentration of H<sub>2</sub>SO<sub>4</sub>, but there is little difference comparing between 10% H<sub>2</sub>SO<sub>4</sub> and 15% H<sub>2</sub>SO<sub>4</sub> for these parameters suggesting that higher concentration of H<sub>2</sub>SO<sub>4</sub> helps growth of oxide layer, which could be used for improving the efficiency of pulse reversed Nb EP process.

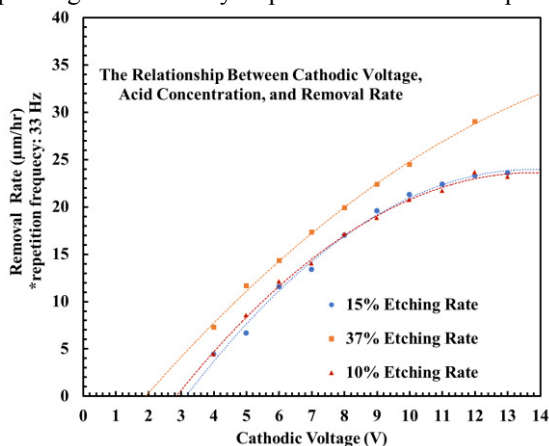


Figure 2: Sample studies reveal that removal rates depend on cathodic voltage and electrolyte concentration in interesting ways.

Applying this mechanistic understanding to the single cell/multi-cell Nb SRF cavity and commissioning the

custom designed pulse controller, different concentration H<sub>2</sub>SO<sub>4</sub> and different pulse structures have been studied for the single cell. The 37% sulfuric electrolyte (for maximum conductivity) leads to rapid removal rate compared with lower concentration sulfuric acid; however the high peak current is challenging for most pulsed power supplies, especially as we project to application on multi-cell cavities with surface areas of order 1 m<sup>2</sup> and more, which indicate potential current requirements of several thousand Amperes. For our initial development work, we use 10% sulfuric as the electrolyte, with 40 ms anodic pulse and 30 ms cathodic pulse, in between there is 20 ms off time.

Figure 3a shows the cathodic coulombs per pulse linearly increases with applied cathodic voltages with same applied anodic voltage and pulse structure. This is consistent with the cathodic current being fully controlled by simple hydrolysis on the surface of the niobium. The total current is determined by the applied potential and the current distribution flowing from counter electrode and resistively through the electrolyte to the cavity. Figure 3b shows the anodic coulomb increases non-linearly with applied anodic voltages with a given applied cathodic voltage and pulse structure. We will address elsewhere an integrated model that is emerging.

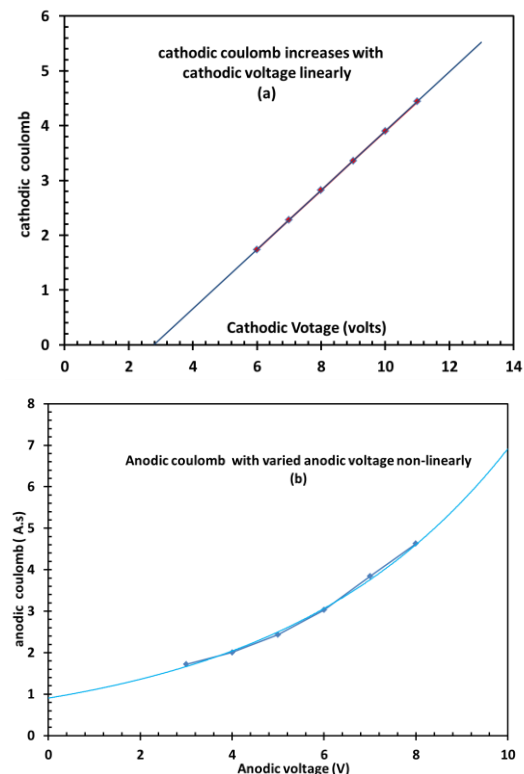


Figure 3: The (a) cathodic coulombs per pulse, and (b) anodic coulomb per pulse increases with applied voltages.

Our measurement for the thickness removal at different locations indicates with there is differential removal along the cavity; the beam tube removal is higher than at the equator when using an unmasked counter electrode. With 8 V cathodic pulse, 4 V anodic pulse, and 6 Hz repetition frequency, the removal rate was 1.3 µm/hr from the

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beamtube and 0.6  $\mu\text{m/hr}$  from the equator area. This differential may either be the result of non-uniform anodization during each pulse or non-uniform removal by each cathodic pulse.

When we intentionally masked the MMO counter electrode at all the beam tubes and kept the part of equator area exposed, the cathodic current and anodic current both decreased significantly, and the time profile of anodic current also changed. It is important to recognize that when the anodic current goes to zero, even while an anodic potential is yet applied, the cavity is considered uniformly anodized to a specific oxide thickness. The anodic current that flows each pulse represents the regrowth of the oxide removed during the intervening cathodic pulse, which need not be uniform.

The BPEP currents with the above parameters with masked and unmasked electrode are compared in Fig. 4. The measurement of the thickness removal at different locations showed improved uniformity of removal, but decreased the removal rate. With the same pulse profile, the removal rate was 0.3–0.4  $\mu\text{m/hr}$  at both beamtube and the equator areas.

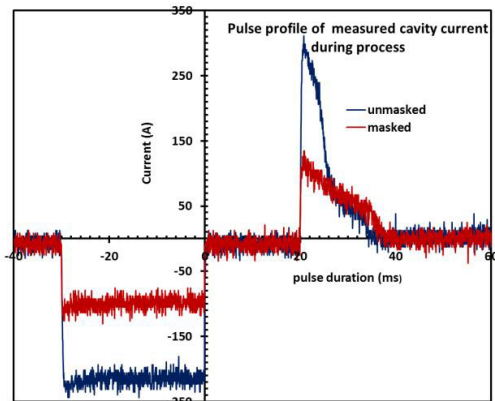


Figure 4: BPEP current for single-cell cavity processing, unmasked and masked counter electrode, 10%  $\text{H}_2\text{SO}_4$ .

### THE RF PERFORMANCE OF Nb CAVITIES PROCESSED BY HF-FREE PULSE REVERSED EP

Single cell cavity RDT-13 was the first cavity processed by BPEP and tested at Jefferson lab. RDT-13 had previously received standard EP treatment and performed well, the gradient reached to 32 MV/m. Testing was complicated by multipacting above 24 MV/m. This cavity was processed in the Jefferson Lab BPEP system, removing 27  $\mu\text{m}$  per differential thickness measurements at the equator area. Figure 5 illustrates that performance following BPEP was equivalent to conventional EP; the gradient reached to 25 MV/m, where the multipacting phenomenon inhibited further testing.

To explore whether HF free pulse reversed EP could be applicable to the light electropolishing that is used to remove surface nitrides following the application of 800°C “nitrogen doping”, the single cell cavity RDT-12

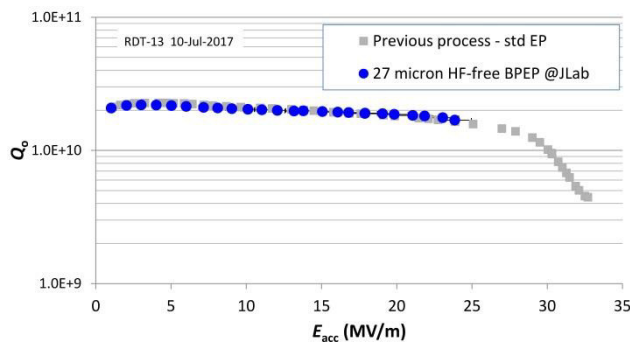


Figure 5: First BPEP-processed cavity at JLab, showing equivalent performance to standard EP.

was taken as a first candidate. This was a legacy cavity left over from early LCLS-II cavity process R&D. RDT-12 received a 40  $\mu\text{m}$  “reset” standard EP, followed by the presently standard 2N/6 800°C doping [9]. The cavity in this state was subjected to 7  $\mu\text{m}$  removal from the equator area with BPEP, using 10%  $\text{H}_2\text{SO}_4$  electrolyte. The subsequent RF performance is presented in Fig. 6, together with the cavity’s prior performance in 2014. While higher field performance continued to be limited by a fabrication flaw that degrades performance in the 8–11 MV/m range, the performance after fresh doping plus BPEP was clearly improved and the 1.6 K data confirm that the BPEP process contributes at most negligible residual resistance.

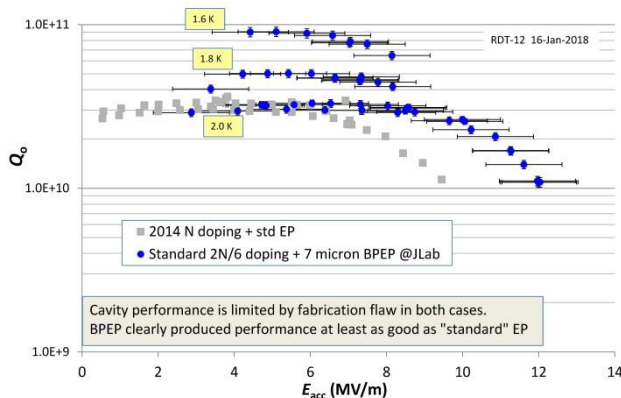


Figure 6: First BPEP processed “nitrogen doped” cavity.

### NEXT

In the next months we will transition to working with 7-cell and 9-cell cavities while continuing to refine process understanding and optimize removal rates and uniformity as we move this technology toward full deployment for use at JLab and then propagation in the broader SRF community.

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