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NEW PROGRESS WITH HF-FREE CHEMICAL FINISHING FOR Nb SRF CAVITIES

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Abstract

Jefferson Lab has implemented a bipolar pulsed electropolishing system for final chemical processing of niobium SRF cavities. This FARADAYIC bi-polar electropolishing (BPEP) has been applied to single cells, a 7-cell CEBAF C100 cavity, and to 9-cell TESLA-style cavities [1]. As a mechanistic characterization of the process emerges, the critical role played by the local current density during each cathodic pulse is becoming clear. This influences system and operational parameter refinement. We present current process parameters, removal characterization, and rf performance of the processed cavities. This is the fruit of collaborative work between Jefferson Lab and Faraday Technology, Inc. directed toward the routine commercialization and industrialization of niobium cavity processing. We also present supporting data from controlled-parameter coupon studies.

INTRODUCTION

Bi-polar pulsed electropolishing (BPEP), as an alternative method to treat Nb surfaces, uses anodic pulses to anodize the Nb surface with an HF-free electrolyte and intervening cathodic pulses to erode the oxide via mechanical action of the hydrogen gas bubbles formed at the niobium surface by electrolysis, resulting in a surface leveling polishing [1-3]. This process eliminates the need for HF and/or fluoride salts to remove the Nb surface oxides and is being studied as a safer, greener and more economic method to prepare Nb SRF cavities [4].

BPEP has been used as final chemical processing of niobium SRF cavity recently by Faraday Technology Inc. and Jefferson Lab. Similar work is also being investigated in partnership with KEK and Nomura Plating. The studies demonstrated that bi-polar pulsed electropolishing could effectively polish Nb SRF cavities, and produce a high-quality surface comparable with conventional EP which uses concentrated HF and H_2SO_4 as electrolyte, and provides comparable cavity performance [5-7]. However, optimization of parameters of the BPEP process for improving the removal rate and uniformity, and achieving highly reproducible high RF performance cavities, and utility processing nitrogen "doped" Nb cavities needs further systematic studies.

Jefferson Lab has developed a low cost BPEP control technique and implemented it in a vertical EP processing system. In this study, the systematic mechanistic and surface studies for guiding the design of the BPEP and the optimization of BPEP process for single cells are reported.

EXPERIMENTAL

Cavity processing at JLab by BPEP was performed inside of a closed chemical cabinet where the cavity is mounted on a vertical stand. The mixed metal oxide (MMO)-coated Ti-clad copper rod serving as a counter electrode was inserted coaxially through the cavity. Hydrogen and oxygen generated during BPEP flow back to the solution reservoir where they are diluted and exhausted. The cavity is set as the ground potential; the counter electrode is driven by a custom-designed pulse generator. Figure. 1 is the wiring diagram for the Jefferson Lab pulse generator and controller system, which is under a patent application [8]. Ultrasonic thickness gauges are used to monitor surface removal at different locations of single cell and multi-cell SRF cavities.

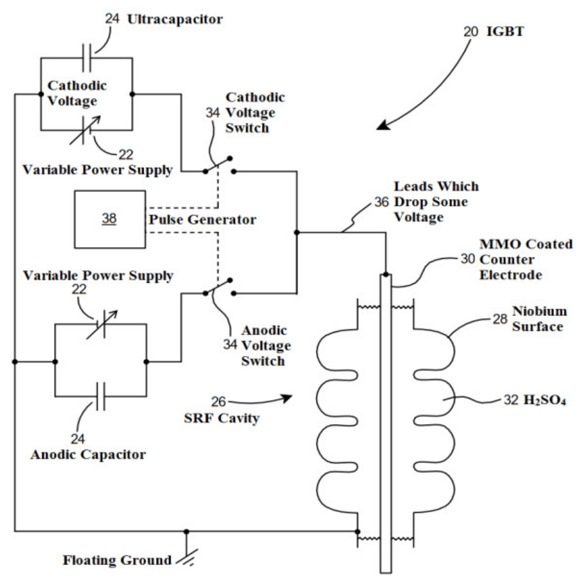

Figure 1: Schematic of the JLab BPEP equipment. [8]

Many standard samples made of high purity niobium with reactive surface of 3.14 cm^2 were fabricated for use exploring process parameters. The counter electrode for such bench-top tests was Ti-clad Cu plate with MMO coating and having reactive surface area of 30 cm^2. The distance between Nb sample and counter electrode was set as 9.8 cm. All the samples underwent 60 µm BCP etching before use. AFM measurements were done on 50 µm × 50 µm areas at three different locations, and the average of root-mean-square roughness (R_q) was calculated for each sample. The surface removal for samples was calculated through loss of mass.

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RESULTS AND DISCUSSION

Mechanistic Studies for Optimization of BPEP

The waveform of BPEP could be designed at certain repetition frequency and structure. In this sample study, the waveform consisted of 3 ms cathodic pulse, then 2 ms off time, the 3 ms anodic pulse, and another 2 ms off time, which results in 100 Hz repetition frequency. A set of 20 samples were treated by BPEP with 15% and 37% sulfuric acid with 4 V anodic voltages, varying cathodic voltage from 4 to 13 volts. A consistent 20 µm was removed from all samples. Our studies revealed that cathodic current density on Nb surface increases with the cathodic voltage, which results in the increase removal rate see Fig. 1 and 2. Each cathodic pulse only partially removes the oxide formed during the anodic pulse. The average surface removal by each pulse is less than 0.1 nm, which suggests that the hydrogen formed may strip off the porous oxide layer only at relatively sparse locations each cycle. See Fig. 3. These results also revealed that the net removal rate strongly depends on the cathodic current density and repetition frequency.

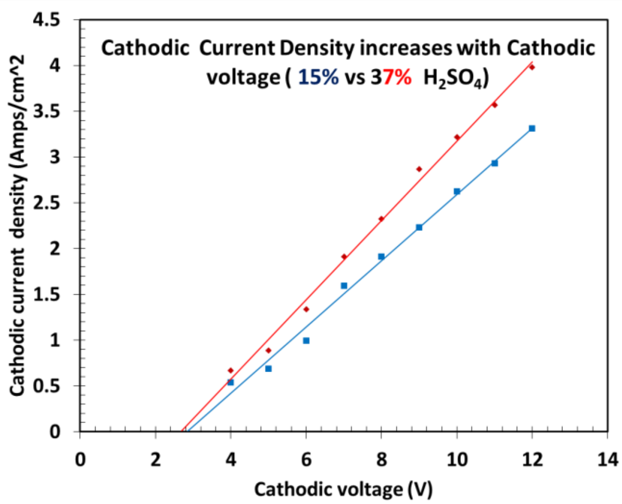

Figure 2: The cathodic current density increases with cathodic voltage for 15% and 37% H_2SO_4.

The surface roughness has been systematically studied for all the samples under different cathodic voltages with 15% and 37% sulfuric acid. See Fig. 4. The lower concentration H_2SO_4 (15%) resulted in a smoother surface than that of high concentrated H_2SO_4 (37%) at lower cathodic voltage.

Using a standard 10 V cathodic pulse with different anodic voltages of 4, 5, 6 and 7 V, our results show no dependence of removal rate or surface finish on anodic voltage.

Optimizing BPEP for Single Cell Cavities

Applying the understanding emerging from sample studies to single/multi cell SRF cavities to improve the removal rate for BPEP processing of cavities indicates that an optimization of repetition frequency and increase of cathodic current density is needed.

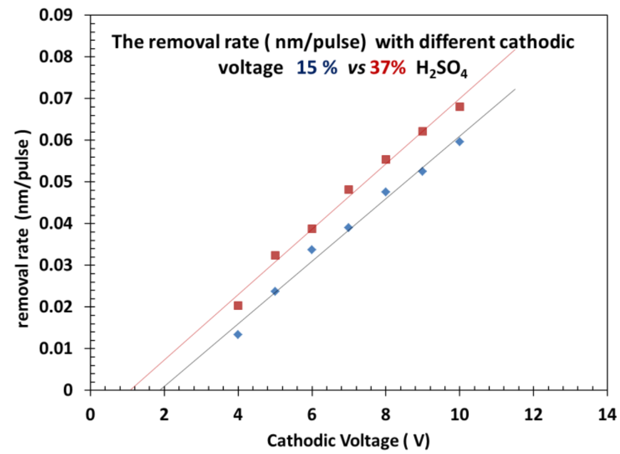

Figure 3: The average removal per pulse increases with cathodic voltage for 15% and 37% H_2SO_4.

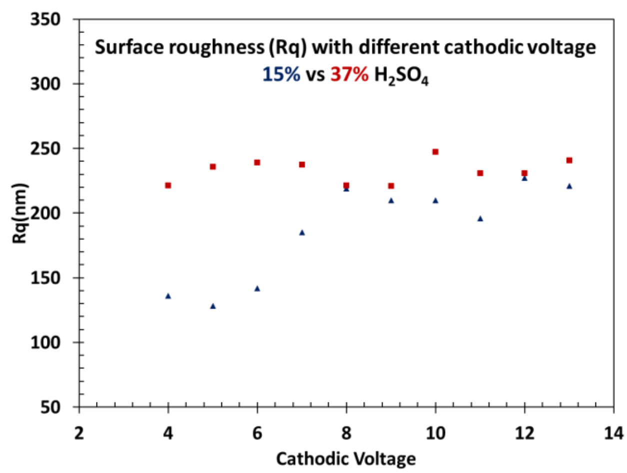

Figure 4: The surface roughness of samples processed by different cathodic voltage for 15% and 37% H_2SO_4.

A systematic study with different cathodic pulse length and voltage in 10% and 37% sulfuric acid was conducted to design an optimized parameter set a for single cell Nb SRF cavity. Figure 5 shows the BPEP current in 10% and 37% sulfuric acid with 4, 7 and 10 V cathodic voltages, with 4 V anodic voltages. The cathodic current density increased linearly with cathodic voltage, as with sample studies. Compared with 10% H_2SO_4, 37% H_2SO_4 not only increased the anodic peak, but also decreased cavity anodization process as a "shrinking" of the current anodic shoulder was observed. Besides, 37% H_2SO_4, which has near maximum electrical conductivity, yields a higher cathodic current at same cathodic voltage, yielding a higher removal rate.

Figure 6 compares the pulse current generated for different cathodic pulse lengths with 37% sulfuric acid. We observed that as the anodic current shoulder shrinks when cathodic pulses decreases to 20 ms and 15 ms there seems no effect on anodic current when comparing with 25 ms and 30 ms cathodic pulse, suggesting that cathodic pulse duration may affect the efficiency of oxide removal at each pulse.

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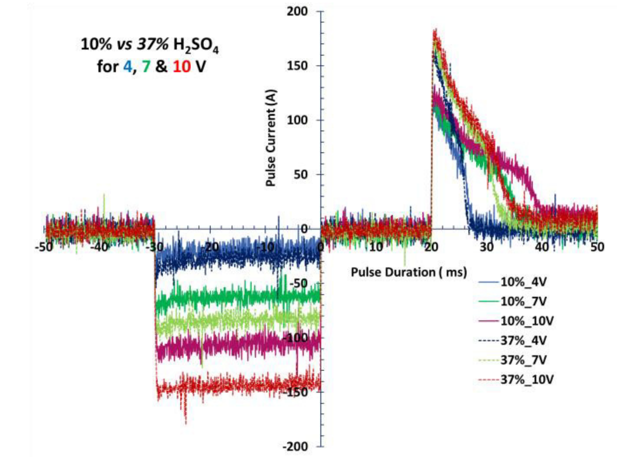

Figure 5: BPEP current with single cell cavity.

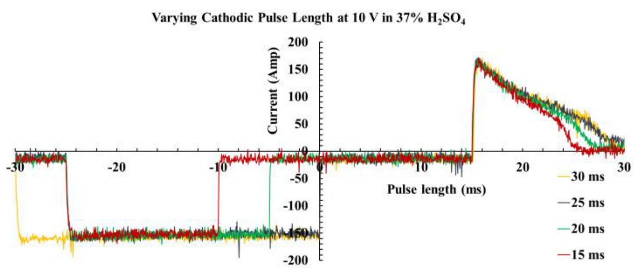

Figure 6: BPEP current with variation of cathodic pulse length.

Therefore, to maximize the removal rate and uniformity of removal for Nb SRF cavities with BPEP, the optimum repetition frequency will be influenced by cavity shape and the reactive surface area of the counter electrode.

Our previous studies noted that beam tube removal is higher than equator when using an unmasked counter electrode, and masking counter electrode at all the beam tubes improves the removal uniformity removal but decreases the removal rate [3].

In this study, a 1.3 GHz nitrogen-doped Nb single-cell was processed with 4 V anodic voltage and 10 V cathodic voltages provided by power supplies, and the counter electrode was masked at the beam tube. For BPEP process for polishing of cavities the voltage was set to a specific value by power supply, but due to voltage drop in the insulated-gate bipolar transistor (IGBT) and the leads, about 2~3 V is lost for cathodic pulse and about 1 V for anodic pulse. The cavity was polished with two parameters in 37% sulfuric acid, one with a 15 ms cathodic pulse and another with a 25 ms cathodic pulse while maintaining 11 Hz repetition frequency. The 15 ms pulse polished at the equator with removal rate of 0.6 µm/hr and 0.4 µm/hr at the beam tubes. The 25 ms pulse polished at the equator with the removal rate of 0.8 µm/hr, and polished the top beam tube with the removal rate of 0.2 µm/hr, and the bottom beam tube with the removal rate 0.6 µm/ hr.

A CEBAF C100 shape 7-cell was polished with 10% sulfuric acid at 6 Hz with a cathodic pulse of 8 V. These parameters were designed to test the capability of our pulse control system for multicell. Further optimization is underway for 7 cells. With masked counter electrode, this 7 cell was polished at a removal rate of 0.1 µm/ hr. The subsequent RF test was complicated by accidental electrode scratching of the cavity; hence RF test result is inconclusive and is not reported here.

The RF Performance of Nb 9 Cell Cavity Processed by BPEP Process

Both JLab and Faraday Tech seek to build experience with multicell cavities, but access has been limited. Under our CRADA, JLab sent Faraday the LCLS-II 9-cell cavity CAV238. Faraday BPEP processed the cavity using 10% H_2SO_4, removing an average of 13.5 µm by weight reduction then shipped it back to JLab for testing, see Fig. 7. The gradient was initially administratively limited by radiation production at 25 MV/m. (3 R/hr + neutron production) Operation in the 8/7/6/π modes to quench were consistent with ~32 MV/m quench field limited in an end cell. After a second HPR and 120°C bake, performance improved nicely. This result suggests that BEPE has no fundamental limit in obtaining a gradient in multi-cell cavities up to 32 MV/m.

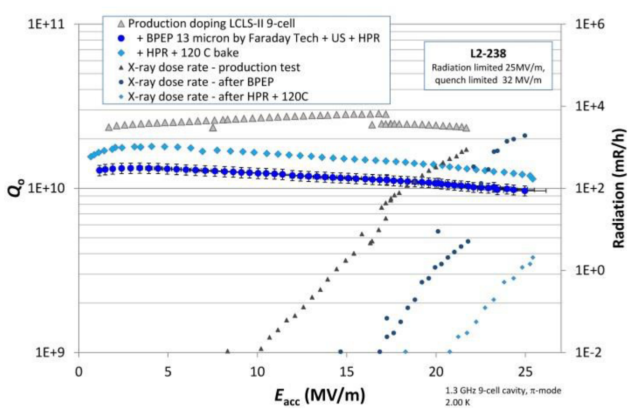

Figure 7: RF test of 9-cell cavity BPEP processed by Faraday.

NEXT

In the near future, we will continue to work with multi-cell cavities to refine process parameters, electrode geometry, and upgrade our pulse control system. We are also planning to start systematic studies of BPEP for N_2 doped samples and single cells.

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