

FERMILAB EP FACILITY IMPROVEMENTS*

F. Furuta[†], D. Bice, A.C. Crawford, T. Ring, Fermi National Accelerator Laboratory, Batavia, USA

Abstract

Electro-chemical Polishing (EP) is one of the key technologies of surface treatments for niobium superconducting cavities. Fermilab has established cold EP method and applied on all single cell cavities (1.3 GHz~3.9 GHz) processed at Fermilab EP facility. Cold EP method allows achieving continuous large current oscillations during EP process and providing the uniform removal over the cell with the variation of $\pm 15\%$. Here we report details of Fermilab EP facility and cold EP method in this paper.

INTRODUCTION

Horizontal EP on single cell cavities at Fermilab EP facility started in 2012. R&D efforts of EP had been paid on precise temperature control over EP process to achieve continuous large current oscillations during the process. In 2014, the efforts were established by maintaining the cell surface at low temperature of 16 C during EP process and named as “cold” EP method. The first article of cold EP method was published in 2017 [1]. So far, more than 200 EP with this cold EP method have been successfully applied on over 60 single cell cavities at Fermilab EP facility and contributed as one of important surface treatments to the cavities achieved high-Q and/or high field during vertical tests. The keys of cold EP method are 1) low concentration of water in the electrolyte, 2) low temperature at the cell surface, and 3) low electrolyte flow rate. In this paper, we will revisit those key factors and describe the details of Fermilab EP facility and current cold EP parameters.

EP TOOL AT FERMILAB

Figure 1 shows EP tool at Fermilab EP facility, CPL (Cavity Processing Laboratory), and 1.3 GHz TESLA shape single cell cavity on the tool. The tool is capable for single cell cavities with frequency of 1.3 GHz or higher. So far, 1.3 GHz, 2.6 GHz, 3 GHz, and 3.9 GHz single cell cavities have been processed on this tool. Fermilab EP process took 2 or 3 days. Day1 was for a cavity installation and instrumentation. Day2 was leak check and EP process. We had Day3 depends on the target removal. The leak check of the tool was performed using sulfuric acid and this also helped to remove all residual waters in the tool.

Electrolyte

EP electrolyte is the mixtures of sulfuric acid (H_2SO_4) and hydrofluoric acid (HF). Traditional ratio of EP electrolyte is $H_2SO_4 : HF = 9\sim 10 : 1$ by volume, and uses $H_2SO_4 > 96\%$ and HF 46~48% by weight. Fermilab uses

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[†] ffuruta@fnal.gov

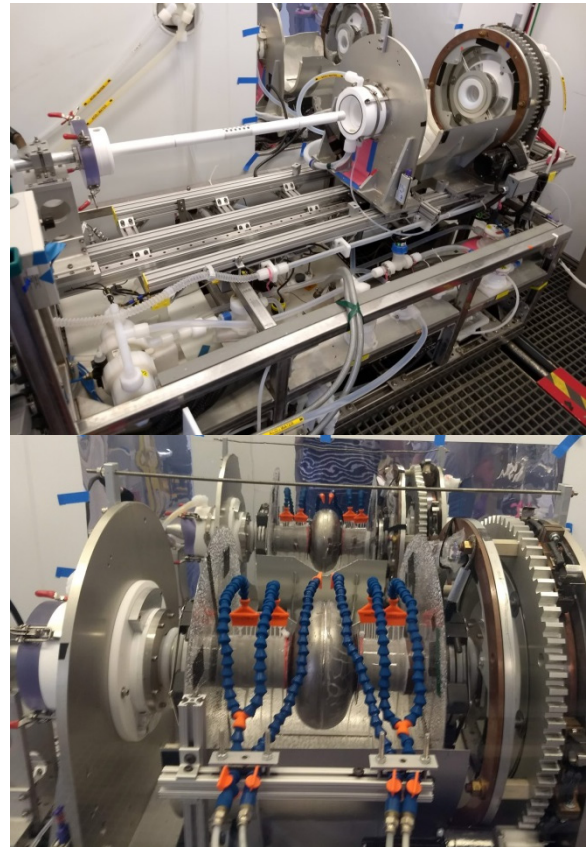


Figure 1: FNAL EP tool (top) and 1.3GHz single cell cavity on the tool (bottom).

EP electrolyte with the ratio of $H_2SO_4 : HF = 13.5 : 1$ by volume. Concentration of each acid Fermilab uses are $H_2SO_4 > 96\%$ and HF $\sim 70\%$ by weight. The ratio of HF to H_2SO_4 is similar to traditional mixtures, but the high concentration of HF provides the first key of cold EP, low concentration of water in the electrolyte, and also minimizes HF evaporation during acid mixing. EP electrolyte was pre-mixed by the company and delivered to Fermilab (~ 110 L/drum). For EP process, a fresh electrolyte of 10L was transferred from the EP electrolyte drum to the acid tank of EP tool. This 10L of electrolyte was circulated during EP process and dumped to the waste drum after the process, no electrolyte was re-used even if the removal was small. The administrative removal limit with 10L of electrolyte is $80\mu m$ on 1.3GHz single cell cavity; this corresponds to the niobium concentration of about 11g/L.

Temperature Control and Monitoring

Temperatures during EP process were monitored at six locations; three thermocouples with insulator are on the cavity (Fig. 2), another two with insulators are on the drain lines of EP end groups at both cavity ends, and the last one is in the acid tank. Wireless thermocouple system is applied on the rotating cavity and the drain lines.

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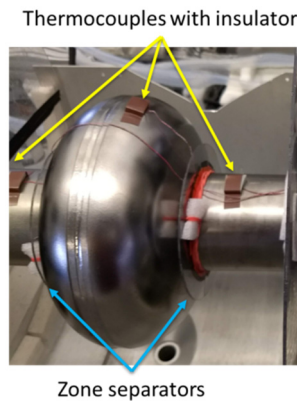


Figure 2: Thermocouples and zone separators on the cavity.

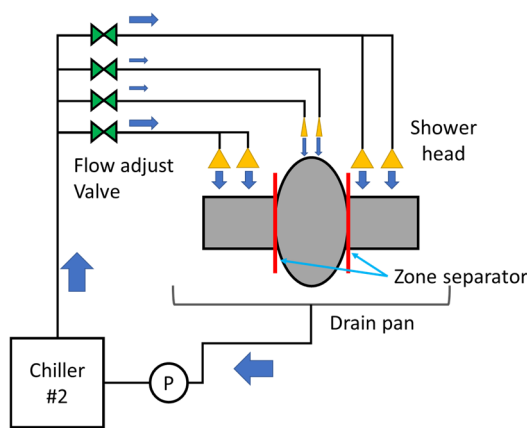


Figure 3: Schematic of cavity outside cooling.

Figure 3 shows the schematic of cavity outside cooling at Fermilab. The chiller unit #2 controls the temperature of cavity outside wall. The 30% propylene glycol is used for the chiller #2 and could provide the outside cooling water of below 0 C without freezing. Zone separators, flexible plastic disks seen on iris in Fig. 2, divide a single cell cavity in three zones (the cavity cell and the beam tubes) and prevent outside cooling water moves from one zone to another. Flow adjusting valves control the amount of cooling water to each zone. With these schemes, we could maintain the cavity cell and the beam tubes in different and low temperatures during EP process (the second key of cold EP). The temperature of electrolyte in the acid tank was maintained below 12C with the chiller unit #1 during cold EP process.

Current Oscillations

EP reaction can be described in two parts; 1) electrochemical reaction develops niobium pentoxide (Nb_2O_5) on niobium surface by applying voltage between cathode (aluminium, >99.5%) and anode (niobium cavity), this part was responsible for decreasing EP current, 2) HF acid removes those niobium oxide layer, this part was responsible for increasing EP current. These two reactions happen simultaneously. The viscous dielectric layer of

niobium salt was produced on niobium surface during EP process and affected to the current behaviours. The horizontal rotational EP established by KEK had succeeded to maintain continuous current oscillations during EP by agitating the viscous layer with cavity rotation and also by circulating the electrolyte [2]. In addition to those baselines of horizontal EP method, Fermilab applied the low temperature conditions during EP process and succeeded to maintain current oscillations much larger and deeper than traditional EP conditions. The low temperature of the cavity outside at the cell surface (~15 C) and the beam tubes (~0 C) brought on less heating of EP reactions and allowed lowering the electrolyte flow rate. This low flow rate also contributed to maintain the agitation in good condition for continuous large current oscillations (the third key of cold EP).

Removal Rate during Cold EP

The thickness measurement by ultrasonic thickness gauge was performed before and after the bulk cold EP of 30 μ m. The current integration was about $2.1 \times 10^5 A \cdot sec$, so average removal was 30 μ m if we assumed the uniform removal over the cavity. The actual average removal over the cell was $34 \pm 3.5 \mu$ m, this demonstrated cold EP method could provide good removal uniformity over the cell with the variation of $\pm 15\%$. The removal at beam tube was about 20 μ m, well suppressed due to the lower temperature at beam tubes. Based on these thickness measurements, the target current integration values were calibrated to the target removals at cell. If the target removal was 10 μ m or less, the whole process of EP was done by cold EP method, but the removal rate during cold EP was small, about 5 μ m/hour over the cell, due to the low temperatures. If the target removal was larger than 10 μ m, hot EP conditions (Table 1) were applied first to make removal rate larger and then cold EP conditions was applied during the final 10 μ m removal. As an example, if the target removal was 120 μ m, the first 110 μ m was completed with hot EP conditions, and the final 10 μ m was completed with cold EP conditions.

Table 1: EP Parameters

Parameter	Cold EP	Hot EP
Target removal	10 μ m or less	>10 μ m
EP Voltage	18 V	
EP Current ^{*1}	15 [A]	40 [A]
Equator temp. ^{*1}	15 [C]	32 [C]
Beam tube temp. ^{*1}	0 [C]	5 [C]
Electrolyte temp. ^{*1,2}	12 [C]	20 [C]
Removal rate at the cell ^{*1}	5 [μ /hr]	12 [μ /hr]
Electrolyte flow rate ^{*1}	1.5~2.3 [L/min.]	
Cavity rotation speed	1 [Revolution/min.]	
Nitrogen gas flow	1 [L/min.]	

*1: average values, *2: at the acid tank of EP tool

Cold EP Parameters and Profiles

Table 1 summarizes the optimized cold and hot EP conditions for 1.3 GHz single cell cavity processed by Fermilab EP tool. The numbers are average values. Figure 4 shows typical current oscillation profiles during cold and hot EP on 1.3 GHz single cell cavity. X axis in Fig. 4 shows time in 5sec/div and Y axis shows current in 200 mV/div, 200 mV corresponds to 8 A, and the bottom X axis line corresponds to current=0. Figure 5 and 6 shows the temperature profiles of cavity wall and acid drain lines and the profiles of EP current, voltage, flow rate, and electrolyte temperature at acid tank during typical 40 μm EP on 1.3 GHz single cell cavity, respectively. As described before, the first 30 μm was done with hot EP conditions and the final 10 μm was done with cold EP conditions.

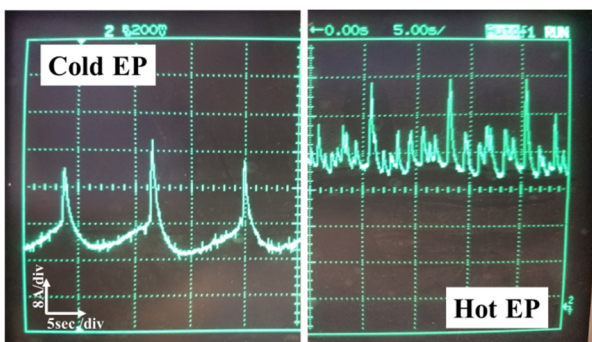


Figure 4: Current oscillation profile during Cold (left) and Hot (right) EP.

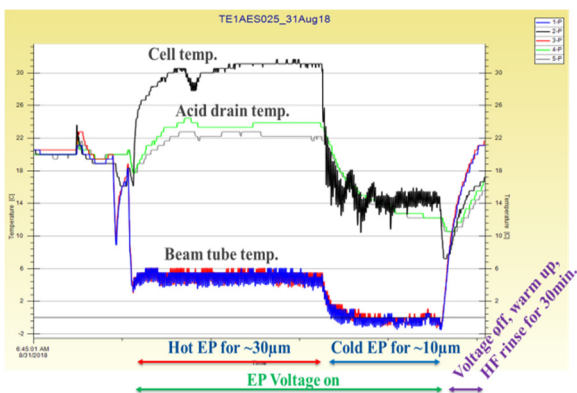


Figure 5: Typical temperature profiles during 40 μm EP on 1.3 GHz single cell cavity.

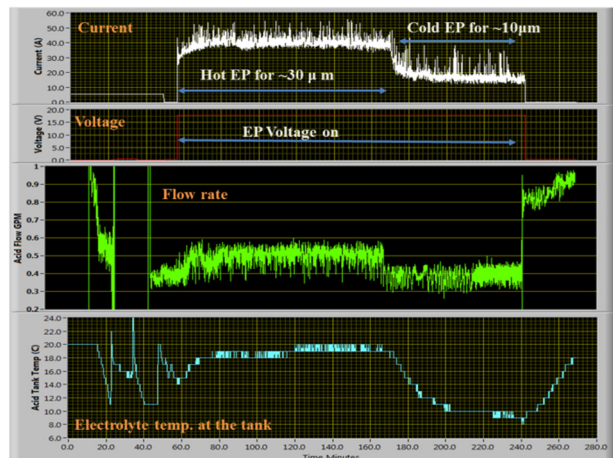


Figure 6: Typical EP profiles during 40 μm EP on 1.3 GHz single cell cavity.

FUTURE PLANS

Figure 7 shows Large EP tool at Fermilab CPL which was transferred from AES to Fermilab and waiting for re-installation. This large EP tool could process from single cell cavities to multi-cell cavities, such as 1.3 GHz 9-cell cavities and 650 MHz 5-cell cavities. Cold EP method will be extended to this large scale EP tool and applied to those multi-cell cavities.



Figure 7: Large EP tool at FNAL CPL (left) and a dummy tube installed for flow test on EP bed (right).

SUMMARY

More than 200 EP on over 60 single cell cavities had been successfully performed using cold EP method at Fermilab. Cold EP method is one of important surface treatments of Fermilab to achieve high-Q and/or high field performances with single cell cavities during cryogenic tests. Uniform removal over the cell with the variation of $\pm 15\%$ had been achieved by cold EP method, this is especially important on the final removal for doped-cavities. Applying Fermilab cold EP method on 9-cell cavities at ANL EP facility is in progress. The re-built work on Large EP tool at Fermilab CPL for 1.3 GHz 9-cell and 650 MHz 5-cell is also under discussions.

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