

# ELECTROPOLISHING ON SMALL SAMPLES AT FERMILAB\*

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## Abstract

The electropolishing process (EP) is considered an essential step in the processing of high gradient SRF cavities. Studies on EP of small samples has been started at Fermilab as part of the SRF materials R&D program.

A simple bench top setup was developed to understand the basic variables affecting the EP. In addition a setup for vertical EP of half cells, based on the Cornell design, was used and another one for dumbbells was designed and tested. Results and findings are reported.

## ELECTROPOLISHING OF NIOBIUM

In the past four years, most of the laboratories and universities involved in R&D programs for the production of high gradient superconducting RF cavities substituted the standard buffered chemical polishing (BCP) process with EP [1,2].

At the moment the introduction of EP is considered the essential step toward the production of over 35 MV/m superconducting RF cavities.

The main improvement of the cavity performance associated to EP is the reduction of the material surface roughness.

Whereas during BCP the chemical reaction mostly happens at the grain boundaries resulting in an enhancement of the material topological features, during EP the electrochemical process smoothes these features resulting in a reduction of the overall surface roughness of the material.

The use of EP has an impact, not only on the performances of cavities, but also on the overall processing sequence that has to be modified, especially those steps involving the chemical treatment of dressed cavities.

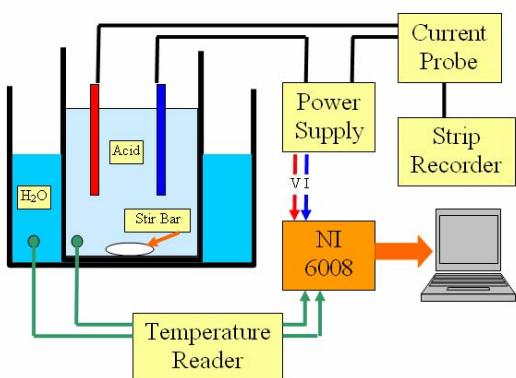


Figure 1. EP electrolytic cell and data acquisition scheme.

While most of the institutions involved in high gradient cavities production are in the advanced process of debugging their EP facilities, at Fermilab we are in the phase of commissioning the FNAL-ANL BCP joint facility.

Meanwhile a program of EP studies on small samples has been launched. The goal of the program is to understand the relevant parameters involved in EP, to understand the technological problems related to this approach and the familiarization with the process itself. After introducing the EP experimental setups developed and used at FNAL, this paper describes a comparative surface finish study on EP and BCP treated samples and a preliminary study on the uniformity of the material removal by EP in half cells.

## EXPERIMENTAL SETUP

Performing the EP on multi cell cavities is a complex task which requires expensive infrastructure [3]. In order to understand the process and evaluate the influence of each parameter it is necessary to work with bench top setups. Moreover, the smaller amount of acid involved and the limited amount of hydrogen generated during the process allows to safely perform the electrochemical etching under a fume hood in the laboratory environment.

Three separate setups have been developed at Fermilab to electropolish: flat samples, half cells and dumbbells; but all of them share the same instrumentation and data acquisition system. The basic setup consists of an electrolytic cell composed of an aluminum cathode and a niobium anode (sample) immersed in a 10:1 mix in volume of  $H_2SO_4$  (96%wt) and HF (49%wt) [4]. The optimal temperature of the process is between 30 and 40 C and, since the reaction is exothermic, cooling must be provided. Given the small amount of acid involved an ice bath in the secondary containment and temperature monitoring using two thermocouples, one in the acid bath

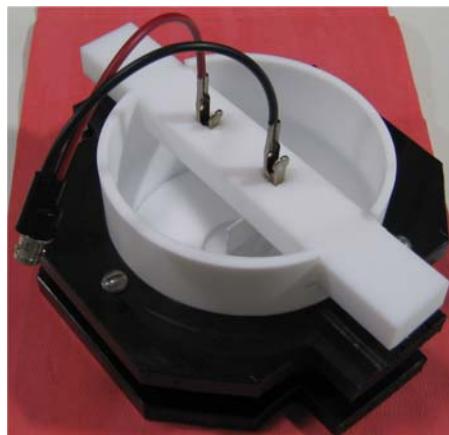


Figure 2. Flat samples setup.

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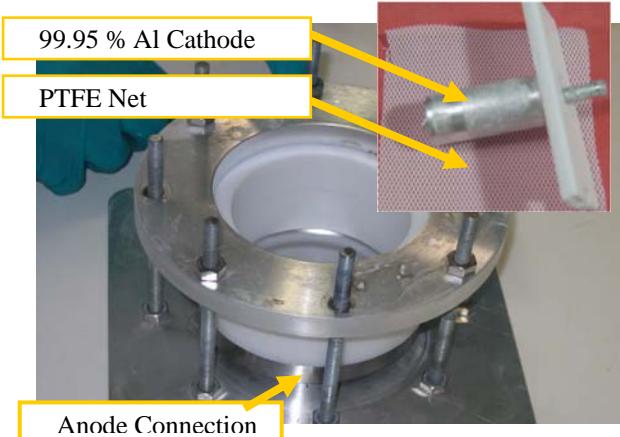


Figure 3. Half cells setup.

and another in the cooling water is sufficient. The EP process can be performed either controlling the voltage or the current.

At Fermilab, for now, we decided to adopt the controlled voltage technique.

#### Instrumentation and data acquisition

The necessary power to activate the electrochemical process is provided by an Agilent series 6000 power supply while a non contact probe is used to monitor the current flow to the electrodes. The current is recorded by a strip chart reader. The data acquisition system consists of a NI USB DAQ and a Labview application which is logging: current, voltage and the temperatures of acid and water. The Labview application, besides storing the data into a laptop, allows to visualize in real time: the current versus time, the current voltage characteristic and the derivative of the current-voltage characteristic versus voltage. These features allow adjusting the parameters during the process resulting in an overall optimized control of the EP [5].

#### Flat samples setup

This setup, shown in Fig. 2, is designed to perform EP on 2" square Nb samples. The Nb anode and the pure aluminum cathode of the same dimensions are connected to the electrical circuit through two alligator clamps which also serve as mechanical support by fixing the electrodes to a PTFE bar which is suspended on top of a PTFE beaker filled with acid. The beaker is placed into a PVC secondary container filled with cold water. The secondary container itself is placed on top of a magnetic stirring table. During the process the acid is mixed using a PTFE coated magnetic stir bar. The cathode is surrounded by a PTFE mesh that blocks the hydrogen bubbles from reaching the Nb surface. The goal for this setup is to understand the influence of: stirring, temperature and voltage-current ratio.

#### Half cells setup

This setup, shown in Fig. 3, is designed to perform EP on the deflecting mode and accelerating mode 3.9 GHz

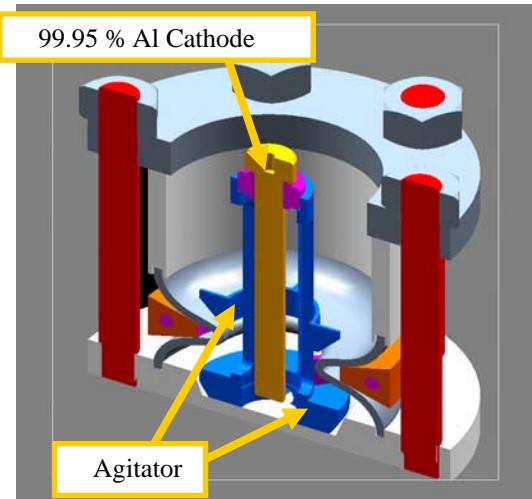


Figure 4. Dumbbells setup

half cells produced at Fermilab and is based on a Cornell prototype [6, 7]. Compressed between a PTFE tube (dam) and a PTFE disk, the half cell acts at the same time as sample and as acid container. The dam must be high enough to accommodate for the foam generated during the process to avoid air-acid interface defects on the sample. The aluminum cathode is suspended from the top and is cylindrical. The setup is placed into a PVC secondary container filled with water and placed on top of a magnetic stirring table. The acid stirring is performed using a PTFE coated magnetic stir bar as in the previous case. The goal for this setup is to understand the etching rate distribution in vertical configuration.

#### Dumbbells setup

This setup, shown in Fig. 4, is designed to perform EP on the deflecting mode and accelerating mode 3.9 GHz dumbbells produced at Fermilab. The setup is very similar to the half cells one, but with different options for the acid agitation. One solution, shown in Fig.4, uses the magnetic stirring table and a PTFE stir bar embedded in an agitator; the other uses a small DC motor to activate the agitator. The goal of this setup is to understand if it is possible to individually electropolish dumbbells before welding the full cavity. This technique would allow avoiding the deep chemical process of the full cavity before the heat treatment.

## CURRENT VOLTAGE CHARACTERISTIC

Due to the high number of variables involved in the fabrication and conditioning of the cavities, it is not obvious how to establish a correlation between surface finish and cavity performance; in general the smoother the surface the higher is the probability to obtain a high performing cavity. At a given temperature, the electrochemical process must be tweaked to the optimal V-I combination in order to obtain the best surface finish in the electropolishing range. The optimal working condition for the electrolytic cell described above is in the region of 10 to 15 volts with temperature ranging between 30 and 40 C.

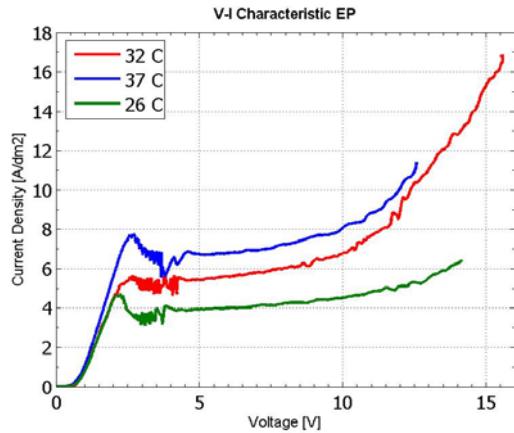


Figure 5. Voltage-Current characteristic.

The peculiar characteristic of this region is the oscillation of current with a frequency between 0.3 and 0.6 Hz (in the FNAL half cell setup). The oscillations are associated to the formation and disruption of the viscous layer in which the electrochemical reaction takes place and are strongly dependent on the acid temperature, the geometry, and acid agitation speed. In some cases the oscillations are not present, but the Nb surface roughness at the end of the process is very small. This phenomenon may be related to possible equilibrium thicknesses in the evolution of the viscous layer, in particular at slow stirring. During the EP process the V-I curve changes as a function of temperature needing adjustments of the working parameters (voltage or stirring). The best results have been obtained applying a voltage of  $\sim 12.5$  V at 30 C with constant current oscillations for 40 minutes. Typical V-I curves are shown in Fig. 5.

## ETCHING RATE DISTRIBUTION

Acid agitation is necessary during both BCP and EP to keep a stable material removal in time, but the high viscosity of the acid mixes used in both processes raises the problem of differential etching in different areas due to the cavity geometry. During the horizontal EP the cavity rotation assures an almost uniform result both at iris and equator in particular if the cathode is masked in the iris region.

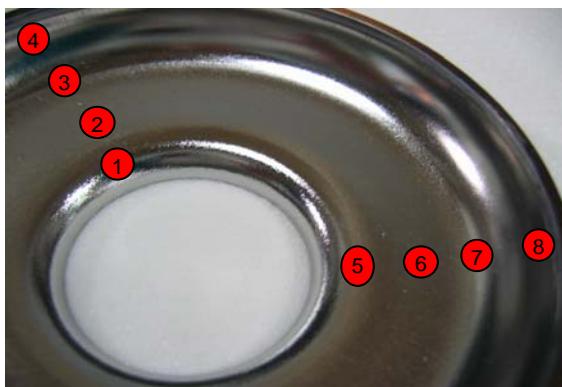


Figure 6. Thickness measurements point on half cell.

Point	Initial [mm]	EP 1 [ $\mu\text{m}$ ]	EP 2 [ $\mu\text{m}$ ]
1	2.734	13	15
2	2.739	10	15
3	2.681	5	8
4	2.676	15	15
5	2.726	10	18
6	2.737	10	13
7	2.668	8	18
8	2.655	13	10

Table 1. Niobium removal during EP on half cells. In the second column is shown the initial thickness while in the third and forth are listed the variations in thickness due to the two successive EP.

During vertical EP the acid mixing is obtained using an agitator placed in the iris area. This technical solution potentially may generate differential etching between irises and equators. Measurements of the material removal in eight points along 2 orthogonal radii of a 3.9 GHz cavity, Fig. 6, were performed using an ultrasound thickness measurement device before and after two successive EP sessions. The results, Tab. 1, show that the material removal data scatter around a rather uniform etching rate in all points except 3 and 7.

Sample #	Step	$R_a$ ( $\mu\text{m}$ )	Etch ( $\mu\text{m}$ )
<b>A</b>	RAW	0.79	-
	EP	0.31	77
	Heat Treat.	0.31	-
	1st BCP	0.67	23
	2nd BCP	1.06	25
	3rd BCP	1.26	25
<b>B</b>	RAW	0.83	-
	EP	0.48	57
	Heat Treat.	0.45	-
	1st BCP	0.63	26
	2nd BCP	0.95	29
	3rd BCP	1.27	24
<b>C</b>	BCP	1.8134	191
	1st EP		146
	2nd EP		59
	3rd EP	0.60	26

Table 2. Comparison of surface roughness  $R_a$  between samples that received an initial deep EP and later BCP treated in steps and a sample that received a deep BCP and later EP treated in steps.

## BCP -EP COMPARISON

The introduction of EP resulted in a significant modification of the cavity processing sequence. In particular the final chemical etching of a dressed cavity is the most significant step to switch to EP. Since this operation involves the removal of only 20  $\mu\text{m}$ , this step could be replaced by the simpler BCP if the resulting surface roughness is acceptable. In order to investigate

this issue, a small study was performed on three samples. One of them (C) underwent a strong BCP, heat treatment and light EP in successive steps; the other two (A and B) underwent a strong EP, heat treatment and light BCP in successive steps. At each step the roughness of the samples was measured using a Taylor Hobson Talysurf Intra device equipped with a 2  $\mu\text{m}$  radius diamond chisel. The resulting Table 2 shows that to restore the original surface roughness of a Nb sample after a BCP treatment of  $\sim 200 \mu\text{m}$  it is necessary to perform a  $\sim 220 \mu\text{m}$  EP. The tests on samples A and B demonstrated that the final light etching of 20  $\mu\text{m}$  can be performed via BCP without compromising the surface smoothness of the material as obtained after the initial EP.

## FUTURE PLANS

The main goal of this work was to familiarize us with the EP process and to better understand its governing parameters.

In addition we started investigating the possibility of performing the initial deep material removal by EP on individual dumbbells before welding them into the final cavity assembly. If the cavity performances are preserved, this technique could be a cost effective alternative for the cavity processing. The setup to perform EP on dumbbells is now ready and the future plan is to produce a 3 cells 3.9 GHz cavity with this technique.

## AKNOWLEDGEMENTS

The authors are very thankful to H. Edwards and H. Carter for the support of this activity and to D. Hicks and D. Burke for the work performed at the FNAL TD MDTL.

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