

# SINGLE CELL ELECTRO-POLISHING AT CEA SACLAY: FIRST RESULTS\*

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## Abstract

The goal of the Work Package 5.1 of the CARE SRF program is to study electro-polishing (EP) of niobium and thus isolate an ideal set of parameters for 1.3 GHz single cell cavity treatment that should be applied to industry. The more challenging aspect concerns acid composition's optimisation, made of a mixture of hydrofluoric (HF) and sulfuric ( $H_2SO_4$ ) acids with possible additional water. Preliminary experiments have been carried out on samples to guide the program on single-cell cavities. In that way, a set-up for horizontal EP has been installed in our laboratory.

This set-up has been successfully qualified with the standard recipe used at DESY since the first EP on TESLA-shaped cavity has reached an excellent gradient above 42 MV/m. The search for new parameters makes then sense. Different configurations have been investigated: higher temperature, lower voltage and new acids' concentrations. Dummy cavities have been electro-polished with a new mixture:

1vol HF(40w%) – 2vol  $H_2SO_4$ (95w%) – 7vol  $H_2O$ ,  
(1-2-7) with high water content.

Different cavities have also been electro-polished and results including surface aspect, RF measurements and impurities forming will be related.

## INTRODUCTION

Electro-polishing is believed to be most desirable final surface treatment for large scale 1.3 GHz cavity production related to projects such as the International Linear Collider.

A general set of parameter is used worldwide (same acid composition, voltage configuration, cavity rotation etc.) Precious practical results were given concerning this set of parameters by KEK [1]. However, some aspects could be ideally improved (acids are concentrated, high voltage is used, impurities are formed, etc.).

Is it possible with a mixture made of hydrofluoric, sulfuric and water to highlight new favorable configurations? As EP on cavity is long and fastidious, a limited number of experiments can be tested. The spadework on the subject has been done by a campaign of measures on samples [2,3] where promising sets of parameters have been isolated.

We are now able to test these parameters on single-cell cavity, since an EP set-up has been installed in our laboratory. First results with standard parameters will be presented with very promising gradients after ethanol rinsing.

Alternative parameters (lower voltage, lower acid concentration) have also been tested.

## EXPERIMENTAL SET-UP

The set-up (see Figure 1) is installed under a ventilated cabinet for safety reasons. Acid is stored in a PVCC tank and circulates via PFA piping and pumps. Acid is cooled thanks to a heat exchanger in PVDF. A constant nitrogen flow is maintained in the tank and in the cavity during EP to dilute hydrogen formed during the process.

EP is carried out at constant voltage and intensity as well as temperature in both acid tank and cavity are monitored. Acid is injected in the cavity through a hole in the middle of the cathode to the bottom. We used no Teflon net around the aluminum cathode for experiments described in this paper.

Furthermore, we use scrubbers filled with Bromo Thymol Blue, and we have a way to roughly evaluate quantities of degassed acid.



Figure 1: Electro-polishing set-up at CEA Saclay.

## QUALIFICATION OF THE SET-UP

Before starting tests with new sets of parameters, we need a 'reference' point with adequate performance after EP at the standard process. In that way, we electro-

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polished 1DE1 cavity (Tesla Shape) with gradient  $E_{acc}$  of 36.8MV/m before treatment at Saclay.

We used DESY parameters [4] with acid at 30°C and 17V voltage. The mixture used is the standard “1-9” (temperature and intensity monitoring during treatment are given in Figure 2): 1 volume of HF(40w%) for 9 volumes of H<sub>2</sub>SO<sub>4</sub>(95w%). The cavity was rinsed with ethanol, as a cure to possible sulfur deposition [5] before a High Pressure Rinsing in clean room.

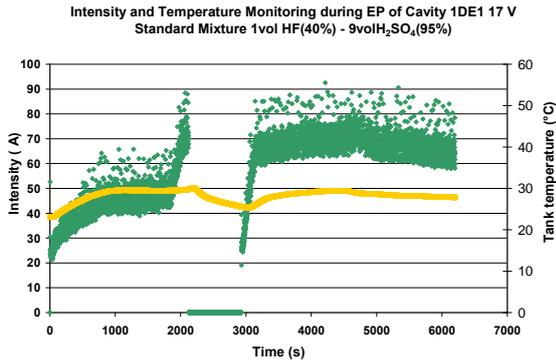


Figure 2: Intensity and temperature monitoring during EP of 1DE1 cavity. We can notice the characteristic current oscillations at high voltage.

The cavity was then sent to DESY where it was baked (125°C 48h) and tested. It reached the excellent gradient of 42.7MV/m at 1.8K (see Figure 3) compared to previous measurements. Red and dark blue curves correspond to tests after baking; light blue and orange ones, before.

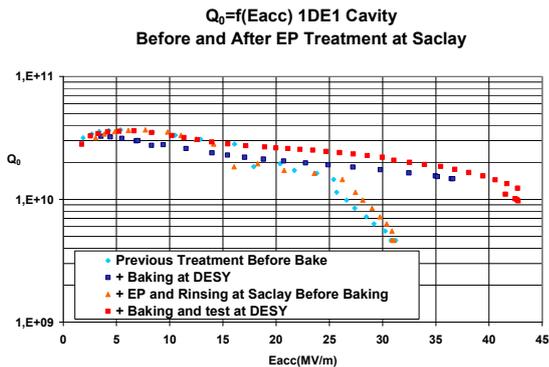


Figure 3:  $Q_0$  Vs  $E_{acc}$  curves for 1DE1 cavity before and after 42  $\mu$ m EP. After baking (125°C 48h) and tests at DESY, the gradient reaches 42.7 MV/m at 1.8K.

### EVALUATION OF ETHANOL RINSING

After this encouraging preliminary result we went on electro-polishing cavities (1DE1 and 1DE3 from DESY) with the same procedure: ~30 $\mu$ m EP + Ethanol Rinsing (30 min) + High Pressure Rinsing. Each cavity was treated one time and different RF tests took place at DESY and Saclay. Results are presented in Figure 4.

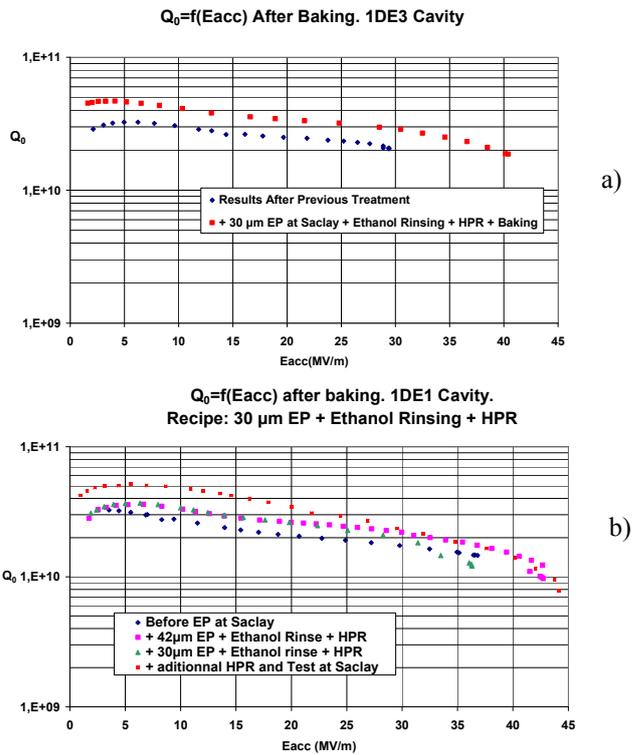


Figure 4:  $Q_0=f(E_{acc})$  for a) 1DE3 and b) 1DE1 cavities.

### EFFECT OF AN UNEXPECTED HIGHER TEMPERATURE

As it is shown in Figure 4b, 1DE1 cavity reached 36MV/m after additional EP sequence. Unfortunately, this result is questioning since we encountered a breakdown of the pump of the cooling system and acid temperature was not controlled and went up to 40°C during this EP. As a result, we noticed a strong degassing, a loss of the current oscillations and the surface of then cavity was mat after treatment. The mat aspect of the surface with the loss of current oscillations is in accordance with preliminary studies on samples.

As 1DE1 reached gradient above 43 MV/m at Saclay after an only additional High Pressure Rinsing (Red dots in Figure 4 b), it is not possible to link the performance with the mat/shiny aspect of the surface.

### LOWER VOLTAGE WITH 1-9 MIXTURE

It is commonly believed that current oscillations are responsible for the good surface state of electro-polished niobium surfaces. Characteristic I(V) curves such as in Figure 5 include two oscillations' areas at high and low voltages. Cavities are generally electro-polished in the oscillation area at high potentials. However, electro-polishing at low potentials would imply some benefits:

- Less electrical power would be consumed.
- Heating of the bath would be less consequent.
- Sulfur production at the cathode should be reduced.

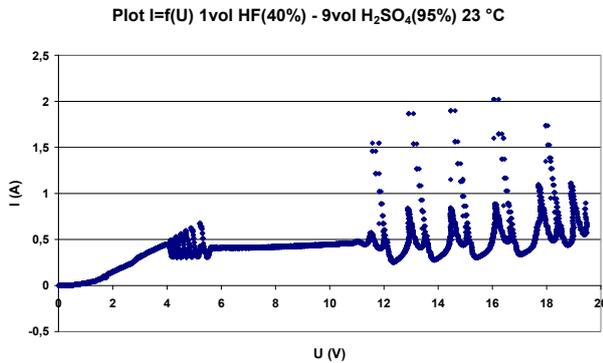


Figure 5: I(V) curve on a sample with standard mixture. Oscillations' area at low voltage is investigated on cavity.

Tests were carried out with a dummy cavity that showed a very bright surface after 100 $\mu$ m electro-polishing. As the process seems promising 1C21 cavity was electro-polished at 5 volts and 80 $\mu$ m were removed at this voltage. It was rinsed with ethanol. After tests at 1.7K, the cavity reached 32MV/m after baking (See Figure 6).

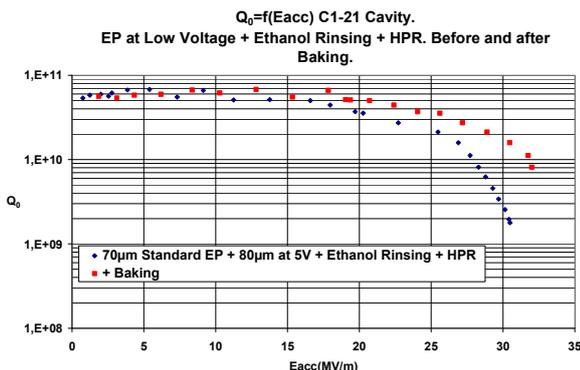


Figure 6:  $Q_0=f(E_{acc})$  at 1.7K. 1C21 cavity after EP at low voltage. Results before and after baking.

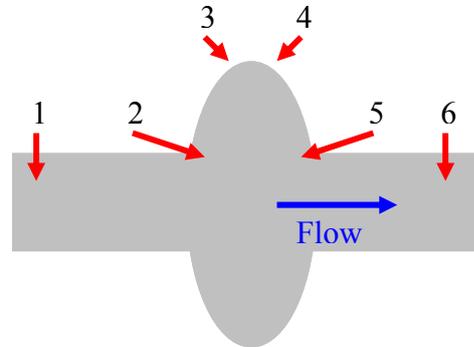
After this first encouraging result, additional EP sequences at low voltage on 1C21 are planned.

## ELECTRO-POLISHING WITH DILUTED MIXTURES

Niobium samples have been successfully electro-polished with the diluted mixture 1vol HF(48w%) – 2vol H<sub>2</sub>SO<sub>4</sub>(95w%) – 7vol H<sub>2</sub>O [3]: Surface state is improved, lifetime of the bath is longer and removal rate is high compared to the one obtained with the standard mixture. Other advantages might be foreseen in the perspective of large scale cavity treatment: easier preparation of mixtures, less expensive and less dangerous process.

A “dummy” cavity has been electro-polished at 8 V with the mixture: 1vol HF(40w%) – 2vol H<sub>2</sub>SO<sub>4</sub>(95w%) – 7vol H<sub>2</sub>O. We replaced aluminum cathode, which would have been strongly corroded in diluted acid mixture [6] with a copper one.

An interesting characteristic has been put forward: the removal rate seems more uniform in the cavity compared to the standard EP. An example is given in Figure 7 after a heavy removal (~100 $\mu$ m).



Location	1	2	3	4	5	6
$\Delta e$ ( $\mu$ m)	60	70	110	120	40	90

Figure 7: Removed thickness  $\Delta e$  in the cavity as a function of a location after an average 100 $\mu$ m removal.

The high removal rate at the equator might be attributed to diffusion phenomena with higher tangential speed at the equator during cavity rotation. However, as our Ultra Sonic device is only 10 $\mu$ m accurate and requires heavy removal material to detect noticeable change, complementary experiments are needed.

Unfortunately, some drawbacks were noticed:

- Sulfur is generated
- Some copper from the cathode deposits on the cavity inner surface. It would be necessary to use a less reactive metal.
- The final surface state after EP depends on the initial surface state.

The most prejudicial drawback is induced by a measurement on sample: a sample electro-polished with such a mixture was sent at DESY for hydrogen measurement. It showed heavy contamination: 13-15 $\mu$ g/g that should lead to Q-disease.

As a consequence, we stopped experimentations on cavity with diluted mixtures to focus on other parameters. Diluted mixture can not be used as a final electro-polishing step because of hydrogen contamination. Nevertheless, due to the high removal rate, long lifetime of the bath and a hint for more uniform removal rate, it could represent a promising alternative for the first stage of electro-polishing, before annealing of the cavity.

## CHANGE IN PARAMETERS

Our present goal is to prove effectiveness of ethanol rinsing with the EP set-up. However, it is a flexible device that facilitates R&D experimentation. It makes easy to investigate parameters such as rotation speed, acid flow and temperature. We started the study of the influence of rotation speed on EP with standard mixture at low voltage. As we notice it in Figure 8, an increase in rotation speed has no obvious impact on current at low voltage with standard mixture. It shows that EP in such

conditions is not driven by diffusion phenomenon. Furthermore EP at high rotation speed would have no real interest.

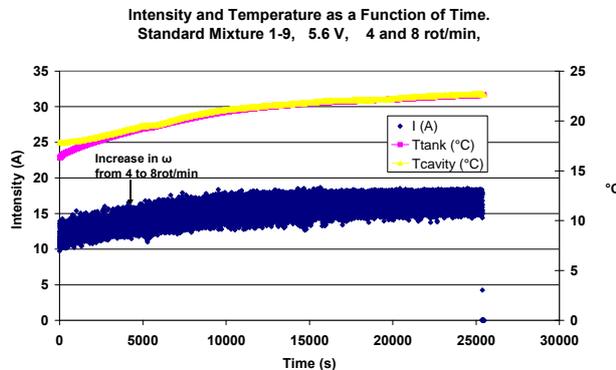


Figure 8: Increase in rotation speed during EP at low voltage with standard mixture.

On addition to this we also intend to test the more concentrated mixture:

3vol HF(40w%) – 6.72vol H<sub>2</sub>SO<sub>4</sub>(95w%) – 0.28vol H<sub>2</sub>O

This electrolyte showed promising advantages on samples: higher lifetime of the bath, increased removal rate, high brightness achieved, no increased hydrogen contamination.

## CONCLUSION

First RF-Tests results with standard EP parameters are excellent: The set-up is qualified and effectiveness of ethanol rinsing as a recipe to prevent sulfur contamination is investigated as a contribution for GDE R&D S0 program. Repeatable gradients over 40 MV/m are promising. Furthermore, the first result with low voltage EP is encouraging. This configuration, such as diluted and more concentrated mixtures will be investigated in detail.

If these tests with different acid concentration show good results, they could also be used for large scale treatment with two major advantages: higher lifetime and higher removal rate.

Nevertheless, the process itself needs to be improved. For example, fluid distribution is non uniform. Modeling is of paramount importance to improve the process.

## AKNOWLEDGEMENTS

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