COMMENTS ON ELECTROPOLISHING AT ETTORE ZANON SPA AT THE END OF EXFEL PRODUCTION

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Abstract

In 2013 a new horizontal Electropolishing facility was developed and implemented by Ettore Zanon SpA (EZ) for the treatment of cavities for the European XFEL series production. More than 300 cavities have been treated. Electropolishing has been used for two applications: bulk removal and recovering of cavities with surface defects. Treatment settings have been analysed and compared with cavities performances to verify possible influences of the various parameters. Main parameters considered are treatment time, voltage and current, that together define average thickness removal. We present here the results of these investigation.

The facility and process in use are also presented, together with possible next upgrade of the system, facing the new production of cavities for the LCLSII project.

INTRODUCTION

Since beginning of 2012, Ettore Zanon SpA has produced 420 1.3 GHz cavities for the EXFEL facility at DESY - Hamburg. At the present time, only final treatments for few cavities are missing before end of production.

BCP Flash Scheme

Cavities with outstanding and stable performances are required for the EXFEL, in order to be acceptable for the accelerator. Minimum goal is 23.6 MV/m with $Q_0 = 1x10^{10}$ and low field emission. Almost all cavities prepared up to now are well above the acceptance level required for module installation at CEA France.

To reach high gradients on a stable basis a surface treatment like electropolishing is required [1]. The inner surface of the cavities must be as smooth as possible, without defects or contaminants.

Two different chemical treatment have been developed for preparation of a cavity surface: Electropolishing (EP) and Buffered Chemical Polishing (BCP) [2].

For the EXFEL cavities two possible options are considered: BCP flash scheme (140 μ m main EP + 10 μ m final BCP) or final EP scheme (110 μ m main EP + 40 μ m final EP). Ettore Zanon SpA followed the BCP flash production scheme. After welding, cavity is degreased in a ultrasonic bath and rinsed with ultrapure water inside a clean room of ISO7 standard.



Figure 1: EP bench developed by E. Zanon.

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ISBN 978-3-95450-178-6



Figure 2: Process parameters during a 145 µm EP for a EXFEL cavity. Voltage, current, inlet and outlet temperatures (one data per minute) are displayed.



Figure 3: Process parameters during a 10 µm test EP with thermocouples connected at each equator: current and temperatures data are displayed.

The first surface treatment applied to the cavities is to remove 140 μ m Niobium with EP in a facility outside the cleanroom. After the electro-chemical process, cavity is rinsed with ultrapure water (UPW) to remove acid residues. Rinsing is completed with a short high pressure rinsing (HPR) inside ISO4 cleanroom [3].

Sulphur is produced during EP by a side reaction and must be removed dissolving it with ethanol [4]. Another problem related to EP is H2 adsorption in the Niobium material. The Hydrogen, embedded in the Niobium bulk material of the cavity, is therefore degased with a 800°C annealing treatment.

EP FACILITY AND TREATMENT

According to specification given by experts from DESY, EP plant (Fig. 1) has been developed by the technical department of Ettore Zanon SpA. The facility is designed for the horizontal EP of 1.3 GHz - 9 cell cavities. The cavity, placed in a frame, rotates at 1rpm during treatment. Rotation is transmitted to the frame of the cavity by a pulley, connected to a motor shaft. Two brass slewing rings allow current transmission and support frame and cavity during rotation.

The polishing bench turns to vertical position for water leak test, rinsing and also during removal of the cathode



Figure 4: Comparison between micron removal by weight and by current calculation.

(in order to leave the cavity filled with water in the meanwhile). Automatic movements are handled by a PLC by means of position sensors.

The cathode is made of Aluminium 99.5%, shielded with Teflon tape in correspondence to irises. It is inserted in the cavity with a distance of 14 mm to the irises. It is also used for acid distribution, thanks to nine holes placed in correspondence to equators. Nitrogen flow along the cavity is guaranteed by a holed tubing that is fixed to the cathode. All parts of the plant in contact with acid are made of fluorinated polymers (PFA, PVDF or PTFE).

Two tanks are used to recirculate acid during treatment (10 l/min), one in the storage area and one just below the cavity. Acid is cooled by a heat exchanger in the return line to the main storage tank. Plant is serviced with ultrapure water (18 MOhm*cm resistivity) and pure nitrogen 99.999%.

Exhaust gases, mostly hydrogen (H2) and hydrofluoric acid (HF) produced during treatment, are sucked and conveyed to a scrubber. To ensure personnel and explosion safety, H2 and HF sensors are installed in the room and in the piping system.

The facility showed high reliability and can perform five bulk treatments per week (140 μ m each) over a long period. A one/two days maintenance every month is necessary to assure stable quality and avoid mechanical problems to the plant. Included in the maintenance is the cleaning with alcohol, to prevent possible contaminants accumulation.

Treatment Details

Acid used for EP is a mixture of sulphuric acid and hydrofluoric acid 9:1. It can be used up to 10 g/l of dissolved Niobium without significant removal rate reduction. Reaction is exothermic and acid must be cooled. To keep current and temperature within acceptable min and max values, an ON/OFF controller is used, so that it start cooling acid when current gets too high. Current is used instead of temperature, because it

ISBN 978-3-95450-178-6

allows a much faster response. In Figure 2 a typical plot for a 140 μ m treatment is shown. The temperature variation is due to the cooling ON/OFF method.

Recently, new wireless thermocouples have been added in the plant, under request of JLab [5]. These sensors are installed directly on the cavity surface and reveal instantaneous temperature at each equator or iris. At the moment, they are only used for monitoring, but the idea is to implement a new temperature control using them.

A test with temperature recording at equators during a 10μ m EP is presented in Fig. 3. In this case, the oscillation of the temperature data is caused by the rotation of the cavity at 1 rpm, because the cavity is half-filled with acid: temperature is higher when the surface is in contact with acid and lower when it is in contact with gases. The actual temperature detected at the cavity surface is higher by ~4 °C, compared to the temperature detected by the old T sensor, that is placed on the outlet piping. The maximum temperature difference between two different equators is ~2 °C.

This is the first test of a series, scheduled in preparation for the treatment of LCLSII cavities. In fact, JLab suggests to have such thermocouples to have more insight into the process. A possible change of process parameters (voltage, temperature, cathode masking) is still under discussion.

According to average removal rate recorded (0.4 μ m/min), EP treatment duration is between 6 hours and 6.5 hours (if more than 140 μ m removal is desired). During EXFEL production, actual average removal is formally controlled by weighing cavity before and after treatment. However, it is also possible to have an online control during treatment. In fact, current value is correlated to the material removed, as shown in the following equation:

$$r\left[\frac{\mu m}{\min}\right] = \frac{I \cdot M_{Nb} \cdot 60 \cdot 10^{5}}{q_{e^{-}} \cdot 5 \cdot N_{A} \cdot \rho_{Nb} \cdot S_{cav}} = 1.484 \cdot 10^{-3} I[A],$$

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where:

 $r = removal rate, as \mu m/min,$ I = current intensity [A],

 $M_{\rm Nb} = Niobium molecular mass, 92.9 g/mol,$

 $q_{(e_{-})} =$ electron charge, 1.6*10⁻¹⁹ C,

 $N_A = Avogadro constant, 6.022*10^{23} mol^{-1}$

 $\rho_{\rm Nb}$ = Niobium density, 8570 g/dm3,

 S_{cav} = inner surface of a 1.3GHz cavity, 91 dm³,

5 =oxidation state of Niobium.

Data from weight measurement and from current calculation are well in agreement, as verified for more than 300 cavities treated (Figure 4).

Electropolishing treatment is composed by a series of operations:

- 1. Cavity is installed on EP bench
- 2. Pressure test with ultrapure water is performed, to check for leaks on piping connections.
- 3. Cavity and piping are drained with nitrogen, to avoid acid water contact.
- 4. Horizontal cavity is filled with acid up to the set level.
- 5. Costant voltage is applied (17 V) and reaction proceeds for approximately 6 hours.
- 6. Cavity is drained with nitrogen and rinsed before removing it from bench.



RESULTS

Figure 5: Correlation between material removal and frequency variation.

The availability of data from so many cavities has been used to verify some hypothesis discussed during cavities production. In particular, the effect of average total material removal was investigated.

As explained in the previous section, total removal is a value related to EP parameters:

- voltage,
- current,
- temperature,
- time.

In our system voltage is set and temperature and time are kept under control. As a consequence, current value is automatically determined by the reaction.

One important issue during production is to determine the final frequency of the cavity, before building it. The final frequency, of course, is modified by the inner surface chemical treatments, that change the internal cavity volume.

The average frequency variation due to EP treatment was estimated during first treatments and that value has been used for cavity composition. As shown in the Fig. 5, data are rather spread, but the mean value 1 μ m = 6 kHz is successfully used. In fact, even with some small variations, cavities are always in tolerance, with respect to the specification given by Desy.

One hypothesis for this behaviour is that the EP done at high temperatures (actual T recorded at equators can be higher than 40 $^{\circ}$ C) does not allow a strict control of the reaction and the actual local removal rate.



Figure 6: Comparison between micron removed and cavities performances.

Another topic discussed during production is the possibility to gain better performances for the cavities, performing a longer EP, which means higher removal of material from the surface.

This doubt arises because only the average thickness removed is known and the actual value removed at equators is not measured (no measurements with thickness gauge are scheduled for series production) [6].

For this reason, to avoid the risk of retreatments, several cavities underwent a longer EP. Moreover, some cavities have a much higher total removal, because a second EP was necessary for production needs. In the plot in Fig. 6, the total micron removed is the sum of all EP performed for each cavity.

From these data, collected from more than 300 cavities, it seems that a higher removal rate does not imply a better performance during vertical test. Here the maximum acceleration gradient is considered, but similar plot is obtained also for usable acceleration gradient (usable Eacc is a parameter in use for EXFEL cavities, that takes into account quality factor and field emission) [7]. If better performances will be required for a future project, simply increasing EP treatment is not an effective solution. However, it must be noticed that these data refer to cavities that performed final 10 μm BCP.

CONCLUSIONS

The completion of EXFEL production has shown that the facility is reliable and can perform five bulk treatments per week (140 μ m each) over a long period.

Moreover, as long as the minimum 140μ m removal is guaranteed, cavities electropolished with this facility perform well during vertical test, above requested goal (23 MV/m). Longer treatments make sense only if necessary for other production needs.

Facing the new production of cavities for the upgrading of LCLSII, the process will be modified and verified according to specifications of Jefferson Lab. For these cavities, final EP after nitrogen doping will also be set up, and final BCP will be discarded.

One of the changes under discussion is the possibility to perform EP at lower temperature, with the aim of avoiding sulphur production and improve correlation between removal and frequency. In fact, more investigation may be interesting in this field, to better understand and control frequency variation behaviour.

ACKNOWLEDGMENT

EXFEL series production was a new experience for everybody involved. We would like to thank the whole staff that made it a successful experience.

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