UPDATE ON EP EXPERIENCES AT DESY WITH THE ELECTRO POLISHING PROCESS AT DESY 1.3 GHZ

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Abstract

Since several years electro-polishing (EP) of superconducting resonators is one of the major preparation techniques in use for high gradient superconducting resonators. At DESY the EP facility is operational since mid of 2003. About 150 resonators have been polished in this infrastructure since. Typical process data, a maintenance plan and reliability of components installed in the DESY apparatus as well as actual RF test results of resonators will be presented. For the XFEL project the industrialization of the electro polishing processes is started. A status report of the industrialization will be given.

INTRODUCTION

For the XFEL it is proposed that 850 Resontotrs will undergo a surface preparation by eletropolishing [Ref.:1]. The DESY Ep facility will serve as an infrastructure on which the design of industrial EP facilities bases on. Intensive data- and error event logging [Ref.:2] allows to point out basic limitations and consumables of the facility. A maintance plan basing on these data can serve for a quotation of costs in industrial production.

SET UP AND MAINTENACE

1) EP set up at DESY

The DESY EP infrastructure was set up in 2003 and serves as major cavity surface treatment infrastructure since. The DESY EP infrastructure can be divided in three major sub units, the EP bench, the storage and supply cabinet and the gas and liquid disposal units.

1.1) EP bench

Since start up of the bench no major problems with the mechanics are observed. The original rotary shaft seals, made from Viton (Fig.:1), show strong corrosion [Ref.:3] after 50h of operation. In 2005 this design was exchanged to shaft seals made from Teflon where no wear out is observed since. The maintenance of these seals is limited to a once a year inspection. The sump located underneath the EP bench is made from PVDF and has to be exchanged every two year due to strong corrosion on the PVDF material. To study the origin of corrosion the sumps exchanged are cut and analysed. On two different sump units different corrosion is observed. According to literature PVDF of different degree of polymerisation can be obtained by different temperatures applied during production.

Figure 1: Corrosion of rotary shaft seals made from Viton the PVDF material.

Depending on the degree of polymerisation different resistance against aggressive media are reported. The sumps in use are made of commercial available plates by in house hot air welding. For sump No 2 high polymer PVDF was in use and showed corrosion in the welding seams which are exposed to different temperatures during welding. After two years about 40% of the wall, receptively weld thickness is corroded. This corrosion is observed even on areas with no direct contact to the acid. For safety reasons the sump tanks have to be exchanged at least every two years of contact with the acid or the evaporated gases.

Figure 2: Left corrosion on welds of sump no 2; Right corrosion on the commercial available PVDF

1.2) Storage and Supply Unit

The pneumatic driven acid pumps are made from Telfon and in use since 2003. No leakage, no corrosion or reduction in performance is observed since. The diaphragm membranes based design of the pumps leads to strong pulsing of the acid pressure. This pulsation results in vibrations of the acid supply tubes. Due to these vibrations the fittings of pipes, valves and sensors have to be controlled and re tightened once a year. In 2007 the pumps are exchanged to a bellows based Teflon pumps , which show a strong reduction of the vibrations inside the acid circuit.

The heat exchangers in use at DESY are made from PVDF and show corrosion on welds like found in the PVDF sumps No 2 as well. They are exchanged every two years to ensure leak tightness between the cooling water and the acid lines

A flow sensor inside the acid supply line is part of the safety control and is one basic parameter out of three parameters in use to steer and control the process. It needed to be exchanged after 4 years of operation due to corrosion on the housing and the impeller (Fig.:3).

Figure 3: Impeller section detached form sensor due to corroded connectors

Temperature sensors embedded in Teflon tubes are installed to control the acid temperatures on different location. These sensors show small deviation in temperature after about 2 years of operation. Due to the fact that these sensors are part of the steering, the process control and the safety circuits build in the DESY EP set up, they are exchanged every two years.

1.3Gas and liquid disposal

Hydrofluoric (Hf) gases are strongly evaporated form the EP acid mixture in use. HF sensors are in stalled in the acid storage cabinet, the housing of the EP bench and inside the exhausting lines that collects all out gassings from the EP process. These sensors serve for personal and environmental safety and are subject of strict control of the authorities. They need to be calibrated twice a year. After 2 years of operation the sensitivity is reduced and the sensors probe heads have to be exchanged.

Gaskets inside the HF feed line towards the Hf absorbers are corroded after about 4 years of operation. The detection of leaking gasket installed in gas lines is complicated due to the limited excess to pipes and connectors during operation. In 2007 a shut down of four weeks was needed to pin point the origin of HF gases detected by the HF sensors. To prevent long shut downs within the production of resonators, gaskets should be exchanged at least after two years of operation.

2. Maintenance plan

Basing on the experiences gained with the DESY EP apparatus so far, a maintenance plan (Table 1) for is worked out. Baseline for this plan is a yearly shut down and maintenance period which sums up to a total of 2 weeks, one week for acid draining.

Table1: Maintenance plan for the DESY EP facility

	clean + control	calibrate	exchange
Hf absorbent	on line		according to HF sensor signals
Shaft seals	1		
Sump			2 years
Heat exchanger			2 years
Flow sensor	1	1	2 years
T sensor		1	1 yearly
Electrode	1		200h
Power supply connectors	1	1	2 years
Exhauster gaskets	1		2 yeras
Pumps	1	1	
PVDF valves	1		3 years
Tubings	1		
HF+ Gas sensors	1	twice a year	2 yeras
Power supply	1		
Level sensors	1	1	2 years

and rinsing of pipes and about one week for the maintenance of the system components and restart of the facility

2 Process data and aging

2.1 System aging

Since start up in 2003 all process relevant data are stored in a data base [Ref.:2]. Since the major process data like acid volume Q; voltage V and the temperature range between inlet temperature T3 and the outlet temperature T4 are kept constant, the over all process data can be correlated and analysed. (Fig.. 3)

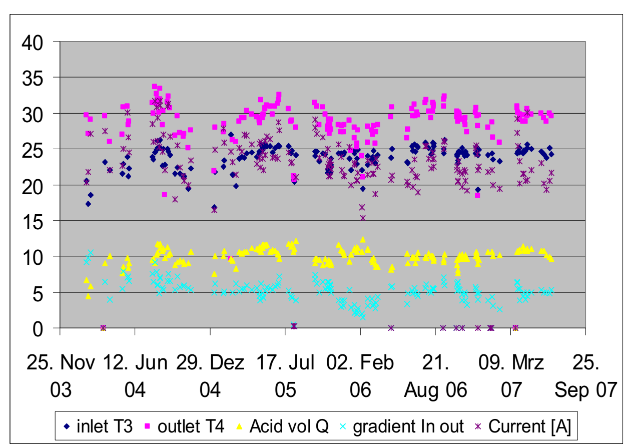

Figure 3: Correlation of average current, acid temperature and temperature gradient since 2003

The data analysis shows that the average current is continuously degrading. Only after a shutdown and maintenance a recovery of the averaged current is observed. After a short operation time, following the shut down, the current seems to fall back to the general tendency of degrading average current observed over the years.

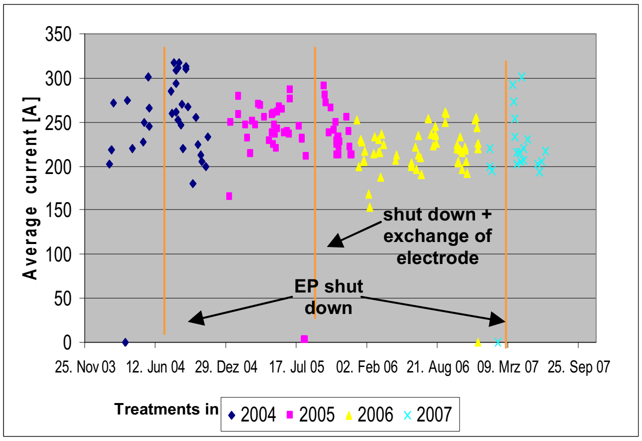

Figure 4: Development of average current [A] during EP processes since 2004

The maintenance of major components like the electrode, cable connectors, rotary current sleeves or a calibration of the power supply results in a short time of higher current flow before the current reduces back to the general tendency again. Actually the origin of this aging can not be explained

The reduction of average current causes reduced removal rates. The process time for the main EP process [Ref.:4], where 160 µm of Niobium have to be removed, had to be extended from 6 hours in 2003 [Ref.5] up to 7 hours in 2007.

2.2 Acid aging

The activity of the acid mixture in use is correlated to the amount of free Hf dissolved in the mixture, the voltage applied and the temerature. These EP parameters define the removal rate. Most EP apparatus use constant voltage condition and define the process via the time of the EP treatment. The aging of the mixture is expressed by the reduction of the removal rate. The analysis of data published, do not show significant correlation between the acceleration gradients and the aid in use time. At DESY the acid in use is exchanges when about 12 gr of Nb per litre acid are dissolved, which actually corresponds to removal rates of 0,3 µm/min.

The removal of material can not be measured online yet. During start up of the polishing process the processing time has to be defined and put into the data set of the EP processors. Aging of the acid requires different data set for different in use time of the acid. To provide more reliable data for process input for the operators, the in use time of an acid barrel and the removal rate, defined

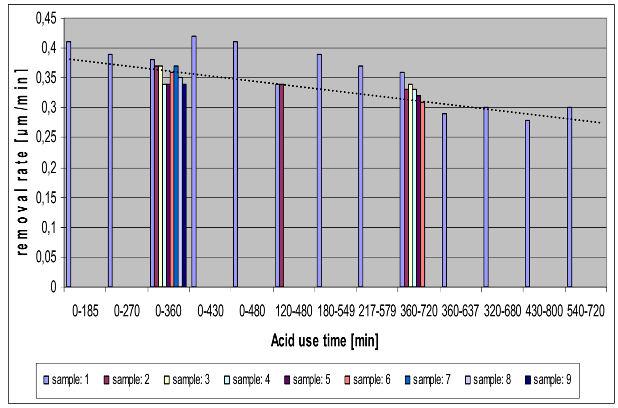

Figure 5: Reduction of removal rates correlated to the in use time of the acid

by the weight loss of the cavity during the process. (average removal rate) are measured (Fig.: 5) and serve for input data definition.

2.3 Optimization of processing time

For an industrial processing of cavities the treatment time is one of the major cost driving factors. To optimize the processes, the DESY data are analysed in respect of gradient and removal of material.

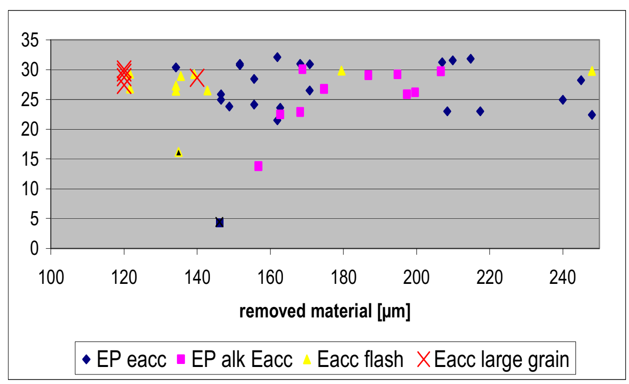

Figure 6: Correlation of max. gradient and removed material since 2004 . (only RF test results 1st power rise before 120 C baking are displayed)

Only the RF measurements of the first power rise after preparation and before 120 C baking are analysed to determine the process related parameters. These data (Fig.:6) indicate that after a total removal of 160 to 180 µm most resonators reach gradients well above 25 MV/m. Four cavities are limited by fabrication errors on welds or H2 contamination below 15MV/m after preparation. Cavities showing gradient of 20 to25 MV/m are loaded with strong electron loading which may cause the limited gradient.

3. Ethanol rinsing

Strong sulphur settling is found in the EP apparatus [Ref.:3]. The sulphur origins form a chemical reaction of the alumina electrode and the EP acid in use [Ref.:7]. Sulphur is able to settle and stick on the cavity surface and is well know as a source of field emission. Studies made on samples show that sulphur removal by High Pressure Rinsing or ultrasonic treatments with ultra pure water is not very efficient while pure ethanol removes the sulphur segregations on the samples effectively (Fig.:7). Since 2006 alcohol rinsing is part of the cavity preparation sequence [Ref.:5] at DESY.

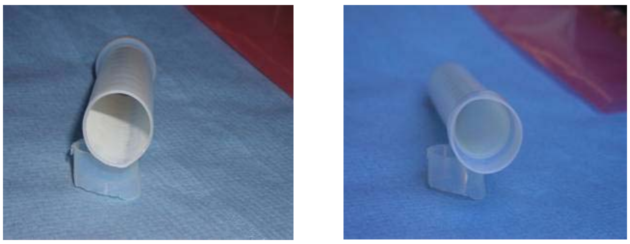

Figure 7: Sulphur removal with ethanol. Left: tube as removed from EP apparatus; right: tube after ethanol cleaning

To study the efficiency of ethanol rinsing RF data of the first power rise, right after preparation and before 120 C baking of the resonators are analysed (Fig.:8). The maximum accepted level of fieldemission (fe Limit) of resonators for application in accelerator modules of the flash linac is set to 1Exp^{-2} mGy /min., measured in the DESY vertical test inserts.

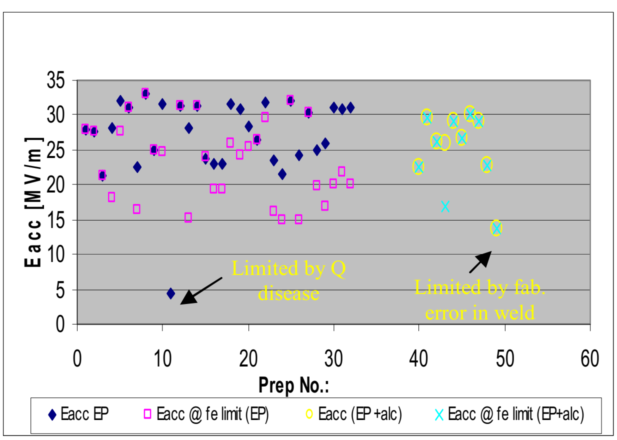

Figure 8: Comparison of test results with and without alcohol rinse applied after assembly before 6 times HPR (only RF test results 1st power rise before 120 C baking are displayed)

The data (Fig.:8) show a clear improvement due to the application of alcohol rinsing. The x ray loading is strongly reduced while the spread in maximum gradient of the cavities is not influenced by the ethanol rinse

INDUSTRIALIZATION

For the XFEL cavities the cavity preparation is proposed to be done by industry. Electropolishing will be a major part in the preparation technique there. To study the transfer from laboratory level to industrial fabrication a study on industrialization of the EP process is placed to industry in February 2007. A total of 30 resonators have to undergo the main electropolishing [Ref.:4] in industry. Two companies (Accel Instruments, Bergisch-Gladbach, Germany and Henkel Elektropolitur, Neustadt Glewe, Germany) build up infrastructure to perform the main EP under industrial condition. The execution of the administrative and legal rules took about four month. After legal permission the installation phase of about 5 month was started. The industrial EP systems are under commissioning now and first cavities are expected to be delivered to DESY by November 2007.

CONCLUSION

The DESY electropolishing set up is running since 2003 and has performed more than 200 electro polishing processes with a total treatment time of more than 470 hours. Basing on the experiences gained since, a maintenance plan is set up. Even the parameters of the EP process are fixed and controlled by a PLC, a continuous reduction of the average current (removal rates) is observed but can not be explained yet.

An ethanol rinse of cavities is made to remove sulphur, origin from chemical reactions of the electrode and EP mixture. Cavities undergone that alcohol rinse showed strong reduction of fieldemission. That ethanol rinse is now a standardized part of the cavity preparation of cavities at DESY

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