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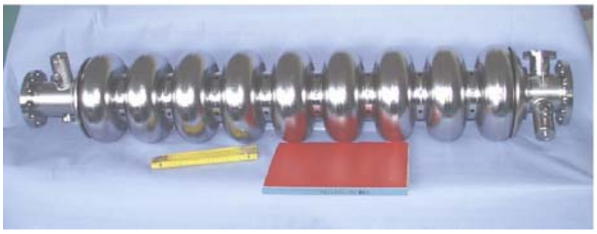

QUALITY CONTROL OF THE ELECTRO POLISHING PROCESS AT DESY

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Abstract

The technology of electro-polishing of super-conducting resonators made from Niobium is foreseen as basic surface preparation technology for the XFEL accelerator project at DESY. Here about 1000 resonators will be build and installed into the accelerator section. For an industrial application of this technique a quality control has to be developed and established. A method to control the acid quality and improve the life time of the acid is under development. We report on the test setup and measurements done on samples and the implementation of this quality control to the DESY electro polishing process.

Contribution to the PAC05, Knoxville, USA

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Quality Control of the Electro Polishing Process at DESY

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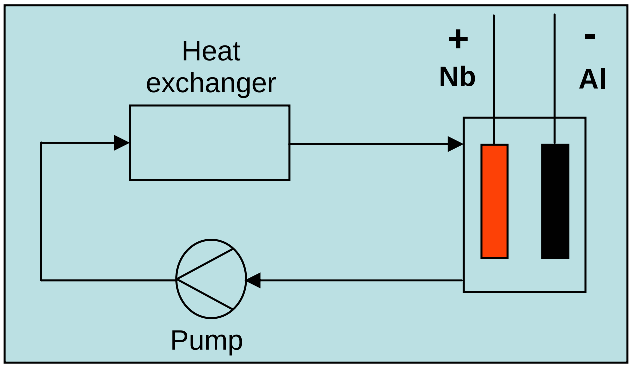
Abstract

The technology of electro-polishing of super-conducting resonators made from Niobium is foreseen as basic surface preparation technology for the XFEL accelerator project at DESY. Here about 1000 resonators will be build and installed into the accelerator section. For an industrial application of this technique a quality control has to be developed and established. A method to control the acid quality and improve the life time of the acid is under development. We report on the test setup and measurements done on samples and the implementation of this quality control to the DESY electro polishing process.

INTRODUCTION

The investigations on the electropolishing (EP)-process are made in the scope of the EU funded research on the introduction of quality control of the EP-process (CARE, contract number RII3-CT-2003-506395).
The EP-process of the DESY-EP-facility displays variations in the current, which are influenced by several parameters. Investigations on the current density vs. voltage characteristic of the acid mixture have taken place to analyze the acid. From this analyze parameters should be found to stabilize and optimize the EP-process.
A defined quantity of acid is poured into a trial case, in which two electrodes (Nb-anode, Al-cathode) are being immersed into the acid. The electrodes are separated by a fixed distance of 26mm. During this process the temperature of the acid is stabilized. A heat exchanger made from aluminium (Pic. 1) is integrated into the acid circuit.



Picture1: Schematic of the experimental set up

The voltage is gradually increased to the final value of 25V by a computer steered power supply. The I-U- values are recorded via computer-controlled mains adapter (Pic. 2).

Picture 2: Computer-controlled mains adapter

Each measuring point is read out 10 times at fixed voltage. The mean value of these data is displayed on the mains adapter unit. This method ensures the accuracy and reproducibility of the measurement.

INVESTIGATION ON REPRODUCIBILITY

To prove the reproducibility and sensitivity of the j/U test set up, the same acid was analyzed ten times within the same boundary conditions. In the voltage range of 5-21V the accuracy of the measurement is calculated to be 1-2% of the current density value (Fig.1).

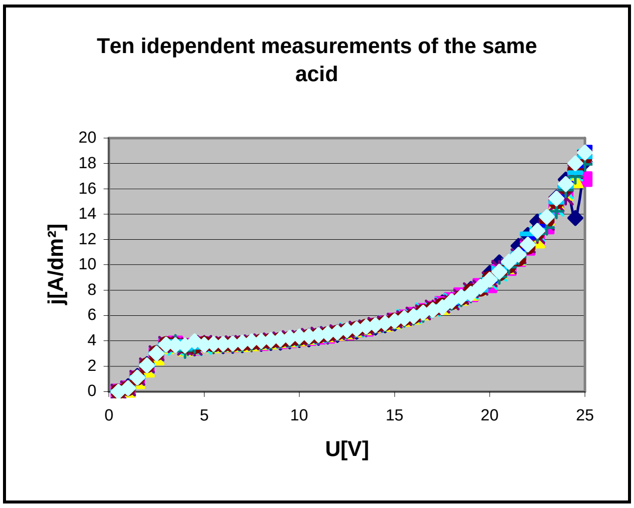


Figure1: Reproducibility measurement

These results show that the reproducibility is good enough to serve as analysis method.

ORIGIN OF CURRENT VARIATION

To use this method for quality control of the EP acid it is necessary to know which parameters influences the change of current. Our experience shows, that there are mainly two parameters that cause a variation of the EP current, process temperature and HF-concentration.

The influence of temperature

In the theory of chemical reactions the increasing of temperature results in an increase of the ion mobility. This implies a descent of the electrical resistance. To control the parameters for the EP mixture used at DESY (HF/H_2SO_4 1/9 with 2,78 mol/l HF) the same sample of acid with nearly unchanged concentration of components within the measurement is analyzed. The acid temperature is stabilized to ± 0,3K. At four process relevant temperatures the j/U values are measured. The results show a strong dependency of current density and temperature density (Fig. 2).

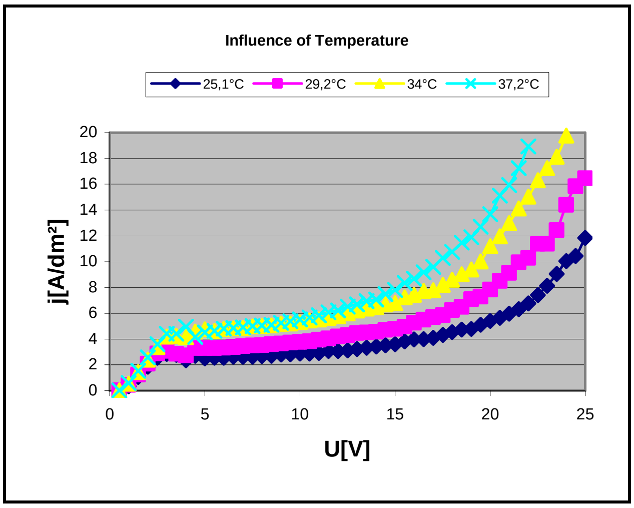


Figure 2: Temperature dependency of the EP mixture

The influence of HF-concentration

Due to the electro-chemical process (Eq.1) [Ref.1] and out gassing of HF, the HF-concentration of the acid decreases.

1st

$$2Nb + 5SO_4^{2-} + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^-$$

2nd

$$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F \bullet 0{,}5H_2O + 1{,}5H_2O$$

3rd

$$NbO_2F \bullet 0{,}5H_2O + 4HF \rightarrow H_2NbOF_5 + 1{,}5H_2O$$

Equation 1: Chemical formula for the EP-reaction

To see the influence of the HF-concentration, acids with HF-contents between 100% (2,78 mol/l) and 20% of the originally HF-concentration are measured at the same temperature.

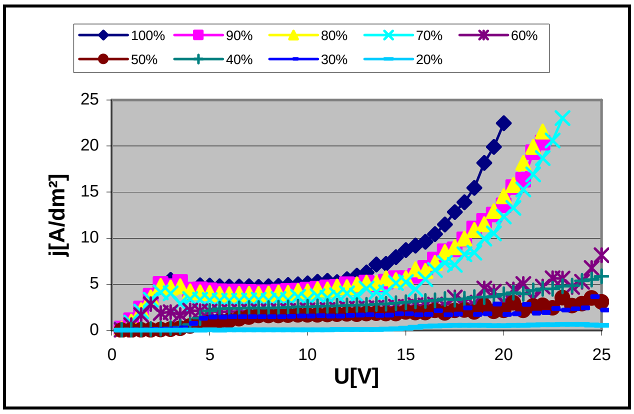


Figure 3: The influence of HF-concentration

The results show a clearly dependence between the HF-concentration and the current. For HF-concentration below 70% of the originally concentration, the current density is significantly lower then for a fresh acid.
To make sure that this curve degradation depends to the HF concentration, an acid with only 50% of the original HF was measured first. For a second measurement the HF concentration is refreshed to 100 %. Fig.4 displays the two U/j curves and shows the correlation to fresh acid (100% HF).

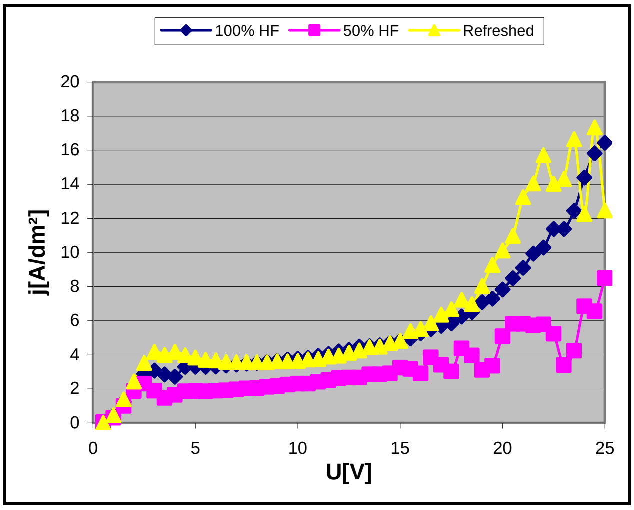


Figure 4: Regenerated acid

These results confirm the assumption that for constant temperature HF concentration is the leading factor for reduction of current density.

QUALITY CONTROL

The test setup, applied at constant temperature, can serve as a quality control instrument. This allows analyzing the entire HF losses (out gassing and chemical losses) of the acid to be tested.

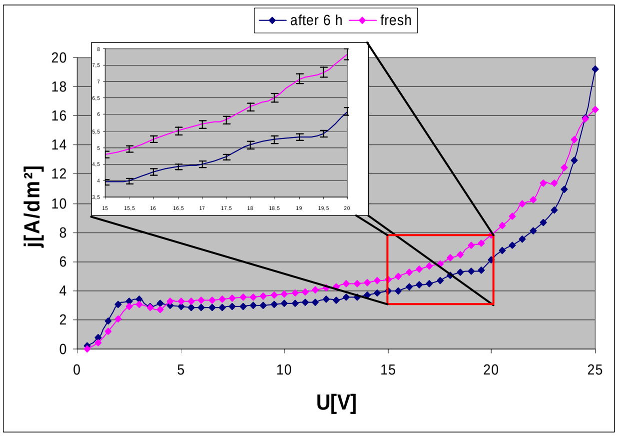


Figure 5: Fresh Acid and after 6 hours

An example for this application is given in Fig.5. The acid in use at the DESY EP setup is controlled before and after usage. The reduction of the current density measured at the DESY EP process confirms with the sampling of the acid in the test apparatus. With standard deviation taken into account, clearly separated curves are found. They allow to re identifying the HF concentration in the acid.

CONCLUSION

It was shown that this method is applicable in the laboratory. Current densities measured in the test setup are comparable to those of the EP facility at DESY (Ref.2).
This analyze setup will allow to design an online measurement for an industrial polish setup. A measurement sample holder integrated into the acid lines of an EP apparatus will allow controlling the acid composition online. More over a system to refresh the HF component can be designed.
The influence of niobium, solved in the acid, as a second component of aging has to be studied to get the full quality control parameter set.

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[2] A. Matheisen, „Electro - polishing surface preparation for high gradient cavities at DESY“, this conference

ACKNOWLEGMENT

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