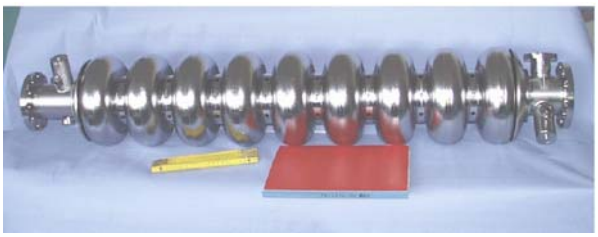
SRF 

Investigation on the aging process of EP-acid

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Abstract

The influence of the aging process of the acid on the current-voltage-characteristic of the acid mixture has been investigated to optimize the EP-process.

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Introduction

The aging process of acid was investigated in the scope of the EU funded research on the introduction of quality control of the electropolishing (EP)-process. The EP-process of the DESY-EP-facility displays variations in the current, which are influenced by the aging process of the acid. Investigations on the current-voltage-characteristic of the acid mixture have taken place to stabilize and optimize the EP-process. At a defined quantity of acid is poured into a trial case, in which two electrodes (Nb-anode, Al-cathode) are being immerged with a constant separation distance. With the use of a computer steered power supply the voltage was gradually increased to the final position of 25 V. The I - U-values are being recorded via computer-controlled mains adapter.

Every measuring point is read 10 times and out of these results emerges the average value. All investigations were repeated and the results compared to assure the reproducibility of it.

1. Investigations of the acid on changes in the current

To assert the changes that the current undergoes during the EP, acid-samples were taken from every fresh barrel before the first and after every following EP in the EP-facility and were analysed via a polarogram. In diagram 1 you can clearly see the decrease of the current during the increasing time of the process.

Observation: During the investigation a relatively low current value for fresh acid was noticed. As for now, we have not found and explanation for that.

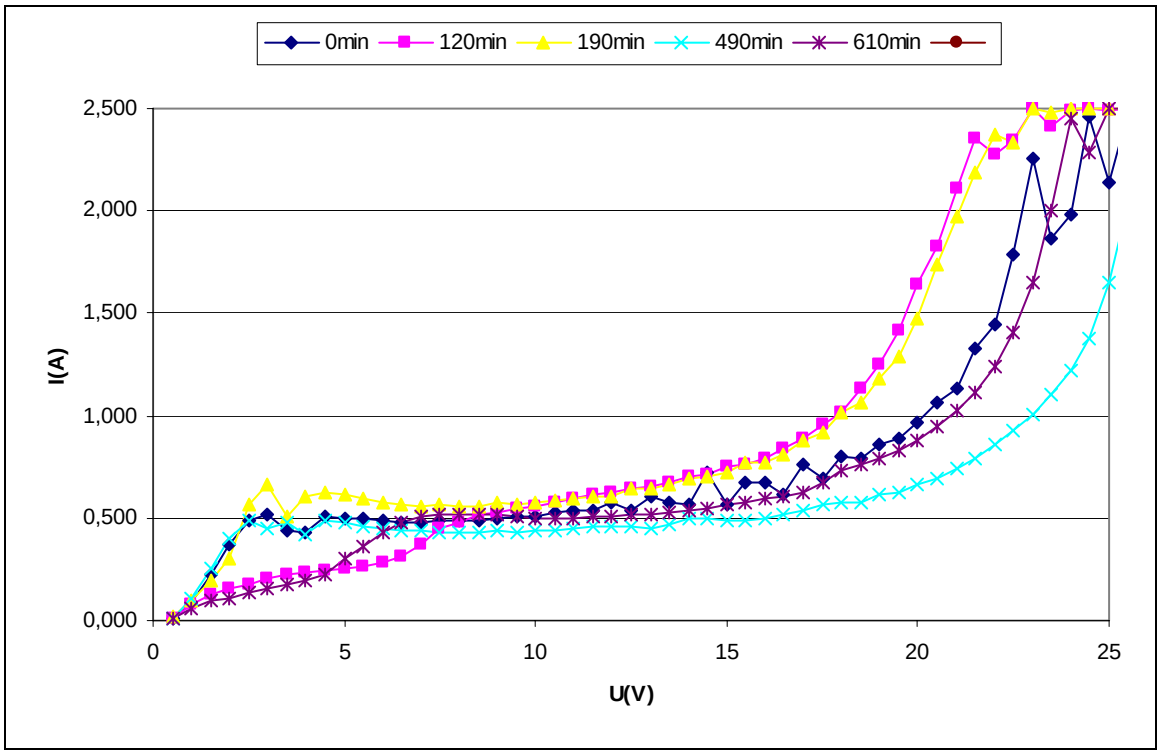

Diagram 1: Barrel 1 at 30°C

2. Investigation of the influence of the HF-content on the EP-process

2.1 Basic considerations

Fresh EP-acid has a HF (hydrofluoric acid)-concentration of 2,78 mol/l. This concentration decreases both through chemical reaction and degassing. The empirically determined limit value, at which the acid counts as used, is at about 10g dissolved Nb per litre of acid, which corresponds to a Nb-concentration of 0,108 mol/l. Of the chemical formula

$$2Nb + 5SO_4^{2-} + 5H_2O \rightarrow Nb_2O_5 + 10H^+ + 5SO_4^{2-} + 10e^- \text{ [Eq.1]}$$

$$Nb_2O_5 + 6HF \rightarrow H_2NbOF_5 + NbO_2F \bullet 0{,}5H_2O + 1{,}5H_2O \text{ [Eq.2]}$$

$$NbO_2F \bullet 0{,}5H_2O + 4HF \rightarrow H_2NbOF_5 + 1{,}5H_2O \quad \text{[Eq.3]}$$

we can conclude a HF-usage of 0,54 mol/l, which is a decrease of the HF-concentration of 19%. At a fill-up quantity of 250kg with a density of 1,8 kg/l, 1.400 g dissolved Nb corresponds to a concentration of 10 g/l.
The decrease of HF through degassing from the acid has not been investigated at this point.

2.2 Results of the measurements

A number of samples with a HF-concentration between 100% and 20% of the original HF-quantity were prepared in the laboratory to investigation the influence of the HF-concentration on the EP-process. We can see from the results (diagram 2-4), that at a HF-concentration of about 70% of the original HF-concentration the level is reached, once fallen below the current (I) decreases notably and therefore the removal rate is strongly reduced. The acid is then used up.

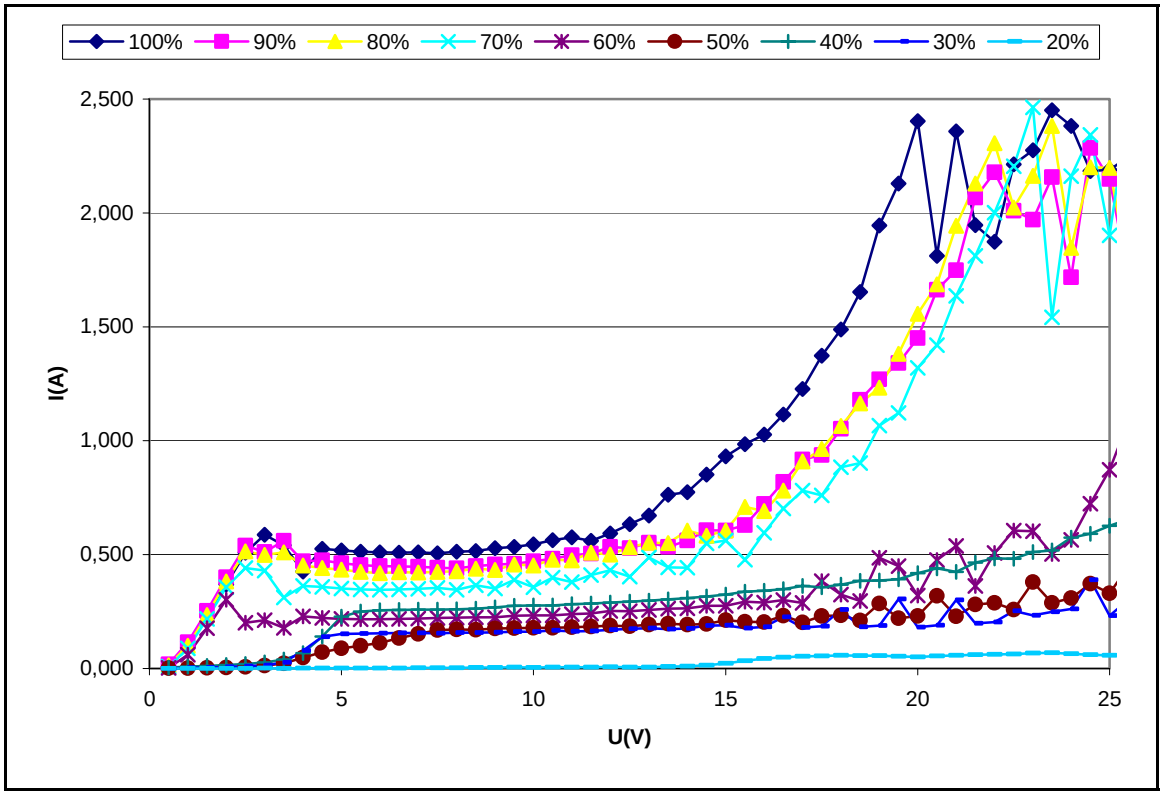

Diagram 2: Test 1 at 30°C, I-U-characteristics of samples with different HF-quantities

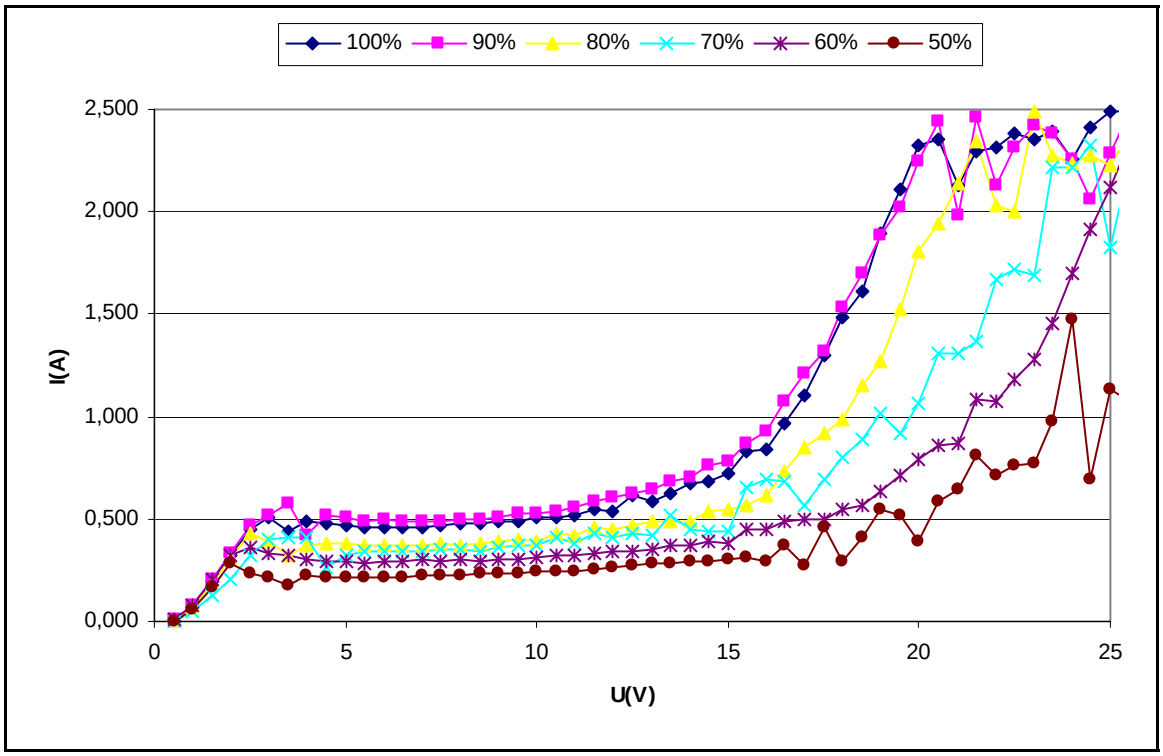

Diagram 3: Test 2 at 30°C, I-U-characteristics of samples with different HF-quantities

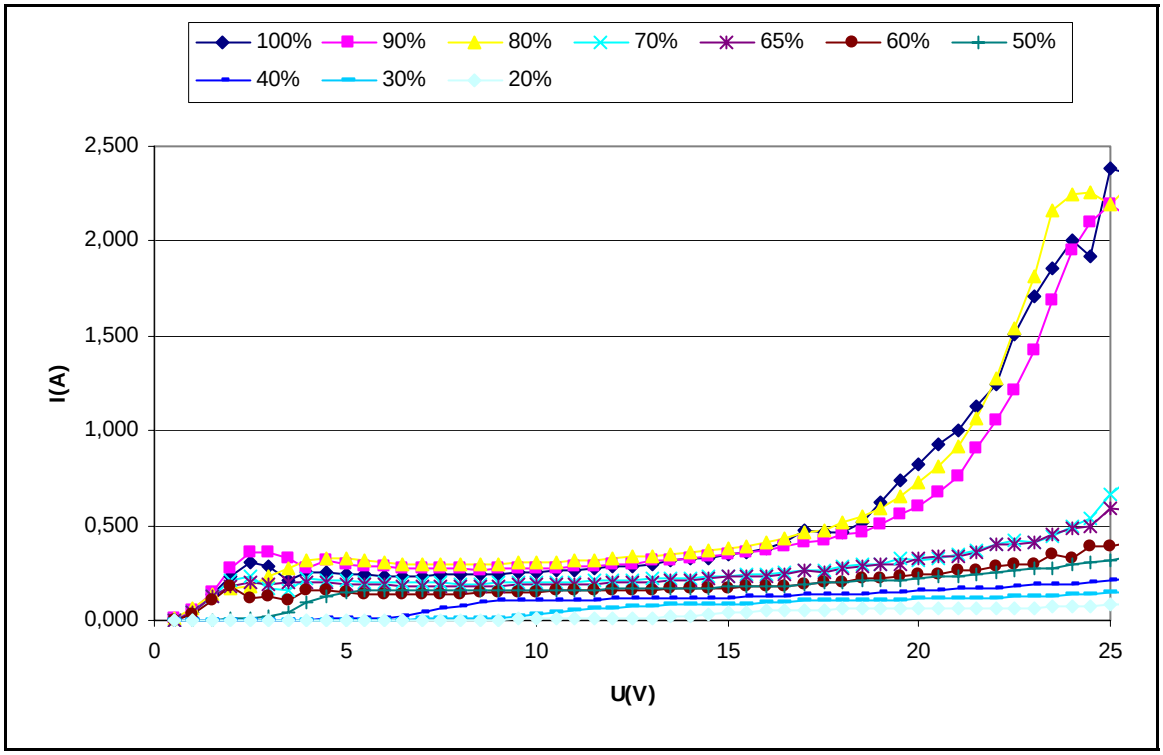

Diagram 4: Test 3 at 20°C, I-U-characteristics of samples with different HF-quantities

2.3 Is a regeneration of the acid possible?

After the proceeding investigations the question whether it is possible to regenerate acid by adding HF and in doing so to extend the durability of the acid was to be answered. To test this possibility an acid with 50% of the original HF-quantity was prepared, measured and finally regenerated to 70% of the HF-quantity by adding HF. The results (diagram 5) show, that it is possible to refresh acid by adding HF. By increasing the HF concentration from 50% to 70% the refreshed acid shows a similar curve to the directly blended acid with the same concentration.

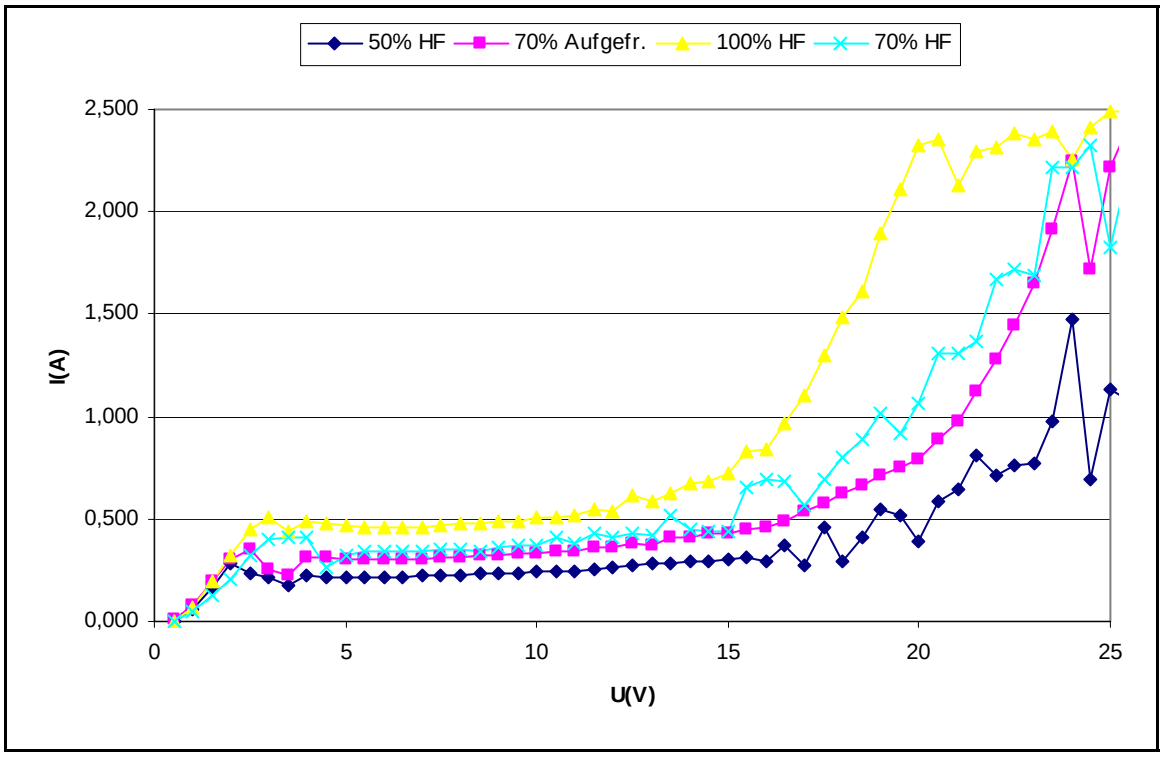

Diagram 5: Investigation on regeneration

3. Summary and prospects

The HF-quantity of the acid decreases through the chemical reaction during the EP. This has a negative effect on the parameters of the current, and therefore on the removal rate, which decreases in the course of the reaction.
A new method has been found, through which the investigation of acid with simple means is possible. It was proved with this method that a regeneration of acid is possible and therefore a longer durability under constant conditions can be reached.
A dosing pump should be build into the EP-facility, which constantly adds small doses of HF and therefore keeps the level of acid constant.
To define the exact amount to be added, the fumes of the EP-process have to be investigated on the HF-value, to level out this loss as well.