STATUS OF THE EP SIMULATIONS AND FACILITIES FOR THE SPL

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Abstract

CERN is assembling a new vertical electropolishing facility in order to process several niobium cavities of beta 1 and beta 0.65 in the context of the HP-SPL R&D programme. Electrochemical simulations are being used in order to define the optimal cathode geometry to process the cavities in a vertical position. Macroscopic properties of fluid dynamics like the Reynolds number and thermodynamics linked to the power dissipated in the process are taken into account to dimension the main system components. All the materials from the different equipments must be compatible with all chemicals within the required working temperature and pressure. To provide safe operating conditions when handling chemicals or processing cavities, specific safety and protection equipment is also foreseen.

INTRODUCTION

In the context of the HP-SPL R&D programme, CERN is assembling a new electropolishing installation. It integrates past experience with monocell Tesla type Niobium cavities and Copper cavities vertical electropolishing [1, 2].

The new electropolishing facility is conceived to process cavities in a vertical position. This allows the cavity to be permanently immersed during treatment avoiding transient phenomena as with half immersed horizontal setups and therefore improving surface finishing. On the other hand, this configuration relies on a strict understanding of the electropolishing conditions like bath composition, flow parameters, temperature control and cathode geometry, in order to avoid irreversible damaging of cavities.

BATH PARAMETERS

The physical, chemical and electrochemical analysis of the electropolishing bath is essential in order to understand and optimize the behaviour of the electropolishing process, and in particular for performing a simulation of current density distribution.

Hereafter are described the methods used to characterise the electropolishing bath. The reported results are relative to a standard bath prepared from 96% m/m of sulphuric acid and 40% m/m of hydrofluoric acid in a volume ratio of 9 to 1.

Physical Parameters

The main physical parameters are the electrical conductivity, temperature, viscosity and density.

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Conductivity was measured with a standard probe; ranging from 100 mS/cm at 15ºC to 130 mS/cm at 25ºC

Operating temperature was measured with a thermometer fully covered with PTFE, with a range from 0 to 100ºC.

In absence of compatible measuring equipment, the viscosity was calculated from literature values of separated components: μ = 23 cp at 20ºC.

Density was measured with a polypropylene volumetric flask; within the temperature range, density doesn't change significantly from the value d = 1.8.

Chemical Parameters

Water content can be determined using Karl-Fischer titration and a Sigma-Aldrich procedure.

Free fluoride content can be determined in a new prepared bath by titration using an ion-selective electrode. However and for used baths with increasing niobium concentration, the method doesn't seem to work. Further studies are ongoing.

Niobium content can be determined by atomic absorption spectroscopy (AAS)

Electrochemical Parameters

The most important of all parameters for a simulation is the polarisation curve. Its shape combines the bath composition, physical parameters like agitation, temperature, density and viscosity, with potential or current inputs.

In order to understand the behaviour of the electropolishing process, the polarisation curve is measured under different conditions. Figure 1 and 2 illustrate some examples, measured with a rotating electrode system for an accurate control of the flow in fast scan mode.

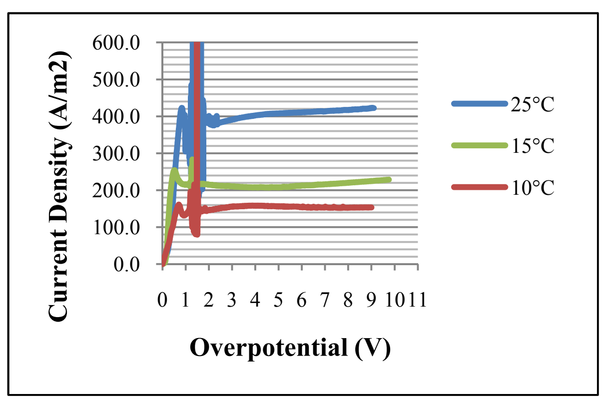


Figure 1: Temperature effect on the polarization curve at 100 rpm – Pt-reference electrode – Copper counter electrode – surface ratio 1 - new bath.

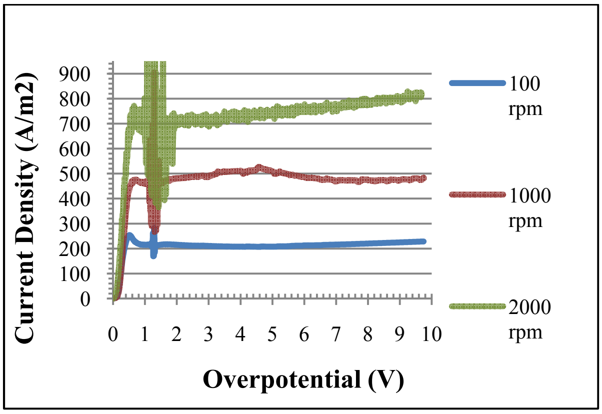


Figure 2: Effect of the agitation on the polarization curve. 15°C – Pt-reference electrode – Copper counter electrode – surface ratio 1 - new bath.

For the simulation software, these fast scans (10 mV/s) can introduce transient phenomena that won't be seen in the real process. Thus a steady state polarisation curve must be built based on the different important areas identified by the fast scan. A steady state polarisation curve and its corresponding fast scan are illustrated in Fig. 3. For each identified area a characteristic potential is imposed and an amperometric measurement is made to define the steady state current density corresponding to the imposed potential.

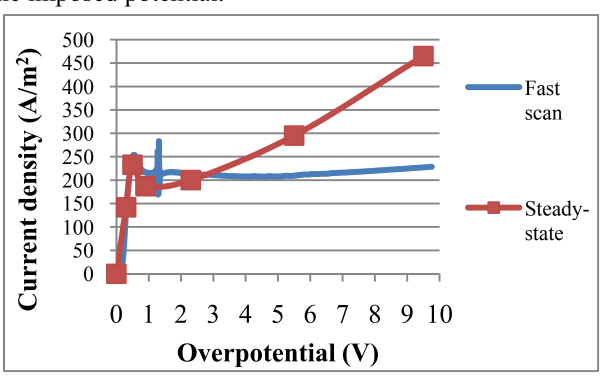


Figure 3: Fast scan (10 mV/s) versus steady state polarization curve. 15°C – Pt-reference electrode – Cu counter electrode – surface ratio 1 - new bath-100 rpm.

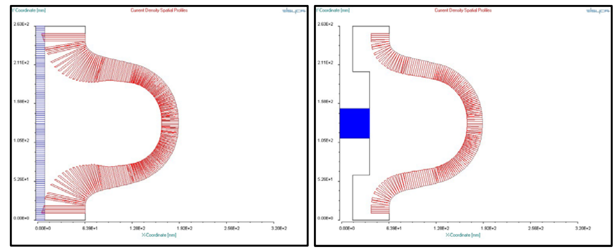
CATHODE GEOMETRY

The cathode design is fundamental for the success of a vertical electropolishing installation. The optimised geometry can be defined with accuracy and in a relative short time using adequate data and simulation software. The results presented hereafter were calculated with real bath parameters and using the ElSyca ElSy2D electrochemical software.

The parameters used were:

- Beta 1 SPL cavity cell geometry
- Steady state polarization curve: 15°C – Pt-reference electrode – Copper counter electrode – surface ratio 1 - new bath-100 rpm
- Bath conductivity: 100 mS/cm

In Fig. 4 are illustrated the current distributions calculated by the simulation software for two different cathode geometries in a single cell SPL beta 1 cavity. In a) a simple copper rod with 20 mm diameter is used while in b) an optimised geometry has been used..



a) b)

Figure 4: Current distribution for two different cathodes geometries.

A profile of current density versus length of the cell gives a better evaluation of the optimization done with the second type of cathode geometry as illustrated in Fig. 5.

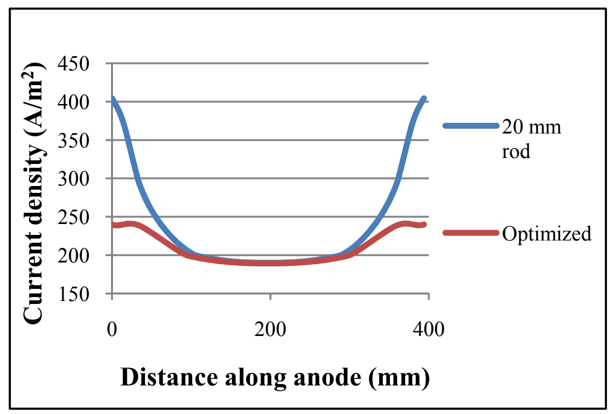


Figure 5: Current density along the cell for a 20 mm rod cathode type and an optimised geometry.

The distribution improves from a ratio of i_{max}/i_{min}= 2.1 to 1.3. Besides the cathode geometry optimization, the simulation software allows a faster understanding of the specific conditions at each point of the cavity and to correlate them with the different working parameters like bath flow and temperature as well as the necessary current and potential to achieve a good electropolishing finishing.

ELECTROPOLISHING FACILITY

The new installation for cavity treatment must answer the technical constraints imposed by a vertical electropolishing concept, but also to safety and environmental directives. The setup is illustrated in Fig. 6.

Working Principle

The electropolishing bath is pumped in a closed loop from a storage tank into the cavity (bottom to top) and back. The bath temperature is monitored inside the storage tank and at the outlet on the top of the cavity; a cooling circuit inside the storage tank compensates the heating of the bath by the input process power. The bath flow is controlled and monitored at the inlet of the cavity. A degassing valve is installed at the top of the cavity in order to remove molecular hydrogen (H_2) as much as possible from the circuit; molecular nitrogen (N_2) can also be introduced inside the storage tank in order to vent H_2 out, but this must be limited to avoid losing free hydrofluoric gas (HF). A secondary pump allows pumping the bath back to the storage tank as gravity flow is not possible. A demineralised water inlet is connected to the circuit in order to rinse the cavity after the electropolishing.

Storage tank bath feeding and draining is done by a single point. The action is defined automatically.

The DC power is supplied to the cavity by external contacts to the cavity surface (anode) and by the cathode which is placed inside the cavity.

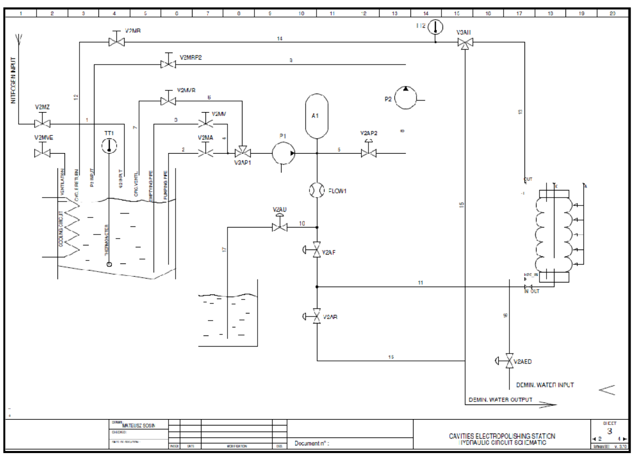


Figure 6: Circuit schematic.

Working Parameters

The setup was designed taking into account two interdependent parameters, bath flow and temperature.

The flow should not be turbulent in any active part of the cavity in order not to disrupt the viscous layer and therefore guarantee homogeneous electropolishing conditions. It should however be sufficiently high to avoid a large temperature differential between the input and the output. Thus, and taking into account the geometry of an assembled cavity and its cathode and respecting a laminar flow, the maximum flow rate shouldn't go over 50 lpm.

A difference in temperature between the inlet and the outlet of the cavity would result in a different polishing rate. A low working temperature is beneficial since it allows for an easier control of the process as it hinders high working current densities. The dissipated power is thus reduced and the heat can then be removed with lower flow rates. This in turn helps in keeping the working current densities at low values.

Materials and Equipment

All piping is made of PVDF PN16 d32 with exception of flexible hoses that are in PTFE PN1.5 1"; valves are in PVDF PN16 d32 with FPM o-rings. Pumps and dampener are made of PFA with FEP o-rings; the main pump and its dampener have a maximum capacity of 50 lpm and the secondary pump of 30 lpm. The storage tank is made of PVDF 12 mm thick assembled by butt fusion jointing and is equipped with a heat exchanger also made of PVDF; the heat exchanger has a nominal power exchange of 2.5 $kW.K^{-1}$. The interfaces between cavity and circuit are also made of PVDF.

Figure 7: Fume hood.

Automatic Control

The system is controlled by a PLC Simatic S7-300 with analog and digital in/out cards. PVSS and Unicos will be used as a supervisory control system installed on a standard PC.

The valves are pneumatically operated with FESTO CPV-10-GE-MP-8 controller and read in their states by PLC with pre-installed end position switches. The main pump is controlled by flow meter feedback. Operation is done via a touch-screen panel.

Safety

The nature of the electropolishing bath restricts the choice of materials in contact as to guarantee the correct operation of the installation and to protect operators from equipment defaults.

To confine the dangers linked to the bath and to the electropolishing process, the entire installation will be assembled in a fume hood ("walk-in booth", Fig. 7). This is connected to the extraction network of the building and to its air washers in order to guarantee the capture of gases and aerosols released by the bath or the process.

The electropolishing set-up will sit on a retention basin to recover any spillage or rinsing water. The recovered volume can be either pumped to a process water treatment station or to a container in case of concentrated products. The storage tank has its own retention tank.

REFERENCES

[1] L. Lilje et al., NIM A516 (2004) 213.
[2] S. Calatroni et al., Proc of the 11th SRF Workshop (Travemunde, Germany, 2003).