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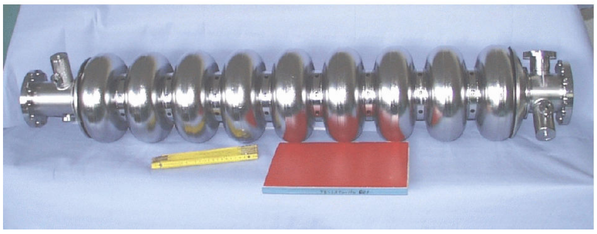

Research and Development on Superconducting Radio-Frequency Technology for Electron Linear Accelerators

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AUTOMATED ELECTROPOLISHING

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Introduction

The development of new forming techniques for the fabrication of seamless TESLA-type cavities requires good control of the Electro-Polishing (EP) process. We have developed a new process for Electropolishing Niobium resonators based on a dynamical automated control that takes into account the surface roughness evolution meanwhile electropolishing. The process consists of a dynamical control of the minimum of differential electrolytic bath conductance extracted from the I-V characteristics (Polarization curve). The obtained results are very good, i.e. a smooth and highly reflecting Niobium surface is obtainable, even by using electrolytes whose composition is not necessarily known. This last result makes the Automated EP tool being particularly interesting, especially when searching for alternative electrolytes, where a large amount of new baths need to be tested.

The Electropolishing Characteristics

Fig. 1 reports a typical I-V characteristic for the electropolishing of Copper in ortophosphoric acid solution in the case of planar and parallel faced electrodes, when edge effects are negligible [Jacquet, P.A., Metal Finishing, 48, 1, 2 (1950)].

The following behaviour is observed at different sections of the polarization curve:

- Over section **Va** and **Vb** the current increases as a linear function of the voltage. The Copper dissolution happens with a too low rate. The process is accompanied by the evolution of Oxygen bubbles sticking to the anode and promoting local pitting of the Copper surface.
- Polishing effect is observed between **Vb** and **Vc**; the Copper structure is brought into relief, as long as the process takes place. However roughness levelling and brilliant surface are obtained at the end of the plateau. Here, at voltages close to **Vc**, there is the minimum of Oxygen bubbles evolution. Even a minimum amount of bubbles can represent a limitation to the achievement of the desired roughness levelling. Migrating toward the top, Oxygen bubbles produce undesired vertical traces depending on the solution agitation.

- At a higher potential the gas evolution becomes stronger and the surface erosion is accompanied by pitting. A better surface quality, although mat, is obtainable at voltages well above Vc, since Oxygen bubbles have not time for sticking to the surface.

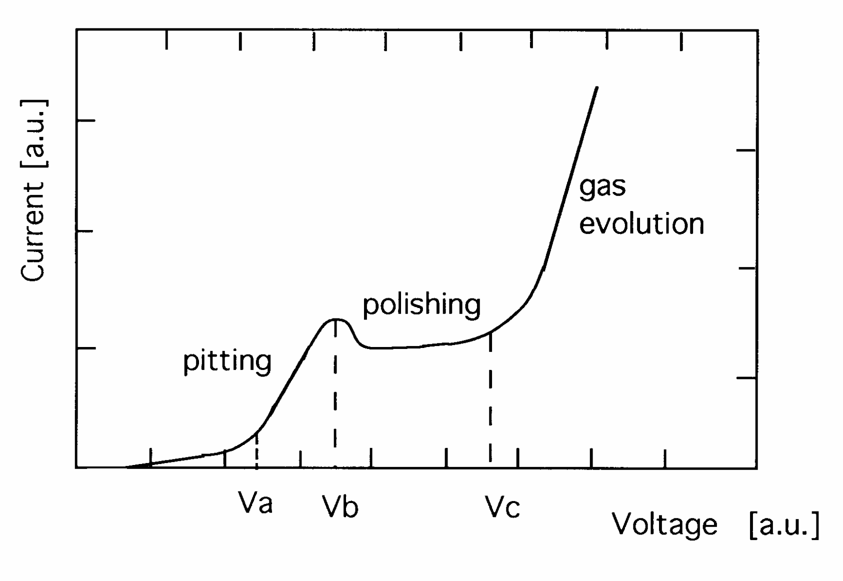


Fig. 1. Typical I-V characteristic for Copper electropolished in Orthophosphoric acid solution.

The behaviour in fig 1 can be however found in many systems metal-electrolytes, even if the plateau will be even extremely narrow, being determined by a minimum of the first derivative of the I-V Characteristics.

Several hypotheses exist for explaining the mechanism of electropolishing in acid solutions. All of them concern with the existence of the thin bluish viscous layer of electrolyte forming in proximity of the anode. The simplest explanation is the one proposed by Jacquet [1]. When a current passes across the electrolyte, the anodic film has higher viscosity and higher electrical resistivity than the bulk of the electrolyte. The thickness of such a film on a rough surface (fig. 2) differs from site to site: above protrusions the film is thinner than above valleys. Hence protrusions dissolve more rapidly than wells.

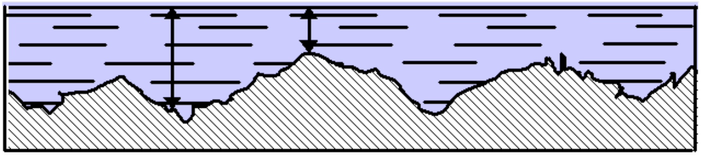


Fig. 2 - The Copper anode coated by the viscous liquid film

The thin layer of electrolyte in the vicinity of the anode assumes a bluish color. Moderate agitation of the solution reduces the thickness of the bluish layer while voltage drops. Vigorous agitation reduces the thickness to a few tenth of a mm and voltage drops even more. Jacquet assumed that a relationship exists between the formation of the viscous layer in the electrolyte and the polishing effect. According to Jacquet, the process should be controlled by maintaining a constant voltage rather than a constant current density; in this case it is not necessary to calculate the total surface area of the sample to be polished. Rigorously speaking, it would be not possible to control the electropolishing process solely on the basis of voltage or current density measurements,

The Applied Voltage **V** is a function of the anode and cathode potentials, the Voltage Drop in the electrolyte ($\mathbf{I}\ \mathbf{R}_{\mathbf{electr}}$) and the voltage drops in the conductors and contacts ($\mathbf{I}\ \mathbf{R}_{\mathbf{Conduct}}$), i.e.

$$V = (V_{\text{anode}} - V_{\text{cathode}}) + I\,\mathbf{R}_{\mathbf{electrol}} + I\,\mathbf{R}_{\mathbf{Conduct}}$$

V depends on the electrode potentials, the electrolyte concentration, the anode and cathode surfaces, the arrangement of electrodes in the bath and the shape and size of the bath. Hence, the "applied voltage” used by Jacquet as a control parameter would not always correspond to the optimum polishing conditions. On the other hand, the potentials of the anode and of the auxiliary electrode do not remain constant - they change as a function of the time of electrolysis and the composition of the electrolyte.

Given that the plateau region in the I-V characteristics gives the best polishing conditions, it is important to examine the effect on the plateau played by the process parameters. The four most important parameters are the electrolyte Temperature, Acid Concentration, Viscosity and Stirring. The temperature does not affect the plateau voltage range, but only the current density; the same holds for acid concentration, the plateau voltage remains unchanged, while the current density increases; the current density is inversely proportional to Viscosity; while stirring increases almost linearly the current density.

The author and his co-workers have proposed one useful technique that makes very easy to find automatically the optimum Electropolishing conditions. The technique consists in locking the minimum of the differential conductance found by numerical derivation of the Polarization curve. The idea is that, since the viscous layer has higher viscosity and greater electrical resistivity respect to the bulk of the electrolyte, by finding the minimum of the differential conductivity of the I-V characteristics, one automatically obtains the right electropolishing voltage. Computer control helps in finding this ideal working point and constantly tuning the process following the evolution of this point.

The I-V characteristic is monitored and controlled by the use of a PLC Fieldpoint programmed in LabView. Working with a PLC gives the big advantage of not loosing the control of the working point during the locking procedure around the minimum of the EP bath differential conductivity. That was indeed the main problem we faced during our preliminary attempts of dynamic control of the EP differential conductivity by a simple PC, due to the fact that standard PCs often interrupt the process just while refreshing so that the dynamic control often is lost. The process is driven in voltage. An automatic program displays the numeric derivative of I versus V. The working point is chosen as the minimum of such derivative, i.e. the minimum of bath differential conductance.

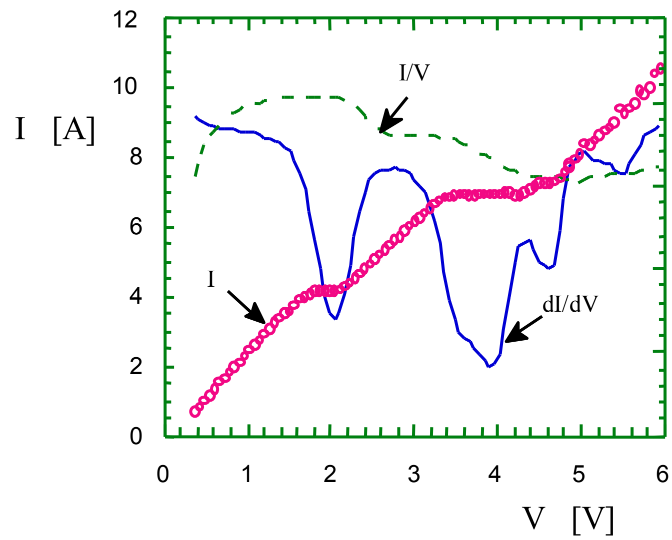


Figure 3: I-V characteristic for a standard electropolishing process (circles); The differential conductivity dI/dV (continuous line) and the ratio I/V (shaded line) are displayed versus voltage. We interpret the first minimum in differential conductance as due to edge effects and to the not uniform distance between electrodes

In fig. 3, we display the differential conductance compared to the ratio I-V and together with the I-V characteristic. Some literature approximates the minimum of dI/dV with the minimum of I/V. Already from fig. 4, it is visible that the two minima differ quite substantially in voltage. In any case that of an electrolytic cell is a non-linear circuit, hence the solution conductivity is a differential quantity while, the ratio I/V has no any physical sense in such a case.

The I-V characteristic evolves with time. Fig. 4 displays the evolution of the current *plateau*.

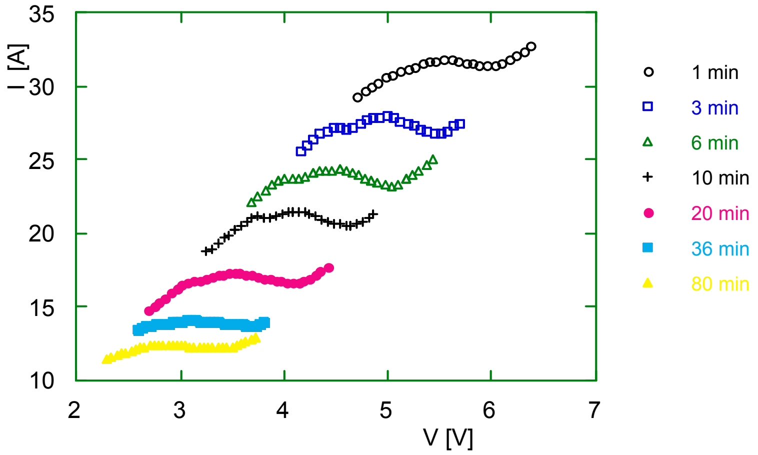


Figure 4: Time evolution of the I-V characteristic plateau

The reason for the plateau decreasing is twofold. The leveling action decreasing the roughness, but also the surface area decreases. Moreover in the hypotheses of an anodic passivating oxide film, the longer is the process, the more stable is the film, the weaker is the etching. By locking the minimum of the differential conductance, we could lower the plateau up to very low values of metal dissolution rate. Hence when the viscous layer becomes too thick, equivalently the voltage becomes too low, the process is reset by a sudden increase of the supplied voltage and by starting the process again from the beginning by plotting the I-V characteristics.

In other words whenever we need to remove hundreds of microns of metal, we use to apply the method of locking the minimum of differential conductance for one hour, than for few minutes we work at a much higher voltage in order to destroy the passivation layer, then we restart locking the minimum for another hour and so on.

At the minimum of the differential conductance there is already low gas evolution. However as soon as the process is started and the plateau starts to get lower, at a certain voltage threshold, gas evolution can even disappear.

We have built a computer program that automatically locks onto the minimum of the bath differential conductance. In this way not only the process is constantly driven according the best parameters, but also we can directly find the best electropolishing current density, without need to know them a priori.

The method suitably applies to whatever metal (Copper, Niobium, Magnesium, Aluminum, Titanium and his alloys, Gold alloys, and many technical alloys), and the operator does even not need to know the electrolyte he his using.

The automated EP Labview program

The rotating cavity EP System built for the Electropolishing cavities is a standard closed loop circuit and it is visible in fig. 5. Monocell and three cell cavities can be easily treated. Copper and niobium cavities can be simultaneously treated. The architecture of the automated EP is sketched in fig. 6. The best working point of the I-V characteristic is searched and dynamically followed by during the whole EP process.

Fig. 5: The rotating cavity EP system built for the treatment of both Niobium and Copper.

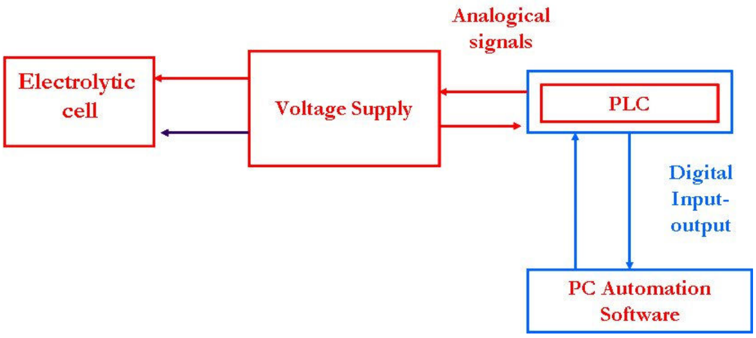


Fig. 6 - Scheme of the automated EP

The apparatus is made of:

- an Industrial power supply controlled by means of an analog current signal;
- a special PLC, the FieldPoint® FP-2010 by National Instruments, that uses the software written by LabView® 7.1;
- The LabView® program written on a computer, and then transferred on the memory of FieldPoint®.

The Hardware consists in a ALINTEL S4000 power supply (100Vmax - 40V max) with remote controller. The 0-10 V applied to the pins at the interface produce 0-100 V in output. The analog readings from the Power supply are converted to digital in a PLC and elaborated by the automation software. The digital outputs from the automation software are converted in voltage signals that drive the Power supply.

The PLC is a LabView® programmable National Instruments Field Point 2010 and it is composed by 2 modules; 1 supply; 1 RS-232 Serial interface; 1 Ethernet 10/100 Mbps. It is possible to drive the process by several PC's connected to the PLC in a network as shown in fig. 7.

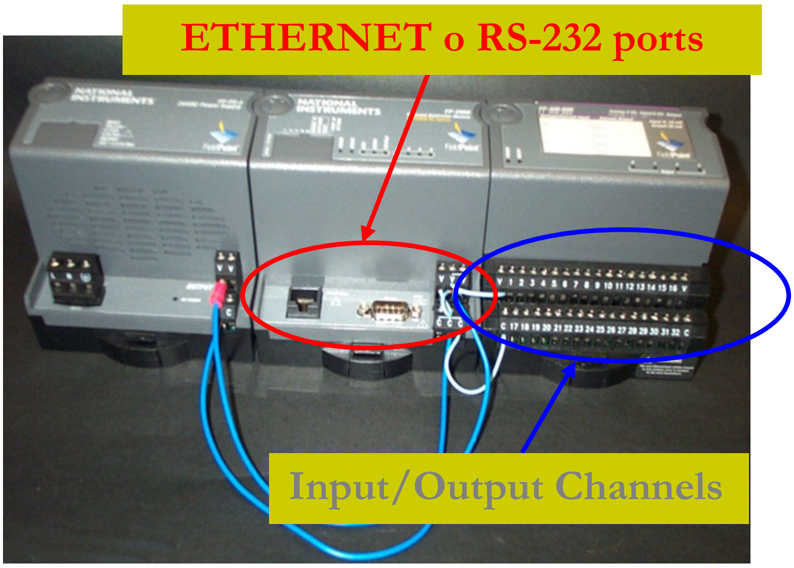
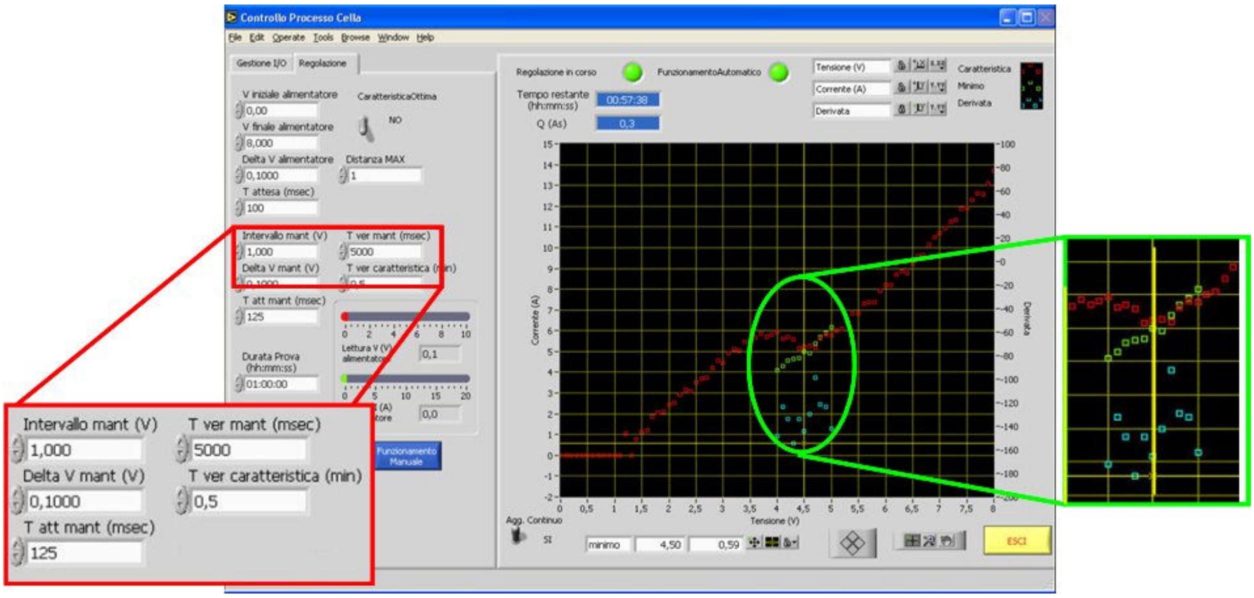


Fig. 7 - The LabView® programmable National Instruments Field Point 2010

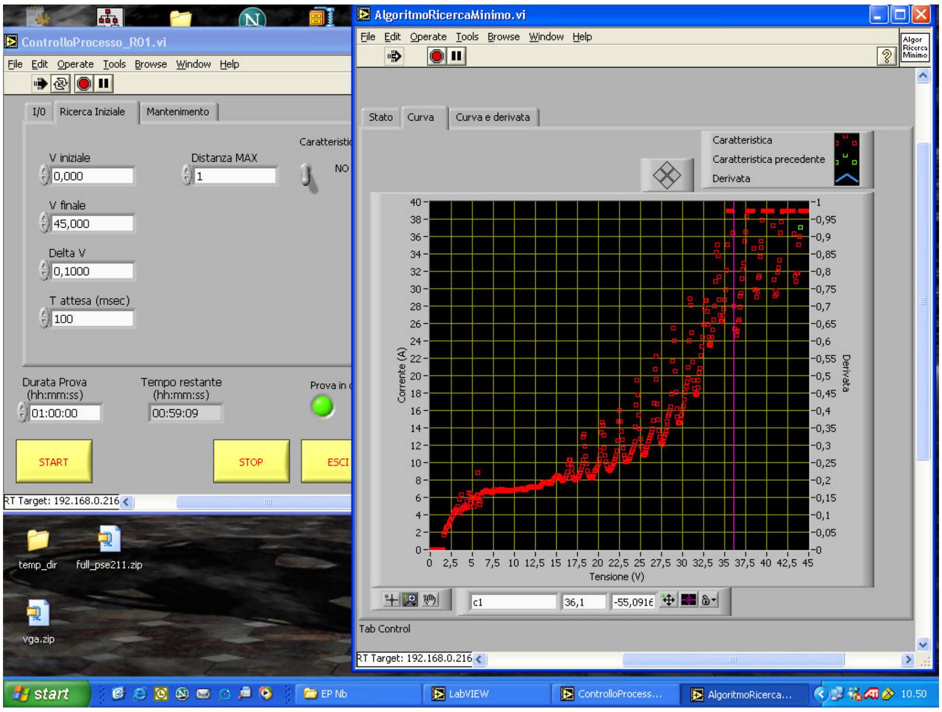
The control software of electropolishing is written in LabView 7.1, that is a graphical language specifically designed for the interface and control the instruments of National Instruments. This language meets a simple graphic interface and a powerful set of function. The core of the program is the algorithm for search and pick the working point. The program initially makes a large scan from two values set by the user. After the scan, the software calculates the derivative of the I-V curve, and sets the voltage that match to minimum point as shown in fig. 8. Then the program maintains the voltage for a time value set by user, after it makes a little scan around the minimum voltage. By recalculating the derivative, the program follows the new voltage minimum. After a time set by user, the program uses the initial data to rescan the characteristic curve. This system allows to the program to adjust the minimum if the working point goes away from plateau. If the point found by program isn't at right place, the user can stop the automatic search and manually sets a new minimum. The program continues the automatic search on the new minimum. All parameters can be changed in real time.



V iniziale alimentatore 0,00
V finale alimentatore 8,000
Delta V alimentatore 0,1000
T attesa (msec) 100
CaratteristicaOttima NO
Distanza MAX 1
Intervallo mant (V) 1,000
T ver mant (msec) 5000
Delta V mant (V) 0,1000
T ver caratteristica (min) 0,5
T att mant (msec) 125
Durata Prova (hh:mm:ss) 01:00:00
Lettura V (V) alimentatore 0,1
I (A) alimentatore 0,0
Tempo restante (hh:mm:ss) 00:57:38
Q (As) 0,3
Agg. Continuo SI
minimo 4,50 0,59

Fig. 8 - *Typical I-V characteristics (red dots) displayed on the computer screen controlling the electropolishing processes. The curve drawn by the blue dots is the derivative of current versus voltage. The numbers on the left are the process parameters; the knobs at the bottom-right are needed for settling the starting point*

The program tested on Niobium, displays the typical oscillations given by the forming and the cracking of the forming oxides and displayed in fig. 9. Oscillation create difficulties on setting the working point, but it is enough to directly start the acquisition of the polarization curve after the oscillation and to search normally the working point on the plateau after the oscillating regime.



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V finale: 45,000
Delta V: 0,1000
T attesa (msec): 100
Durata Prova (hh:mm:ss): 01:00:00
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Fig. 9 – Typical oscillating regime in the I-V characteristics when Electropolishing Niobium

The programme has been successfully used for the Electropolishing of a monocell cavity and it is a unique tool for the development of the research of new Hydrofluoric-free electrolytes for the Electropolishing of niobium. The Automated EP tool has been satisfactorily used by our laboratory and it is available for whole CARE partners working in the WorkPackage 5.3 on Electropolishing.

Acknowledgements

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