ELECTROPOLISHING OF SEAMLESS 1.5 GHZ OFHC COPPER CAVITIES

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Summary   The development of new forming techniques for the fabrication of seamless TESLA-type cavities requires a good control of the electropolishing process. We have developed a method for electropolishing resonators in vertical position without rotating the piece. The process is driven by controlling the differential conductance of the I-V characteristic. Some investigation has been also carried out on the effect of bath temperature on surface finishing quality.

Introduction

Although the mechanism of electrolytic polishing is not yet understood in all aspects, the process is generally considered to include both a leveling action and a brightening action. The modern techniques are based on the work of Jacquet [1] who investigated methods for polishing metals for metallographic examination which would be capable of producing a smooth surface without distorting the crystal lattice, which is inevitable concomitant of the formation of a damaged surface layer due to the mechanical polishing.

During the process products of anodic metal dissolution react with the electrolyte to form at the metal surface a film that can be or gaseous or liquid, in the latter case usually a layer of solution containing a high concentration of salts of the anode material. At least two layers are encouraged to form on the specimen surface: a relatively thick viscous layer that is nearly saturated with the dissolution products, while a thinner film beneath it is responsible for the polishing on a finer scale. A film of anodically discharged gas usually Oxygen is commonly observed to be a blanket on the outside of the viscous film. The film has a higher resistance to the passage of current, and the surface consists of depressed and elevated microareas. Such a film will be thinner on microprojections and thicker over the microdepressions of the metal surface. The high current density areas will thus be dissolved away by electrolysis so that the entire surface of the metal will tend to be flatten out and

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evened out. Polarization effects may also come into play which tend to make the depression relatively cathodically passive and the protrusions anodic, due to the differences in the concentration of the anodic film. More rapid ionic and molecular diffusion through the polishing film may also contribute to the leveling action. Atoms of the metal being removed in electropolishing pass through the polarized anodic film as metal salts and enter the electrolyte, either to be dissolved therein, deposited on the cathode, or to precipitate as sludge.

Current-Voltage relation in electrolytic polishing.

Electropolishing solutions for Copper are often acid-base, however other electrolytes may be alkaline, cyanide or metal salt systems. The I-V characteristics of Copper in these various electrolytes is determined not only by the different composition, but also by the operation temperature, stirring rate and mutual orientation of electrodes.
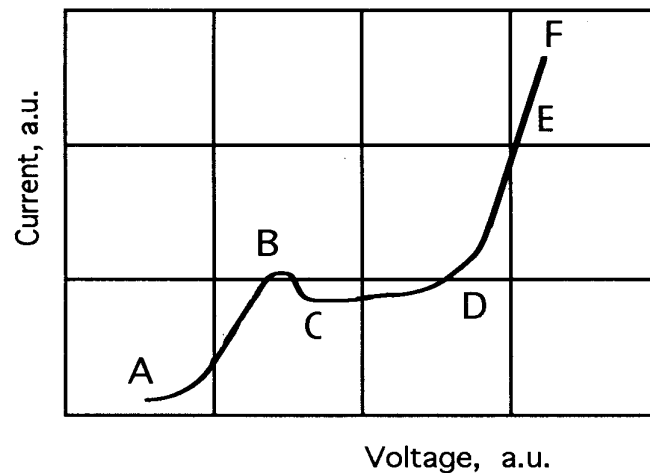

Fig. 1: Volt-Ampere characteristic for Copper electropolished in a phosphoric acid solution. This curve is typical for those electrolytes that form an ionic film (after P.A. Jacquet [1]).

It is well-known that the best results for electropolishing Copper are obtainable with cyanides. However such approach is often unpracticable, because of the highly poisonousness of cyanides and the large quantities of solution needed . Hence we used the classical bath based on phosphoric acid, consisting of 55% Phosphoric acid and 45% n-buthanol. Phosphoric acid is indeed the major constituent in many of the acid type solutions suitable for Copper electropolishing. However more recently organic acid and addition agents are added to reduce many of the disadvantages in the straight acid-type solutions. The I-V characteristics of Copper in phosphoric acid is typical and it indicates

the range of Current and voltages where polishing occurs. Five distinct regions can be distinguished (fig. 1).

In the region A-B current density increases with potential, some metal dissolves, and the surface has a dull etched appearance. The region B-C reflects an unstable condition, while region C-D indicates a stable plateau at which the polishing film, previously formed, reaches a point of equilibrium and polishing occurs; during the latter stage, current density remains constant and optimum polishing conditions occur. This condition takes place as soon as the anodic film growing speed equals the diffusion speed in the electrolyte. The increasing of resistance with thickening of viscous film explains the constant current density versus voltage. The plateau takes places up to when the film is not broken. In the region D-E, gas bubbles evolve slowly, breaking the polishing film and causing severe pitting. Polishing with rapid evolution of gas occurs in the region E-F.

Even a minimum amount of bubbles can represent a limitation to the roughness leveling. Migrating toward the top, bubbles produce undesired vertical traces depending on the solution agitation. The main problem of surface quality lays in avoiding pitting and vertical traces, the former due to gas bubbles stationing on the anode, the latter due to gas migration toward the top. In our experience, whenever appliable and if not too energetic, stirring significantly improves quality of results.

We have developed a method for controlling the electropolishing process, by searching the best working point of the I-V characteristic and by locking on it during the whole process. The process is driven in voltage and a computer monitors the I-V characteristic. 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 in differential conductance of the bath, that corresponds to the point of maximum resistance of the viscous layer.

Temperature effect on surface finishing quality.

Traditionally electropolishing of copper in Phosphoric acid, buthanol solution is performed at room temperature. Some literature [2,3] treating such topic reports that electrolyte temperature should be as low as possible, since high temperature decreases the film viscosity, hence its capability to remain sticked to the anode. The reason for low temperature is said to be twofold: firstly the anode current density is lowered giving a slower and more controllable polish. Second the width of the polishing plateau is greatly increased at low temperatures, making the conditions for polishing easier to find and to maintain. We have found that all this will be maybe valid for other polishing solutions, but not for Copper in a phosphoric acid electrolyte. We have seen indeed that higher

temperatures strongly improve both smoothness and reflectivity of treated Copper. We have electropolished several samples at different electrolyte temperatures: passing from - 5°C to + 60°C, the average roughness R_a^* decreases monotonously from 3000 KÅ to 700 KÅ. Moreover since higher temperature means lower viscosity of solution, the density of pitting due to Oxygen bubbles becomes lower and lower. We saw also that the higher dissolution speed associated with higher temperature, is a benefit in terms of time saving and it does not mean at all that the process is less controllable. Even we saw that temperature practically does not influence the plateau width. Last, the method of locking around the minimum of differential conductance is so sensitive that even if the plateau is very narrow, the working point is automatically followed by computer. We have chosen however a working temperature of 50°C, since at 60°C, it starts forming Copper Pyrophosphate clusters where Oxygen bubbles nucleate.

Experimental technique: the apparatus.

Beta 1 multicell cavities are traditionally made of half-cells EB-welded together. The search for lowering the manufacture costs has recently asked for alternative forming solutions in order to suppress electron beam welds. In particular in the case of Niobium sputter-coated seamless ninecells, electropolishing of the resonator interior is a step that cannot be neglected. The problem has been solved elsewhere by filling half the resonator with the electrolytic solution, disposing the resonator in horizontal position and by rotating the whole piece around the axis. In order to minimize the solution amount, to avoid that the solution for the cavity exterior would get in contact with the interior of the cavity, and in order to not rotate the piece in the acid, we have developed a way to make electropolishing in vertical position without rotating the piece. It is well known that if electropolishing is performed in such a configuration the half cell below the equator, comes out well polished and brilliant. The halfcell above is mat, pitted, strongly oxided and coated by Copper powder. That being caused by the gas evolution that remains trapped in each cell.

Iris is instead the most shining part of the resonator. Starting from this we have noticed that iris is the bottle neck for the escape way of all bubbles nucleated during the process. Just where there is a concentrated dynamic flow of bubbles, there is the best polishing action.

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* The average roughness R_a according to standards DIN 4768, ISO/DIS 4287/1 is defined as the arithmetic average among all values of the roughness profile within the evaluation length L_m,

$$R_a = \frac{1}{L_m} \int_0^{L_m} |y|\, dx$$

Hence we have built a cathode as sketched in fig. 2, in which acid is forced by a pump up to a nozzle made by two planar disks in correspondence of the equator. The pumping speed must be regulated so that the solution reaches equator helping bubbles to be carried out.
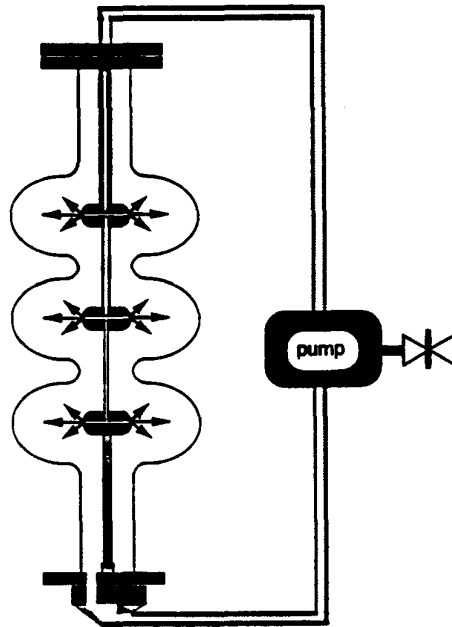

Fig. 2   Sketch of the apparatus for electropolishing seamless resonators. The acid solution is sprayed against the cavity walls.

The *plateau* in the I-V characteristics is easily visible. Locking on the minimum of differential conductance gives as a result a very shining and brilliant surface all over the resonator. Such a method has been successfully applied also to seamless Niobium monocells in lactic acid solutions.

Acknowledgments

References

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