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COMPARATIVE CHARACTERISATION OF SURFACES STATES AFTER CHEMICAL AND ELECTROCHEMICAL TREATMENTS.

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Abstract

This report describes several approaches aiming at a convenient characterization and comparison of surface morphology of niobium after surface treatments. These techniques can be applied to niobium samples in order to optimize the electropolishing process: thanks to numerical estimation of various parameters (optical quality, gloss, roughness and topology parameters) we can easily compare samples surface and rank them. First results already show that slight modifications of the electropolishing conditions have a large impact on the achieved surface states.

INTRODUCTION

This report stands for the Work Package WP5-1 of the CARE SRF Joint Research activity of the sixth framework program. This work package is dedicated to the improvement of the electropolishing process through optimization of the electrochemical bath composition in regards to the achieved surface states. In a first step this optimization program is conducted on samples and will be then adapted to a mono cell test bench. If this work appears/remains conclusive, it would be a valuable tool available for the industrial preparation of cavities for the TESLA project.

Optimization of surface treatment requires that we have an efficient way to compare surface states obtained when varying the etching conditions. Unfortunately it is impossible to tests every change on real cavities, so we have to find out a set of parameters which will give an estimation of the surface state, even if we still don't know which one is the more accurate for RF applications. Several possibilities exist: optical comparison of photographs, roughness and topologic analysis of the surface morphology, and gloss estimation. There are some tools which help to traduce these parameters into relative figures, convenient for comparison.

SURFACE MORPHOLOGY ANALYSIS

Surface roughness and topology analysis

Although roughness is not relevant to predict RF behavior due to local relief, it gives valuable information about the existence of preferential etching at grain boundaries or etching pits. A smooth surface is also known to retain less dust contamination, and thus is highly desirable.

Mechanical microscopy and roughness analysis as well as topologic analysis of the data with conformal equivalent structures have already been described elsewhere [1]. Meanwhile acquisition of the data requires a sophisticated apparatus (mechanical microscope), and a work station for the calculation of the 1rst to 4th order momentum (i.e. the classical roughness parameters: Ra, Rq, Sk, Ek), the developed surface, and the parameters of the ellipsoids from the conformational equivalent structures. These tools are very useful for the analysis of the internal state of cavities [1] but have showed to give rise to heavy procedures uncompatible with quick evaluation of surface state. Anyway, roughness (σ_{rms}) measurement made on several samples exhibit values about 1-2 µm, which is higher than values generally found in literature [2, 3] for electropolished samples where $\sigma_{rms} \sim 0.5$ µm. This might be in relation with the large measured area in our case ($5mm^2$), and shows once more that EP gives rise to smooth surface only at small scale. At larger scale, large variations appear.

Therefore we have concentrated on simpler tools more likely to help us for a rough, but rapid evaluation of the surface state.

OPTICAL OBSERVATION

Direct observation of samples can be made all along the etching process with an optical microscope (Leica DMRM) and a gloss-meter (Labomat Refo-3D). Both are standard laboratory light equipment, very convenient, and relatively not expensive.

Optical microscopy

We have taken photographs at various scales with various electropolishing (EP) parameters (elapsed time and/or removed thickness, bath composition, anode-cathode distance, see table 1 for examples). Comparison of samples made in close conditions only based on visual evaluation is difficult. But the

quantification of qualitative data like photographs can be done with the help of Müdge grid. This technique is based on the comparison of data, two by two, and quotation, according to a simple rule:

If A >> B, then A = 3 and B = 0
If A > B, then A = 2 and B = 0
If A ~ B, then A = 1 and B = 1 ("good ")
If A ~ B, then A = 0 and B = 0 ("bad")

Then each score is added up and one gets a numerical evaluation of each data. Application of this technique to the photographs from table 1 is shown in table 2, along with some other data. See also figure 3..

Gloss measurement

Gloss measurement is a standardized method once dedicated to the evaluation of paints and describes the capacity of a surface to reflect directed light. In general determine it by the photoelectric measurement of specularly reflected light from the surface in accordance with international standards (ISO 2813, ASTM D 523, DIN 67530). The intensity of the reflected light is dependent on the angle of illumination and material properties, but also on the measuring optics (dimensions of the light source and receptor aperture, receptor sensitivity, calibration of the instrument by comparison to a standard). In particular angle of observation are very important: 20° is well adapted to high gloss material (B_{60} > 100), while 60° fits better for less glossy material. As etched sample are sometimes very rough we have systematically measured the two orientations [4].

Anyhow, this simple measurement does not take into account additional phenomena like haze or scattered light. Like roughness, it is only a partial description of the surface state and needs to be completed by other means.

Experimental

We have systematically studied electropolishing of samples versus time in various conditions: voltage, anode-cathode distance, and fluorhydric acid concentration (40% or 46 %). For every other parameters we are close to the classical EP as described in [5] for instance : the bath composition is HF 1 volume- H_2SO_4 9 volumes, the temperature is 30°C ± 2°. The ratio of Nb surface to the volume of bath as well as to the surface of cathode are close to the ones used for cavities (see e.g. [6]). The experimental set-up is described on figure 1.

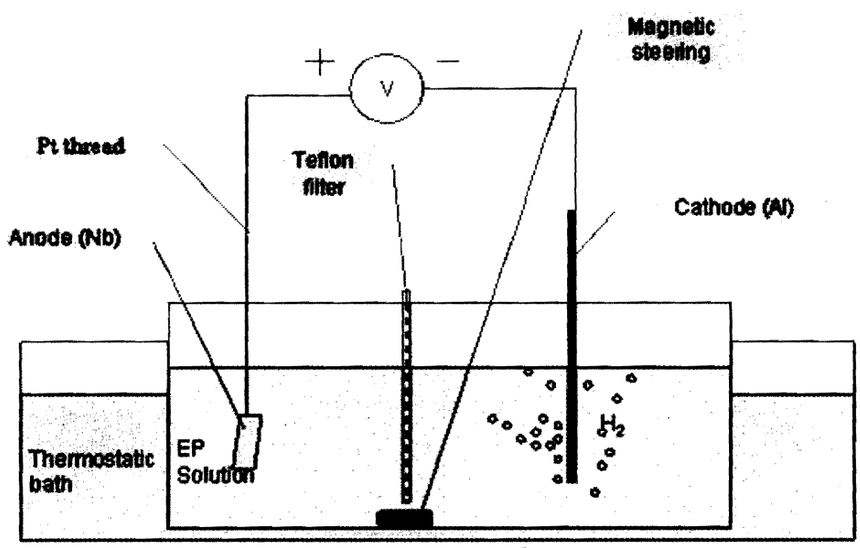

Figure 1 : experimental Set-up

Table 1 : summary of data for two different anode-cathode distances.

1HF(46%)-9 H_2SO_4 (95.5), V= 16 V									
$d_{anode\text{-}cathode}$ = 5.5 cm					$d_{anode\text{-}cathode}$ = 2.5 cm				
Removed thickness (µm)	Gloss		Original micrographes magnification		Removed thickness (µm)	Gloss		Original micrographes magnification	
	B20 =	B60 =	X200	X500		B20 =	B60 =	X200	X500
0	10	56		0*	0	11	61		0*
58.5	137	301		12*	41.6	78	194		6*
122.6	421	466		40*	95.7	133	255		30*

**Müdge "score" (MS). NB in this serie of tests the highest score reaches 40*

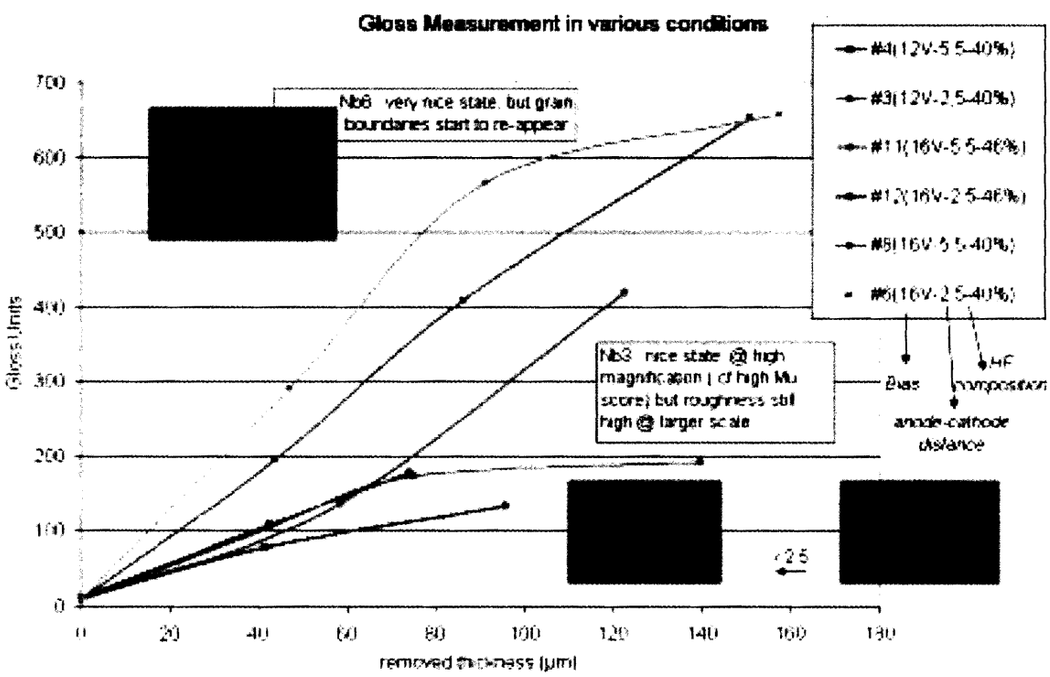

Figure 2: Gloss measurement for a series of EP in various conditions

We notice a continuous improvement of the surface at least during the first 100 µm removal, an improvement which is also observed for cavities. After 150 µm, in some cases we observe a stabilization of the results, as well for gloss than fore Müdge scoring (see figure 3). Obviously further experiment are needed in order to know if heavy EP leads to a degradation of the results on samples as well on cavities. This point is very important for the future finishing procedure of cavities preparation.

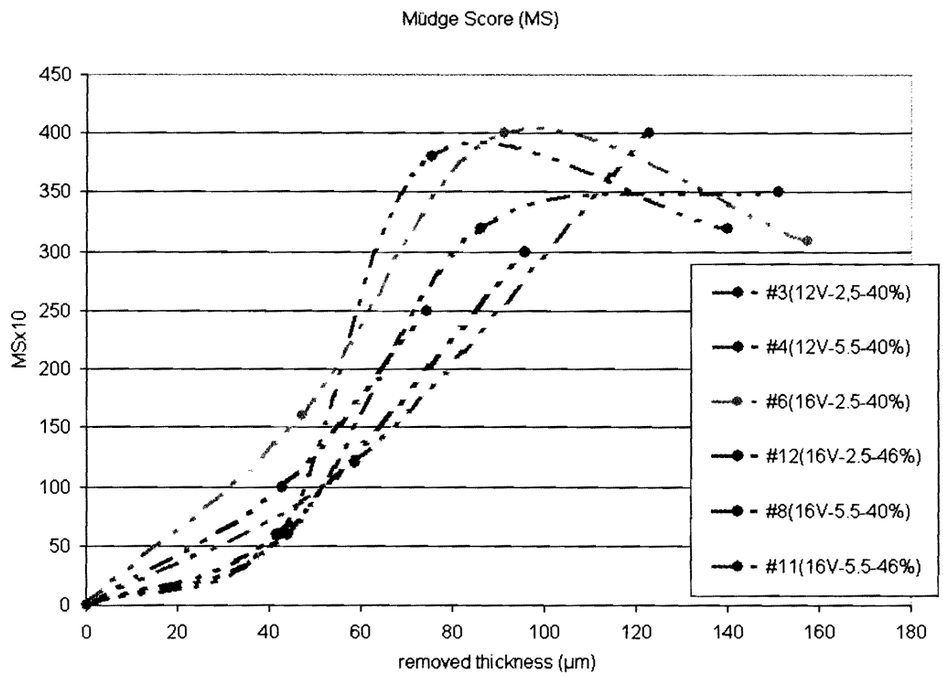

Figure 3 Müdge score for the same series of experiment (color refers to the same sample)

CONCLUSIONS

Obviously, gloss measurement is a very convenient and effective tool for comparative evaluation of surfaces, but it is mostly influenced by microrouhnes. Localized defects can hardly be detected this way. For instance, at large removed thickness, some grain boundaries etching tends to reappear, but hardly influences the gloss (at least for "good" samples with 100-150 µm removed). Photographs or visual inspection is still necessary to complete this information. More experience is needed for larger material removing. We can already infer from this first set of results that heavy etching might be harmfull, and that slight changes in EP condition (cathode-anode distance, bath composition) have a large influence.

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