

PLASMA ELECTROLYTIC POLISHING AS A PROMISING TREATMENT REPLACEMENT OF ELECTROPOLISHING IN THE COPPER AND NIOBIUM SUBSTRATE PREPARATION FOR SRF*

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Abstract

Superconducting radio frequency (SRF) cavities performances strongly depend on the substrate preparation. Currently, the conventional protocol of SRF surface preparation includes electropolishing (EP) as the main treatment achieving low roughness, clean and non-contaminated surfaces, both for bulk Nb and Cu substrates. Harsh and non-environmentally friendly solutions are typically used: HF and H₂SO₄ mixture for Nb, and H₃PO₄ with Butanol mixtures for EP of Cu. This research is focused on the application of a relatively new technique “Plasma Electrolytic Polishing” (PEP) for the SRF needs. PEP technology is an evolution of EP with a list of advantages that SRF community can benefit from. PEP requires diluted salt solutions moving to a greener approach in respect to EP. PEP can in principle substitute, or completely eliminate, intermediate steps, like mechanical and/or (electro) chemical polishing. Thanks to the superior removing rate in the field (up to 3.5 μm/min of Nb, and 10 μm/min of Cu) in one single treatment roughness below 100 nm Ra has been obtained both for Nb and Cu. In the present work a proof of concept is shown on Nb and Cu planar samples.

INTRODUCTION

The PEP technique was described for the first time in 1979 [1], but only in recent years it has really gained the interest of industries and research institutes. Dental implant, multi-metal, alloys, and semiconductor polishing are only some of the current fields of applications of PEP and different recipes were developed to apply PEP on stainless steels alloys, aluminium, titanium, and others [2-4]. Theoretically, the PEP technology can polish any metal structure and presents several advantages compared to other polishing methods. In particular, PEP uses low-concentration salts solution electrolyte, so no harmful gases are produced. Moreover, PEP is a very fast process capable to generate a good surface quality, with no mechanical stress and no thermal distortion or damage [5]. Despite these advantages, there are still very few works in literature and hardly any on the treatment of copper and niobium, the two materials of most interest in the SRF field.

From EP to PEP

The process setup of PEP is quite similar to the standard electropolishing: the metal part to be polished is immersed into the electrolyte and connected to the positive pole of

the power supply (anode). A second electrode is connected to the positive pole and works as a cathode (see Fig. 1).

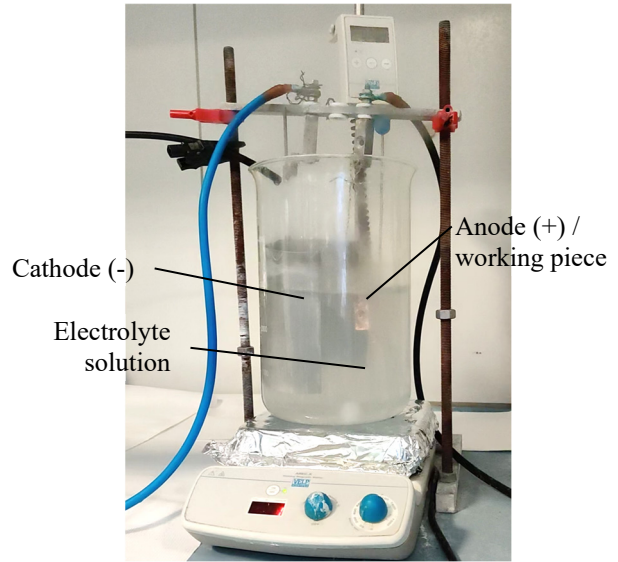


Figure 1: Chemical stand for the PEP study used at LNL.

The two principal differences between EP and PEP are a higher voltage regime and an electrolytic solution with a low conductivity. In particular, this last point is one of the advantages of PEP, especially in the case of Nb. The electrolytes are based on an environmentally friendly solution of various salts, normally in concentration range of 2-10 %. No HF and H₂SO₄ are necessary for the Nb polishing anymore. Moreover, the PEP solutions are less viscous than EP ones, thus simplifying the process plant requirements.

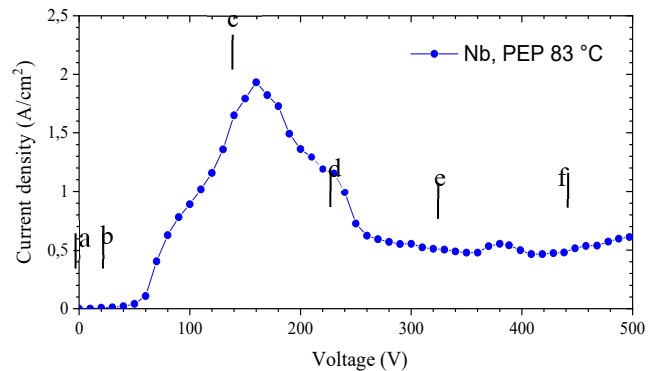


Figure 2: Current-voltage characteristics of Nb in PEP electrolyte developed at LNL (T=83 °C).

Figure 2 shows a typical current vs voltage curve obtained in this work for Nb. The general behaviour is quite

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similar for all the metals, even for, the so called “valve metals” as Nb and Ti, due to the fact that their high resistive oxides are more “tricky” to polish by PEP compared to “non-valve metals” as Cu and Fe. A review on the difference and challenges for PEP in valve metals is presented in [6]. The first section of the curve “*ac*” is the usual electrolysis process, described using classical electrochemistry. The conventional electropolishing occurs in the first part of the curve “*ab*” and is well described in [7]. The second section of the curve “*cd*” can be called transition mode, in which an unstable vapour-gaseous envelope (in some literature is called plasma-gas layer) is formed around the anode. The unstable vapour-gaseous envelope (VGE) produces a non-uniform contact between sample and electrolyte with consequent current instability. The VGE becomes stable in the zone “*de*” in which PEP takes place. The typical PEP voltage values for Nb and Cu are around 300 V, generally between 200 and 350 V. Increasing the voltage we enter zone “*ef*” in which the current slowly increases, strong arcing takes part, and the plasma becomes unstable.

Mechanism of PEP

Regarding the PEP mechanism, there are two main points in which the scientific community have not found a common agreement, yet: the description of the VGE conductivity and the polishing mechanism [8]. About the VGE conductivity one of the first theories proposes VGE as a water-vapour film in which the high electric field produces gas ionization [9]. The responsible of the conduction are the metal ions from the anode and electrolyte ions. Other theories [10] emphasize that the current density is too high to be only ionic and suggest the formation of a glow discharge in which conduction is also contributed by electrons. Furthermore, there are other authors that instead propose a non-homogeneous thickness of the VGE with consequent formation of “electrolyte bridges” [11] or “streamer discharges” [12] in which an electron avalanche produces ionization and a consequent conductive channel in the VGE that connects the anode surface peaks/defects with the electrolyte.

The polishing mechanism is not fully understood, and this is a second main point in which there is not fully agreement in the literature. In the streamer theory, the principal mechanism of polishing is the local melting caused by an increment of the temperature in the workpiece in proximity of the channels, due to the plasma discharge. A further increment of temperature produces then a gas explosion that removes the melted metal, levelling the surface. A similar mechanism is related to the glow discharge model proposed by Vaňa et al. [10]. In this model, the discharge melts the anode/workpiece surface where the VGE thickness is lower, firstly removing the peaks and producing a very rapid smoothing effect. However, it should be underlined that the role of the electrolyte is essential, since a mirror-like surface is achievable only with the proper electrolyte. We mention that there also exist some works that do not involve melting to describe the PEP mechanism and use only electrochemical considerations to explain how the VGE layer leads to a rapid peak dissolution.

Risk Assessment Comparison

Processing with a PEP technology can open a new greener and safer way, if compared to the conventional EP and BCP in the case of Nb. As it was mentioned before, those solutions contain concentrated Sulfuric, Phosphoric, Nitric, and Fluoridric acids. The working conditions are extremely difficult, since in both cases hazardous solutions produce harmful gases. Moreover, the proper disposal of exhausted solutions is very expensive, due to the toxicity and hazardousness of the solutions themselves, and it significantly impacts the cost of the process. Finally, the use of strong corrosive agents also increases the cost of the plants and limits the number of compatible materials for their construction.

These reasons are not only pushing the SRF community to look for alternative solutions for the polishing of accelerating cavities since several years [13, 14], but are of more general interest for all industrial applications where the polishing of metal parts is required- in particular for those metals such as Nb, Ti, V, Ta, which tend to form chemically very stable oxides. The PEP using aqueous solutions of salts in a relatively low concentration, may be an answer to this problem.

EXPERIMENTAL

A series of planar samples of Nb RRR300 and OFHC Cu were used, with ~ 9 and 7 cm^2 area, respectively. Before each polishing treatment, a proper degreasing with ultrasound and commercial soaps was applied. The discharge was sustained by two power supplies ITECH IT6018C-500-90 (of 16 kW each) connected in parallel. 3 L of solution were used as electrolyte, and heated and stirred by a commercial magnetic stirrer (see Fig. 1).

The surfaces were studied by visual inspection, linear profilometry by Veeco Dektak 8 and SEM micrography by Coxem CX-200 plus. The roughness evaluations were done on 1 mm distance, with at least 3 different position of the measuring lines, arithmetic (R_a) and root mean square of the surface roughness (R_q) values were obtained during these scanning. The removing rates are calculated with the double weighting method.

RESULTS ON NIOBIUM

The Nb dissolution in case of EP or chemical buffer polishing (BCP) is always conducted in the presence of fluorhydric acid, that converts niobium oxide into soluble fluoride compounds, permitting further removal processes. In PEP, an alternative solution must be used because of the high conductivity of the HF. In literature, at least one work on Nb polishing by PEP is already present [15]. However, from the article, it is difficult to evaluate the final surface quality to compare it with standard Nb EP. Moreover, the process reproducibility appeared to be an issue: in our test no polishing of Nb was possible with the recipe described.

An original solution for Nb PEP polishing (currently under patenting evaluation) has been developed at LNL. The solution is simply composed by a salt dissolved in water, like most of the PEP solutions reported in literature. The

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solution can perfectly polish pure Nb in PEP regime producing a smooth surface even after few minutes of treatments. In the next paragraphs results on bulk Nb are described more in detail.

Fast Polishing Comparison (BCP, EP, PEP)

To compare the ability of the different polishing techniques to smooth rapidly the Nb surface, tests on 4 samples obtained from the same Nb RRR300 sheet were done.

Table 1: PEP, EP, BCP Comparison

Parameters	BCP (1:1:2)	EP (1:9)	PEP
Solution	HF:HNO ₃ :H ₃ PO ₄	HF:H ₂ SO ₄	Diluted salts
Voltage	-	18 V	300 V
Current density	-	0.025 A/cm ²	0.4-0.6 A/cm ²
Power density	-	0,45 W/cm²	150 W/cm²
Temperature	15 °C	30 °C	78 °C
Treatment time	5 min	20 min	2 min
Thickness removed	6.5 μm	6.5 μm	7 μm
Removing rate	1.3 μm/min	0.3 μm/min	3.5 μm/min

In Table 1 the parameters used for each sample are reported. What is immediately evident is the very high power density required by PEP (150 W/ cm²) and the erosion rate of PEP: 3 times faster than BCP and 10 times faster than EP. Even more outstanding is how the PEP polishing mechanism acts mainly on peaks and allows to cut the initial roughness more than 50% in just 2 minutes of treatment. In 20 minutes EP reduces roughness less than 30% (see Fig. 3). The visual comparisons confirm the roughness characterization. In Fig. 4 it is visible how all the treated samples appear already shining after 7 microns of removed material, but only on PEP surface the image reflected is well defined.

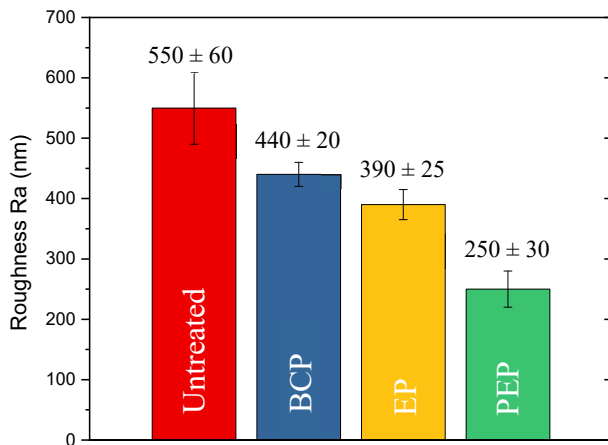


Figure 3: Roughness data values Ra of Nb samples after removing ~ 6-7 μm with 3 different polishing techniques.

In Fig. 5 SEM micrographs of all 4 samples are reported. The scratches present in the initial surface are removed by all polishing techniques in the 7 microns of treatment. On

BCP we have preferential etching on certain grain orientation as expected. On the EP treated sample, lamination lines of the Nb sheet still remain visible. The PEP sample instead, is already perfectly smooth and no preferential etching is present

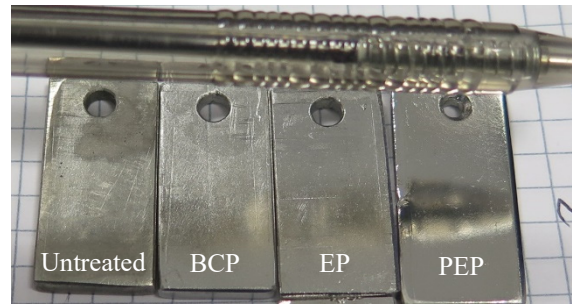


Figure 4: Visual comparison of the Nb surface after fast polishing (7 microns removed) with 3 different techniques. Starting from left: initial surface, BCP, EP and PEP.

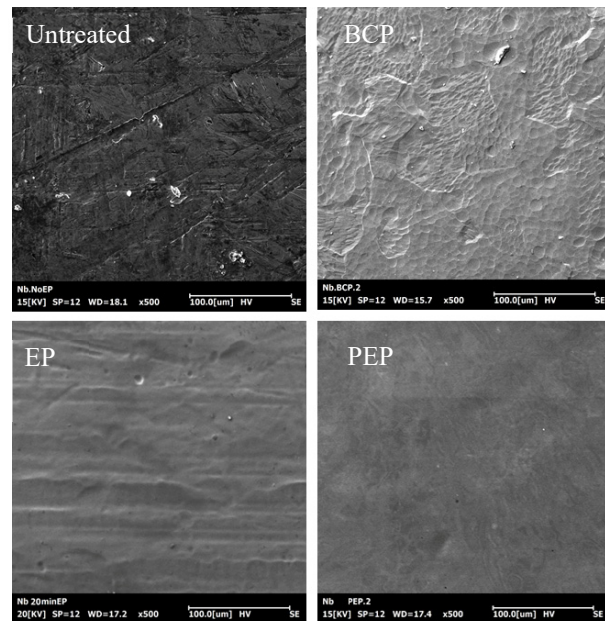


Figure 5: SEM micrographs at same magnification level the Nb surface after fast polishing (7 microns removed) with 3 different techniques. Starting from upper left in clockwise direction: initial surface, BCP, PEP and EP.

Long Polishing Comparison (EP, PEP)

In common cavity preparation protocols as EXFEL [16] 150-200 microns of Nb are removed during the polishing process in order to eliminate the whole damaged top layer during the different forming processes. The PEP removing rate remains stable with time and in less than 1 hour 150 microns were removed. While the EP process takes almost 10 hours. In addition, the roughness of the PEP sample decreased to 70 nm (see Fig. 6), as the EP sample has a roughness of 200 nm. The SEM micrographs (Fig. 7) support the profilometric characterization: microroughness and grain boundaries of the EP sample appear to be much more prominent than those of the PEP sample.

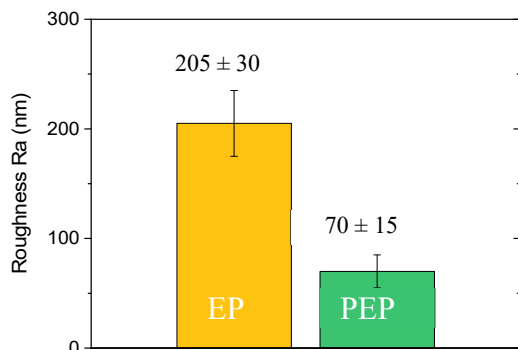


Figure 6: Roughness data values Ra of Nb samples after removing ~ 150 μm with EP and PEP.

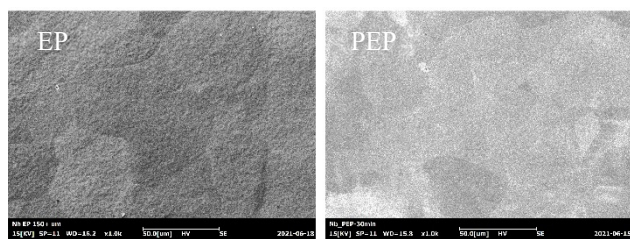


Figure 7: SEM micrographs at same magnification level of the Nb surface after long polishing (150 microns removed) with EP (on the left) and PEP (on the right).

Shape Factor

The removing rate seems to depend on the geometry of the anode. In the same set-up and parameters, an erosion rate that varies from 1,5 up to 6 μm/min depending on the surface shape was obtained. It is still unclear, how precisely the removing rate can vary depending on the shape. The lower polishing rate (and best finishing) has been obtained with cylindrical anodes/working pieces. We speculate that on cylindrical shapes the VGE can be more uniformly covering the surface rather than on flat samples, in which the sharp edges are going to be smoothed and rounded, due to the higher local current densities and discharges, as well as in the EP.

RESULTS ON COPPER

The current recipes of Cu surface polishing were standardized years ago, and since then, nothing has changed significantly. The removing rate values of EP on Cu do not exceed 0,5 μm/min in the non-agitated mode at 25°C with a current density of 0,01-0,03 A/cm². The values can of course vary, depending on the cathode/anode distance, working temperature, and solution composition (for e.g. organic moderators can decrease current density, due to their non-conductivity).

The SUBU solution developed by CERN [17] is a good tool in case of complex and big surfaces to polish the surface. Its removing rate can reach 1,5 μm/min, but it decreases dynamically with the time, depending on the saturation of the working solution. At LNL it is used as a finishing step after EP of 6 GHz copper cavities, to ensure more uniform polishing across the surface. According to

the authors, the mechanism of polishing is similar to the EP, by means of forming a viscous layer during the processing.

The copper PEP is not a common task in the field, that is why there is a limited quantity of materials regarding it [18-21]. Currently, we have developed two solutions, that we are evaluating for the patenting. On this paper, however, all the results reported only refer to one solution. Removing rate can reach up to 23 μm/min in some cases. Longer treatments are decreasing it down to 10 μm/min, most likely due to the degradation of the solution.

Fast Polishing Comparison (SUBU, EP, PEP)

Similar to Nb, a comparison between PEP and traditional SUBU5 and EP has been done. The used parameters are reported in Table 2.

Table 2: SUBU5, EP, PEP Comparison

Parameters	SUBU5	EP (3:2)	PEP
Solution	H ₂ NSO ₃ H 5 g/l; NH ₄ -citrate 1 g/l Butanol 50 ml/l; H ₂ O ₂ 50 ml/l;	85 % H ₃ PO ₄ : n-Butanol	Diluted salts
Voltage	-	2-6 V	300 V
Current density	-	0.01-0.03A/cm ²	0.3-0.5 A/cm ²
Power density	-	0,12 W/cm²	135 W/cm²
Temperature	72 °C	25 °C	70-80 °C
Treatment time	5 min	30 min	2 min
Thickness removed	7.5 μm	8 μm	8 μm
Removing rate	1.5 μm/min	0.2-0.5 μm/min	3-10 μm/min

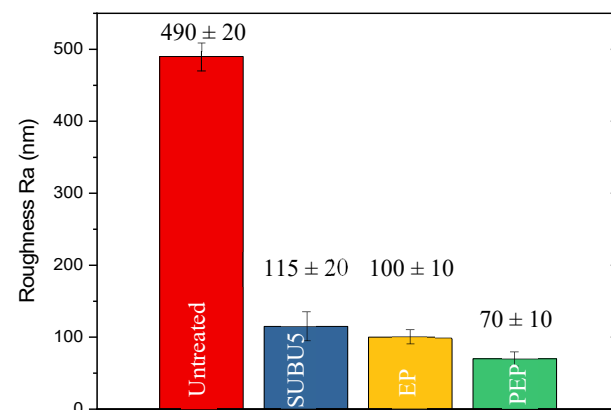


Figure 8: Roughness data values Ra of Nb samples after removing ~ 6-7 μm with 3 different polishing techniques.

The results on copper confirm the trend seen on Nb. The PEP presents an erosion rate 10-20 times higher than EP depending on the process conditions and 2-4 times higher than SUBU5. Regarding the smoothing ability, the superiority of PEP is clearly visible from the roughness measurements (Fig. 8), where PEP is the only technique to present values below to 100 nm Ra (70 nm). However, since EP and SUBU5 are still able to achieve roughness of 100 nm Ra, visual inspection (Fig. 9) and SEM

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micrographs (Fig. 10) show similar morphologies, especially for PEP and EP. It is however appreciable to a trained eye, the greater smoothness of the surface treated with PEP. The SUBU5, in general as all chemical polishing techniques, emphasizes the different grain orientations, but less than BCP for Nb.

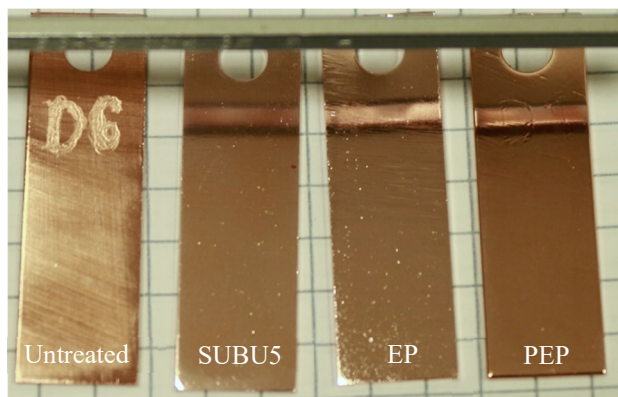


Figure 9: Visual comparison of the Cu surface after fast polishing (8 microns removed) with 3 different techniques. Starting from left: initial surface, SUBU5, EP and PEP.

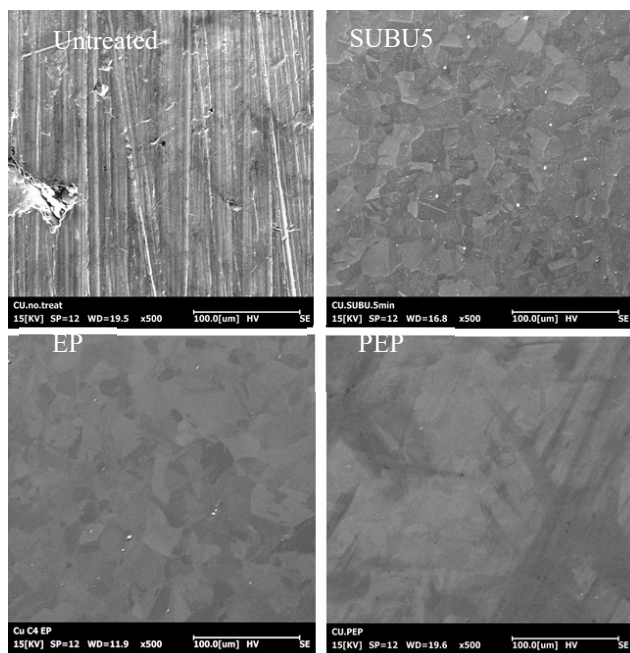


Figure 10: SEM micrographs at same magnification level of the Cu surface after fast polishing (8 microns removed) with 3 different techniques. Starting from upper left in clockwise direction: initial surface, SUBU5, PEP and EP.

Long Polishing Comparison (EP, PEP)

In Figs. 11 and 12 the results for 2 different copper samples are reported. Here 200 and 170 microns, respectively by PEP and EP, were removed. The roughness of EP sample still remains around 100 nm as was obtained after 8 microns (see Fig. 8). For PEP sample a remarkable value of Ra 40 nm was obtained. However, it is proper to report the presence of pitting on a small portion of the

sample treated by PEP. The EP sample instead is pitting free.

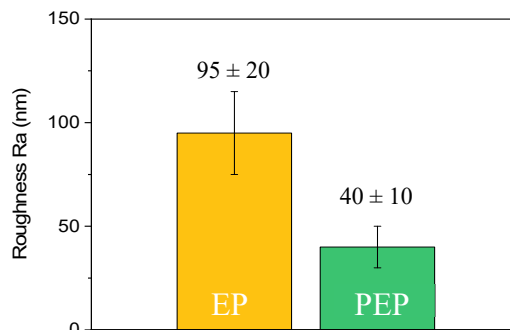


Figure 11: SEM micrographs at same magnification level of the Cu surface after long polishing with EP (170 microns removed) and PEP (200 microns removed).

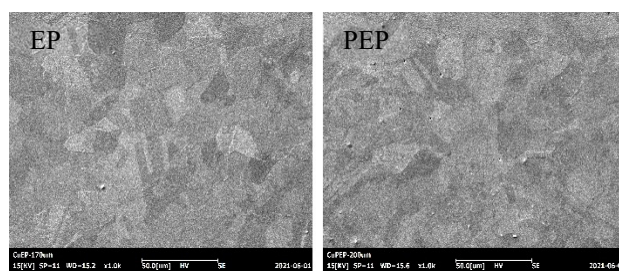


Figure 12: SEM micrographs at same magnification level of the Cu surface after long polishing with EP (170 microns removed) and PEP (200 microns removed).

Comparison with Literature PEP Solutions

There are at least 6 reported solutions for copper PEP in literature [18-21], but any work specifies the minimum achievable roughness. From our tests, the best solution was found to be the one described in [20] based on $(\text{NH}_4)_3\text{PO}_4$. The polishing is good, but it is not possible to obtain the EP mirror like finishing typical of EP and SUBU (see Fig. 13). The roughness after 12 microns removed is 180 microns, twice as the PEP solution developed by LNL. The removing rate, on the other hand, is half of the LNL solution.

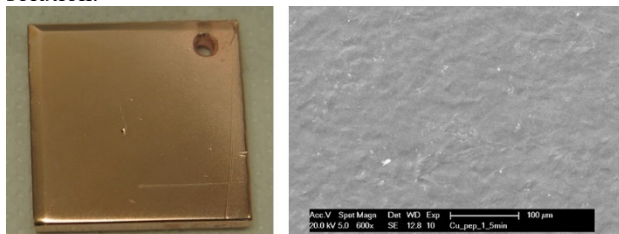


Figure 13: Cu sample treated by PEP with recipe described in [19]. On the left a picture of the treated surface, on the right a SEM micrograph. With this solution only a matte surface is achievable.

Jet Polishing

An attempt of Jet polishing study was done, in the framework of PEP scalability to large areas as accelerating cavities. The set-up used was simple and it is visible in Fig. 14 for a copper planar sample. The surface to be treated is

placed at positive potential (anode), while the cathode (negative) is an aluminum ring inserted in the tube that sprays the electrolyte solution directly to the anode surface, closing the electric circuit. The solution is recycled using a PTFE membrane pump. The same set-up has been used on a half-cell 6 GHz with a noticeable improvement of the surface (see Fig. 15), confirming the possibility of a successful PEP jet polishing on Cu surface. At the moment, jet polishing on Nb has not been successfully tested, yet.

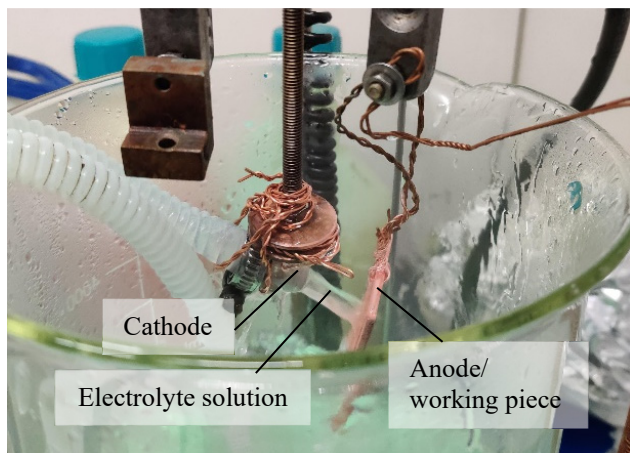


Figure 14: Jet polishing set-up.



Figure 15: 6 GHz half-cell treated by PEP jet polishing. On the left the cavity before the treatment, on the right the cavity after PEP jet-polishing.

BATH TEMPERATURE

The solution temperature is an important process parameter to consider. With a low temperature bath, the working piece can reach temperatures above 400 °C (see Fig. 16) according to the literature [19]. Moreover, higher solution temperatures lead to lower current densities (see Fig. 17) and lower surface roughness, according to our experience.

The best polishing results are obtained with a solution temperature around 80-90 °C. Above 90 °C, the boiling process has a significant impact on the operation of the process, thus we have avoided this regime.

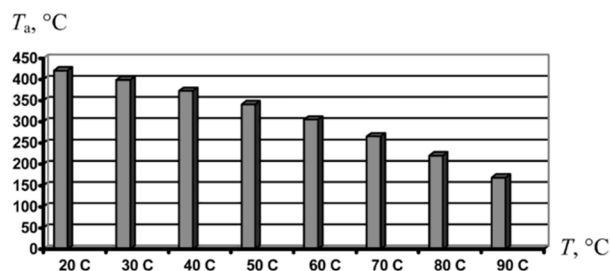


Figure 16: Anode temperature depending on the working temperature under 250 V [19].

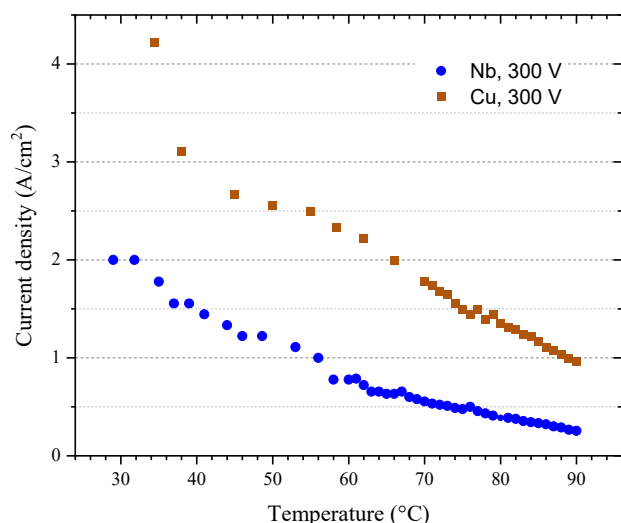


Figure 17: Current density depending on the bath temperature for Nb and Cu.

SCALABILITY TO LARGE AREAS

The complex scalability of the PEP process to large areas is definitely very challenging. The advantages of handling dilute salt solutions instead of solutions with large concentrations of hydrofluoric and sulfuric acid, the very high erosion rate and the excellent surface finish, conflict with the high-power demand and large amounts of gas (hydrogen released at the cathode and electrolyte vapor) to be removed during the PEP process.

If we think of treating a 1.3 GHz single cell cavity, in a simple horizontal rotating configuration (with only half a cell immersed in the electrolyte), the power required to sustain the PEP discharge is about 20 kW, scaling the parameters used in this work. A nine-cell elliptical cavity becomes difficult to achieve under these conditions. However, we believe that there is still plenty of room for improvement on the process. To reduce the power request there are several possibilities yet to be explored: pulsed PEP, raising the bath temperature to reduce the current density, or even lowering the working voltage after triggering the plasma as proposed in [19]. Another possible

way is the jet polishing of which in this work is reported a proof of concept on copper. In particular, we believe that this configuration may be the more appropriate for closed and complex surfaces such as multi-cell cavities.

CONCLUSION

A novel, eco-friendly polishing technology for niobium and copper has been demonstrated at the planar sample level.

The PEP technique has shown to have a superior surface finishing and a higher erosion rate compared to the classical polishing techniques for copper and niobium. A mirror-like finishing is achievable in just a few minutes and for long treatments, the roughness Ra decrease below 100 nm.

The scalability to large areas such as 9 cell elliptical cavities, is extremely challenging and further optimization and exploration of alternative set-up as jet-polishing are mandatory before evaluating this possibility. However, PEP can be already taken into consideration as alternative of standard treatments for the polishing of small parts such as high frequency cavities, couplers, the QPR samples and more generally any metal components in which low roughness is required.

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