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APPLICATION OF PLASMA ELECTROLYTIC POLISHING ONTO SRF SUBSTRATES*

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Abstract

A new promising approach of SRF substrates surface treatment has been studied - Plasma Electrolytic Polishing (PEP). The possible application of PEP can be used not only on conventional elliptical resonators, but also on other components of SRF such as, for example, couplers or the Quadrupole resonators (QPRs). However, SRF application of PEP represents a challenge since it requires a different approach to treat the inner surface of elliptical cavities compared to electropolishing. In this work, the main problematics and possible solutions, the equipment, and the polishing system requirements will be shown. A proposed polishing system for 6 GHz elliptical cavities and QPRs will be shown and discussed.

INTRODUCTION

The PEP processing is a potent method to treat metal surfaces for various purposes: fast cleaning, derusting, polishing, surface preparation for the film deposition, etc. It is wide use for the dental implants treatment field [1]. Thus, being significantly known in some particular industrial fields, the technology it is not well studied and requires additional investments for even more potential benefits. The scalability from the laboratory technology to the needs of SRF is not a trivial task. The possibility of Nb and Cu PEP has been already demonstrated for SRF and published earlier [2], and further studies are being shown in detail [3]. The aim of this work is to share the obtained experience regarding the application of the PEP technology for the SRF needs with the community. A list of possible setups is discussed.

EXPERIMENTAL SETUPS

All the processes were carried out at the facility of Materials Science and Technology Service for Nuclear Physics at Legnaro National Laboratories (LNL) of INFN. The PEP is a technology, that is very similar to EP in terms of the equipment requirements: DC power supply, two electrode system (anode and cathode), the electrolytic solution and connection. However, additional requirements are necessary, such as more powerful power supplies, with high current and high voltage output. At LNL, two DC power supplies of 16 kW, 500V, 90A each, are currently available. They are connected in series, to gain additional current values.

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The different polishing object may require different setups, that is why this study is aimed to demonstrate the comparison and basic schemes of possible setups during PEP processing.

Generic Laboratory Setup "EP-like"

The "EP-like" setup was developed and currently is operation at LNL due to its simplicity and convenient usage in the laboratory environment. It consists of a 5 L beaker, a metallic stand (for electrical connection), and a custom cathode (planar, cylindrical, mesh-type shape). The connection is done similarly as in EP, where the electrodes are defined according to the polarity of the DC power supply (negative – cathode, positive – anode, a sample). The process is carried out at 60℃ with an agitation by magnetic stirrer to improve heat dissipation. The average temperature of the solution is constantly controlled, to avoid low or too high temperature regime. This setup can treat relatively small pieces, depending on the metal nature and composition of the solution (up to 70 cm^2). A scheme of the current setup is shown below (e.g. Fig. 1). An Additional cooling for continuous treatments is necessary and it can be achieved by using jacketed beakers or nitrogen fluxing.

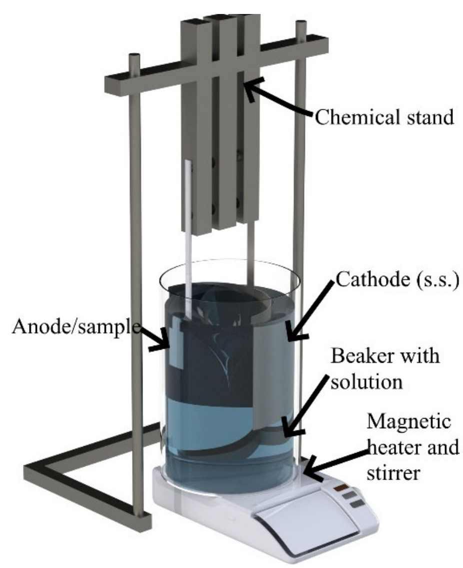

Figure 1: Generic laboratory setup "EP-like".

PROS: Simplicity, fast installation, low volume of solution allow faster studies of possible solution composition. Faster pre-heating. Glass transparency permit observation of the discharge process.

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CONS: A significant disadvantage is the limitation of the sample dimensions, lower volume can significantly decrease time of a treatment, so additional cooling is necessary.

Generic Industrial Setup "EP-like"

The main difference from the previously described laboratory setup is the size of the working bath: it can be increased by a significant value, without limitation (e.g. Fig. 2). Moreover, the bath is convenient to produce in 316L stainless steel that will be chemically stable with most of PEP solutions publicly available. A second role of metallic bath is a cathodic role inside the electrolytic process. Such a setup will not have any problem of lower cathode surface area. It is convenient to have a thermo-heating element inside the bath, and a water cooling coil. It is also necessary to have a dedicated aspiration due to the high-boiling process, and thus gas formation.

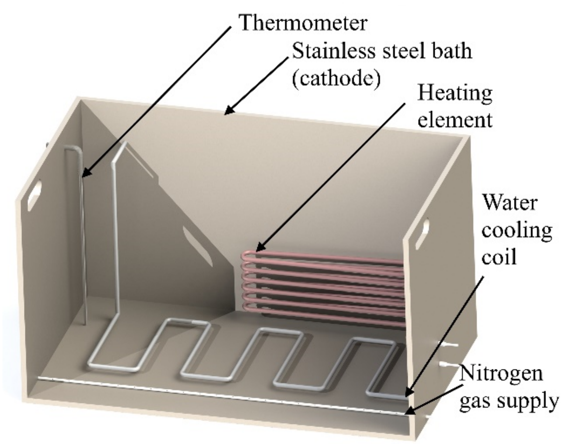

Figure 2: Industrial setup for PEP.

PROS: Much higher values of surface area are possible to treat, multiple samples co-polishing (including different metals) of any shape (excluding holes and caves).

CONS: Once the setup is made, it is fixed for one solution, so the study work is not convenient to perform. No possibility to control and observe process.

Jet-PEP Setup

In literature, a positive experience of application of PEP is presented as a technique to treat pipe-like samples (e.g. Fig. 3) [4]. One of the most efficient ways is a Jet polishing, that founds some attention, and possibly can be applied in any cylindrical shape objects. At LNL a simplified setup for the proof of concept (e.g. Fig. 4) was produced.

The experimental setup is based on the same principle of PEP polishing, but the plasma is ignited during jet fluid stream of an electrolytic solution onto the samples surface from the gun (polishing head). The function of this gun is to provide the solution and negative charge supply. The polishing speed (removing rate) share parameters dependencies of conventional PEP, with the addition of flux speed and distance between cathode and anode.

PROS: A working solution to treat inner surfaces. Application of the fluid can be local, so it can be applied on defective zones.

CONS: The setup is less common, and the quantity of articles on this topic is limited.

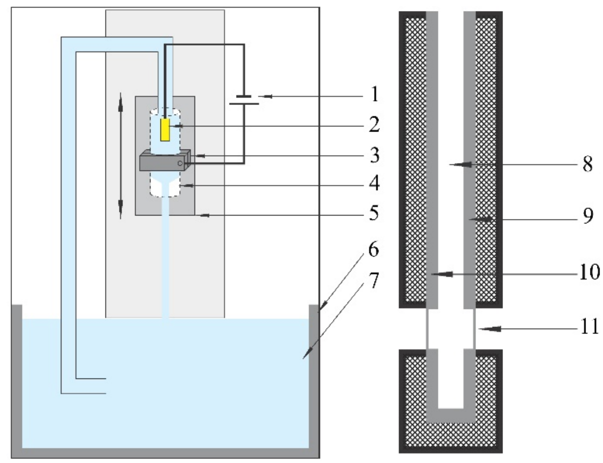

Figure 3: Proposed design of inner surfaces by Jet PEP [4]. 1 – DC power supply, 2 – polishing head (cathode), 3 – tube clamping, 4 – tube (anode), 5 – spindle drive with vertical axis, 6 – basin, 7 – electrolyte, 8 – fluid, 9 – metal core (cathode), 10 – isolator, 11 – adjustable polishing head gap.

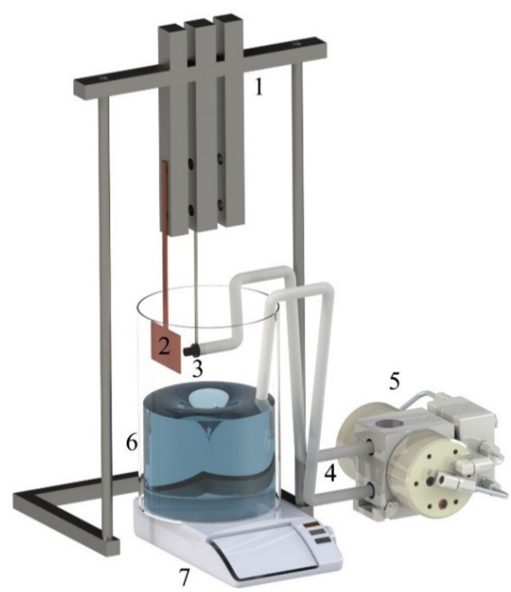

Figure 4: Jet PEP setup build at LNL; 1 – chemical stand, 2 – anode (treated sample), 3 – spraying head (cathode), 4 – in and out tubes connection, 5 – PTFE pump, that flux the solution onto the anode surface, 6 – beaker with electrolytic solution, 7 – magnetic stirrer and heater.

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Double Cathode Setup

A double cathode setup was suggested by S.I. Bahayeu [5] as a possible way to polish internal surface of the tubes (for instance elliptical cavities). In case of a low cathode : anode ratio plasma envelope may change to cathodic heating mode. This configuration can solve this issue. In this configuration, two cathodes are connected both negatively to the power supply outlet. The external cathode role is to dissipate the heat and current flows. The inner cathode is placed inside the tube sample, centred accordingly (e.g. Fig. 5).

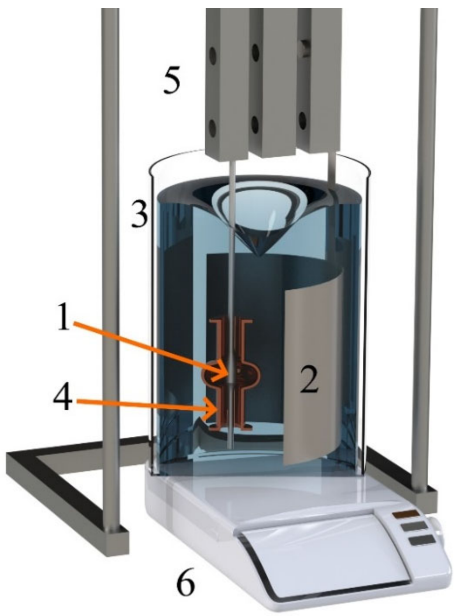

Figure 5: Double cathode setup for PEP of cylindrical objects; 1 – inner cathode, 2 – external cathode, 3 – beaker with electrolytic solution, 4 – 6 GHz cavity, anode, 5 – chemical stand, 6 – magnetic stirrer and heater.

PROS: May permit inner surface cleaning in classical baths setups, easy to project.

CONS: May work not-uniformly across the inner surface. Temperature control may be an issue during the processing. Temporary covers for external surface may be needed to ensure only inner processing and decrease power output values. Centring components are needed, and connection to DC power supply is slightly complicated.

RESULTS & DISCUSSION

he aim of this work is to develop a reproducible and easy to operate setup for the PEP of 6 GHz and 1.3 GHz cavities, and the QPR samples. Basically, it is an engineering question since the possibility and the state of art is already presented. We have collected our library of successful and less successful experiments. We are continuing our research for two paths: a further solution and sample PEP studying and its optimization, and the development of a specific equipment to perform PEP of SRF workpieces. The brief resume of these results and a discussion is shown below.

Samples Discussion

At LNL it was conducted a series of polishing, including different solutions and sample shapes, since 2020. Currently, we can polish almost any piece with a relative surface area up to 100 cm^2. It can be increased, but a proper cooling is required. Overall, a variety of planar samples were successfully treated including Nb and Cu metals. A half-cell Cu 6 GHz cavity was successfully polished with a Jet-polishing setup [3]. The cylindrical shape samples were studied as well: both a Nb and Cu/Nb QPR samples were polished in the laboratory setup at LNL (e.g. Fig. 6).

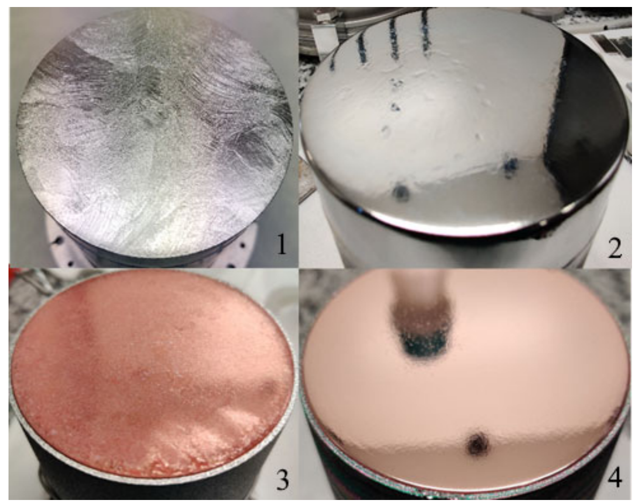

Figure 6: The QPR samples polished with PEP; 1 – Nb sample, previously treated by BCP, 2 – Nb Sample after PEP, 3 – Cu/Nb QPR sample, 4 – Cu/Nb QPR sample PEP.

Process & Setup Parameters

Even though the aim of this work is to focus on the equipment during PEP processing and requirements, all the parameters have a significant value on the dynamism of the process and the final result of the treated surface.

The temperature plays a significant role in the PEP processing, since it has a valuable impact on the surface quality and uniformity (lower temperatures lead to higher current densities flows) and may initiate electrochemical process instead of a plasma processing. It can also limit the time of treatment in the desired temperature working window. Based on our experience, a stable temperature between 75-85℃ is preferable both for Nb and Cu. Moreover, for Nb processing it was found that lower temperatures might be responsible for salt/oxide production during PEP. Lower temperatures are also decreasing uniformity and stability of plasma discharges. The current densities (and most likely removing rates), as well, are connected with the working temperatures (e.g. Fig. 7).

The cathode / anode surface area ratio is suggested to have a 1:10 ratio [6], in order to avoid discharges on the cathode, instead of treated surface of anode, as well as not to obtain cathodic heating. A possible way to increase the ratio is to use cathode with enlarged areas values (high roughness, mesh system).

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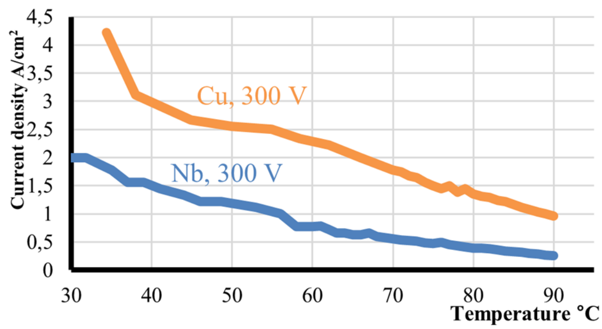

Figure 7: Current density and temperature chart for the developed solutions at LNL of Cu and Nb PEP.

The working distance still requires additional focus from the community. It was found that closer than 2 cm distance can provoke direct discharges between electrodes through a non-PEP mechanism, ruining the surface as a result, causing oxidation or local melting. This phenomenon can be avoided by a proper agitation of the solution and controlling the distance between electrodes. Contrary to EP, in the case of PEP the distance does not play a limiting role [7].

The working voltage. Depending on the nature of the treated metal and the used solution, the working voltage window may be wider or thinner. For Cu we managed to reobtain a plasma envelope at 260-350 V range, and for Nb it is slightly tighter window of 280-330 V. Generally, the process can be started with a higher working voltage, and then decreased steadily without negative effect on the surface quality [8].

The current density, similarly as voltage, can differ depending on the conductivity of the selected solution and metal nature. Generally, 0,2-0,6 A/cm^2 became a standard window [7]. However, in case of copper, we have received a slightly higher values of 0,3-1 A/cm^2 in some cases. The current density is in direct connection with the solution temperature, as shown earlier.

The chemical composition plays a significant role in the PEP processing, allowing certain reactions, or modifying pH of the solution. The Main components of the PEP solutions are metal and ammonium-based salts, that are the source of ions inside the solution and, as a result, a certain conductivity value. However, it is also known the usage of weak and strong acids in a tiny quantity (such as citric, phosphoric, lactic etc) [6]. Anions of sulfuric acid is generally suitable for the variety of metals and alloys, mostly for stainless steel. It is also suggested to add moderators and surfactants [7], that can possibly improve the final polishing results, similarly to EP. Additives can be alcohols, organic salts, organic viscous compounds similarly like in EP. There are two options for dissolution of metals: a) a conversion into insoluble compound, b) dissolution into soluble complex-agent compounds. Our developed solutions are using these principles, in case of Nb – formation of a white insoluble and partially soluble yellow compound. In case of copper, only soluble compounds were noticed.

CONCLUSIONS

It was shown the main setups of PEP equipment adapted for study, semi-industrial processing, and possible configuration for some specific shapes were shown. The obtained solutions and modes among which specifically developed setups, can benefit SRF community providing powerful, ecological modern way to treat SRF substrates.

Additional studies are still necessary in case of Jet processing.

FUTURE WORK

Further studies are scheduled for the implementation of the already developed solutions and recipes of PEP onto elliptical and non-elliptical complex shapes SRF substrates surfaces, in particular: 6 GHz, 1.3 GHz cavities, and the QPR samples. The main goal is to achieve comparable substrate surface within simplification of a conventional protocols. JET-PEP requires additional studies as a main perspective way to treat internal surface of elliptical cavities. Possible collaborations and the community impact can contribute to the application of PEP as technology of choice inside for SRF.

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REFERENCES

[1] H. Zeidler, F. Boettger-Hiller, J. Edelmann, and A. Schubert, “Surface Finish Machining of Medical Parts Using Plasma Electrolytic Polishing”, *Procedia CIRP*, vol. 49, pp. 83–87, 2016. doi: 10.1016/j.procir.2015.07.038

[2] E. Chyhyrynets, “Plasma electrolytic polishing technology as a modern, powerful, and green way to prepare surfaces for SRF”, presented at the 9th Int. Workshop on Thin Films and New Ideas for Pushing the Limits of RF Superconductivity, virtual conference, Mar. 2021, paper 7973. https://indico.jlab.org/event/405/contributions/7973/

[3] C. Pira *et al.*, “Plasma Electrolytic Polishing as a promising treatment replacement of electropolishing in the copper and Niobium substrate preparation for SRF”, presented at the 2021 Int. Conf. on RF Superconductivity (SRF'21), virtual conference, July 2021, paper THOTEV06, this conference.

[4] M. Cornelsen, C. Deutsch, and H. Seitz, “Electrolytic Plasma Polishing of Pipe Inner Surfaces”, *Metals*, vol. 8, no. 1, p. 12, Dec. 2017. https://doi.org/10.3390/met8010012

[5] S. Bahayeu, private communication, Apr. 2021.

[6] K. Nestler, F. Böttger-Hiller, W. Adamitzki, G. Glowa, H. Zeidler, and A. Schubert, “Plasma Electrolytic Polishing – An Overview of Applied Technologies and Current Challenges to Extend the Polishable Material Range”, *Procedia CIRP*, vol. 42, pp. 503–507, 2016. doi: 10.1016/j.procir.2016.02.240

ISBN: 978-3-95450-233-2 ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-SUPTEV002

[7] P. N. Belkin *et al.*, "Mechanism and technological opportunity of plasma electrolytic polishing of metals and alloys surfaces", Appl. Surf. Sci. Adv., vol. 1, p. 100016, 2020. https://doi.org/10.1016/j.apsadv.2020.100016

[8] I. S. Kulikov *et al.*, *Electrolytic Plasma Processing of Materials*. Minsk: Belarusskaya Nauka, 2010, ISBN: 978-985-08-1215-5 (in Russian).