INVESTIGATION OF AN ALTERNATIVE PATH FOR SRF CAVITY FABRICATION AND SURFACE PROCESSING

O. Hryhorenko^{1†}, C. Z. Antoine², T. Dohmae³, F. Brisset⁴, D. Longuevergne¹
¹Université Paris-Saclay, CNRS/IN2P3, IJCLab, 91405 Orsay, France
²Université Paris-Saclay, CEA-IRFU, 91191 Gif-sur-Yvette, France
³High Energy Research Organization (KEK), 1-1 Oho, Tsukuba, Ibaraki, Japan
⁴Université Paris-Saclay, CNRS-IN2P3, ICMMO, 91405 Orsay, France

Abstract

The preparation of SRF cavities includes a lengthy, costly, and safety issued electrochemical polishing (EP or BCP) step to remove the damaged layer coming from the cavity fabrication. We have shown that most of the damage layer is originated from the rolling process during the preparation of the sheet material, while subsequent deep drawing tends to leave only um thick damage layer. We propose a 2-steps mechanical process that allows us to easily get rid of the thick damage layer on the sheets before cavity forming. The process has been established on samples and extended to large disks ready for 1.3 GHz half-cell forming. The polished sheets will be then sent to KEK for half-cell forming and subsequent surface and material analysis before proceeding to half-cell welding. Former studies on the sample demonstrated that damages induced by forming can successfully be removed by recrystallization and less than 10 µm final chemistry.

INTRODUCTION

During the production of the superconducting radiofrequency (SRF) cavities made of Niobium (Nb), the inner surface is significantly damaged. To recover the surface properties and obtain optimal performances in term of quality factor and accelerating field, a layer of 150-200 μ m has to be removed [1]. Regarding the order of magnitude and comparing it with the value of the standard surface processing, we can conclude that the damaged layer is caused mainly by the preparation of 3.5 mm sheets from 1 cm diameter rolls (rolling process), see Fig. 1. The following cavity forming steps (deep-drawing, EB welding) leave significantly thinner damaged layer [2].



Figure 1: Finite element simulation of rolling process [3].

Nowadays, buffered chemical polishing (BCP) and electropolishing (EP) are the main techniques used for surface processing. However, the increased demand for the number of SRF units for the future large-scale accelerators (FCC, ILC), see Fig. 2, requires higher performances, higher reliabilities, and higher repeatability to initiate the construction. For such ambitious goals, traditional surface processing needs new strategies, due to issues with safety regulations, environmental impacts, and limitations in the quality of the final surface state (level of roughness, remaining defects),



Figure 2: Number of SRF cavities versus year [4].

Metallographic polishing (MP) is a possible candidate as an alternative surface processing not only for bulk Niobium, but also to prepare the substrate for thin film deposition. Thin films seem to be a future path of SRF due to their potential to show higher performances, whereas the bulk Nb approaches his theoretical limit. This new surface preparation technique has the potentials for cost savings. smooth surface preparation, and improved reliability and repeatability of the process, compared to the standard treatment of the bulk Niobium. However, instead of polishing complex geometries, metallographic polishing can only be applied on flat surfaces. Hence, based on such limitations we have to perform the polishing on Niobium sheets before forming. An alternative path of SRF cavity fabrication is thus required and is composed of several steps as depicted in Fig. 3.

MOPFDV001

 ^{*} Part of this work supported by the European Nuclear Science and Applications Research-2 (ENSAR-2) under grant agreement N°654002.
 † hryhorenko@jjclab.in2p3.fr

20th Int. Conf. on RF Superconductivity ISBN: 978-3-95450-233-2

SRF2021, East Lansing, MI, USA JACoW Publishing ISSN: 2673-5504 doi:10.18429/JACoW-SRF2021-M0PFDV001



Figure 3: Sketch of the cavity fabrication.

SURFACE PROCESSING

2 Steps Metallographic Polishing Preparation

Metallographic polishing has been performed on commercial polishing devices manufactured by the French company LAM PLAN [5], see Fig. 4. The devices are automatic machines with a rotating sample holder and a rotating disk with different diameters giving possibility to polish the samples up to ø150 mm (MASTERLAM 1.0) and up to ø350 mm (MM 9100).



Figure 4: Photographs of a polishing machine MASTER-LAM 1.0 (left) and a polishing machine MM 9100 (right).

The 2-steps recipe was developed on MASTERLAM 1.0 with ø 300 of disk. The protocol consists of the following steps:

Step1: Rigid composite disk (RCD) in combination with a $3\mu m$ polycrystalline diamond (dia.) as an abrasive to remove the damaged layer after fabrication.

Step 2: Microporous polyurethane cloth in combination with a 50-nm silica colloidal abrasive dilution (20% in H_2O) with a high pH=10 to recover SRF surface properties.

In order to polish Nb disk dimensions compatible for 1.3 GHz cavity fabrication, see Fig. 5, the recipe was transferred and optimized on a larger machine MM 9100 with a diameter of lapping/polishing disk of 1000 mm. The polishing recipe had to be adapted to the new polishing parameters accessible with larger machine. As an example, the impossibility to create sufficient pressure on large surface in order to keep reasonable removal rate, imposed to adapt **Step 1** by increasing the diamond grain size from 3 μ m to 6 or 9 μ m. Table 1 summarizes the pressure drop and removal rate change versus the increase of the polished areas.

MOPFDV001

320



Figure 5: Photographs of the mirror polished surfaces properties of which are compatible with SRF requirements.

Table 1: Polished	Area,	Pressure	and	Removal	Rate	Char-
acteristics						

Area, cm ²	Pressure, g/cm ²	Removal rate, μm/min
18	400	3 (dia. of 3 µm)
60	300	1.1 (dia. of 3 µm)
125	200	0.6 (dia. of 3 µm)
855	150	0.25 (dia. of 6 µm)
855	150	0.5 (dia. of 9 µm)

Surface Characterization with Microscope

The surface state was systematically investigated by a laser confocal microscope VK-X 200 Series commercialized by KEYENCE. The polishing run has been periodically interrupted in order to reveal possible embedded particles, especially during **Step 2**, as silica colloidal has tendency to de-pollute the surface versus time. The optical images with laser microscope were used to quantify the level of embedded particles after the first step and were used to define the de-pollution time (at least 2 hours) to recover the SRF properties and reveal all embedded particles, see Fig. 6.



Figure 6: De-pollution efficiency of silica colloidal solution versus time. Red dots correspond to embedded diamonds calculated from an optical image with the laser confocal microscope.

Figure 7 shows the digital interference contrast images after each step of polishing. Surface roughness after **Step 1** was better than for **Step 2**, 70 ± 10 nm and 100 ± 50 nm respectively. The increase of roughness is caused by the associated chemical reaction in the solution, which is promoting the removal of particles during depollution step and also is causing grains appearing. The performance of such processing has been already evaluated under RF and the first results are presented at the 19th SRF conference [6].



Figure 7: Digital interference contrast (DIC) images of Nb surface state after **Step 1** (left) and **Step 2** (right).

SURFACE FORMING

Forming Studies on Samples

A set of surface processed samples, in particular after metallographic, chemical, and mechanical polishing has been used to perform and study the impact of forming on the quality of the surface and how the forming procedure could be improved to maintain surface quality. The R&D studies have been done on a dedicated test-bench, see Fig. 8. The specific curvature of the die gives a possibility to simulate the shape of the cavity iris on the small samples. Cavity half-cells are normally formed by the pressure of 20~30 tons (6.5~10MPa) which corresponds to 0.2~0.3 tons for our samples (15x40x3 mm).

The forming of the samples was performed in conventional and alternative ways. The conventional way is using the standard protocol, where material formed by dies using oil lubricant as interface. In an alternative way, a urethane sheet of $30 \ \mu m$ is used to protect the pre-polished surface.



Figure 8: Photo of the dedicated test-bench used for bending studies.

Roughness and EBSD Characterization

In order to evaluate the impact of the conventional and alternative way of forming, roughness and crystalline damage (face) investigations have been done before and after forming with laser confocal microscope and electronic microscope equipped with the EBSD camera.

To ensure that the microscope measurements have been done on the same locations, the samples, before and after forming, were marked by a dedicated tool. Figure 9 shows the DIC images with measured roughness before and after conventional and alternative forming for different polished surfaces. In the case of the conventional forming, the deformation and damages (scratches) of the grains are caused by the surface friction (contact zones). On the contrary, for the alternative forming technique, the urethane sheet preserves the quality of the polished grains and new grain boundaries are formed, which leads to the increase of roughness. To evaluate the damages of the polished surface, an EBSD analysis was used. Figure 10 shows the signal of secondary diffracted electrons depending on the orientation and damages in the grains. The black color represents the locations with high residual stresses where electrons do not diffract anymore. Hence, the grains which were in the contact zone during conventional forming are completely damaged. However alternative forming shows very limited damages at grain boundaries. Samples polished at KEK didn't show diffraction patterns, confirming that the surface damages introduced by the polishing procedure are already too high and too deep.



Figure 9: The DIC images of the polished surfaces before and after bending.



Figure 10: EBSD maps of the polished surfaces before and after bending.

CONCLUSION

Two steps metallographic polishing recipe compatible with SRF applications has been successfully extended to large sheet dimensions compatible with 1.3 GHz elliptical cavities. At the same time, forming activities performed at KEK showed promising results of alternative forming, as EBSD maps show that the urethane sheet preserves the surface from additional damages. Hence, forming of 1.3 GHz half-cells with the alternative technique could be envisaged. This work will be continued in the framework of KEK-FJPPL program.

ACKNOWLEDGEMENTS

The authors are very grateful and appreciate the support from the members of LAM PLAN team. Especially we

and DOI

ler.

publi

MOPFDV001

20th Int. Conf. on RF Superconductivity ISBN: 978-3-95450-233-2

and DOI publisher. © Content from this work may be used under the terms of the CC BY 4.0 licence (© 2022). Any distribution of this work must maintain attribution to the author(s), title of the work,

would like to thank William Magnin for the excellent assistance in the lapping and polishing tasks. Part of this work was supported by the European Nuclear Science and Application Research-2 (ENSAR-2) under grant agreement N° 654002.

REFERENCES

 [1] C. Antoine, "Materials and surface aspects in the development of SRF niobium cavities", EuCARD Report No. Eu-CARD-BOO-2012-001, 2012. http://cds.cern.ch/record/1472363/files/Eu-CARD-BOO-2012-001.pdf

[2] C. Z. Antoine, "Material and surface aspects in SRF technology", Lectures on SRF 2018, Gif-sur-Yvette, France, 2018.

- [3] C. Z. Antoine and R. Crooks, "Reducing electropolishing time with chemical-mechanical polishing", in *Proc. 14th Int. Conf. RF Superconductivity (SRF'09)*, Berlin, Germany, Sep. 2009, paper TUPP0071, pp. 405-405.
- [4] O. Hryhorenko, C. Z. Antoine, M. Chabot, and D. Longuevergne, "Metallographic polishing pathway to the future of large scale SRF facilities", in *Proc. 19th Int. Conf. RF Superconductivity (SRF'19)*, Dresden, Germany, Jun.-Jul. 2019, pp. 828-832. https://doi.org/10.18429/JACoW-SRF2019-THP002
- [5] https://www.lamplan.com/
- [6] O. Hryhorenko, "Development and optimization of mechanical polishing process for superconducting accelerating cavities", Orsay, France, 2019.