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Niobium micro-mechanical polishing for superconductive radio-frequency applications

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Abstract

For the development of surface finish improvement of Superconducting Radio-Frequency (SRF) niobium components, a qualification study of Micro-Mechanical Polishing (MMP) was carried out. The motivation of mechanical polishing was the possibility to recover RF surfaces by changing their topographic structure, the smoothening of surface defects and the limitation of material removal quantity by further electrical or chemical etching. The machining and polishing technique on niobium are very difficult and challenging due to its high ductility and the use of mechanical polishing with abrasive is usually avoided for SRF application because of the embedded particles risks. Nevertheless, the aim of this study was to qualify a combination of the MMP method with the subsequent chemical treatment that removes surface impurities. Several niobium specimens were MMP polished with two different micro-tools. The surface topography was measured and evaluated by Scanning Electron Microscopy (SEM) to identify the incrusted micro-tools size and the affected layer thickness to be removed. The chemical and electrical polishing was performed. SEM observation and surface roughness measurement were repeated to confirm the contamination removal and surface finish gain when using MMP. The qualification was encouraging for the MMP and electropolishing combination where all inclusions were removed and the uniform mirror-like surface of Ra about 0.02-0.03 µm was obtained. First trials on 1.3 GHz accelerating cavities were realised to evaluate the RF performance of MMP electropolished surface.

Micro-mechanical polishing, Niobium, SRF applications, SEM

1. Introduction

The surface quality of niobium SRF parts is a very important issue that highly contributes to the performance of accelerating components [1-2]. The low core roughness and smoothness of the surface is required because the peaks and other sharp topographic features can cause non-field-emitter quenches which generate higher surface resistance and could be a reason of the drop of the quality factor of cavities (Figure 1c) [1, 3]. The usual surface treatment of niobium SRF parts is the chemical etching by Buffer-Chemical Polishing (BCP) or Electropolishing (EP). However while the BCP or EP are essential to remove the plastically deformed layer from surface issued from the manufacturing process (150 – 200 µm) [2, 4, 5], there is no possibility to significantly improve surface structure or smooth the surface defects. The only technique to recover surface topography is the mechanical polishing with abrasive particles. Several researches have been performed on this topic and the most investigated method is centrifugal barrel polishing (CBP) [6-10]. The limitation of the CBP technique is the non-uniform material removal along the cavity profile, the abrasive pollution and the high treatment time (90-300 hours) [7]. The abrasive contamination on the niobium RF surface can affect the required high thermal conductivity (> 10 W/m·K at 2K) [11] and the residual resistivity ratio (RRR>300). Therefore the abrasive removal is an important issue. Navitski et al. [6] and Palczewski et al. [10] have already investigated a combination of centrifugal barrel polishing with EP or BCP polishing to obtain a chemically clean surface. In case of Palczewski et al. [10], the CBP removed about 130 µm/93 hours and the additional EP of 10 µm were sufficient to regain clean surface structure. Nevertheless the Electron beam welds were not smoothened enough and some voids appeared, that could be caused by the CBP polishing or welding procedures. This is the risk of high mechanical polishing removal that can result in opening of welds porosities and in filling the voids by polishing media that is detrimental to RF performance. Navitski et al. [6] faced the non-uniform material removal along the cavity shape. Three times higher material removal was observed in the equator area compared to iris (100 µm/30 h). The MMP technology basically removes only the surface roughness so the material removal is imitated to 20 – 40 µm (depending on material and initial roughness). That could be also advantageous for surface recovering of cavities with low RF performances and where the required material removal is the lowest possible to avoid mechanical failure or buckling of the structure.

This study is mainly focusing on qualification of the MMP technology with the BCP or EP post-treatment to get mirror-like surface finish and abrasive-free surface for cavities recovering or defects smoothening of other SRF parts.

2. Material and Method

The MMP technology is the combination of mechanical and physical processes. The mechanical process is provided by machine movements that creates the flux. The flux is composed by the micro-tool particles are specifically designed that according to treated material and initial surface roughness of the workpiece. The physical process corresponds to the catalyst that activates the micro-tools action [12].

The time of treatment is depending on the initial surface roughness and it varies between 30-60 hours. The target of MMP technology is mainly to remove the primary and the secondary roughness to get a mirror-like surface finish. That means that material removal is the secondary effect of the process. Two types of micro-tool particles were chosen: Micro-tools_1 and Micro-tools_2 (commercial products); and two time durations were investigated: Long MMP (~ 50 h) and Short MMP (~ 25 h).

The qualification was performed on several niobium specimens. The size and shape variated from simple sheets with dimensions of 12x18x4 mm (Figure 3) to very complex welded parts such High-Order Mode (HOM) coupler (Figure 1a) or Field antenna (Figure 1b). The surface roughness and dimensions were measured before and after MMP treatment by optical machine VEECO WYKO – NT 3300 (ISO 4325) and by contact device Mitituyo on all specimens. The surface pollution was evaluated by SEM on the Zeiss Sigma SEM machine with Field Emission Gun (FEG). The SEM observation was performed after MMP and then after BCP & EP treatment to confirm micro-tools removal.

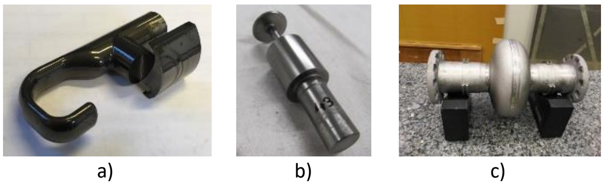
a) b) c)

Figure 1. The niobium specimen SRF parts treated by MMP : a) HOM coupler for Crab RFD cavities; b) Field antenna and c) 1.3 GHz niobium accelerating cavity .

After the qualification tests of MMP, first trials on the accelerating 1.3 GHz cavities were realised. For the set-up design and the cavity orientation adjustment, two copper 1.3 GHz cavities (spun and welded) were polished because of similar material properties and lower price than niobium cavities. According to these results, the niobium 1.3 GHz mono-cell (Figure 1c) was treated in order to obtain the best state of surface and for the further RF testing.

3. Results

3.1. Qualification of surface topography

The mean arithmetic surface roughness (Ra) and the average maximum height of the profile (Rz) were measured by the contact device Mitituyo. The surface parameters were evaluated before and after MMP treatment in order to identify the potential of mechanical polishing itself and afterwards, the surface topography was re-measured after BCP and EP. Figure 2 represents the surface parameters results for all treatment steps. Each column is an average of the all testing samples roughness (5 measurements/sample/treatment). The Micro-tools_1 showed higher performance achieving Ra in tens of nanometres 0.013 ± 0.002 µm. Moreover the same order of magnitude was kept after EP with Ra 0.053 ± 0.012 µm. The BCP treatment deteriorated slightly the surface roughness to a Ra 0.203 ± 0.013 µm (Micro-tools_1) and 0.514 ± 0.042 µm (Micro-tools_2). However for both micro-tools, the surface roughness difference between Long and Short MMP was almost eliminated by the BCP. So the Short MMP with BCP resulted in the same Ra as the Long MMP with BCP in a half of time. Concerning the Rz, from initial average Rz about 4.30 µm the Micro-tools_1 Long MMP reached about 0.11 µm and after BCP 1.29 µm and EP 0.38 µm. So the elimination of the primary roughness by MMP is well demonstrated (Rz < 1.0 µm). Figure 3 shows the shiny and glossy effect of the BCP and EP on MMP treated samples.

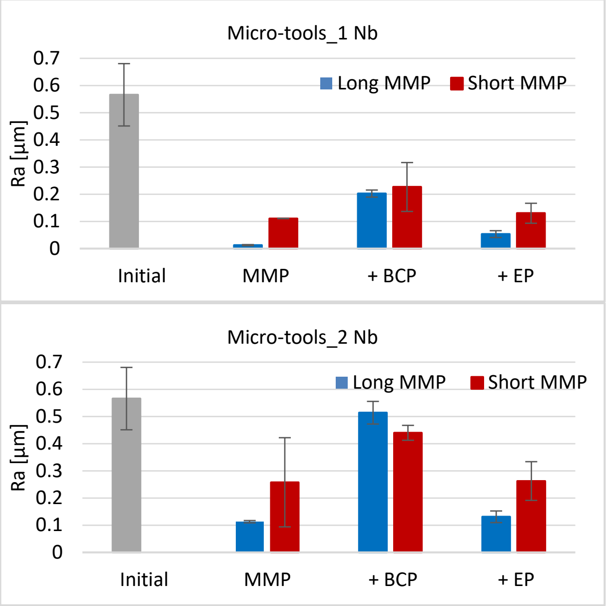

Figure 2. The surface roughness Ra for each polishing treatment.

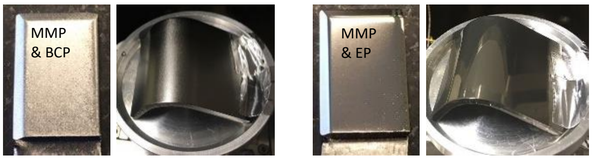

Figure 3. The comparison of the surface aspect of the niobium samples after MMP and BCP & EP.

3.2. Micro-tools pollution removal

The SEM analyses were conducted with FEG. Samples were mounted on sample holder and introduced into the vacuum chamber of the SEM. The chamber was then flushed with nitrogen and evacuated until a vacuum of at least $2\cdot10^{-5}$ mbar was reached. The BSE contrast was used to detect foreign material and the SE contrast was applied to determine the form of foreign material and their possible incrustation into the Nb surface.

3.2.1. Micro-tools_1 treatment

After the Long MMP treatment, the polished samples revealed the incrusted micro-tools all over the surface with the size below 10 µm (Figure 4). On the micrometre-scale, some other particles were found as well and their nature was identified by Energy Dispersive X-ray (EDX). After the Short MMP, the inclusion density was lower nearly by 70 %.

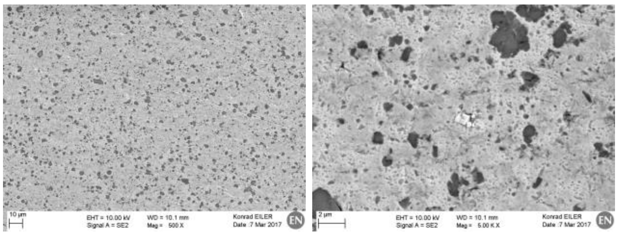

Figure 4. The SE overview of surface state after long treatment type Micro-tools_1 on Nb sample.

According to maximum size of particles about 10 µm, the BCP and EP material removal was defined to 20 µm depth to ensure removal of all affected layer. Figure 5 shows the surface after MMP & EP. The surfaces were free of inclusions and no other contaminations or pollutions were observed.

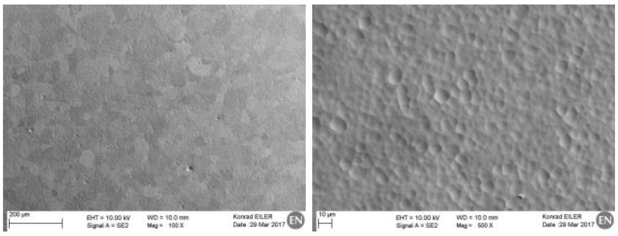

Figure 5. The SE overview of free-inclusions surface state after MMP & EP on the same Nb sample as in Figure 4.

After MMP & BCP, the surfaces exhibited the typical post-BCP structure with revealed grain boundaries due to etching. Moreover, some residual fine Micro-tools_1 of 1 µm size were observed (Figure 6). In order to get rid of them several additional BCP etching and High-pressure water rinsing were done but only the subsequent EP removed all the residual particles remained still on the surface. Therefore, the EP treatment can be considered as more convenient method for the MMP polished parts.

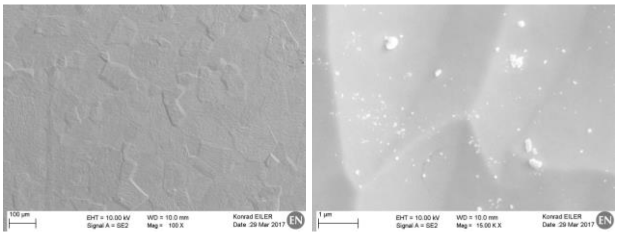

Figure 6. The detailed SE images of surface state after MMP & BCP for small < 1 µm Micro-tools_1 particle dispersed on the surface.

3.2.2. Micro-tools_2 treatment

The same surface pollution was observed with the dark micro-tools_2 incrusted into surface (size < 5 µm) and other contaminations (Figure 7). The BCP and EP material removal was defined to 20 µm depth as well because of some other metal particles incrusted on the surface with size of 10 µm.

After EP and BCP, the MMP surfaces were free of inclusions (Figure 8).

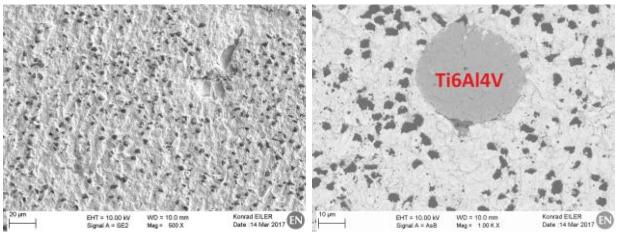

Figure 7. The surface is mostly covered with Micro-tools_2 inclusions around 5 µm in size and some various contaminations such as Fe, CuSn, Si and Ti.

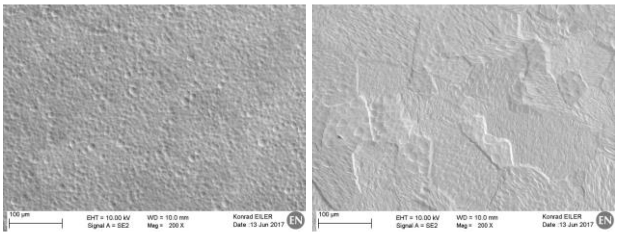

Figure 8. The SE overview of free-inclusions surface state after MMP & EP (left) and MMP & BCP (right).

3.2.3. First trials on the 1.3 GHz cavities

As the MMP technology showed a good potential in term of the surface finish and it was confirmed that the EP is able to completely remove all undesirable micro-tools particles incrusted in the surface, first trials on the 1.3 GHz cavities were realised.

Two seamless copper cavities were used for the test of MMP set-up designs: horizontal and vertical. The special clamping system and additional support were fabricated to hold the cavity in the good position and to enable the inner flux passing. The main objective of this polishing was to remove the maximum of material. The MMP took 60 hours. The part was rotated after 30 hours to get suitable polishing uniformity. The interior surface roughness was measured by optical machine VEEKO and the thickness profile evaluation was also realized (Figure 9). During the polishing of the first cavity in horizontal position, the most significant material removal was in the equator area (100 µm). Moreover, the weight of the abrasive flux in the equator area and with the high movement of all the assembly, the fatigue crack on iris appeared. It could be also caused by the initial very small thickness of iris area (1.5 – 2 mm). Anyway, an additional support of the equator was used for the vertical treatment to avoid this crack of fatigue. Concerning the results of surface roughness, the mirror-like topography was obtained by the vertical position (Figure 10). The Ra on iris area was about 0.088 µm and on the equator about 0.027 µm. The horizontal position reached also low surface roughness: 0.119 µm on iris and 0.585 µm on equator. The material removal in horizontal position was more uniform all along the cavity profile compared to vertical position where the thickness decreasing on cut-off tubes was nearly negligible.

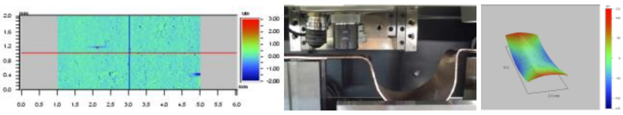

Figure 9. The optical measurements of the surface topography at the iris and cut-off areas.

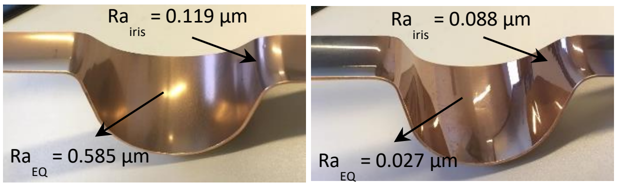

Figure 10. Comparison of state of surface after horizontal MMP (left) and vertical MMP (right) positions.

After comparing the beneficial and limitation aspects of both positions, the vertical one was chosen for further investigation mainly because of the high surface finishing and the better stability of the cavity fixation during MMP process.

4. Discussion

The open question of the MMP potential for improving the manufacturing of RF parts is the radio-frequency performance. Therefore the next niobium 1.3 GHz cavity (spun and welded) was chosen for the MMP vertical treatment. The cavity has been already tested by RF so there is a possibility to directly compare the influence of MMP processing. The MMP time and the Micro-tools_1 composition was adapted to get the best surface quality. The contamination removal will be provided by EP to keep the mirror-like surface and to obtain free-inclusion surface. The cavity MMP polishing was already realised and the next steps is the EP followed by the RF test. Figure 11 shows the surface aspect before and after MMP of the RF cavity surface.

Figure 11. The niobium 1.3 GHz cavity treated by MMP technology.

5. Conclusion

The mechanical polishing performed by MMP technology, provides an efficient way to homogeneously polish parts of complex geometry by immersing them into a suspension charged with abrasives. Like most mechanical polishing procedures, it leaves incrusted particles on the polished surfaces. Both MMP Micro-tools left abrasive inclusions on all analysed surfaces, independent of the duration of the treatment. The treatment duration has only affected surface roughness.

Concerning the surface topography reached by MMP, the Micro-tools_1 treatment achieved shiny surface with the Ra in level of nanometres (0.018 ± 0.007 µm). The MMP & EP is able to keep the glossy surface finish and reach the inclusions free surface with Ra < 0.05 µm. The MMP & BCP removes completely the Micro-tools_2 particles, but it leaves some fine residual Micro-tools_1 on the surface (not incrusted). For the cavity and thin wall structure parts, some possible geometrical deformations can be observed during the MMP due to stress relaxation, so a re-tuning would be needed.

To conclude the MMP technology showed a high potential for the mechanical polishing on niobium parts mainly in term of the mirror-like RF surface quality. Next investigations should evaluate the RF performance of the MMP & EP combination.

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