SURFACE ANALYSES AND OPTIMIZATION OF CENTRIFUGAL BARREL POLISHING OF Nb CAVITIES

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Abstract

Detailed microscopy investigations of the niobium surface quality after centrifugal barrel polishing (CBP) have been performed applying metallographic techniques. The results imply the need for further optimisation of the polishing procedure, mainly to reduce the thickness of the layer that is damaged at the surface as well as pollution by the polishing media. The most realistic application of CBP is a combination using CBP initially to remove surface defects followed by chemical polishing to obtain a chemically clean niobium surface.

INTRODUCTION

Centrifugal barrel polishing (CBP) is an acid-free surface-polishing technique based on abrasive media. It considerably reduces the usage of chemicals in the preparation of Nb cavities, typically requiring only a final light electropolishing (EP) or buffered chemical polishing (BCP) step and achieving considerably smaller roughness than in chemical treatments alone [1, 2]. CBP addresses in particular the removal of pits, welding spatters, deep scratches, and inclusions of foreign material that occasionally occur in the production process and often remain unaffected by the EP/BCP treatment. A mirror-smooth surface without chemical contamination is also an important enabling step for thin-film technologies of SRF cavities.

A combination of CBP and EP/BCP was already applied and tested on single- and nine-cell Nb cavities with resulting quality factor Q_0 of cavities of more than 10^{10} and accelerating gradients E_{acc} of 25 to 40 MV/m [3, 4]. Accordingly, similar polishing is also considered for the accelerating structures of the future International Linear Collider (ILC) [5] as an alternative or partial replacement of the established EP procedure.

Dedicated studies of the CBP process using a "coupon" cavity and applying microscopy and metallographic techniques have been performed and reported here.

EXPERIMENTAL DETAILS

CBP Machine

The CBP machine (Fig. 1) has been purchased by the University of Hamburg and is used in the ILC-HiGrade Lab at DESY within a common R&D program [6]. The machine is custom built for this purpose by Mass Finishing Inc. and can be used to polish up to two single- or nine-cell 1.3 GHz SRF cavities at once. During the polishing process the cavity, approximately 50% filled with a polishing media, rotates around the central shaft of the machine at up to 110 rpm, while at the same time counter-rotating around its own axis with the same speed. The rotation speed of the main shaft determines the centrifugal force acting between the polishing media and the cavity walls, while the counter-rotation of the cavity lets the polishing media move across the surface and polish it.

Different polishing media and variation of the rotation speed is used to achieve an optimum polishing result.

Figure 1: CBP machine in the ILC-HiGrade Lab.

Coupon Cavity

Since quality of the inner surface plays a key role for the RF performance of the cavities, it is necessary to characterise the surface after the applied polishing procedure. The inner surface is, however, hidden in the cavity and difficult to access by conventional microscopy techniques. To overcome this, a special "coupon" cavity has been fabricated in cooperation with our colleagues from KEK, Japan. This is a niobium single-cell cavity with six openings into which removable samples (coupons) can be placed at the most interesting regions of the cavity (Fig. 2). The usage of the coupons allows polishing characterisation and optimisation by direct measurements of the surface roughness, removal rate, and removal profile as well as further detailing the amount of contamination left behind after the polishing process. This allows detailed surface studies after a series of CBP tests

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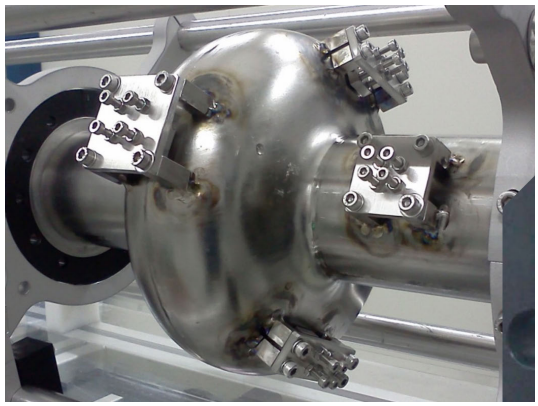

Figure 2: Coupon cavity used for the characterization of the CBP polishing. The protruding structures indicate the position of the removable samples.

as well as after follow-up chemical treatments to determine the best combination.

Sample Preparation

Polycrystalline niobium discs (diameter 8.6 mm, thickness 3 mm, and RRR of 300) with a central thread on the back side and a small off-axis pit (diameter 1 mm) on the front side (Fig. 3) have been used as coupon samples. The central thread allows fixation on a suitable holder during CBP in the cavity and is compatible with sample holders appropriate for the different measurement techniques discussed below. The pit on the front side permits the determination of the removal rate and enables a sample repositioning with an accuracy of ~500 µm in different measurement techniques.

The CBP was performed in 4 steps with different mixtures of polishing media as derived from best FNAL, JLAB, and previous DESY experience [1, 2, 4]:

Step 1: Ceramic angle-cut triangles (KM, 9 x 9 mm^2), surfactant (TS compound), and de-ionized (DI) water;

Step 2: Plastic cones (RG-22, 12.5 mm), TS compound, and DI water;

Step 3: 600 mesh Al_2O_3 cubic hardwood blocks (5 mm), and DI water;

Step 4: Colloidal SiO_2 (40 nm) and cubic hardwood blocks (5 mm).

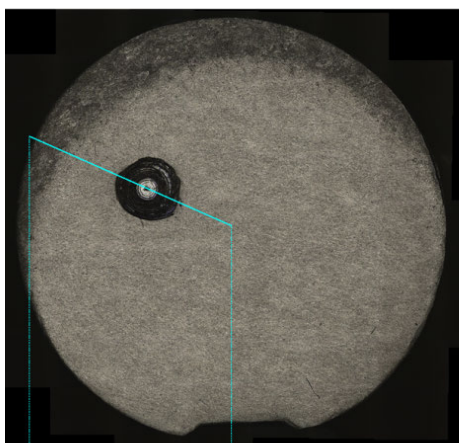

Figure 3: Front-side view of the coupon-sample (diameter 8.6 mm) with an off-axis pit for the determination of the removal rate.

Table 1: CBP removal rate and processing time at different polishing steps

	Removal at the tube, µm/h	**Removal at the equator, µm/h**	**Processing time, h**
Step 1	3	9	8
Step 2	1	2.5	15
Step 3	0.1	0.7	30
Step 4	<0.1	<0.1	40

After the CBP, the coupon cavity is rinsed by DI water and cleaned in an ultrasonic bath first with a cleaning agent (TICKOPUR R-33) and then in pure DI water.

Measurement Techniques

The surface quality of the samples after the CBP was thoroughly investigated with several techniques. At first, scanning electron microscope (SEM, Philips PSEM 500) with energy dispersive x-ray analysis (EDX, Bruker AXS, XFlash Detector 5010) and confocal 3D laser scanning microscope (Keyence VK-X100K) were systematically applied to control the successive removal of surface defects and to reveal possible residues of the CBP process. Based on the profilometry results, the removal rate, polishing quality, and surface roughness were determined.

RESULTS AND DISCUSSION

Surface Quality

In Fig. 4 the evolution of the niobium surface after sequential CBP steps is compared. The final surface of CBP-polished niobium is rather smooth on average but contains many inclusions and still deep grooves with sharp edges around them due to the mechanical polishing process.

Typical SEM images of the final CBP surface (Fig. 4, right column) show many insulating inclusions (up to ~10^6 cm^{-2}) with a size of up to 40 µm and sharp rims. EDX investigations of the surface after the CBP reveal inclusions containing aluminum (Fig. 5). Therefore, these defects are most likely Al_2O_3 particles that are used during the steps one to three of the CBP process.

The amount of inclusion is highest in the area of the end-tubes of the cavity, where the polishing speed is also lowest (see Table 1). Although the Al_2O_3 material most probably originates from step 3 of the polishing, the reason for the embedding should be in too rough surface after two initial steps. Comparing the surface roughness it can be seen that the surface of the equator area is smoother than in the other two areas containing a lot of deep grooves. This is clearly visible starting already from

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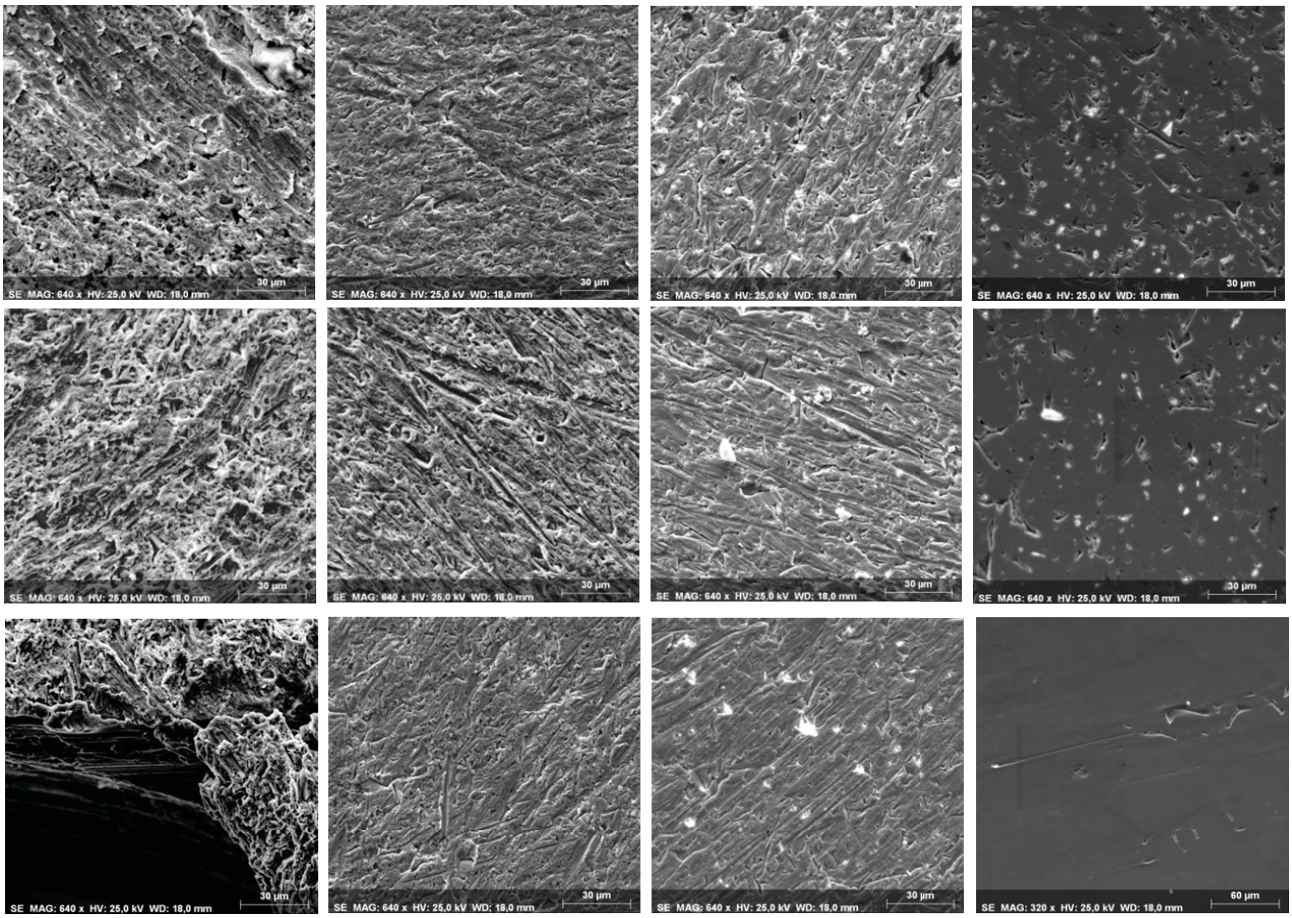

Figure 4: SEM images of the surface of niobium coupons after step 1 to 4 (from left to right column correspondingly) polished in the location of the end tube (upper), tapered cell area (middle), and equator area (lower row).

step 2.

The amount of material polished away at step 3 is definitely not enough to remove the grooves. Polishing media from step 3 might fill also the grooves and stay there, appearing at the final step. Low-pressure rinsing with DI water is definitely not enough to remove the embedded particles.

The lamination effect may also play an important role. Since the surface after the first two steps is very rough and sponge-like, some polishing media can penetrate into the softened surface and get encapsulated by the lamination of the upper surface layer under the pressure

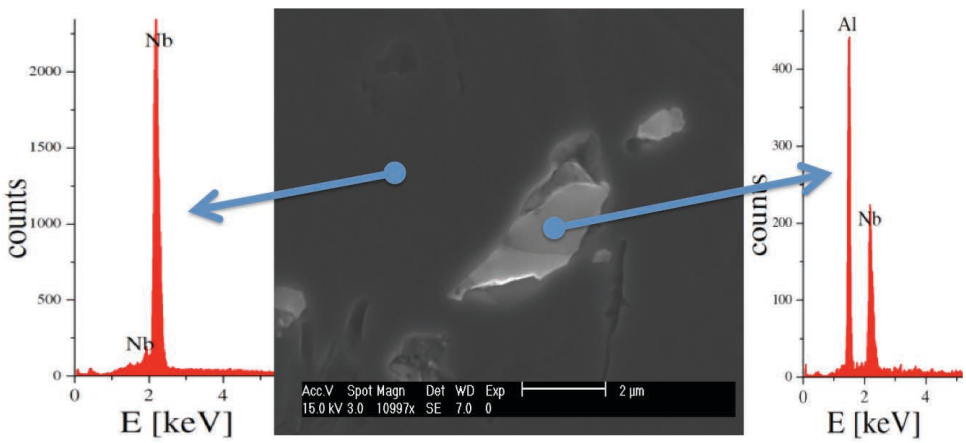

Figure 5: Al_2O_3 inclusions found embedded in the Nb surface as identified by SEM/EDX analyses on the CBP sample from the tube area.

of the polishing media sliding across the surface. During the last fine polishing steps the embedded media gets released.

The polishing media, released at the final polishing step, is mostly rougher than the colloidal silica and causes scratches on the surface. Removing the polishing media during step 4 and replacing it significantly reduces the scratching of the surface, as confirmed by the SEM investigations (Fig. 6). However, this additional procedure increases, the number of polishing steps and the polishing time.

Current results indicate a necessity of removing the embedded particles/scratches by additional chemical polishing to reach a chemically clean and scratch-free niobium surface. These findings confirm the results of cold RF tests of the cavities, which were successful only after some tens of µm of additional EP polishing [1].

Strong shearing and lamination of the upper surface layer, especially at the first polishing step, have been observed on the coupons (Fig. 7). The resulting damaged layer is not removed by the final CBP polishing; it is simply smoothed and must be removed by additional chemical polishing.

In order to determine the thickness of the surface-damaged layer, investigations of the coupons with a

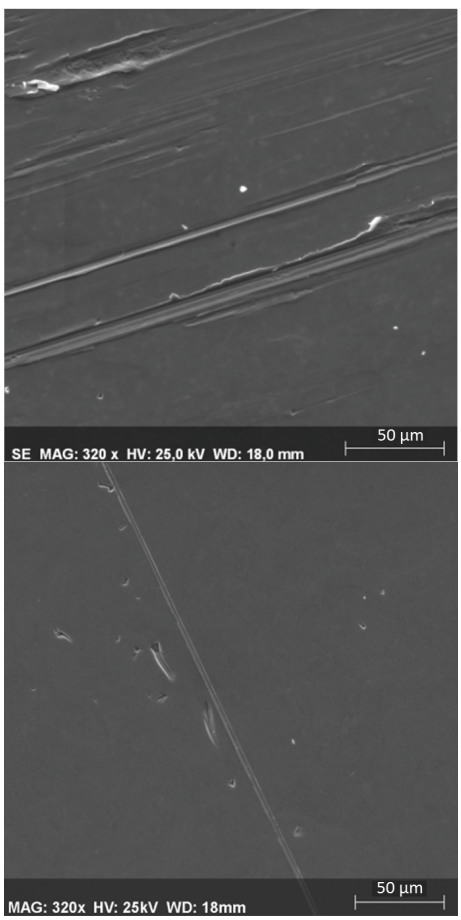

Figure 6: SEM images of the equator surface before (top) and after (bottom) renewal of the polishing media at the final polishing step.

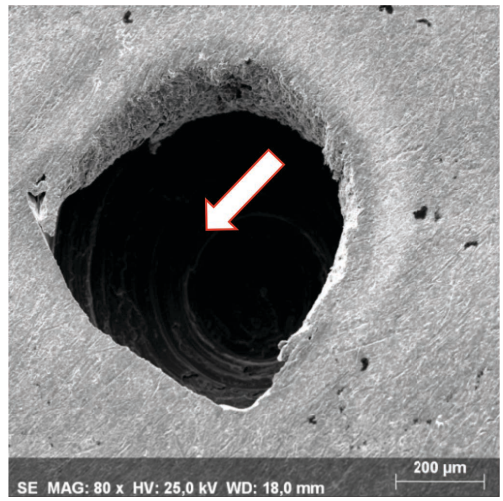

Figure 7: SEM image of the coupon surface around the off-axis hole indicating shearing and lamination of the upper surface layer.

metallographic technique have been performed. The coupon samples were cut and the cross-sections mechanically and chemically polished to reveal the grain structure of the material. Laser scanning microscopy of the cross-sections reveals the presence of small irregular grains within the surface layer of up to 40 µm depth as shown in Fig. 8. A final BCP or EP polishing step should be applied to remove this surface-damaged layer.

An example of successful removal of the surface inclusions and scratched upper surface layer by an additional 20 µm BCP polishing is confirmed by SEM and laser scanning microscopy as presented in Fig. 9. The inclusions have been removed completely, revealing the grain structure of niobium. The roughness of the surface after the BCP increased, however, by a factor 4 [7].

Investigations of a coupon sample after pure CBP and

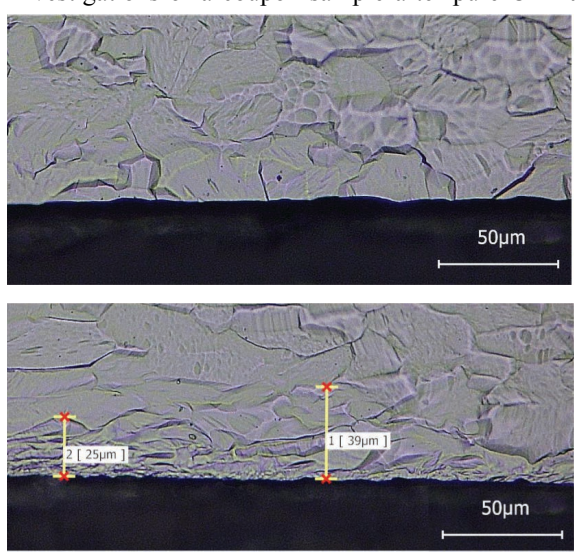

Figure 8: Laser scanning microscopy images of the cross-sections of the initial coupon-material (top) and after four-step CBP polishing (bottom) revealing a surface damaged layer (bottom side of the cross-sections).

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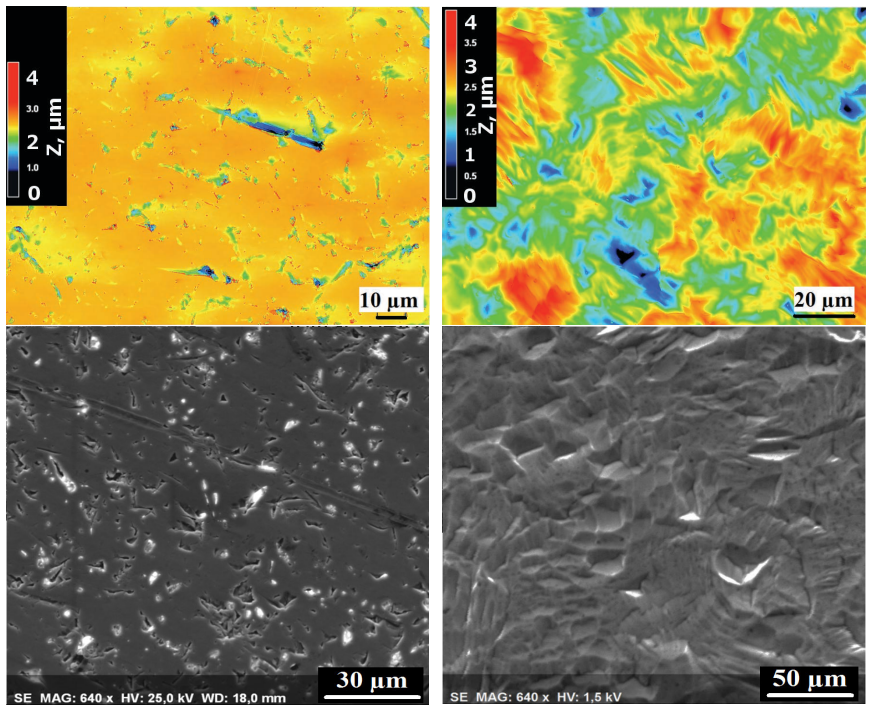

Figure 9: Laser scanning (top) and SEM (bottom) images of the CBP sample before (left) and after (right) 20 µm additional BCP polishing.

after the additional 20 µm BCP polishing using field-emission scanning microscopy confirmed a significant shift of the activation level of field emission from 60 MV/m to much higher field values of more than 175 MV/m [7].

CONCLUSIONS

Microscopic investigations of the CBP polishing procedure indicate the necessity of further optimisation of the polishing to reduce the surface-damage layer, amount of contamination by the polishing media and consequently the amount of required chemical post-treatment. An improvement and control of the homogeneity of the polishing and removal rate across the whole cavity surface has also to be considered. Since the removal rate at the iris region is a factor three smaller than at the equator, the polishing time should be determined by the slowest polishing rate at the iris. To remove just the surface layer of 100 µm, which is usually done in the established polishing procedure, requires a polishing time of more than 30 hours. This would lead, however, to the removal of nearly 300 µm at the equator.

The thickness of the damaged layer might be reduced by e.g. lower centrifugal force (lower rotation speed of the machine). However, this would decrease the polishing speed.

Since chemistry-free polishing seems not to be feasible at the moment, the most realistic application of CBP is a combination of step 1 and/or 2 of the CBP for removal of defects and BCP or EP polishing to obtain a chemically clean niobium surface.

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