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R&D ACTIVITIES ON CENTRIFUGAL BARREL POLISHING OF 1.3 GHZ NIOBIUM CAVITIES AT DESY/UNIVERSITY OF HAMBURG*

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Abstract

In this paper the status of research activities at ILC-HiGrade Lab (DESY/University of Hamburg) on Centrifugal Barrel Polishing (CBP) of 1.3 GHz Niobium Cavities is presented. We focus on CBP based on the polishing recipe reported by Fermi National Laboratory and Jefferson Lab [1]. The aim is to gain a better understanding of the limitations of this technique, detailed characterization of the treated surface after each polishing step using a "coupon" single cell cavity. Plastic deformations upon initial CBP steps, embedded polishing media and residual damage upon final polishing were investigated at different areas of the cavity.

INTRODUCTION

Current serial production technology of superconducting radio frequency (SRF) Nb cavities represents a complicated process which includes a number of technological steps starting from purification of raw niobium and production of plane sheets up to mechanical fabrication of cavities, chemical and thermal processing and finally testing.

Forming steps can cause mechanical damage of approximately 120 µm into the interior surface of the cavities. Electron-beam welding of the cavities can produce weld beads on the interior surface of the cavity [2]. Electropolishing (EP) is presently the preferred route for preparing the final cavity interior surface.

Despite rigorous control of technological steps according to the respective technical specification, some cavities quench at low gradients, particularly due to defects in the equator areas of the cells. Some of the defects, such as pits, welding sputters and inclusions of foreign material are difficult to remove by chemical polishing.

Centrifugal barrel polishing (CBP), a mechanical processing of the interior of the cavities has a high potential to revive such cavities. It can be used instead of "bulk" EP in a standard mass production to remove the damaged surface layer as it is not sensitive to material inclusions. At the same time, it can effectively remove protrusions and reduce the contour of the weld bead. A smooth surface provided by CBP is a good starting condition for the following EP. A moderate "final" EP treatment (10-40 µm) might be enough to reach high surface quality and, therefore, better cavity performance. In addition the usage of hazardous reagents would be significantly reduced. However, the processing recipe of CBP and the optimal amount of "final" EP are to be estimated. Here we continue this study by further exploring CBP process, in particular, the surface roughness and plastically deformed layer at the coupon surface using metallography technique. The attempts to reduce the damaged layer are also considered.

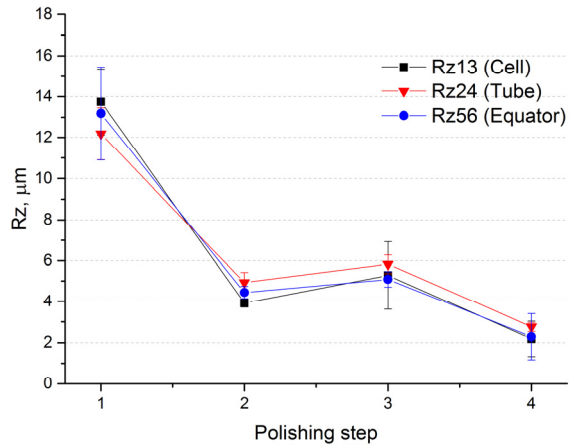

Figure 1: Roughness R_z (peak-to-valley height, µm) of Tube (red triangles), Cell (black squares), and Equator (blue circles) area coupons measured after 1-4 steps of CBP.

PREVIOUS WORK

We have investigated JLab/FermiLab four-step CBP recipe [1] in detail. A mirror-smooth surface has been achieved on single- and 9-cell cavities by applying CBP in a four-step process (here steps are numbered as CBP#1, CBP#2 and so on), firstly with ceramic triangles (Duramedia ACT), then plastic cones (VF-RG 22), alumina mesh (#600) and finally colloidal silica (40 nm) as abrasives. Earlier we reported some of the results at [3]. In particular, the surface of the coupons installed at irises, cells and equators after each polishing step was investigated with microscopic (SEM) and spectroscopic (EDX) techniques. Abrasion rate (the rate at which material is removed) and pollution with polishing media were analysed. The origin of large deep scratches left after the final polishing step was identified, and partially eliminated (by multiple changing of the polishing solution). Cross sectioning of some coupons after the final treatment by metallographic method revealed the presence of a thick (up to ~40 µm) damaged layer on the surface.

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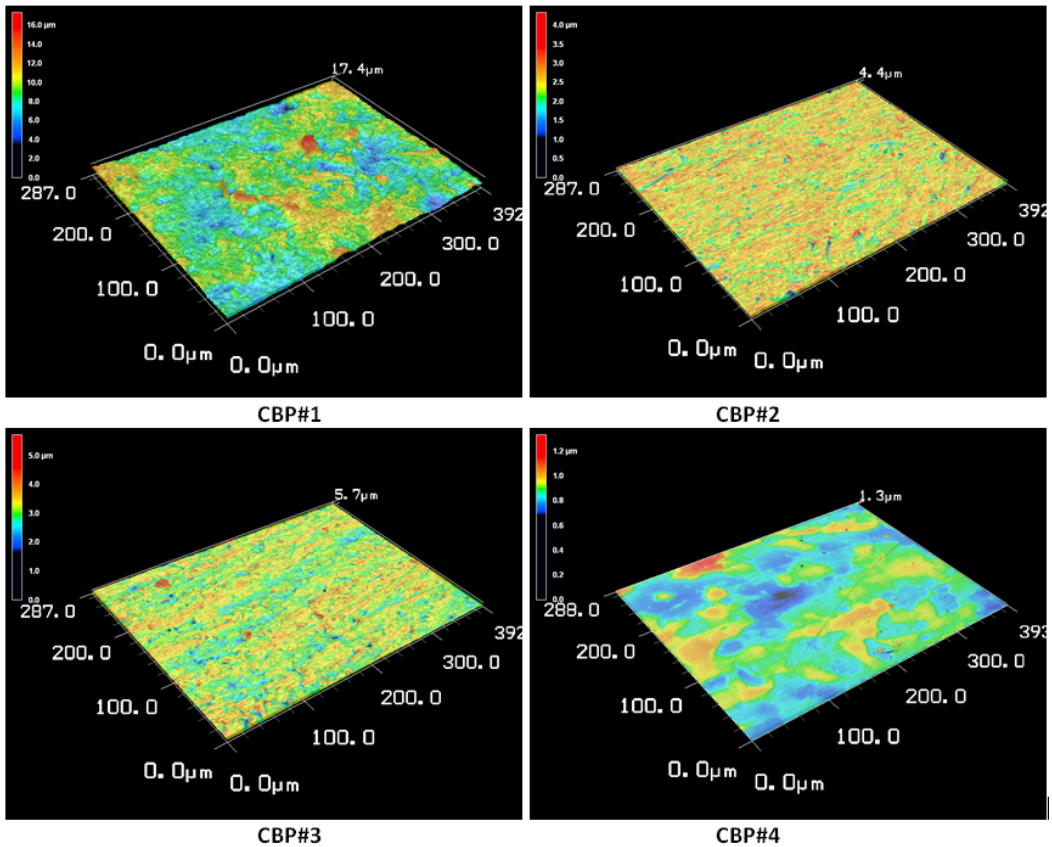

Figure 2: The surface topography (3D view) of the Equator coupon after each step of CBP treatment (3D laser scanning microscope, Keyence VK-X100). Note different scale for vertical (colour) axis. A mirror smooth surface is shown in differential interference contrast (DIC) mode.

CURRENT RESULTS

Surface topography of Nb coupons after each CBP step was studied with 3D laser scanning microscope (Keyence VK-X100). Roughness (a peak-to-valley distance) of the coupons after various steps in three regions of the cavity is shown in Fig. 1. The roughness upon polishing is supposed to decrease gradually from one CBP step to the next. However, after CBP#3, both the average peak and valley heights were found to increase as compared to the preceding step. In Fig. 2 the surface topography evolution upon 4-step CBP treatment of one of the equator coupons is presented. It is seen that the surface gets gradually smoother with each step (except CBP#3 where separate protrusions are clearly visible;see the scale bar for height) until the crystal grains get pronounced and a mirror surface is achieved upon CBP#4.

Metallography

Since many important macroscopic properties of metallic materials are highly sensitive to the microstructure, the damaged layer at the coupon surface after various CBP steps was systematically explored using a metallography technique. Metallographic preparation of the sample surface includes various methods of grinding, polishing, and etching thus allowing the observation of the internal structure of metals and alloys via optical microscopy. Optical images of the coupon cross sections after CBP#1 for the different cavity regions are shown in Fig. 3. A cross section of the unpolished coupon is shown for comparison. We found that the depth of significant (plastic) deformation after the first CBP step varied from 15 μm (for the end-tube region of the cavity) to 30 μm (equator area), while local plastic deformations reach depths up to 25 μm and 70 μm, respectively. After CBP#2 no deformed grain boundaries were observed, since at the determined material removal rate of 1 μm/h (tube area) and 2.5 μm/h equator area) the damaged layer produced during CBP#1 was removed. In addition, a separate study showed that CBP#2 on its own does not produce any plastic deformation that could be determined by the metallography techniques employed.

Additional CBP runs were performed with a lower rotation speed (70 rpm instead of 100 rpm) of the main shaft than used in the FNAL/JLAB recipe with the aim of reducing the depth of surface damage. The abrasion rate and the depth of significant deformation were estimated. It was found that the abrasion rate at CBP#1 decreased by a factor of three while the depth of significant defor

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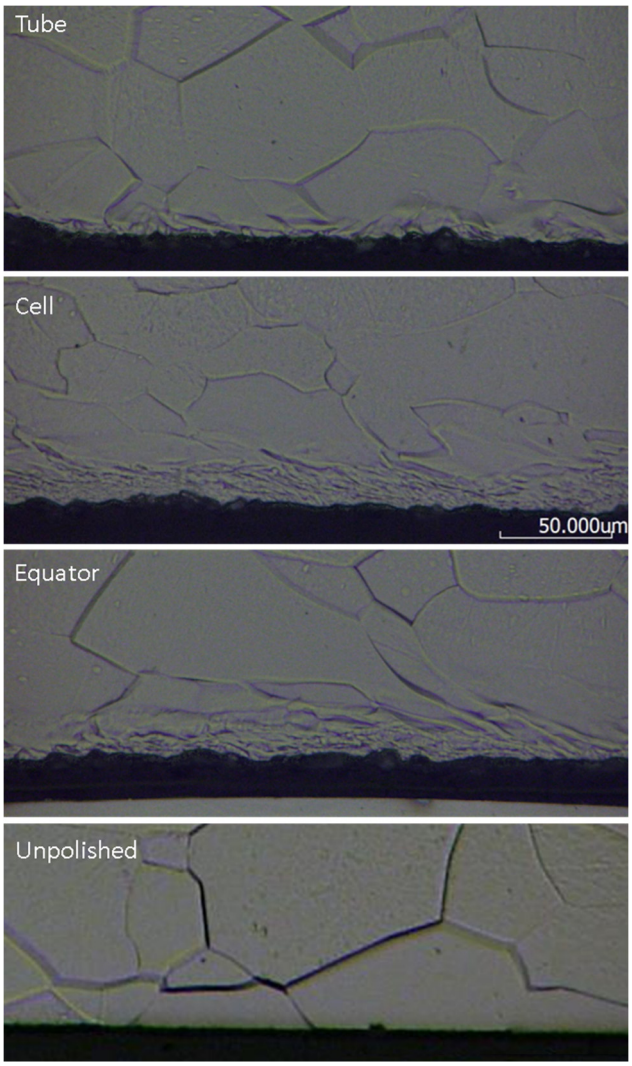

Figure 3: The internal structure of the coupon cross-section plane near CBP (CBP#1, 100 rpm) treated surface of the Tube, Cell and Equator coupons (3D laser microscopy). A cross section of the unpolished coupon is shown for comparison.

mation in most crucial areas of the cavity decreased by up to 50 % (see Fig. 4).

Cooling Experiments

The formation of niobium hydrides responsible for the Q-disease phenomena observed in performance of Nb cavities was explored using the CBP-treated Nb coupons. It is possible to study the hydrides due to the surface-relief features which are caused by an irreversible plastic deformation during the formation of the niobium-hydride crystals at low temperature. A Nb coupon after the full CBP procedure (4 steps, mirror-smooth surface) was cooled down in vacuum to 10 K by using COOLPOWER™12/45 cold head with COOLPAK ™4000 compressor unit manufactured by Leybold Vakuum GmbH. After the cooling system was switched off, it was left to warm up naturally. The temperature log during cooldown is shown in Fig. 5.

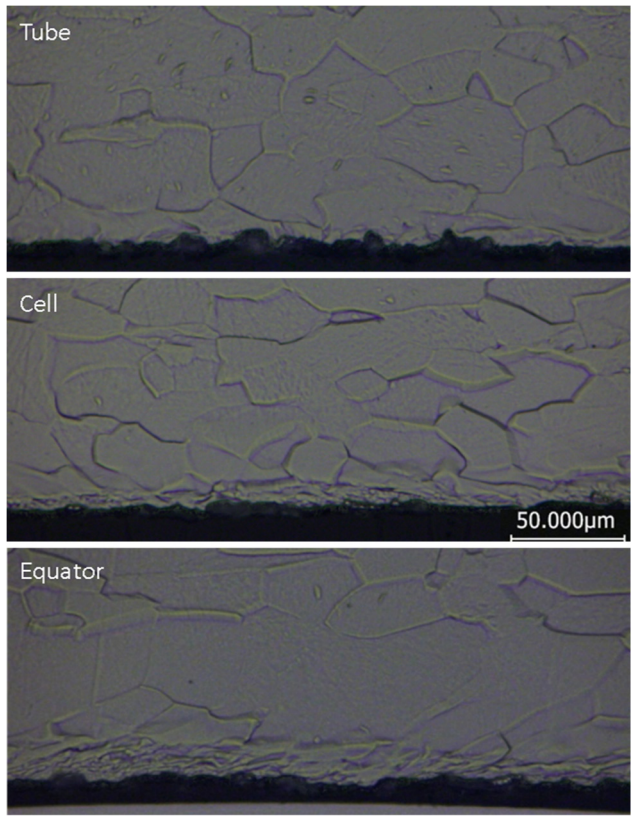

Figure 4: The internal structure of the coupon cross-section plane near CBP (CBP#1, 70 rpm) treated surface of the Tube, Cell and Equator coupons (3D laser microscopy).

Before and after the cooling, the coupon was inspected by a 3D laser-scanning microscope in a differential interference contrast (DIC) mode. The DIC images of the surface are shown in Fig. 6. After the cooling, areas of irregular deformation were found at the surface. These areas could be identified as "footprints" left after niobium hydride formation.

The topographic features of the "footprints" reach 250 nm in height and 20–40 µm in-plane. A scratch defect of the niobium surface can be seen in both images. The location of the footprints along the scratch is in good agreement

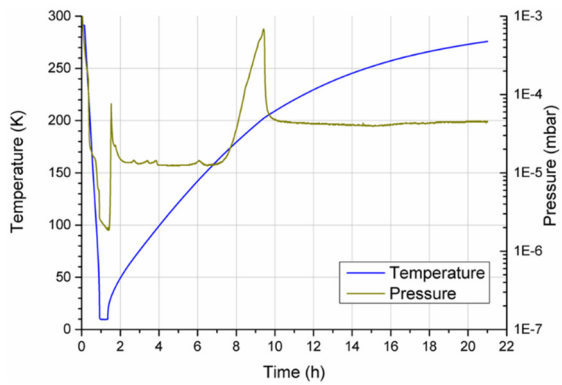

Figure 5: Temperature (blue line) and pressure (green line) during the cooling experiment.

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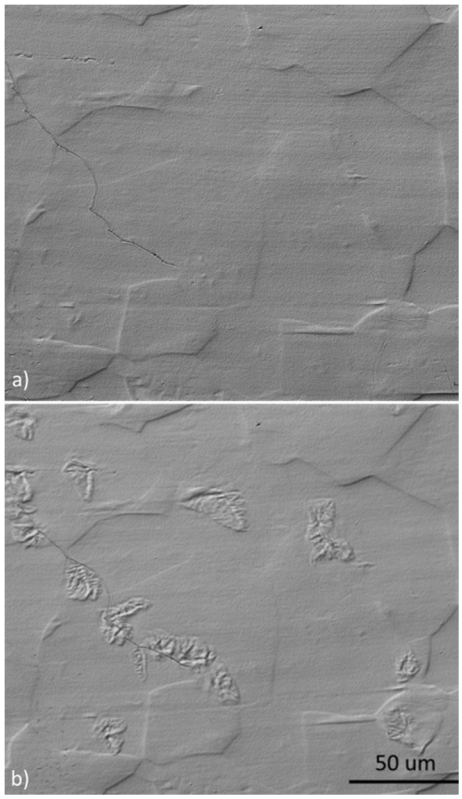

Figure 6: Laser images of the same region of the coupon surface before (a) and after (b) the cooling.

with theory as the hydrides are expected to precipitate preferentially near the surface defects [4, 5].

The mechanical properties of the Nb surface after the full cycle of CBP have been studied. Indentation microhardness measurements are a well-known and reliable test method, frequently applied to bulk materials and thin films to gain a better understanding of the connection between the mechanical properties, the microstructure and the chemical composition. While the metallographic method is sensitive to mostly large plastic deformations, the microhardness technique is also very sensitive to microscale defects invisible via metallography. Moreover, due to the high surface roughness obtained after the first CBP steps, performing hardness measurements at small loads is not feasible owing to the small dimensions of the indentations and the low optical reflectivity of the sample surface.

Microhardness was measured by the Vickers method at various loads (5-2000 gf). It allows calculation of the hardness for different depth of indentation. Our results (see Fig. 7) showed an increase in Vickers hardness (HV) from 60 for the reference sample to 130 and 110 for the surface of the Tube- and Equator-area coupons, respectively, after the full cycle of CBP. The increase in hardness indicates that a layer of plastic deformation and dislocation is still present in the cavity surface even if it looks mirror-smooth to the naked eye. Since the surface of the Tube-area coupon contains a much higher quantity of abrasive particles embedded into Nb and therefore more dislocations, the microhardness value in these areas is higher. One should note that the metallographic cross-section of the coupons treated by CBP#1 demonstrated the maximal amount of visible plastic deformations in the Equator area, while the current investigation of the coupons after the four CBP steps shows the maximal mechanical damage in the Tube area, i.e. the opposite result. This means that during CBP#1, the crucial parameter determining the surface quality is the abrasion rate, while for the last two CBP steps the density and shape of the abrasive particles are the major factors.

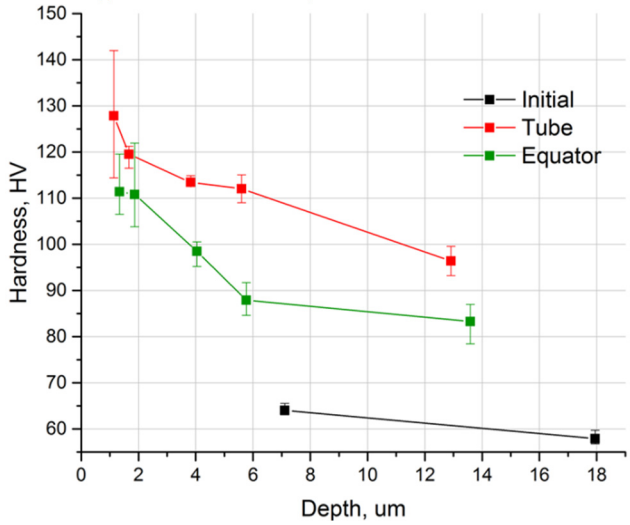

Figure 7: Microhardness values vs. indentation depth of the coupons (Tube, Cell, Equator regions) after four CBP steps.

Electropolishing

Electropolishing is required for the post treatment of the coupons after CBP. The EP set up was commissioned and tested on coupons. The first experiments on fine-grain Nb samples decreased the root-mean-square roughness from 2 μm to 0.5 μm, while the height of peaks and depth of valleys decreased from 30 μm to 3 μm and from 24 μm to 11 μm, respectively. These measurements were made over an area of 710×530 μm^2. The same roughness parameters within a single grain are 4.9 μm, 17 μm and 20 nm over an area of 10x10 μm^2. The individual grains constituting the crystal structure of the material became clearly pronounced after this treatment. The average height of separate grains measured at grain boundaries was 215 nm. Several EP treatments of single-crystal cut-outs from Nb ingots melted with an electron beam were also performed with similar results.

CONCLUSION

In summary, the current results of this study show that a large plastically deformed layer at the Nb surface is present after CBP#1; its depth varies over the cavity surface and is maximal at the equator area. Although the layer of significant deformation is removed by subsequent CBP#2, its initial presence might have facilitated hydrogen absorption into Nb.

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If only CBP#1 polishing is used for the cavity surface preparation, it should be followed by chemical polishing with at least 70 μm depth. Such a procedure reduces the advantage of the CBP as a replacement for bulk chemical polishing. On the other hand, CBP#2 polishing can be used as the single polishing step in a single-step CBP. In this case, the duration of the polishing step should be increased (~3 times) to compensate for a lower removal rate of CBP#2 comparing to CBP#1 of basic recipe. Using CBP#1 followed by CBP#2 in the industrial production would not be optimal due to doubling of the cleaning, preparation and control procedures required.

The thickness of the plastically deformed layer can be decreased by reducing the rotation speed of the barrels during the CBP process. However, this results in reduction of the abrasion rate.

Formation on niobium hydrides upon cooling to liquid nitrogen temperature was observed on the sample surface. High temperature baking should be used to degas hydrogen after CBP.

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