DETAILED SURFACE ANALYSIS OF INCREMENTAL CENTRIFUGAL BARREL POLISHING (CBP) OF SINGLE-CRYSTAL NIOBIUM SAMPLES*

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Abstract

We performed Centrifugal Barrel Polishing (CBP) on single crystal niobium samples/coupons housed in a stainless steel sample holder following the polishing recipe developed at Fermi Lab (FNAL) in 2011 [1]. Post CBP, the sample coupons were analyzed for surface roughness, crystal composition and structure, and particle contamination. Following the initial analysis each coupon was high pressure rinsed (HRP) and analyzed for the effectiveness of contamination removal. We were able to obtain the mirror like surface finish after the final stage of tumbling, although some defects and embedded particles remained. In addition, standard HPR appears to have little effect on removing embedded particles which remain after each tumbling step, although final polishing media removal was partially affected by standard/extended HPR.

INTRODUCTION

Superconducting radio-frequency (SRF) cavity technology is one of the key technology driving current particle accelerators. Modern SRF cavities are made out of high purity niobium sheets which are then formed into cavities. To achieve state–of–the art performance, these cavities must go through a multi-step process which condition the inner surface to support a high Q and maximum acceleration gradient (E_{Acc}). The usual method to treat the inner surface included initial bulk buffered chemical polish (BCP) and/or electropolishing (EP), followed by a high temperature bake to remove hydrogen, followed by another light BCP/EP, high pressure rinse (HPR), and low bake. This technique can achieve $Q_o > 2 \times 10^{11}$ and $E_{Acc} > 40 \frac{MV}{m}$ for a ILC 1.3 GHz cavity at 2K [4].

One of the alternate techniques to reduce bulk and light chemistry is mechanical polishing, pioneered at KEK in Japan [2]. In addition to reducing chemistry, recent work at FNAL by Cooper *et. al* [1] has shown that centrifugal barrel polishing (CBP) can reduce the surface roughness by a order of magnitude lower than chemistry alone [3]; which in turn possibly raises the Q thereby improving the performance.

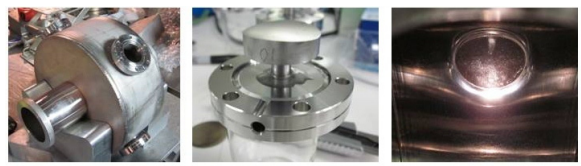

Figure 1: Sample mounting of niobium coupon in CBP holder. Left, the stainless steel holder - diameter is 9 inches; middle, coupon mounted on a 2 $\frac{3}{4}$ conflate flange before CBP, the hole in the flange contains a set screw to adjust the height of the rod; right, coupon mounted inside SS container - looking though the opposite coupon holder port.

Most CBP recipes rely on cavity testing with trial and error to ascertain a recipe for optimal performance. While this technique works, there should be a physical quantity which can help guide further recipe refinement. These CBP quality factor could include visual appearance, surface roughness, contamination size and shape, and/or crystal structure deformation. There are two readily available ways of ascertaining this information; one is to CBP a cavity until it reaches a "good" performance and then sacrifice the cavity by cutting out samples which can then be placed in a analysis equipment such as atomic force microscopy (AFM), scanning election microscopy (SEM), and electron back scatter diffraction (EBSD); the other would be to perform the analysis on sample housed in a host sample holder. While the first may be ideal, the second is cheaper and throughput can be much higher.

In this work, we perform CBP on single crystal niobium samples/coupon housed in a stainless steel coupon holder following the mirror smooth CBP recipe developed at FNAL, and analyzed the coupons with multiple surface characterizing techniques. We were able to reproduce the mirror finish published yet our mirror finish also contained scratches and embedded media. Our results indicate that each step in the process removes all signs (embedded media) of the previous steps, yet residual embedded media from each step always remain. The effects of high pressure rinsing were also evaluated with promising results for complete colloidal silica removal with multiple/extended

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Table 1: FNAL recipe comparison of removal rates on a fine grain cavities and large grain coupon removal rates following the same recipe at JLab. The spread in our removal rates comes from the variation between coupons.

FNAL recipe	processing time (hours)	published removal rates ($\frac{\mu m}{h}$)	coupon removal rates ($\frac{\mu m}{h}$)
9mm x 9mm triangles (Course)	10	11	27-31
RG-22 cones (Medium)	12	3	7-9
400 mesh alumina (Polish 1)	15	>1	1-1.5
800 mesh alumina (Polish 2)	20	>1	0.5-1
colonial silica (Polish 3)	40	negligible	negligible

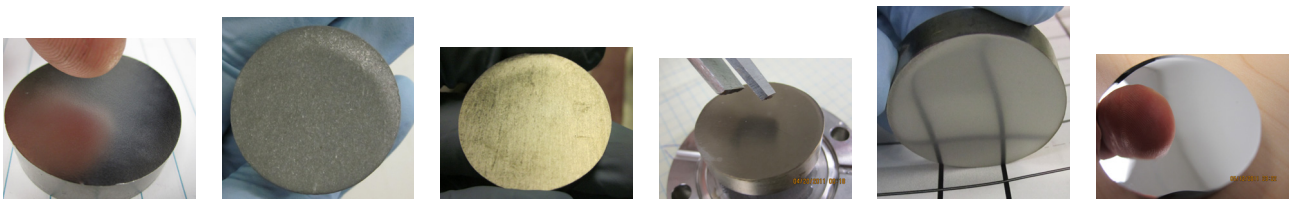

Figure 2: Image of a representative coupon for each CBP process start from the PRE-CBP BCP sample on the left and moving right, 9mm×9mm KM + 40:1 DI water:TS Compound, RG-22 cones + 40:1 DI water:TS Compound, 400 mesh alumina + wood blocks + DI water, 800 mesh alumina + wood blocks + DI water, and colonial silica (40nm) + wood blocks.

rinsing cycles.

CENTRIFUGAL BARREL POLISHING (CBP)

All single crystal samples were CBP'ed inside a stainless steel cavity holder designed by Peter Kneisel at JLab [5], and mounted into a CBP machine manufactured specifically for SRF polishing by Mass Finishing [6]. We mounted each $1\frac{3}{8}$ coupon on a niobium rod using high temperature mount wax (South Bay Technology Quick Stick 135 mounting wax) fitted into a $2\frac{3}{4}$ conflate flange, see Figure 1 middle. Because the holder has been used in the past with smaller coupons the edges of the SS holder have been warn down, leaving an exposed edge (Figure 1 right) which deviates from the curvature of the coupon. Each coupon was cut out of single crystal piece of niobium with a surface orientation of 110, verified by EBSD.

Following the FNAL recipe, each step was performed with the sample holder filled 50% with media and then topped off with liquid (DI water + TS compound or DI water only or colidal silica). The details of the recipe and removal rates comparison are shown if Table 1, with the image of the coupon after each step shown in Figure 2. Between each CBP step the coupons were rinsed with acetone to remove the mounting wax. This allowed the removal rates to be measured at the center of the sample using an Olympus Panametric NDT 25DL Plus ultras sonic thickness gage. Since the coupon holder contained 4 samples, all samples went through the first two step, and then for each addition step a coupon was removed; leaving only one coupon for the final step.

COUPON ANALYSIS

Scanning Electron Microscopy (SEM)

SEM was used to find the contamination level and embedded media for each step of CBP. For the first step using KM triangles (Al_2O_3 and SiO_2) both about 8-10 μm in diameter were left behind. Step two left behind SiO_2 about 1-3 μm in diameter, in addition Al_2O_3 was also found (not shown), suggesting it was left behind from the first step (or contamination). Step three and four left behind Al_2O_3 in the range of 3-5 μm. The last step was unable to remove all the Al_2O_3 from the third or forth step. Example embedded media indicative from each step is show in Figure 3. Colloidal silica is only found on our sample in crack/fishers on the surface which contain Al_2O_3 particles, or where it looked like a peace of Al_2O_3 used to be embedded (see section on HPR).

Atomic Force Microscopy (AFM)

Each coupon was analyzed by AFM over multiple 50 μm × 50 μm section to find the RMS height and Zmax (highest point above the RMS height). The compiled data is shown in Figure 4. One can see the first step creates a surface rougher than the BCP surface, yet after the medium CBP step the RMS surface roughness and Zmax are recovered. Two surprising feature of the data is polishing step 1 (4 from Figure 4) may not be needed, and the manufactured media would appear to have a more uniform finish than the first two polishing steps.

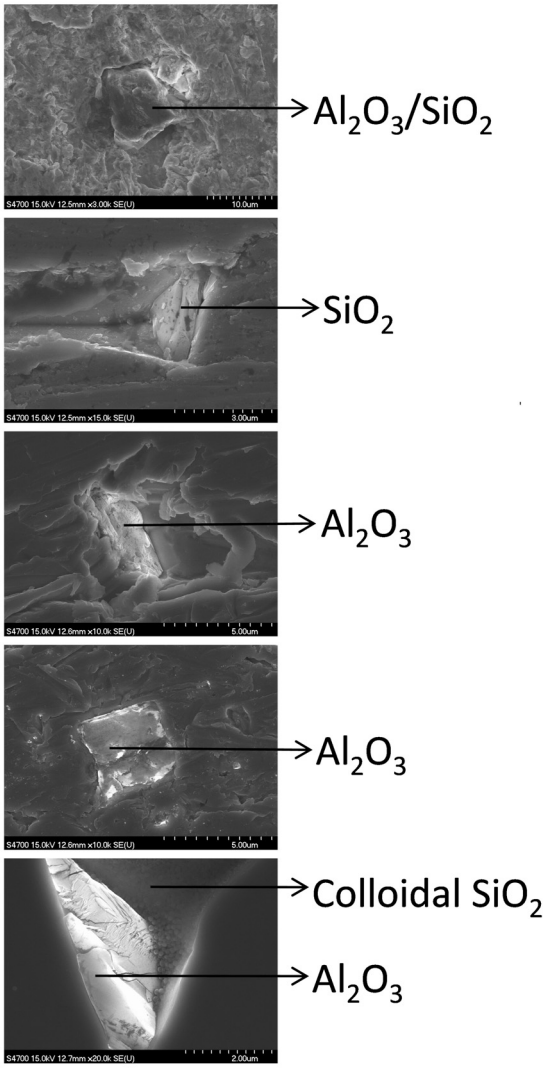

Figure 3: SEM image of embedded media from each CBP step moving - from top to bottom: course, medium, polish 1, polish 2, polish 3. Particle size and description found in text.

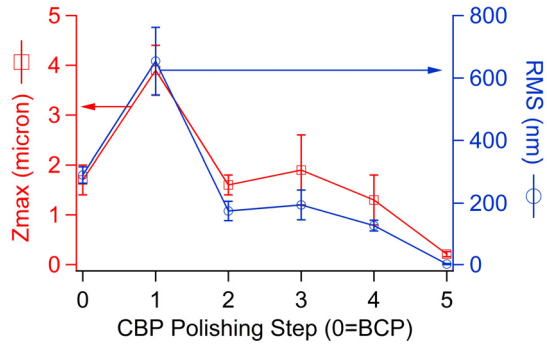

Figure 4: AFM data for each step in the CBP process. RMS height taken over multiple a 50m X 50 m section of each coupon, the error bars represent the span of measurements from multiple sampling areas. Zmax for each coupon, the error bars represent the span of measurements from multiple sampling areas.

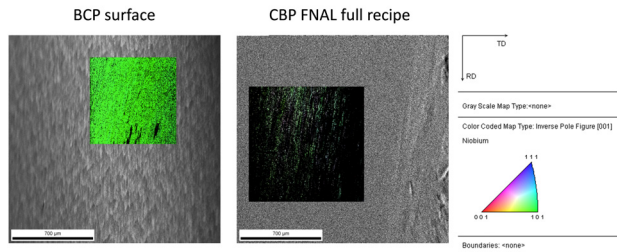

Figure 5: Electron back scatter diffraction (EBSD) before CBP on a fresh BCP surface (left) and after the FNAL CBP recipe (middle). The gray background is the SEM image of the surface, the color insert (right) indicates the appearance of a periodic crystal structure crystal orientation is present at the surface. BCP C.I.=0.87 and CBP C.I.=0.20

Electron Back Scatter Diffraction (EBSD)

Each coupon was analyzed with EBSD to ascertain the surface crystal structure for all CBP step. The initial BCP surface showed a clear 110 crystal orientation (average confidence index $C.I. = 0.87$), but every step of CBP destroyed the crystal orientation, at least within the 40nm probing depth of EBSD (Figure 5). Only after the full recipe did any coupon show any sign of crystal structure with EBSD, although still not nearly as periodic as the BCP surface which showed a $C.I. = 0.87$ (CBP $C.I. = 0.20$).

High Pressure Rinsing (HPR)

After each polishing step, the coupon underwent a cleaning process of a low pressure DI water rinse, ultrasonic cleaning in acetone for 10 minute, ultrasonic cleaning in DI water with Micro–90 detergent for 10 minute, and a final ultrasonic cleaning in DI water 10 minute. The initial cleaning was unable to remove any embedded media, see previous sections. After the initial surface analysis, all coupon underwent a high pressure (HPR) rinse in DI water at 1250 PSI and at a distance of 3.5-3.75 inches for 1 hour to evaluate contamination removal. The HPR system setup used the standard single cell rinsing cycle for Jefferson laboratory where the coupons/cavity rotates around the spay head in a 360 degree pattern at 2 rpm, and the rising wand moves up at a stepping rate of 0.5 inches per minute for a total of 80 passes, about twice as long as a standard single cell. The HPR was unable to remove the embedded media from the first four steps, but it did seem to remove at least some portion of the colloidal silica (Figure 6 left). After an additional 6 hours HPR, in the same way as before, more colloidal silica was removed (Figure 6 right). Where the HPR removed the colloidal silica small residue circles remained, presumably (but not verified) from the glycerol

suspension, but this was also reduced by extended HPR.

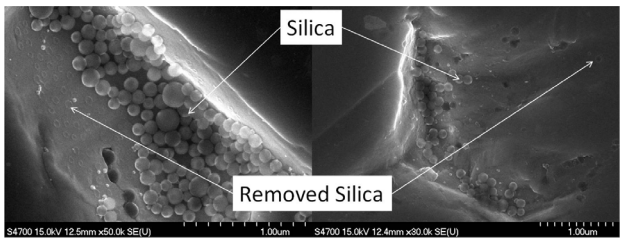

Figure 6: SEM image surface CBP coupon after a 1 hour HPR cycle (left) and an additional 6 hour HPR (right), see text for HPR information.

CONCLUSION

We have presented a systematic study of the CBP surface of single crystal niobium coupon using the 2011 FNAL recipe. We found the removal rates were 2-3 time higher than what was published for standard fine grain niobium cavities. This is possibly due to the samples being large grain rather than the published fine grain, or a property of the stainless steel sample holder. We found all tumbling steps left behind media embed which were not removed by HPR, but in the final polishing step the residual media could at least be reduced. In addition, it appears the first polishing step could be removed from the recipe, without effecting the final surface roughness.

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