STUDY ON PARTICULATE RETENTION ON POLISHED NIOBIUM SURFACES AFTER BCP ETCHING*

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Abstract

Niobium surface defects and inclusions can be introduced during the manufacturing processes used in the production of SRF cavities. Bulk removal methods (sanding, polishing, etc...) are frequently utilized to remove or smooth away these defects on the surface of the niobium metal. It is hypothesized that these mechanical removal methods are capable of trapping performance-degrading particulates, which are then exposed during subsequent chemical processing, potentially contaminating the cavity prior to RF testing. This paper summarizes results of a series of surface roughness and etching experiments performed to determine the relationship between the extent of polishing and trapped particulate, and to determine a method for mitigating this particulate contamination through BCP etching. The relationship between these experiments and RF cavity performance will be explored as well.

INTRODUCTION

The construction of the Facility for Rare Isotope Beams (FRIB) driver linac will require more than 350 niobium superconducting radio frequency (SRF) cavities. Four types of cavities will be used in the linac: 80.5 MHz β = 0.041 and β = 0.085 quarter-wave resonators, and 322 MHz β = 0.29 and β = 0.53 half wave resonators. Each of these cavities must be fabricated with utmost precision to ensure proper functionality of the cavities during RF testing and during the operation of the completed accelerator. Of the many critical aspects of these RF cavities, the surface finish of the niobium exposed to RF fields is paramount to the proper functionality of these cavities during testing. Unfortunately, despite the best of intentions and the most well-defined procedures and quality assurance protocols, issues can arise that compromise these niobium surfaces. Weld spatter deposited on the niobium during electron-beam welding of cavity components, foreign debris and inclusions imbedded in the niobium during sheet rolling or part stamping, and gouges or scratches occurring due to mishandling or machining errors can all impact the quality of the cavity surface [1]. Fortunately, these defects can frequently be fixed by means of mechanical abrasion. While these repairs are necessary, unfortunately, the use of abrasive media to repair the niobium surface can cause lasting degradation of the surface finish, and, as a result, potential degradation of cavity performance due to particulate contamination on the cavity surface. This study seeks to examine the lasting impact of these repairs, and to determine a method for mitigating the consequences by means of processing and analyzing a series of niobium samples.

EXPERIMENTAL

Two lots of niobium samples were used in this experiment. Each lot consisted of a series of small grain, high-RRR (>250), 3 mm thick sheet stock samples, shear-cut to approximately 2.5 cm square. Each sample was stamped with a serial number, photographed, degreased with acetone, ultrasonic cleaned, and precision measured for thickness (±0.0025 mm) with an NDT Systems Model TG900 ultrasonic thickness gauge.

As is typically performed with RF cavities, following cleaning, the samples were chemically etched with a standard solution of buffered chemical polish (BCP). The BCP mixture used at FRIB is a 1:1:2 mixture of concentrated hydrofluoric (49% w/w), nitric (70% w/w), and phosphoric (85% w/w) acids. The first lot of samples was divided into eight sub-lots. The sub-lots were each submerged in BCP and etched for between 60 and 480 minutes, removing up to 300 microns of niobium from the surface of the samples. Upon completion of the etching, the samples were thoroughly rinsed with ultrapure water (UPW) and set to dry in an ISO 5 cleanroom. Once dry, the surface roughness (both R_a and R_z) at six different locations on each sample was measured with a Fowler 54-410-500 X-Pro Portable Surface Roughness Tester.

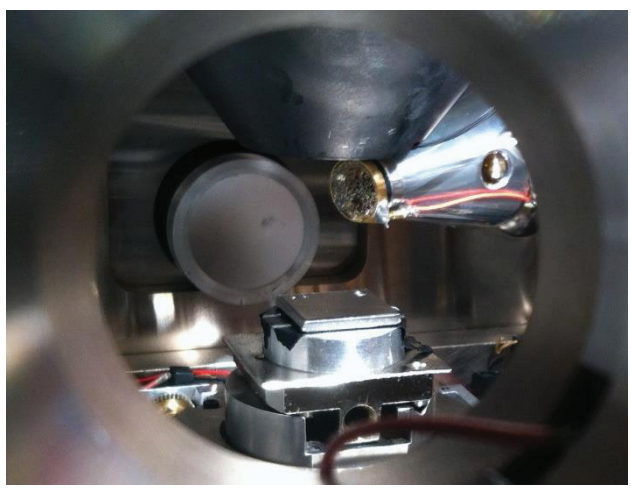

Figure 1: A niobium sample on the microscope stage as seen through the SEM's vacuum viewport.

To simulate a portion of an RF cavity repaired with an abrasive media, the second lot of samples was "polished"

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for five seconds per sample with a pneumatic Dyno-File tool equipped with a medium-grit aluminium oxide conditioning belt. Ultrasonic thickness measurements showed that the abrasion did not remove an appreciable amount of material from the sample surface. Following the abrasion of the samples, they were divided into sub-lots, and ultrasonic cleaned once more. Once clean, the abraded samples were etched in the same manner as the as-received samples, then were rinsed and dried in the cleanroom and measured for surface roughness.

Once the surface roughness characterization of the samples had been performed, a representative set of samples were analyzed with a CamScan 44FE Field Emission Gun Scanning Electron Microscope to allow the topography and particulate retention of the samples to be observed in great detail (Fig. 1).

DISCUSSION

The development of the surface roughness for the as-received samples followed the same well-defined pattern that had been reported previously for chemically polished niobium surfaces [2, 3]. The surface roughness, R_z, prior to etching was very small, though it steadily increased for the first ~100 microns of etch removal. At this point, it began to plateau, and stayed relatively constant for the remainder of the etching procedure. The more surprising outcome was the evolution of the surface roughness for the abraded samples. Not surprisingly, the initial surface roughness of the abraded samples was significantly higher than was observed on the as-received samples (Fig. 2).

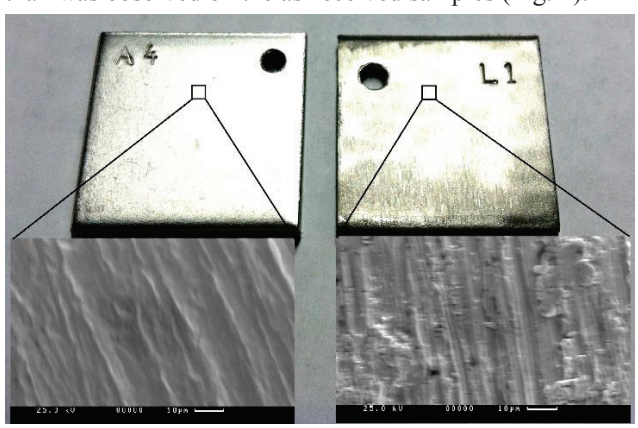

Figure 2: As-received niobium sample (left) compared to an abraded sample (right). Note the difference in surface roughness, both in the picture and in the SEM scans.

Once etching was commenced, the surface roughness fell sharply during the first 50 microns of removal, at which point the roughness began to climb, then plateau in a manner consistent with the as-received samples. The surprising finding was that the surface roughness of the abraded samples never converged with the as-received samples (Fig. 3). A similar pattern was observed with R_a measurements. The implication here is that under the conditions explored in this experiment, once a niobium surface has been abraded, the surface roughness will not return to a state consistent with a surface that has not been altered with an abrasive media. As can be seen in Figure 4, while the samples look very similar to one another with the naked eye, microscopy shows that they are not. Note the difference in the overall surface finish, and observe the valley that runs through the center of the image. These crevasses were seen in several locations on the abraded samples up until at least 100 microns of niobium had been removed from the surface. This has the potential to have serious consequences on RF cavity performance [4], since the increased surface area of a rougher material allows more room for particulates to become trapped on the surface, potentially resulting in field-emission and cavity quenching during operation.

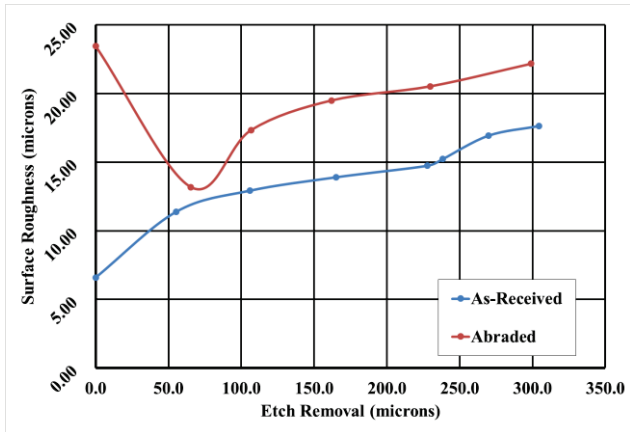

Figure 3: Plot of etch removal vs. surface roughness for the as-received and abraded niobium samples for different etch removal amounts.

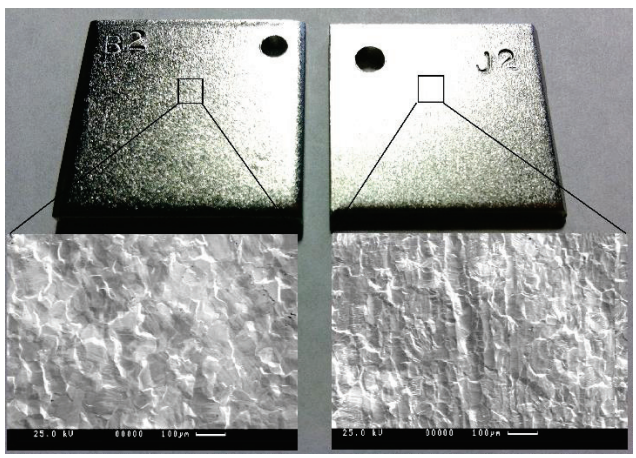

Figure 4: As-received niobium sample (left) compared to an abraded sample (right) following 70 microns of etching. Abraded sample has visibly worse surface finish under the microscope.

In addition to the concerns about the roughness of the niobium surface, there was also evidence to suggest that on top of the passive role that abrading can play in particulate retention, particulate was actively being ground into the surface of the niobium. These particulate were only exposed after significant amounts of etching had been performed (Fig. 5). While these particulate are not necessarily a *guarantee* of a bad cavity test, they are

certainly not preferable, and can contribute to less than optimal cavity performance, especially at high-field.

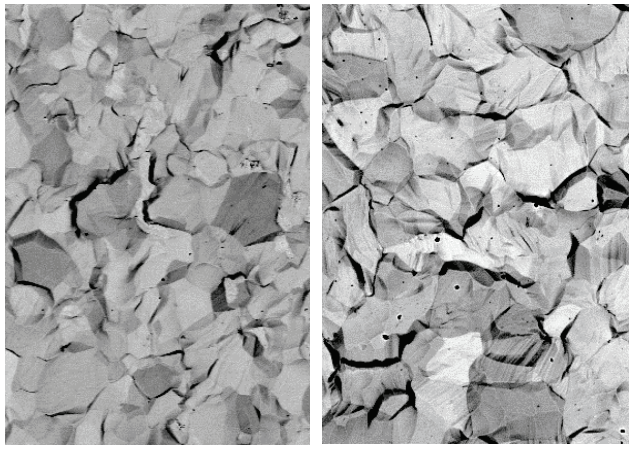

Figure 5: SEM scans of an as-received sample (left) and an abraded sample (right) after more than 100 microns of etching. Note the unusually high concentration of black particulate spots on the abraded sample.

SRF Cavity Repair: A Case Study

A β=0.085 quarter-wave resonator intended for use in the ReA3 coldmass was recently processed in the FRIB chemical etching and cleanroom facility. Once assembled and pumped down, the cavity demonstrated a large weld leak between the helium vessel and RF space along one of the cavity's flanges. At this point, the cavity was sent for repair at an electron-beam welding facility. During welding, suspected contamination in the weld area caused a significant amount of weld spatter to be deposited on the RF surfaces inside of the cavity.

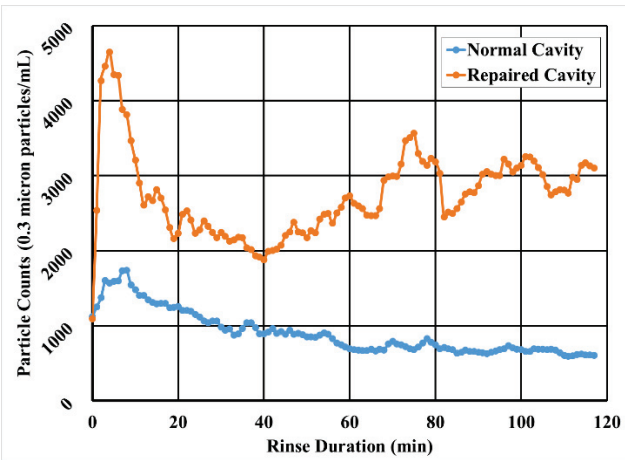

Figure 6: Liquid particle count comparison between a normal and repaired cavity during high-pressure rinsing.

The same abrasion method used in this experiment was used to remove the spatter and smooth out the resulting imperfections on the cavity surface prior to a chemical etch and high-pressure rinse (HPR) being performed. Liquid particle counts taken during the HPR were more than five times higher than were historically observed for a cavity of this type (Fig. 6). HPR was continued for an additional two hours, at which point the particle counts began to fall back to an acceptable range. Unfortunately, the subsequent cavity assembly and leak check revealed another leak requiring additional repair, so no comparison between cavity test results could be established. Despite the fact that no test results were gathered for this repaired cavity, data gathered over the course of many cavity processes indicates a correlation between low particle counts during HPR and superior cavity performance.

CONCLUSION

While the repair of surface defects on SRF cavities is often a necessity, careful consideration should be given to the methods and materials used. If abrasives must be used, as small of a surface area as possible should be affected. Care should also be taken to adjust processing procedures following these repairs to ensure the highest quality product possible. At a minimum, extended etching and high-pressure rinse cycles are recommended prior to assembly for RF testing. The necessity of repairs should be considered as well. Frequently, damage or inclusions on welded surfaces can cause defective welds; these must be repaired. However, if these issues are seen on other cavity surfaces, chemical polishing alone can often mitigate the damage without causing long-term impacts to the quality of the cavity surface. These less aggressive repair solutions should be pursued whenever possible.

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