STUDY CORRELATING NIOBIUM SURFACE ROUGHNESS WITH SURFACE PARTICLE COUNTS

C. Compton, L. Dubbs, K. Elliott, D. Miller, R. Oweiss L. Popielarski, K. Witgen
Facility for Rare Isotope Beams (FRIB), Michigan State University, East Lansing, Michigan, USA

Abstract

A study has been initiated at Michigan State University (MSU) to relate the surface preparation of Superconducting Radio Frequency (SRF) resonators and surface particle counts, using niobium samples. During fabrication, undesired surface roughness can develop on the internal surfaces of the resonators. The final cavity finish will be product of material forming, machining, welding, chemistry, high-pressure rinsing, and handling of the niobium material. This study will document niobium samples treated with MSU standard processing procedures; first measuring the surface roughness, then polishing samples with defined techniques, processing, and measuring surface particle counts. The samples will include as received niobium, machined surfaces, welded surfaces, and surfaces with characterized surface imperfections (scratches).

INTRODUCTION

The Facility for Rare Isotope Beams (FRIB) at Michigan State University is an approved ~550M$ project funded by a cooperative agreement between Michigan State University (MSU) and The US Department of Energy (DOE) for advancement in the study of rare isotopes. The driver linac for the FRIB project is an 200 MeV/u superconducting linac with final beam power reaching 400 kW. There are four types of resonators used; two quarter-wave resonators (80.5 MHz, Beta= 0.041 & 0.085) and two half-wave resonators (322 MHZ, Beta= 0.29 and 0.53) [1]. The resonators are house in rectangular, bottom loaded cryomodule with internally built focusing solenoids.

Acquisition strategies have been drafted for the major components of the FRIB linac, including the procurement of the 341 required SRF cavities. As part of the acquisition specifications, a study was initiated to correlate niobium surface roughness with post-processing particle counts and define the acceptance criteria. The study will also establish a mechanical polishing procedure to be used on SRF cavity surfaces as a means to repair surface defects. Surface defects are defined here as surface irregularities occurring from fabrication or processing of the cavities. Surfaces can become damaged as a result of machining (tool marks, tool breaks) electron-beam welding (sputter, undercutting), and cavity mishandling (scratches, dings).

The study will use small niobium samples, measuring both surface roughness and surface particle counts as the samples are exposed to polishing methods and standard SRF cavity processing.

Using a QIII+SPD (Pentagon) surface particle counter, surface particles will be measured before and after the samples are processed. Samples will be prepared in lots of three, with all samples in a lot receiving the same treatments. In addition, a lot of control samples will follow all sample processing runs.

SAMPLE PREPARATION

The initial study will use flat poly-crystal (~50μm), high RRR (>250) niobium samples. Samples will be 2 inches by 2 inches by 0.079 inches thick. The defined polishing technique for a given sample lot will be applied to a 1 inch by 1 inch square, centered area on the sample. Surface roughness measurements will be taken on samples as-received, after polishing, and after processing.

A Fowler Profilameter (54-410-500) was used to measure the surface roughness. Two small holes are used to mount samples to process tooling. A portion of the samples will be halved and electron-beam welded together, with a full penetration weld, to study the welded surface. All samples were processed using the same processing procedures, equivalent to procedures used in standard FRIB cavity processing; see PROCEDURE section. Data will be collected and recorded from all samples tested. Data presented is an average of the data collected from the three samples per lot.

Prior to processing, a selection of samples, with and with outs welds, will receive a surface polish, exploring multiple polishing methods and media. In addition, a selection of samples will be prepared with scratches to define acceptable polishing practices in the event scratches are observed on cavity surfaces. Scratches will be "man-made" using a scribing end mill to produce scratches with uniform width and depth across several samples/lots. Examples of the three types of samples are shown in Figure 1.

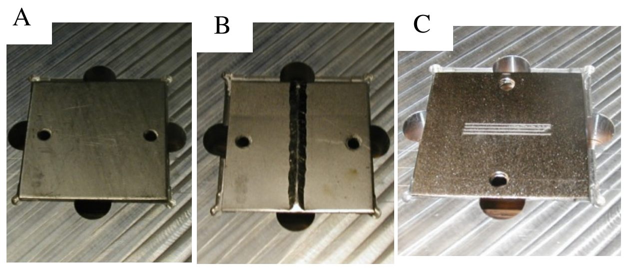

Figure 1: Samples are fabricated from high RRR niobium, 2” x 2” x 0.079”; A: As-received sample, B: Welded (full penetration) sample, C: Scratched samples.

SAMPLE PROCESSING

Samples, with the exception of control lots, will be treated with a defined polishing treatment, followed by standard cavity processing.

Sample Polishing

Several different polishing treatments were explored with two types chosen for the initial round of this study. A selection of samples will receive hand polishing using Scotch-Brite® media, utilizing three different levels of abrasion (very fine-Aluminium Oxide, super fine-Silicon Carbide, super fine-Aluminium Silicate). The second treatment will use a pneumatic tool (Dyno-File®), also utilizing three different levels of abrasion (coarse, medium, and very fine; all Aluminium Oxide). Sample lots will be prepared in multiple polishing sequences, preparing samples only receiving coarse polishing to samples cascading from coarse to fine polishing. Samples are held in a fixture using labelling marks to define orientation. A frame template is place atop the sample and used to ensure the 1 inch by 1 inch area has received polishing. All samples are polished using standard polishing techniques in which the sample is polished in one direction until present surface marks are removed. If additional polishing is done (with finer abrasive), the direction of the polish will be rotated 90° and repeated until previous marks are removed.

Sample Processing

After the samples have received their pre-processing treatments and measurements (electron-beam weld, scratched, polished, surface roughness) they are subjected to the current processing procedures foreseen for FRIB cavities. All samples are pre-cleaned (acetone→ Micro90 wash→ UPW rinse → methanol) and soaked in tap water for >12 hours. The water soak will allow the detection of any surface contaminants present from sample fabrication or preparation. Samples are then etched in 1:1:2 BCP for 100 minutes (~100-150µm removal). During etching, the acid is agitated and chilled to < 18°C. After etching, samples are rinsed in ultra-pure water and moved into a cleanroom environment. Samples receive a second rinsing and assembled onto the high-pressure rinse tool, shown in Figure 2. Samples are high-pressured rinsed for 22 second each (5.25 sec/in^{2,} proposed rate for FRIB processing) and allowed to dry in a Class 100 environment (>12 hours). After drying, surface particle counts are made and recorded.

Particle Counter

Particle counts, using the QIII+SPD (Pentagon) as shown in Figure 3, were taken prior to sample etching and at the conclusion of the processing cycle. The surface counter was used under cleanroom HEPA filters and purged until a zero background signal was obtained, at the 0.3µm scale. All samples were then measured by having the surface particle counter probe scan the polished surface area of 1 in^2 at the middle of each sample (multiple channels; 0.3, 0.5, 1,5, and 10 µm).

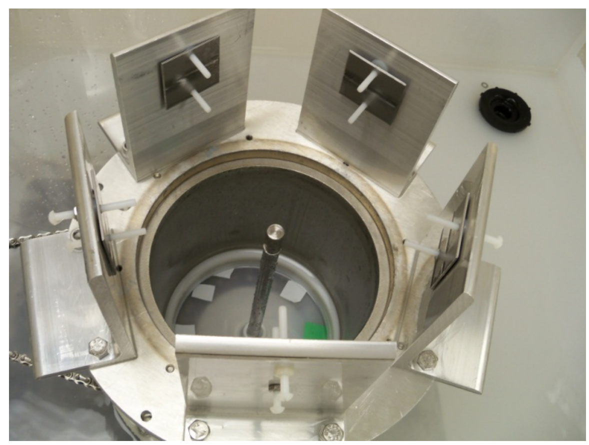

Figure 2: High-pressure rinsing of niobium samples; 5 samples on tool per run (5.25 sec/in^2)

Figure 3: A QIII+SPD surface particle counter will be used to measure particles on samples before and after processing.

RESULTS

When looking at the particle count data from the pre-processed samples, particle counts correlate well with the sample polishing history; as shown in Tables 1 and 2. The samples receiving a three stage polishing treatment (coarse→medium→fine) produced smoother surfaces with fewer particles than samples given a single stage coarse polish. This was observed in both the as-received and welded samples. As-received samples receiving no additional polishing also measured low particle counts.

After Processing, the non-scratched samples showed no measurable surface particle counts, as shown in Table 2. The surface roughness of the samples did increase after processing, but was consistent with surface roughness observed in other BCP treated surfaces. Samples receiving scratches had measurable surface particles both pre and post processing.

Table 1: Table shows sample lot preparation information including: pre-polishing and polishing treatments and before and after surface roughness measurements

Sample lot #	Pre-polishing treatment	Polishing treatment*	As-received/Polished average surface roughness (micron)	Processed average surface roughness (micron)
1	As-received	N/A	0.312	1.612
2	As-received	Hand (1)	0.301	1.602
3	As-received	Hand (2)	0.264	1.486
4	As-received	Hand (3)	0.218	1.514
5	As-received	Dyno-File (1)	1.455	1.508
6	As-received	Dyno-File (2)	1.307	1.559
7	As-received	Dyno-File(3)	0.886	1.452
8	Welded	N/A	0.852	1.037
9	Welded	Dyno-File (1)	1.497	1.215
10	Welded	Dyno-File (2)	1.546	1.408
11	Welded	Dyno-File(3)	0.9122	1.096
12	Scratched	N/A	76.2 depth	3.833
13	Scratched	N/A	152.4 depth	6.349

* (1) = coarse, (2) = coarse→medium, (3) = coarse→medium→fine

Table 2: Table shows surface particle measurements of sample lots before and after processing

	Before Processing		After Processing	
Sample lot #	Average Particle Counts (0.3μm)	Average Particle Counts (0.5μm)	Average Particle Counts (0.3μm)	Average Particle Counts (0.5μm)
1	0	0	0	0
2	0.33	0	0	0
3	0.33	0	0	0
4	0	0	0	0
5	9.8	6.6	0	0
6	7.3	4.5	0	0
7	1.3	0.6	0	0
8	0	0	0	0
9	3.0	1.7	0	0
10	1.1	0.6	0	0
11	0	0	0	0
12	2.67	0.52	1.67	0.33
13	5.0	3.0	5.11	3.33

DISCUSSION/ FUTURE WORK

The initial results of the study are encouraging. The Q3 counter is shown to be a good tool for measuring surface particles. The counter provided data that was consistent across a sample lot (1 lot = 3 samples) and made sense when comparing across multiple sample lots.

The after processing data confirms that the high-pressure rinse system is affective in removing particles at surface roughness levels equivalent to BCP treated samples. As the surface roughness increases, post processing particles were measured. The data from the scratch samples supported the idea as scratch depth increases, particles can become trapped and hard to remove. Further tests are needed to determine the scratch depth limitation.

Further research will continue with efforts to correlate surface roughness (with a focus on scratch depth) to post processing particle counts. The study will define an accepted procedure for polishing welds or surface defects that will be drafted into a production specification. Future samples will also include investigating formed material mimicking both rolled and deep drawn surfaces found in FRIB cavity designs.

ACKNOWLEDGEMENTS

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REFERENCES

[1] M. Leitner *et al.*, in *Proceedings of the 15th International Workshop on RF Superconductivity, Chicago, IL, USA, 2011*, MOPO009.