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SUMMARY OF FRIB CAVITY PROCESING IN THE SRF COLDMASS PROCESSING FACILITY AND LESSONS LEARNED*

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Abstract

Baseline coldmass production for the linear particle accelerator at the Facility for Rare Isotope Beams (FRIB) is nearing completion. This paper will review the processing of cavities through the FRIB superconducting radio frequency (SRF) coldmass production facility focusing on chemical processing and high-pressure rinsing. Key processing data will be compiled and correlations between processing variables and cavity RF testing results will be examined.

SUMMARY OF CAVITY PROCESSING

Superconducting radio frequency (SRF) cavity production for FRIB began in November of 2014. With ten beta=0.53 half-wave resonator (HWR) cavities left to certify for the project at the time of writing, the run of production is nearing completion. Cavities undergo many processes before being assembled to a coldmass. The focus of this paper will be on the chemical etching and high pressure rinse processes.

A total of 1135 processes for 349 unique cavities were performed in the chemical etching facility (summarized in Fig. 1). This consumed 87 barrels or approximately 16500 liters of 1:1:2 buffered chemical polish and removed 600 kilograms of material from cavities. A total of 504 high pressure rinse processes were performed and are summarized in Fig 2.

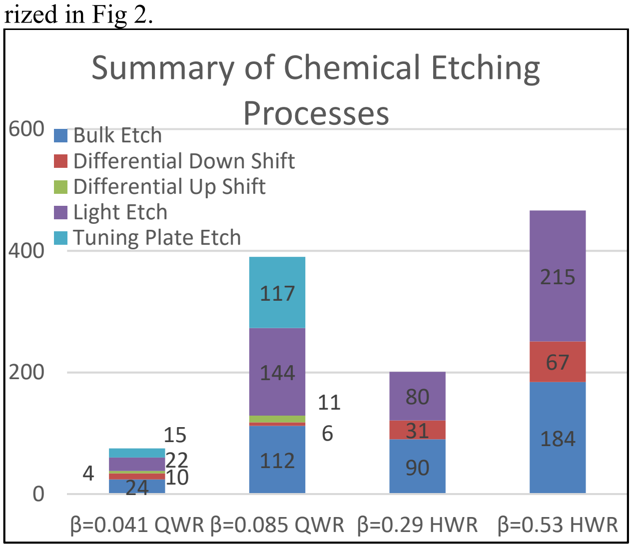

Figure 1: Bar chart summarizing chemical etching processes for FRIB cavities.

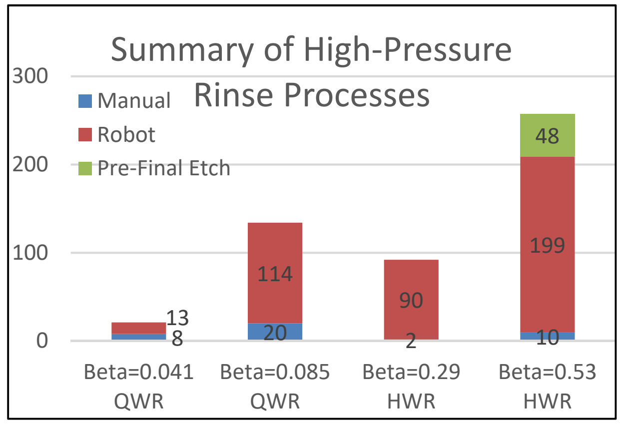

Figure 2: Bar chart summarizing high-pressure rinse processes for FRIB cavities.

PROCESS IMPROVEMENTS

Continuous process improvement is critical to the success of any project. Either unforeseen problems arise from the translation of conceptual designs to real world applications, or ideas for improvement become apparent when processes are put into practice. Many changes were made to the chemical and high pressure rinse processes to improve cavity performance.

Improvements to Chemical Etching Process

Acid Injection Quill Alteration One issue that plagued both HWR type cavities early in production was the presence of divots that formed on short plates after bulk etch processing. An example of these divots can be seen in Fig. 3.

Figure 3: Borescope image of an etching feature that formed during the bulk etching process due to the close proximity of the end of the acid quill to the short plate.

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All etching processes of FRIB cavities utilize etching quills inserted into the cavities to guide acid flow around the cavity space [1]. These quills are installed to the rinse ports of HWR's and extend into the cavity space stopping close to the short plate. The lengths of these quills were designed based on cavity 3D models/ manufacturing drawings. In practice, cavity fabrication variances resulted in the lengths between the short plates and the rinse ports to differ from cavity to cavity. If this distance was significantly shorter than drawings, upon quill installation, the ends of the quills would come close enough to the short plate to affect acid flow patterns which resulted in features such as those seen in Fig. 3.

This was a significant issue that had potential adverse effects on cavity testing performance. The fix for this issue was to simply shorten the quills and having operators verify that the quills were not making contact with the short plate upon quill installation to the cavity.

Acid Vapor Residue Reduction Another improvement of the chemical etching process related to the reduction of acidic vapour residue. It was noticed during borescope inspections of cavities being reworked due to failed vertical tests that a localized white, cloudy surface may be contributing to increased field emission. An example of the acid vapour residue can be seen below in Fig. 4.

Figure 4: An example of acid vapour oxidation seen after a bulk etch process.

It was posited that this acid vapour residue may be forming in the time between the end of the acid etching step of the etch process and before the cavity fills with ultra-pure water during the initial rinsing step. Further investigation needs to be performed to determine the nature of this residue (niobium oxide, phosphate, salt, etc.).

In an attempt to remedy this issue, procedural steps were changed to reduce the time that residual acid remained on cavity surfaces. The first step was to drain the acid as quickly as possible by draining all acid through the outlet set of etching quills. The acid drains more quickly out of the outlet set of quills than the inlet set of quills due to larger holes in the quills and the presence of weep holes at the quill base. Secondly, when the cavity is approximately half full of ultra-pure water during the cavity rinsing step, the cavity is rotated 180 degrees to rinse the top half of the cavity. These steps reduced the duration of the cavity surfaces being exposed to residual acid by almost half.

Qualitatively, improvement was seen during borescope inspection after etch processes. There was noticeable reduction in the amount of oxidation seen on cavity surfaces. Improvement has also been noted during vertical testing in the form of reduced conditioning times.

Improvements to High Pressure Rinse Processes

Total Organic Carbon (TOC) Measurement In March of 2016 a robot was installed to high pressure rinse cavities. This was a significant upgrade from the manual system previously employed to perform high pressure rinses, the benefits of which were discussed previously [2]. This high pressure rinse system has proved very reliable with less than six weeks of down time in three years, has not damaged a single cavity during processing, and has also proved to be flexible so making changes have had little impact on employee resources.

One key change that was made to the process to increase reliability was the addition of TOC measurements. In November 2017, there was a string of four cavity tests that failed due to high field emission at operating gradient. It was found that seals inside the high-pressure rinse pump failed earlier than expected which resulted in either oil or debris from the seals breaking down to contaminate the ultra-pure water being pumped to cavities. In addition to the failed cavity tests, there were several more cavities that were processed, assembled and ready for vertical testing that would need to be reprocessed. The deterioration in water quality was not seen in liquid particle count (LPC) measurements which, up to that point, was the only quality control check for the rinse process. Testing of the contaminated water yielded TOC measurements of 1000 ppb which is the maximum value of the TOC analyser. For reference, typical TOC measurements seen after cleaning and recertification of the system are under 200 ppb. This added quality control test will prevent cavities from being rinsed with contaminated water in the future and reduce downtime related to cleaning the system if the issue is caught as early as possible.

Rinse Process Changes Many changes have been made to the process over the course of production. Some examples include increasing the rinse time, reducing the speed of the wand moving through the cavity, changing the speed at which the wand arm rotates, changing the hole pattern on the nozzle, changing the port rinsing order, and altering the start and end points of the wand path through the port. All of these changes were made with simple programing or equipment changes. Many of these changes were made in response to poor cavity test results and were made based on intuition (ex. more rinsing means cleaner cavity surfaces) and were validated with data after several processes.

One example that improved the quality of cavity rinsing was the addition of a pre-final etch high pressure rinse. The goal of this added rinse process was to ensure that rinsing of the cavities after the degreasing process was thorough and consistent. There was some concern that residual Micro-90 degreasing agent (used in the ultrasonic cleaning process) remained in the cavity even after low pressure

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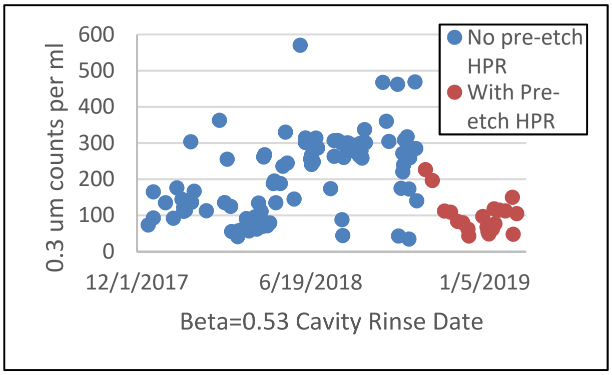

Figure 5: Scatterplot comparing LPC (0.3 um counts/ml) data collected at the end of final rinse processes during final cavity processing before and after the pre-etch HPR process was implemented.

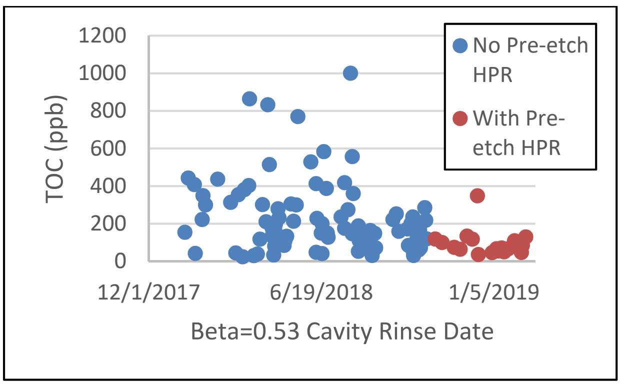

Figure 6: Scatterplot comparing TOC (ppb) data collected at the end of final rinse processes during final cavity processing before and after the pre-etch HPR process was implemented.

rinsing. Figure 5 and 6 show the quality control data collected at the end of high pressure rinse processes over time.

There is an obvious reduction in both LPC's and TOC after the addition of a pre-final processing light etch. This data supported adding the extra rinse step to the normal cavity production router. Intuitively, cleaner cavities will result in better performing cavities. As of the writing of this paper there was not enough test data to perform a statistical analysis.

FINAL ETCH NIOBIUM CONCENTRATION VS TESTING RESULTS

It has been reported elsewhere that the final etch of a superconducting cavity should be performed with BCP containing less than 20 g/L of niobium [3]. The concern is that higher niobium concentration can increase the formation of non-water soluble niobium phosphates on cavity surfaces. Final etching processes at FRIB have been performed without regard to niobium concentration of the acid. Anecdotal experience at FRIB led to the belief that niobium concentration had no impact on cavity performance over the course of FRIB baseline production. With a total of 420 final etching processes that were followed by cavity tests, there is a very large data set that can be analysed to determine if there is a correlation between niobium concentration of acid during final etch processes and cavity test performance. Four different cavity vertical test quality indicators were examined: field emission onset, field emission at operating energy gradient, quality factor at the operating energy gradient, and the maximum energy gradient. It is acknowledged that there are many variables that can affect testing results, but if there is a correlation it is likely that it would appear due to the quantity of data. Cavity test data was split into the separate cavity types (beta=0.085 QWR, beta=0.29 HWR, and beat=0.53 HWR). Select graphs for the beta=0.53 HWR test quality parameters can be seen below in Figs. 7 .. 9. Correlation data is presented for three cavity types in Table 1. Beta=0.041 data is omitted due to the small data set.

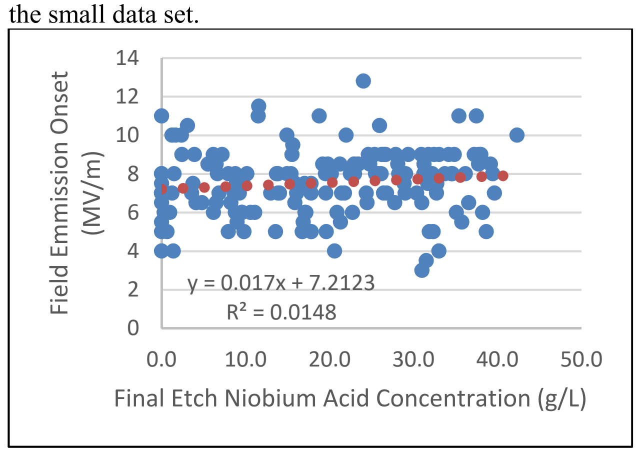

Figure 7: Field emission onset data for all beta=0.53 cavity vertical tests.

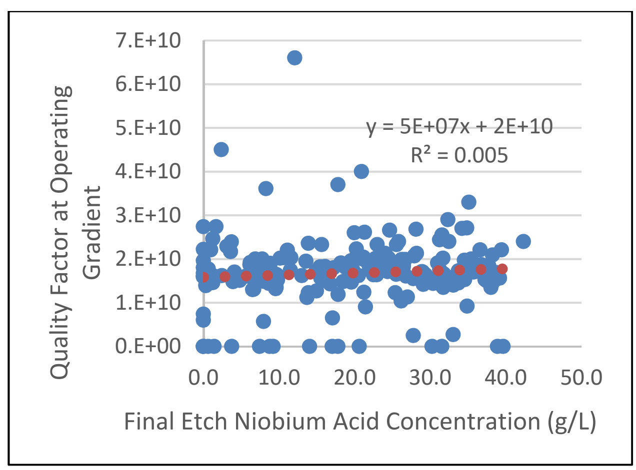

Figure 8: Quality factor data for all beta=0.53 cavity vertical tests.

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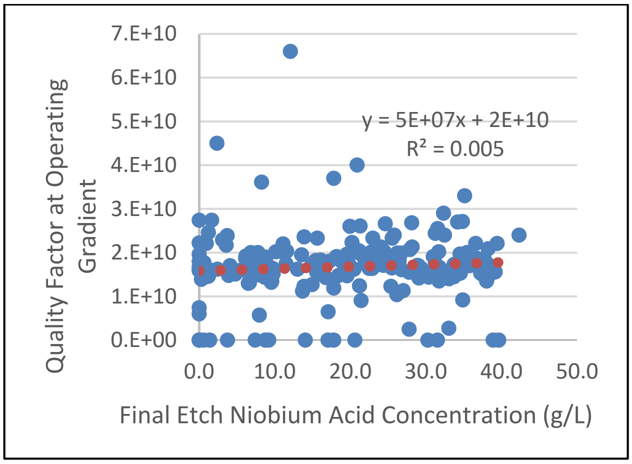

Figure 9: Quality factor data for all beta=0.53 cavity vertical tests.

Table 1: Final Etch Acid Concentration versus Test Data Correlation Summary

Cavity Type/ Test Parameter	Correlation Coefficient
Beta=0.085 QWR	
Field Emission Onset	0.16
X-rays at Operating Gradient	-0.02
Quality Factor at Op. Gradient	0.00
Maximum Field	-0.01
Beta=0.29 HWR	
Field Emission Onset	0.05
X-rays at Operating Gradient	0.06
Quality Factor at Op. Gradient	0.01
Maximum Field	-0.12
Beta=0.53 HWR	
Field Emission Onset	0.12
X-rays at Operating Gradient	-0.02
Quality Factor at Op. Gradient	-0.01
Maximum Field	0.05

As the correlation data shows, there is no relationship between any test data and the niobium concentration of the acid. This is significant because this shows that acid does not need to be changed prior to these processes which improves operational flexibility and reduces acid costs.

CONCLUSION

Baseline cavity production for the FRIB particle accelerator is nearing completion. To date, more than 1100 etching processes have been performed on 349 unique cavities and over 500 high pressure rinse processes have been performed. This has provided production staff at FRIB valuable experience that has been leveraged to help improve processes. The chemistry acid injection quills have been altered and procedural changes have improved cavity surface quality. Increased data collection and added rinse steps have improved rinsing reliability and quality. Additionally, all of these processes have provided a plethora of data that can be used to test certain notions regarding cavity processing and either confirm them as correct or offer an alternate view on such topics. For example, based on production data and test results, there is no correlation between niobium concentration in BCP and cavity vertical test data. It is the hope of the authors that insights such as this will prove beneficial to others in future projects.

ACKNOWLEDGEMENTS

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