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IDENTIFYING SPECIFIC CRYOMODULE AND CLEANROOM PARTICULATE CONTAMINATION: UNDERSTANDING LEGACY ISSUES AND PROVIDING NEW FEEDBACK STANDARDS*

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Abstract

While the techniques used to provide "UHV clean" and "particle-free" beamline components, including SRF cavities, continue to evolve, "real-world" operating machines must deal with actual accumulated and latent contamination issues that produce non-trivial cryogenic heatload, radiation, activation, and degradation via field emission. We have developed a standardized and automated particulate contamination assay method for use in characterizing particulates found on beamline components and in cleanroom assembly environments. We present results from using this system to analyze samples taken from reworked cryomodules from CEBAF. Particulate sizes are much larger than anticipated. Utility for feedback on sources to enable improved source reduction is explored.

INTRODUCTION

Performance standards for SRF-based particle accelerator systems continue to evolve. Surface material engineering continues to push boundaries in both dissipated heat and peak sustainable surface fields. The chief extrinsic limiting phenomenon, however, remains surface particulate contamination which become electron field emission sources when residing on locations with high surface electric field. While the techniques used to provide "UHV clean" and "particulate-free" beamline components, including SRF cavities, continue to improve, "real-world" operating machines must deal with actual accumulated and latent contamination issues that produce non-trivial cryogenic heatload, radiation, activation, and degradation via field emission.

In order to build specific actionable knowledge of contaminates found on CEBAF beamline components, we have developed a standardized and automated particulate contamination assay method for use in characterizing such particulates and assessing "clean" assembly environments. The process of sample collection and automated analysis is described in a companion contribution to this conference [1]. Here we review results from using this system to analyze samples taken from inter-cryomodule beamline girders, reworked cryomodules from CEBAF, and some initial results from current particulate source identification work in the JLab cleanroom activities.

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SAMPLING SYSTEM

As described in [1,2] we have developed a system for characterizing particulate contamination around the use of commercially available forensic standard clean carbon tape spindles that are conveniently analyzed in a scanning electron microscope (SEM). All of the work described here uses these samples, which are tracked via unique serialization numbers.

Samples were collected under controlled conditions, and reference environmental samples were collected to help discriminate particulates derived from the intended sampled surface from particulates arriving to the analyzed spindle independently during the collection process (see Fig. 1).

Figure 1: Standard sample spindle and example of particulate sampling from cavity interior.

CEBAF CRYOMODULE PARTICULATE ASSAYS TO DATE

Beamline Girders

During the servicing of diagnostic instrumentation on the CEBAF beamline in 2015-2016, a "C50" cryomodule known as "Franklin" and installed in 2009, was accidentally vented in an uncontrolled way. Subsequently, rf performance was severely degraded and showing very high field emission effects. This cryomodule was then the worst performer in CEBAF, and thus was selected for the next available rework cycle.

The adjoining "C20" cryomodule "Independence" followed in 2017 with removal for rework, being replaced by the refreshed Franklin module. Independence had been on the CEBAF beamline since installation in 1992.

We collected particulate samples from the inside of the adjoining girders prior to their re-processing. These girders have been on the CEBAF beamline since initial construction in 1992. The girders were isolated and

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brought into the cleanroom for sequential disassembly and interior surface sampling for particulates.

Figure 2 notes the locations that were sampled on the two girders which bracketed Franklin in CEBAF, and also similarly on the outboard girder adjoining Independence. Four additional beamline girders were sampled prior to their rework. These were the girders bracketing cryomodules in zones NL07 and NL23. The latter were girders that had been installed as part of the 12 GeV Upgrade project and bracketed the C100-6 cryomodule which was pulled for re-cleaning due to field emission loading.

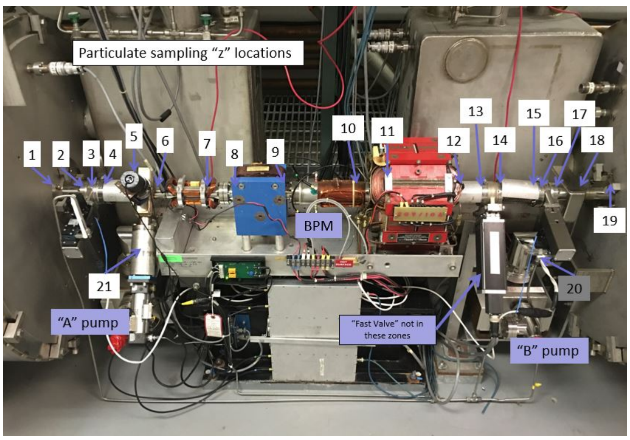

Figure 2: Particulate sampling locations on CEBAF warm girder.

Perhaps the most dramatic and telling sample is the one collected by shaking the bellows at sample position #13. The debris that fell on the spindle required no microscope to discern. See Fig. 3.

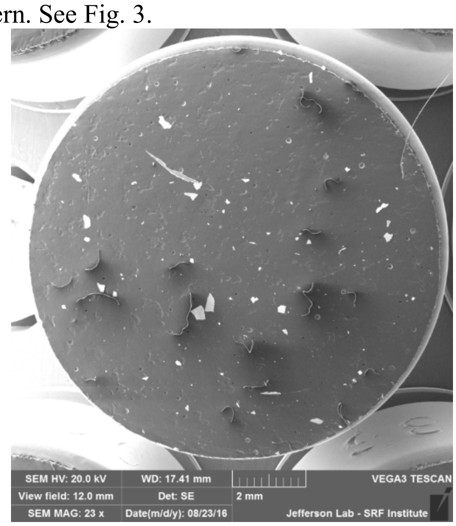

Figure 3: Sampled particulates from girder 1L12 from shaken bellows at location 13 in Fig. 2.

The following types of particulates were found in these girders:

- Cu flakes and long slivers
- SS flakes
- Zinc pieces and slivers
- Sand
- Ni
- Molybdenum sulfide
- Ta flakes
- Ti
- Cu/Ag flake with hydrocarbon fibers
- Si/Al/Mg & Si/Ca/Al at ion pumps
- Si/Mg/O
- Large hydrocarbon pieces – sheet-like

The size of the coper and stainless steel particulates found averaged over 50 µm. It is clearly understood that the particulate load present in these girders is incompatible with reliable operation of a superconducting linac. Remediation is being implemented at every opportunity, and increasingly stringent measures are being implemented to minimize transport opportunities for existing particulates on the CEBAF beamline.

Original Style Cryomodules

As the cryomodules Franklin and Independence were being disassembled for rework, samples were collected from each of the four cavity pairs and the modules' ion pump manifold. Having no prior experience, we chose to take maximum use of the opportunity and collected a total of 293 samples from Franklin and 185 from Independence. Both of these cryomodules have the original 5-cell cavity pairs, waveguide higher-order-mode couplers, and many indium seal joints. There were approximately 60 standardsampling locations per cavity pair. See Fig.4.

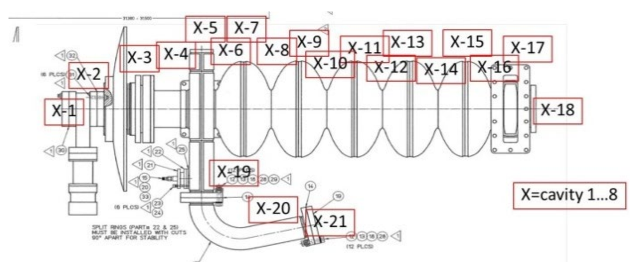

Figure 4: Typical sampling locations.

Material character of the particulates found inside the cavities corresponded well with the large particulates found in the girders. This is consistent with the chief issue of cryomodule performance degradation being one of the transport of pathological contamination from the girders into the SRF cavities.

The metallic particulate load was ~3X higher in Franklin than in Independence. Mineral and masonry type particulates were a factor of 10 higher in Franklin than Independence. Interestingly, hydrocarbon signature particulates were most prevalent in Independence. Over 100 polymer or elastomer particulates were found in Franklin and adjoining girders, but essentially none in Independence.

While fabrication standards have evolved since the original CEBAF construction, the large particulates found well exceed any intended standard and are consistent with introduction after installation. While performance of the SRF cavities can be limited by micron-scale particulates, analytical pursuit of such scale particles was not motivated due to the presence of so many much larger particulates.

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C100-6

One C100-style cryomodule has been removed from CEBAF, disassembled, the cavities re-rinsed with ultra-pure high pressure water in the new JLab facilities, and then reassembled. The cryomodule, C100-6 was removed from the CEBAF zone NL23 due to degradation following a beamline vacuum incident, after which, field emission loading and the radiation production was excessive. This module is due for reinstallation in CEBAF in the Fall of 2019.

C100-6 NL23 was bled up in the tunnelfrom girder 24 to girder 23 then brought to Test Lab with end CF flanges not hermetically sealed. Because of handling issues, the pump drops were not sampled. Due to the ongoing LCLS-II construction work, sampling occurred in a portable cleanroom, not in an ISO 4 cleanroom as with previous work. Also, since these cavities were only going to be re-rinsed then reassembled, no samples were collected from inner iris regions of the cavities, only from the easily accessible beampipes.

A total of 68 spindle samples were collected and all were analyzed. Samples were collected from each cavity as each was sequentially disassembled from the string. Three samples were typically collected from each end of each cavity and an additional sample was collected from the waveguide fundamental power coupler as illustrated in Fig. 5. Four samples were collected from the beamline endcaps. Eight witness samples were collected to monitor the environment in which sampling occurred. These samples were analyzed using the upgraded fully automatic system that finds and characterizes the elemental composition of all particles greater than about 4 µm dimension [1].

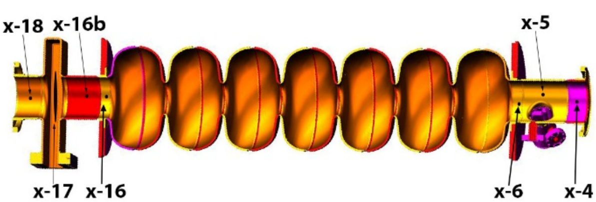

Figure 5: Sampling locations on cavities from C100-6.

The automated scans found and identified an average of 84 to 587 particulates greater than 4 µm per spindle from the 58 cavity samples. For comparison, the endcap samples which had been open to the environment for an extended time were found to have an average of 5366 particulates. Detected particles ranged in size from 4–480 µm, with 77% of the detected particles in the 4–10 µm range.

The new automated analysis records the elemental composition of particulates found. Since particulates are often aggregates, as described in [1] we look for patterns of occurrence of key elements as our first slice through the data. The vast majority of the particulates on beampipe samples showed presence of silver, steel, aluminum or copper. For some reason, cavity 2 samples showed more Al that typical. Also, rather surprisingly, cavity 6 (of eight) showed 25–580% more particulates than the other cavities, with a wider variety of materials represented as well (including an assortment of minerals), suggesting a very general contamination exposure event sometime during its preparation for assembly circa 2011. Only the known-compromised endcap samples showed the same variety of contaminating materials.

Another curious finding was that particulate composition was different on samples collected from the fundamental power coupler (location #17) than any other location, and this was roughly true for all eight cavities. For example, numerous hydrocarbon fibers with length 0.2–2.0 mm were collected from the waveguides. This suggests that perhaps at assembly, the copper-plated stainless steel waveguide sections with bellows and integrated ceramic rf window were not cleaned to the same standards as the SRF cavities. If this is true, then the data also suggests that this built-in contamination did not propagate up into the beamline. (Note that from assembly, through operation, and disassembly, the cavities are always oriented with the plane of the FPC waveguide flange being vertical.)

Sample SEM micrographs of particulates collected from C100-6 are presented in Fig. 6.

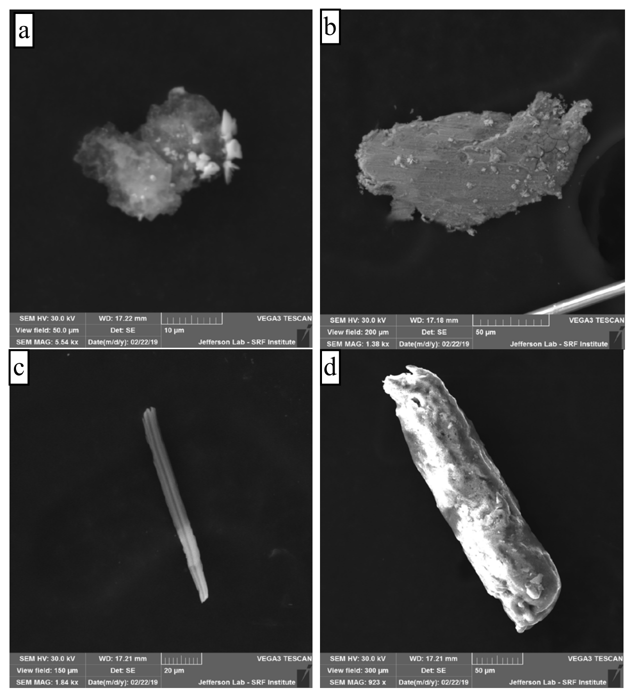

Figure 6: Particulates collected from C100-6 beamline, a) silver, b) copper, c) steel, and d) titanium.

Process Development

As reported in [1], these standard spindle samples were used to investigate particulates associated with LCLS-II copper-plated bellows flexing, cleaning, and assembly. See Fig. 7. This work provided valuable feedback concerning the effectiveness of cleaning procedures and the sustained integrity of the copper plating with bellows flexure.

We look forward to engaging structured procedure development/refinement efforts using our new standardized and automated system.

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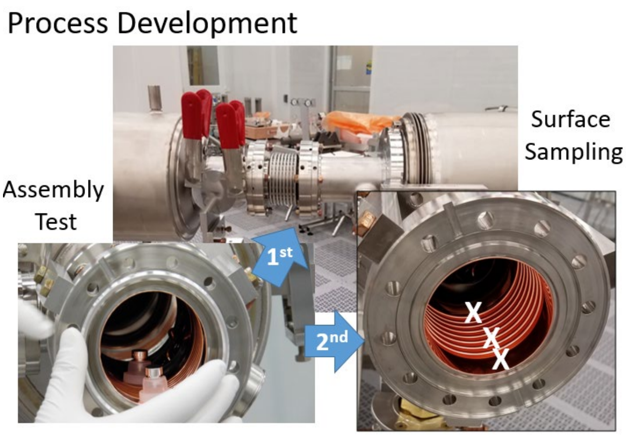


Figure 7: Particulate assessment of LCLS-II bellows assembly.

CHARACTERIZATION OF KNOWN SOURCES

We recently began using this particulate assessment system to identify and characterize particulates with well-defined sources. The intent is to eventually bridge the gap between sources and on-process contamination, so that source attribution and migration routes become increasingly clear, enabling effective remediation and continuous improvement.

One simple exercise was to simply characterize the particulates emitted by the anti-static N_2 blow-off guns used routinely in the JLab cleanroom. A 1-minute, direct flow exposure on the carbon-tape capped sample, followed by five 1 sec bursts yielded on analysis 12 particulates 7–20 µm all having the same EDS spectrum dominated by silicon, with traces of Na, Mg, Al, K, Ca, Ti & Fe (Fig. 8). While investigation continues, we suspect the particulates may represent breakdown fragments of the point-of-use filter on the N_2 gun.

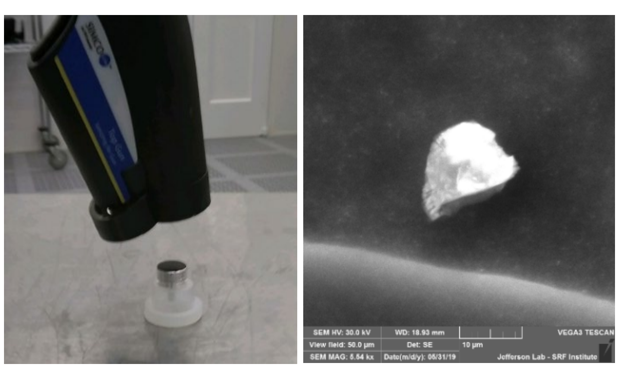
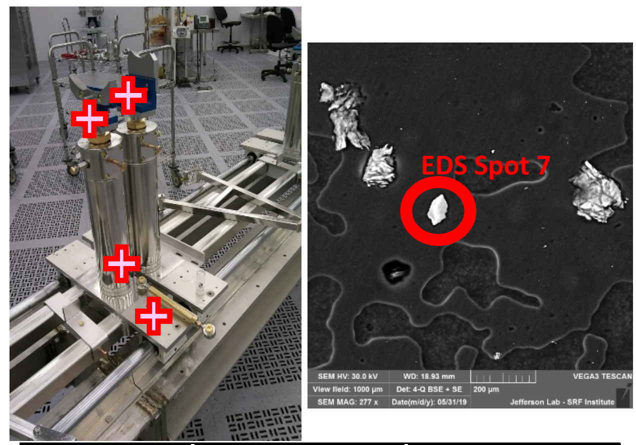


Figure 8: Particulate from N_2 gun test.

In another exercise, a white 5 mil Class 10 clean room glove was donned in the entry room. The glove was used to enter the door into the cleanroom and then the surface analytical area. Each finger of the glove was then applied to the exposed GSR carbon tape. On analysis, 28 particulates were found. The majority of the particulates were identified as mineral – silicate or calcite.

The analysis system can also be used for quality control checks. As an initial trial, one spindle was used to collect contact samples from the cryomodules string assembly rail used for LCLS-II cavity strings. The rail was approached in the “cleaned and prepared for string assembly” condition. The carbon tape sample was touched to the base, pedestal, and top mounting hardware as indicated in Fig. 9. 16 different spots were selected for characterization on the resulting spindle. The following materials were found: polymer or elastomer, copper, aluminum, silicate, calcite, copper phosphate, copper zinc, iron, aluminum sulfate, and an organic.



EDS Area	Category	Type
EDS Spot 1	Polymer or Elastomer	Polymer or Elastomer
EDS Spot 2	Salt	Copper Phosphate
EDS Spot 3	Metallic	Copper
EDS Spot 4	Polymer or Elastomer	Polymer or Elastomer
EDS Spot 5	Mineral	Calcite
EDS Spot 6	Polymer or Elastomer	Polymer or Elastomer
EDS Spot 7	Polymer or Elastomer	Polymer or Elastomer
EDS Spot 8	Metallic	Aluminum
EDS Spot 9	Salt	Steel Flouride
EDS Spot 10	Mineral	Silicate

Figure 9: Particulate sampling from cavity string assembly rail.

We find that with each use of this analysis system we promptly find unexpected results. These are all new clues for building the understanding which will enable reliable engineering of processes that ensure that SRF cavities are created, delivered, and maintained particulate free for accelerator operations.

CONCLUSION

The primary finding of the investigation of particulates found in CEBAF is that particulate sizes are much larger than anticipated. While past efforts to control particulate contamination considered the battle to be control of particulates smaller than 5–10 µm, in reality, beamline elements, including cavities in cryomodules, were found to contain a generous number of much larger particulates. Some of these appear to have obvious sources, others do not.

Feedback from this work has motivated changes in CEBAF vacuum pumping systems, cryomodule assembly procedures, and beamline maintenance work procedures.

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We intend to routinely sample cryomodules and other linac beamline components for particulate contamination, continuing to build a picture of issues, sources, and trends.

We anticipate using the same standardized particulate assessment system for ongoing feedback to active process refinement for cryomodule production and maintenance procedures.

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