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STANDARDIZED BEAMLINE PARTICULATE CHARACTERIZATION ANALYSIS: INITIAL APPLICATION TO CEBAF AND LCLS-II CRYOMODULE COMPONENTS*

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Abstract

Despite continuously evolving efforts to minimize and manage the occurrence of particulates in operational SRF accelerator systems, the presence of electron field emission from contaminating particulates on SRF surfaces with high surface electric fields remains a consistent challenge. Jefferson Lab has recently initiated a standardized particulate sampling and characterization practice in order to gain more specific and systematic knowledge of the particulates actually present. It is expected that patterns emerging from such sampling will strengthen source attribution and guide improvement efforts. Initial samples were gathered from a cryomodule and adjoining warm girders that were removed from the CEBAF tunnel for reprocessing. The collection and analysis techniques were also used to characterize particulates on the inside of LCLS-II string components. Samples are transferred to clean industry-standard forensic GSR carbon tape spindles and examined via automated cleanroom SEM scanning for particle localization trends and sizing. The particulates are subsequently analyzed with EDS for elemental composition. A catalogue of found particle types is being accumulated. The methods used and results obtained from these initial applications will be presented.

INTRODUCTION

That extrinsic particulates on the surface of high electric field regions of superconducting rf (SRF) cavities present performance-limiting effects via electron field emission has been very well established. Significant efforts are made to obtain clean "particulate-free" interior cavity surfaces. Effective cleaning processes have been developed by the community, largly using ultrasonic detergents and high pressure rinsing with ultra-pure water. See, for example references [1-5]. In addition, careful attention has been applied to development and control of assembly and handling techniques which attempt to minimize contamination of clean surfaces.[6-8]

Success with this contamination control is indicated by the absence of field-emission-induced x-radiation during high-field operation of the SRF cavities. Higher field applications require higher standards of maintained cleanliness to assure success. Field-emission free multi-cell cavity performance above 40 MV/m has been demonstrated at multiple labs. The very non-linear exponential field emission current response has been successfully modeled by Fowler-Nordeim theory.

Practical reality to date, however, is that all SRF accelerators exhibit more x-ray production and attendant extra heat load attributed to particulate-induced electron field emission than desired. Geometries that allow acceleration of emitted electrons significantly above 10 MeV also encounter activation and material degradation due to neutron production.[9]

Accelerator cleanliness standards have clearly evolved over the past 30 years. An effort has recently begun at Jefferson Lab to increase the systematic characterization of particulates on the CEBAF beamline, both in the older sections constructed in the early 1990's and in more recent assemblies. An important preliminary investigation on a cryomodule from the JLab FEL identified significant contamination.[10] We seek now to implement a standardized particulate collection and characterization protocol in order to provide better feedback to all potential contributors to the problem.

SAMPLE COLLECTION METHOD

We take as a working assumption that the subject particulate contamination has arrived at the surface after the sampled beamline surface was previously clean and free of such contaminants. So, the particulates are expected to be at least initially non-adhered to the surface. For the collection of each sample, we employ a section of fresh cleanroom wipe rated for ISO-4 use, wetted with reagent isopropyl alcohol. Approximately 2 cm^2 of the wipe is rubbed gently over the surface to be sampled. All other contact with the collecting wipe is avoided. The collection area of the wipe is then pressed against a freshly exposed clean carbon tape, with intent to transfer and adhere collected particulates to the carbon tape.

We take advantage of the commercial availability of standardized and serialized forensic gunshot residue (GSR) analysis spindles. These are guaranteed clean. Each spindle is topped by carbon tape. A standard holder accepts an array of these GSR spindles for examination under scanning electron microscope (SEM). At Jefferson Lab, we collect particulate samples onto GSR spindles under ISO-5 or better conditions, then transport the samples to our Tescan VEGA3 XMH SEM, located in the JLab cleanroom suite, for analysis. (See Fig. 1.)

SAMPLE ANALYSIS METHOD

In order to establish a sustainable, systematic, and rather high-volume analysis of samples collected from multiple sources, we aim to automate as much of the analytical work as possible. Once an array of spindles is

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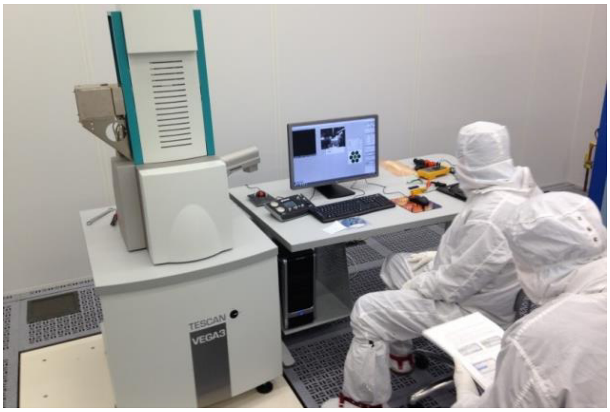

Figure 1: Tescan Vega3 SEM used for particle analysis.

loaded into the SEM, an initial scanning routine sequences through the spindles and for each one accumulates a macro SEM image of the carbon tape, assembled via stitching of multiple images each acquired with a 3 mm field of view. This initial scanning now runs unattended and requires 1.5 hrs per sample spindles. The resultant browsable images provide quick feedback and easy reference for any captured particulates larger than about 25 µm.

A second automated routine increases the contrast against the carbon tape background in the acquired SEM image and is able to locate and size particulates, generating a table of location coordinates. Energy dispersive x-ray analysis (EDS) is then used to obtain elemental composition information of individual particulates. EDS data is collected in TEAM with an EDAX Apollo X air cooled silicon drift EDS detector. Typical scans have a deconvolved and fitted background under 1000 counts and detected major x-ray peaks above 10^5 counts. To date, all EDS data have been collected manually. OEM software has recently been acquired which should enable automated EDS characterization at coordinate locations handed-off by the SEM particle ID software and consolidate the back end analysis into the acquisition process.

For the spindles analysed so far, EDS characterization has been completed for all particles larger than 1 mm, and regions were sampled where particles were evident from the macroscans. If no particles are observed at low magnification, five or more separate local regions are selected for examination at magnification higher than 500X. Standard reports in pptx format are generated for each EDS acquisition. The pace of manual EDS analysis is ~1 spindle sample per day, allowing at most 4/week with other SEM usage.

Surveys of the initial samples have allowed the creation of a catalog of types of particulates found. Programs have been written which mine data from the pptx files and import them into a common database structured by the types of particles found. In this way we anticipate being able to handle an increasing number of samples with higher efficiency.

INITIAL SYSTEMS SAMPLED

CEBAF Beamline Girders

During the servicing of diagnostic instrumentation on the CEBAF beamline in 2015-2016, the cryomodule in north linac zone 12, known as "Franklin," 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. While there had been no previous assessment, it was suspected that particulate load of some sort had been transported from one of the adjoining warm beamline girders into the SRF cavities. The decision was taken to remove and re-clean both girders that bracketed Franklin during the time that the cryomodule was swapped out with a freshly reworked one.

In addition, we took the opportunity to obtain particulate samples from the inside of these girders prior to their re-processing. These girders have been on the CEBAF beamline since initial construction in 1992. The girders were isolated and sealed under local clean conditions in the tunnel, transported to the Test Lab, exterior magnets and instrumentation removed, exterior cleaned, and then brought into the cleanroom for sequential disassembly and interior surface sampling for particulates. (See Fig. 2.)

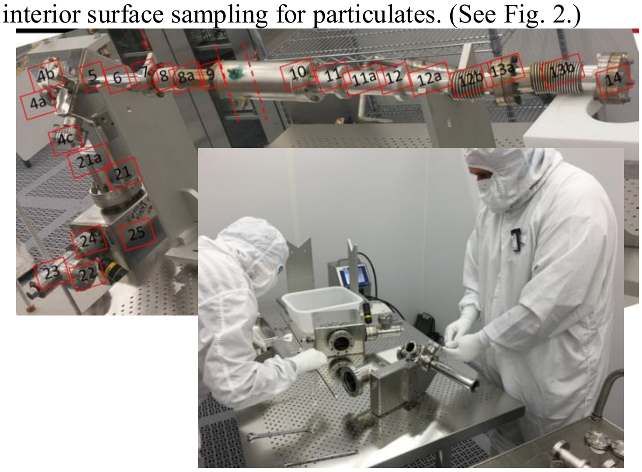

Figure 2: CEBAF beamline girder during particulate sampling in cleanroom.

Contaminated Cryomodule

As the cryomodule Franklin was being disassembled for rework, samples were collected systematically from each of the four cavity pairs and the module's ion pump manifold. (See Fig. 3.). Having no prior experience, we chose to take maximum use of the opportunity and collected a total of 293 samples. A subset of the sampling locations is illustrated in Fig. 4.

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Figure 3: Sample collection during Franklin disassembly.

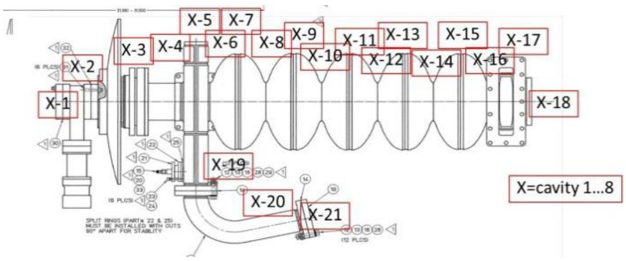

Figure 4: Particle sampling location key for each cavity removed from Franklin.

LCLS-II Beamline Bellows

During assembly of the cavity string for JLab's LCLS-II cryomodule CM3, one of the beamline copper-plated bellows was unrestrained during evacuation for leak checking, collapsing longitudinally under the external atmospheric load. The decision was taken to vent the string and remove, inspect, re-clean, and reassemble the affected components. We took the opportunity to collect seven samples from inside of and near the collapsed bellows and, for comparison, a nearby properly-constrained bellows during the disassembly. (See locations in Fig. 5.) We were interested to look for any evidence of particulate-generating abrasion and also to obtain a baseline response from our standardized characterization protocol in this very different system.

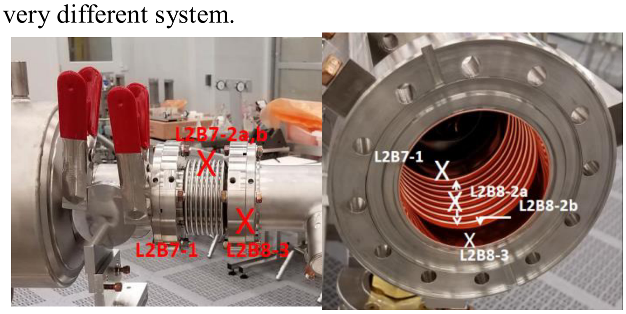

Figure 5: LCLS-II bellows sampling locations.

RESULTS AND DISCUSSION

To date, 69 GSR spindles have been scanned and over 1400 particles have been characterized. All of the particulate characterization is proceeding in the same way. A common catalog of 39 types of particles found has been assembled. This then supports differential comparison of occurrence of such types from different circumstances. While it is yet too early to interpret the observation frequency of different particulate types in a strict quantitative density sense and analysis of the full set of samples remains yet for the future, we are able to make some preliminary observations.

Very clearly, the particulate load in the sampled girders from CEBAF is far above any current standard for SRF accelerator beamlines. Hundreds of copper and stainless steel particulates larger than 40 µm were found on the collected samples. Distinct particles of aluminium, silver, titanium, zinc, nickel, and minerals were also common. Particulates of soil and clay were found rather frequently. Non-distinct polymers were also observed, some of which may have been associated with the collection media.

Essentially all of the materials found in the girders have also been found on the beamline elements in Franklin, although with apparent reduce frequency. There is a suggestion of higher density on the downstream side of the module, closer to girder 13 (from whence the suspected contamination breeze blew), but this merits further confirmation. Figure 6 illustrates a large steel particle found in the niobium beamtube central to the third cavity pair in the cryomodule. One is left to speculate how it came to arrive there.

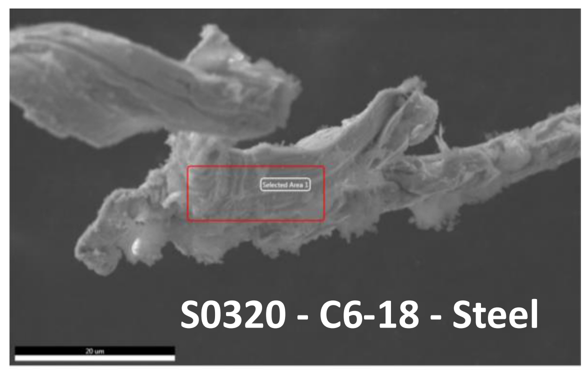

Figure 6: Steel particle retrieved from the beam tube between cavities 5 and 6 in Franklin, ~20 × 100 µm.

Examination of samples from the LCLS-II copper plated bellows found no evidence for abrasion-induced shedding. More steel and copper loose particulates were found on the sample from small radius surfaces on the un-flexed bellows L2B8 than the compressed L2B7. The size of the particulates found on the LCLS-II bellows was considerably smaller than was typical in the other sample sets.

SUMMARY AND FUTURE WORK

Application of the systematic particulate characterization method described here has only just begun. While some of the circumstances examined so far are rather pathological, we seek next to establish meaningful baselines for particulate characterization of surfaces produced by present typical production processes and then to provide targeted feedback toward identifying and mitigating remaining uncontrolled sources whether arising during assembly, installation, or accelerator operations.

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REFERENCES

[1] P. Kneisel and B. Lewis, "Advanced Surface Cleaning Methods - Three Years of Experience with High Pressure Ultrapure Water Rinsing of Superconducting Cavities," in 7th International Workshop on RF Superconductivity (JACoW, Gif-sur-Yvette, France, 1995), p. 311. http://accelconf.web.cern.ch/AccelConf/SRF95/papers/srf95l02.pdf.

[2] C. E. Reece, E. Ciancio, K. A. Keyes, and D. Yang, "A Study of the Effectiveness of Particulate Cleaning Protocols on Intentionally Contaminated Niobium Surfaces," in 14th International Conference on RF Superconductivity (JACoW, Berlin, Germany, 2009), p. 746. http://accelconf.web.cern.ch/AccelConf/SRF2009/papers/thppo062.pdf.

[3] R. L. Geng and A. C. Crawford, "Standard Procedures of ILC High Gradient Cavity Processing at Jefferson Lab," in 15th International Conference on RF Superconductivity (Chicago, IL USA, 2011), p. 391. http://accelconf.web.cern.ch/AccelConf/SRF2011/papers/tupo015.pdf.

[4] A. Yamamoto, et al., "Advances in Superconducting RF Technology for the ILC," Applied Superconductivity, IEEE Transactions on 23, 3500706 (2013)

[5] I. M. Malloch, C. Compton, and L. Popielarski, "Study on particulate retention on polished niobium surfaces after BCP etching," in Particle Accelerator Conference 2013 (JACoW, Pasadena, CA, 2013), p. 823. http://accelconf.web.cern.ch/AccelConf/PAC2013/papers/wepac17.pdf.

[6] S. Berry and O. Napoly, "Assembly of XFEL Cryomodules: Lessons and Results," in LINAC 2016 (JACoW, East Lansing, Michigan, 2016), p. 646. http://accelconf.web.cern.ch/AccelConf/linac2016/papers/we1a02.pdf.

[7] L. Zhao and A. V. Reilly, "Particulate study on materials for cleanroom assembly of SRF cavities," in LINAC 2016 (JACoW, East Lansing, Michigan, 2016), p. 512. http://accelconf.web.cern.ch/AccelConf/linac2016/papers/tuplr022.pdf.

[8] S. Berry, et al., "Clean Room Integration of the European XFEL Cavity Strings," in International Particle Accelerator Conference 2014 (JACoW, Dresden, Germany, 2014), p. 2474. http://accelconf.web.cern.ch/AccelConf/IPAC2014/papers/wepri001.pdf.

[9] C. Hovater, et al., "Operation of the CEBAF 100 MV Cryomodules," in LINAC 2016 (JACoW, East Lansing, Michigan, 2016), p. 65. http://accelconf.web.cern.ch/AccelConf/linac2016/papers/moop11.pdf.

[10] R. L. Geng, J. F. Fischer, E. A. McEwen, and O. Trofimova, "Nature and Implication of Found Actual Particulates on the Inner Surface of Cavities in a Full-Scale Cryomodule Previously Operated With Beams," in SRF2015 (Whistler, BC, Canada, 2015), p. 164. http://accelconf.web.cern.ch/AccelConf/SRF2015/papers/mopb035.pdf.