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AUTOMATION OF PARTICULATE CHARACTERIZATION*

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Abstract

Foreign particulates residing on high electric field surfaces of accelerator cavities present sources for field emission of electrons that limit the useful dynamic range of that cavity. Developing the methods and tools for collecting and characterizing particulates found in an accelerator enables process development towards creating and maintaining field emission free SRF cavities. Methods are presented for sampling assemblies, components, processes, and environmental conditions utilizing forensic techniques with specialized tooling. Sampling activities to date have produced an inventory of over 850 samples. Traditional SEM + EDS analysis of this volume of spindles is challenged by labor investment, spindle sampling methods, and the subsequent data pipeline which ultimately results in a statically inadequate dataset for any particulate distribution characterization. A complete systematic analysis of the spindles is enabled by third party software controlling SEM automation for EDS data acquisition. Details of spindle creation, collection equipment, component sampling, automating particle assessment, and data analysis used to characterize samples from beamline elements in CEBAF are presented.

INTRODUCTION

Functional operation of SRF cavities requires a clean RF surface [1]. Any material on the high electric field surfaces of the cavity has the potential to become an electron field emission source. Studies have been conducted to determine what types of materials may emit due to high electric field exposure [2]. It is understood that the size and shape of the particulate is generally of highest consequence [3].

In an effort to gain useful specific knowledge regarding the contaminating particulates found in CEBAF beamline assemblies, including accelerator cryomodules, we have developed an efficient and systematic routine to collect and analyze such contamination. To date, this system has primarily been used to characterize components after removal from the CEBAF beamline [4]. Such contaminants are largely a legacy issue from which we continue to learn. We have also begun to use the system to characterize current particulate sources that present challenges in the JLab cavity and cryomodule production processes. Our intent is to develop this system into a quality assurance and continuous improvement tool – identifying particulate sources early so that effective targeted controls may be implemented.

We want to do more than simply count and size particulates present. We want to characterize them sufficiently to have some idea as to their source, and also perhaps their migration path from generation to the location found forensically. The task then becomes one of accumulating familiarity with the sources for the types of materials actually represented in the particulates found and, as possible, association with candidate sources from which controlled representative particulates have been collected.

To have any hope of succeeding, we recognized that automation of characterization and data processing is essential. Only a system capable of producing reliable statistics on 10's of samples per week analysing 100's of particulates per sample will suffice. Producing valid statistics requires a reliable collection process with negligible, or easily distinguishable, features from the sampling population and sufficient observations to establish quantitative differentiation.

The elements of an automated scanning electron microscope (SEM) particulate identification system for multi-sample analysis with elemental characterization utilizing electron-dispersive X-ray spectroscopy (EDS) are presented. Presently, the resulting database is being incorporated into JLab's Pansophy system [5].

METHODS AND MATERIALS

Standard Sample Collection

Application of forensic sampling techniques to enable process development for field emission free cavities is built on the use of standard commercially-available gunshot residue (GSR) forensic spindles and an SEM. Creation of collection spindles, handling, and automated analysis were all performed in a clean environment with cleanroom techniques. Several types of spindles were created: witnesses, controls, component sampling, and process evaluations. Witness samples were created alongside collection spindles to provide background measurements of airborne environments.

Controls were of two types: process and library. Process controls created a collection spindle by directly sampling some activity or environmental contributor other than airborne particulate. Library controls were intended as material references for capturing the contributions from a known process component. If the component could fit in the SEM, like a stainless steel bolt or bellow, then direct SEM examination characterized the bulk EDS spectra. Intentional particle generation with a flat chisel scribe produced particulate characteristic of the components' material type. Contact library controls sampled the surface of components with carbon tape.

There are several methods that could be used to sample a sensitive component for particulates. The simplest method is to directly sample the parts with carbon tape [6]. Direct sampling has a limited surface area and may leave a

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residue on the sampled component but likely has excellent particulate retention if the sampled surface is flat enough and free of any adhesive wetting agents. More elaborate methods of flow assays allow for collection from the entire surface area of the part but are challenged mainly by process development: reliable background measurements, implementation, and efficacy. A compromise between the two sampling methods is to fabricate a component sampling wand with a non-interacting swab. The component sampling wand we used was fabricated from SS tube and VCR fittings to capture an isopropyl pre-wetted ITW Alpha Wipe as the sampling surface. The synthetic wipes are a non-abrasive knap that has adequate texture and surface area to accommodate particle retention up to transfer onto the carbon tape. The swabbing wand collection surface area is an integrating medium for consolidating large components' surface area into a single collection spindle. The GSR collection spindles are standard SEM 12 mm aluminium stubs topped with carbon tape prepared by the vender under clean conditions that are sequentially stamped with numbers on the side.. Each spindle is retained on the inside of a glass vial's plastic top. The examination surface of the collection spindle is provided with a plastic cover slip that is removed just prior to creating a collection sample. Control samples of the collection spindles as received did not identify any particulates, validating the product as an ideal sampling vehicle. Particulates were collected from components with the swabbing wand or by shaking the components over a transfer wipe and then transferring the collected particulates to a collection spindle. (See Fig. 1.)

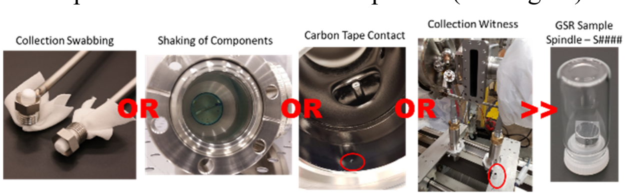

Figure 1: Sampling methods for spindle creation.

The selection of assemblies installed in CEBAF for sampling was determined by machine performance. Identified problematic assemblies were removed from CEBAF service for reprocessing. Some of the assemblies removed have been in service since the 1990's and were prepared with processes known to be inferior to the current standard operating procedures developed, for example, for the LCLS-II project.

Opportunistically, the assemblies were dissected while creating particulate samples before reprocessing. The assemblies were externally cleaned and transferred to the cleanroom for particulate sampling. Setup for collecting particulate samples occurred at least 12 hours before sampling in an effort to reduce ambient contributions. Tools were cleaned, blown to zero counts, and then the area was vacated to allow recovery of the environment before disassembly or component sampling. Particulates were collected from the assembled components with the swabbing wand first and then from disassembled components collecting any observable debris independently on a unique collection spindle. If a suspicious region of oxide, stain, or surface residue was identified on a component then a collection spindle was independently created isolating the distinguishable feature from the other particulates collected on the given component. Once the assembly had been completely disassembled and sampled for particulates, the individual components were cleaned and then reassembled with the current standards. The reprocessed assembly was then reinstalled into CEBAF service.

SEM Standard Parameters

A Tescan VEGA XMH3 scanning electron microscope (SEM) with a LaB_6 filament and equipped with an EDAX EDS detector was used for particulate analysis. This SEM is located integral to the JLab cleanroom suite. Standard procedure upon loading collection spindles was to collect a low mag image of the whole spindle, 12 mm field of view, and panoramas upon first examination and any auditable event. A specific instance of an auditable event occurred with the first set of spindles processed in 2016 revealing that standard SEM venting compromised carbon tape particulate retention, especially for large particulates.

Configuration of the beam for particle analysis requires a balance in resolution and beam density. Obtaining high resolution SEM images builds an inventory of morphological characteristics for the collected particulates. Classifying particulates types with EDS requires sufficient beam power in the EDS volume of the microscope. Typical SEM LaB_6 beam configurations are presented in Table 1.

Table 1: Beam Parameters for 16–17 mm Working Distance

Acc Voltage (kV)	Beam Intensity	Spot Size (nm)	Probe Current (pA)	CPS on Carbon	CPS on Metal
20	15	200	30–50	0.5–5k	1–10k
30	15	150	50–150	5–20k	50–100k

Particulate Identification

Automatic identification of particulates with EDAX Genesis Particle Software via grey scale thresholding was conducted on 242 spindles. Particulates are identified in the image by selecting a region of the pixel grey scale histogram combined with dimension constraints. Only minimum and maximum particle sizes were implemented as dimension constraints. The detectable particle size is dictated by the image field of view and pixel resolution. In Team, particulates were selected by the SEM operator, primarily with SE imaging, focusing on the largest particulates for EDS analysis.

Identifying particulates with grey scale thresholding was explored with SE, BSE, and SE + BSE detectors. Methods were developed to accommodate particle identification for each imaging mode. A combination of SE and BSE detectors was found to produce the best contrast and provide the most dynamic range for isolating different particle types from the carbon tape features. A single phase was implemented for balanced to dark field images acquired with SE + BSE detectors mixed in a 25/75 to 60/40 ratio. Metallic

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particulates were readily selected, though some of the polymeric/elastomeric and hydrocarbon-based materials were not contiguously identified with a single phase. A more elaborate 4 phase scheme was developed for balanced to bright field SE only images. The increased number of phases allowed for selection of bright and dark particulates, distinguishing from carbon tape features and isolating particulates of all material types.

Carbon tape features often have contrast similar to some types of particulates. Carbon tape features are blocked by a combination of image detector settings and by a pre-scan pass filter on spectra. The automated particulate software rejects particulate that quantify as only carbon and oxygen with a carbon signal above 96% atomic percentage. Other pre-scan features allow blocking particulate types with low x-ray yields (CPS) and insufficient total counts in a specified energy range (0.1–6 keV).

The volume of features identified became excessive for some heavily burden collection spindles even when implementing pre-scan filters. For a pre-scan of 2 seconds, approximately 43,200 particulates could be screened in a day, and with a spectra collection live time of 20 seconds, ~ 4,320 particulates can be quantified for material typing per day.

SYSTEM PERFORMANCE

A system has been developed to support an on-going particulate analysis program for forensics analysis of assemblies, monitoring environments, and particulate centric process development activities. The system utilizes commercially available components with minimal custom tooling. Creating collection spindles is a time consuming process due to meticulous process controls necessary to eliminate cross contamination from environmental and sampling sources. Setup time and component disassembly are main drivers for the total time commitment for generating collection samples. The sampling environment and assembly design can contribute significantly to the time allocation. Generally 30–50 collection spindles could be created in an 8 hour shift, average direct labor per collection spindle creation ~ 10–15 minutes including setup and clean up.

The Tescan SEM has a standard GSR stage available that was purchased for the particulate analysis program. The Tescan GSR stage can carry up to 52 spindles per pump-down cycle. It is standard procedure to register the collection spindles' rotation with the serial number centered on the spindles' set screw. Stage loading time is proportional to the number of collection spindles to be characterized, ~2–3 minutes per collection spindle, plus some allotment for routine venting and pumping cycles on the SEM. A chamber pump down is generally less than 5 minutes to a cross over pressure of 5×10^{-3} Pas. Venting with particulate samples in the chamber takes 15–20 minutes to let the turbo pump spin down naturally, no gas breaking.

Characterization of the collection spindles was proceeded by programming the Tescan for panorama's and the Genesis software for automated particle characterization. Low mag inspection images were manually collected during the programming of the stage. The entire direct labor invested in each spindle during automated SEM characterization generally is 5–7 minutes. On several occasions, 20–30 spindles were loaded, inspected, and both software programs configured in 4–5 hours. The tool time associated with the imaging automation was fixed, 3–5 minutes per low magnification image and 30–50 minutes per panorama (depending on image quality). The panoramas generated by the Tescan VEGA software could be passively collected in several hours. Automated spectra collection time was proportional to the number of potential particulates identified. Referring to the simple grading method of red, yellow, and green labelling; red collection spindles must have a max particulate cut-off or have morphological filters enabled to prevent excessive particulate counts. Some heavily burden spindles took more than 48 hours to acquire spectra on more than 10,000 particulates in the specified size range. For a run of 22 process evaluation spindles, 3 with a red disposition (limited to a maximum of 1500 particulates per spindle) and the remainder split between yellow and green dispositions, the average time per spindle was ~ 2 hours, producing data for a total of 9426 particulates in ~ 40 hours.

Initially during development of the automation software, collection spindles were analyzed manually with EDAX Team. Each spindle would have at most 20–30 particulates selected by the operator starting with the largest particulates. Elemental spectra were collected in TEAM with a 20 second live time. Data was processed in TEAM and exported by EDS location to PowerPoint files. On average, 3 spindles were completed in an 8 hour shift. A Visual Basic program was written to mine the spectra settings, elemental channel data, and then the program created a separate PowerPoint file to group all the particle images and spectra analyzed in a batch. The elemental channel data was reviewed with accompanying particle images and spectra to dimension particulates and assign a material category and type. Processing the data from PowerPoint files to CSV import files typically took ~1.5 hours per spindle. An Access database was created to compile the collection spindle creation records, spectra settings, elemental channel data, & particle dimensions with material category typing data. The process is illustrated in Fig. 2.

Reporting data was focused around labelling particulates and generating statistics for dimensions, distributions, and occurrences. This method of reporting is thorough and time consuming. Compilation of the manual data identified prevalent types of particulates, and relative occurrences in the types of particulates, establishing the elemental palette for the automated software.

The automated software output consisted of a table of particle ID, relative stage coordinates, dimension data, elemental channel data for 26 elements, and Genesis material assignment. The automated data generated was combined with the manually collected data in Access until a standard desktop computer's resources were exceeded. Another Visual Basic program was written to homogenize the reporting of identified particulates' category and typing between the manually assessed and automatically labelled particulates. Development of an Oracle database with Pansophy query

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interface is underway to create a completely automated solution from acquisition to reporting and datamining. The automated data flow is presented in Fig. 3.

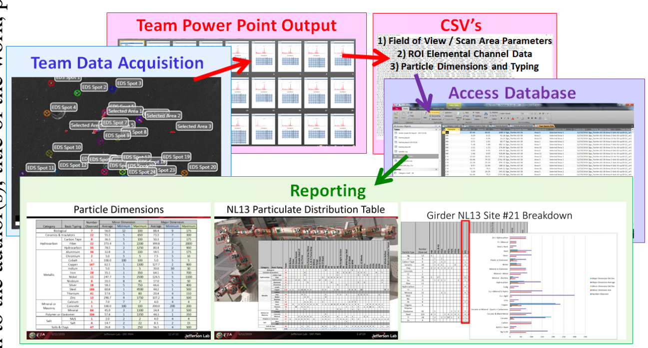


Figure 2: Manual particulate characterization data flow.

The automated data is compiled with spindle attributes enabling correlation of collection, component, location, and assembly data with accumulated particulate statistics.

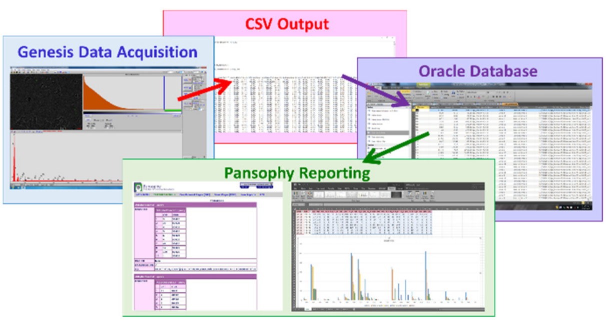


Figure 3: Automated characterization of particulates data flow.

Particulate Reporting

A case example is presented for a set of collection spindles created from a VTA test stand issue. There was a problem with a pump cart that caused a vacuum failure to an actively pumping cavity on a test stand. During disassembly of the vacuum line it was found that there was a residue of dust and particulate. The suspicious area was wiped with an ITW Alpha wipe producing a collection of the particulate for anaylsis. The wipe was transferred to surface analytical portion of the cleanroom in a heat sealed bag where it was removed and sampled with collection spindles. Figure 4 demonstrates reporting data from one area scanned manually on one of the two collection spindles created from the vacuum line particulate.

On the left is the low magnification inspection SE image of the whole collection spindle as viewed in the SEM. On the right in Fig. 4 there are several related SE images of decreasing field of view, the lowest magnification is the upper right image, the middle magnification is the lower left image on the right, and the bottom right image is the

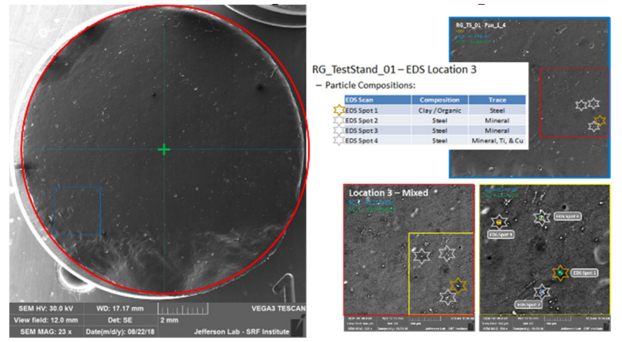
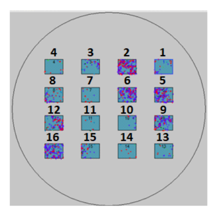


Figure 4: Example of reporting data from 1 EDS area for a manual collection.

field of view that acquired the EDS spectra of the particulate identified by color coded stars. There are three grey stars surrounding the steel particulates and a gold star surrounding the clay particulate. The only valid statistical statements that can be readily made from manually collected data are the observed frequencies which only become strong statements with sufficient sampling, increased direct labor. For a similar amount of direct labor the automated system can produce a particulate typing report similar to Fig. 5.



Class Rank	Class Description	Count	Average Diameter (um)
2	Iron	44	9.12
15	Calcium Carbonate	1	2.57
22	Carbon Tape	60	10.54
23	Pass Filter	134	0
25	SiO	1	3.64
27	Clay 2	19	13.7
29	ZrO + C	526	5.87
30	Calcite + Fe P K Cl S	5	8.36
32	Steel	50	12.82
34	Steel + C	987	3.74
36	Calcite + AlMgSiO	1	3.45
NA	Unclassified	0	0

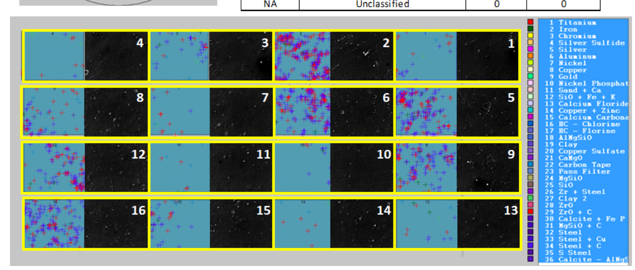


Figure 5: Example of reporting data for particle typing.

In Fig. 5 on the upper left is a graphical representation of the spindle and sampled fields of view. The fields of view are also presented at the bottom of the figure placed next to the corresponding SE image. A legend on the bottom right provides the association of the symbol color with specific particulate type. The automated typing dataset elucidates strong statements about particulate distribution on the spindle, observed frequency, & size distribution.

Although faster and easier to execute, the automated data tends to supress the cross contamination contributions. One might be tempted to expand the category and typing scheme to capture Steel + Cu, Steel + Ag, Steel + Cl, & any other permutation of steel with trace element(s). An EDS map of steel particle with various type of trace contamination is presented in Fig. 6.

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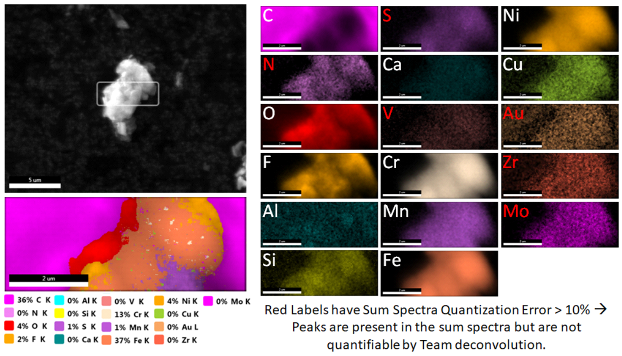

Figure 6: EDS map of a steel particle with trace elemental contaminants.

It can be seen that there are several elements present that are not in any steel specifications (e.g. F, S, & Ca). Distinguishing each variation in material type creates a verbose typing system that will become cumbersome to report due to the distinct variations of individual dominant particulate types.

A solution is provided in Fig 7, counting the number of particulates that exceed atomic % thresholds for each individual elemental channel [7].

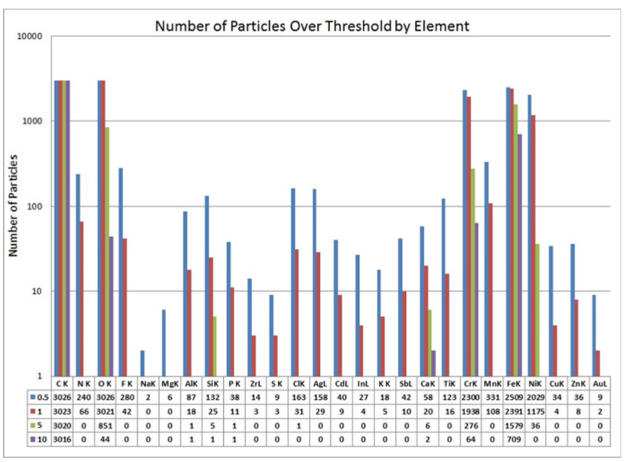

Figure 7: Example of reporting data for elemental channel counting. The four color bars for each elemental channel represents thresholds of 0.5, 1, 5, & 10 At %.

The channel counts are interpreted by looking at the distribution of elemental peaks with respect to each other. This captures the total particulate elemental volume of the spindle in the form a characteristic spectrum or relative distributions. To decode the distribution, individual elemental channels are dissected based on the composition of particulate types and material category typing statistics. For example, in Fig. 7 above, iron is most often steel and occasionally just Fe from material and typing statistics. Looking at chromium and nickel indicate the probable number of particulates in the Fe elemental channel that are steel. The disparity in small to large threshold suggest that there are likely some elemental Fe particulate and some small elemental Cr and Ni. Majority of the particulates collected were metallic.

CONCLUSION

A fully automated particulate characterization system has been developed. The system has been designed for throughput with minimal labor. A database and reporting system has been implemented for manual, automated, and combined manual and automated datasets. Manual inspections provide for targeted inquiries of particulate types and appearance with high resolution SEM images and X-ray spectra. Every particulate is manually selected from a field of view and spectra reviewed before reporting to PowerPoint. Isolation of cross contaminates on the surface of particulates is possible with the high resolution images and control over spectra excitation volumes. Collecting data manually cannot provide the throughput for real time process feedback due to acquisition, processing, and analysis burden. Ultimately the dataset produced with manual inspections are subjective and lack statistically integrity. The automated system produces a dataset capable of providing statistical statements about relative occurrences and distributions without a significant direct labor investment. The automated particulate analysis system will be utilized in continued forensic studies of assemblies in service and deploying into service. Significant improvements in field emission behaviour will be realized by analyzing process particulate evaluations.

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