NATURE AND IMPLICATION OF FOUND ACTUAL PARTICULATES ON THE INNER SURFACE OF CAVITIES IN A FULL-SCALE CRYOMODULE PREVIOUSLY OPERATED WITH BEAM*

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Abstract

We report on the preliminary results of the first study of found actual particulates on the inner surface of 5-cell CEBAF cavities in a full-scale cryomodule previously operated with beam. The procedure of particle collection is illustrated. The nature of studied particulates is presented. The implication of the findings will be discussed in view of reliable and efficient operation of CEBAF and future large-scale SRF accelerators.

INTRODUCTION

Field emission in an SRF cavity often has its root in small foreign particulates, lodging on the cavity inner surface. Although it is known that not all the surface particulates are active field emitters, the general practice nowadays is to avoid particulate contamination by careful cleaning and handling of individual cavities, as well as clean room assembly of cavity strings. Despite these elaborate processes, adequately clean cavity surface is still difficult to obtain for a beam-ready cavity placed in the accelerator tunnel. Consequentially, degradation in field emission onset is sometimes observed from vertical qualification test of an individual cavity to its test in a cryomodule. Moreover, as will be shown in this contribution, new particulates, shed from beam line components, may travel and arrive at the cavity surface, after a cryomodule has been placed in the accelerator tunnel. The nature of these "traveling beam-line particulates" landed on the inner surface of a beam-accelerating cavity is largely unknown for two reasons: (1) lack of access to such surfaces; (2) lack of a workable procedure for investigation without destroying the cavity.

In the decades-long history of large scale SRF machine operation in CEBAF, it has been known that the field emission in cavities placed in the tunnel might deteriorate over some time of beam operation. Among the consequences of this deterioration is an increased machine trip rate via its charging effect on cryogenic ceramic RF windows. In order to keep the trip rate to stay within the tolerable limit, the operation gradient of some cavities must be lowered. This results in an apparent "gradient loss", or more precisely a loss in "usable gradient", which in turn reduces the attainable beam energy for stable machine operation. Historically, two practical counter-measures are used in CEBAF to restore the "lost" gradient: (1) Helium processing; (2) cryomodule refurbishment [1-3]. Each of these two countermeasures has its own advantages and dis-advantages. Helium processing is less expensive and can be done *in-situ* in accelerator tunnel; but sometimes it produces a rather small effect. It is also possible that a cavity may be degraded from helium processing, as observed in some helium processed 7-cell cavities, newly installed for the 12 GeV energy upgrade of CEBAF [4]. Cryomodule refurbishment is more effective in raising the gradient capability of the original CEBAF cavities, for a superior cavity surface is obtained from reprocessing these cavities wit modern cleaning techniques and assembly procedures; but the cost for refurbishment is relatively high.

The root cause of the field emission deterioration problem in CEBAF is not known and has not been studied before. Recently, there has been an increased interest in gaining a clear understanding of the problem. A driving force is the need to run the 418 SRF cavities together stably for the upcoming physics run. Among these cavities, 80 of them are new 7-cells installed for the 12 GeV upgrade. They are operated at a nominal gradient of 17.5 MV/m. The corresponding peak surface electric field is ~40 MV/m, which is significantly higher than the typical values of 25-30 MV/m in the original 5-cell cavities. Due to the exponential nature of field emission, *the new 7-cell cavities are therefore at a higher risk as compared to the original 5-cell cavities, if the root cause is the same*. It should be noted that trips rooted from emission in the new 7-cell cavity cryomodules have its unique symptoms such as beam line vacuum spikes etc. In this contribution, we report on the preliminary results of our effort in identifying actual particulates on the inner surface of 5-cell CEBAF cavities in a full-scale cryomodule, which was previously operated with beam. The nature of the found and studied particulates is presented. The implication of the findings will be discussed in view of reliable and efficient operation of CEBAF and future large-scale SRF accelerators.

CAVITIES

The cavities were originally embedded in cryomodule FEL2, which was previously operated with beam. This module is slated to be refurbished in a similar fashion as the past 11 refurbishment modules, all of which successfully raised the acceleration voltage per module from 20 MV to 50 MV. It should be mentioned that the module FEL2 (now known as C50-12) suffered a vacuum accident at one point, in which the cavity inner surface was exposed to a shockwave and atmospheric pressure. This resulted in a large degradation in field emission onset

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for the first few exposed cavities. Subsequent helium processing helped somewhat, but the module could only deliver 35 MV, as opposed to the initial value of 40 MV [5]. It should also be mentioned that, just prior to the refurbishment, the module was additionally exercised in an effort to improve the field emission onset by plasma cleaning at room temperature [6].

Some detailed information about these cavities is listed in Table 1. For readers who are unfamiliar with the original CEBAF cryomodule architecture, Ref. [7] provides a good source of information. Briefly, two cavities are assembled into a cavity pair which is enclosed in a large liquid helium tank, making a *cryo-unit*. Four cryo-units are assembled in series and form a cryomodule. Each end of the module is fashioned with a cryogenic end can, for the purpose of supplying and returning cryogens.

In our present studies, only two pairs, each next to the supply end can and return end can, were selected for particulate collection. Particulate collecting wand penetrated each cavity from the flange away from the center joint, which remained untouched during the particulate collection process. In total, four batches of samples, each from the first and last cavity pair.

Table 1: Cavities and their Locations in Cryomodule

Location*	Name	Cryo-unit	Particulate Collection Sample Batch#
1	IA080#	CU#212	1
2	IA355#	CU#212	2
3	IA367	CU#211	N/A
4	IA366	CU#211	N/A
5	IA365	CU#210	N/A
6	IA366	CU#210	N/A
7	IA290	CU#209	4
8	IA351#	CU#209	3

* Cavity location number starts at the liquid helium supply end can side of the cryomodule.

Cavity has large grain size of ~ mm, apparently heat-treated at a temperature of >1250ºC.

PARTICULATE COLLECTION AND TRANSFER PROCEDURE

The procedure of particulate collection from the cavity inner surface and transfer to a carbon tape for inspection is illustrated below:

- Cars wash with de-ionized water the hermetically sealed cavity pair to remove airborne particles and debris from cutting apart the vacuum vessel.
- Bag the washed cavity pair and then transport to a potable clean room set up in the high bay area.
- Remove covering bag and dry over night with the portable clean room in full operation. Monitor the particle count inside the portable clean room (should have zero count when nothing is moving).
- Wipe the blank-off flange attached to the beam line isolation gate valve with solvent soaked lint-free rags.
- Remove blank-off, visually inspect the “air side” of the isolation gate valve. Wipe the exposed surfaces thoroughly to remove any cooper chips/dust from gasket crushing.
- Attach the freshly cleaned venting manifold to the isolation gate valve and evacuate the venting manifold using a scroll pump.
- Valve off the scroll pump. Open the gate valve. Vent the cavity slowly for overnight (there are two filters in the manifold, one in the narrow bleeding line and one right immediately next to the 6 inch Conflat attached to the gate valve).
- Remove venting manifold to expose the cavity inner surface. Start to monitor the particle count in the working space near the gate valve opening.
- Visually inspect the gate valve housing and the inner surface of the cavity beam tube.
- Wrap a piece of fresh lint-free clean room cloth around a clean rod to make a wiping wand.
- Spray solvent at end of wand to wet the cloth.
- Insert wand into cavity through gate valve throat.
- Press wand against cavity wall within line of sight, including the beam tube surface, first three irises, wipe back and forth to collect particles.
- Retrieve wand from cavity.
- Peel carbon tape [8] from paper.
- Press carbon tape against cloth area that was in contact with cavity surface.
- Re-attach the carbon tape to the host paper in its original place.
- Place tape in a sealed container; transport it to the lab for scanning electron microscope (SEM) inspection (FE SEM, Hitachi S-4700 with EDS).
- Peel off the cover paper on the back side of the carbon tape. Attach tape to sample holder for SEM. Lift off the cover paper from the particulate-bearing side of the tape.
- Load tapes in SEM for inspection.

To illustrate the key step of particulate collection from the cavity inner surface, an example is given in Fig. 1.

Figure 1: Particulate collection from cavity inner surface.

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Due to large number of particulates transferred onto the carbon tape surface, we randomly selected particulates for examination. Roughly speaking, about 100 particulates are selected for each cavity (total number of examined particulates amounts to ~500). Initially, most of selected particulates are studied with EDS for elemental analysis. Later on, with characteristics of the populous "clay" and stainless-steel particulates established, DES was skipped sometime to expedite the process.

Besides samples collected as described above, control samples were also prepared by repeating all the steps except the step of "pressing want against cavity wall, wipe back and forth". The control samples allowed us to rule out false ID of particulates due to cross contamination introduced by the handling. It should be also mentioned that, over the entire procedure, the worker is fully gowned with clean room garments and gloves.

RESULTS

Particulates Being Chased After

Prior to our efforts, three types of particulate were on the "hunting" list: (1) Indium, (2) Titanium, and (3) Carbon.

Indium earns the top position as many of the cavity vacuum joints are sealed with crushed indium wires. As it turns out, there is no single indium particle observed among the numerous particulates examined.

Titanium and carbon are the other two candidates for the following reasons: (1) Ion pumps are used in the warm beam lines next to a cryomodule. An ion pump must be turned back on after it is tripped off during beam operation; (2) Viton O-ring elastomers are used in beam line gate valves. There are many times during the machine operation and maintenance that a gate valve must be opened/closed.

Titanium-bearing particulates are indeed observed. But to our surprise, these particulates tend to bear also tantalum (see Fig. 2). Ultimately, it was brought to light that the beam-line ion pumps next to the cryomodules contain 50% titanium and 50% tantalum in the electrodes. The case then becomes clear that these titanium/tantalum particulates are originated from the beam line ion pumps.

It should be mentioned that titanium/tantalum particulates are observed in all four cavities. It is tempting to blame the vacuum accident mentioned earlier for sending these particulates into the cavity, but there is no evidence of higher probability in finding them in the vicinity of the outermost beam tunes from either end of the cryomodule. Therefore, we suspect that these titanium/tantalum particulates travel, through some mechanism not presently known to us, from the ion pump toward the center of the cryomodule and ultimately landed on the cavity surfaces. It is noticed that all the observed titanium/tantalum particulates have a typical size of less than 10 μm in a characteristic polygon shape with sharp edges. It is also noticed that the ratio of titanium to tantalum varies from particle to particle, with the extremes tend to be dominated by tantalum.

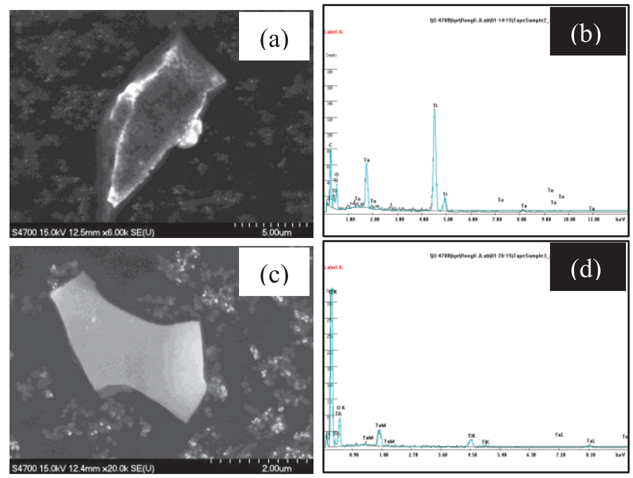

Figure 2: Titanium/tantalum particulates. (a) This 10 μm sized particulate is collected from niobium beam tube surface of cavity IA355; (b) EDX spectrum of particulate shown in (a). (c) This 4μm-sized particulate is collected from the first iris of cavity IA351; (d) EDX spectrum of particulate shown in (c).

Carbon particulates are also found, but none of them bear any element of fluorine, suggesting these carbon particulates are not originated from the Viton O-ring elastomer used in the beam line gate valves. The exact origin of these carbon particulates has to wait till further studies. Table 2 gives a summary of particulates on our "hunting list".

Table 2: Particulates being Chased After

Particulate	Rationale	Outcome
Indium	Indium seal used in CEBAF cavities	Null
Titanium	Ion pumps used in warm beam line next to cryomodule	Found, plus Tantalum from all four cavities
Carbon	Viton O-ring elastomer used at in cold and warm beam lines	Found, but interpretation complicated

Particulates in Massive Amount

Two types of particulates, in unexpectedly massive amount, were discovered. First, particulates containing such elements like silicon, calcium, aluminum, sodium, potassium and magnesium etc., are found in all studied cavities. These particulates bear elements consistent with that of "clay". Second, particulates containing iron, nickel, chromium and silicon etc., are also found in all studied cavities. Clearly, these are stainless-steel particulates. Fig. 3 gives two examples, one each for a "clay" and stainless-steel particulate. The size of these two types of particulates can be as large as 50 μm. Both the "clay" and stainless-steel particulates carry their own distinct physical appearance. A "clay" particulate is typically "grainy" with apparent fissures over its surface.

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A stainless-steel particulate typically has directional textures, suggesting these particulates are shed from the galled spots of the machined surfaces of the beam line stainless-steel components.

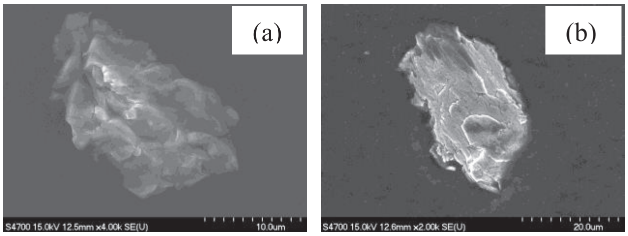

Figure 3: (a) A “clay” particulate collected from the beam tube of cavity IA080; (b) A stainless-steel particulate collected from the beam tube of cavity IA351.

A clear locational dependence is observed for these populous “clay” and stainless-steel particulates: *the farther away from the end of a cavity, the less chance of collecting these particulates*. Given the small area in a rag for effective particulate collecting, we estimate that the efficacy for particulate collection from a cavity wall and transfer to a carbon tape should have small variation. We believe the observed locational dependence reflects the particulate density profile along the beam axis of the cavity string. As we approached the cavity pairs from both the end next to end cans and the end away from the end cans, one may conclude that all these “clay” and stainless-steel particulates are originated from somewhere external to the cavity pair, as opposed to somewhere external to the cryomodule. Again, this observation suggests that *the vacuum accident alone is not enough to account for the found actual particulates*. Table 3 gives a summary of particulates in massive amount.

Table 3: Particulates in Massive Amount

Particulate	Remark	Note
“Clay”*	Found in all studied cavities, can be as large as 50 µm	This might be a direct result of the vacuum accident the module suffered
Stainless-steel	Found in all studied cavities, can be as large as 50 µm	Does this mean an intrinsic particulate shedding from a stainless-steel surface?

*A “clay” particulate contains such elements like silicon, calcium, sodium and magnesium etc.

Other Particulates

Some rare particulates are found. A few interesting examples are given in Fig. 4. The copper particulate shown in Fig. 4(a) indicates our procedure is capable of capturing and transferring micron-sized fine particulates. The niobium particulate shown in Fig. 4(b) suggests its origin from the inner surface of the niobium cavity itself. The spherical shape of the iron particulate shown in Fig. 4(c) suggests that it might have been previously an active field emitter which was melted by the emission current. The near spherical particulate with granule sub-structure shown in Fig. 4(d) suggests incomplete melting might have occurred due to field emission current or perhaps pre-disassembly plasma cleaning.

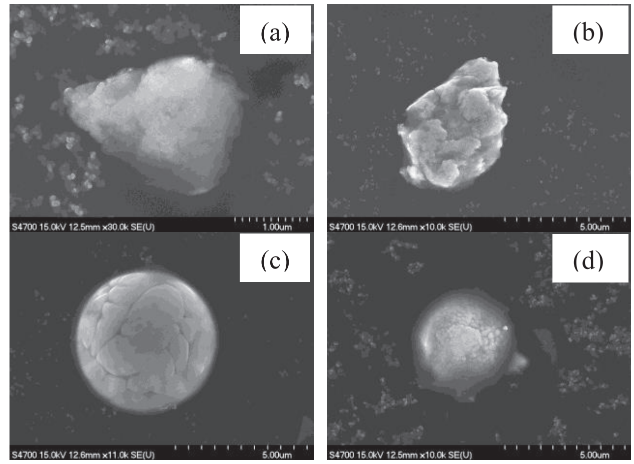

Figure 4: (a) A 2 µm sized copper particulate collected from the beam tube/first iris region of cavity IA351; (b) A niobium particulate collected from the first iris of cavity IA080; (c) A spherical iron particulate collected from the beam tube of cavity IA080; (d) A near spherical particulate with granule sub-structures, consisting of multiple elements including iron, chromium, nickel and sulfur, collected from the second iris of cavity IA290.

IMPLICATIONS

The first data we obtained regarding actual particulates on the inner surface of cavities in a full-scale CEBAF-style cryomodule previously operated with beam give direct evidence that beam line ion pumps can shed particulates bearing elements of electrode materials. There is also indirect evidence that these a-few-micron-sized particulates can travel, under some unknown mechanism, toward and within SRF cavities, with a “penetration depth” of at least 2-cavity length.

We proposed several schemes for elimination of new titanium/tantalum particulate arriving at CEBAF cavities.

- Procedure control of turning on/off of ion pumps.
- Shut off ion pumps completely.
- Implement baffles for shielding particulates from going into cavity.

The beam line ion pumps are already attached during initial CEBAF cryomodule assembly to provide essential function of maintaining the cavity vacuum. When placed in the CEBAF tunnel, these pumps also serve the purpose of beam line vacuum monitoring for machine fault protection. An alternative vacuum-monitoring device, such as a full-range vacuum gauge, must be added if an ion pump is shut off. The loss of effective pumping from shutting off ion pump is inevitable. This ultimately boils down to the question of how much pump pumping is actually provided by these pumps in the backdrop of the cryo-pumping from the cold cavity surface. Further analysis is underway to find out the effective pumping by

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the ion pumps and by the cryo-pumping due to cold cavity surfaces.

The discovery of massive amount of "clay" particulates is not a surprise to us, given the vacuum accident the module suffered. However, the evidence in hand suggests that the shock wave effect alone from the vacuum accident is not sufficient to account for the characteristics of the locational dependence of the found particulates. The discovery of massive number of stainless-steel particulates is a surprise. These particulates also follow the similar locational dependence as that for the "clay" particulates. At the moment, the best fit explanation to these "clay" and stainless-steel particulates seems to be contamination from environment and tooling during the cavity pair and string assembly.

The origin of the found carbon-bearing particulates is a mystery to us. However, the Viton O-ring elastomer can be ruled out. Therefore beam line gate valves in CEBAF should not be blamed for shedding these particulates.

The fact that not a single indium-bearing particulate was found indicates that there is no fundamental case against indium seals from the particulate contamination point of view.

CONCLUSIONS

The first attempt to study actual particulates on the inner surface of cavities in a full-scale cryomodule previously operated with beam is successful. A procedure for particulate collection, transfer and examination was developed and applied. The procedure is proven to be capable to capture small particulates down to the size of a couple of micro-meters. A strong case against beam line ion pumps is emerging. Small particulates of a few micro-meter in size shed off the ion pump electrodes somehow travel a fairly long distance and arrive at the cavity surface. At the moment, the titanium/tantalum particulates stand out as the top root cause to the apparent loss of the "usable gradient" in cavities placed in CEBAF tunnel. Therefore, we are presently focused on the mitigation against these titanium/tantalum particulates. As was pointed out earlier, the new 7-cell CEBAF 12 GeV upgrade cavities are operated at a much higher peak surface electric field, these cavities are at a higher risk of gradient degradation, if the arrival of new titanium/tantalum particulates is allowed to continue.

It is known that gas adsorption can also turn on new field emission by activating field emitters [9]. However, this scenario is not favored, because the past experience with the 5-cell CEBAF cavities has been that, after a room temperature thermal cycling, the field emission in a module tends to get worse, instead of better as expected from deactivation of field emitters by gas desorption [10]. We are presently in the process of examining the field emission behaviors of the newly installed 7-cell 12 GeV upgrade cavities, in which emitter activation by gas absorption has neither been established nor ruled out so far due to limited data.

What we have learned so far may ultimately lead to stoppage of the loss of the usable gradient in CEBAF, which is important to achieve and maintain CEBAF's energy reach in a cost effective manner. Another natural consequence of stopping new field emitters is a reduced trip rate and therefore an increased machine reliability and availability. For the original CEBAF 5-cell cavities, one can expect a reduced RF arc fault trip rate. For the new 7-cell cavities, one can expect a reduced field emission induced beam line vacuum fault trip rate. Given the large number of cavities (418 total) installed in the CEBAF machine, even a small gain in field emission reduction may lead to a large improvement in machine performance. Understanding the root cause of the trip rate is important for CEBAF operation and may also benefit future large-scale SRF linacs for photon science and energy production, in which the requirement for the machine trip rate is significantly more demanding as compared to that of CEBAF.

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