CEBAF Particulate Field-Emitter Control

Rongli Geng July 15, 2015

We thank John Fischer, Chris Dreyfuss, and members of cryomodule assembly group for what they have done in supporting the particulate collection process. We thank Olga Trofimova for her valuable collaboration in producing high quality microscopy data and in offering insightful analysis.

Outline

- Background
- New effort and data
 - -particulate field-emitter collection and identification
 - from cavities previously operated with beam
- Implications and opportunities

Background

- There have been concerns/speculations of field emission degradation over long term after SRF cavities/cryomodules being placed in accelerator tunnel.
 - Known mechanism: work function lowering by accumulation of monolayers of cryosorbed gas species on surface of high electric field region.
 - Gas loading from warm beam line components.
 - Gas release from field-emitted e⁻ bombardment of cold surfaces.
 - Suspected mechanisms: beam-line component particulate shedding and subsequent in-vacuum migration into cavity.

Background (continued)

- Past experience with CEBAF cavities/cryomodules suggests "new field emission" appearing randomly over long period of post-installation operation.
 - The response to such a scenario is to lower the cavity gradient so as to keep the cavity trip rate at acceptable level.
 - A natural consequence of such reaction: apparent "loss" of operation gradient.
 - Understanding of such a scenario is desired in order to find a countermeasure for stopping the gradient loss.

Background (continued)

- "Cryosorbed gases" mechanism is not favored in case of CEBAF.
 - Because one expects to see improvement in field emission when the cavities are thermally cycled (20 K or above) and then re-cooled down, as a result of thermal-desorption of gas species from high electric field regions).
 - Response of CEBAF cavities to thermal cycling has been opposite: field emission being worsened after thermal cycling (oral history).

 "Beam-line particulate shedding and migration" mechanism is a favored model to interpret the field emission behavior of installed CEBAF cavities.

The New Effort

- An effort has been undertaken since September 2014, aiming for understanding and control of CEBAF particulate field emitters.
- The opportunistic cavities are those from the current C50 re-furbishment cryomodule (C50-12).
- These cavities were previously operated with beam (in FEL); were loaded with field emission; (were treated by the trial plasma cleaning?)
- Despite their unique history, these cavities are considered to be acceptable for the intended studies (more later).

Summary of Findings

Particulates being chased after Rationale Result of hunting Particulate note Indium In seals used in CEBAE cavities Null and cavity pairs Titanium Clear evidence of Ti/Ta lon pumps used in warm beam Found, plus Tantalum line just next to the end plate of in all ("outer" as well as particulates from beamline ion "inner") cavities studied cryomodule pump. Top priority of further study. Carbon Found Viton O-ring elastomer seals Interpretation is complicated used at ends of cavity pairs

Particulates unexpected but discovered

Particulate	Remark	note
Stainless-steel	In huge number quantities	New discovery of a new intrinsic source of particulate/field emitter?
"clay"	In huge number quantities	Cavity string vacuum accident?
niobium	Occasionally observed	

Summary of Findings (continued)

- The deeper into the cavity, the less number of particulates collected for each cavity inspected.
 - Sources of particulates are external to cavities.
 - Particulate "penetration depth" effect?
- No particular orientation dependence.
 - ➤ The observed particulates are unlikely related to the vacuum accident that occurred sometime during its service in FEL.
- Ti- & Ta- bearing particulates are found even in second cavity from the end of cryomodule.

Ti- & Ta- particulates somehow can migrate a long way from the ion pump chamber into the cavity space.

Implications and Opportunities

- Focus on sources of particulate external to cavities.
- Beam-line ion pumps (IP's) shed Ti- & Ta- bearing particulates. These particulates migrate toward SRF cavities, with a "penetration depth" of at least 2 cavity length.
 - Procedural control of IP turning on/off.
 - ✓ Isolate cavity RF surface by closing gate valve when IP is turned on/off.
 - Replace beam line IP with "non-particulate-shedding" alternative. ✓ NEG pumps.
 - Implement "particulate shield" between ion pumps and cavities.
- Stainless-steel surfaces shed particulates. These particulates migrate toward SRF cavities.



Surface treatment for reduced particulate shedding (Electropolish)

Surface modification for reduced particulate shedding (Nb coating)

C50-12 Rework Cryomodule Cavities

Received 11/21/14 Rongh (From John Fischer)



TH

TH = Top Host (Wavesuide) Renound: L/R defined for view when gate valve knows facing downward

Cavity Table

Cavity Location*	Cryounit name	Cavity name	note	Particle collection
1	CU#212	IA080	Second cavity pair dis-assembled	Batch1
2	CU#212	IA355		Batch2
3	CU#211	IA367	Third cavity pair dis-assembled	
4	CU#211	IA366	Preferred pair for integrated Q0 test	
5	CU#210	IA365	First cavity pair dis-assembled	
6	CU#210	IA363		
7	CU#209	IA290	Last cavity pair dis-assembled	Batch4
8	CU#209	IA351		Batch3

*Cavity location starting at liquid helium supply end can side

From Drawing provided by Fischer: CU209: Cavity at left IA290; at right IA351

Cavity Venting in Portable Clean Room



Collecting Particulates



First Collection from IA080

Cavity is "Large grain" apparently post purified at > 1250 C This cavity is the first from the Supply End can side



IA080 Collection Areas – Sample Batch 1



Gate valve housing surface near knifedge at blank-off side (two big Cu particles were noticed by unaided eyes)

Note

We do not list C or O in compositional analysis as they are not separable from background due to use of carbon tape for hosting particulates. (exceptions to be noted)

The focus of compositional analysis is the metal elements.

Non-metal elements other than C, O are also spelled out.

When multiple elements present, list all in random order. (it can be done if needed)

Not all particulates present on tape are analyzed due to overwhelming number of particle counts on some samples. General impression of particulate density is given as a qualitative dependence of particulate density with probing depth.

Ca, Na, Mg, Si



Most probably air-borne particles?

IA080 Inner Surface SS Beam Tube Fe, Cr, Ca, Na, Mg, Si



Ca, Na, Mg, Si



Fe, Cr, Mg, Si



Fe, Ni, Cr, Si



С, О



Tape artifact?

Ta, (Ti)



Composition to be determined 10feb15

K, Na, Al, Si



Clay particle mixed with Si residual?

Fe



Melted iron ball?

Al, P



Check EDS again 10feb15

Ca, S



Cr, Fe, Ca, Na, Si



Dirty stainless steel particle

Fe, Cr, Ni, Na, Mg, Al, Si



Ca, Mg



Ca, Na, Mg, Si



Cr, Fe, Mn, Ca, K, Na, Mg, Si



IA080 Inner Surface SS Beam Tube Ca, Al, Si, S



dust

7/15/15, 2015 OPS Stay Treat, JLab

Typical of stainless steel



 Quick EDS analysis showed typical fingerprint of stainless steel: Fe, Ni and Cr peaks (and C due to the carbon tape substrate).

TapeSample1 area2 x3K1a_q00

С, О



 Quick EDS analysis showed C and O peaks in the ratio close to that of a substrate. No significant charging was registered. This could be a carbon tape artifact.

TapeSample1 area3 x3K p7_q00

Background type EDS

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Rongli Geng

С, О



 Quick EDS analysis showed C and O peaks in the ratio close to that of a substrate.
No significant charging was registered. This could be a carbon tape artifact.

TapeSample1 area3 x4K p2_q00
IA080 Inner Surface SS Beam Tube

Al, Si, Ca, Na Typical clay



Quick EDS analysis showed
Al, Si, Ca and Na peaks
typical for clay or/and dust
particles. C and O peaks
were present due to the
carbon tape substrate.
Particle demonstrated
significant charging.

TapeSample1 area2 2K1e_q00

IA080 Inner Surface Nb Beam Tube

Al, Si, O, C





C N	54.10	62.49	0.2554	1.0123	0.4662	1.0003
ОК	39.59	34.33	0.1040	0.9933	0.2644	1.0001
AlK	3.17	1.63	0.0235	0.9178	0.8042	1.0013
SiK	3.14	1.55	0.0254	0.9371	0.8640	1.0000
Total	100.00	100.00				
Element	Net Inte	. Bko	d Inte.	Inte. Err	or 1	P/B
СК	14.38		0.06	2.71	228	3.83
- I.						
οĸ	13.62		0.06	2.79	210	5.67
O K Alk	13.62 3.69		0.06	2.79 5.45	210	5.67 1.00

Quantify composition 10feb15

IAO80 Inner Surface Nb Beam Tube Ta, Ti



IA080 Inner Surface Nb Beam Tube Si, (K)



Melted Si ball? Check size of Si ball from Huitian barrel polishing

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IA080 1st Iris from Gate Valve Al, Si, Cl, Ca, Ti



IA080 1st Iris from Gate Valve Nb



Clear proof that the wiping/transferring technique works

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IA080 1st Iris from Gate Valve Mg, Al, Si, S, Cl, Ca



clay

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IA080 2nd Iris from Gate Valve Si, Ti, Cu, Mg



Quantify composition 10feb15

IA080 2nd Iris from Gate Valve Ta, Ti



IA080 2nd Iris from Gate Valve Cr, Fe



Comparison of center area of four tapes



S4700 15.0kV 12.6mm x50 SE(U)

1.00mm

S4700 15.0kV 12.1mm x50 SE(U)

1.00mm

1.00mm

Nb Beam tube

Tentative Conclusions

- Found Ti-bearing particles (from Ion pump?)
- Found Ta-bearing particles (from ion pump?)
- Found Cu-bearing particles (from gate valve Cu gasket?)
- Found Al-bearing particles (clay, could come from environment)
- Found Cr-, Fe-, Ni- bearing particles (from beam line flanges?)
- Found lots of "dirt" particulates (source yet to be determined, could be from sample handling)
 - Later on, SEM inspection focused on central areas for minimized possibility of cross-contamination
- No indium particulates found so far
- General trend: the deeper into the cavity, the cleaner



E-mail from Olga on January 16, 2015:

... I have finished Batch 2 and, as usual, will send you the results in several emails. The tendency is perfectly clear: each next sample is cleaner than the previous!

IA355 SS beam pipe 6 o'clock, 1 Fe, Ni, Cr



IA355 SS beam pipe 6 o'clock, 2 Fe, Ni, Cr



IA355 SS beam pipe 6 o'clock, 3 Si, Ca, Na, Mg, K



IA255 SS beam pipe 6 o'clock, 4



IA355 SS beam pipe 6 o'clock

C, O peaks (high level of O), traces of Al and Si. Sample charges heavily.



\S-4700\bpr\Rongli JLab\01-09-15\TapeSample2_1 area3 10K 4c eds4.spc .abel : .cquisition Time : 10:43:20 Date: 9-Jan-2015													
V : 15.00 Tilt: 0.00 Take-off:30.00 AmpT : 102.4 etector Type:SUTW, Sapphire Resolution:131.05 Lsec:95													
DDAX ZAF Quantification (Standardless) Element Normalized EEC Table : User c:\edax32\eds\genuser.sec													
Element	Wt %	At %	K-Ratio	Z	A	F							
C K O K AlK SiK Total	65.36 33.56 0.64 0.44 100.00	71.80 27.67 0.31 0.21 100.00	0.4347 0.0785 0.0048 0.0037	1.0071 0.9883 0.9129 0.9317	0.6601 0.2366 0.8156 0.8958	1.0003 1.0000 1.0002 1.0000							
Element	Net Inte	e. Bk	gd Inte.	Inte. Er	ror	P/B							
C K O K AlK SiK	13.71 5.76 0.42 0.29		0.06 0.13 0.08 0.09	2.78 4.37 18.71 24.22	21	17.17 15.58 5.00 3.11							



IA355 SS beam pipe 6 o'clock

TapeSample2_1 area3 x2K 5_q00



 C, O peaks. Sample charges heavily. EDS results are similar to the previous particle.

IA355 SS beam pipe 6 o'clock Fe, Ni, Cr, Si



TapeSample2_1 area3 x10K ar1f_q00

IA355 SS beam pipe 3 o'clock Si, Ca, Na, Mg



IA355 SS beam pipe 3 o'clock

TapeSample2_2 area1 x13K 4_q00



 EDS results are similar to the previous sample: Si, Ca, Na, Mg peaks.

IA355 SS beam pipe 3 o'clock Ti, Si, Mg



IA355 SS beam pipe 3 o'clock Fe, Ni, Cr, Si, Mg, Na



IA355 SS beam pipe 3 o'clock Si, Ca, Na, Mg



IA355 SS beam pipe 3 o'clock

TapeSample2_2 area4 x5K 3_q00



 Quick EDS analysis showed Si and O peaks (C peak is always present due to the carbon tape substrate).
 Silicon oxide particle.

IA355 SS beam pipe 3 o'clock Fe, Ni, Cr, Si



TapeSample2_2 area5 x25K fr1b_q00

Ca, Mg



Ti, Ta, (Si?)



Ta, Ti, (Si?)



Fe, Ni, Cr



 Quick EDS analysis showed typical fingerprint of stainless steel: Fe, Ni and Cr peaks (and C due to the carbon tape substrate).

TapeSample2_3 area2 x1K p2_q00

Fe, Ni, Cr, Si



Ti, Ta



C, S, Ca, Na

Weight percent?





S, Ca



Si oxide (a grain of sand?)


IA355 Nb beam pipe

Cu, Cl, S



IA355 Nb beam pipe

Ag, Al, Ca, S, Cl



IA355 Nb beam pipe

Mn, Fe



Ca



Si, Ca



Al, Fe, Zn, Mg, K, Ca, Si,



Post-emission "melted" alloy ball?



- Quick EDS analysis showed:
- top particle produced C, O, Al, Si peaks;
- bottom particle produced C & O peaks in ratio similar to that of a carbon tape substrate, hence it's probably a tape artifact.

TapeSample2_4 area4 x2K p9ab_q00



 Quick EDS analysis showed C and O peaks. O peak is higher than that of a substrate. Particle is charging significantly.

TapeSample2_4 area4 x3K p6_q00

IA355 1st iris Ca, Al, Mg, Na, Fe, Si, S



Ca, Na, Si



C and O



Quick EDS analysis showed
C and O peaks in the ratio
close to that of a substrate.
No significant charging was
registered. This could be a
carbon tape artifact.

TapeSample2_4 area4 x20K p7_q00

C and O



Quick EDS analysis showed
C and O peaks. O content
was much higher, than that
in the carbon tape
substrate. Both particles
showed significant charging.

TapeSample2_4 area4 x500 p4ab_q00

C and O



Quick EDS analysis showed
 C and O peaks. O content
 was much higher, than that
 in the carbon tape
 substrate. Particle showed
 significant charging.

TapeSample2_4 area4 x3500 p10_q00

IA355 3rd iris

C and O



Quick EDS analysis showed
 C and O peaks. O content
 was higher, than that in the
 carbon tape substrate. At
 high magnifications the
 thread was charging.

TapeSample2_6 area1 x150 thread_q00

IA355 Conclusion

- Clear trend: less particles with increasing probing depth
 - Implying particles from strong assemly (instead of cryomodule venting from vacuum accident)?
- First detection of spherical particles from iris
 - "post emission" melted field emitter?
- Established stainless steel particles being the dominant species
- Ti, Ta detected still despite long distance from beam line ion pump
 - Unique shapes with shape edges/tips
 - Implying Ti, Ta particles mobility and long "mean free path"?
- Some rare species detected
 - Cu, S
 - Ag
 - Zn

IA351 Collection Areas – Sample Batch 3



Tape 5 Control sample#2 (prepared after changing glove with new cloth)

----- Forwarded Message ------

Subject:Batch3 first results Date:Thu, 22 Jan 2015 16:08:07 -0500 From:Olga Trofimova <olgatr@jlab.org> To:Rongli Geng <geng@jlab.org>

Hello Rongli, I am sending you the results for samples 0 and 1 from batch3. As you see, sample 0 is contamination free -- i could not see any contaminating particles on it. Just to be sure, I took a couple of images of suspicious dark spots, but quick EDS showed nothing but regular C and O peaks typical for the tape. As for the sample 1, it's a different story. "Genview" pictures revealed a lot of contaminating particles, so I had to perform selective analysis. I picked several particles or groups of particles in different spots of each area and did imaging and EDS analysis on them. It occurred, that most of contaminating particles were stainless steel flakes. I took several full EDS spectra on them, but eventually kept taking quick EDS. If I saw typical stainless steel signature emerging, I stopped and wrote it down in my notebook. So, when you see a particle image, which does not have corresponding EDS spectra, it is a stainless steel flake. There were a couple of atypical contaminating particles though: one gave Cu peaks, another -- Ta and W. As usual, I am sending several emails with data. Best regards, Olga.

Rongli Geng

Fe, Ni, Cr, Si



Fe, Ni, Cr



Fe, Ni, Cr



С, О



Fe, Ni, Cr



Fe, Ni, Cr, Si



Ti, Ta



Fe, Ni, Cr, Si



Cu



TapeSample3_1 area3 x2K p5_q00



 Quick EDS analysis showed typical stainless steel signature of Fe, Cr and Ni peaks.

TapeSample3_1 area3 x4K p6_q00



 Quick EDS analysis showed typical stainless steel signature of Fe, Cr and Ni peaks.

Fe, Mn, Si, Al



Ca



Ti, Ta



Fe, Cr



IA351 Nb beam pipe & 1st iris Si, Al, Na, K, Ca







Cu



IA351 2nd iris

Zn, Cl, K, F


Fe, Ni, Cr



Fe, Ni, Cr



Si, Al, Mg, K, Fe



Post-emission "melted" alloy ball? *Al and Si peaks in combination are typical for clay and could be the signature of "environmental" contamination. Mg, K and traces of Fe could occur in clay too.*

Rongli Geng

Si, Al, Mg, Zn, Ca, K, Cl, S, P, F



Cu, S, P



Cu, Ti, P or Cu, S, P



Two possible interpretation of EDS results. To pick correct interpretation additional data or cross-reference is needed.



Si, K, Al, Na



Ti, Ta



Fe, Ca, Na, Al, Si, P, F



С, О, Та, Та



TaL

9.00

TaL TaL

keV

TaL

8.00

7.00

TapeSample3_3 area3 x4K p5_q00



 Quick EDS analysis showed C and O peaks with high content of O. particle was charging significantly.

С, О, Ті, Та



Fe, Ni, Cr



Ca, Si, S, N



Si, Al, Na, N



Fe, Cr, Si



Fe, Cr, Si



Si



Si, Al, Na, K



Fe, Ni, Cr, Nb



IA351 Conclusion

 Clear trend: less particles with increasing probing depth ??? (Definitely so. This phenomenon could be even observed directly on "genview"=low mag images of different sample areas.)

Fe, Cr, Ni, S



Post emission "melted" stainless steel ball?

Ca



Post emission "melted" Ca stain?

Ca, Na, Mg, Si, S



Post emission "partial melted" crystal?

C, N

Any possibility of F instead of N?



Post emission "melted" Carbon ball?

Si, Al



Si, Al



Ca, K, Al, Si



Post emission "partial melted" crystal?

Ca, Na, Al, Si, Cl



Post emission "partial melted" crystal?



High C and low O contents and low charging could indicate that this is a carbon tape defect (carbon tape artifact).

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NaCl



Quick EDS showed C,O
(from the substrate) and
peaks of Na and Cl.
Together with really
significant charging it may
indicate that this particle is
NaCl.

TapeSample4_1 area4 x5K p8_q00

С



Post emission "partial melted" carbon particulate?

С



Post emission "partial melted" carbon particulate?



Post emission "partial melted" carbon particulate?

C, Ta



Post emission "partial melted" C/Ta particulate?

С


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С
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Post emission "partial melted" carbon particulate?

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С,О



 Quick EDS showed peaks of C and O in the proportion similar to that of the carbon tape substrate. Charging is minimal. This may indicate that the feature is a carbon tape artifact.

TapeSample4_2 area1 x5K p5_q00

С, О



Quick EDS showed peaks of
C and O in the proportion
similar to that of the carbon
tape substrate. Charging is
minimal. This may indicate
that the feature is a carbon
tape artifact.

TapeSample4_2 area1 x11K p2_q00

С, О



TapeSample4_2 area3 x5K p3_q00

Note: charging is higher than in previous 2 cases.

С, О



Quick EDS showed peaks of C and O in the proportion similar to that of the carbon tape substrate. Charging is minimal. This may indicate that the feature is a carbon tape artifact.

TapeSample4_2 area3 x5K p4_q00

С, О



Quick EDS showed peaks of
 C and O in the proportion
 similar to that of the carbon
 tape substrate. Charging
 occur probably because the
 feature is elevated above
 the substrate.

TapeSample4_2 area4 x5K p1_q00

Appendix I

- Cover FPC waveguide ports with plastic caps (confinement of indium flakes and protect ceramic window)
- "car wash" assembly in production chem room power wash station
- "whole body "cover assembly with plastic sheet
- Transport to portable clean room in TLA high bay
- Remove plastic sheet
- Dry over night





- Wipe gate valve conflact joint area with solventsoaked clean room rags
- Remove 6" blank
- Visual inspect "air side" of the gate valve (photo at right)



Particulates on inner surface of Gate valve

- Wipe gate valve conflact joint area with solventsoaked clean room rags
- Remove 6" blank
- Visual inspect "air side" of the gate valve (photo at right)
- Attach venting manifold (each component freshly UHV cleaned). Two fliters in manifold (one next to pop-off, other, ceramic filter, next to 6" flange)



- Evacuate manifold with a pump station (scroll pump only)
- Manifold vacuum < -30 in Hg



Regular filter in bleeding line

- Valve off scroll pump
- Manifold vaccum maintaining at < -30 in Hg
- Open gate valve
- Pressure quickly rose.
 Ultimately settled at -3 in
 Hg (photo at right)
- Open mini-angle valve and needle valve in the bleeding line
- Pressure ultimately rose to 2 in Hg and stayed there



Pressure quickly rose to -3 in Hg after gate valve was opened – indicating cavity pair was already vented to near atmospheric 7/15/15, 2015 OPS \$P\$(CESSURE) prior to gate valve was opened \$6

- Close mini-angle valve and needle valve in bleeding line
- Valve in scroll pump to evacuate cavity pair to < -30 in Hg
- Valve off scroll pump
- Pressure quickly rose indicating a big leak in cavity pair
- Cavity pair vented overnight
- Pressure at -2 in Hg after over night venting
- Remove manifold
- Visual inspect inner surface of gate valve housing and beam tube inner surface



Appendix II

Sample Preparation Procedure

- Wrapping new cloth around rod to make wiping wand
- Spray solvent at end of wand to wet cloth
- Insert wand into cavity through gate valve throat
- Press wand against cavity wall, wipe back and forth to collect particles (this step is skipped for control sample)
- Retrieve wand from cavity
- Peel carbon tape from paper
- Press carbon tape against cloth area that was in contact with cavity surface
- Re-attach carbon tape to paper in its original place

Control Samples

Same sample preparation procedure except NO WIPING



View at low magnification "bubbles in tape" visible View at high magnification EDS shows C and O only

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