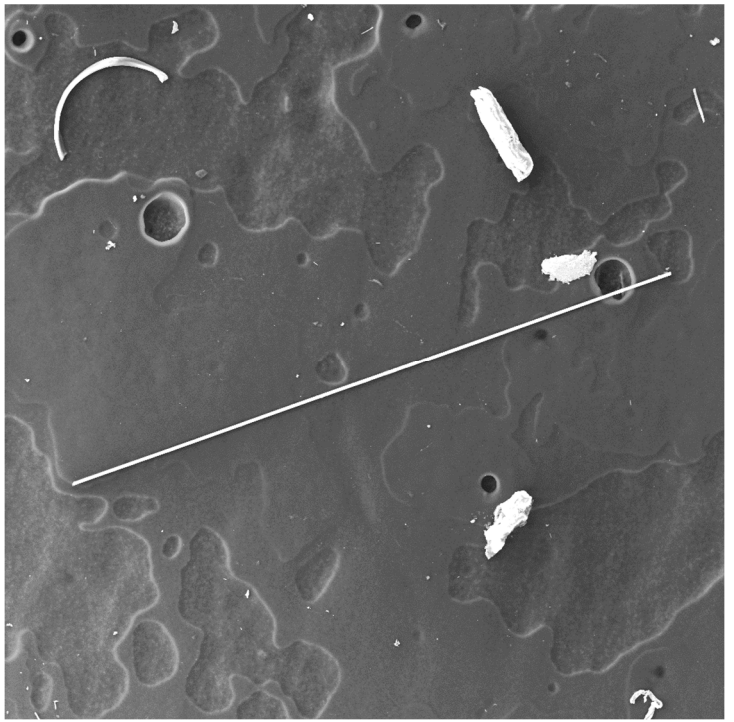
Identifying Specific Cryomodule and Cleanroom Particulate Contamination

Understanding Legacy Issues and Providing New Feedback Standards

Using a new automated particle detection and characterization system to examine samples



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U.S. DEPARTMENT OF ENERGY | Office of Science

JSA

Identifying Specific Particulate Contamination

- Particulates on SRF surfaces are **bad news**
- Particulates on high electric field surfaces become **electron emitters**
  - —>> heat, radiation, dark current, activation, secondary effects
- Particulates on beam vacuum surfaces **move**
  - —During assembly, venting, servicing, maybe from beam charging
- We've known "they" were there for a long time
- Standards have evolved – we now want to have **zero tolerance**
- Operating machines have a built-in legacy
- CEBAF was built to 1989 specification for 5-7 MV/m SRF performance
- "What really is in there?" "How can we best manage it?"
- "How do we migrate to >100 MV field emission-free cryomodules?"

Identifying Specific Particulate Contamination

- JLab has had a sustained interest in characterizing actual particulates present in order to target improvement efforts.
- Initial report on new systematic approach was provided in Lanzhou at SRF'17.

- Since then we've developed an **automated system** to systematically gather much more data
  - Look back at Poster TUP030 "Automation of Particulate Characterization", J. Spradlin *et al*.

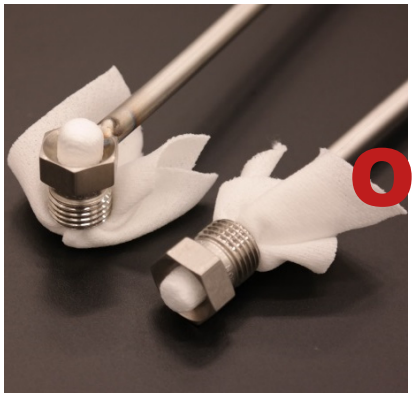
- **CEBAF systems tested to date**:
  - Two original-style cryomodules
  - Five warm inter-cryomodule beamline girders
  - One C100-style cryomodule

- **Cleanroom quality checks**

Identifying Specific Particulate Contamination

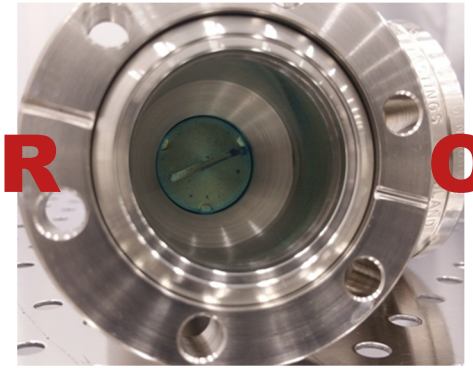
- We use commercially available controlled clean samples
  - Forensic gunshot residue (GSR) collection samples, 12 mm diameter
  - Certified clean carbon tape on serialized aluminum spindles
- Types of sample collection onto carbon tape:
  - swabbing, shaking of components, carbon tape contact & environmental witness

Swabbing



OR

Shaking of Components

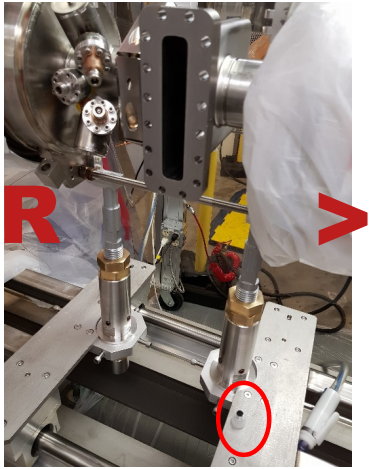


OR

Carbon Tape Contact

OR

Collection Witness



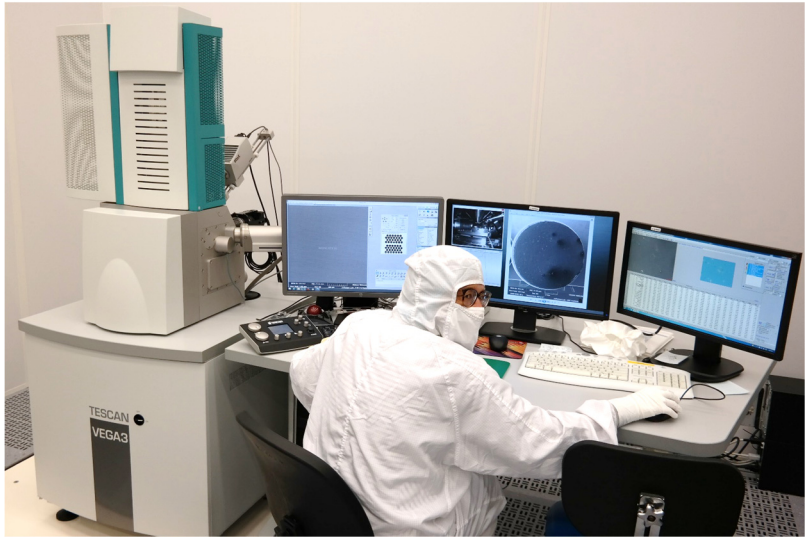
>>

GSR Sample
S####

Identifying Specific Particulate Contamination

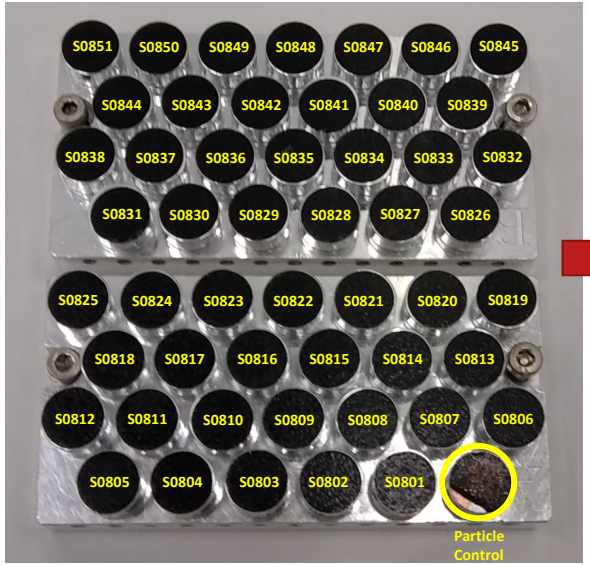
- Tescan VEGA XMH3 scanning electron microscope equipped with an EDAX EDS detector, located in the JLab cleanroom suite
  - Locate and size individual particulates
  - Elemental composition via X-ray spectroscopy

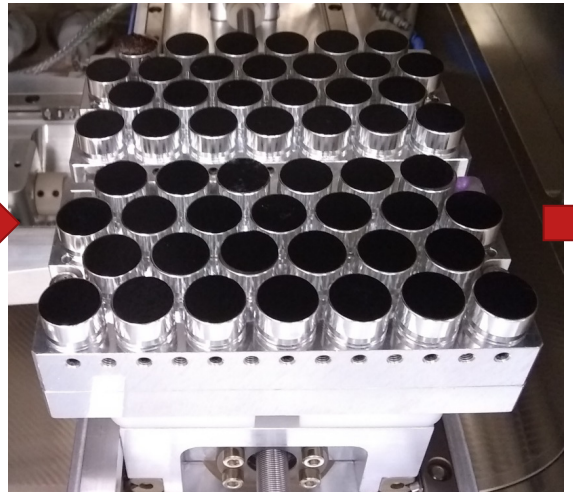
- **Manual operation**
  - Find and characterize ~25 of the largest particulates per spindle
  - Three spindles per day
- **Automated operation**
  - 4 hr setup time for ~30 spindles
  - Up to ~4300 analyzed particulates/day

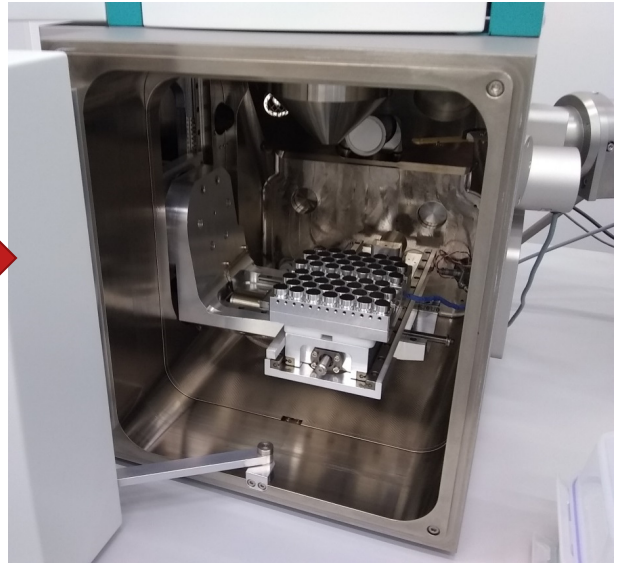


Identifying Specific Particulate Contamination

- System can process up to 51 GSR Samples & one Control Sample in single load of the SEM.







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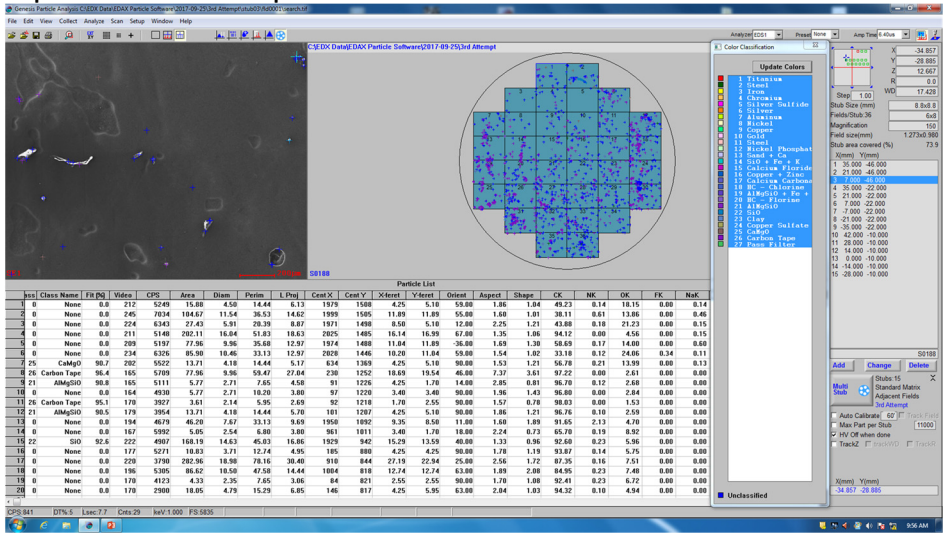
Identifying Specific Particulate Contamination

Data acquisition routine

- Mix of SE and BSE used to automatically locate and size particulates
- EDS Analysis of Particulate composition with EDAX Automated Particle Software
  - Analyze all identified particles in current window that are not Carbon Tape
  - Weight the composition of 24 different elements
- Step to next window on same sample, repeat to cover whole sample
- Step to next spindle, repeat

Example of 205× Completed Scan in EDAX Automated Particle Software



- **Resolution – @205× – 36 Fields of View**

  Window Size = 1.75 × 1.36 mm

  Particulate Sizes = ~ 4 µm to 1.4 mm

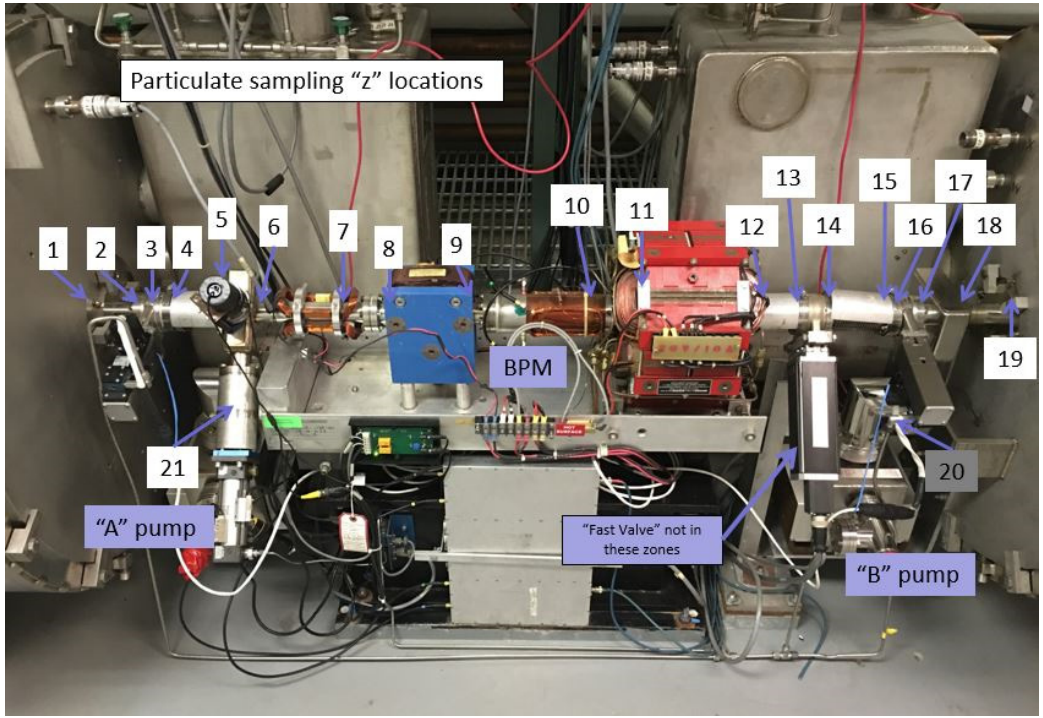
Identifying Specific Particulate Contamination

- Relative number of particulate counts per spindle with particular elemental presence may be meaningful
- Instead of typing particles and counting
- → Count number of times elemental composition signal is greater than a threshold
  - Particulates found are frequently aggregates
  - Overestimates number of physical particles but characterizes cross-contamination
  - Quantified At% is diluted as particle size decreases
    - Smaller particles have a larger background signal from carbon tape
- Larger particulates are presumed worse for Accelerator operations than smaller ones of same composition

Identifying Specific Particulate Contamination

Five warm girders between cryomodules were sampled prior to cleaning and reassembly. 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 sheet-like hydrocarbon pieces



Identifying Specific Particulate Contamination

Two original-style CEBAF cryomodules were sampled when removed for rework.

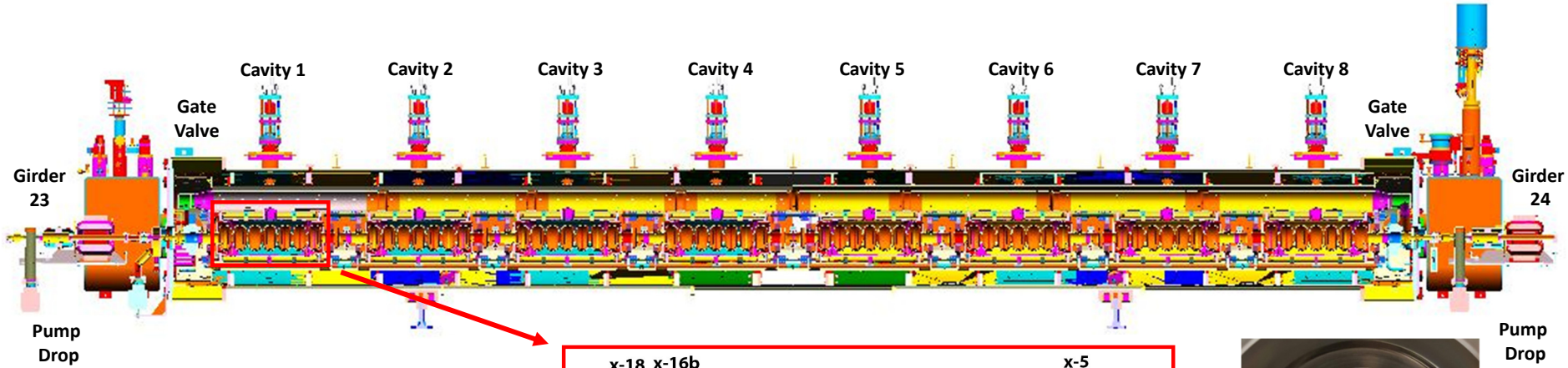
- "**Franklin**" had previously been reworked and reinstalled in 2009.
  - —Suffered a known vacuum servicing accident that transported particulates into cavities.
- "**Independence**" had been on the linac beamline since 1992.
  - —Relatively weak performer, suffered no known acute incidents.

Composition of the particulates found inside the cavities matched the large particulates found in the girders, consistent with the chief issue of CEBAF cryomodule performance degradation being transport of contamination from the girders into the SRF cavities.

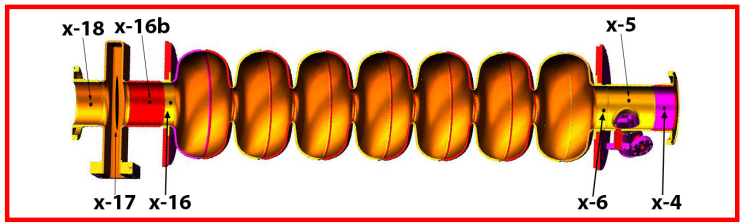
- —The metallic particulate load was ~3× higher in Franklin than in Independence.
- —Mineral and masonry type particulates were a factor of 10 higher in Franklin than Independence.
- —Interestingly, hydrocarbon signature particulates were most prevalent in Independence.
- —Over 100 polymer or elastomer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

Identifying Specific Particulate Contamination

- **C100 cryomodule** in CEBAF zone NL23 was removed for cleaning
- 68 particulate samples were collected during disassembly



- C100-6 NL23 bled up in tunnel from girder 24 to girder 23 then brought to Test Lab with end CF flanges not hermetically sealed.



S0867 = C1-4 → Cavity 1 HOM Beam Pipe

Analysis of Particulates Sampled from C100-6

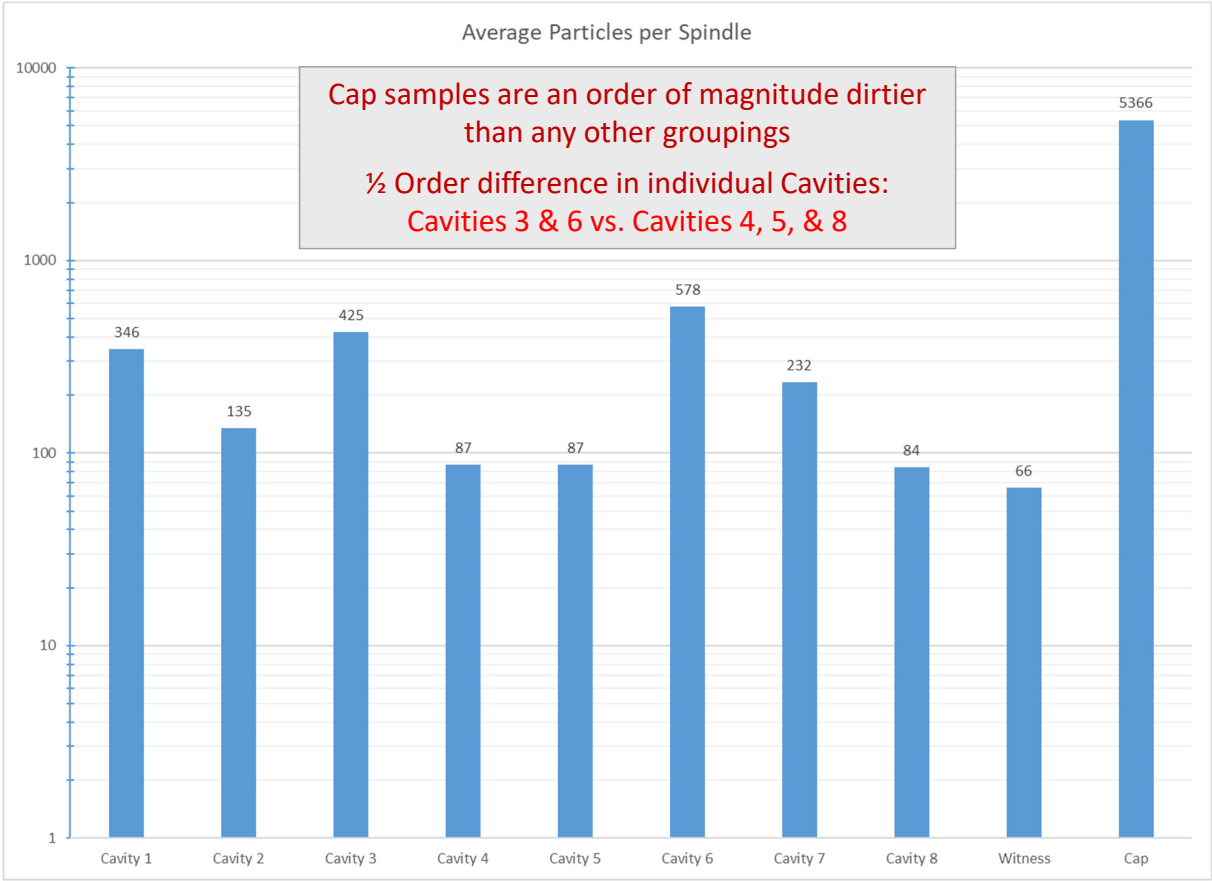
Summary Findings

- The automated scans found and identified an average of 84 to 587 particulates greater than 4 μm per spindle from the 58 cavity samples.
- Detected particulates ranged in size from 4–480 μm.
- The vast majority of the particulates on beampipe samples showed presence of silver, steel, aluminum or copper.
- For some reason, cavities 2 and 5 samples seemed cleaner than typical.
- Rather surprisingly, cavity 6 (of eight) showed up to 5× more particulates than the other cavities, with a wider variety of materials represented as well (including an assortment of minerals), suggesting a general contamination exposure event sometime during this cavity's preparation for assembly circa 2011.
- Waveguide samples were shocking.

C100-6 NL23 205× Particulate Results – Avg # per sample

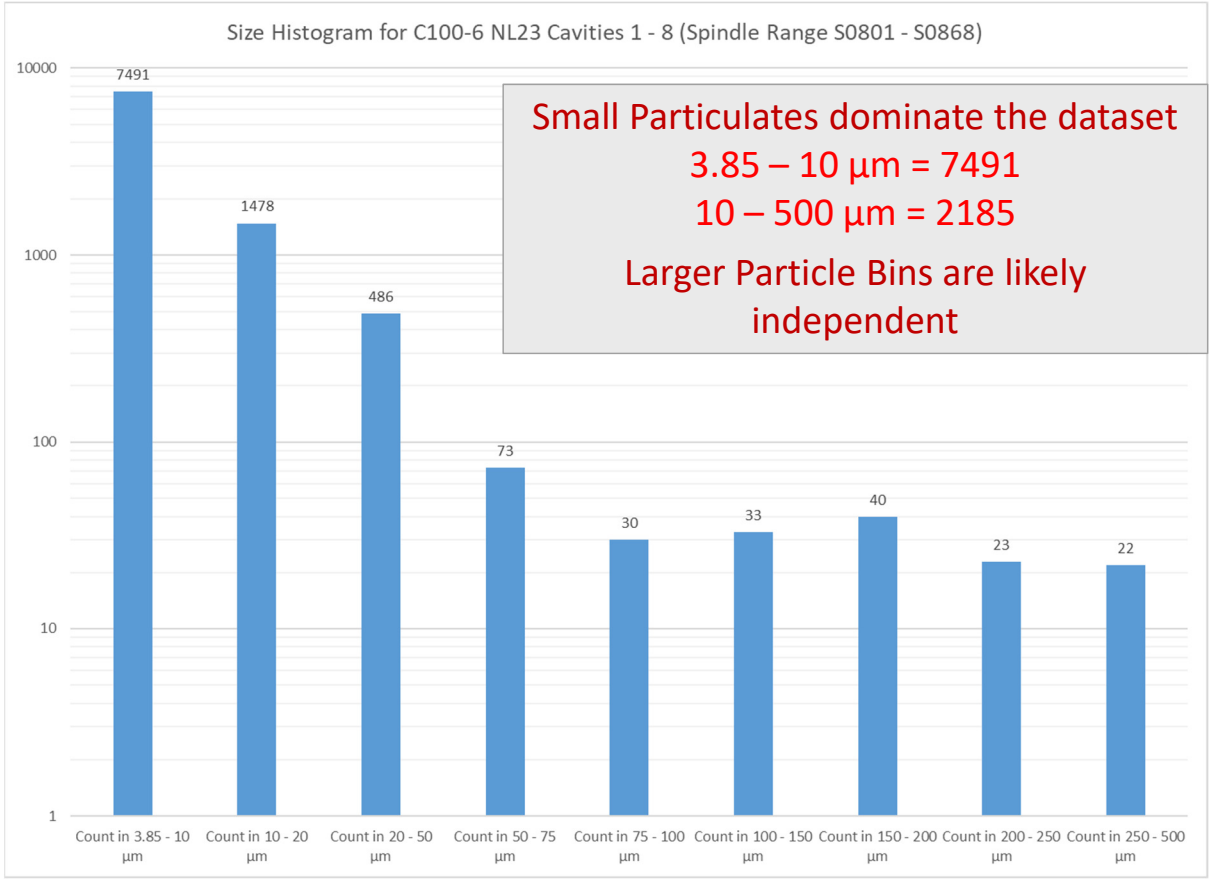
Number of Spindles per Grouping

Cavity 1 = 7
Cavity 2 = 5
Cavity 3 = 6
Cavity 4 = 5
Cavity 5 = 5
Cavity 6 = 4
Cavity 7 = 8
Cavity 8 = 15
Witness = 8
Cap = 4



C100-6 NL23 205× Particulate Results

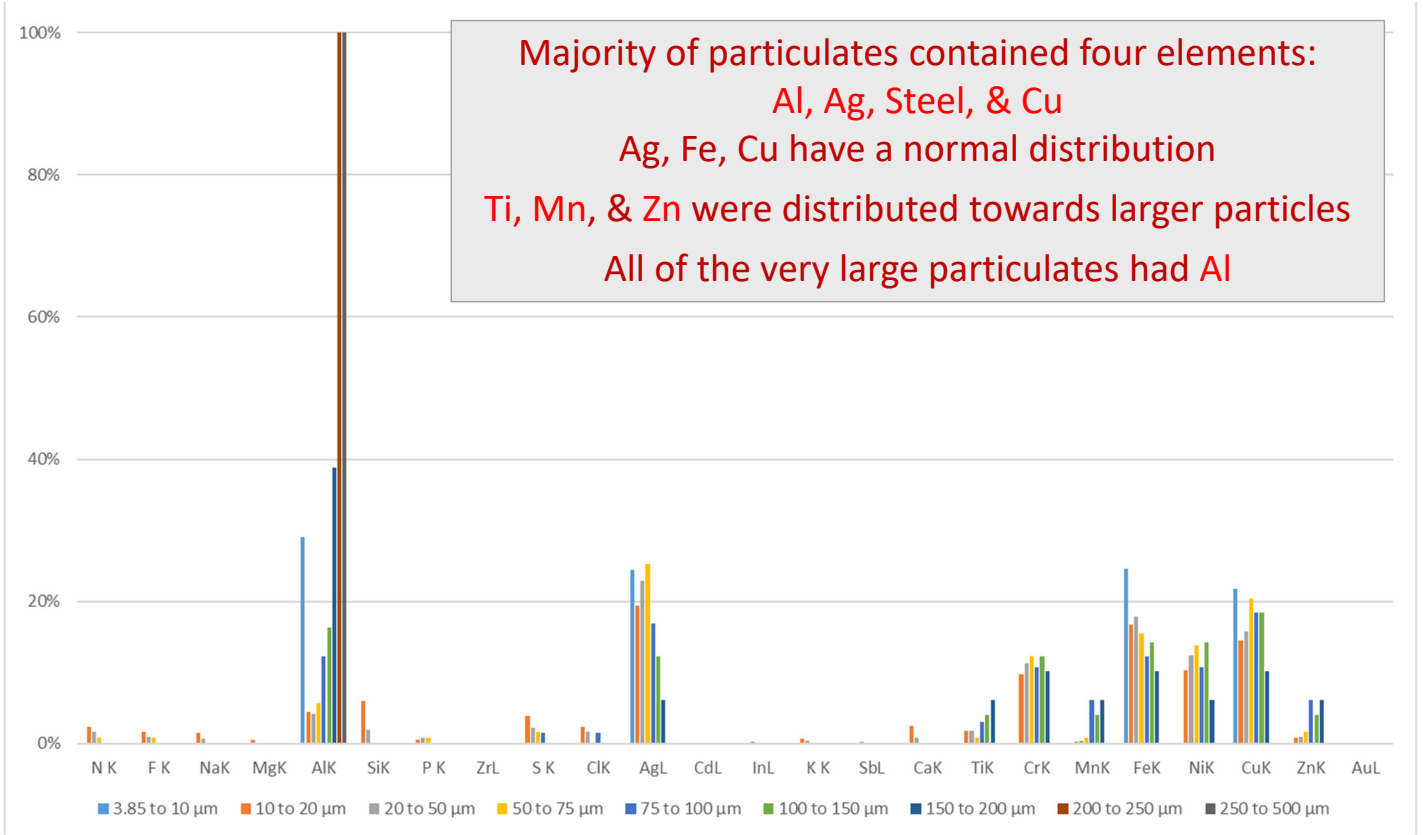
Auto-detected particulates
from the 58 cavity samples
Size Range
3.9 – 480 µm



C100-6 NL23 205× Particulate Results - Material and size distribution

Percentage of Particulates with Element At% over the threshold of 5% that were in selected Size Range

For 24 tracked elements

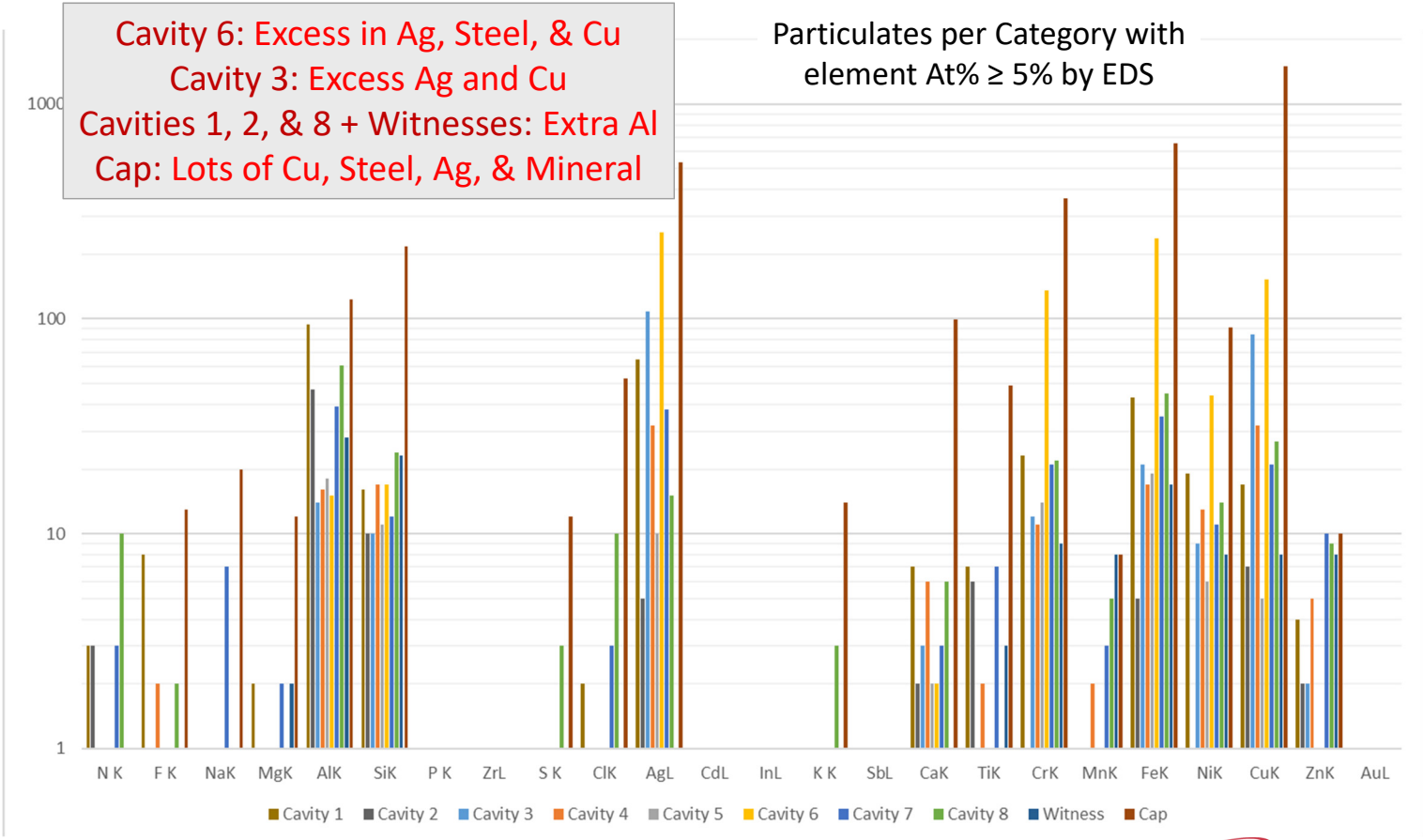


C100-6 NL23 205× Particulate Results – Content varied by cavity!

Cavity 6: Excess in Ag, Steel, & Cu
Cavity 3: Excess Ag and Cu
Cavities 1, 2, & 8 + Witnesses: Extra Al
Cap: Lots of Cu, Steel, Ag, & Mineral

Number of Found "Particulates"
Cavity 1 = 2421
Cavity 2 = 673
Cavity 3 = 2551
Cavity 4 = 439
Cavity 5 = 436
Cavity 6 = 2321
Cavity 7 = 1979
Cavity 8 = 1280

Witness = 527

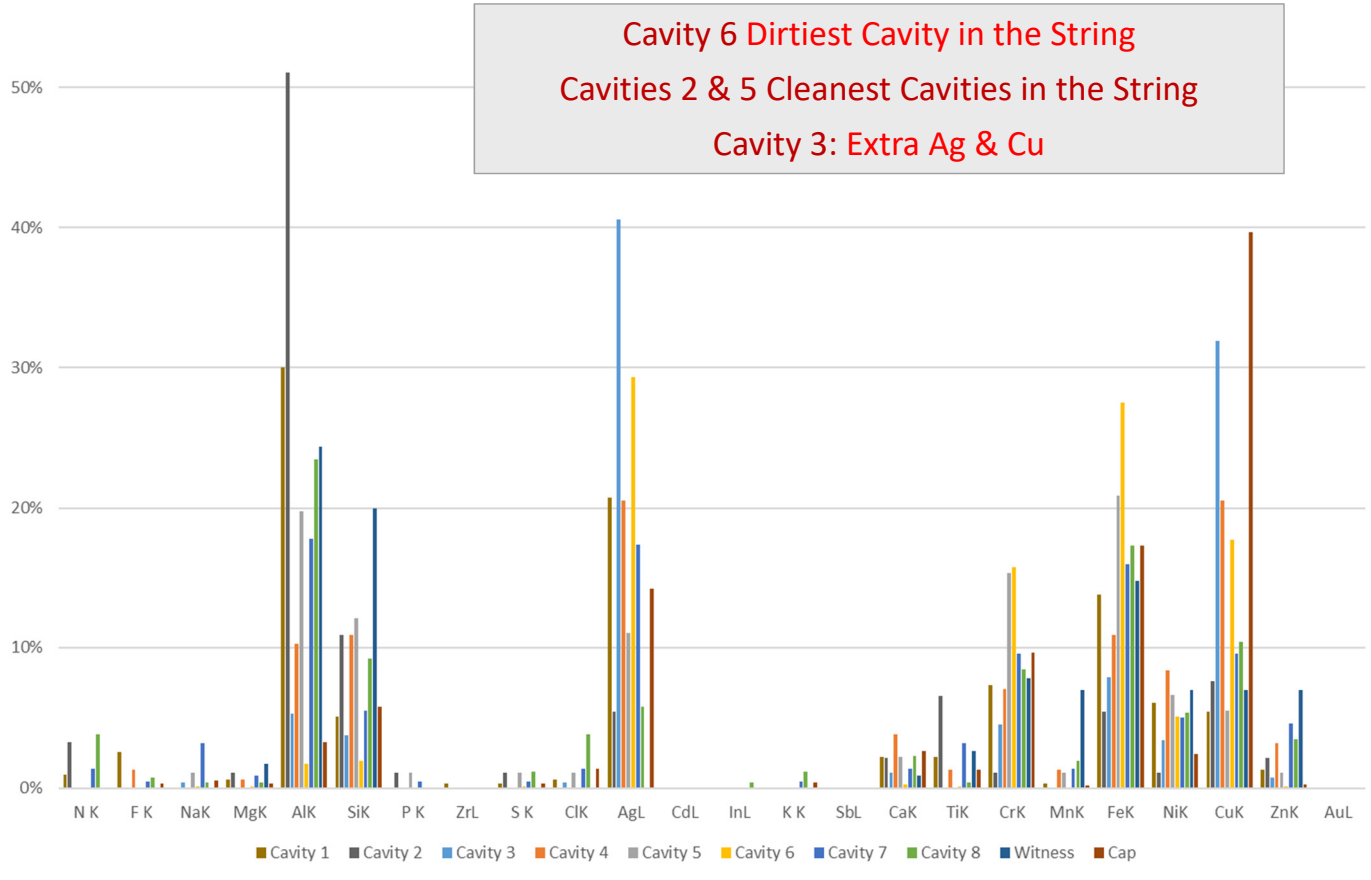


C100-6 NL23 205× Particulate Results – Content varied by cavity

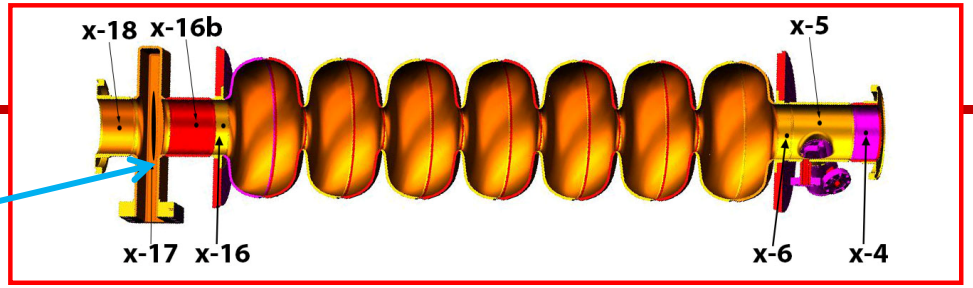
% Particle per Element
For At% Threshold of 5%

<u>Occurrences over Threshold</u>
Cavity 1 = 313
Cavity 2 = 92
Cavity 3 = 266
Cavity 4 = 156
Cavity 5 = 91
Cavity 6 = 862
Cavity 7 = 219
Cavity 8 = 260

Witness = 115

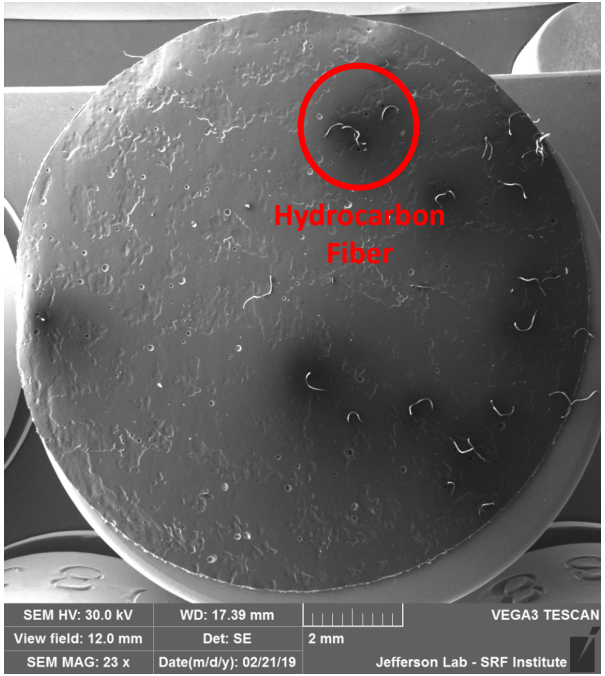


Fibers found in Waveguide Coupler

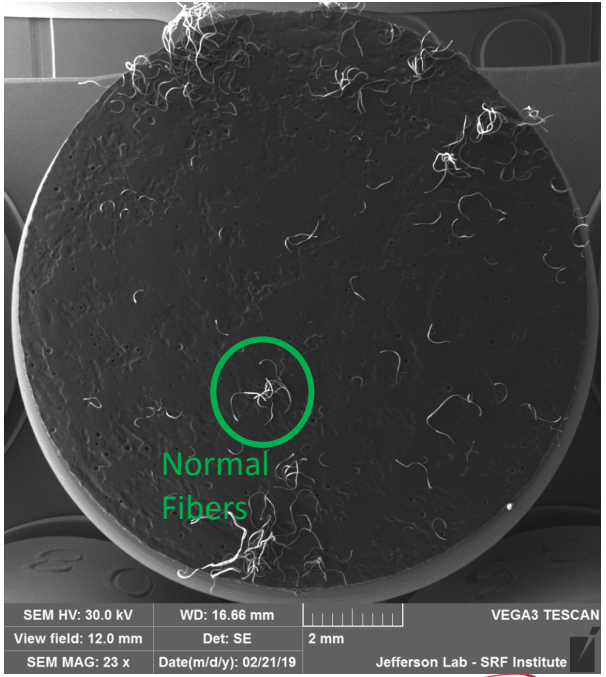


- **Waveguide samples have higher occurrence and density fibers.**
- Two different collection methods:
  - Waveguide Shaken
  - Contact Swabbing
- Fibers likely came from attached Cu/plated transition waveguides.
- Some fibers from “shaken” sample show hydrocarbon halo.

S0831 – Waveguide Shaken

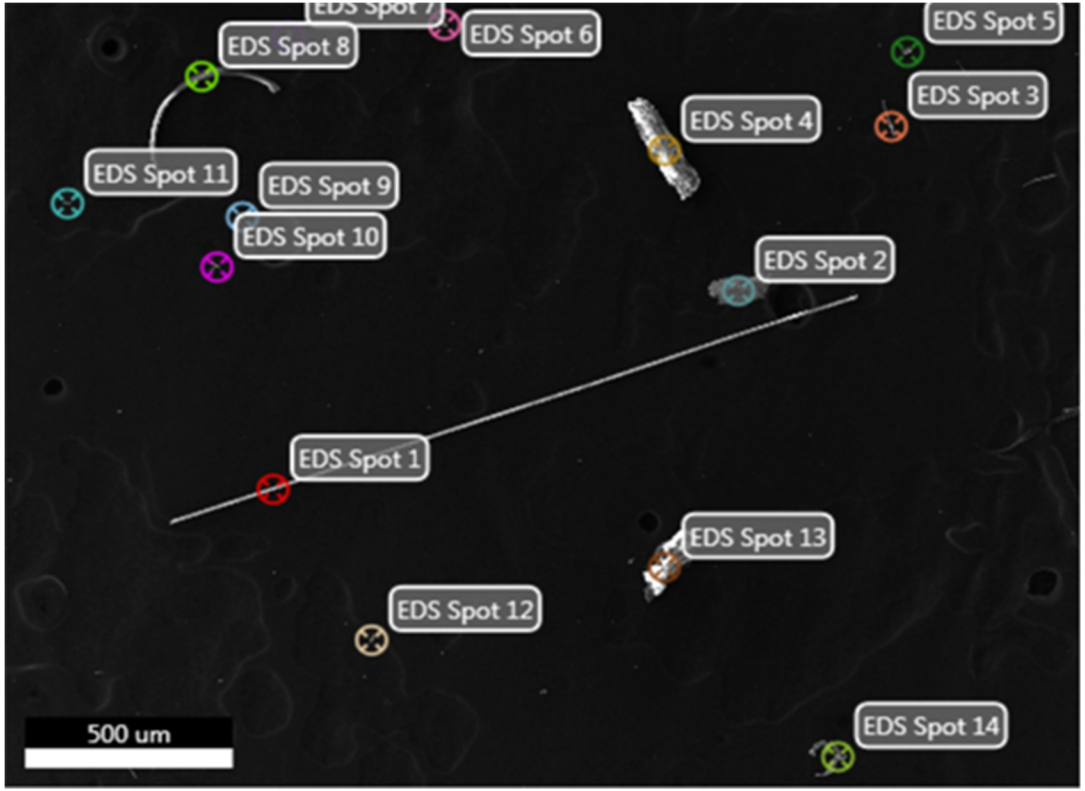


S0828 – Waveguide Bottom C7-17



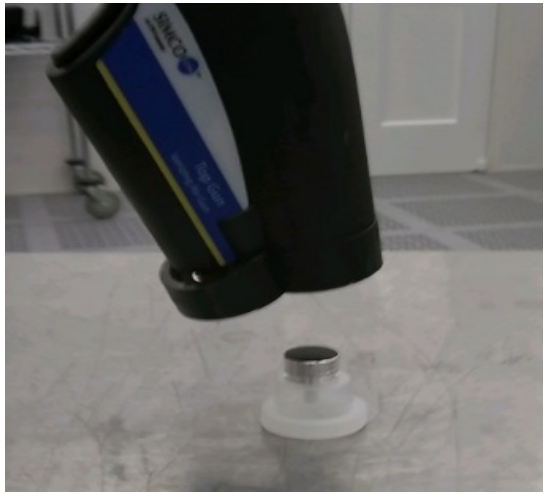
C100-6 NL23 Representative Particulates

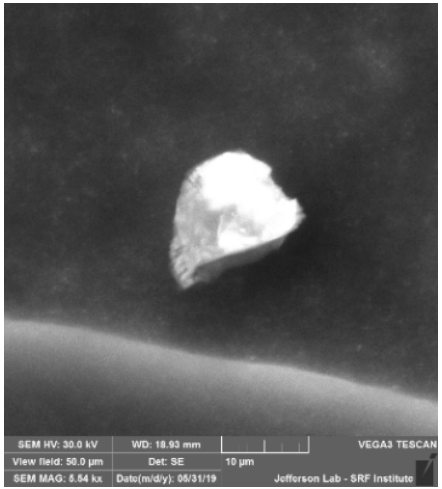
- **Fiber Glass** – EDS Spot 1
- **Copper particle with nickel, silver, steel and chlorine** – EDS Spot 2
- **Steel** with **copper** & **MoS** – EDS Spot 3
- **Titanium** with **aluminum** and **Iron** – EDS Spot 4
- **Silver** with **sulfur** and **chlorine** – EDS Spot 5
- **Fiber** – EDS Spot 8
- **Mineral** – EDS Spot 13



Identifying Specific Particulate Contamination - Quality control checks

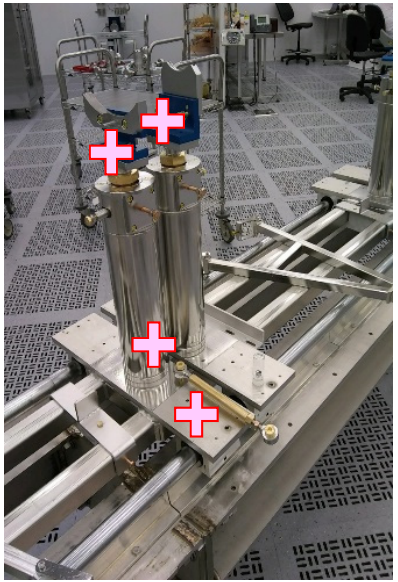
- Check of ionized N_2 blow-off gun
  - 1 min sustained + 5 1-sec bursts directed at fresh sample
  - 12 particulates 7–20 μm all having the same EDS spectrum dominated by **silicon**, with traces of Na, Mg, Al, K, Ca, Ti & Fe.
  - Seems to represent breakdown fragments of the in-line N_2 filter media

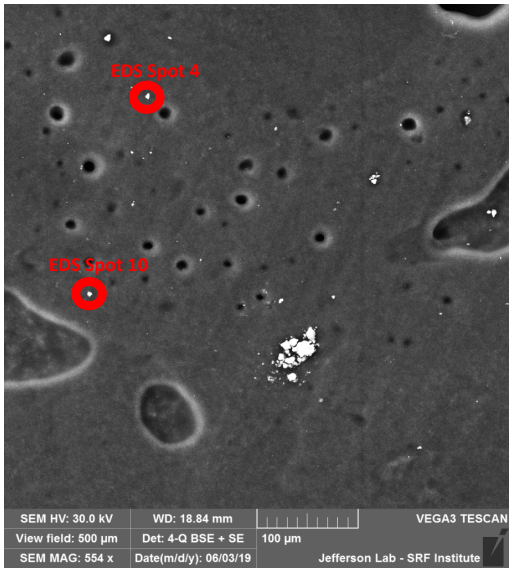




Identifying Specific Particulate Contamination - Quality control checks

- Spot check of LCLS-II string assembly rail – “Cleaned and ready”
  - Carbon tape sample was touched to the base, pedestal, and top mounting hardware.
  - 16 different areas were selected for characterization on the resulting spindle.
  - **Materials 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	Mineral	Calcite
EDS Spot 2	Metallic	Copper Zinc
EDS Spot 3	Salt	Copper Zinc Phosphate Sulfate
EDS Spot 4	Salt	Aluminum Sulfate
EDS Spot 5	Metallic	Iron
EDS Spot 6	Organic	Organic
EDS Spot 7	Metallic	Aluminum
EDS Spot 8	Metallic	Aluminum
EDS Spot 9	Metallic	Copper Zinc
EDS Spot 10	Salt	Aluminum Sulfate
EDS Spot 11	Metallic	Copper Zinc
EDS Spot 12	Mineral	Silicate
EDS Spot 13	Mineral	Silicate
EDS Spot 14	Mineral	Silicate
EDS Spot 15	Mineral	Silicate
EDS Spot 16	Mineral	Silicate

Identifying Specific Particulate Contamination – Process development

- Standard spindle samples were used to investigate particulates associated with LCLS-II copper-plated bellows flexing, cleaning, and assembly.
- This work provided valuable feedback concerning the effectiveness of cleaning procedures and the sustained integrity of the copper plating with bellows flexure.

Identifying Specific Particulate Contamination - Summary

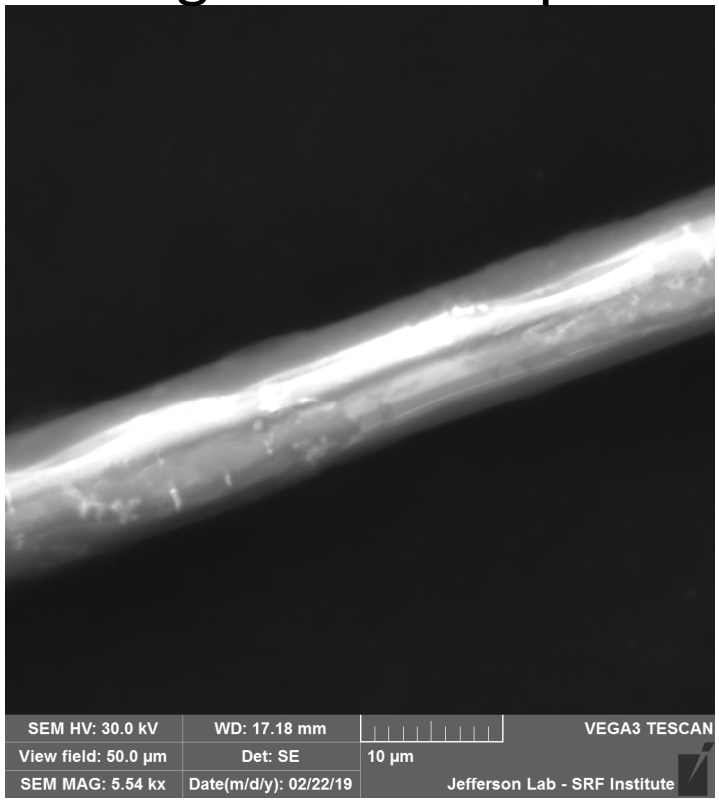
- Particulates found in CEBAF are much larger than anticipated.
- While past efforts to control particulate contamination considered the battle to be control of particulates smaller than 5–10 μm, in reality, beamline elements, including cavities in cryomodules, were found to contain a generous number 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.
- 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 have begun using this standardized particulate assessment system for QC and feedback for procedure refinement.

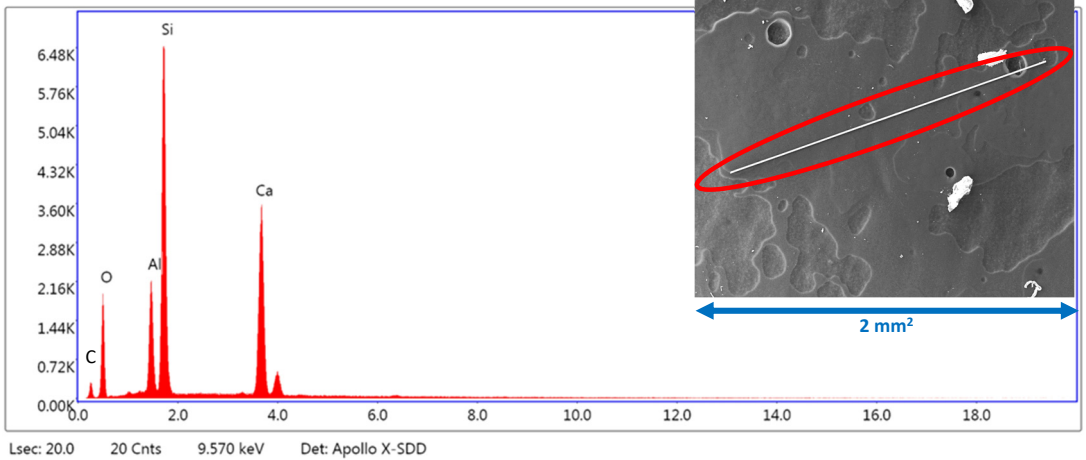
Identifying Specific Particulate Contamination

- Brief **Gallery of horrors** – found on beamline, outboard of C100 cryomodule gate valves

C100-6 NL23 Representative Particles

Fiberglass – EDS Spot 1

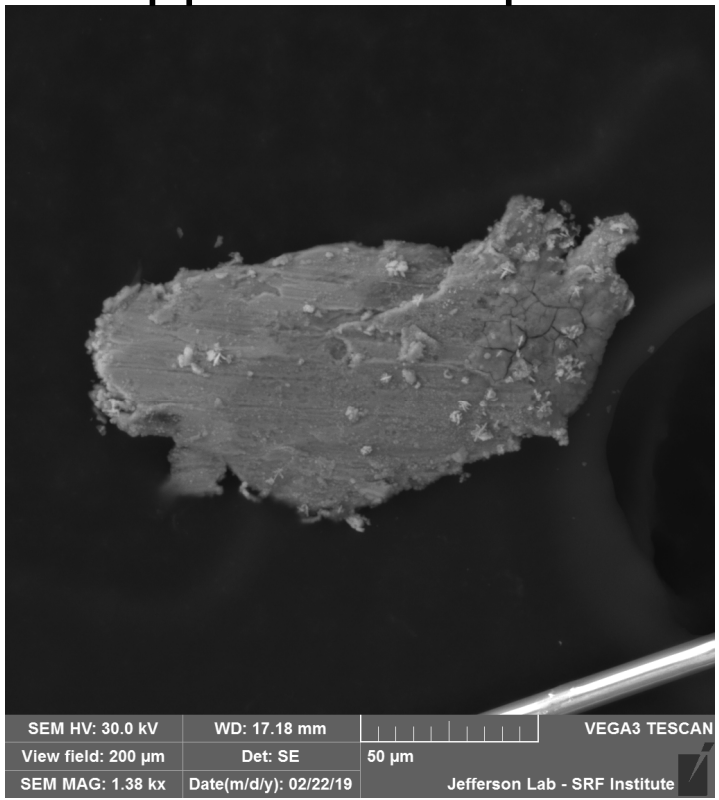


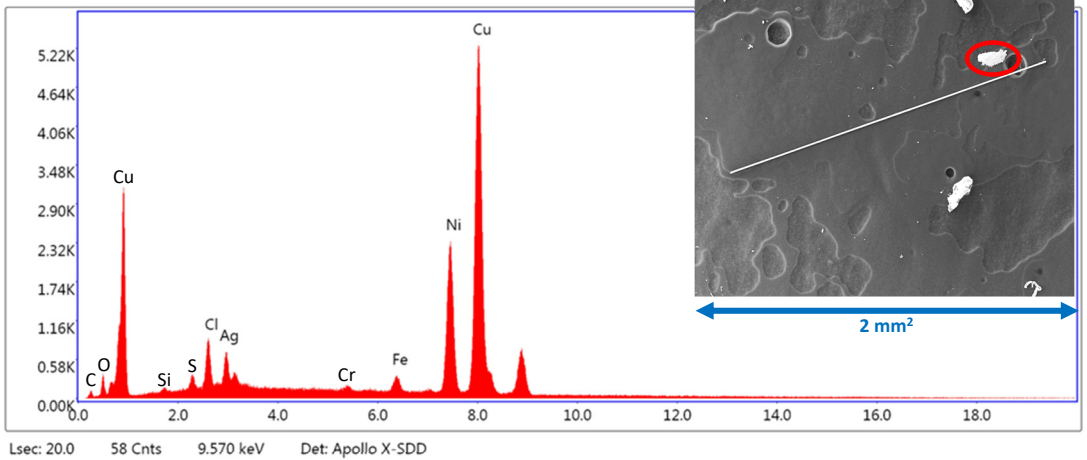


Element	Weight %	Atomic %	Net Int.	Error %
O K	48.47	64.48	641.52	10.01
AlK	8.81	6.95	909.97	6.72
SiK	25.93	19.65	3004.57	5.79
CaK	16.80	8.92	2043.97	2.58

C100-6 NL23 Representative Particles

Copper – EDS Spot 2

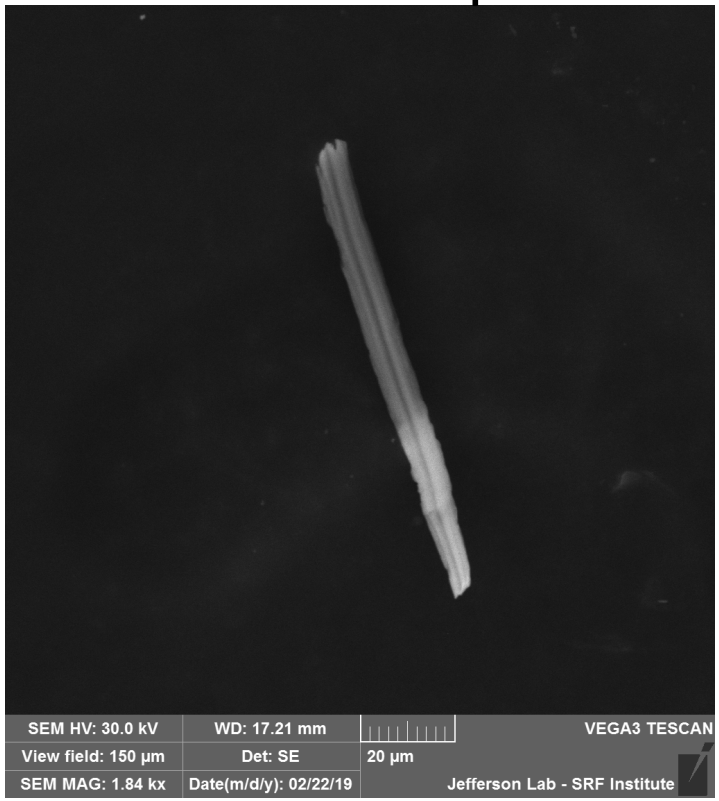


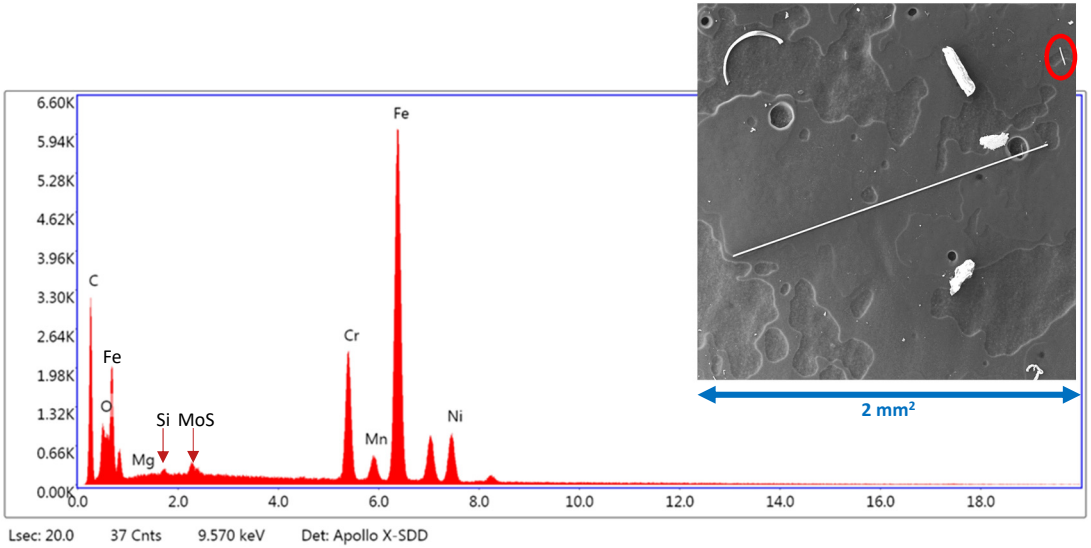


Element	Weight %	Atomic %	Net Int.	Error %
ClK	3.45	5.99	346.31	10.43
AgL	4.47	2.55	262.51	8.02
FeK	2.01	2.22	196.12	7.06
NiK	22.69	23.83	1768.73	2.15
CuK	67.39	65.40	4235.29	1.45

C100-6 NL23 Representative Particles

Steel – EDS Spot 3

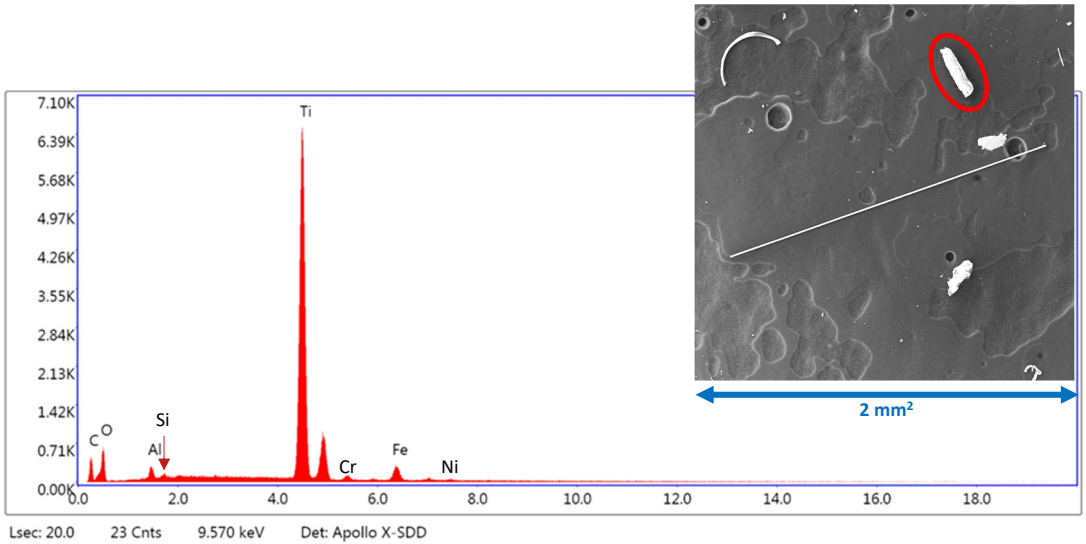




Element	Weight %	Atomic %	Net Int.	Error %
C K	47.67	69.36	1065.99	7.56
O K	18.13	19.80	489.58	14.27
MgK	0.12	0.09	12.66	69.74
CrK	5.89	1.98	1496.95	2.52
MnK	0.64	0.20	142.62	10.48
FeK	23.41	7.33	4440.19	1.42
NiK	4.14	1.23	622.00	3.17

C100-6 NL23 Representative Particles

Titanium – EDS Spot 4

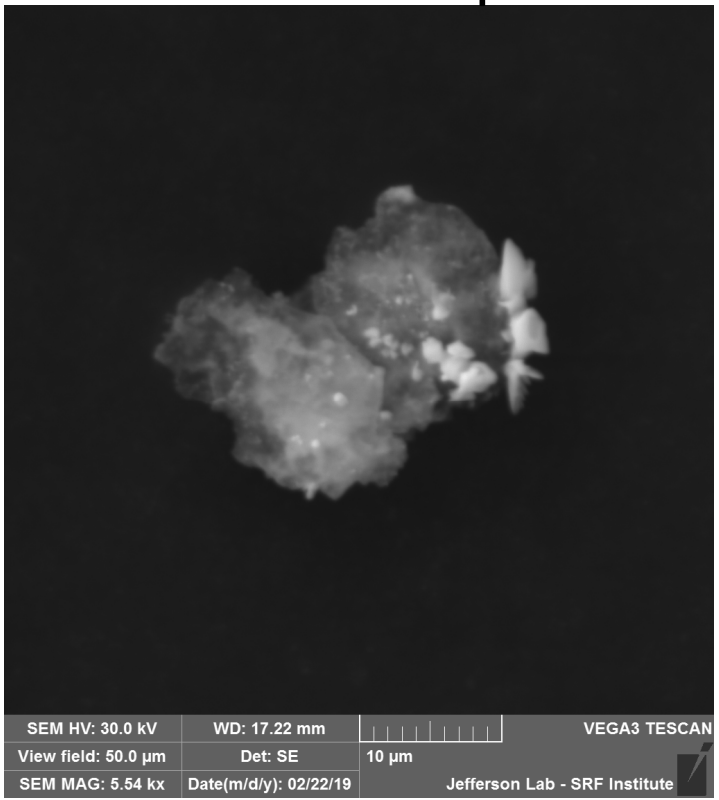


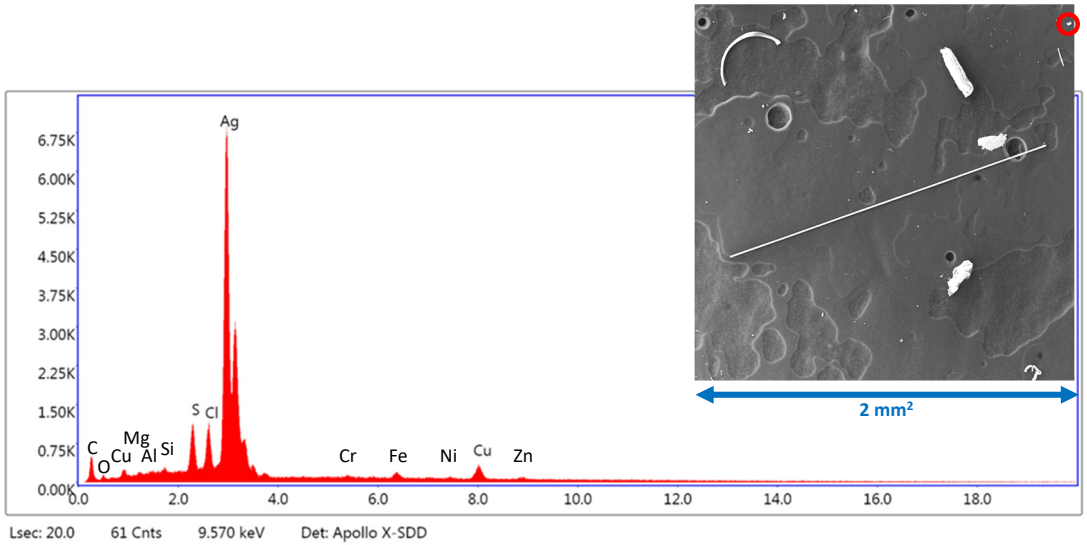
Element	Weight %	Atomic %	Net Int.	Error %
C K	18.49	32.07	151.75	8.83
O K	37.13	48.36	215.66	11.52
AlK	1.28	0.99	77.38	13.12
TiK	40.50	17.61	4072.60	1.44
FeK	2.60	0.97	156.60	8.44

One of the largest Titanium particles found to date with atypical appearance.

C100-6 NL23 Representative Particles

Silver – EDS Spot 5



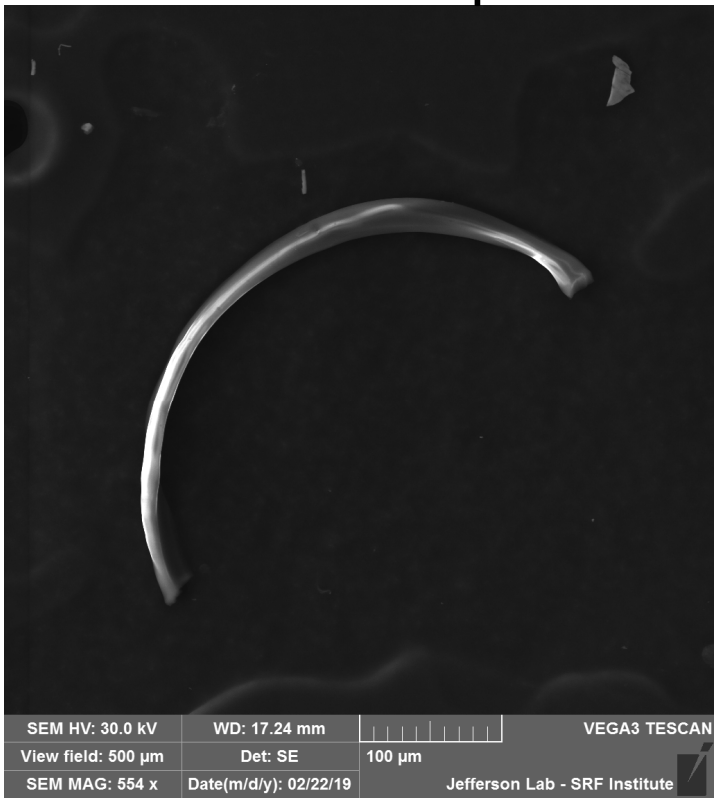


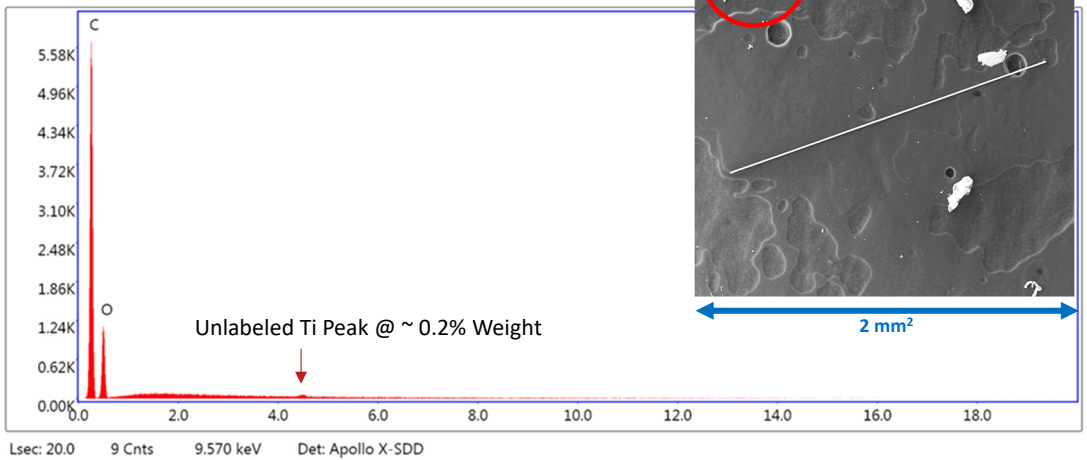
Element	Weight %	Atomic %	Net Int.	Error %
S K	5.63	15.12	475.17	6.99
ClK	4.17	10.14	359.62	8.19
AgL	85.36	68.18	3779.07	1.80
CuK	4.84	6.56	199.26	7.73

Trace Mineral and Steel

C100-6 NL23 Representative Particles

Fiber – EDS Spot 8





Element	Weight %	Atomic %	Net Int.	Error %
C K	70.02	75.68	1828.88	4.31
O K	29.98	24.32	391.20	11.09

Trace Ti @ ~ 4.2 keV