

DebrisSat Laboratory Analyses

January 5, 2015

Paul M. Adams¹, Zachary Lingley², Nathan Presser², and Gouri Radhakrishnan¹

¹Space Materials Laboratory, Physical Sciences Laboratories

²Electronics and Photonics Laboratory, Physical Sciences Laboratories

Prepared for:

Space and Missile Systems Center
Air Force Space Command
483 N. Aviation Blvd.
El Segundo, CA 90245-2808

Contract No. FA8802-14-C-0001

Authorized by: Space Systems Group

Distribution Statement A: Approved for public release; distribution unlimited.

Report Documentation Page

Form Approved
OMB No. 0704-0188

Public reporting burden for the collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to a penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.

1. REPORT DATE 05 JAN 2015	2. REPORT TYPE Final	3. DATES COVERED -			
4. TITLE AND SUBTITLE DebriSat Laboratory Analyses		5a. CONTRACT NUMBER FA8802-14-C-0001			
		5b. GRANT NUMBER			
		5c. PROGRAM ELEMENT NUMBER			
6. AUTHOR(S) Paul M. Adams, Zachary Lingley, Nathan Presser, and Gouri Radhakrishnan		5d. PROJECT NUMBER			
		5e. TASK NUMBER			
		5f. WORK UNIT NUMBER			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) The Aerospace Corporation 2310 E. El Segundo, CA 90245-4609		8. PERFORMING ORGANIZATION REPORT NUMBER TOR-2015-00876			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Space and Missile Systems Center Air Force Space Command 483 N. Aviation Blvd. El Segundo, CA 90245-2808		10. SPONSOR/MONITOR'S ACRONYM(S) SMC			
		11. SPONSOR/MONITOR'S REPORT NUMBER(S)			
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release, distribution unlimited					
13. SUPPLEMENTARY NOTES The original document contains color images.					
14. ABSTRACT The DebriSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite. The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then. DebriSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace. Tests were conducted at the Arnold Engineering Development Complex Range G Two-Stage Light Gas Gun Facility. The 50 kg target was a constructed by the University of Florida from materials representative of a modern LEO satellite. The Aerospace Concept Design Center advised on the selection of materials for various subsystems.					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 106	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

Abstract

The DebrisSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite. The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then. DebrisSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace. Tests were conducted at the Arnold Engineering Development Complex Range G Two-Stage Light Gas Gun Facility. The 50 kg target was constructed by the University of Florida from materials representative of a modern LEO satellite. The Aerospace Concept Design Center advised on the selection of materials for various subsystems. The test chamber was lined with “soft catch” foam panels to trap fragments for size distribution analysis. A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis. Aerospace also placed SEM stub witness plates into the soft catch panels for post test retrieval and analysis. The test was conducted with a pressure of 1-2 Torr of air and used a ~600 gram projectile with a nominal velocity of 7 km/s.

The SEM stubs, witness plate assembly and DebrisSat fragments were covered with black soot and were contaminated with soft catch foam fragments. The SEM stubs were also covered with a thin film of condensed soft catch vapor, similar to that seen with the Debris-LV test which also used soft catch foam panels. Deposits on the SEM stubs and witness plate assembly are predominantly carbon and consist of agglomerates of nano carbonaceous material. These deposits are primarily from the soft catch (similar to Debris-LV) though the C-C composite honeycomb face sheets and MLI are also possible sources. Disordered graphitic carbon is present based on Raman spectra and TEM lattice fringe images. Graphitic carbon was also seen in Debris-LV deposits even though there were no carbon containing materials in the target. The witness plate assembly was covered in a black layer of loose “soot”, even under the protective Whipple plates. Removal of the loose material by rinsing with isopropyl alcohol revealed a thin adherent coating on one side of the support posts indicating early directional deposition from DebrisSat. The coating was carbonaceous (disordered graphite) with nano metal droplets. Fluorine from Teflon wire insulation was also common in the SEM stub and witness plates deposits. Nano droplets of metallic materials (Al, Fe, Cu, Zn, Ge) were also present indicating melting as a result of the impact. Solidified molten metal droplets were also seen in Pre Preshot and Debris-LV debris. Aluminum was from the Al honeycomb, nadir and zenith panels, structural core and COPV liner. Aluminum oxide particles were also present. Iron was from stainless steel tubing and solenoids. Germanium was from the solar cells and copper was from wiring and solenoids. The source of the zinc has not been identified. The solidified molten nano metal droplets are crystalline based on TEM lattice fringes and consist of only a few crystallites.

The witness plates show a significant decrease in reflectance (95% to 6%). Soft catch contamination was seen in the LWIR reflectance spectra of DebrisSat fragments, SEM stubs and witness plate. As a result, it was not possible to get a clean spectrum of the debris generated by the hypervelocity impact. An additional “oxide” band, which may be from a form of aluminum oxide, was seen on some samples.

Acknowledgements

DebrisSat Team Members:

J.-C. Liou: NASA Space Debris Program Office, NASA JSC

AEDC Range G Light Gas Gun Staff

Charles Griffice: Aerospace

Marlon Sorge: Aerospace

Patti Sheaffer: Aerospace

Financial Support:

Charles Griffice and Tom Huynh

Discussions with:

Patti Sheaffer

Charles Griffice

UV-VIS-NIR Spectroscopy

Dianna Alaán

Aerospace Technical Operating Report No.
TOR-2015-00876

Debrisat Laboratory Analyses

5 January, 2015

Paul M. Adams¹, Zachary Lingley², Nathan Presser²
and Gouri Radhakrishnan¹

¹Materials Processing Department
Space Materials Laboratory

²Microelectronics Technology Department
Electronics and Photonics Laboratory

Physical Sciences Laboratories

Introduction

- The DebrisSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite.
 - The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then.
 - In 2009 a Cosmos 2251 upper stage collided with an Iridium 33 satellite.
 - Produced 2000+ trackable fragments (>10 cm).
 - 8 other known collisions, some only known long after occurrence.
- DebrisSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace.
- Tests were conducted at the Arnold Engineering Development Complex, Tullahoma, Tennessee.
 - Two-Stage Light Gas Gun Facility - Range G.
 - Largest such facility in the United States.
 - All tests used a ~600 gram projectile with a nominal velocity of 7 km/s.



Introduction (cont.)

- Two trial tests were conducted prior to DebrisSat.
 - Pre Preshot. February 2014
 - Debris-LV (Pre Shot). 1 April 2014
- Debris-Sat was conducted 15 April 2014
 - The 50 kg target was constructed by the University of Florida from materials representative of a modern LEO satellite.
 - Aerospace Concept Design Center advised on selection of materials for various subsystems.
 - Test chamber was lined with “soft catch” foam panels to trap fragments for size distribution analysis.
 - A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis.
 - Aerospace also placed SEM stub witness plates into soft catch for post test retrieval and analysis.
 - Test conducted with a pressure of 1-2 Torr of air.



Introduction (cont.)

- Documentation to date.
 - Aerospace TOR-2014-03201, Time-resolved Spectroscopy of Hypervelocity Impact Flash on DebrisSat, Gouri Radhakrishnan.
 - Aerospace ATM-2014-03659, DebrisSat Hypervelocity Impact Fragmentation Modeling, Naoki Hemmi.

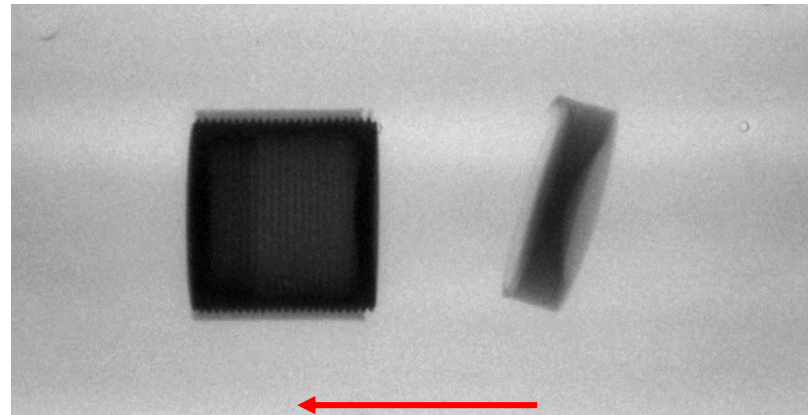


- Background
 - “Darkening” of satellites has been observed as a result of suspected hypervelocity impacts.
 - The material and processes responsible for the darkening is unknown.
- Objectives
 - Materials collected on witness plates in the DebrisSat test were analyzed in order to identify the source and conditions responsible for the darkening.
 - UV-VIS-NIR-LWIR reflectance spectra were measured of post test debris for comparison with pre test sources to determine the spectral signature of material generated by a hypervelocity impact.
 - Possibly determine if a hypervelocity impact occurred based on remotely sensed spectra?
 - Can the source be identified – does it have a unique signature?



Projectile

Images by AEDC

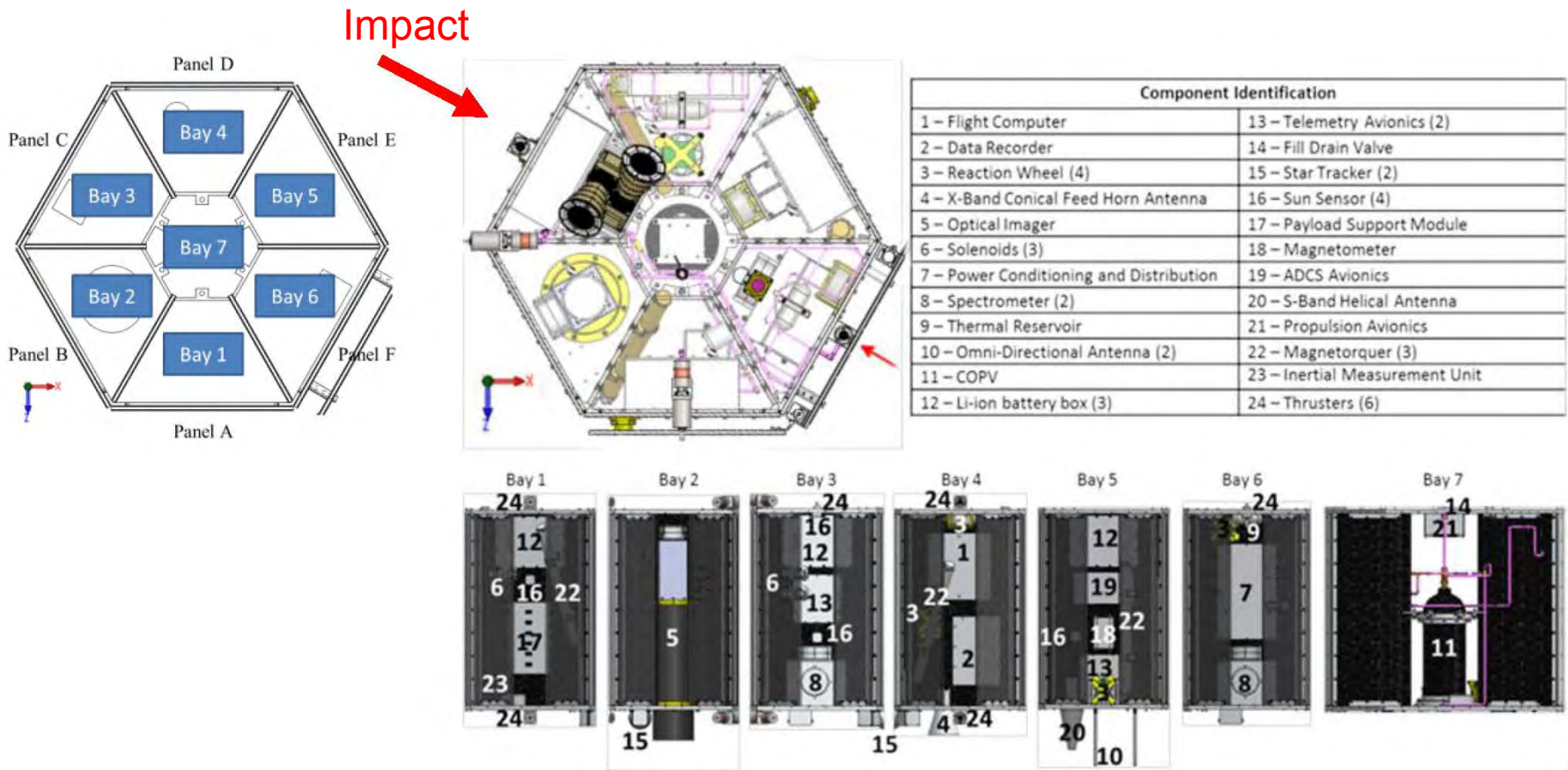


Flash X-ray of projectile in flight

- Constructed from three pieces: Outer Nylon shell (sabot) with 2 part hollow aluminum insert.
- ~600 grams, 8.6 cm diameter X 10.3 cm long – size of a soup can.
- Velocity ~ 6.8-6.9 km/s.
- The Nylon base separated from the Nylon-aluminum body during flight.



DebrisSat Construction



Projectile impacted normal to Bay 3

Figures from U. of Florida

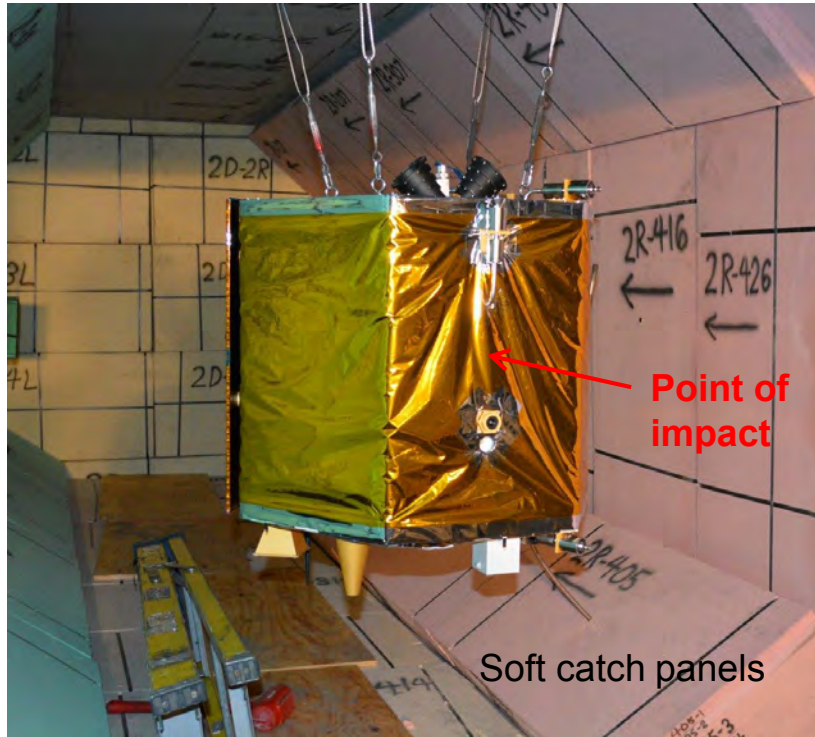


Materials Present on Debrisat

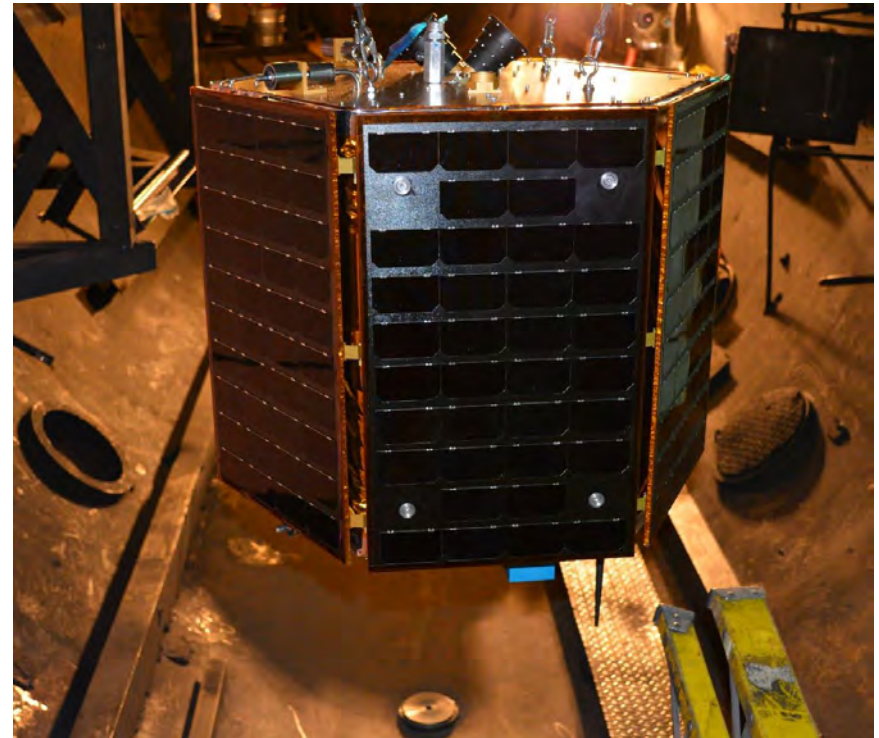
- Nadir and zenith panels: Aluminum 6061
- Face panels and structural ribs: Aluminum 5052 honeycomb with carbon fiber/epoxy face sheets
- COPV tank pressurized to 2 torr with air
- Optics : SiO_2 , sapphire
- Solenoids : Cu
- Stainless Steel (316, 304) : Fe, Cr, Ni
- Printed circuit boards (electronics hardware)
- Li-ion batteries: Cu-polyimide (no Li or electrolyte)
- Solar cells: Ge substrate with GaAs/InP/GaInP
- Kevlar, polyurethane, Mylar, Kapton



Installed in Chamber: Pre test



Looking down range.
DebrisSat is covered with
multi layer insulation (MLI).



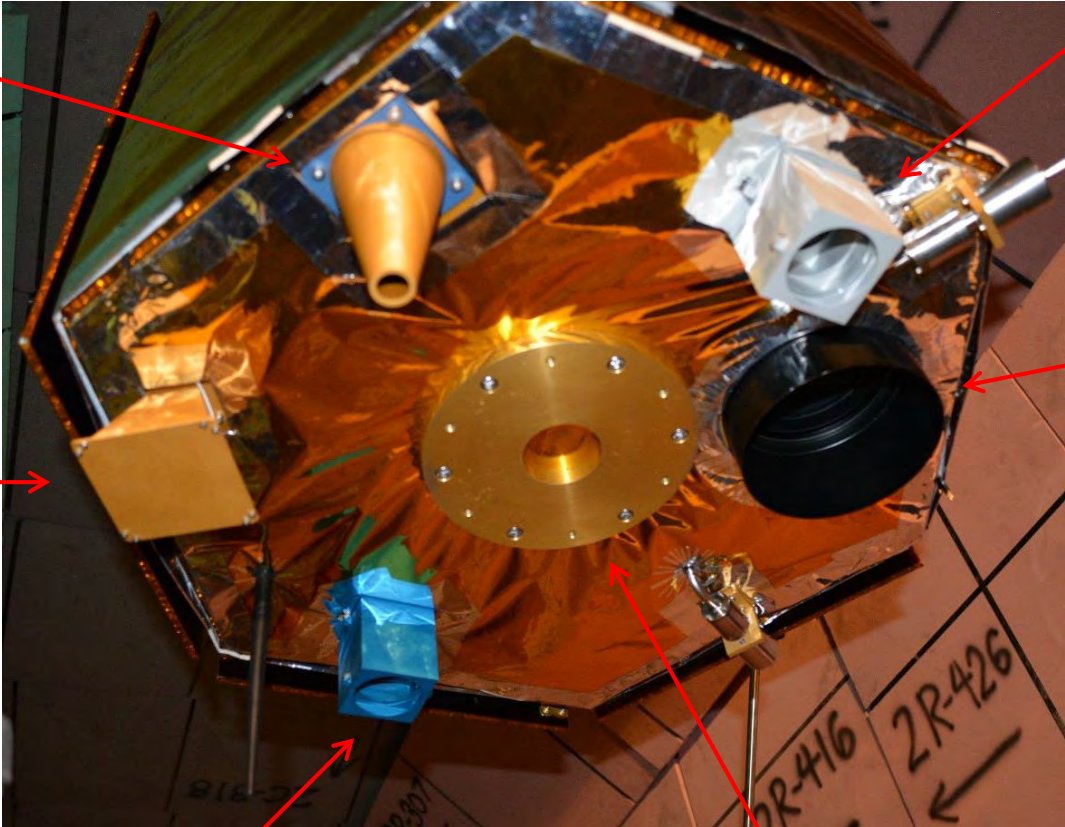
Looking up range.
Solar panels - undeployed



Nadir / Under Side

S-band
cone antenna

Spectrometer
Baffle (Bay 3)



X-band
horn antenna

Optical imager
sun shade

Spectrometer
Baffle (Bay 6)

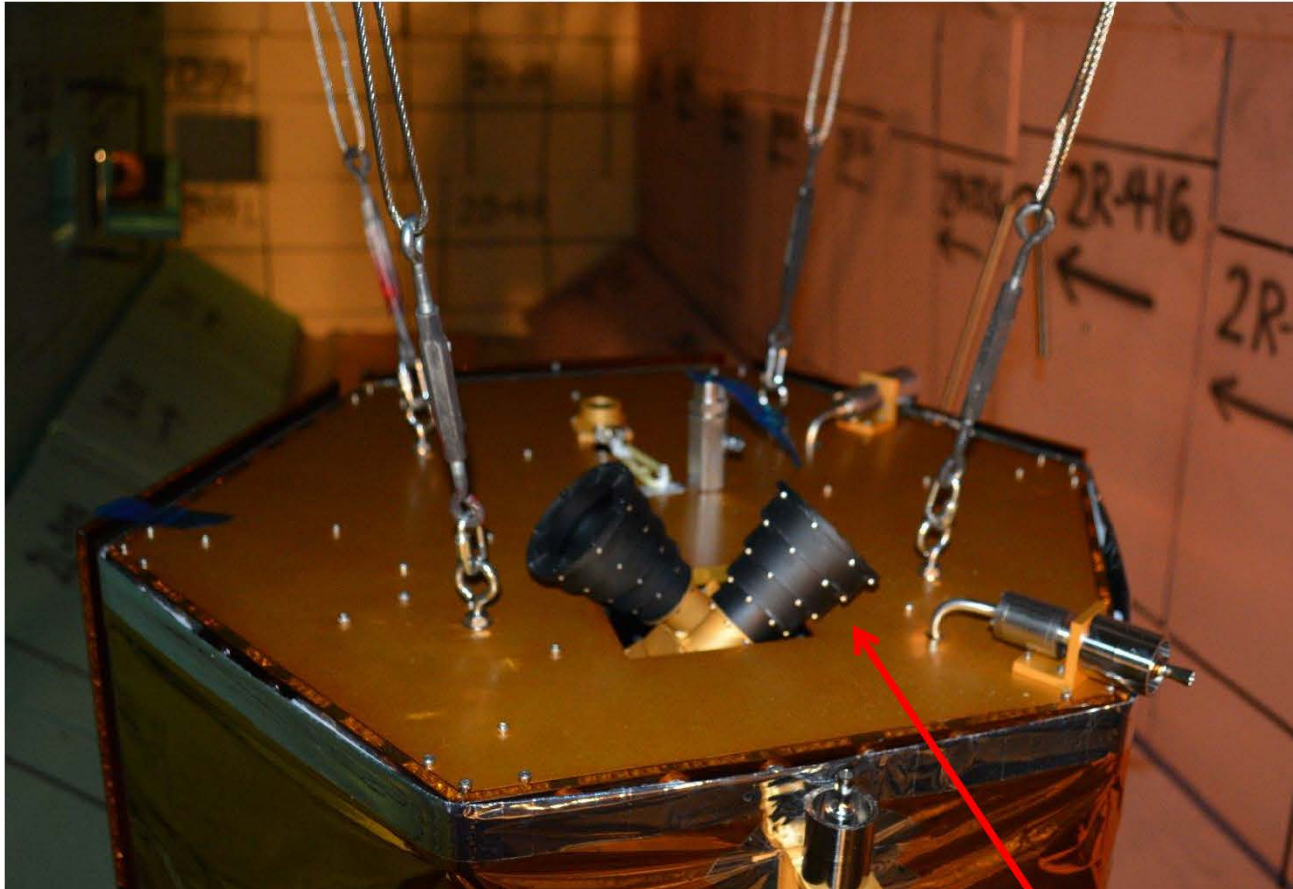
Adapter plate

Aluminum components had various types of anodized finishes (clear, black, blue, gold) to aid in post test fragment identification.



SBU Marking

Zenith / Top Side



Star trackers



Witness Plate: Pre Test

Whipple Plates



Witness Plate Samples:

Direct Exposure

(4) 1" fused silica

(1) 1" Z-93 painted Al

(1) 1" Aluminum

Multi layer insulation (not shown)

Protected Under Whipple Plates

(2) 1" fused silica

(1) 1" Z-93 painted Al

(1) 1" Aluminum

(1) 1" NaCl

(1) Cu sheet

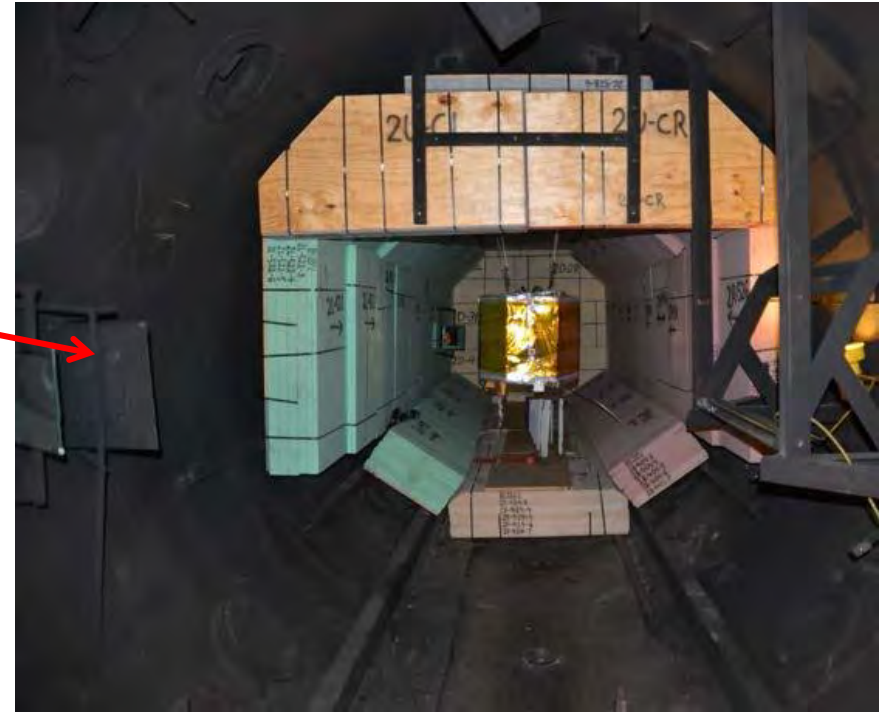
Ge ATR crystal (for FTIR)

Solar cell

Witness plates located in same position in chamber as Debris-LV.
~3 meters up range of DebrisSat.



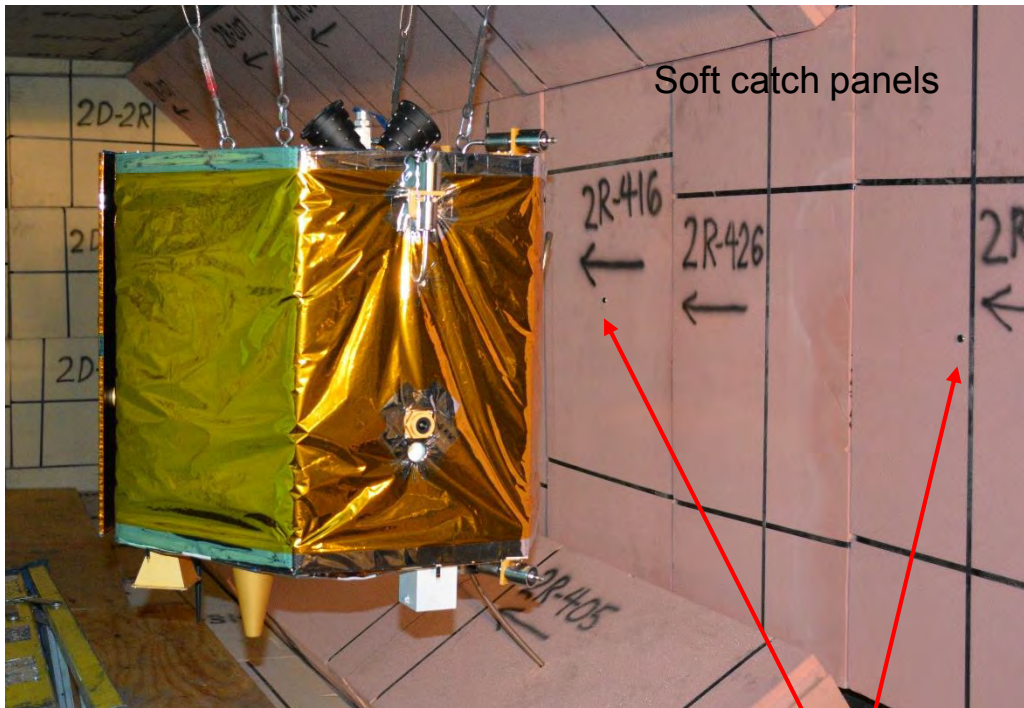
Witness Plates Mounted in Chamber



Plates mounted about 3 meters up range of DebrisSat

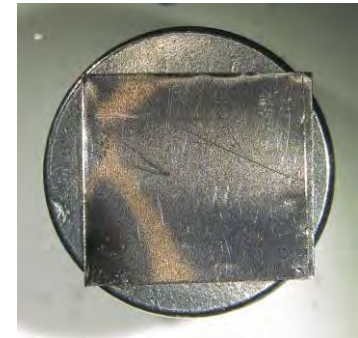


SEM Stub Witness Plates Placed into Soft Catch Panels



Soft catch panels

12.5 mm Al SEM stubs



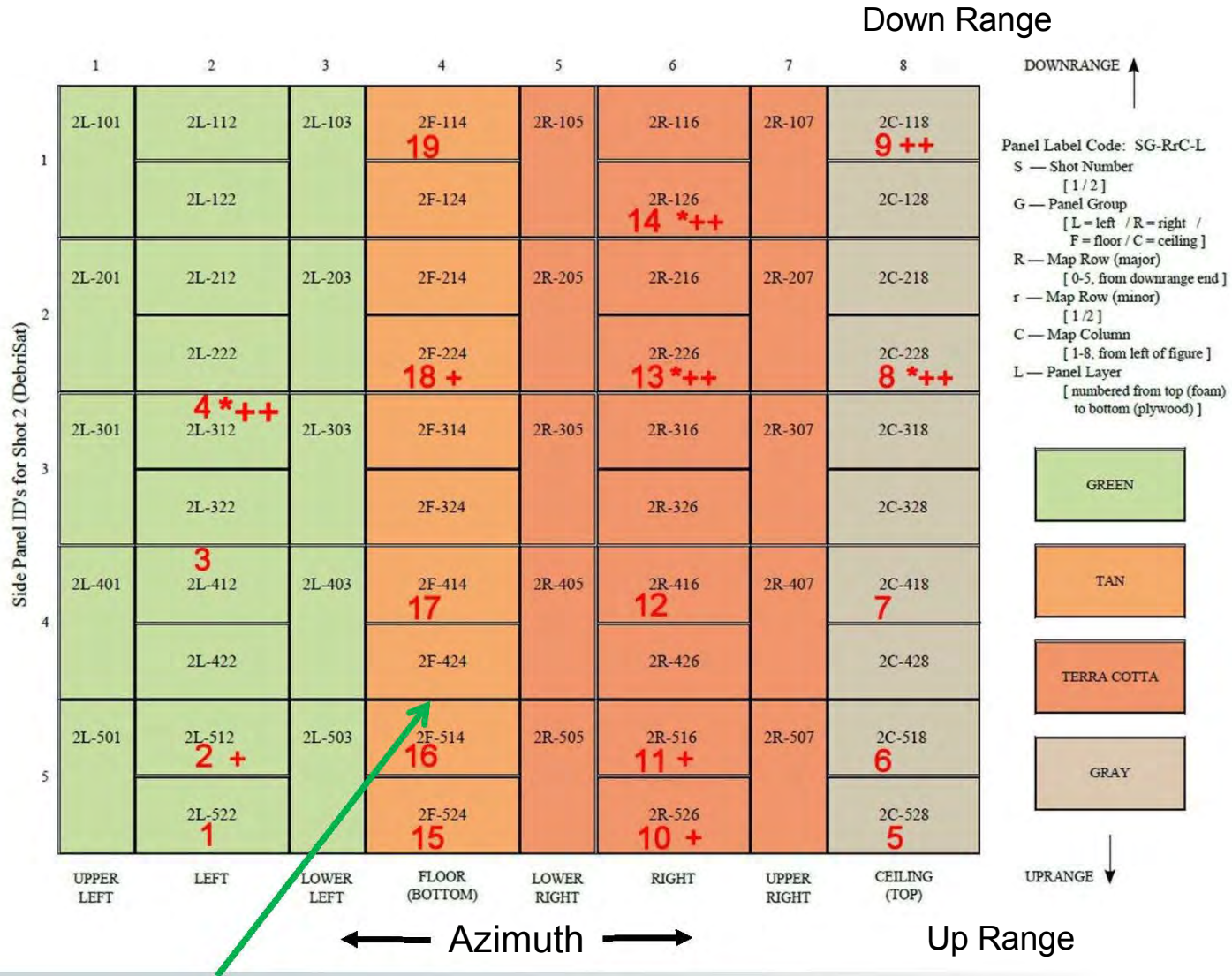
with Ta sheet

SEM stubs

- 24 Aluminum SEM stub witness plates (12.5 mm dia) placed in soft catch with RTV adhesive.
- Tantalum sheet (9 mm x 9 mm) epoxied to front surface in order to distinguish Al debris from stub.
- Identified on back with engraved numeral.
- 11 stubs recovered (+); 5 with Ta sheet present (++), 6 in place (*)
- Remaining stubs embedded in soft catch. – to be recovered at U. of F.



SBU Marking Location of SEM Stubs (red) on Soft Catch Panels



Nominal location of DebrisSat is above 2F-424 and 2F-514

stubs recovered (+), with Ta sheet present (++), in place (*)

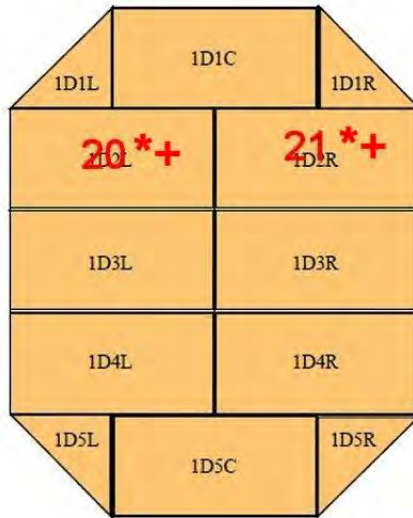
Intact recovered stubs with Ta (*++) tended to be furthest from impact site



Location of SEM Stubs (red) on Soft Catch Panels

Down Range
End Panels

End Panel ID's for Shot 1 (pre-test)



DOWNRANGE
(looking downrange)

Panel Label Code: SERC-L
 S — ShotNumber [1/2]
 E — PanelGroup [D=downrange]
 R — MapRow [1-4, from top of figure]
 C — MapColumn [L=left / R=right / C=center]
 L — PanelLayer [numbered from top (foam) to bottom (plywood)]



Upper Up Range
End Panels



Lower Up Range
End Panels

UPRANGE
(looking downrange)

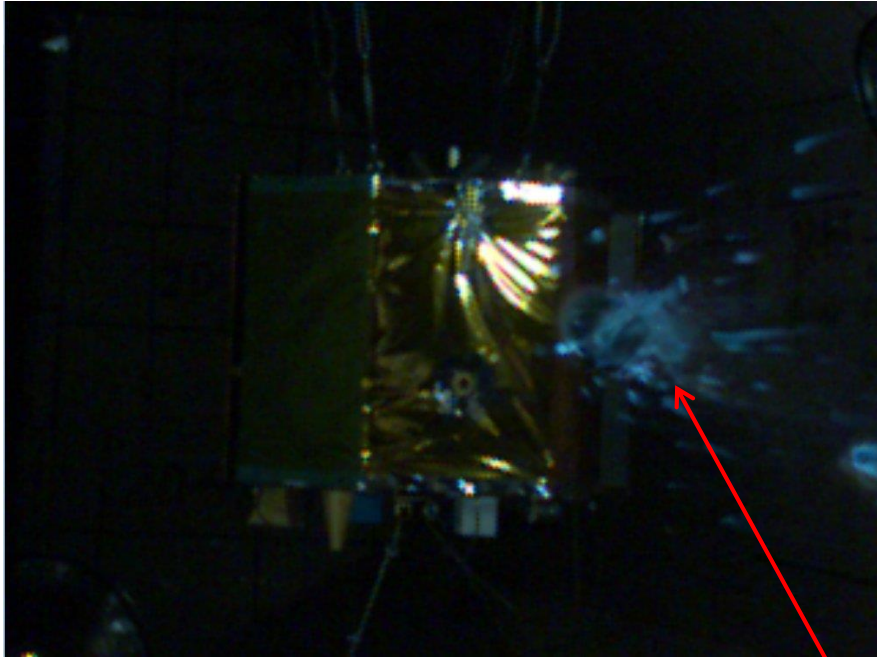
Panel Label Code: SEGC-L
 S — ShotNumber [1/2]
 E — PanelGroup1 [U=uprange]
 G — PanelGroup2 [F=floor / C=ceiling]
 C — MapColumn [L=left / R=right]
 L — PanelLayer [numbered from top (foam) to bottom (plywood)]

•stubs recovered (+), with Ta sheet present (++) , in place (*)

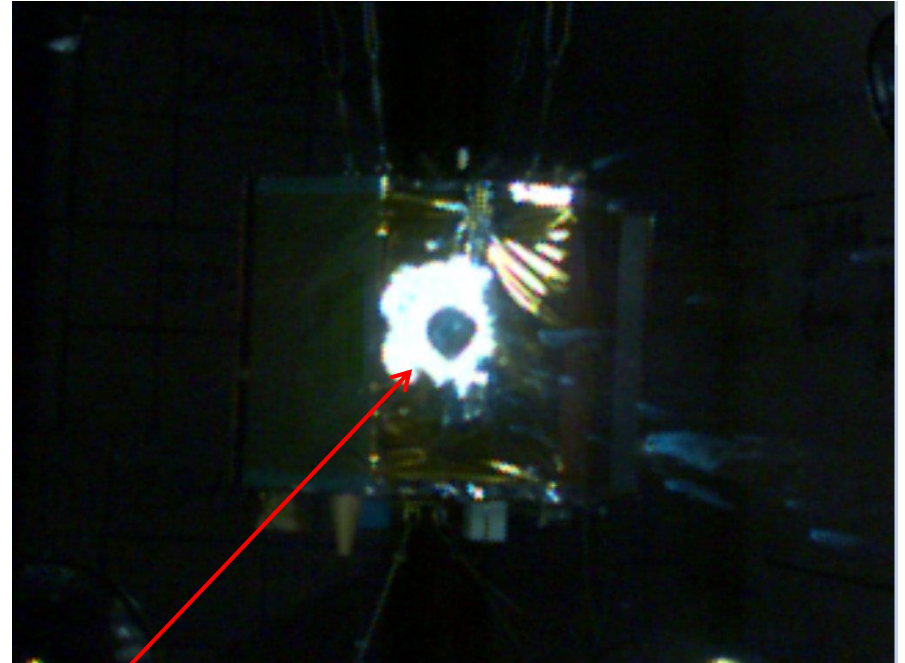


DebrisSat Impact: Frames from High Speed Video

$T = -180 \mu\text{S}$



$T \sim 0 \mu\text{S}$



Projectile

High speed camera (65,000 fps) images from NASA



DebrisSat Impact: Frames from High Speed Video

$T = +45 \mu\text{S}$



$T = +270 \mu\text{S}$



High speed camera (65,000 fps) images from NASA



Laboratory Results

(Supplemental Information and Additional
Analyses in Appendix)



Laboratory Methods

- Scanning Electron Microscopy (SEM)
 - High resolution imaging.
 - Atomic number contrast.
- Transmission electron microscopy (TEM)
 - Ultra high resolution – lattice imaging (crystallinity)
 - Electron diffraction – crystallinity – phase identification
- Energy Dispersive (X-ray) Spectroscopy (EDS) in the SEM/TEM
 - Semiquantitative elemental composition.
 - Elemental mapping and line scans.
- Fourier Transform Infrared (FTIR) spectroscopy
 - Identification of chemical functional groups.
 - Correlation with LWIR hyperspectral remote sensing signatures.
- Raman Spectroscopy
 - Identification of forms of carbon
- UV-VIS-NIR Spectroscopy
 - Measurement of darkening at UV-VIS-NIR wavelengths.

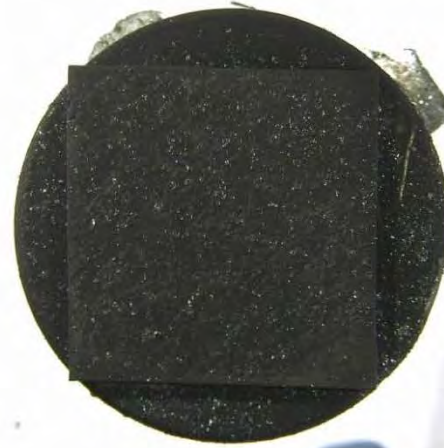


SBU Marking SEM Stubs (post test)

8



9



13



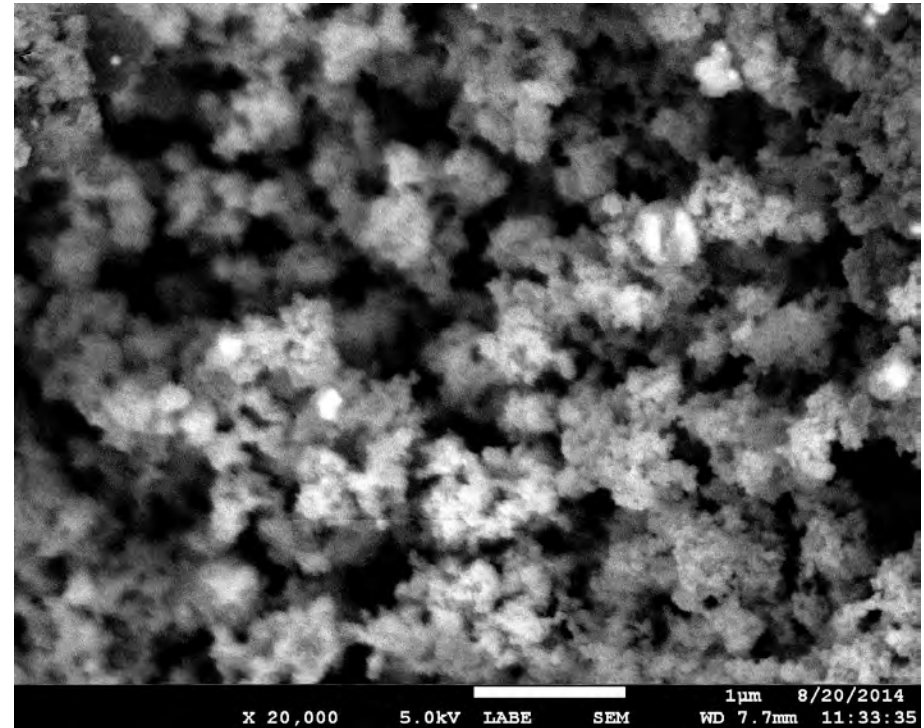
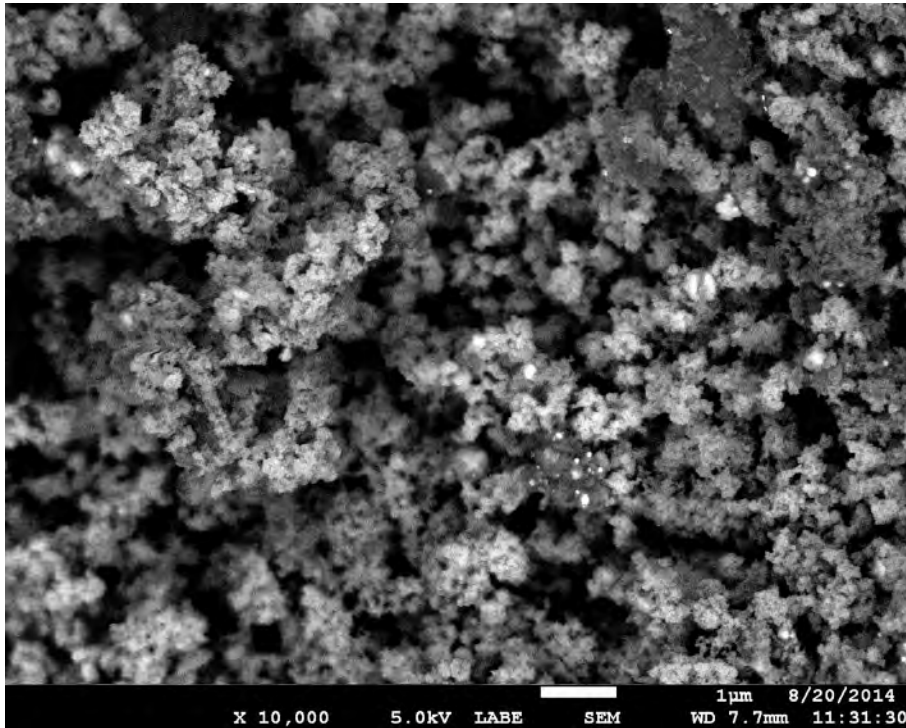
14



Stubs are covered with a black soot-like substance



SEM Stub 4: Backscatter Electron SEM (10KX, 20KX)



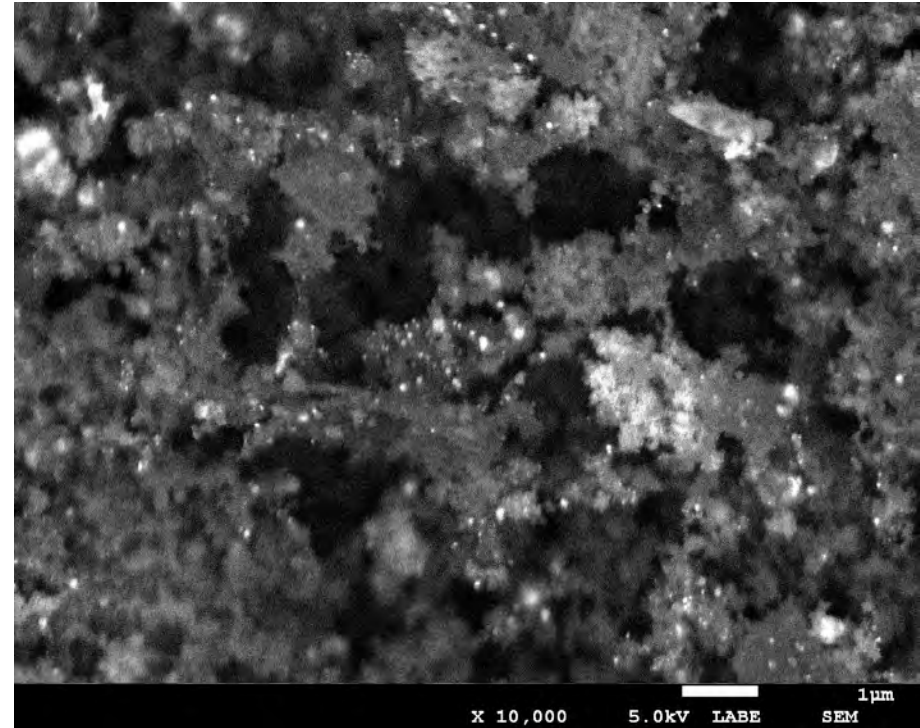
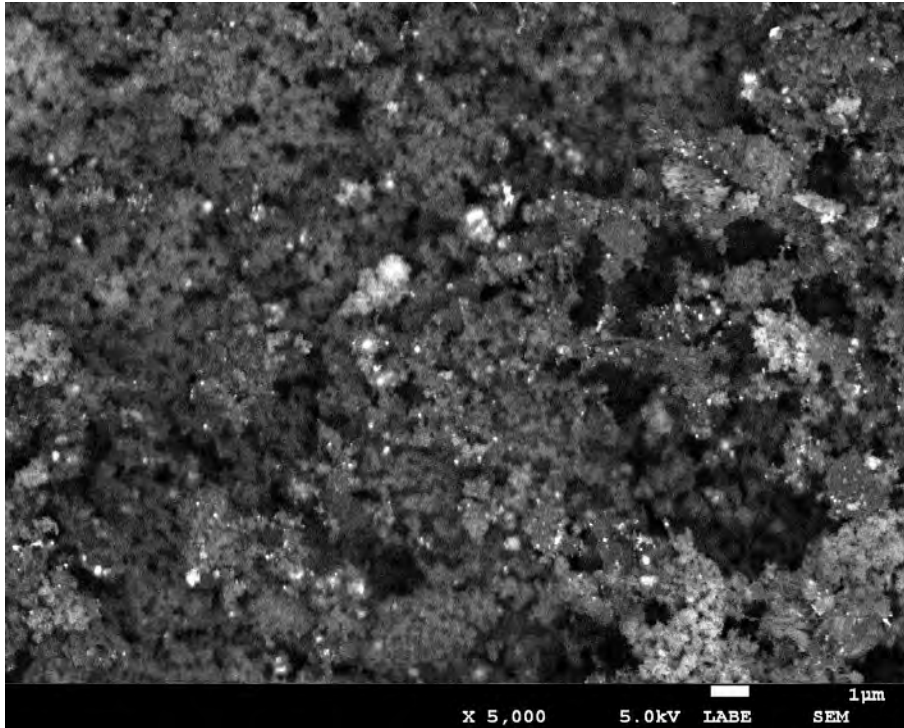
Material on surface consists of nano-sized agglomerates

Located 9 feet down range – 9 O'clock



SEM Stub 9

Backscatter SEM 5KX, 10KX



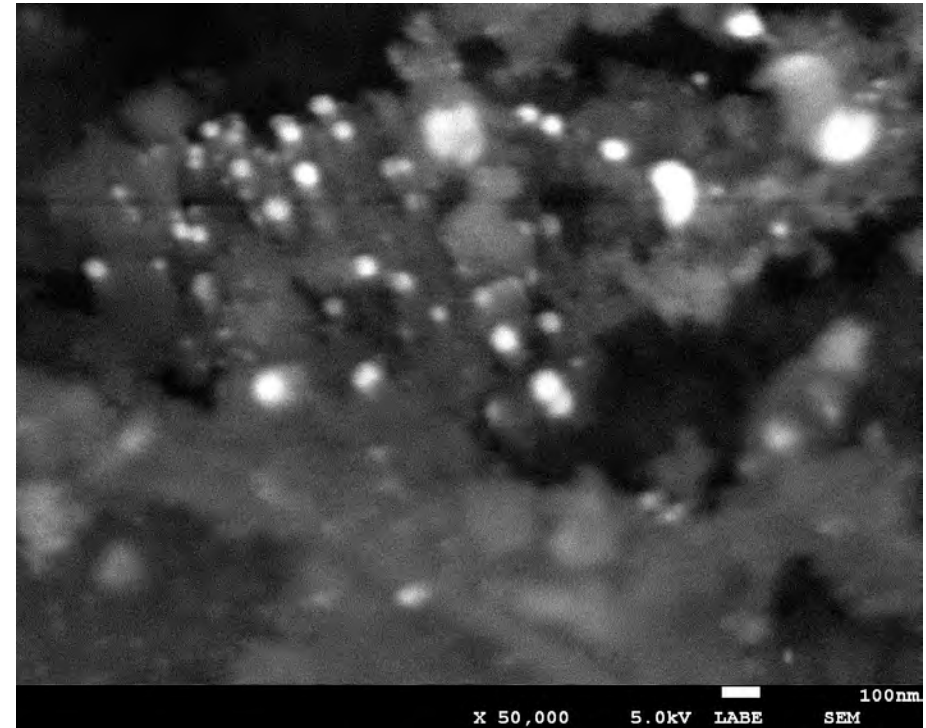
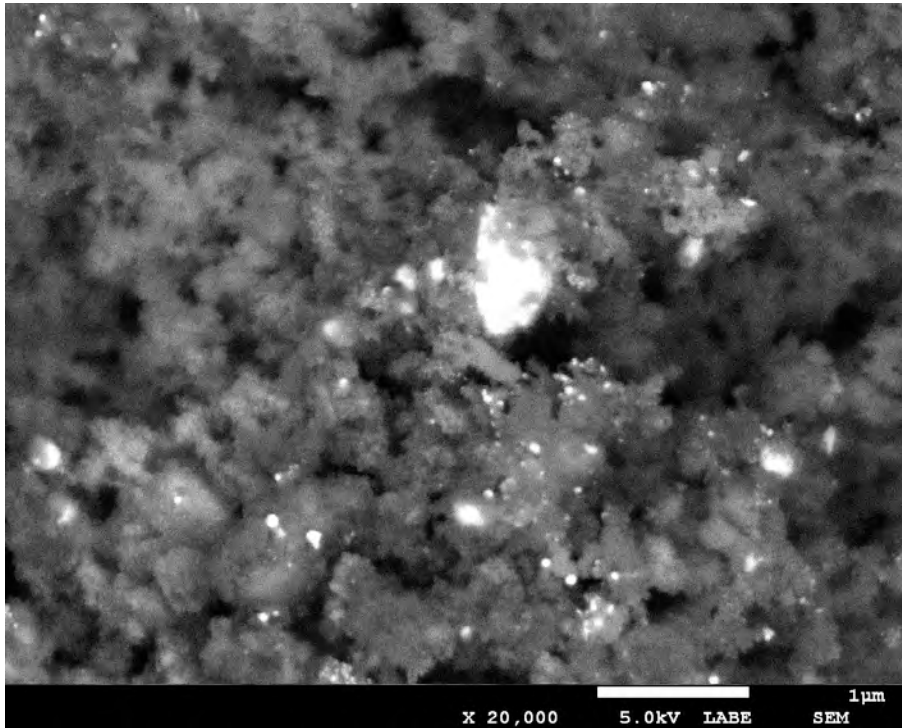
Nano-sized bright areas are high Z (Fe, Cu, Zn Ge, Al) mixed with darker carbonaceous debris.

Located 17 feet down range – 12 O'clock



SEM Stub 9

Backscatter SEM 20KX, 50KX



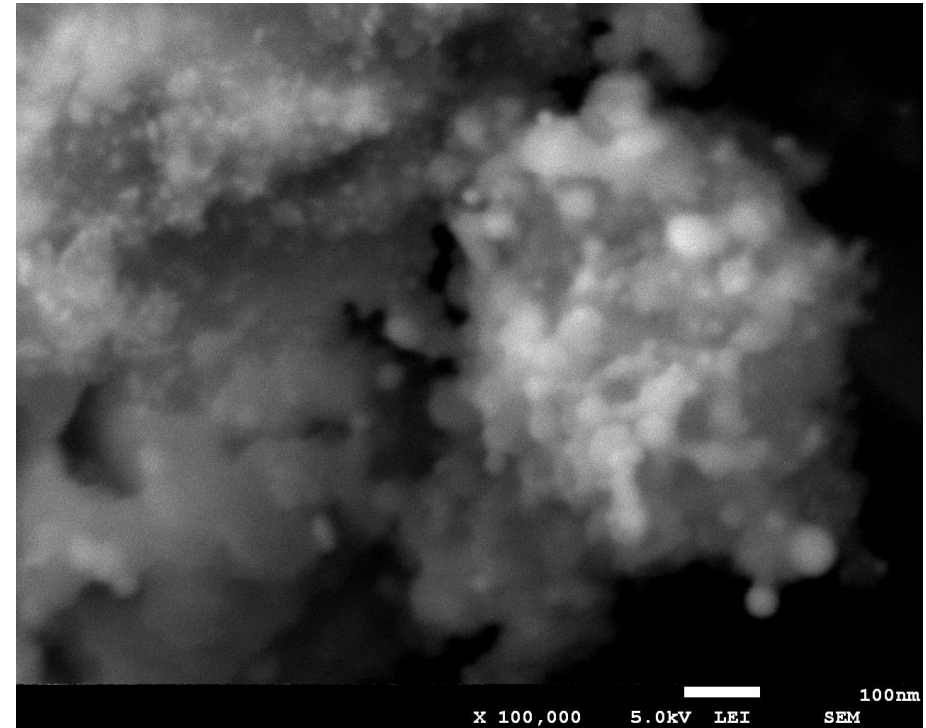
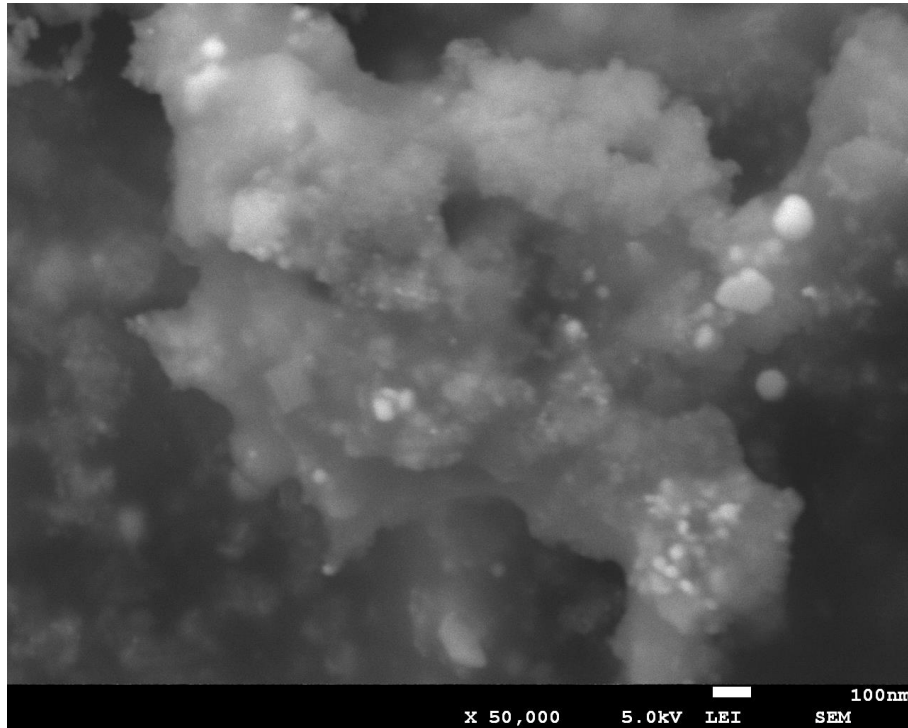
Nano bright areas appear to be solidified molten droplets of high Z material (Fe, Cu, Zn Ge, Al) mixed and covered with carbonaceous debris.

Located 17 feet down range – 12 O'clock



SEM Stub 13

Secondary Electron SEM (50KX, 100KX)

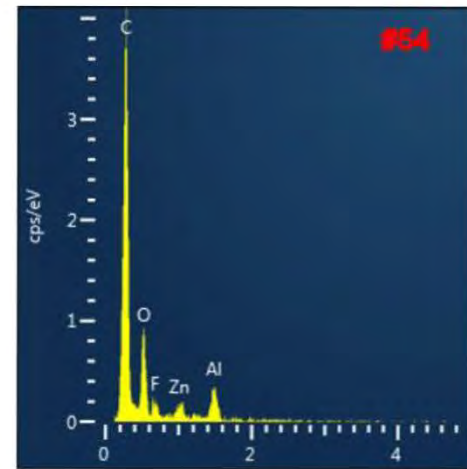
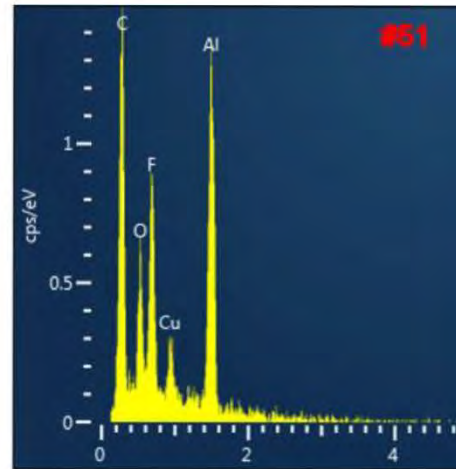
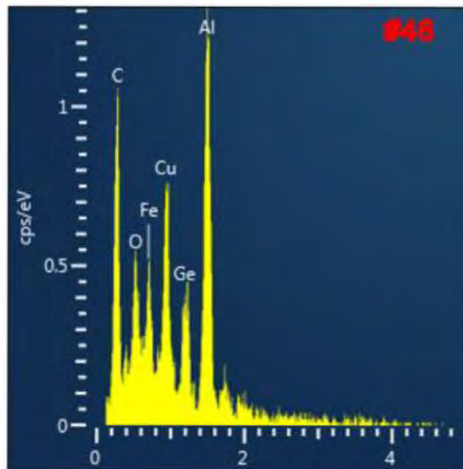
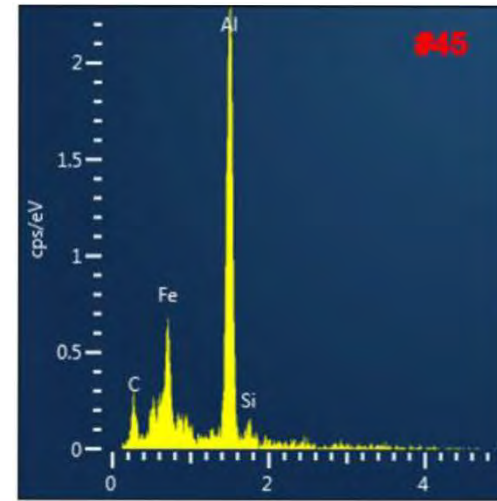
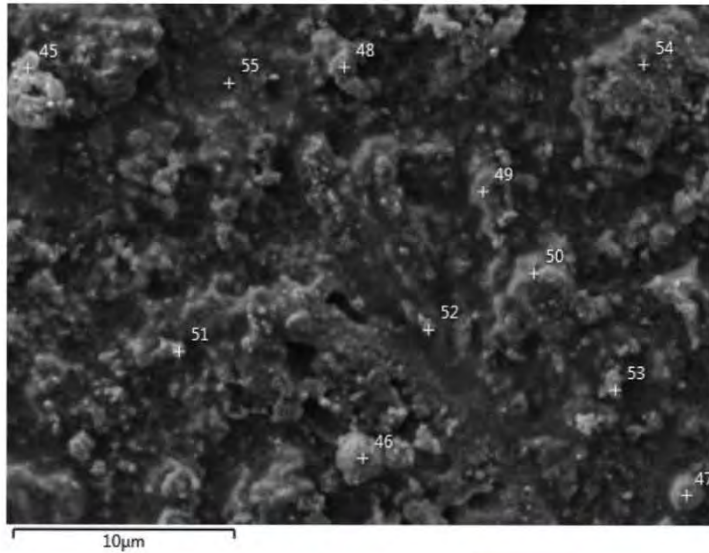


Note nano-scale particulates

Located 11 feet down range – 3 O'clock



SBU Marking SEM Stub 13 SEM EDS

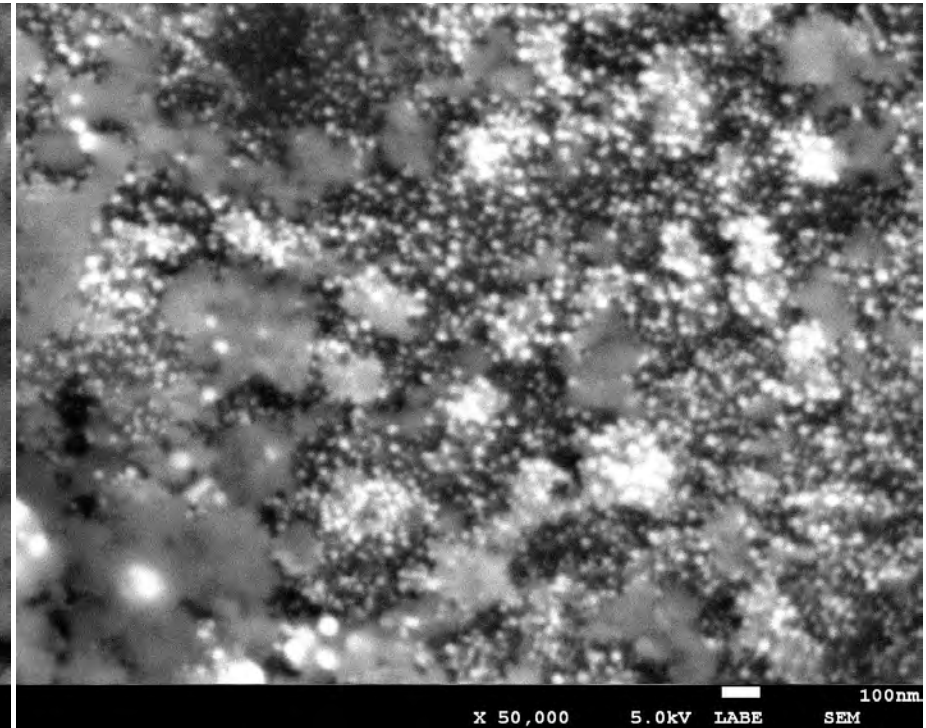
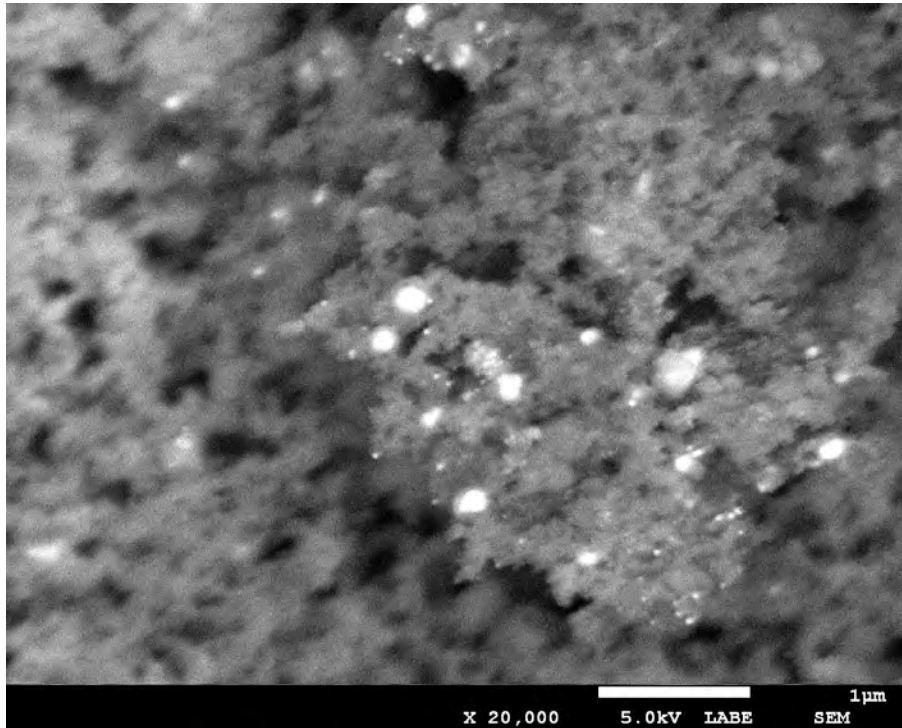


Al, C, F, Cu, O, Fe, and Ge are common.
Bright nano droplets contain Fe, Cu, Ge.



SEM Stub 14

Backscatter SEM 20KX, 50KX



Majority of the deposit is an agglomeration of nano carbonaceous material

Note higher Z nano particles

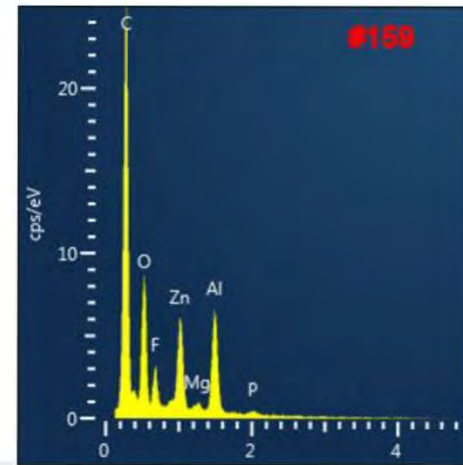
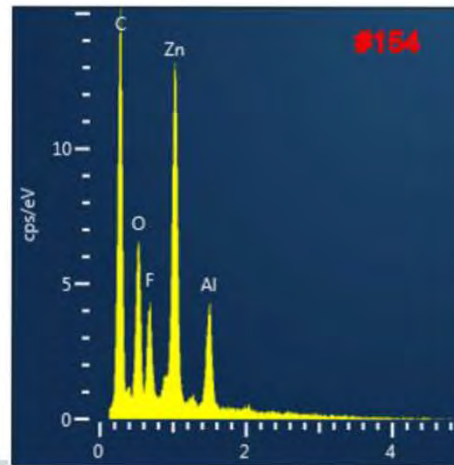
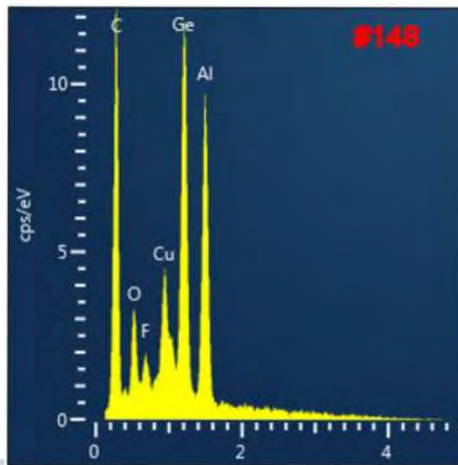
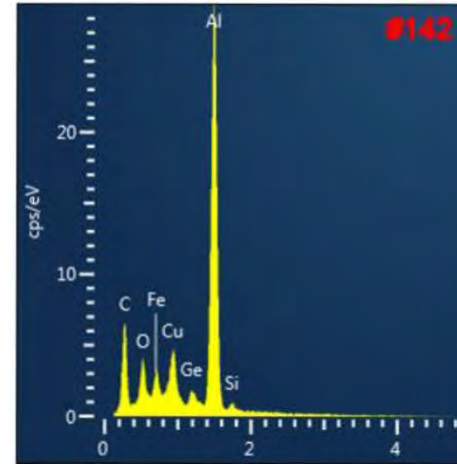
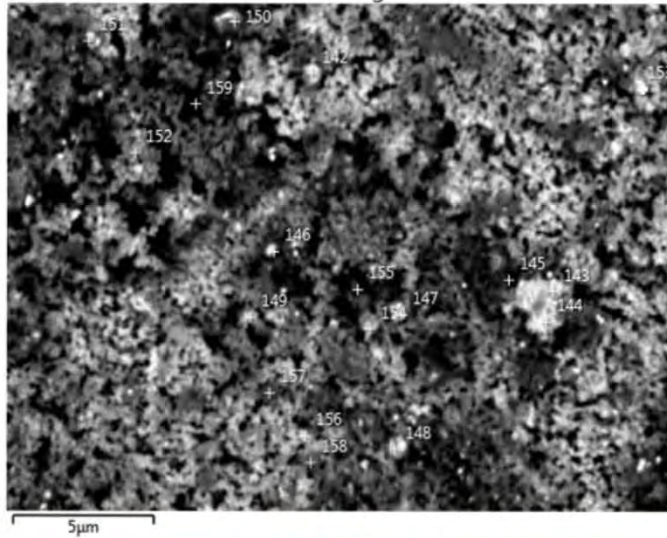
Located 15 feet down range – 3 O'clock



SBU Marking

SEM Stub 14

SEM EDS (5 KV)



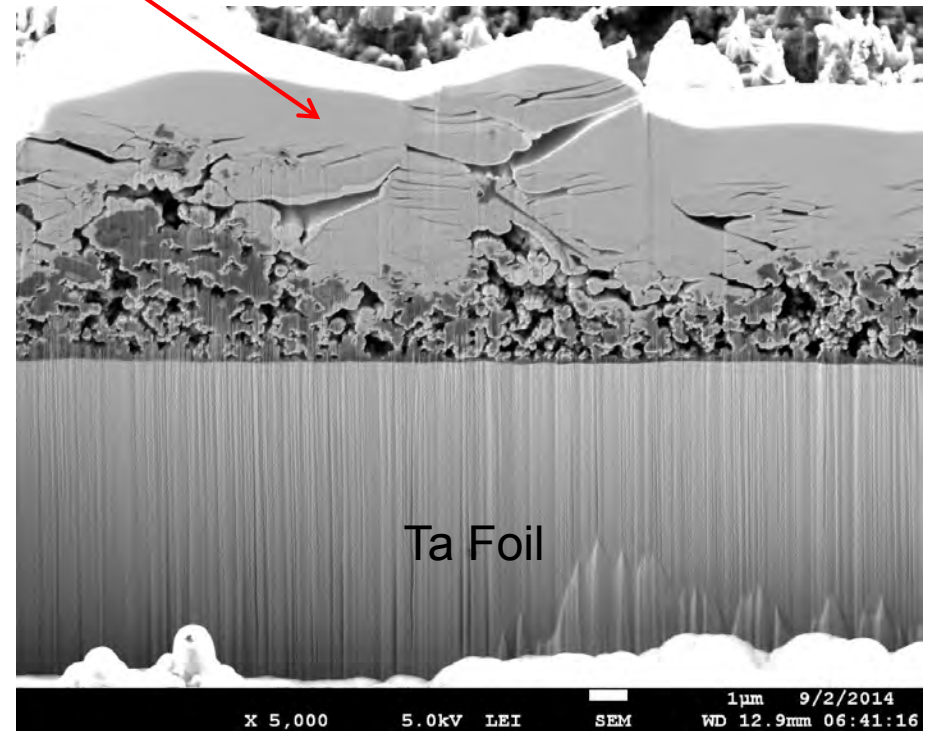
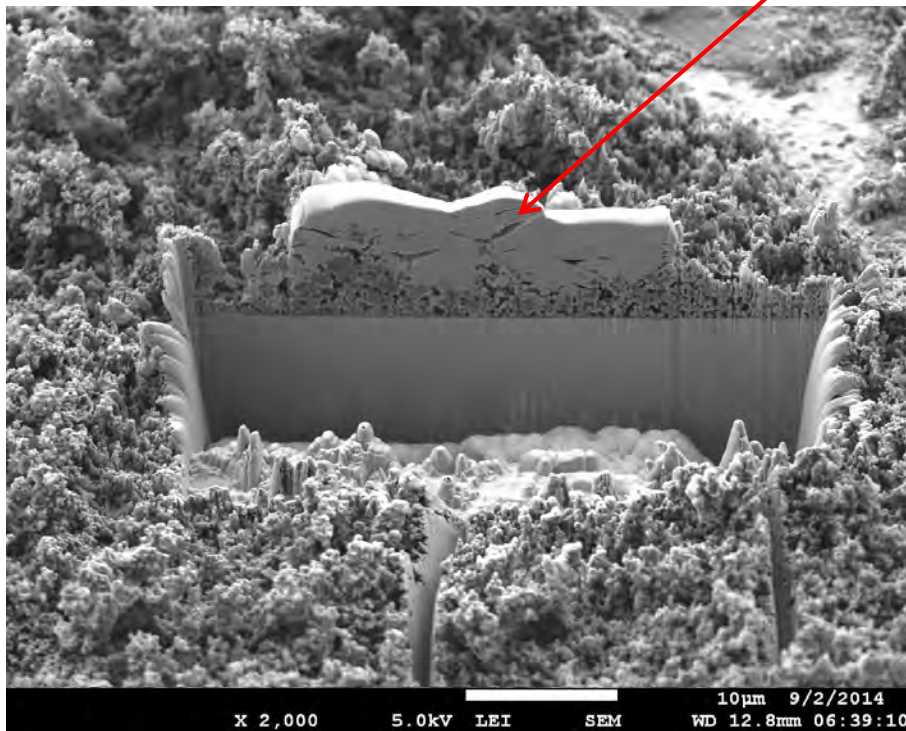
C is ubiquitous - Al, Zn, Ge, F and Cu are common.



SEM Stub 14: Focused Ion Beam (FIB) Cross Section

Secondary Electron SEM 2KX, 5 KX

Protective Pt Layer



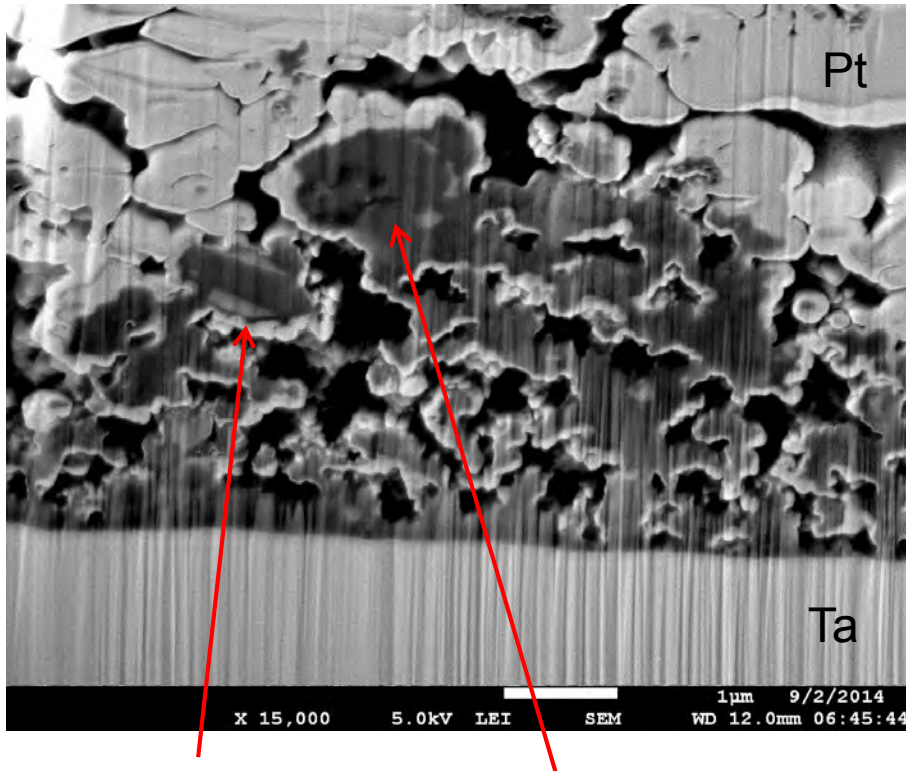
45 degree tilt

32 Deposit is several microns thick consisting of loose debris which is composed of agglomerates of nano particles.

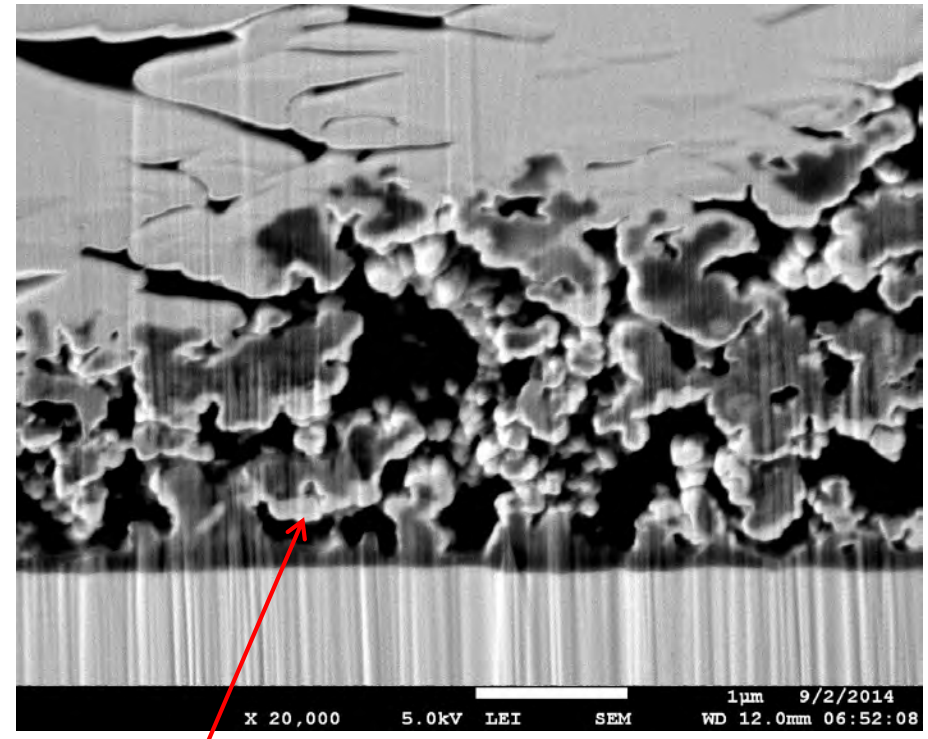


SEM Stub 14: FIB Cross Section

Backscatter Electron SEM 15KX, 20 KX



Higher Z fragments in carbonaceous matrix



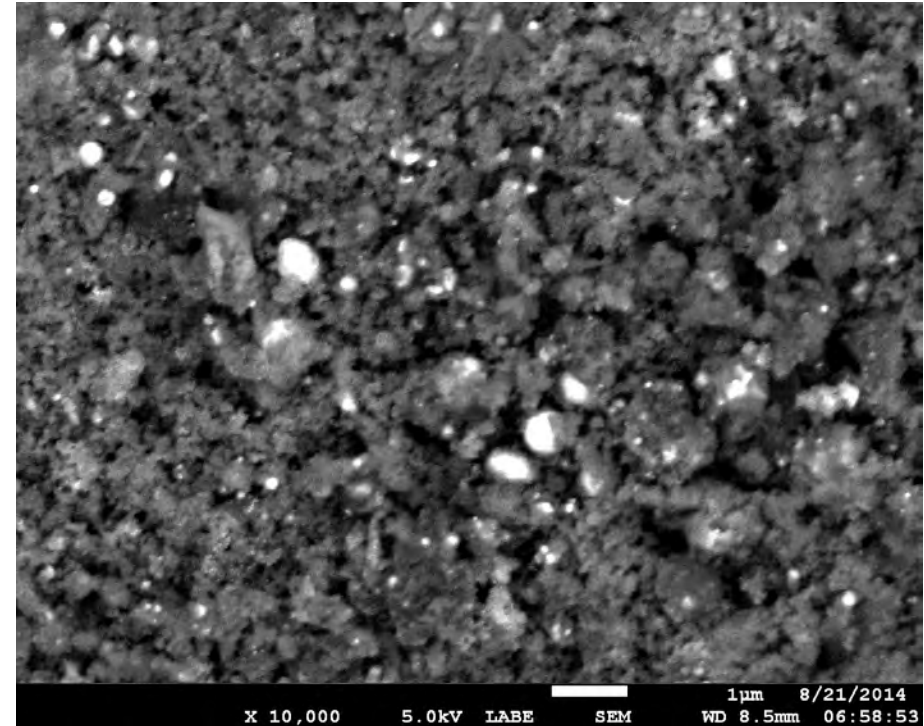
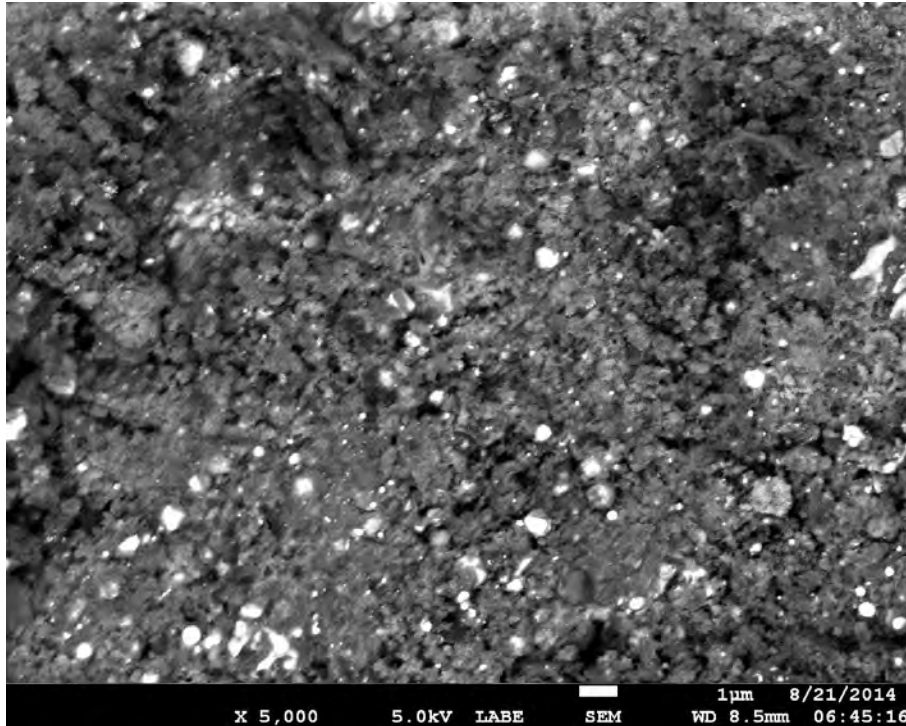
Light rind on particles is probably Ta ejected into cavities during the trenching operation or infusion of Pt.

EDS analysis was hampered by relatively large sampling volume and introduction of significant Pt, Ta and Ga. C, O and some Al detected.



SEM Stub 20

Secondary Electron SEM 5KX, 10KX



Material on surface consists of nano-sized agglomerates. Darker areas are carbon rich. Bright spots contain Fe, Cu, Zn, Ge and represent solidified molten droplets partially covered with carbonaceous nano particles.

Located on down range back wall.



Summary of Area EDS Analyses

v	Stub #4	Stub #13	Stub #14	Stub #9	Stub #20
C	57.7	78.0	61.9	61.1	61.7
O	20.1	11.9	16.1	18.8	16.2
F	7.4	1.8	4.4	5.7	2.9
Na	0.90	0	0	0.35	0
Mg	0.27	0.12	0.38	0.36	0.39
Al	5.4	3.6	5.8	8.7	12.5
Si	1.1	0.53	0	0.88	0.45
P	0	0.11	0.30	0.28	0.35
S	0	0	0	0.03	0
Cl	0.13	0.09	0.12	0.10	0
Cr	0	0	0.03	0.16	0.38
Fe	0.28	0.22	0.28	0.64	1.42
Cu	0	0.09	0.14	0.25	1.27
Zn	3.4	0.16	7.7	1.9	2.0
Ge	0.07	0	0	0.18	0.40
Ta	3.4	3.4	2.9	0.40	0
Distance Down Range	9 ft	11 ft	15 ft	17 ft	19 ft
Azimuth*	9 O'clock	3 O'clock	3 O'clock	12 O'clock	End Panel

All values atomic %

Ta is from the underlying Ta sheet

* Facing down range

Averages of (2) 12 mm² areas

No consistent trends in elemental distribution.



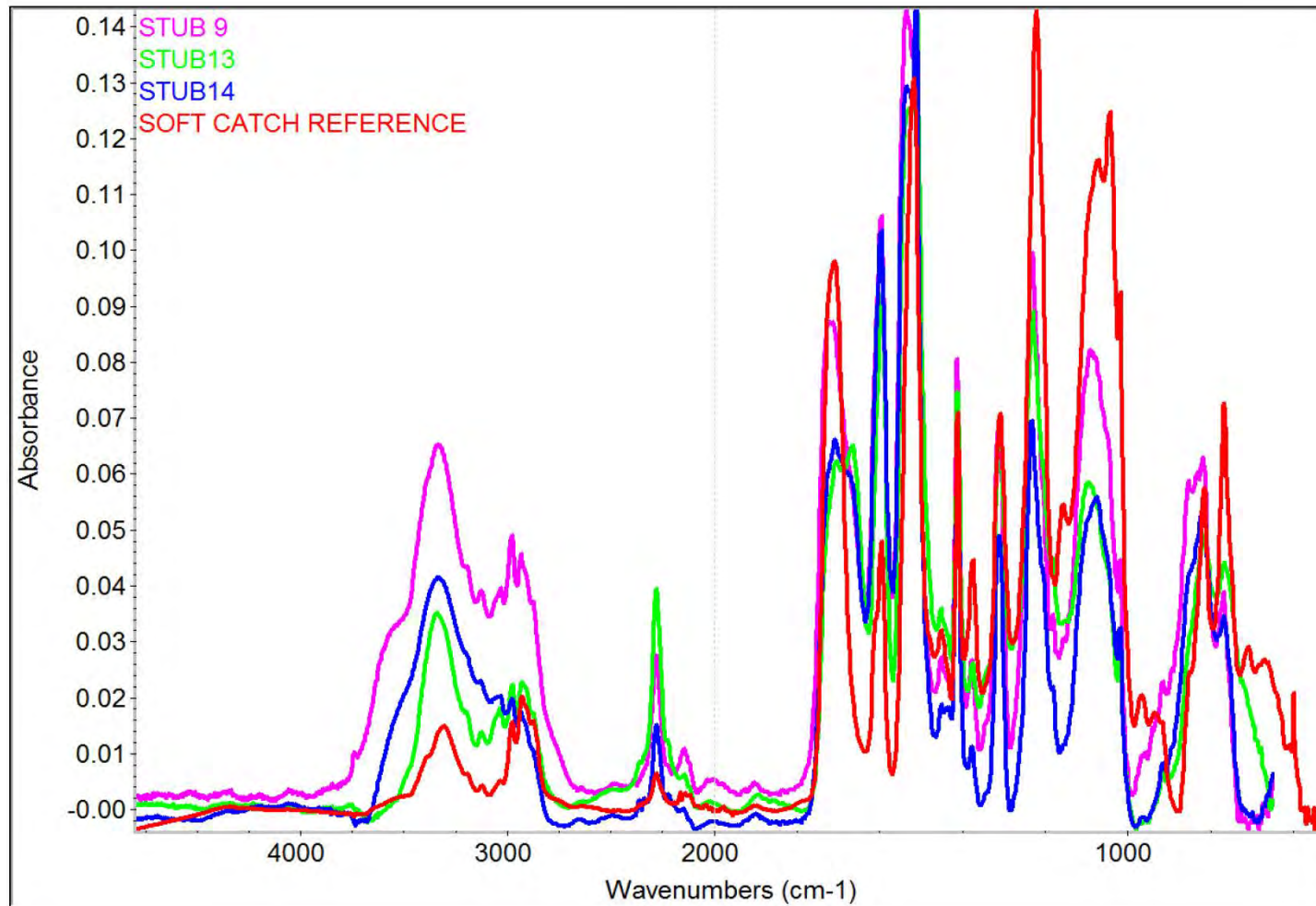
Summary of Area EDS Analyses

- The majority of the deposits consist of carbon and oxygen (77- 89 at%).
 - Primarily from pyrolyzed soft catch (based on Debris-LV results).
 - Some contribution from MLI and C-C composite face sheet.
- Fluorine (1.8 - 7.4 at%) is from Teflon wire insulation and?
- Metal nano particles are common
 - Aluminum (3.6 - 12.5 at%) is from the aluminum honeycomb panels, structural core, nadir-zenith panels and COPV tank.
 - Zinc (0.2 - 7.7 at%) is from an unidentified source.
 - Iron (0.2 - 1.4 at%) is from stainless steel tubing and solenoids.
 - Copper (0.09 - 1.3 at%) is from wiring and solenoids.
 - Germanium (0.07 – 0.40 at%) is from the solar cells.



SBU Marking Post Test: SEM Stubs

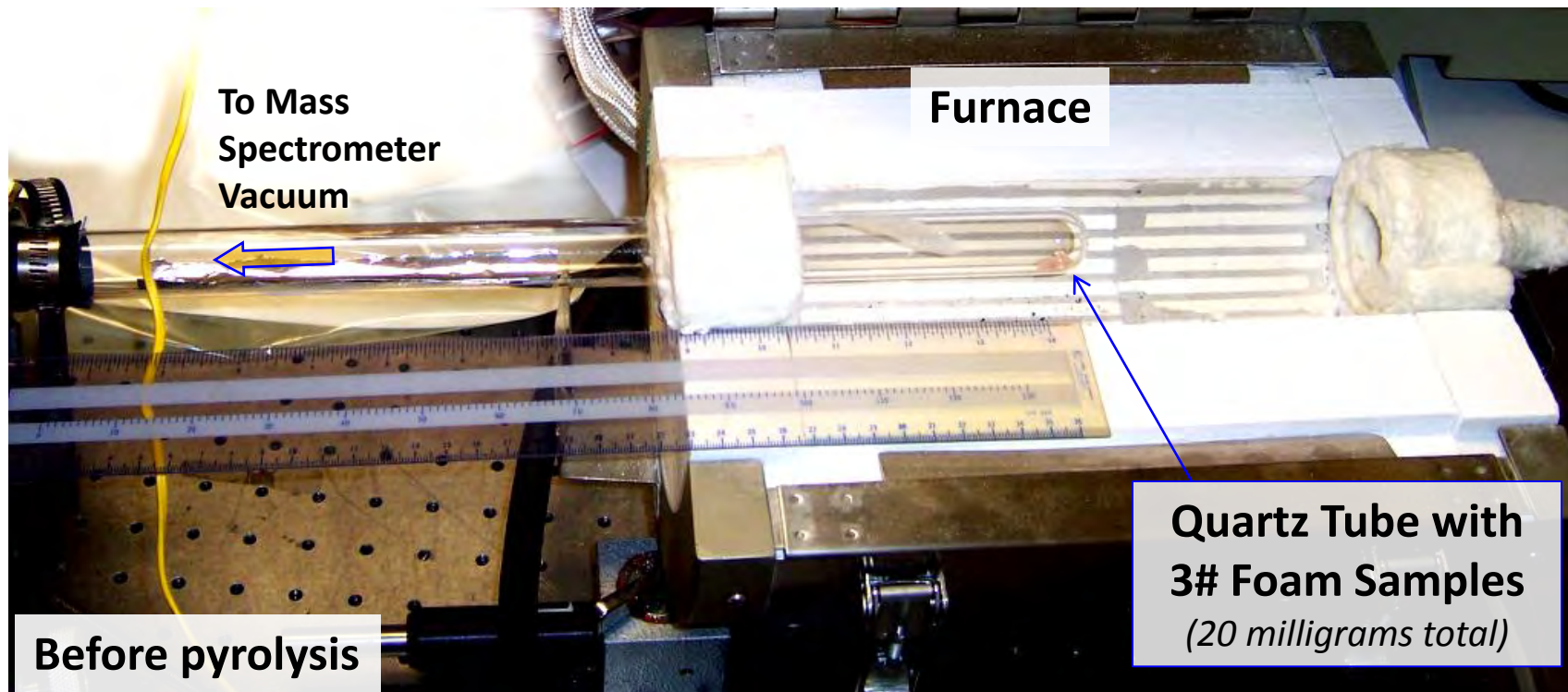
FTIR – Qualitative Diffuse Reflectance



Significant soft catch signature is present on SEM stubs. Some, but not all, is in the form of soft catch foam fragments which give samples a sparkly appearance.



Laboratory Foam Pyrolysis Experiment



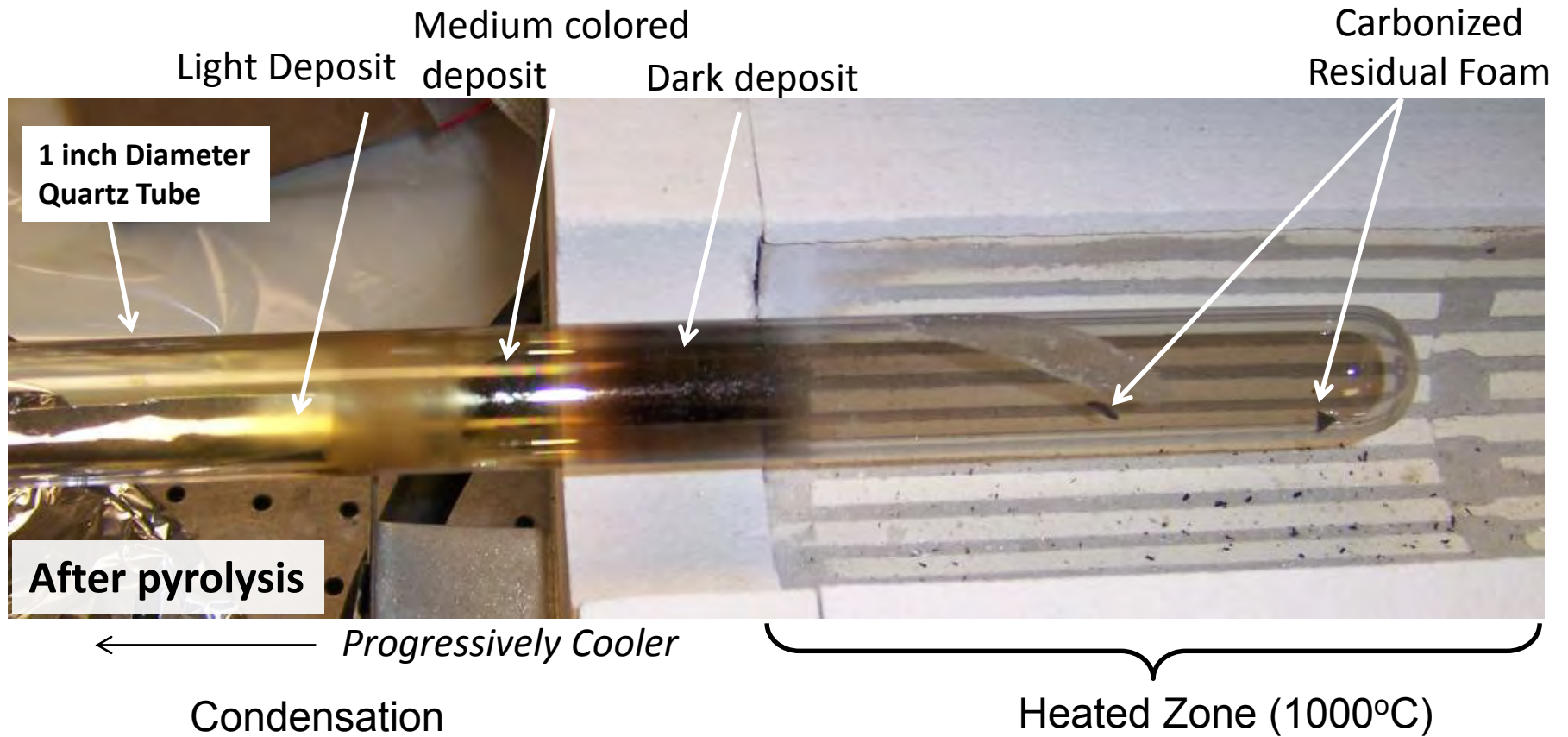
~ 0.001 Torr Throughout Pyrolysis $\Rightarrow \lambda \sim 10$ inches

30 milligram pieces of 3# foam pyrolyzed in a quartz tube under vacuum in order to simulate exposure to plasma from hypervelocity impact.

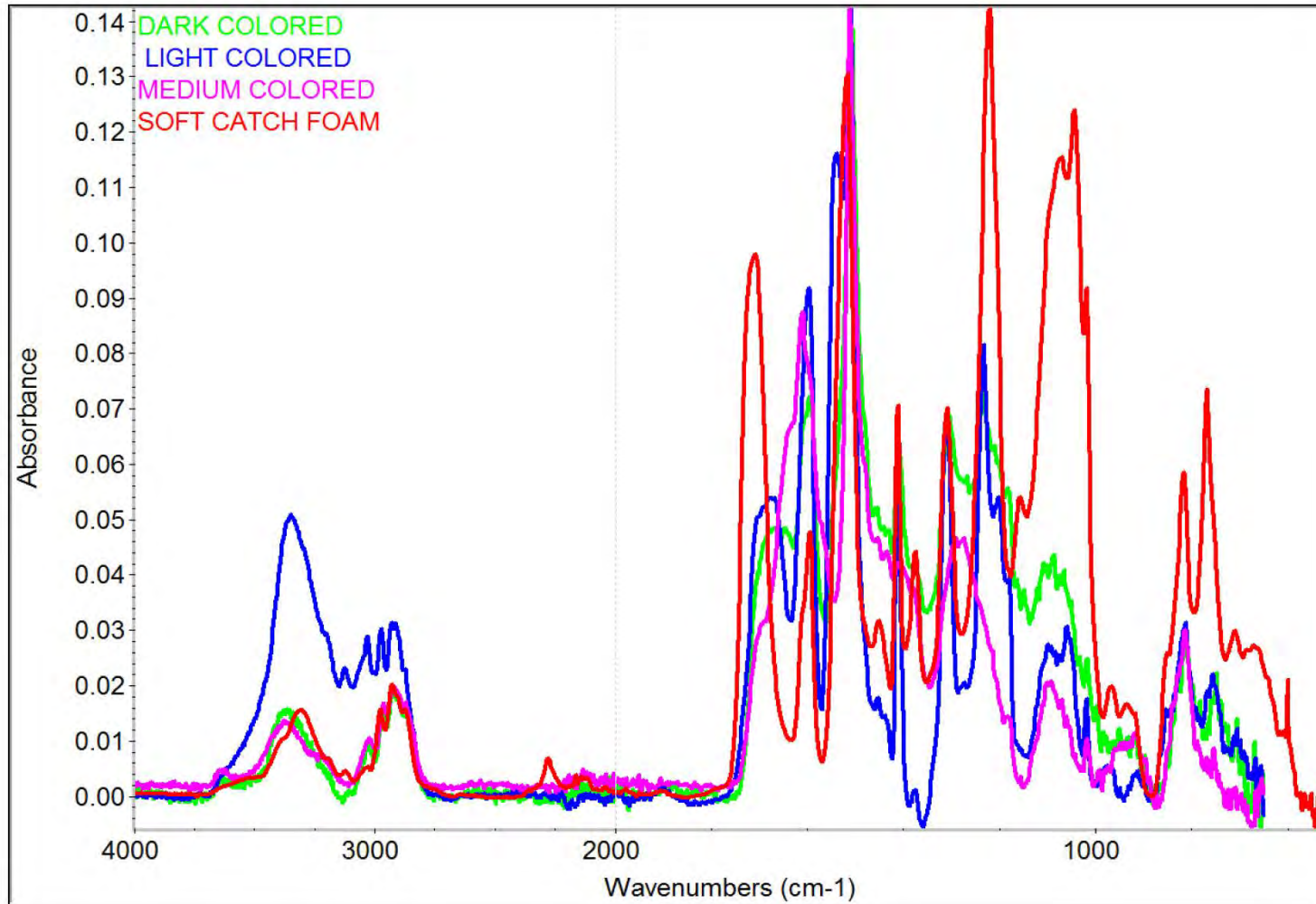
Condensate residues in cool portion of tube outside the furnace were analyzed by FTIR .



Laboratory Foam Pyrolysis Experiment



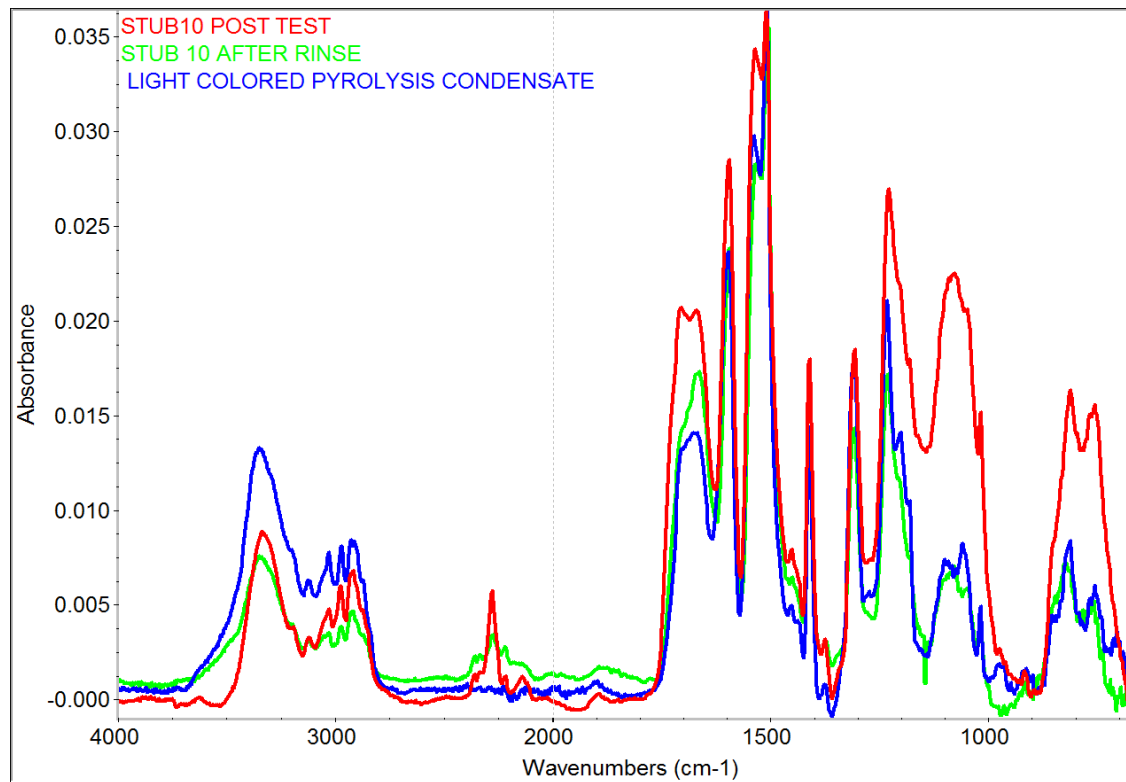
SBU Marking Condensate Removed from Tube



Various colors of condensate have similar but slightly different spectra that resemble soft catch foam.



Debris-LV Stub 10 vs. Pyrolysis Condensate

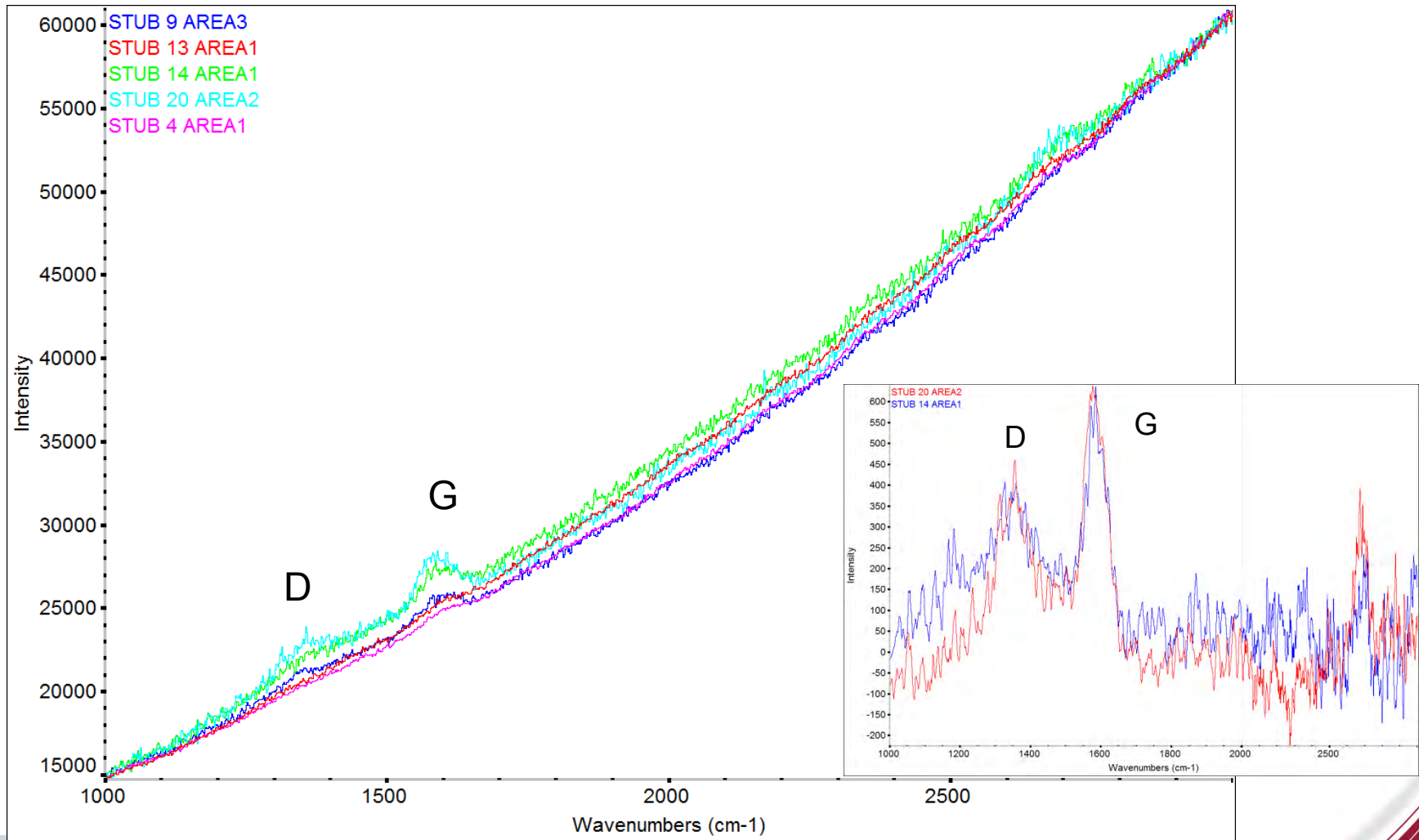


- Spectrum of soft catch condensate is similar to that on SEM stubs, before and after rinsing stubs with isopropyl alcohol (IPA). Alcohol rinse only removed loose fragments.
- SEM stubs are covered with a thin layer of soft catch condensate in addition to fragments. Probably a result of close proximity to soft catch panels exposed to plasma.

Since DebrisSat also used soft catch - SEM stubs are probably also covered with a thin layer of soft catch vapor condensate. DebrisSat stubs were not rinsed with IPA.



SEM Stubs: Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed in some areas.
Graphitic bands also seen in Debris-LV deposits.



Witness Plate: Post Test



Witness Plate Samples:

Direct Exposure

- (4) 1" fused silica **(D)**
- (1) 1" Z-93 painted Al
- (1) 1" Aluminum
- Multi layer insulation

Protected Under Whipple Plates

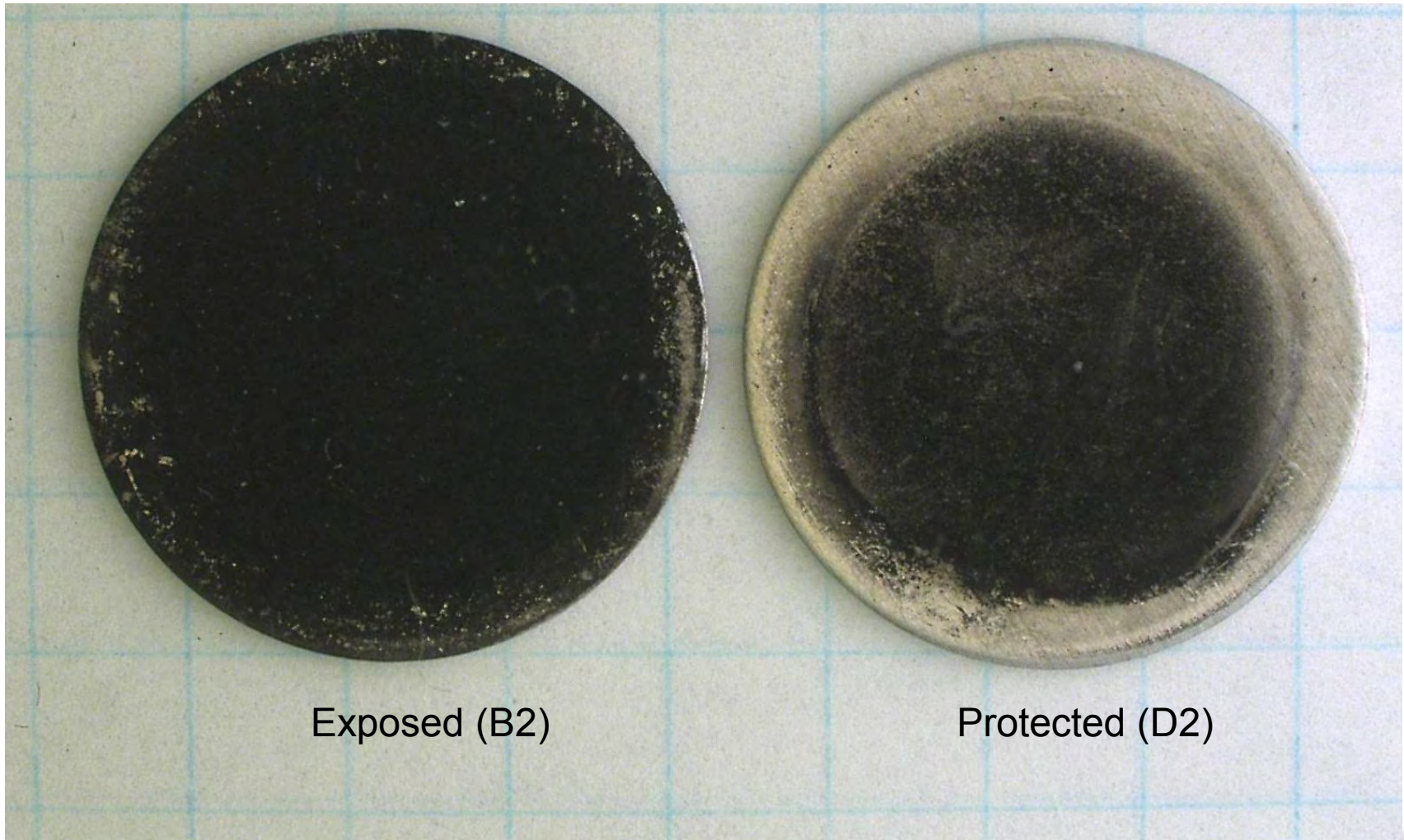
- (2) 1" fused silica
- (1) 1" Z-93 painted Al
- (1) 1" Aluminum
- (1) 1" NaCl (cleaved)
- (1) Cu sheet
- Ge ATR crystal **(D)**
- Solar cell

(D) = destroyed

Top Whipple plate received a significant impact. Assembly is completely covered with black soot. Many witness plate samples were fractured/destroyed.



Witness Plate Post Test : Aluminum Disks

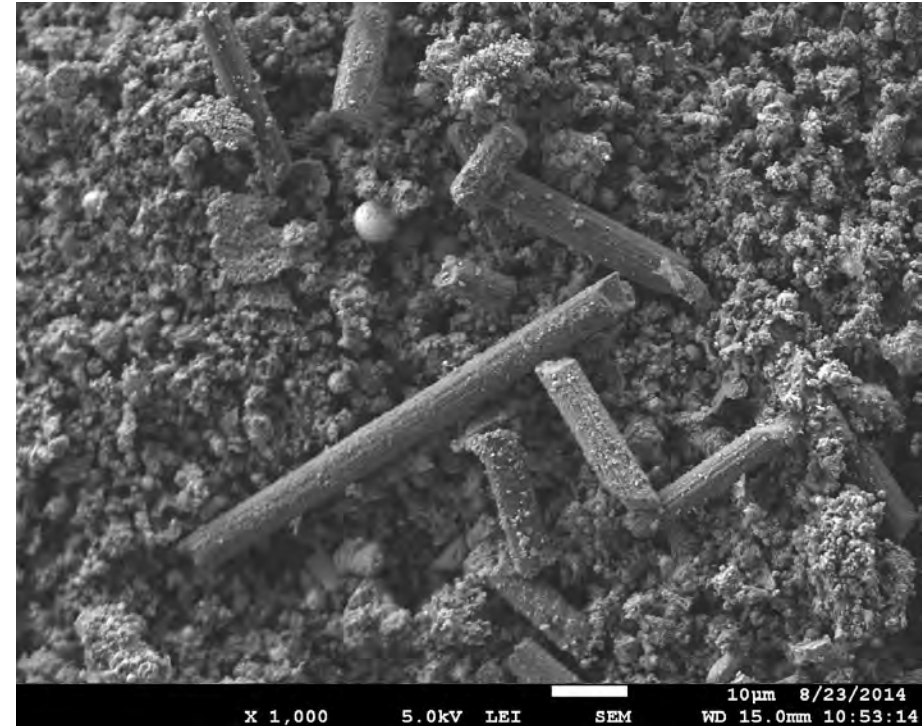
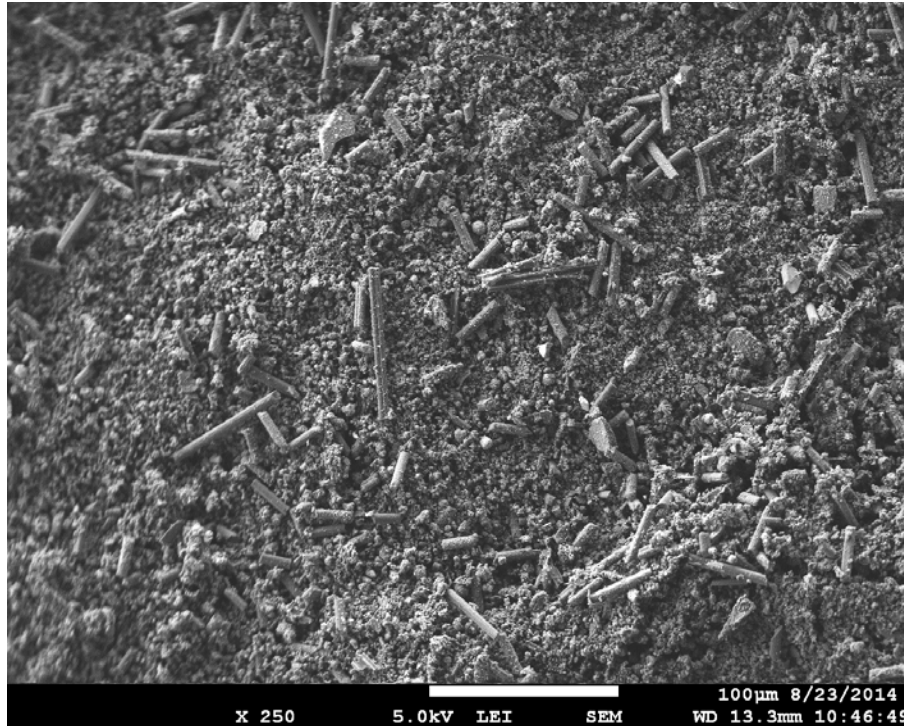


Significant accumulation of debris, especially on exposed disk



Witness Plate Post Test : Aluminum Disk B2 (exposed)

Secondary Electron SEM Images (250X, 1KX)

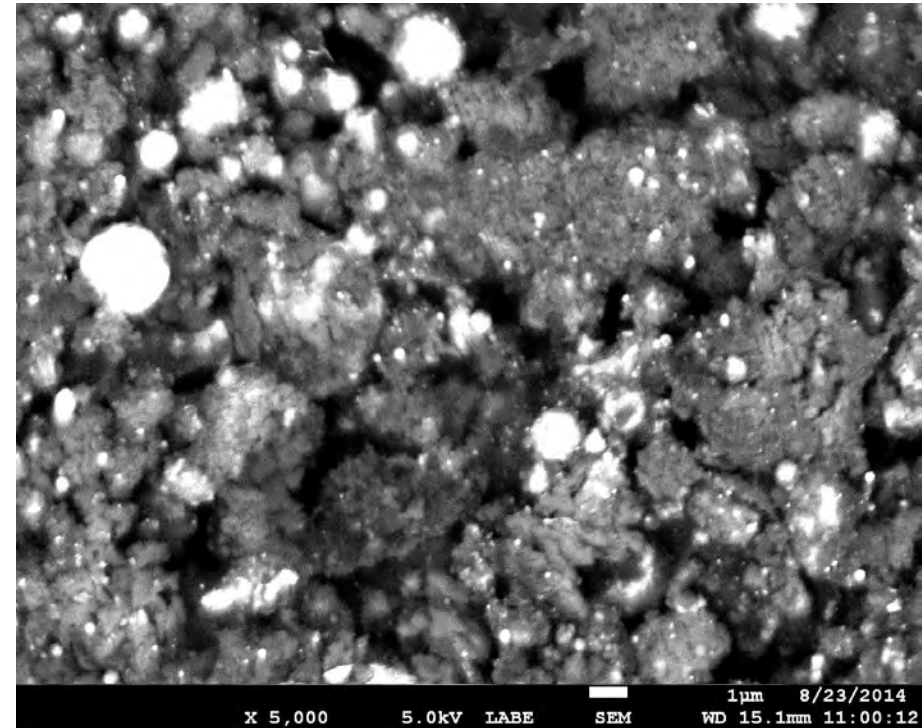
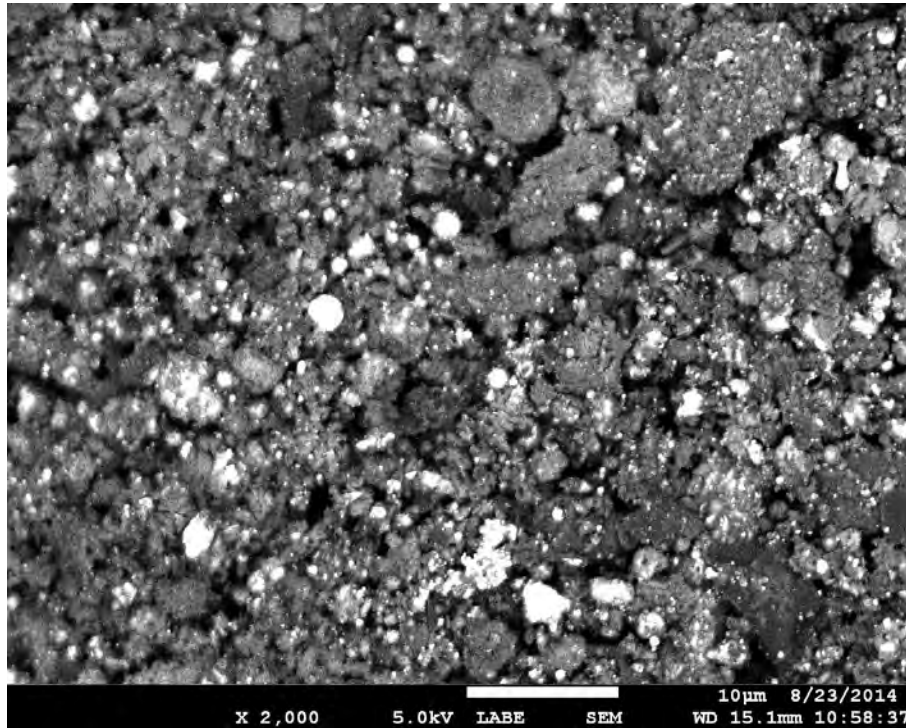


Note fragments of carbon fibers and round solidified droplets. Carbon fibers are from the composite face sheets on the honeycomb structural panels.



Witness Plate Post Test : Aluminum Disk B2 (exposed)

Backscatter Electron SEM Images (2KX, 5KX)

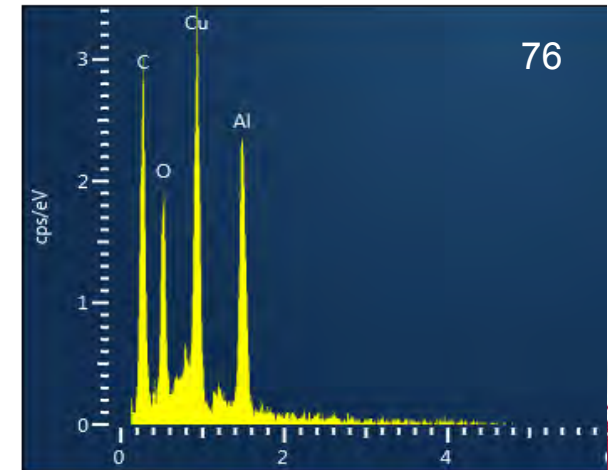
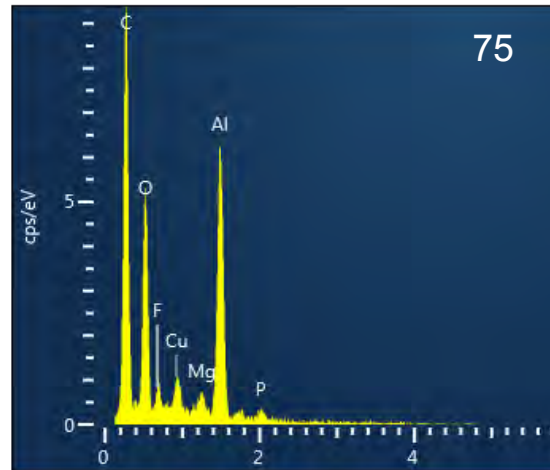
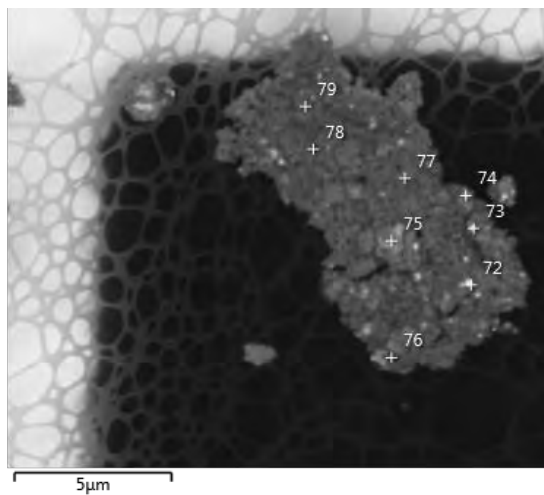
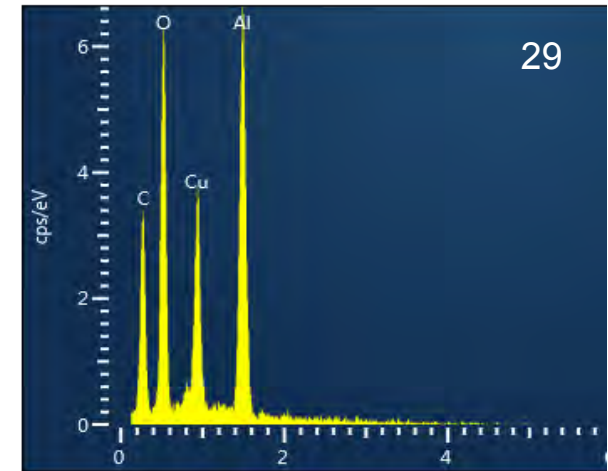
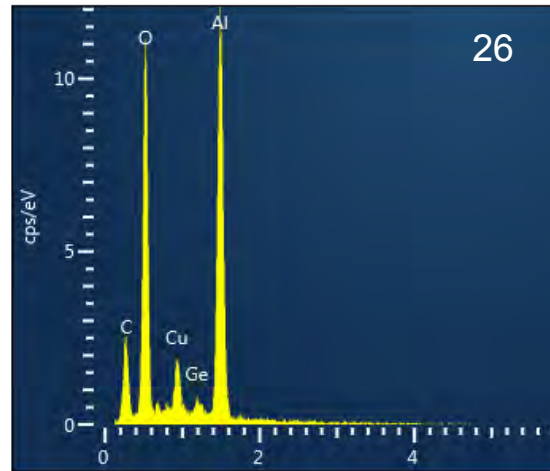
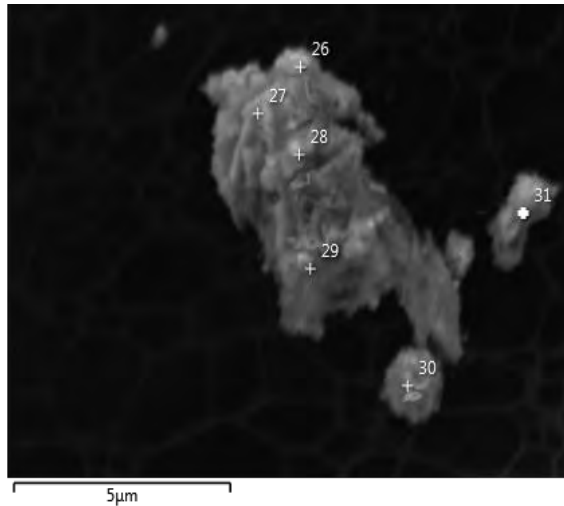


Round solidified droplets are higher Z (Al, Fe, Cu, Ge, Zn) and indicate melting of material as a result of impact.



Witness Plate Post Test : Aluminum Disk B2 (exposed)

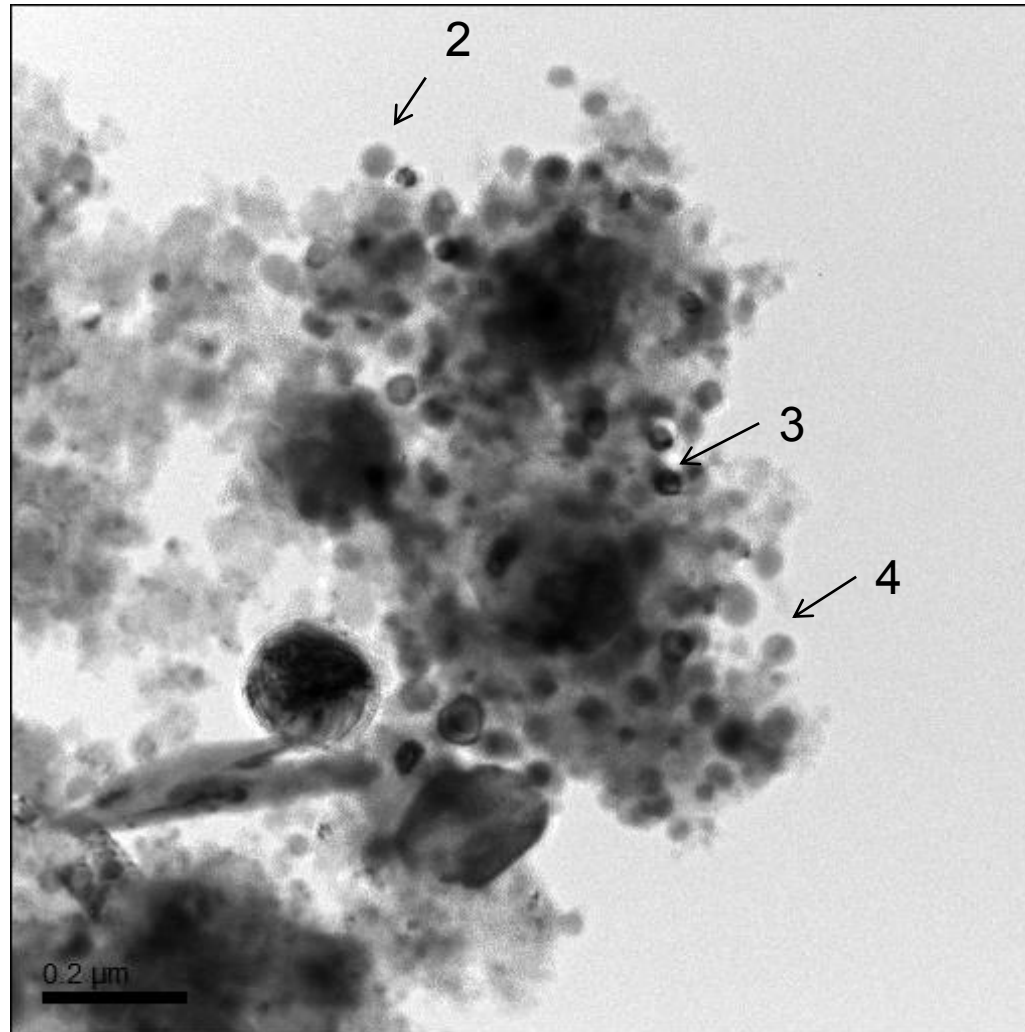
SEM Images of particles suspended on lacey carbon films on Cu TEM grid



Particles are rich in C, O, Al and Cu. Minor C and Cu can be from grid but significant amounts are in the particles. Many particles appear to be aluminum oxide.



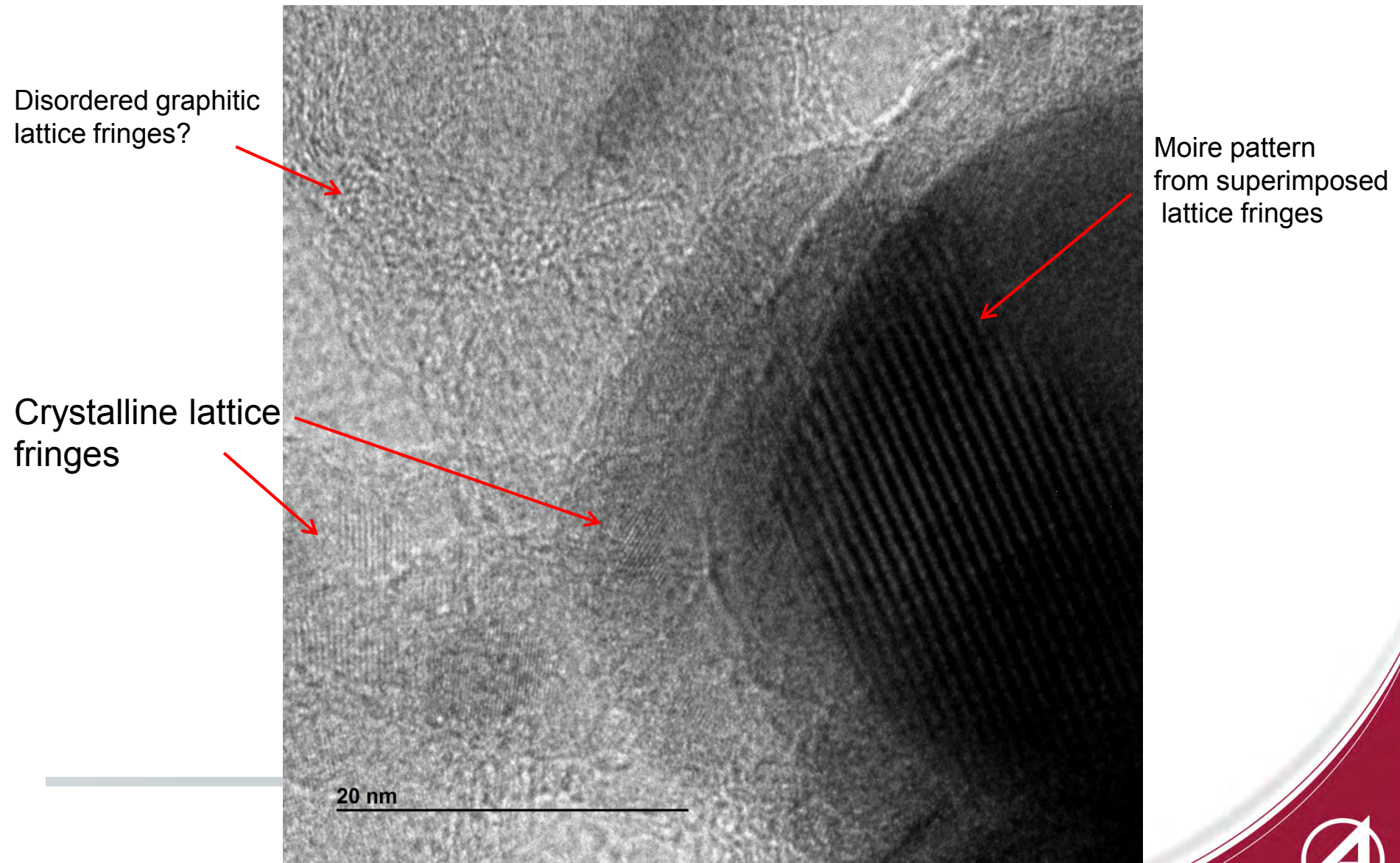
Witness Plate Post Test: Al Disk D2 Debris Transferred to TEM Grid: Bright Field (BF) - TEM



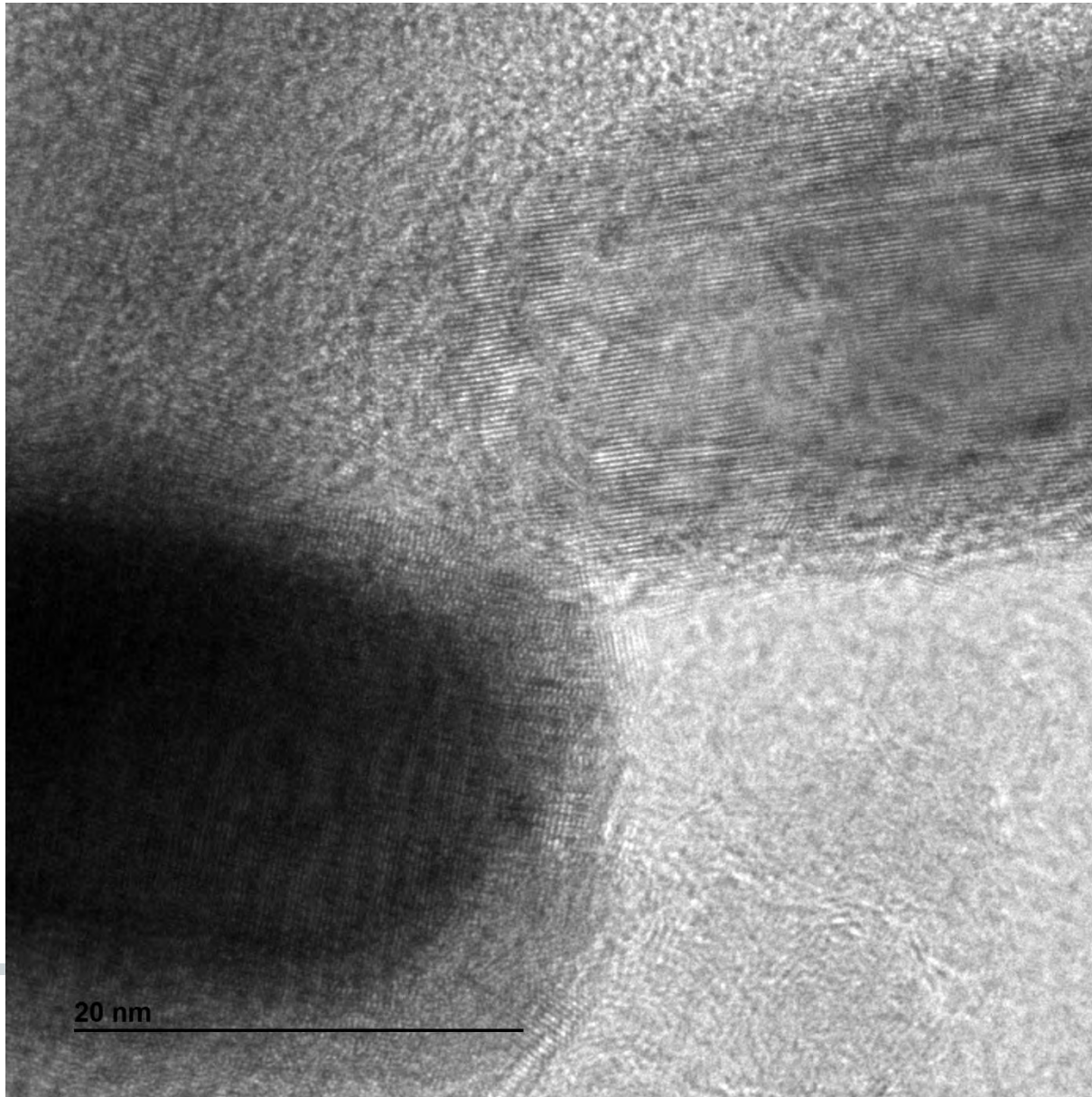
Material is an agglomeration of nano particles. Diffraction contrast in some particles indicates crystallinity.



Witness Plate Post Test D2: Area 3 BF-TEM



Witness Plate Al Disk D2 : Location 4 Area 6 BF-TEM

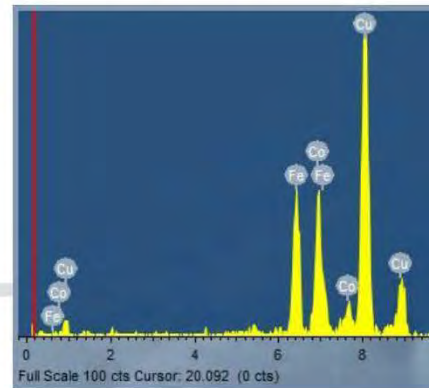
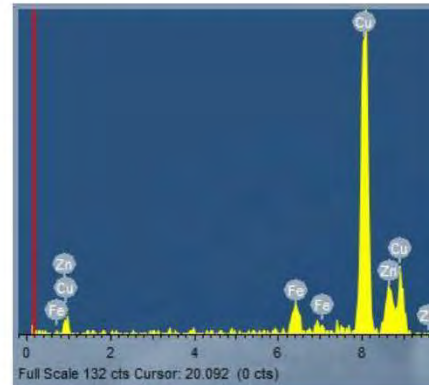
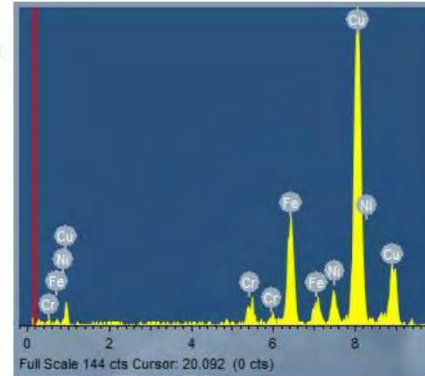
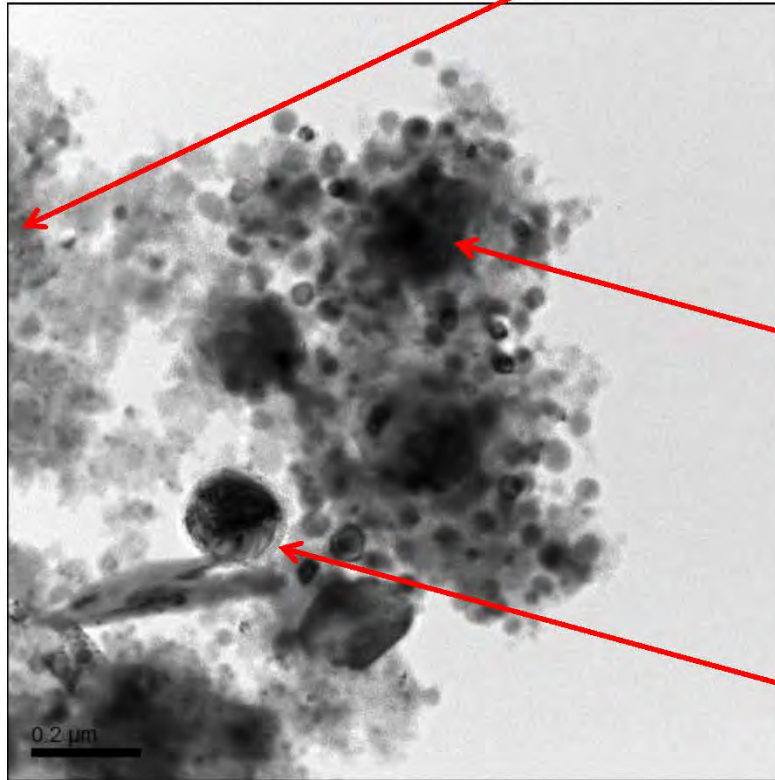


Crystalline lattice fringes (0.256 nm).
ZnO (002) = 0.260 nm

Particle is nominally a single crystal.



Witness Plate Post Test: D2 Location 5 TEM - EDS

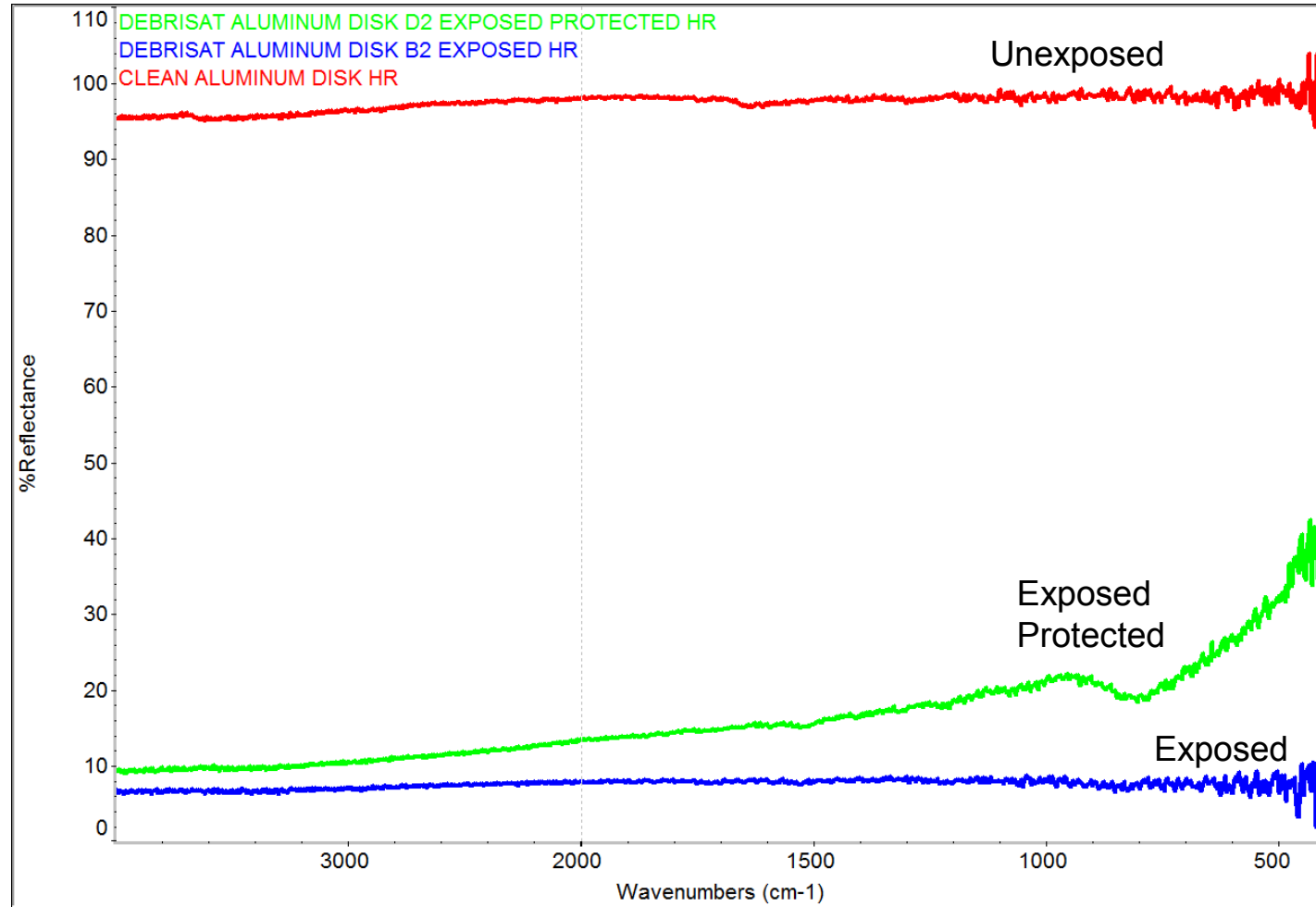


Particulates contain significant Cu, Fe and Co



Witness Plate Post Test: Aluminum Disks (B2, D2)

Quantitative LWIR FTIR Hemispherical Reflectance

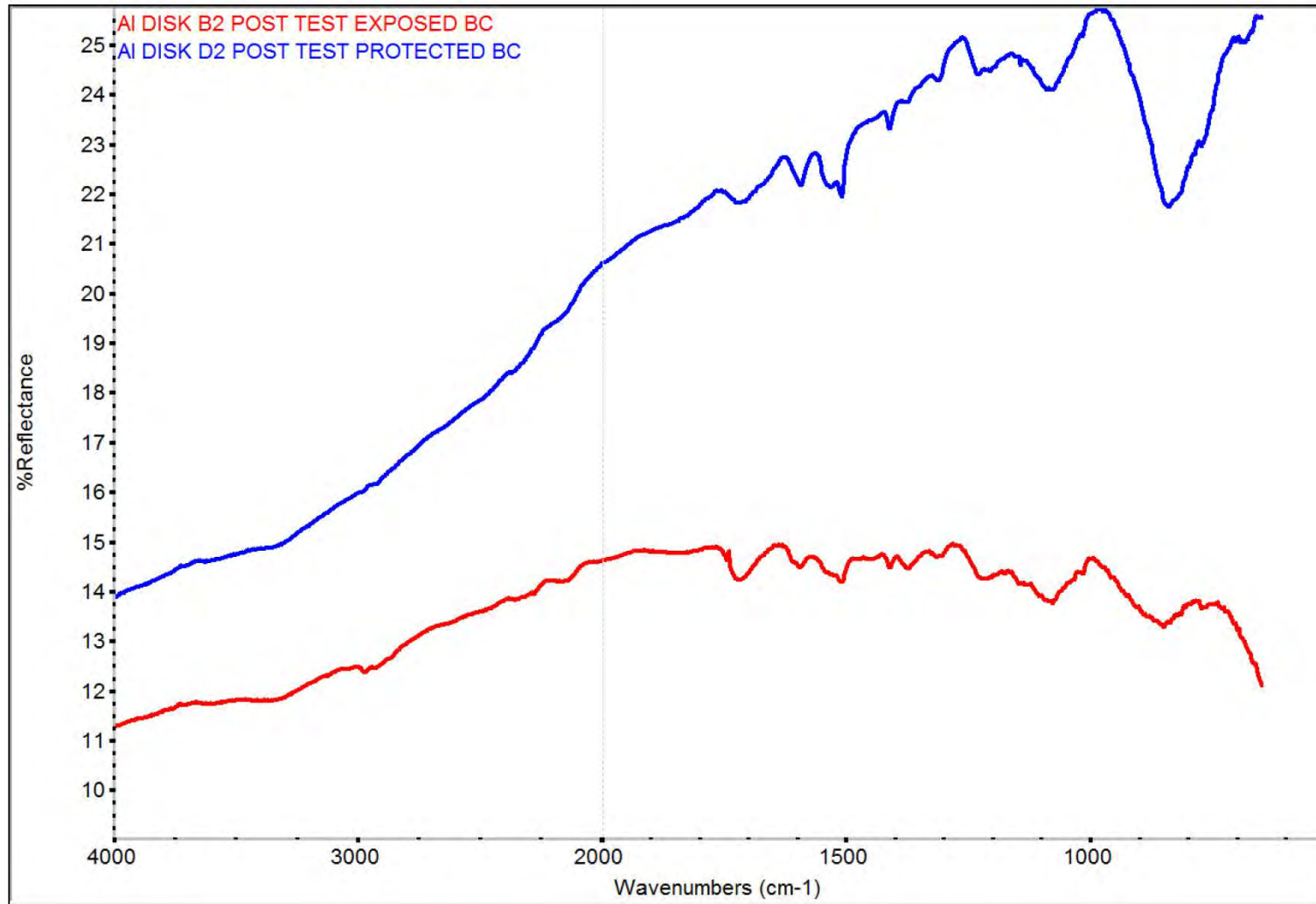


Significant darkening from 95% to 6% reflectance.



Witness Plate Post Test Aluminum Disks

Qualitative LWIR FTIR Biconical Reflectance



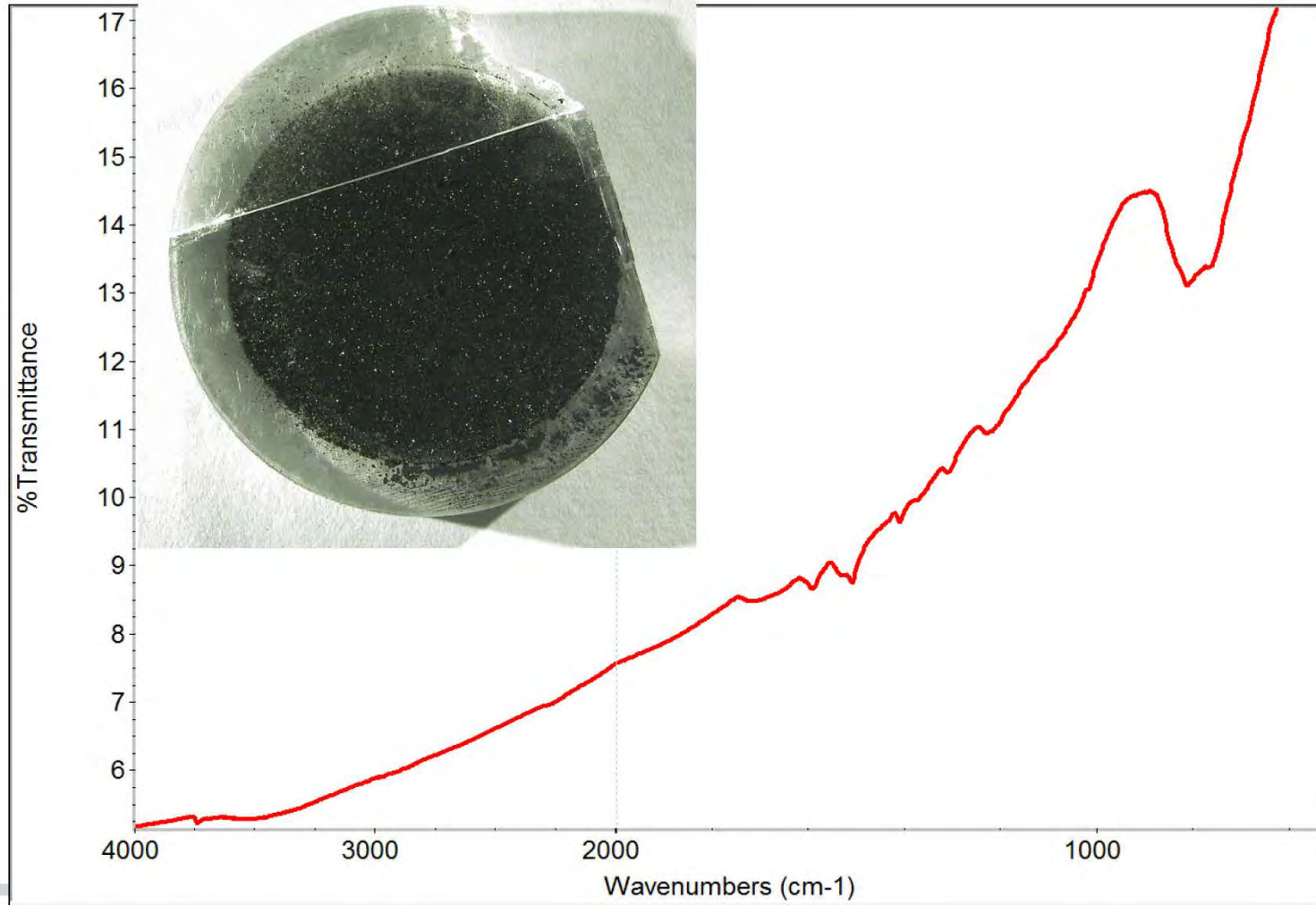
Soft catch contamination plus additional “oxide” band at 800 cm⁻¹. May be an aluminum oxide. Similar band seen in Debris-LV.



SBU Marking

Post Test: Protected NaCl Disk

LWIR FTIR Transmission

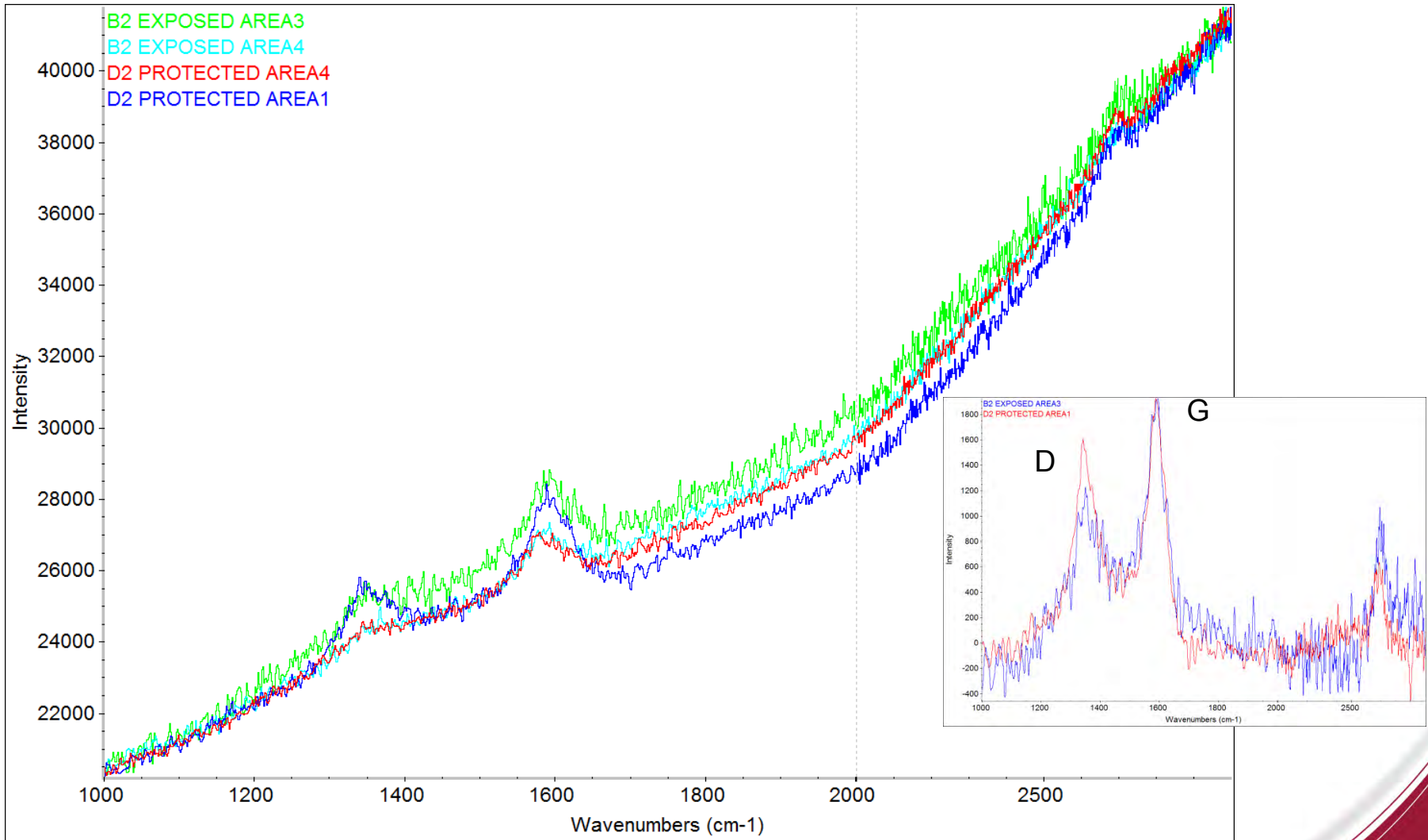


Significant darkening with absorption band at 815 cm⁻¹ similar to band seen by biconical reflectance.



Witness Plate Post Test AI Disks

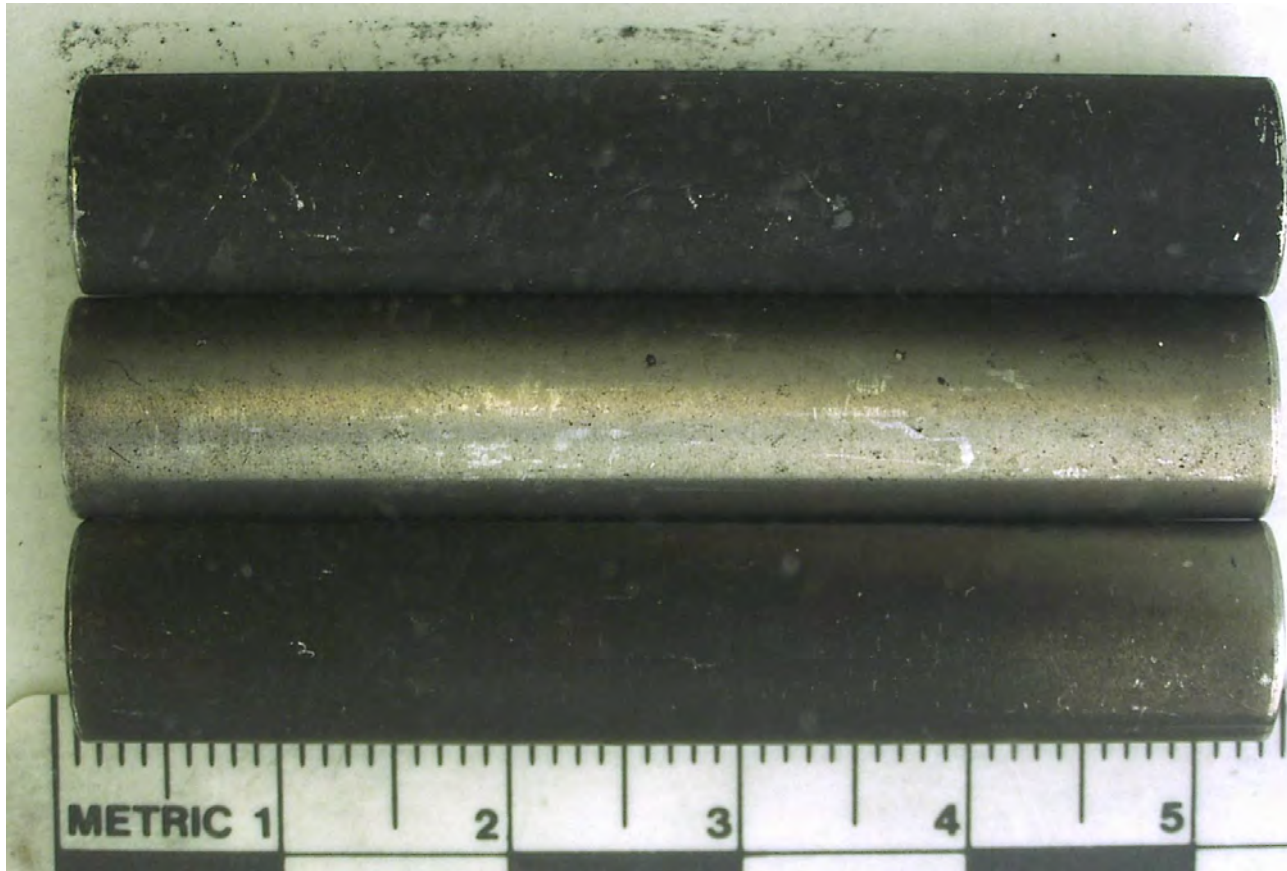
Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed in some areas. Similar to SEM stubs.



Witness Plate Whipple Plate Support Posts



Unrinsed covered
with black soot

Rinsed Side 1

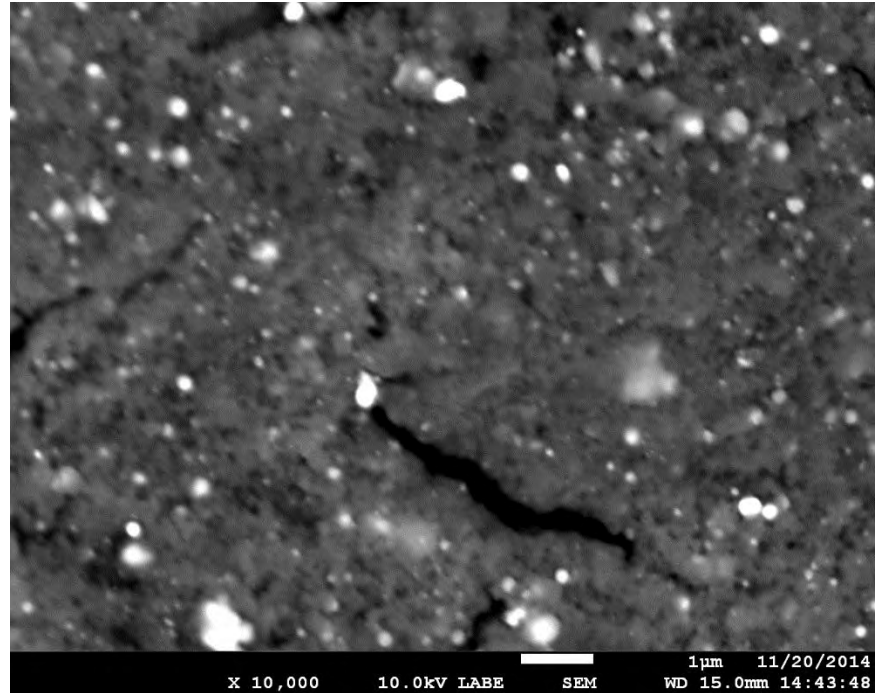
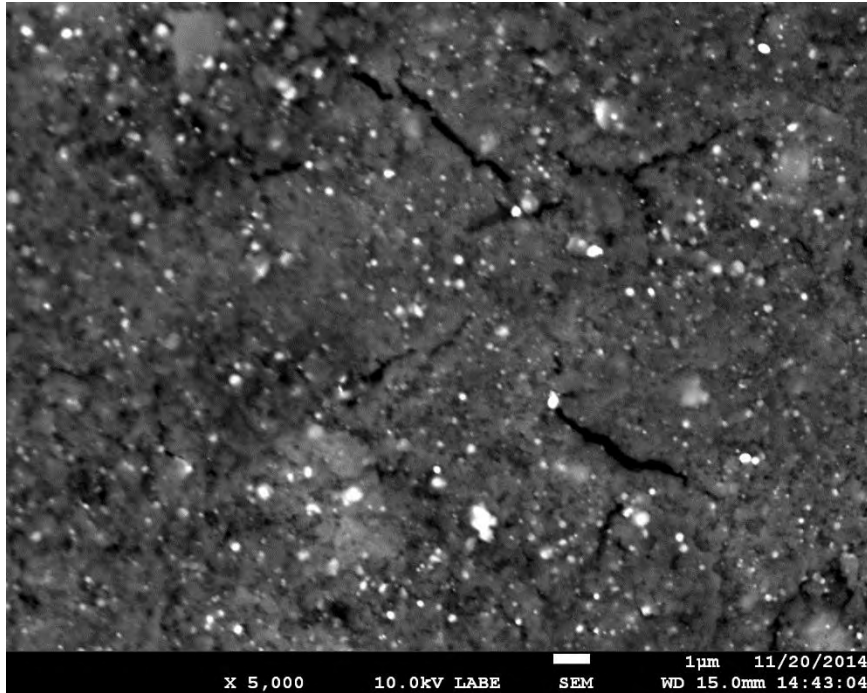
Rinsed Side 2

After rinsing with isopropyl alcohol the loose black soot is removed. One side is relatively clean but a resistant black coating remains on one side indicating directional deposition.



Rinsed Whipple Plate Support Post (surface)

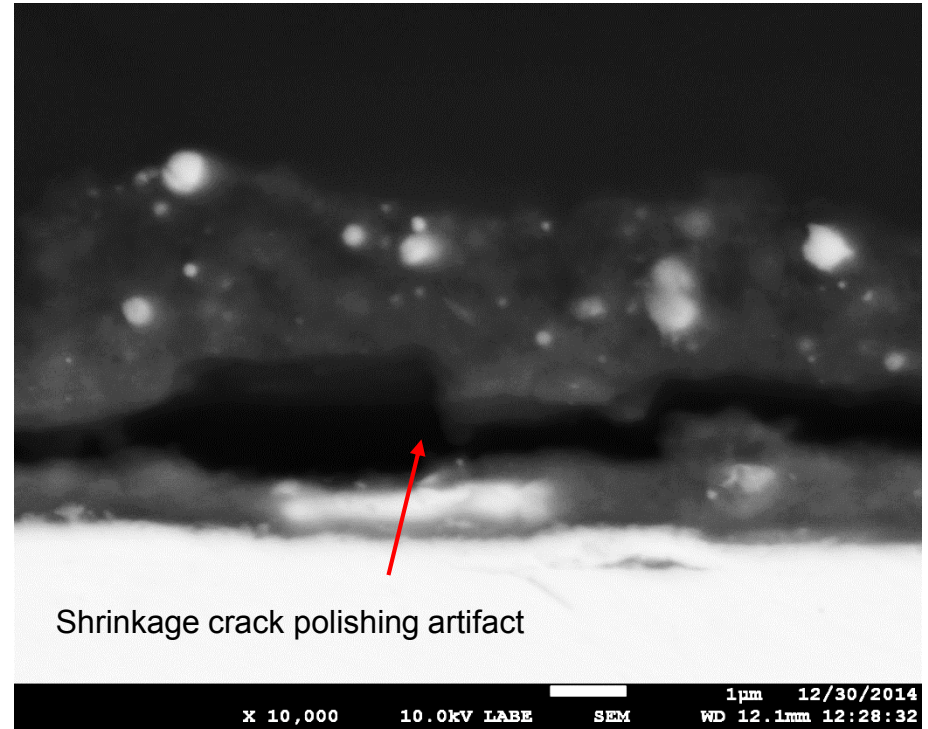
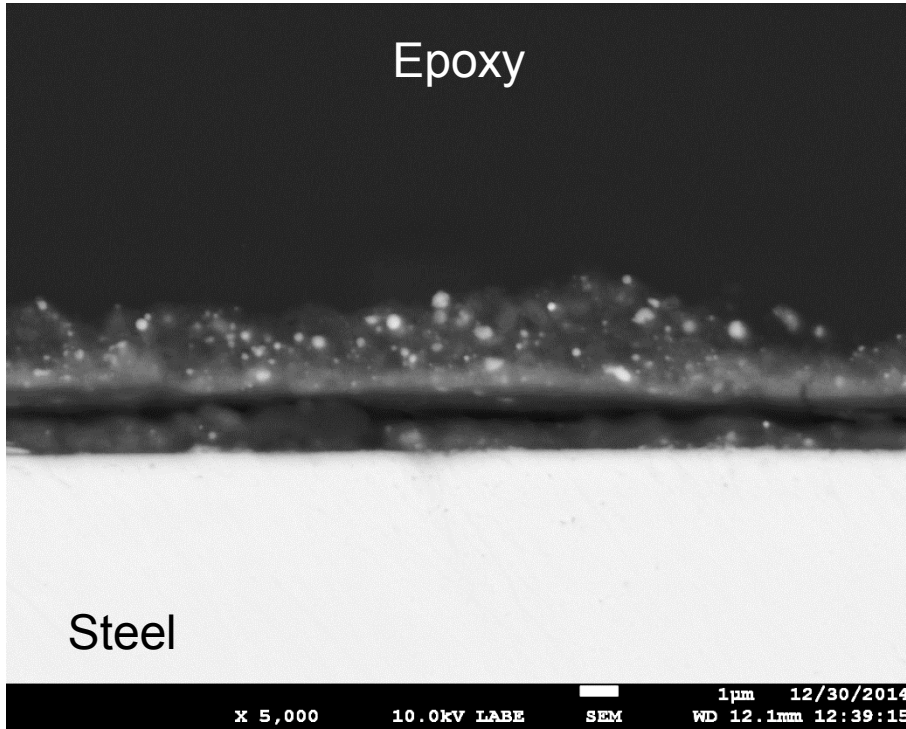
Backscatter SEM 5KX, 10KX



High Z (Al, Fe, Cu) nanoparticles embedded in carbonaceous matrix.



Rinsed Whipple Plate Support Post (polished cross section) Backscatter SEM 5KX, 10KX

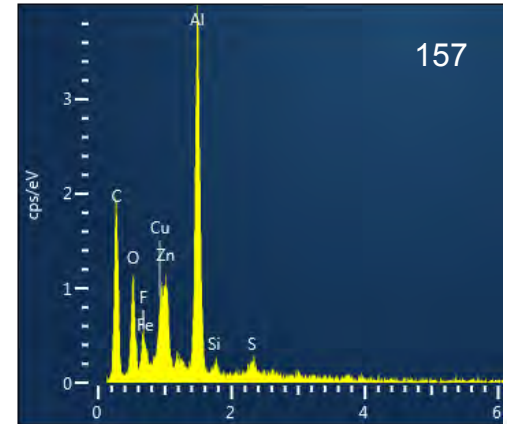
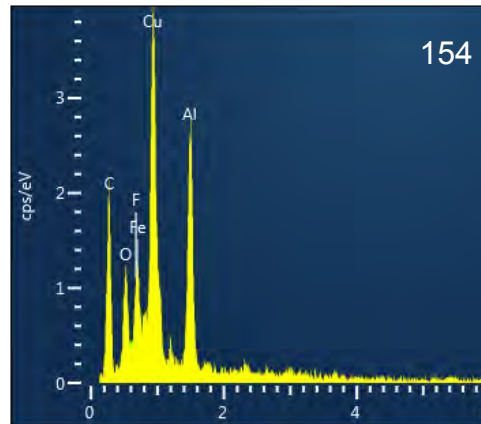
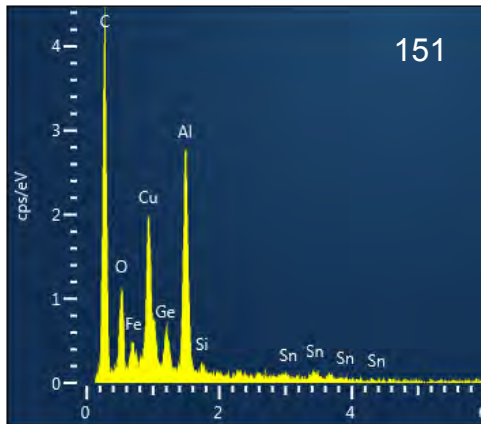
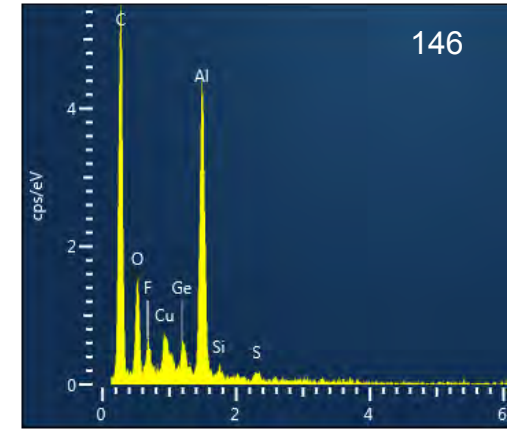
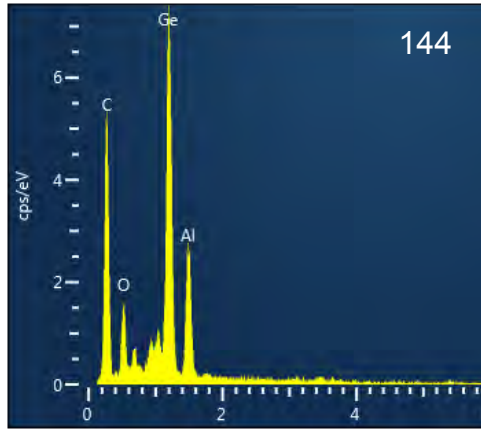
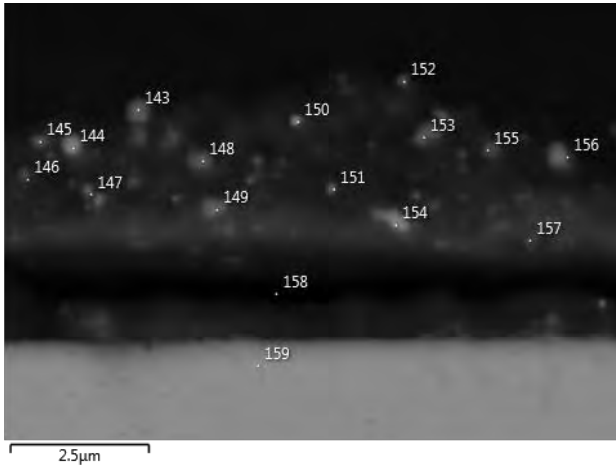


Metal nano droplets in a carbonaceous matrix 2-3 microns thick.



Rinsed Whipple Plate Support Post Deposit

Backscatter SEM EDS Spectra

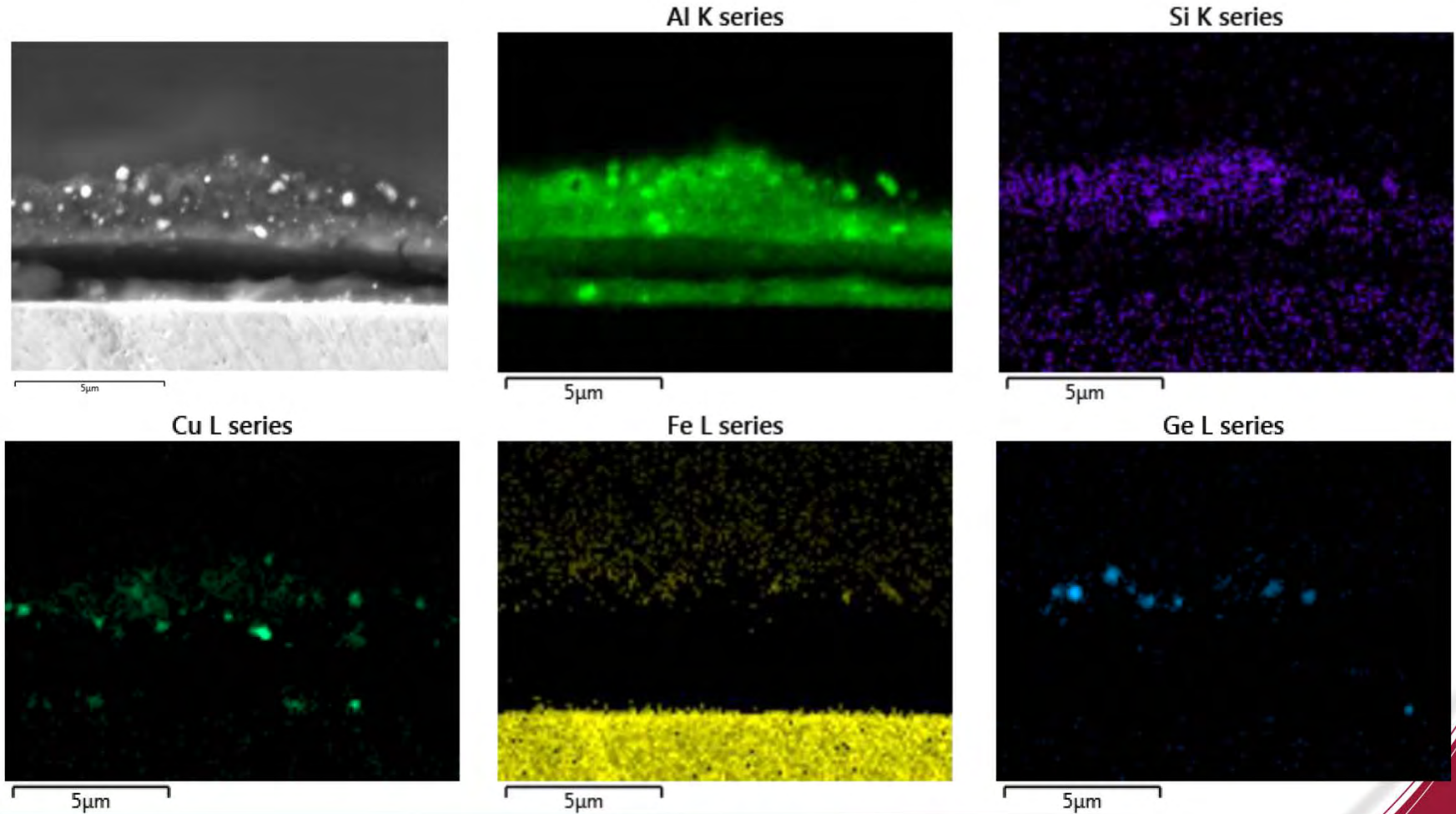


High Z nano particles in carbonaceous matrix contain Al, Cu, Ge, Fe and Zn.



Rinsed Whipple Plate Support Post Deposit

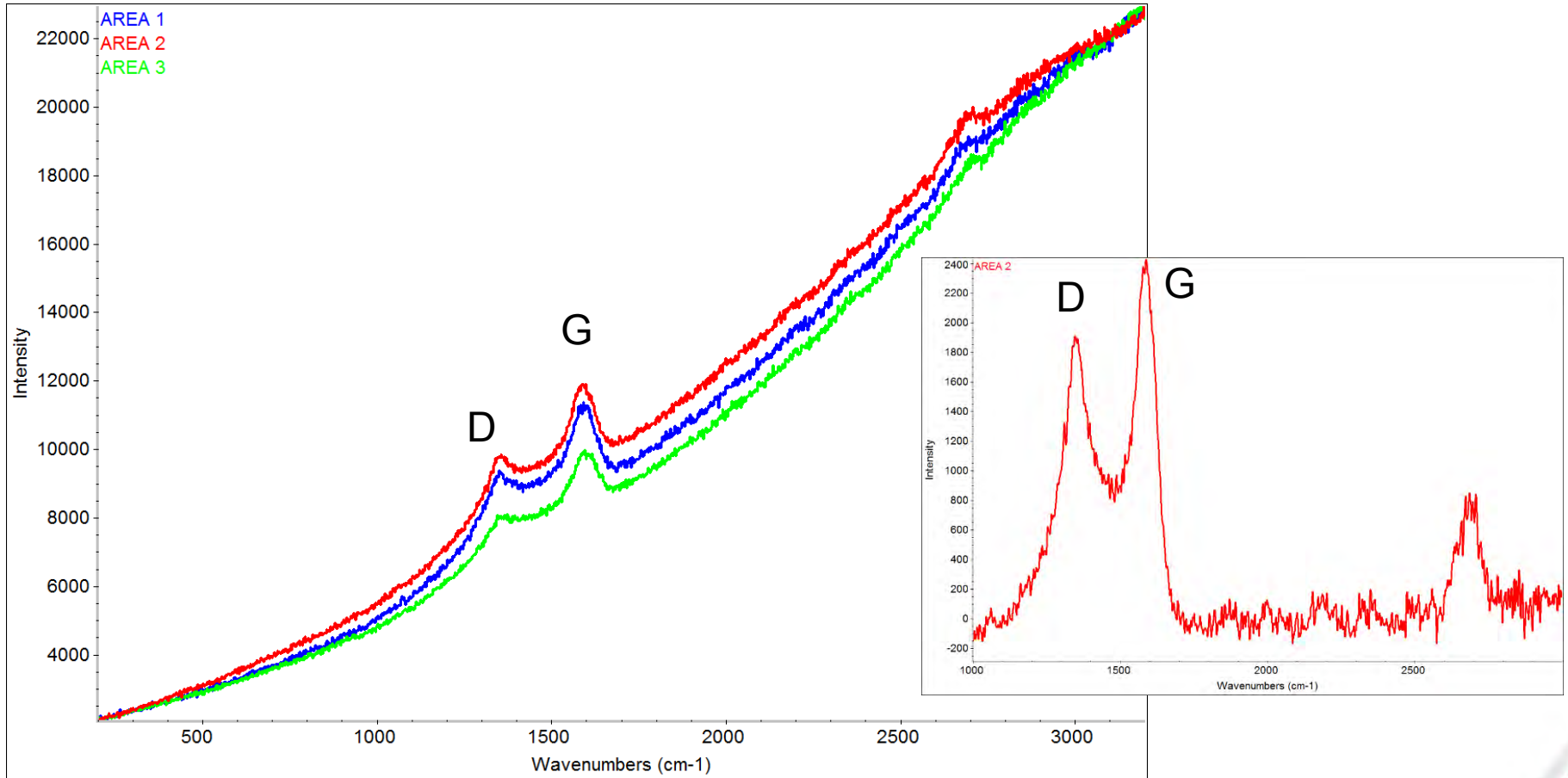
Backscatter SEM and EDS maps



High Z nano particles in carbonaceous matrix contain Al, Cu, Ge, Fe,



Witness Plate Spacer: Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed on dark side of spacer.



Summary of Observations

- SEM stubs, witness plate assembly and DebrisSat fragments were contaminated with soft catch fragments.
- SEM stubs also had a thin film of condensed soft catch vapor.
 - Similar to Debris-LV.
- Deposits on SEM stubs and witness plate assembly are predominantly C and O and consist of agglomerates of nano carbonaceous material.
 - Deposits are primarily from the soft catch – similar to Debris-LV
 - C-C composite honeycomb face sheets and MLI are also possible sources.
- Disordered graphitic carbon is present based on Raman spectra and TEM lattice fringe images. Similar to Debris-LV.
- The witness plate assembly was covered in a layer of loose “soot”, even under the protective Whipple plates.
 - Removal of the loose material by rinsing with isopropyl alcohol revealed a thin adherent coating on one side of the support posts indicating early directional deposition from DebrisSat.
 - Coating was carbonaceous (disordered graphite) with nano metal particles.



Summary of Observations (cont.)

- Fluorine from Teflon wire insulation is also common.
- Nano droplets of metallic materials (Al, Fe, Cu, Zn, Ge) are also present indicating melting as a result of impact. Nano metal droplets also seen in Pre Preshot and Debris-LV.
 - Al is from the Al honeycomb, nadir and zenith panels, structural core and COPV liner.
 - Aluminum oxide particles are also present.
 - Al projectile is another source of aluminum.
 - Fe is from SS tubing and solenoids.
 - Ge is from the solar cells
 - Cu is from wiring and solenoids
- Solidified molten nano metal droplets are crystalline based on TEM lattice fringes and consist of only a few crystallites.



Summary of Observations (cont.)

- Witness plates show a significant decrease in reflectance (95% to 6%)
- Soft catch contamination seen in LWIR reflectance spectra of DebrisSat fragments, SEM stubs and witness plate.
 - Not possible to get a clean spectrum of the debris generated by the hypervelocity impact
 - Additional “oxide” band at 800 cm^{-1} seen on some samples.
- Other laboratory analyses documented:
 - Aerospace TOR-2014-03201, Time-resolved Spectroscopy of Hypervelocity Impact Flash on DebrisSat, Gouri Radhakrishnan.
 - Aerospace ATM-2014-03659, DebrisSat Hypervelocity Impact Fragmentation Modeling, Naoki Hemmi



Appendix 1

Supplemental Information and Analyses



Introduction (cont.)

- Pre Preshot was conducted February 2014.
 - Validated performance of projectile to meet velocity goal of 7 km/s.
 - Confirmed operational status of test chamber and facility.
 - Target was primarily designed to catch the projectile.
 - Multi-shock shield supplied by NASA.
 - Multiple bumper panels of fiberglass, stainless steel mesh and Kevlar.
 - **No “soft catch “ panels were installed** (unlike Debris-LV and DebrisSat).
 - Test conducted with a pressure of ~1-2 Torr nitrogen.
 - A witness plate assembly was provided by Aerospace in order to catch and sample debris.
- Additional Documentation
 - Aerospace TOR- 2014-03082, DebrisSat Pre Preshot Laboratory Analyses, P. M. Adams and P. M. Sheaffer, December 23, 2014.



Introduction (cont.)

- Debris-LV (Pre Shot) conducted 1 April 2014
 - Further validated performance of projectile and facility and served as a dress rehearsal for the DebrisSat test.
 - The 15 kg target consisted primarily of empty tanks and was constructed by Patti Sheaffer from materials representative of a launch vehicle (LV) upper stage.
 - Primarily aluminum and titanium with lesser amounts of copper and stainless steel.
 - Test chamber was lined with “soft catch” foam panels to trap fragments for size distribution analysis.
 - A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis.
 - Aerospace also placed SEM stub witness plates into soft catch for post test retrieval and analysis.
- Additional documentation
 - Aerospace TOR-2014-03577, Debris-LV Hypervelocity Impact Post-Shot Physical Results Summary, P. M. Sheaffer.
 - Aerospace TOR-2015-00928, Debris-LV Laboratory Analyses, P. M. Adams, P. M. Sheaffer, Z. R. Lingley and G. Radhakrishnan.



Laboratory Instrumentation

- Field Emission Scanning Electron Microscopy (FESEM)
 - JEOL JSM-7600F SEM.
 - In-lens secondary electron detector (SEI mode).
 - High resolution imaging.
 - Lower secondary electron detector (LEI mode).
 - Less charging.
 - Enhances topography.
 - Backscatter electron detector (LBE mode)
 - Atomic number (Z) image contrast.
- Energy Dispersive (X-ray) Spectroscopy (EDS) in the SEM/TEM
 - Oxford X-Max silicon drift detector (SEM).
 - Oxford SiLi detector (TEM)
- Transmission Electron Microscopy (TEM)
 - JEOL JEM-3100F TEM.
 - Oxford INCA EDS.



Laboratory Instrumentation (cont.)

- Fourier Transform Infrared (FTIR) spectroscopy
 - Nicolet 6700 spectrometer.
 - Harrick Scientific “praying mantis” diffuse reflectance accessory.
 - Qualitative reflectance.
 - Mercury cadmium telluride (MCT) detector.
 - Fast analysis with excellent signal to noise.
 - Can only analyze small samples (< 1”).
 - Labsphere hemispherical reflectance accessory.
 - Quantitative reflectance.
 - Long scan time with poor signal to noise.
- Raman Spectroscopy
 - Renishaw inVia Raman microscope.
- UV-VIS-NIR Spectroscopy
 - Perkin Elmer Lambda 900 Spectrometer
 - Diffuse transmission and reflectance with integrating sphere.



Considerations for SEM and EDS Analyses

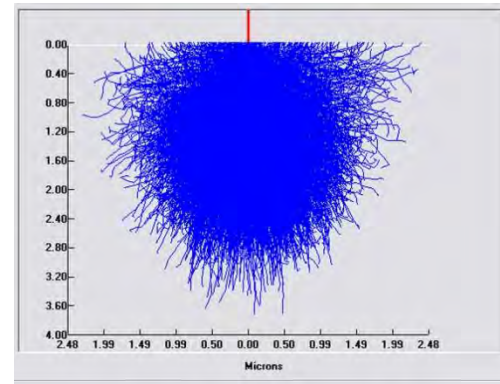
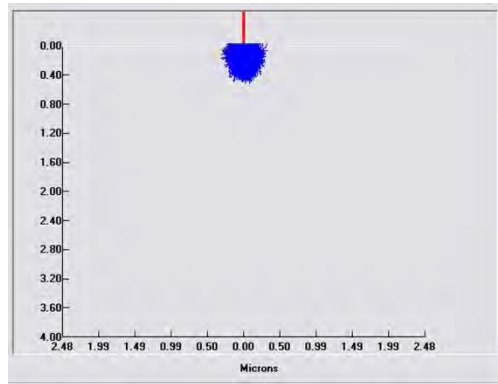
- A preliminary look at Debris-LV and DebrisSat SEM stubs indicated that a thin coating, containing low atomic number (Z) materials (carbon), was present.
- This presented challenges and options for SEM-EDS analyses. Low vs. high voltage (5 KV vs. 15 KV).
- Advantages of 5 KV: high resolution imaging of thin low Z surfaces, EDS from surface material and not substrate.
- Disadvantages of 5 KV: beam does not penetrate beneath the surface (subsurface is hidden), does not excite X-rays from medium Z elements (Cl to Ti), high Z element K series not excited but L series X-rays are.
 - Most medium Z elements not expected .
- Advantages of 15KV: Can see a limited distance beneath the surface, excites X-rays from just about all elements.
- Disadvantages of 15 KV: loss of detail in low Z surfaces, for thin coatings may penetrate through to the substrate

Use both 5KV to 15 KV to obtain more comprehensive information.

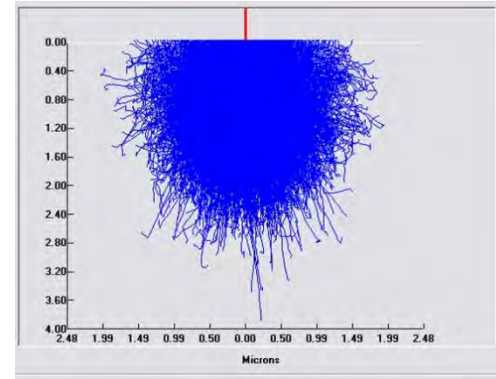
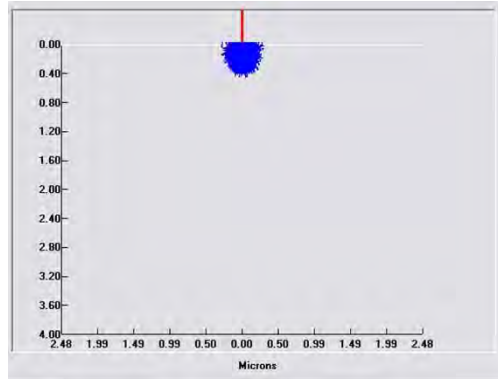


Electron Penetration into Materials

C



Al



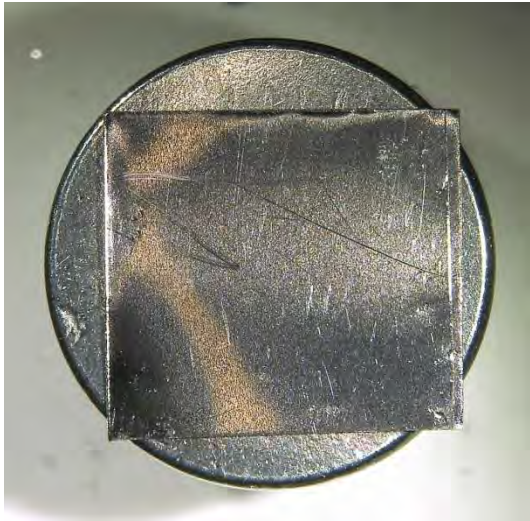
5 KV

15 KV

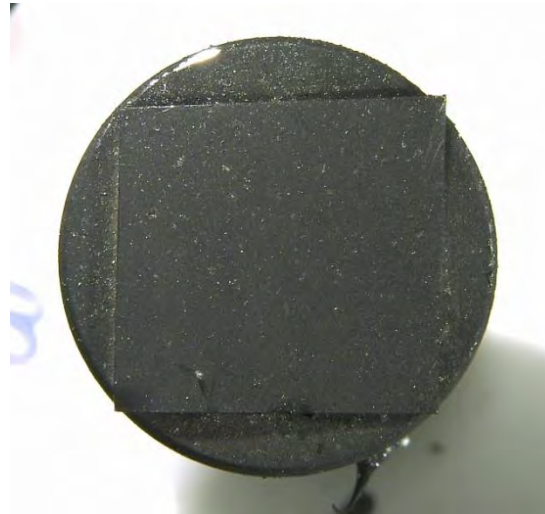
- Electron penetration influences depth of X-ray generation (EDS).
- Secondary emitted electrons have low energy and image primarily the surface.
- Backscattered emitted electrons have greater energy and image partially into the surface and have atomic number information.



SEM Stubs



Unexposed



Exposed #13



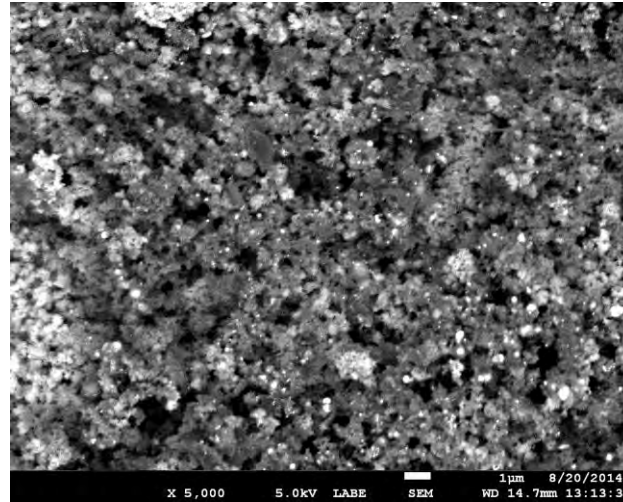
Exposed #14

Note significant darkening of post test stubs

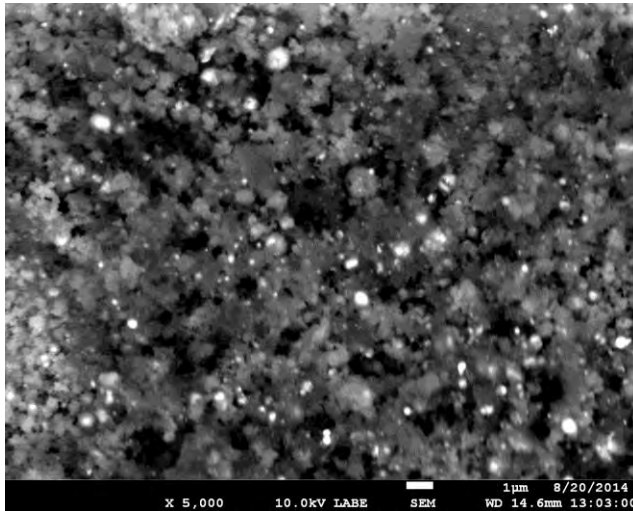


SEM Stub 4: Backscatter Electron SEM (5 KX)

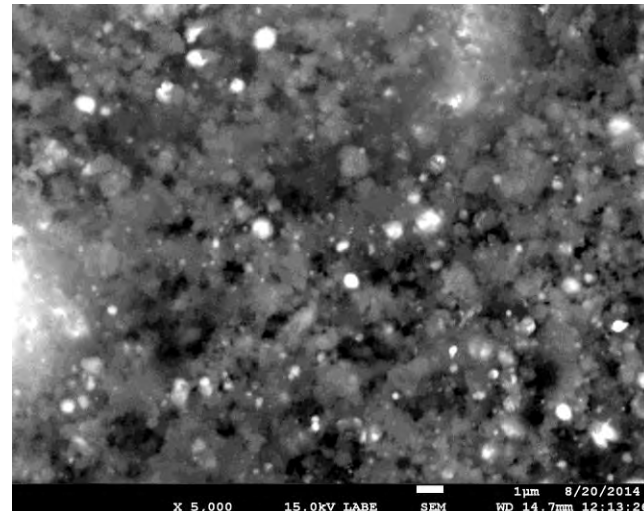
Brighter areas are higher atomic number (Z).



10 KV



15 KV

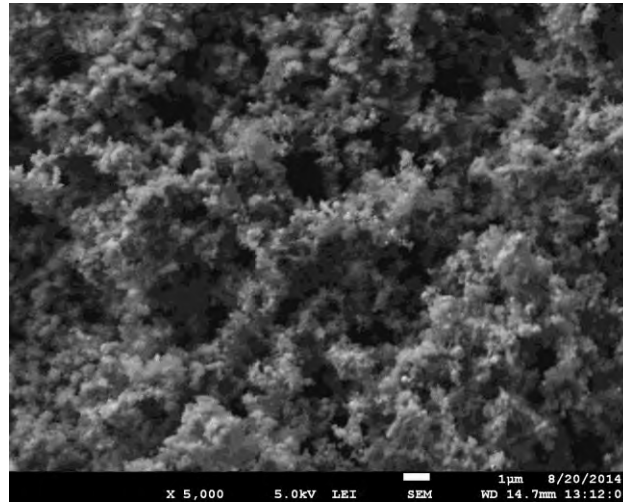


Images are from the same area.

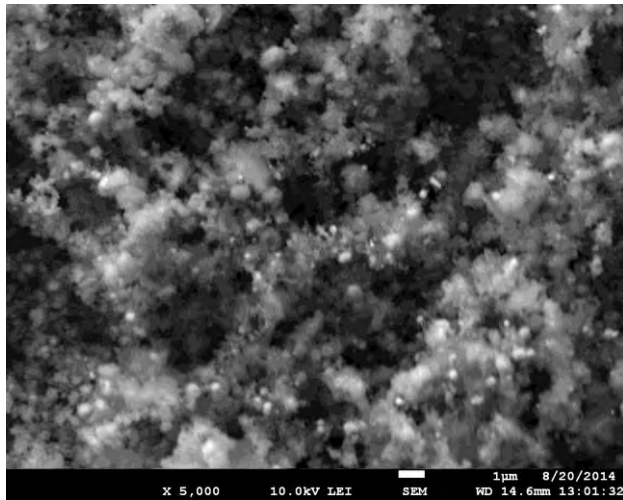
Greater penetration and less surface detail with increasing energy.



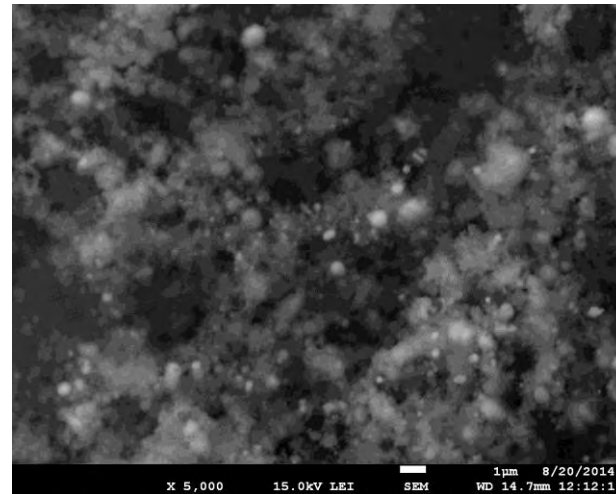
SEM Stub 4: Secondary Electron SEM (5KX)



10 KV



15 KV



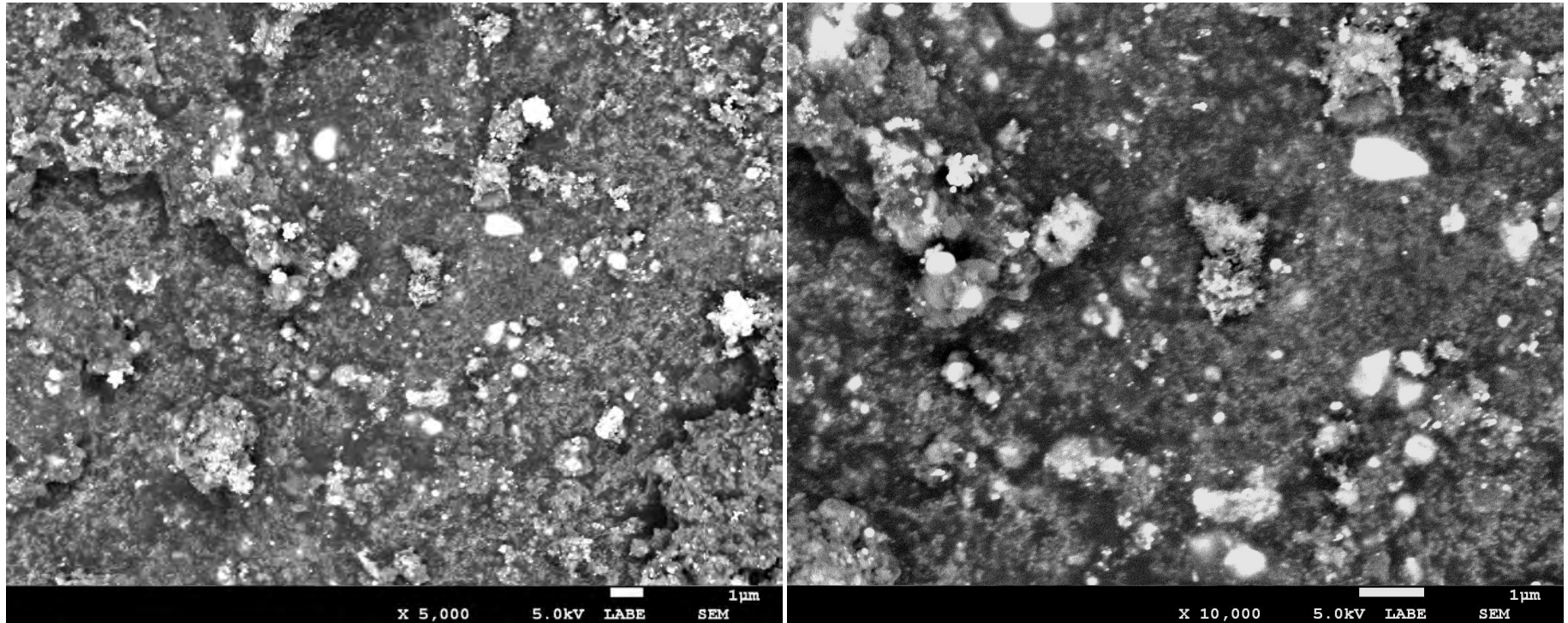
Images are from the same area as previous slide.

Greater penetration and less of surface detail with increasing energy.



SEM Stub 13

Backscatter SEM (5KX, 10KX)



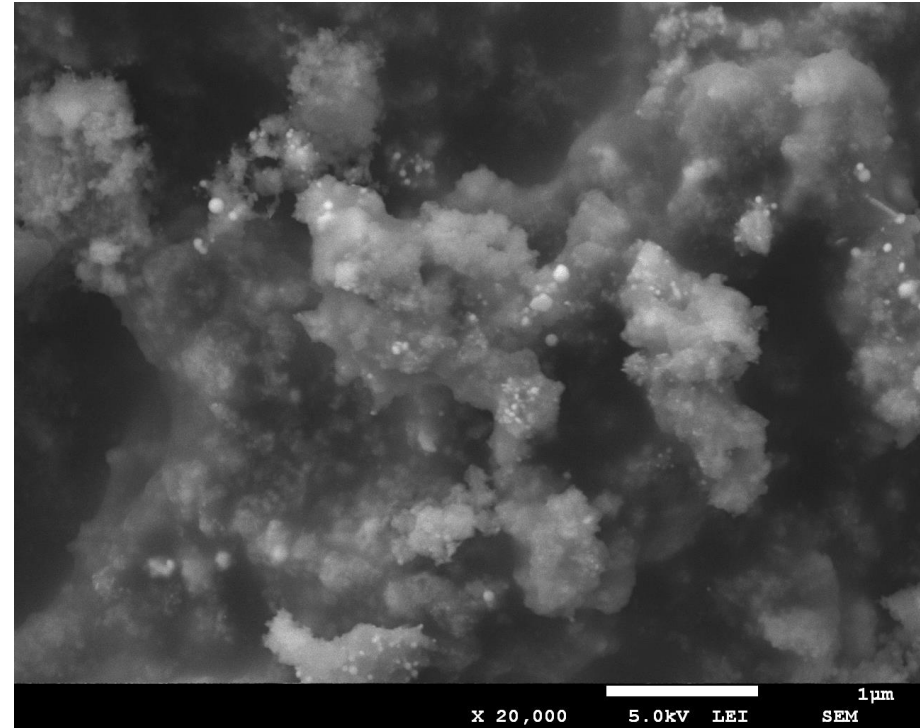
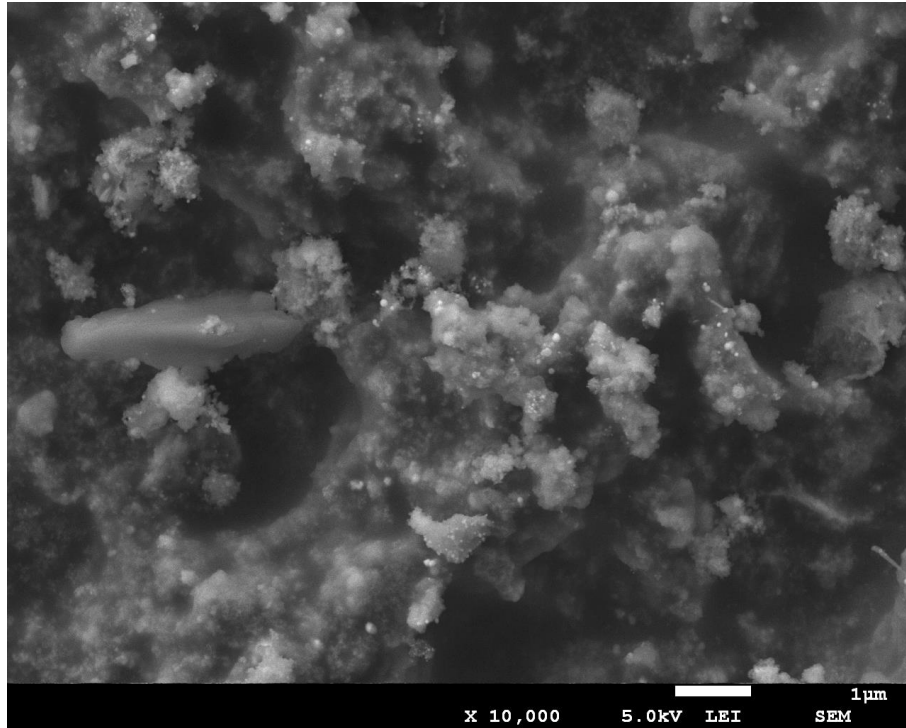
Material on surface consists of nano-sized agglomerates

Located 11 feet down range – 3 O'clock



SEM Stub 13

Secondary Electron SEM (10KX, 20KX)

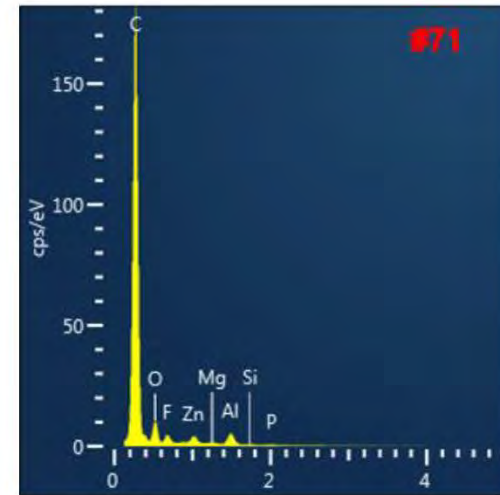
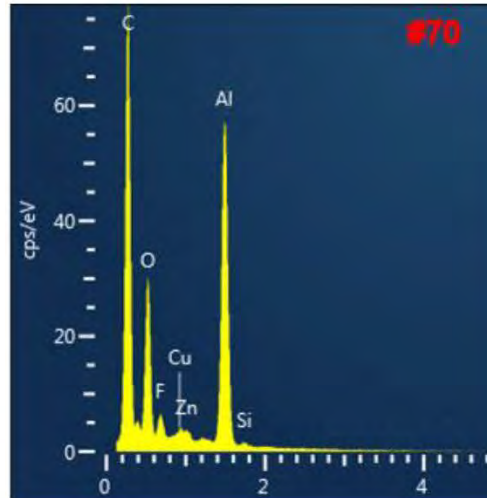
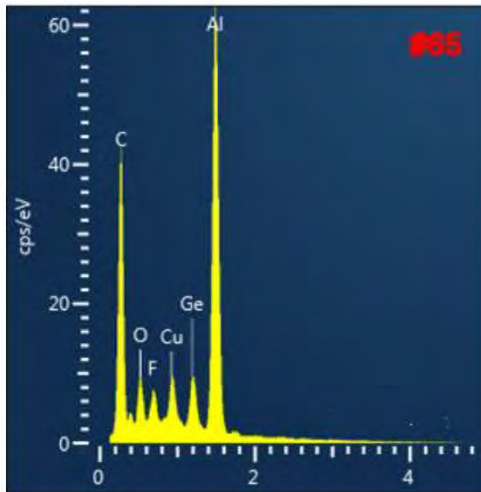
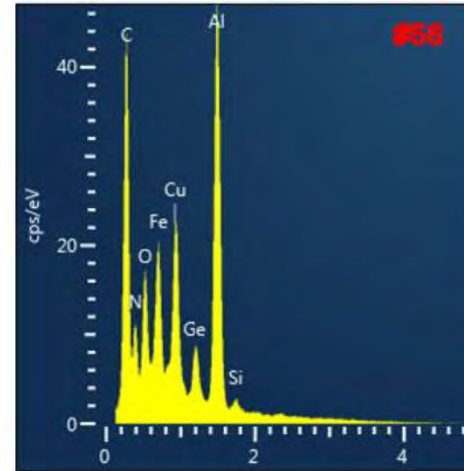
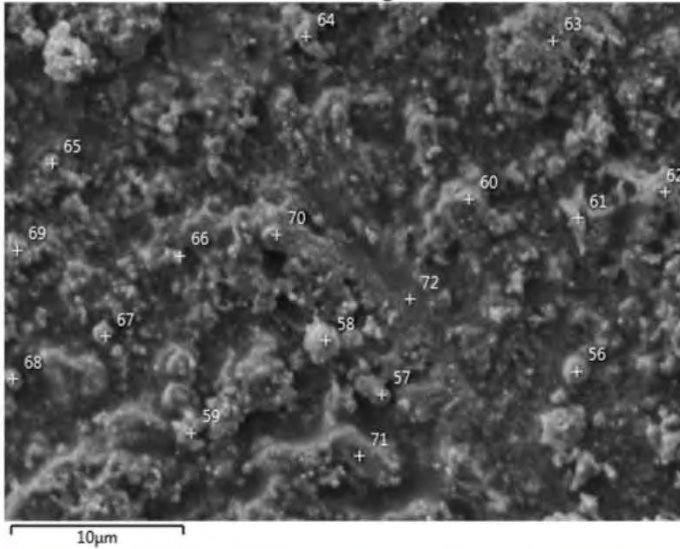


Note nano-scale structures



SBU Marking SEM Stub 13

SEM EDS

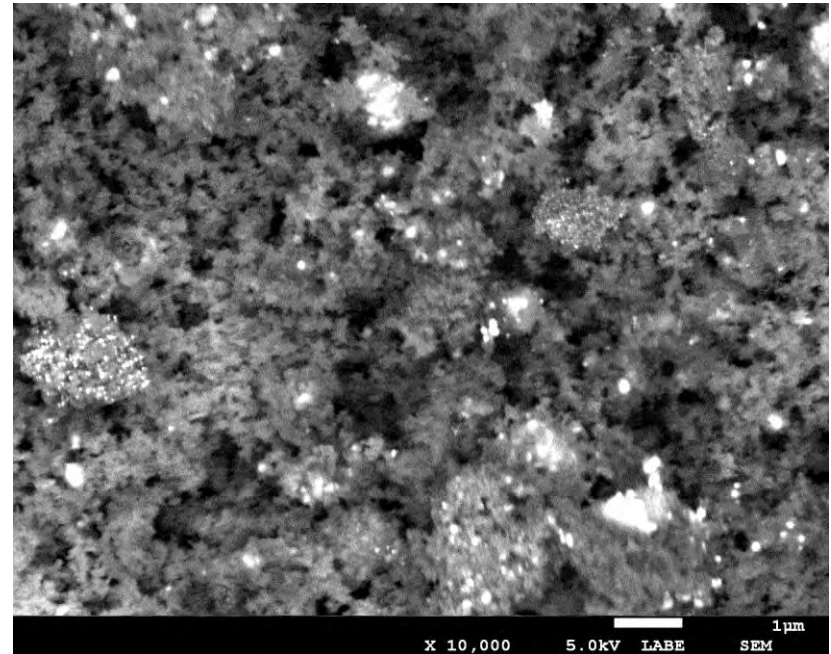
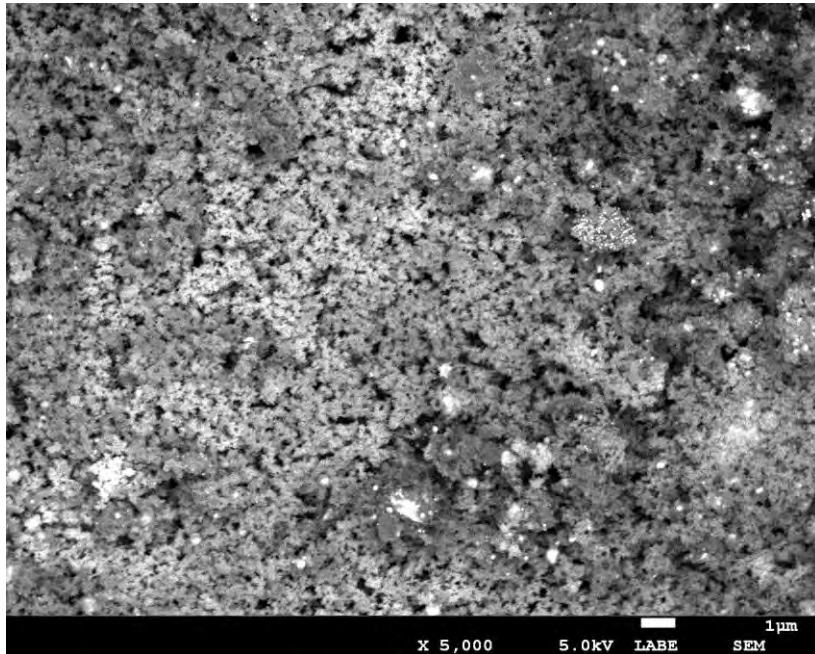


Al, C, Cu, O, Fe, and Ge are common.



SEM Stub 14

Backscatter SEM 5KX, 10KX

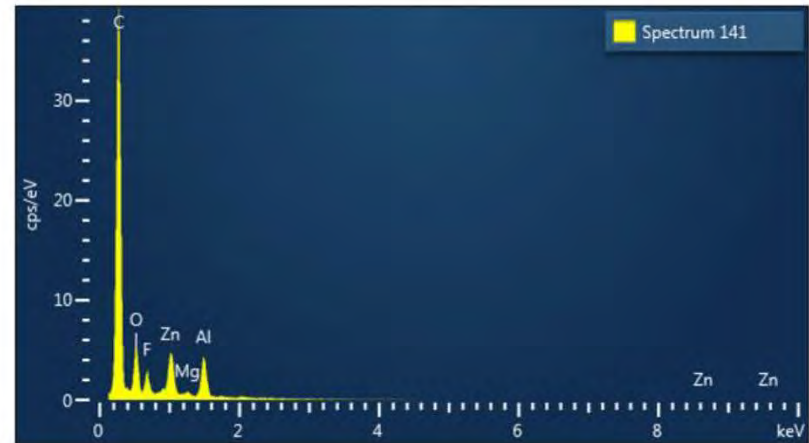
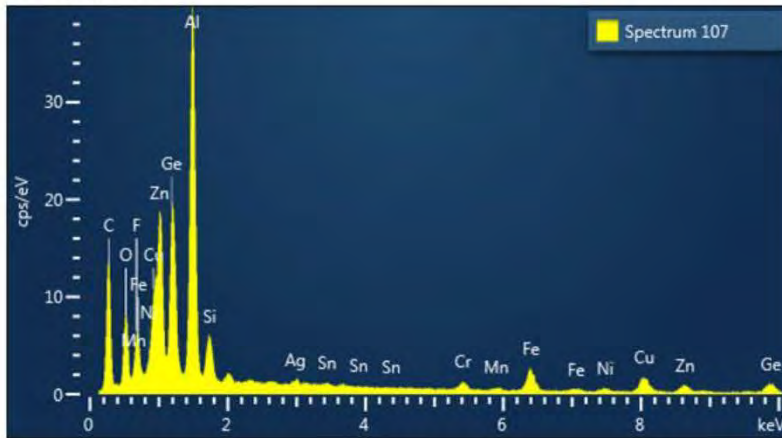
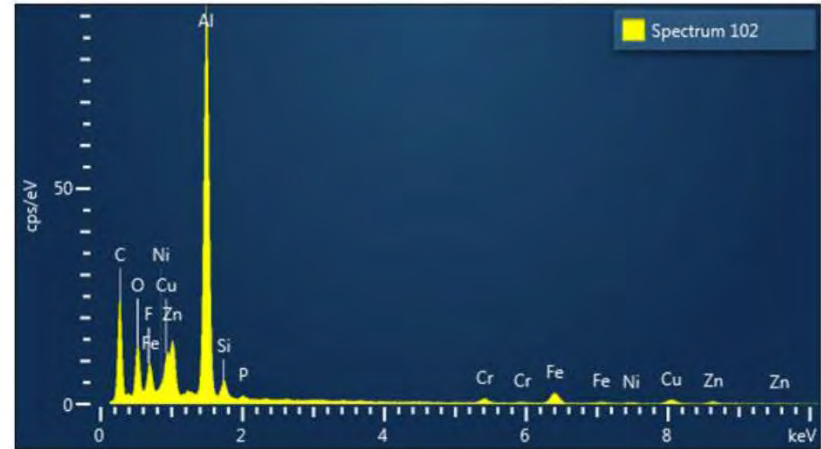
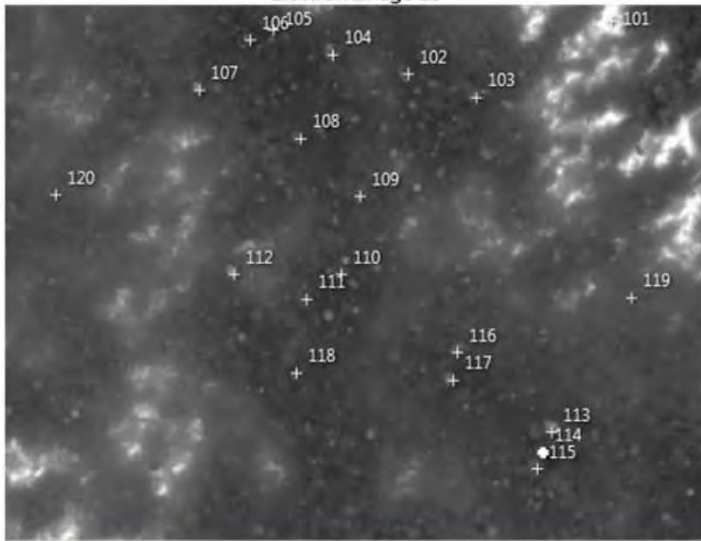


Material on surface consists of nano-sized agglomerates

Located 15 feet down range – 3 O'clock



SBU Marking SEM Stub 14 SEM EDS (15 KV)



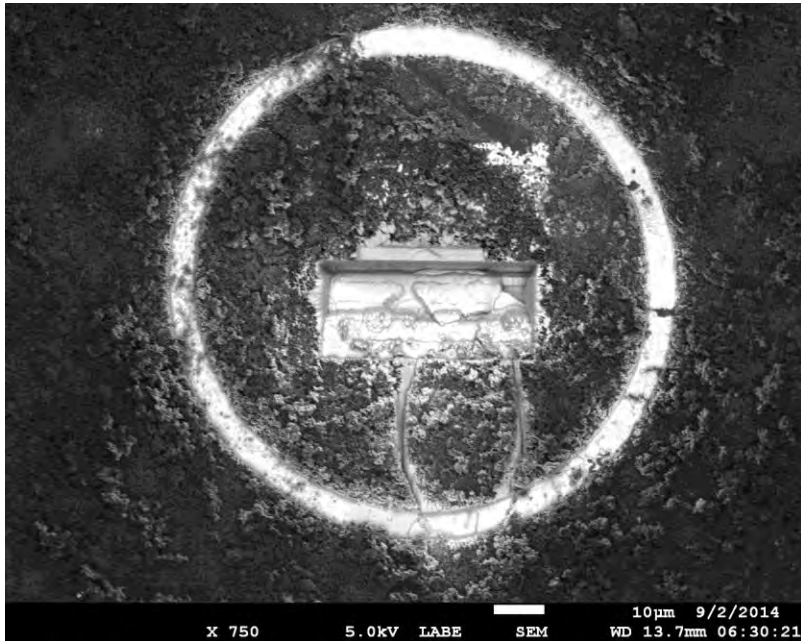
Al, C, Cu, O, Fe, Zn and Ge are common.



SEM Stub 14: Focused Ion Beam (FIB) Cross Section Backscatter SEM

Circle trenced down to Ta to facilitate site location

Protective Pt layer deposition



Vertical wall

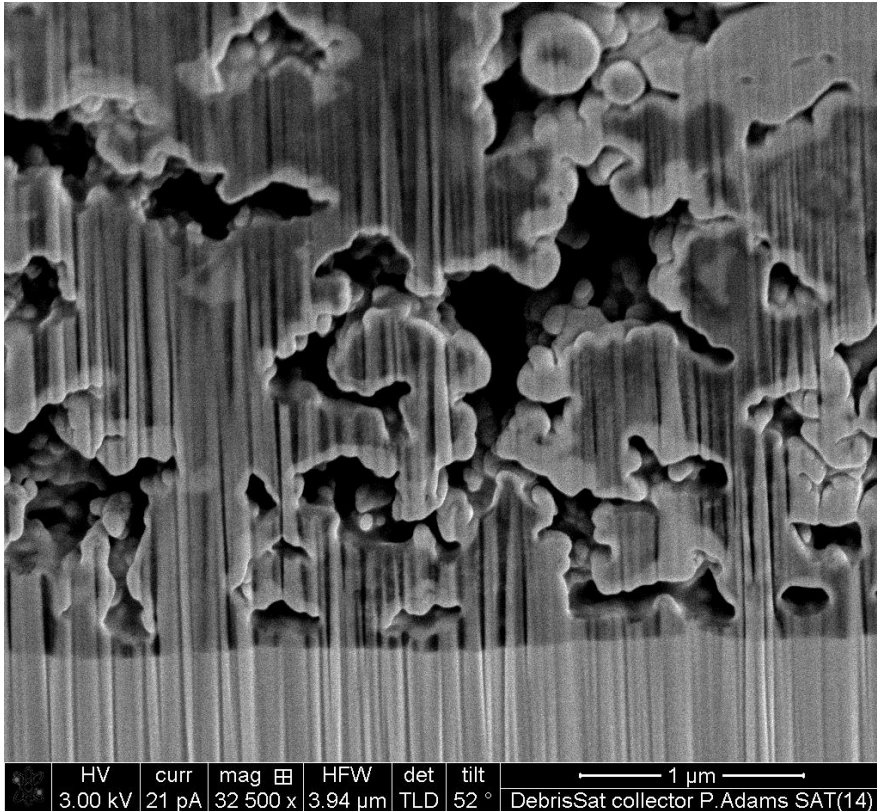
50 deg angled trench

Plan View

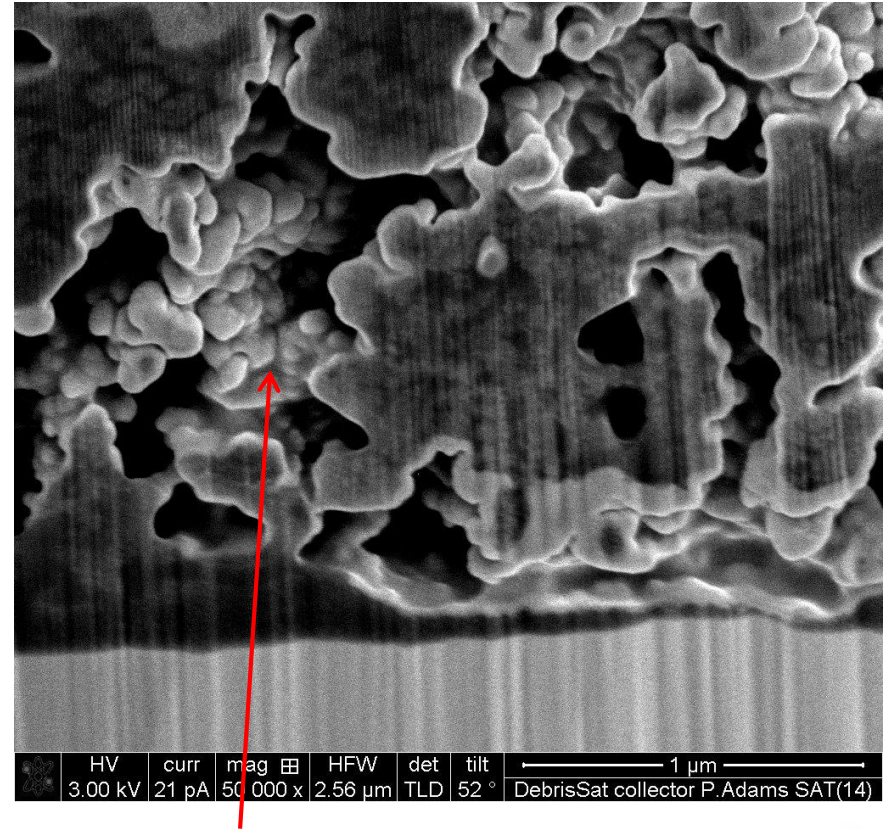


SEM Stub 14: FIB Cross Section

SEM 32.5KX, 50 KX



Striations (curtains) are artifacts of FIB preparation.



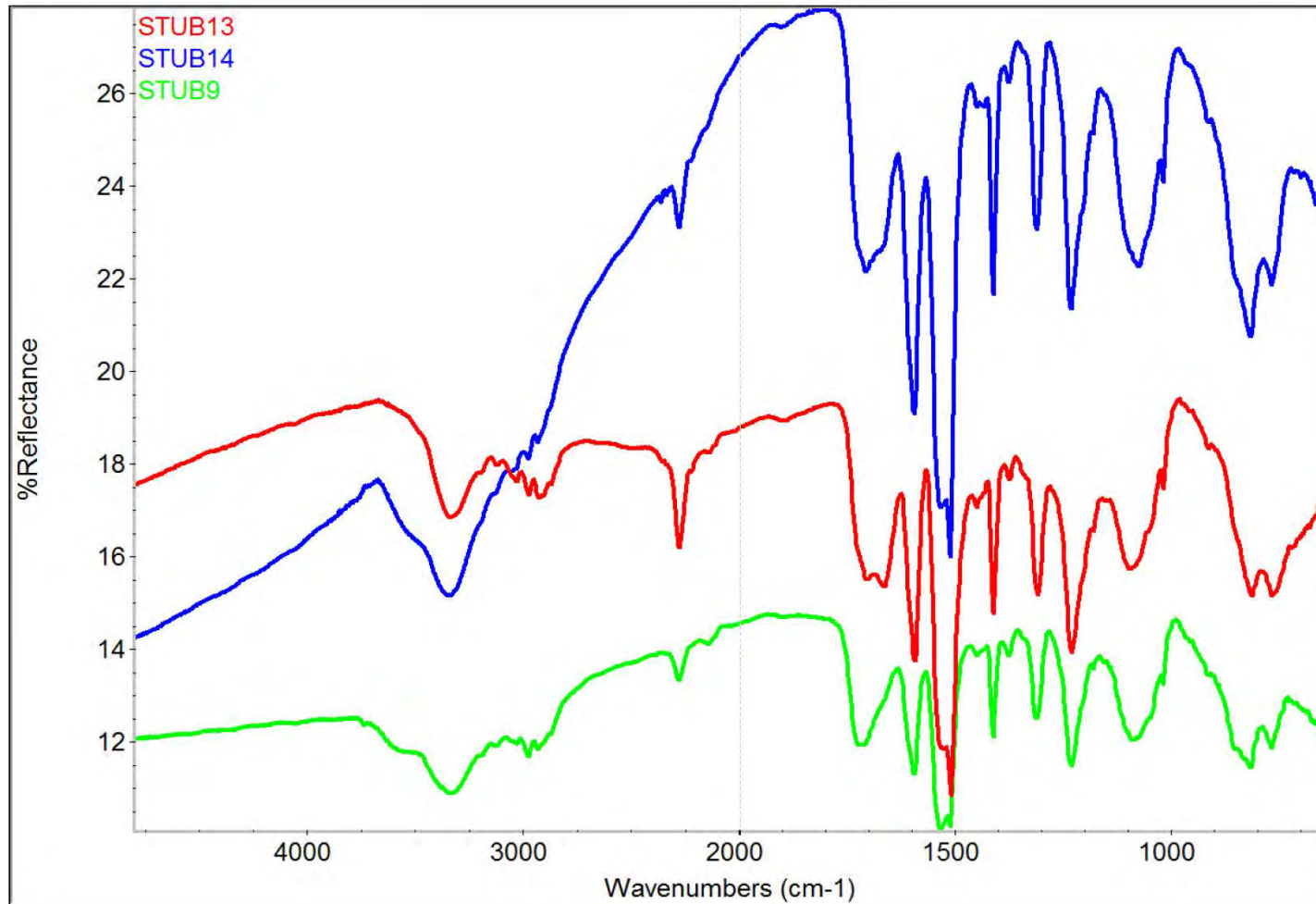
Back deposition of Ta from trenching into voids?

View of Side Wall



SBU Marking Post Test: SEM Stubs

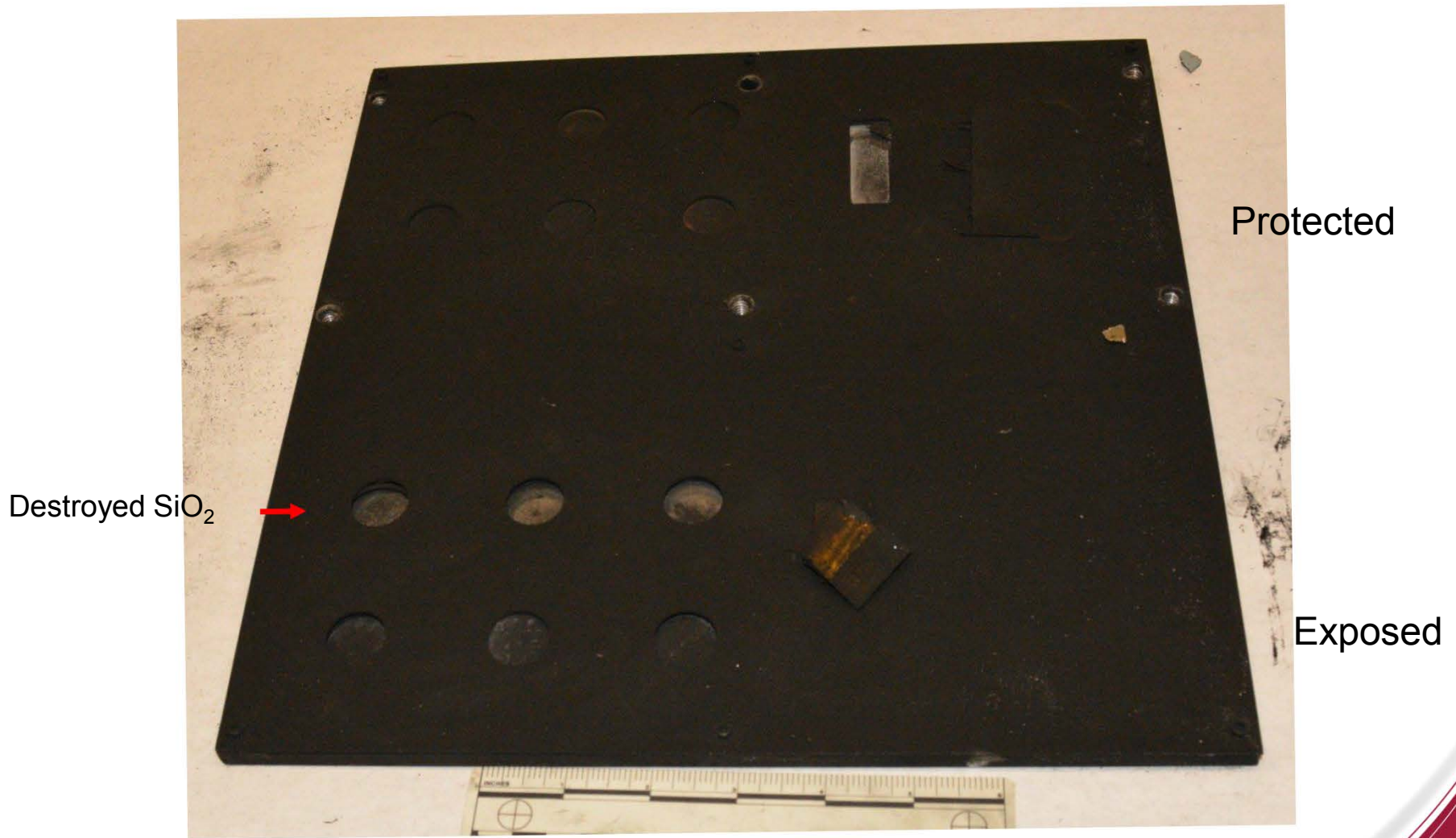
FTIR – Qualitative Diffuse Reflectance



Significant soft catch signature is present on SEM stubs.



SBU Marking Witness Plate Assembly: Post Test

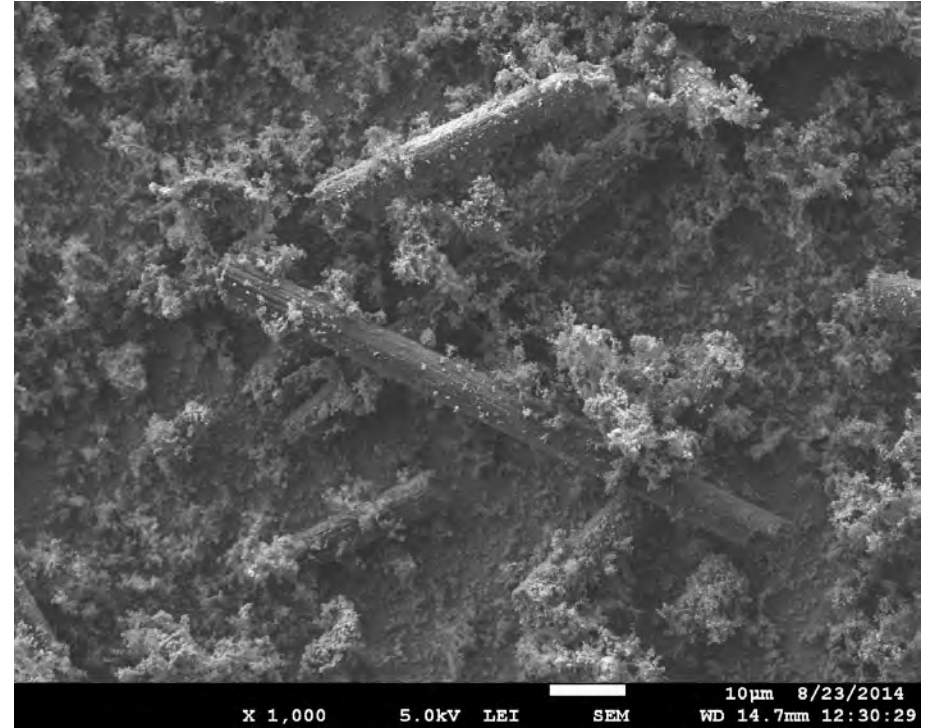
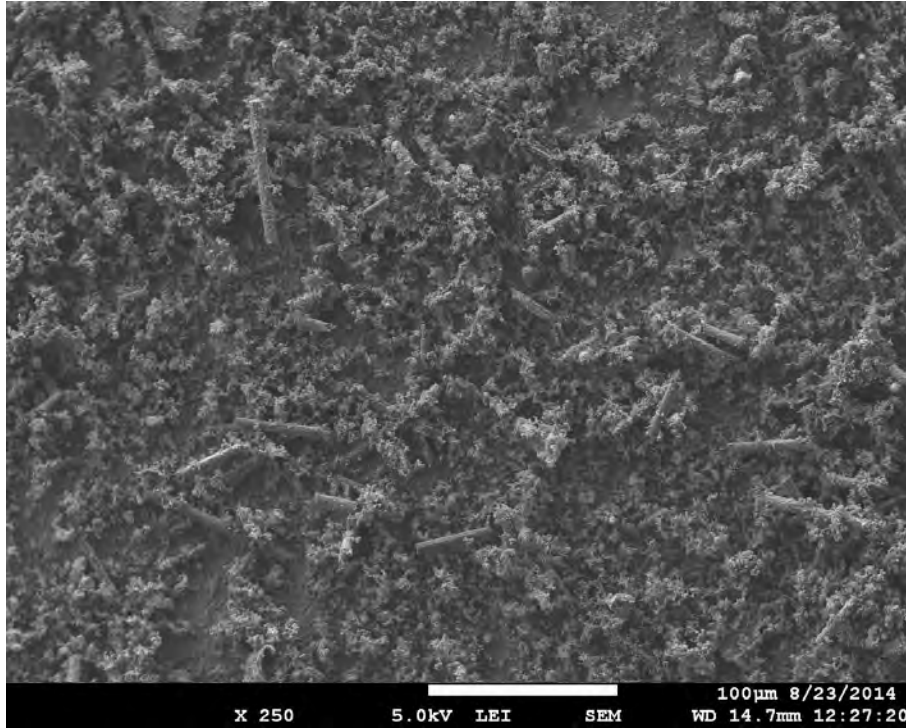


Witness plate is completely covered in black sooty debris – even protected areas under the Whipple plates.



Witness Plate Post Test : Aluminum Disk D2 (protected)

Secondary Electron SEM Images (250X, 1KX)

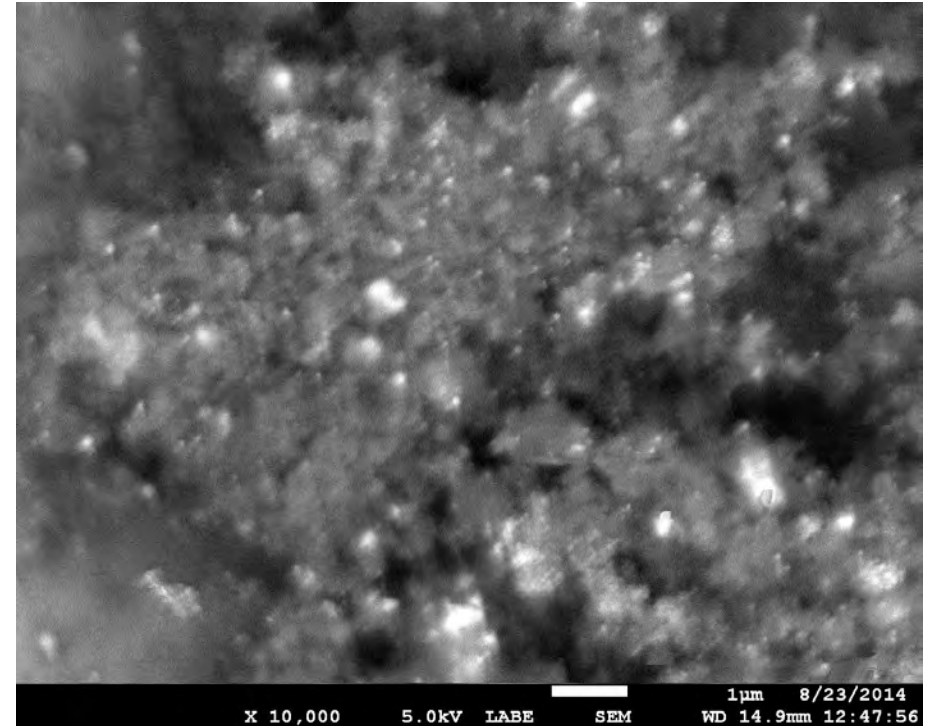
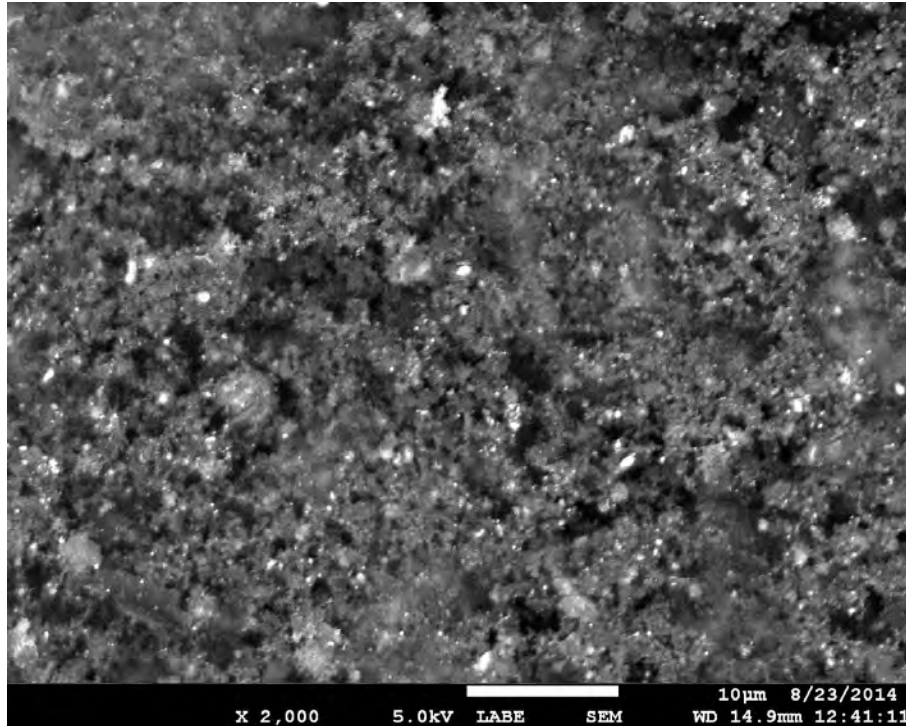


Note fragments of carbon fibers.



Witness Plate Post Test: Aluminum Disk D2 (protected)

Backscatter Electron SEM Images (2KX, 10KX)

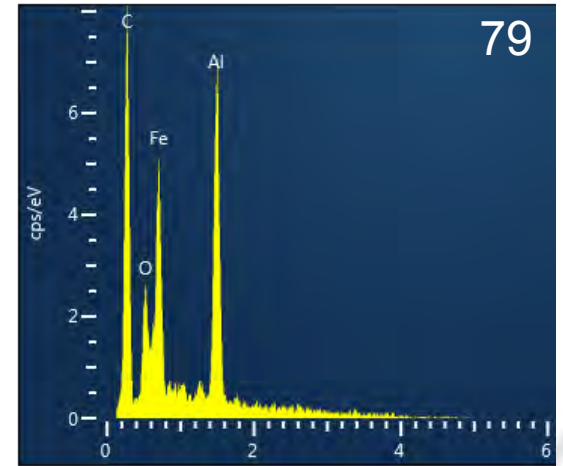
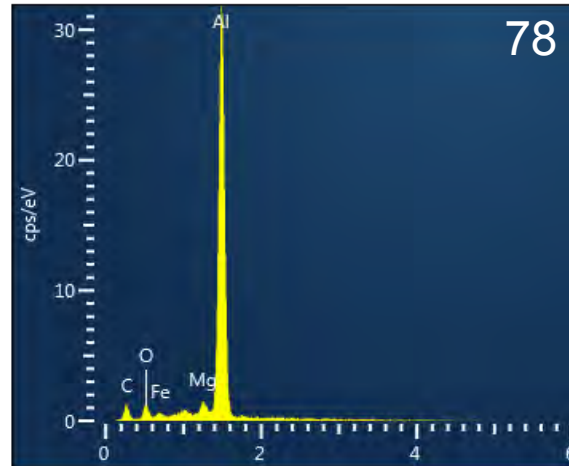
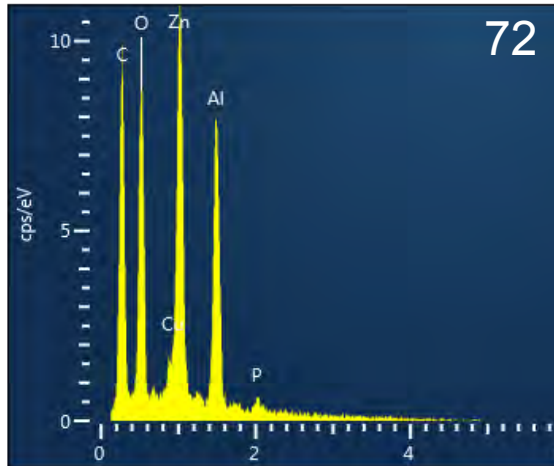
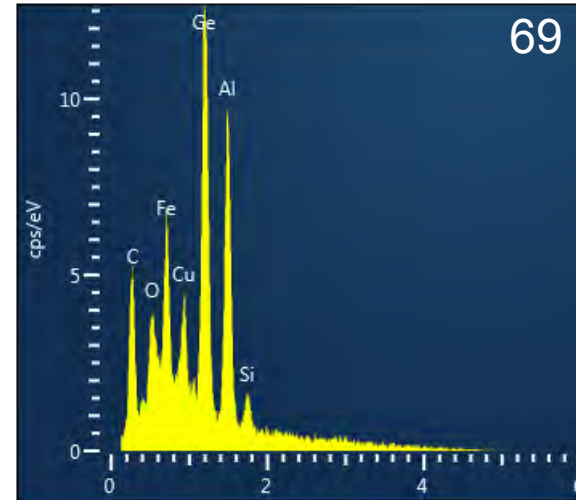
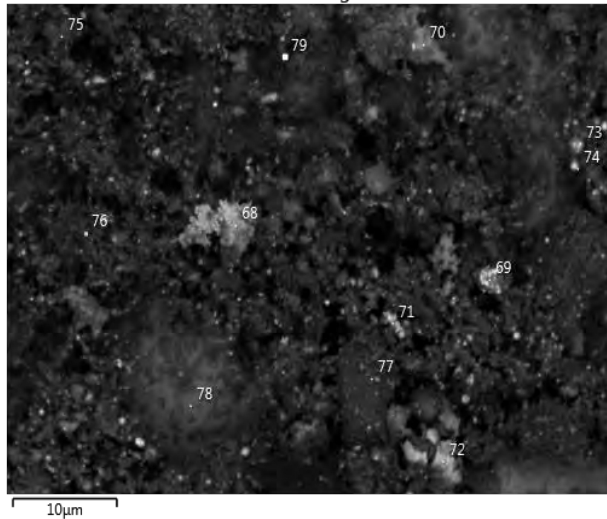


Agglomerates of nano carbonaceous material and solidified molten droplets of higher Z material



Witness Plate Post Test : Aluminum Disk D2 (protected)

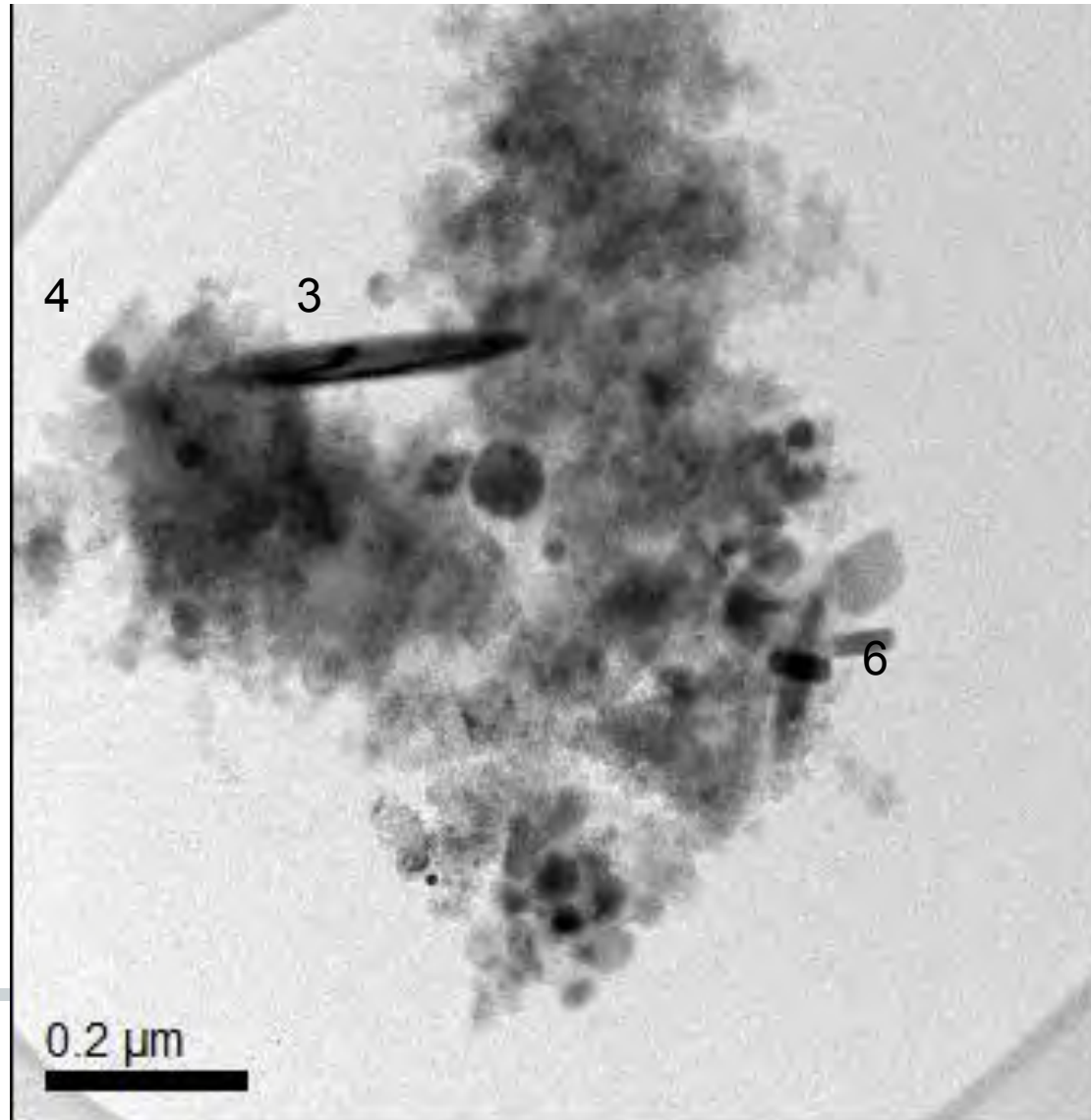
SEM EDS Spectra (5KV)



Al, C, Cu, O, Fe, Zn and Ge are common.



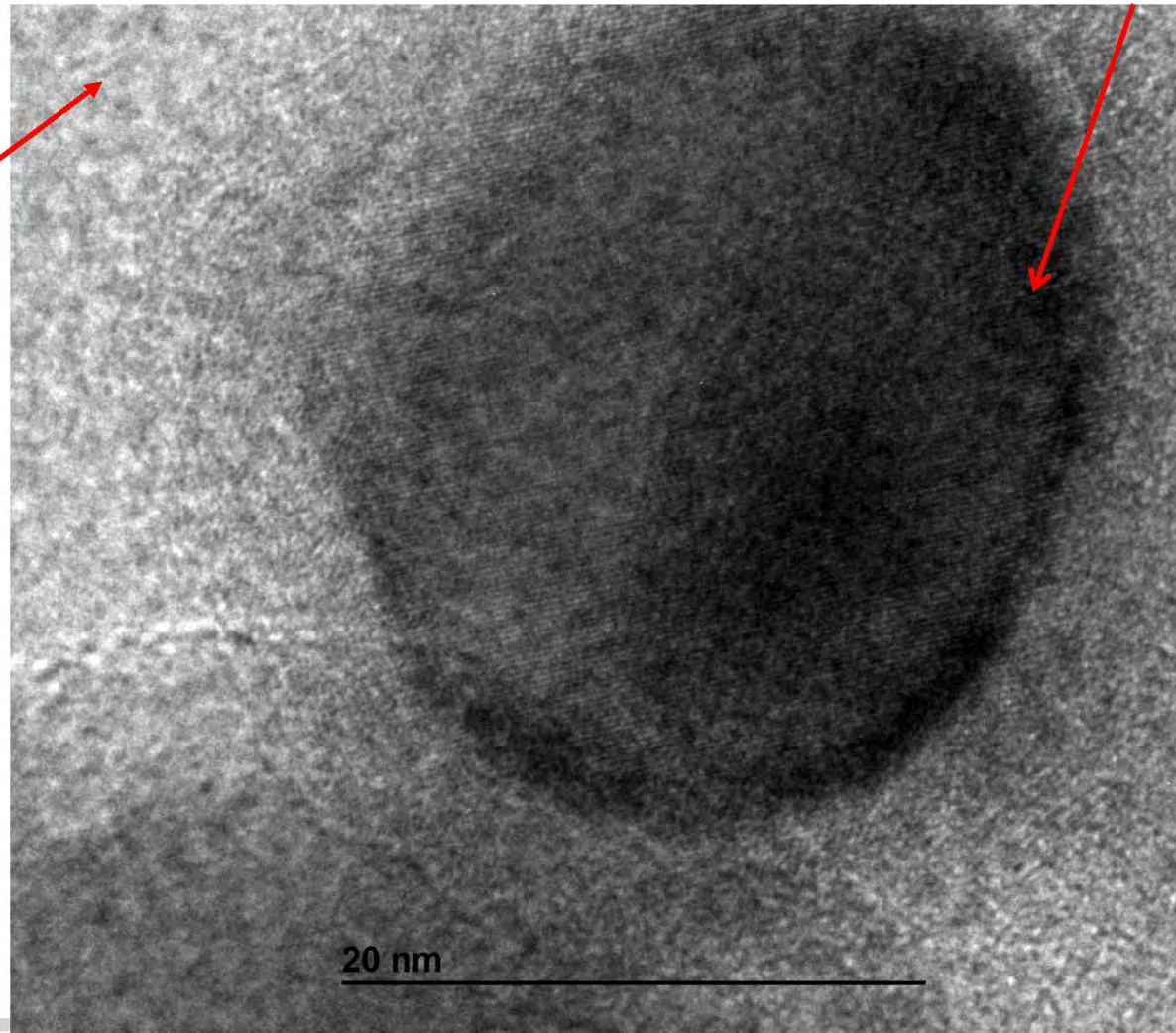
Witness Plate Al Disk D2: Loose Material on TEM Grid: BF TEM Location 4



Witness Plate Post Test D2: Area 2 BF-TEM

Crystalline lattice fringes

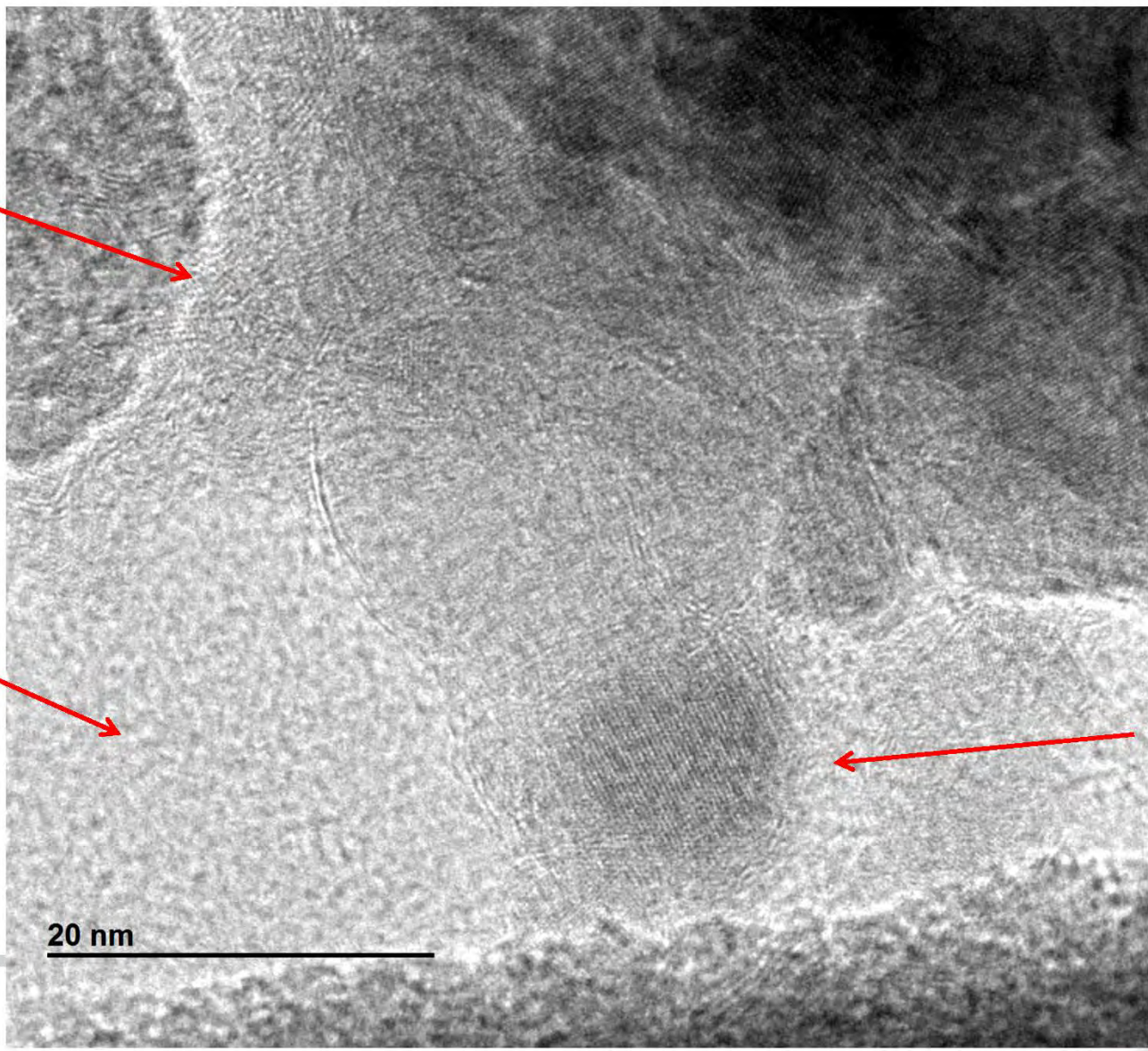
Amorphous C
support film



Particle is nominally a single crystal or consists of only a few crystalline grains.



Witness Plate Al Disk D2: Location 4 Area 3 BF-TEM



Disordered graphitic carbon fringes?



Amorphous ultra-carbon support film



Crystalline lattice fringes



20 nm



Witness Plate Al Disk D2 : Location 4 Area 4 BF-TEM

Crystalline
lattice fringes



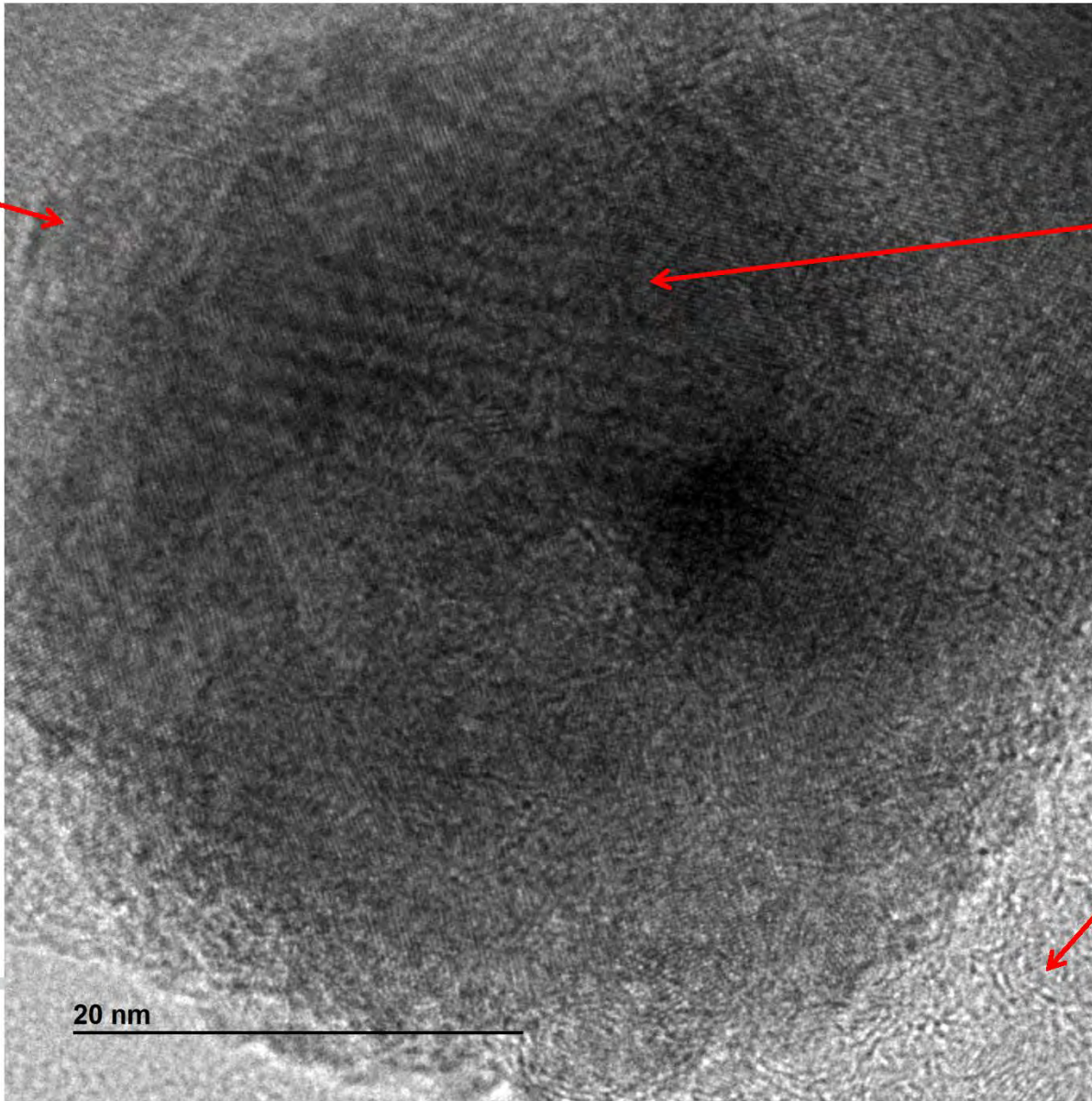
Large spacing
Moire fringes from
overlapping
lattices



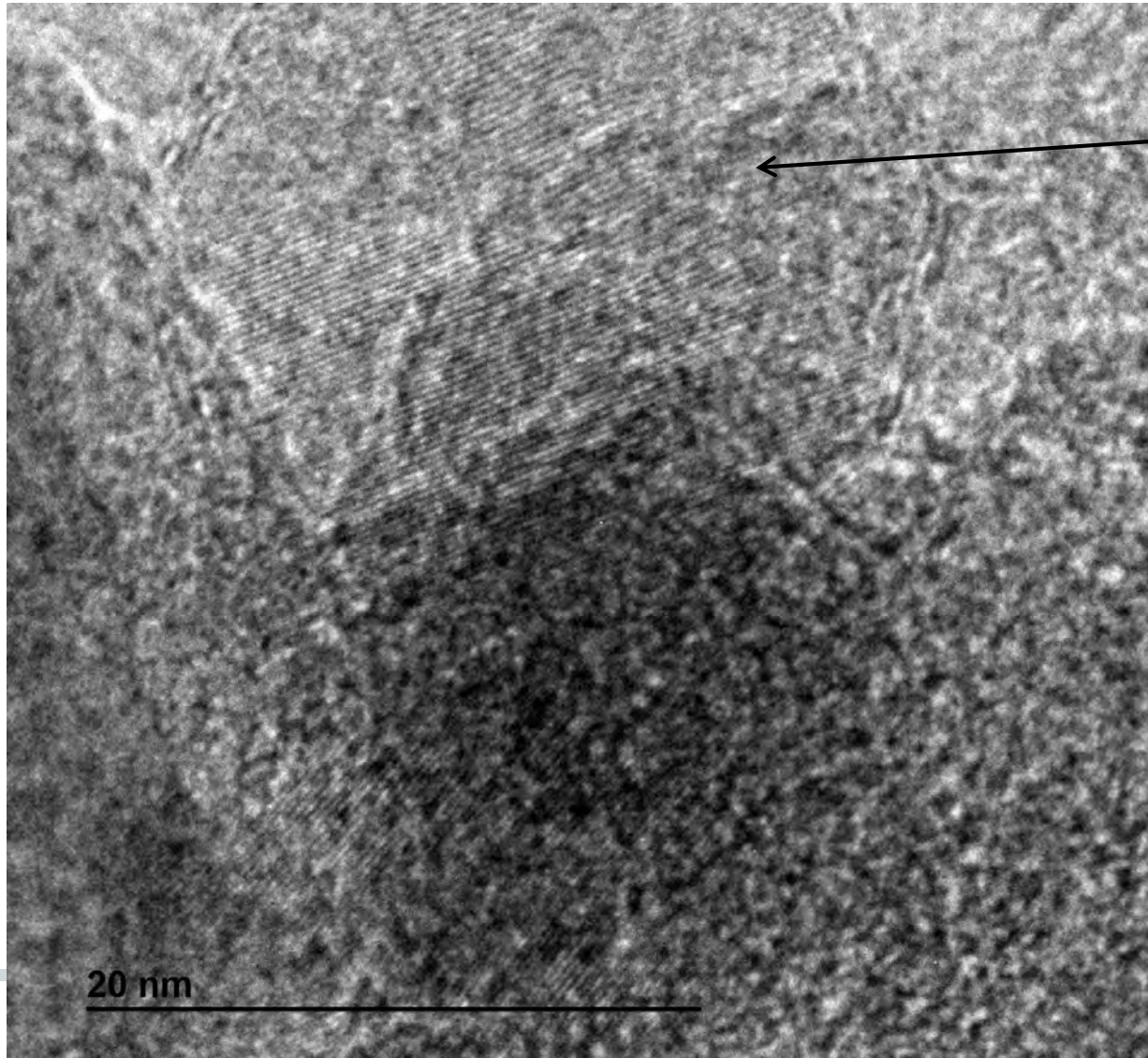
Disordered
graphitic carbon
fringes?



20 nm



Witness Plate Post Test: Al Plate D2: Area 4 BF-TEM



Crystalline lattice fringes

Particle is nominally a single crystal

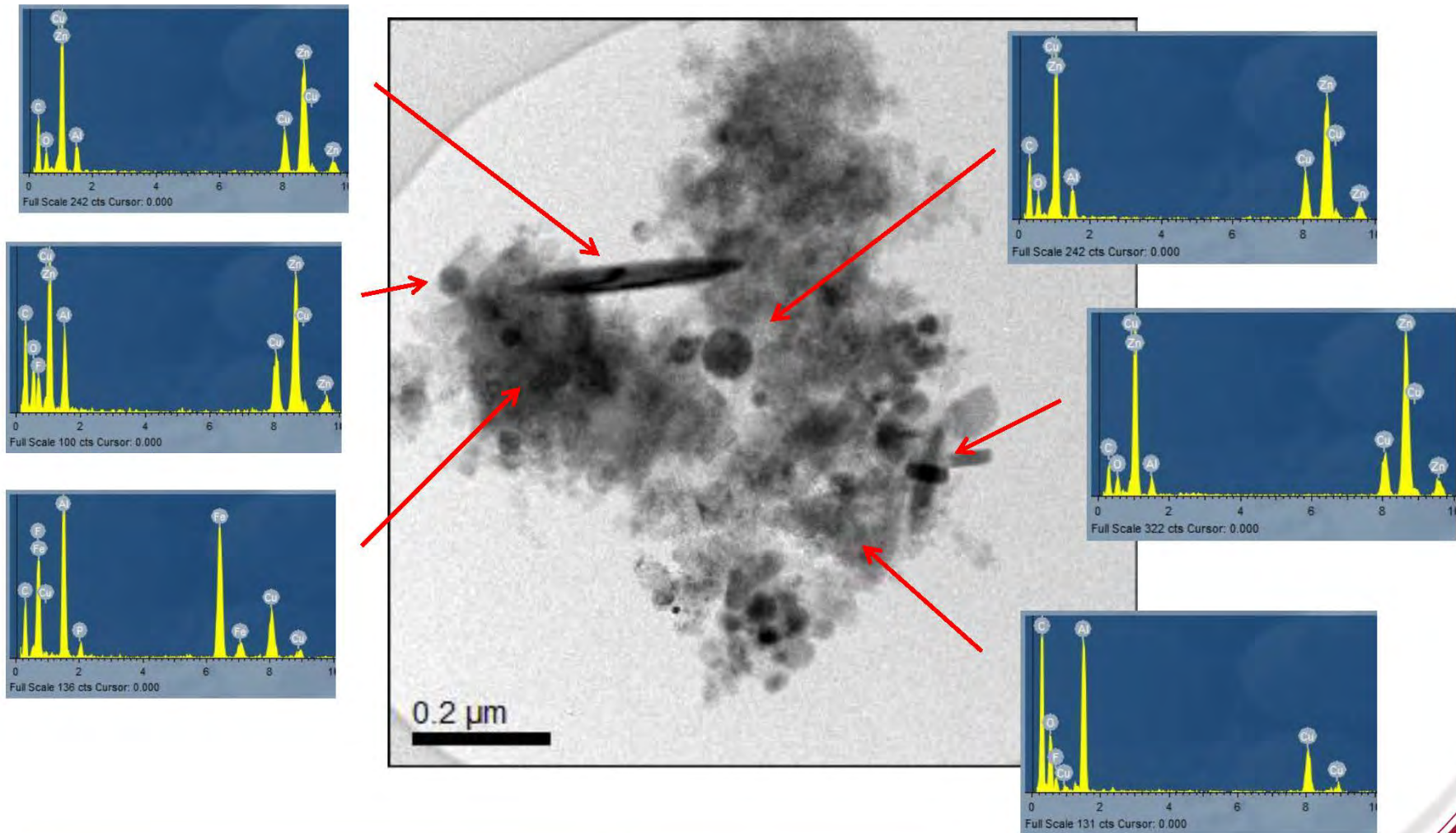




Spot patterns indicate crystallinity

Patterns cover about 0.7 micron diameter area

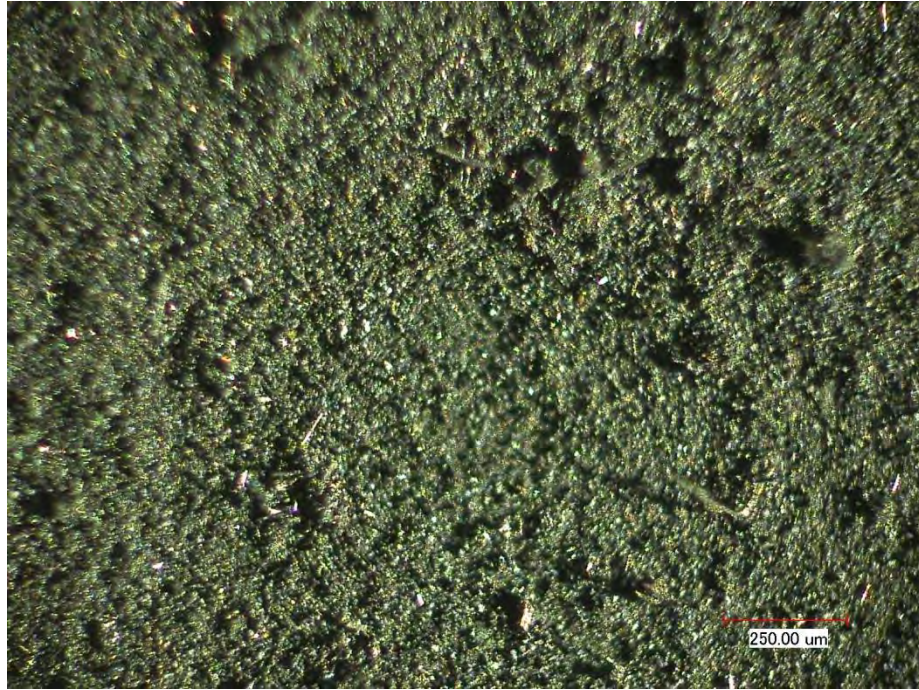
Witness Plate Al Disk D2 : Location 4 TEM-EDS



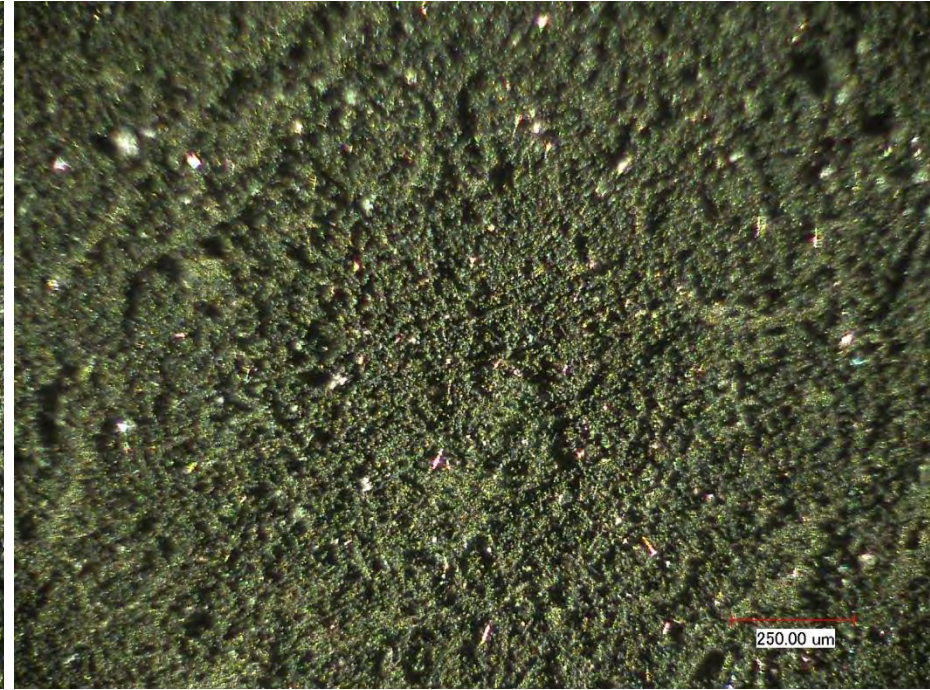
Discreet crystalline particles contain Zn. Al and Fe also detected.
Cu is probably from X-rays scattered from the microscope pole piece.

Witness Plate Post Test : Aluminum Disks

(Optical Images – 150X)



Exposed (B2)

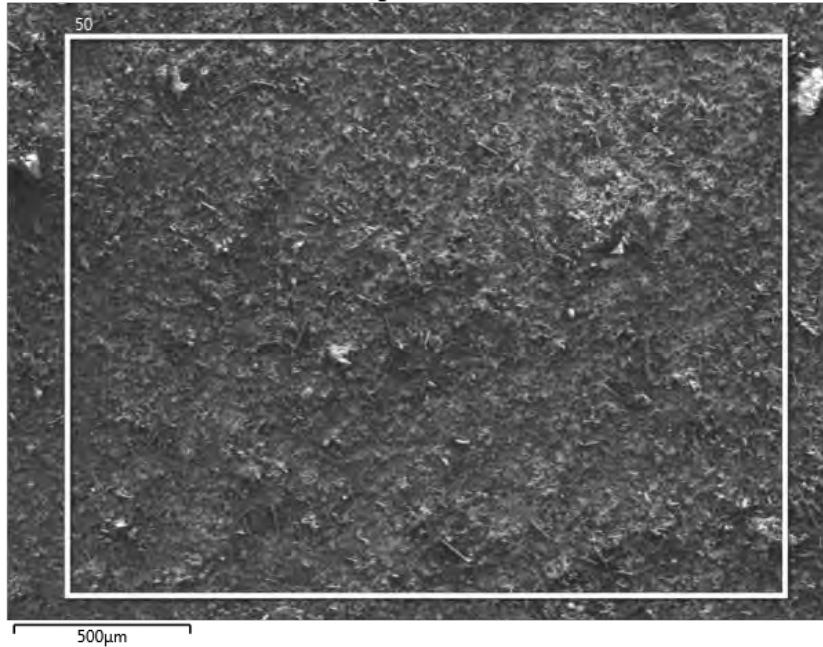


Protected (D2)



Witness Plate Post Test : Aluminum Disk B2 (exposed)

SEM EDS Average Composition (10KV)



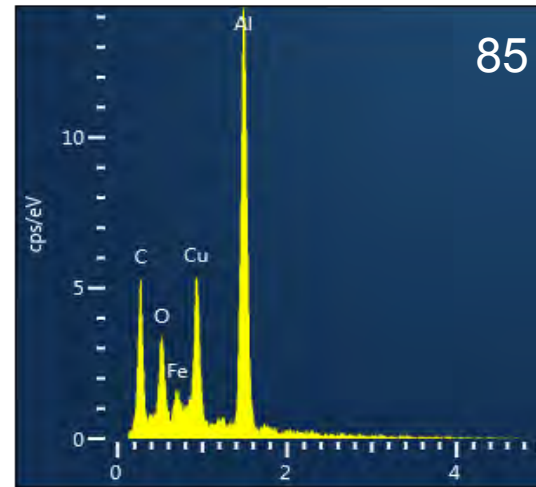
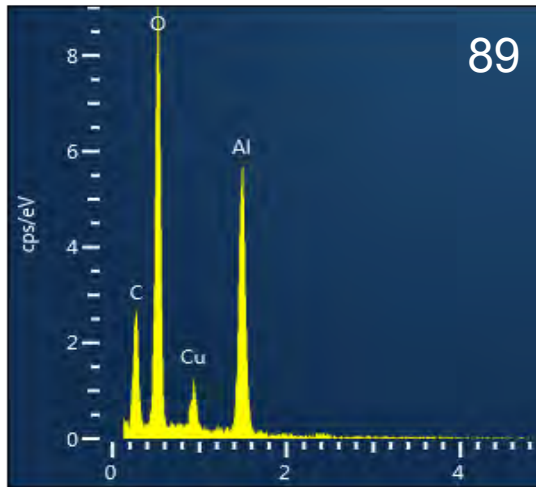
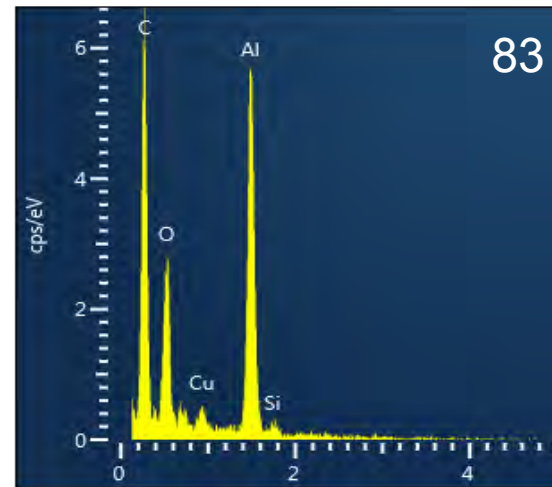
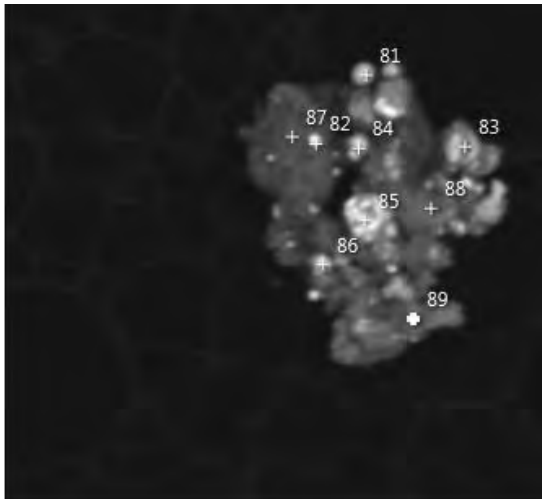
	Atomic %	Atomic %
C	63.67	64.82
O	15.89	15.58
F	1.22	1.61
Na	0.00	0.00
Mg	0.22	0.27
Al	13.74	12.72
Si	0.46	0.43
P	0.34	0.41
S	0.07	0.06
Ca	0.00	0.00
Cr	0.34	0.35
Fe	1.51	1.29
Cu	2.11	1.83
Zn	0.00	0.00
Ge	0.37	0.47
Ag	0.08	0.11
Sn	0.00	0.07
Total	100.00	100.00

Elemental abundances are similar to SEM stub witness plates on soft catch panels.



Witness Plate Post Test : Aluminum Disk B2 (exposed)

SEM Images of particles suspended on lacey carbon films on Cu TEM grid



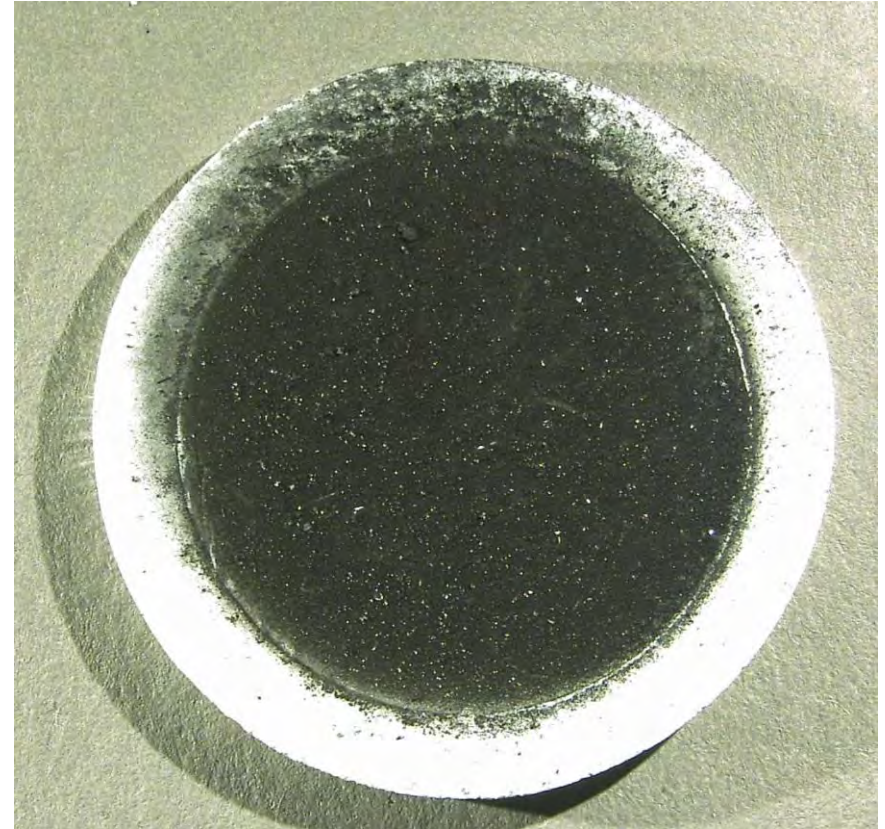
Al, C and O the are most common constituents, also minor Cu.



Witness Plate Post Test: Z93 Painted Aluminum Disks



Exposed

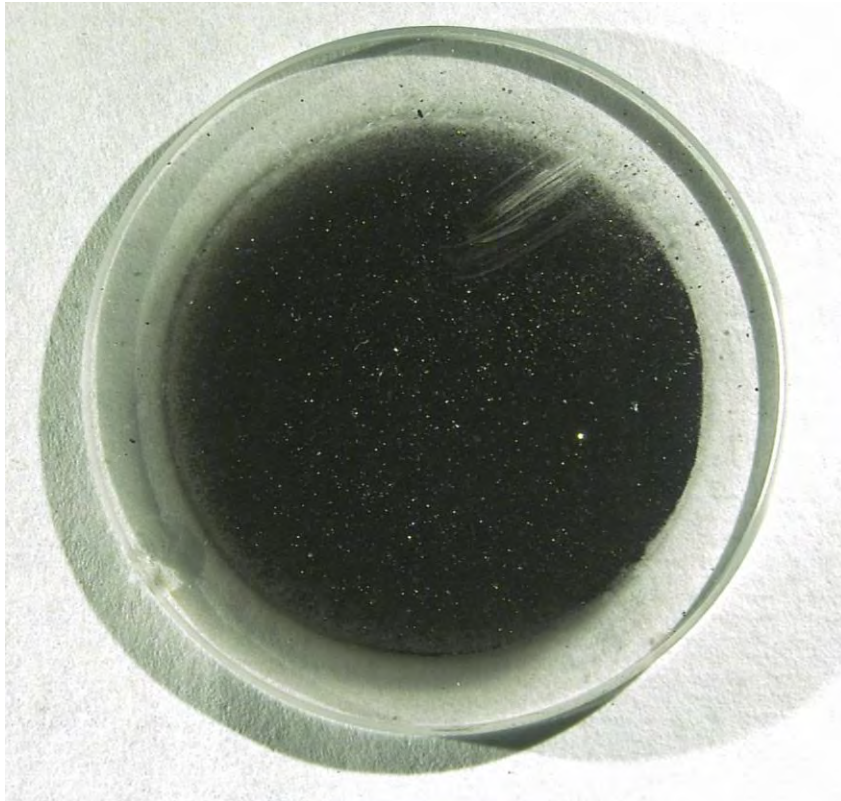


Protected

Z-93 paint consists of ZnO in a potassium silicate binder



Witness Plate Post Test : Fused Silica Disks



Protected (C1)

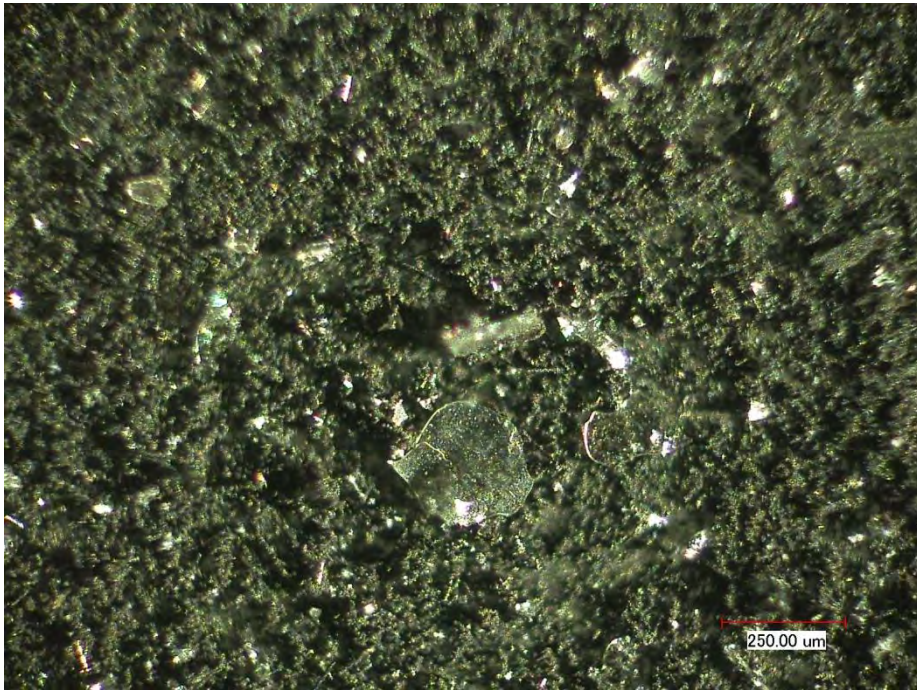


Protected (D1)

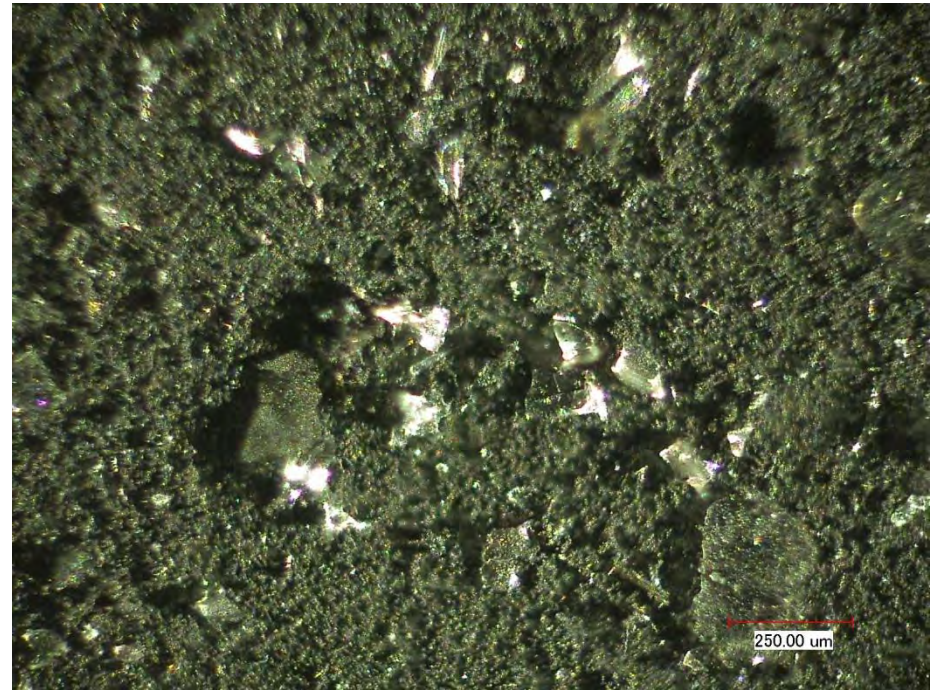
The directly exposed disks were destroyed



Witness Plate Post Test : Fused Silica Disks – Optical Images (150X)



Protected (C1)



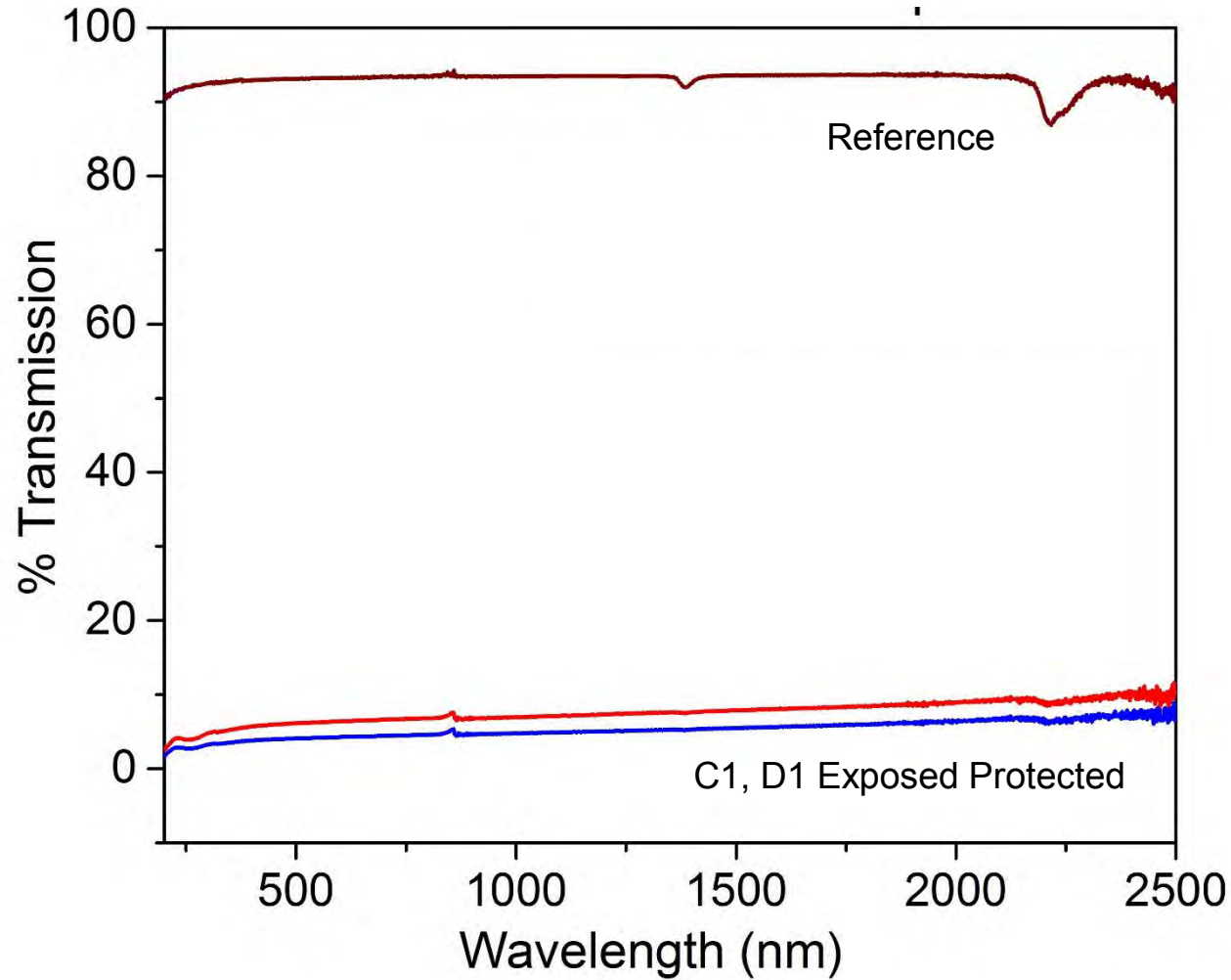
Protected (D1)

Clear angular fragments are soft catch foam debris.



Witness Plate Fused Silica Disks

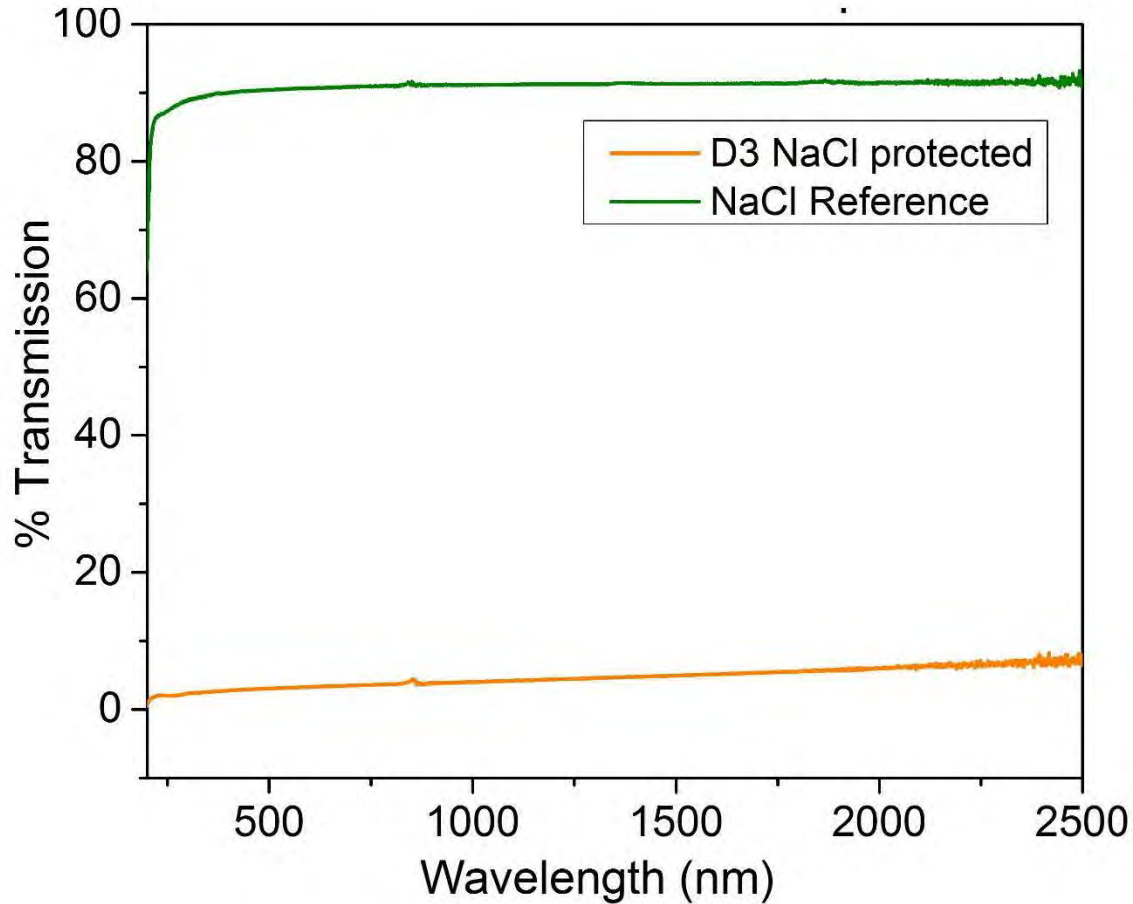
UV-VIS-NIR Transmission



Significant decrease in transmission from 94% to 8%.



SBU Marking
Witness Plate NaCl Disk
UV-VIS-NIR Transmission



Significant decrease in transmission from 90% to 12%.



Appendix 2



SBU Marking Technical Reports Addendum Asset Summary



TRAAS ID #: 2015011406454314820

Report Name: DebrisSat Laboratory Analyses

Aerospace Report Number: TOR-2014-00876

Start Date of Test: 2014-05-01

Created By: 14820 Adams, Paul M

JO: 850672

End Date of Test: 2014-12-31

First Aerospace Author / PI: 14820 Adams, Paul M

Program: DebrisSat

Description:

Keywords:

Asset:	Manufacturer:	Model:	Usage Start Date:	Usage End Date:	Asset Comment:
AAA123	PERKIN ELMER CORPORATION	LAMBDA 900	2014-05-01	2014-12-31	
Date:	Calibration Due Date:	Comment:	Certificate Number:		
2013-04-17	2014-08-17	TMT-NORMAL	fa6f52f7eed80b468d87b46e4dfec1aa		
2014-08-18	2016-03-13	TMT-NORMAL	777773c04920e64b8f107f76514d6a40		
AAA126	PERKIN ELMER CORPORATION	PELA-1000	2014-05-01	2014-12-31	
Date:	Calibration Due Date:	Comment:	Certificate Number:		
2013-04-17	2016-04-17	TMT-NORMAL	ed846df747a19149bb42006784ea95a7		
ABH857	RENISHAW	INVIA	2014-05-01	2014-12-31	
Date:	Calibration Due Date:	Comment:	Certificate Number:		
2013-07-18	2014-07-13	TMT-NORMAL	fd9ec878f3a7246a89e074b2d36e529		
2014-07-16	2200-01-01	SAE	e6467a74b10adc47b9ae2e6d00f01835		
2014-07-16	2015-12-13	TMT-NORMAL	af200c8b9e350842a2f66701f5d97550		



SBU Marking

Asset: ABN670 Manufacturer: JEOL (USA) INC. Model: JEM3100F Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-01-09	2014-05-04	TMT-NORMAL	885d0189ca15864aba20d3b3e6d5eb0a
2014-05-08	2015-12-06	TMT-NORMAL	0f70eaaa9351b42901343965adb1738

Asset: ABW501 Manufacturer: THERMO-NICOLET Model: 6700 Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2014-03-17	2015-08-16	TMT-NORMAL	c0ad25e310e49243ae8a043b67c2c0f1

Asset: ACR364 Manufacturer: JEOL (USA) INC. Model: JSM-7600F Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2014-02-05	2015-07-05	TMT-NORMAL	d498d9360ad1224f85d04309c6869674

Asset: ACR429 Manufacturer: OXFORD INSTRUMENTS Model: X-MAX Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-02-08	2014-06-08	TMT-NORMAL	106f65ed9dfc8043869a5da06587e7fa
2014-06-02	2015-12-27	TMT-NORMAL	f840182f4f92e2449d541c92c3465ec2

*Support and Auxiliary Equipment are not calibrated.



DebrisSat Laboratory Analyses

Approved Electronically by:

Frank L. Knight, SYSTEMS DIRECTOR
ENGINEERING
ENGINEERING DIRECTORATE
OFFICE OF EVP/SSG

David J. Gorney, EXECUTIVE VP
OFFICE OF EVP/SSG

Technical Peer Review Performed by:

Shant Kenderian, DIRECTOR DEPT
MATERIALS PROCESSING DEPT
SPACE MATERIALS LABORATORY
ENGINEERING & TECHNOLOGY GROUP

Steven C. Moss, DIRECTOR DEPT
MICROELECTRONICS TECHNOLOGY
DEPT
ELECTRONICS & PHOTONICS
LABORATORY
ENGINEERING & TECHNOLOGY GROUP

External Distribution

REPORT TITLE

DebriSat Laboratory Analyses

REPORT NO.	PUBLICATION DATE	SECURITY CLASSIFICATION
TOR-2015-00876	May 4, 2015	UNCLASSIFIED

Thomas Huynh
SMC/ENC
thomas.huynh@us.af.mil

Mitch Nolen
AEDC
mitchell.nolen.ctr@us.af.mil

Jesse Edwards
SMC/ENC
jesse.edwards.4@us.af.mil

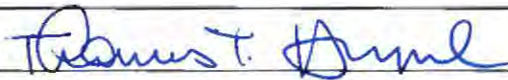
Brian Roebuck
AEDC
brian.roebuck.ctr@us.af.mil

J.-C. Liou
NASA-JSC
jer-chyi.liou-1@nasa.gov

John Opiela
NASA-JSC
john.n.opiela@nasa.gov

Heather Cowardin
NASA-JSC
heather.cowardin@nasa.gov

APPROVED BY
(AF OFFICE)



DATE

16 Feb 2015