CVD ^{13}C Concentrator Target, Genesis Sample 60002
Particle Contamination Documentation
Interim Report

This report represents the completion of initial image documentation of the Genesis-flown CVD ^{13}C-diamond concentrator target 60002. Additional characterization of an abundant contaminant will be added to the final report.

Results Summary

Mosaic images in both bright field and dark field illumination reveal contaminant particles similar to some features found in the SiC targets. These can be generally grouped as: dried salt/mud areas, transparent angular-outline fragments, black irregular fragments, straight black fibers, dried droplet circular features, clusters of transparent spheres ("bubbles"), and curved fibers. Similar to the SiC targets, there is a noticeable lack of crushed collectors, which are commonly observed on wafers. Compared to the SiC targets, the areal density of particles is apparently higher. Also, the diamond exhibits a transparency which contributes to imaging differences. Our overall impression is that the diamond surface is undamaged by crash-impacted debris, and the contaminant particles are mostly crash-related lacustrine sediments and sample return capsule (SRC) debris, perhaps easily removable.

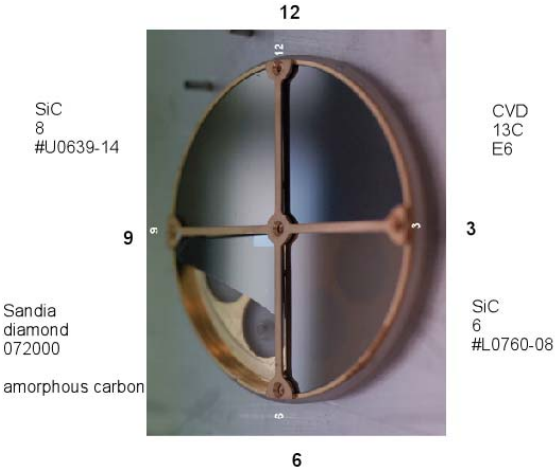


Fig. 1. Post-recovery mounted targets showing cross arm clocking designation. Note that the CVD ^{13}C target was flown between two SiC targets at 12 and 3 O'clock gold cross arms.

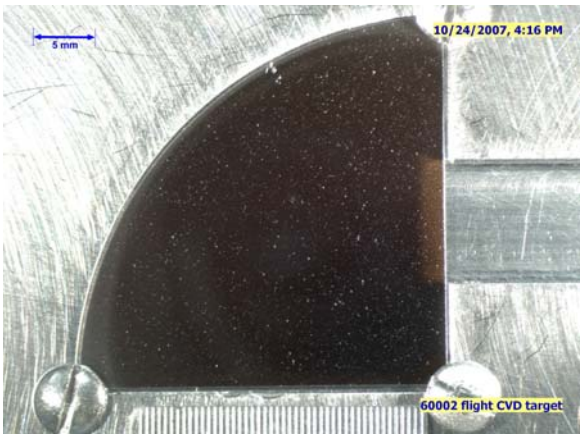


Fig. 2. 60002 overview image in RSV container.

The presumed lakebed materials include the salt/mud deposits and some of the dried droplet features. Presumed SRC debris materials include the black fibers (compare to SRC backshell carbon fibers), the orange-red fragments of color similar to RTV and silicone made brittle, and thin film particles that resemble Kapton tape. Similar to the SiC targets, the most intriguing features, which are not yet observed on wafer fragments, are the clusters of transparent spheres. These "bubbles" appear to be thin-wall structures, based on spherical reflections from numerous small fragments of broken bubble shells. Many of the small size transparent debris fragments may be derived from these bubbles. We are currently in the process of obtaining chemical information on these unusual features.

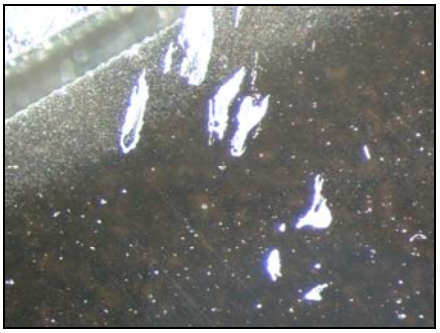


Fig. 3. Salt/mud deposits on outer edge of quadrant. Probable salt composition is halite, gypsum and carbonates. Field of view is 2.5 mm. Available light illumination.

Fig. 4. Surface debris (blue-white), debris spray (tan-white), backside reflection (mottled). Field of view is 2.5 mm. Available light illumination.

Fig. 5. Debris near quadrant edge. Blue color occurs in areas of high solar wind flux. Scale bar is 500 µm. Bright field illumination.

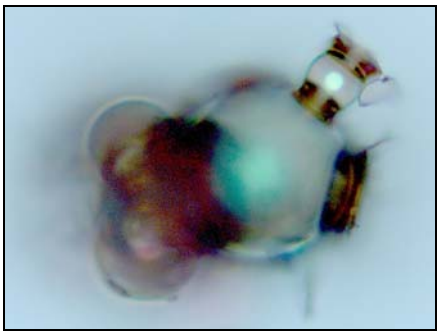


Fig. 6. Unusual 100-µm contaminant particle resembling cluster of bubbles. Fragments of such particles are abundant. Bright field illumination.

Particle size distribution summary: The CVD target has apparent higher areal density of particles, when compared to the SiC target. Using a 50X objective with a particle image resolution of ~ 0.3 µm, two areas of approximately 1 mm^2 were imaged for particle analysis. Results from the two areas on the CVD target are compared with single areas from each of the two SiC targets.

Substrate	Area, mm^2	Particles	Particles/mm^2
SiC 60001	1.183	275	232
SiC 60003	0.983	299	304
CVD scan 1	1.179	466	395
CVD scan 2	1.179	858	728

Particle size distribution, as well as particle areal density, varies between the two areas measured on the CVD target. Figures 7 & 8 illustrate that 90% of the particles are below 5.0 µm diameter in size based on the scan 1 and below 2.3 µm diameter in size based on scan 2. Extended detail on particle size distributions is included in the body of this report.

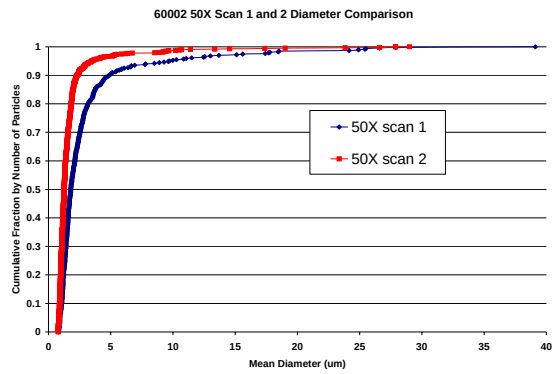


Fig. 7.

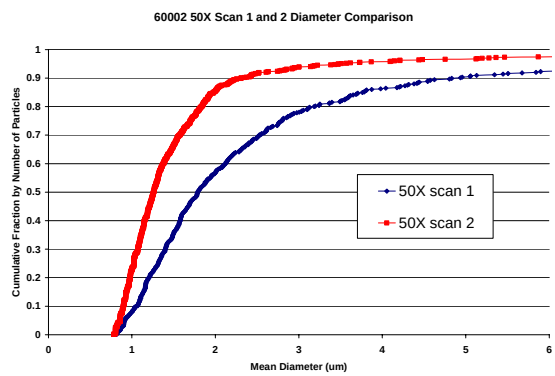
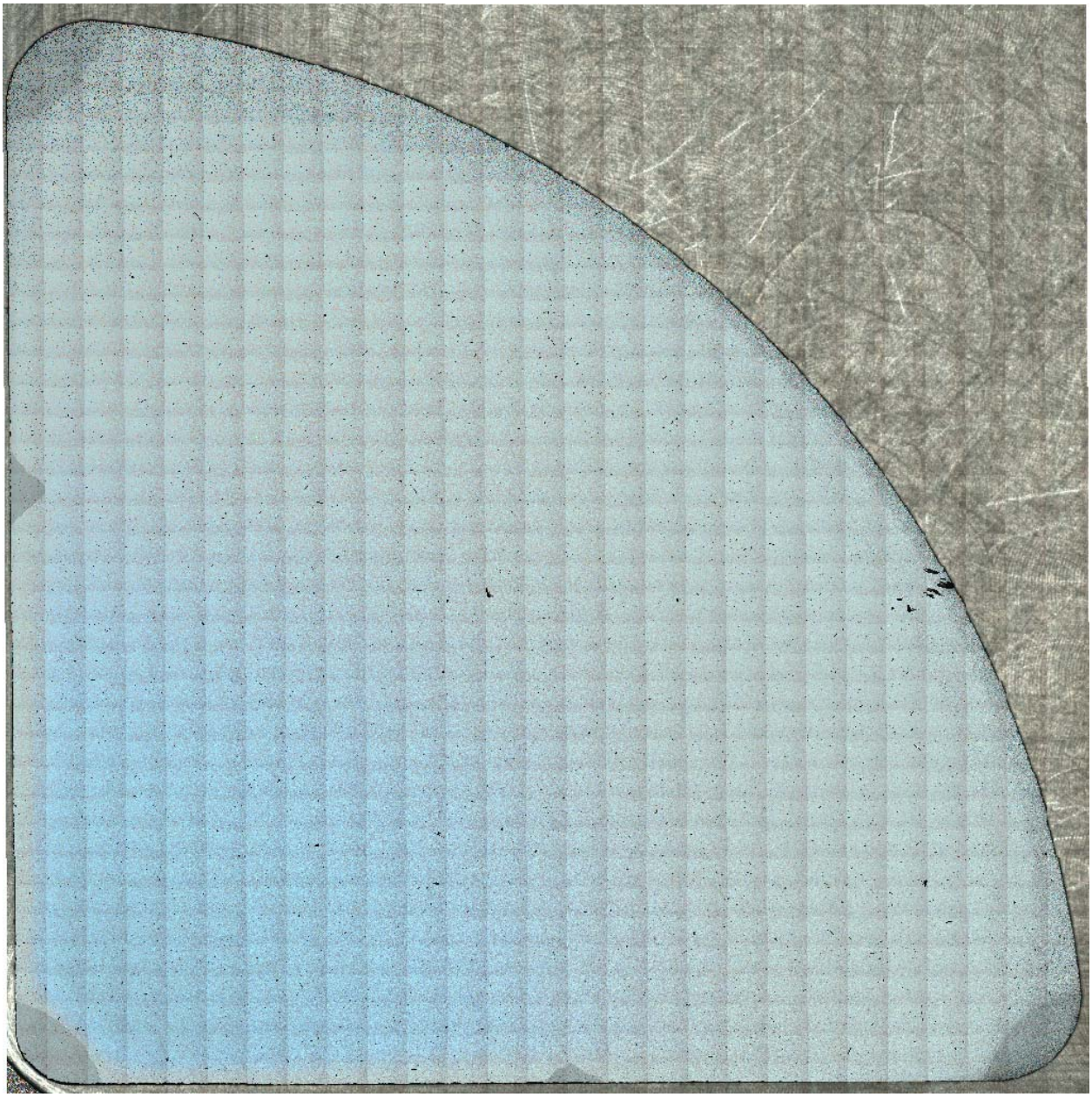


Fig. 8.

Outline for Detailed Data:

5X mosaic images, bright field & dark field
Feature map
Feature images
50X mosaic images and particle size statistics
Appendix 1: quadrant dimensions

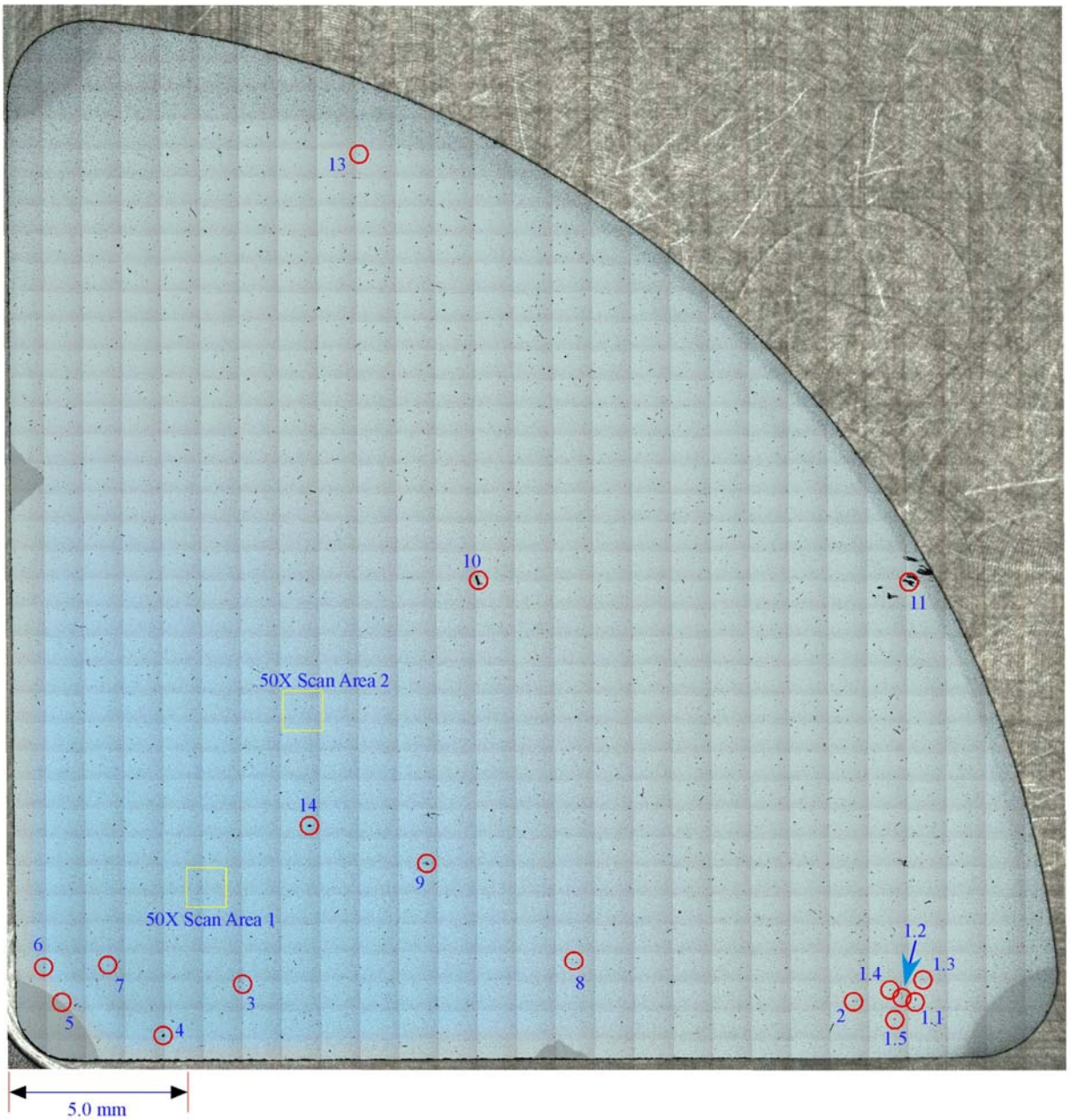
5X Mosaic Scans and Feature Map



60002 bright-field mosaic microscope image, 5X objective. Each rectangle is an individual image that can be viewed at a high resolution. The ~ 50 MB bitmap image (not attached in this report) can be used to zoom into various locations and highlight an area for viewing. The mosaic contrast was not on auto and remained constant for all frames; thus, this color variation (sea aqua-blue to bluish-green), with radial distance from focal point, is real.

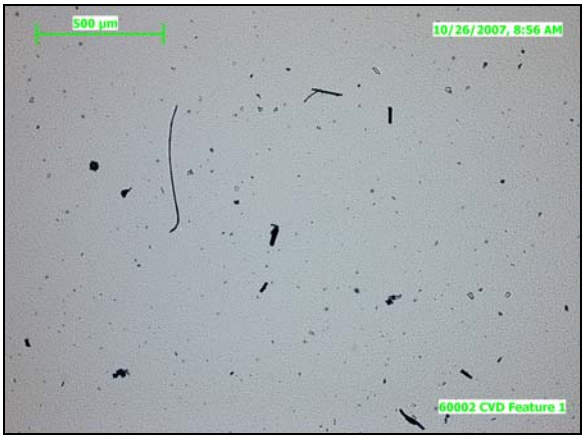
60002 dark-field mosaic microscope image, 5X objective. Each rectangle is an individual image that can be viewed at a high resolution. The ~ 50 MB bitmap image (not attached in this report) can be used to zoom into various locations and highlight an area for viewing.

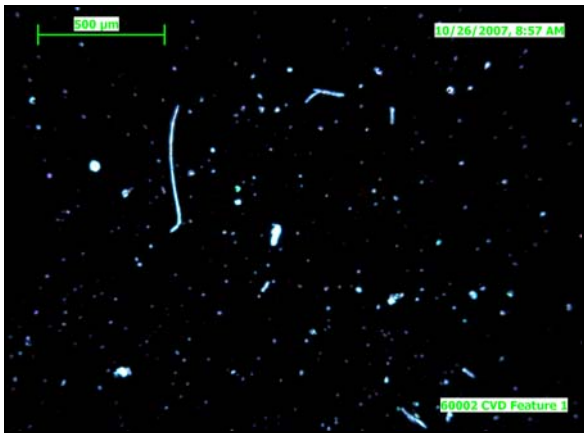
60002 CVD Concentrator Target Feature and Scan Area Map



This feature map corresponds to the following images in the next section. This map also shows the two areas that were scanned with the 50X objective. Detailed particle counts on the two scanned areas can be found in the last section of this report.

60002 Target Features



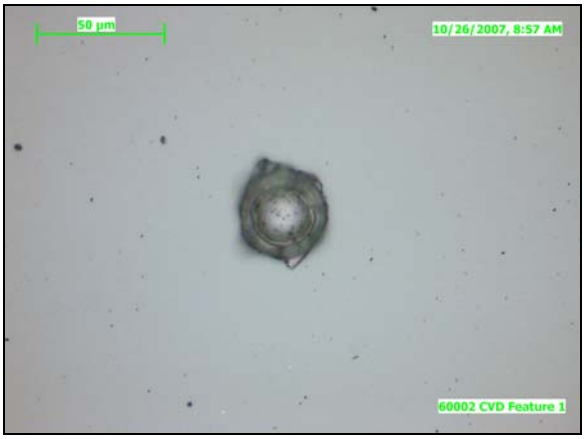


Area of features 1.1, 1.2, 1.3, 1.4 and 1.5: Area of fibers, debris, and bubble fragments, bright and dark field views (photos 60002B5X001_01 and 60002D5X001_02). Scale bars are 500µm.

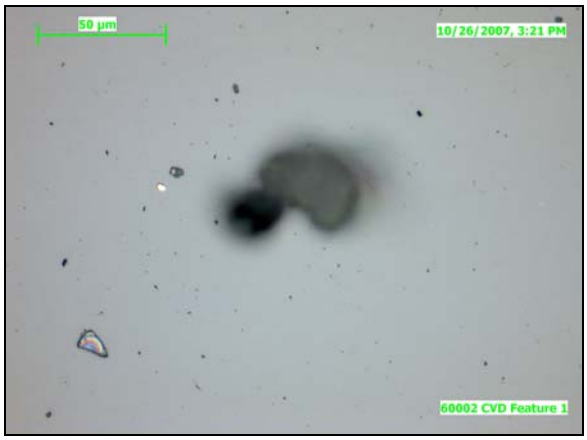
Feature 1.1: Possibl**e** collector fragment with thin, wrinkled, foil-like edge (platinum foil? gold or aluminum coating?). Scale bar is 50µm (photo 60002B50X001_03).

Feature 1.2: Thin, smooth curved fiber, bubble fragment, bright spot. Scale bar is 50µm. (photo 60002B50X001_05).

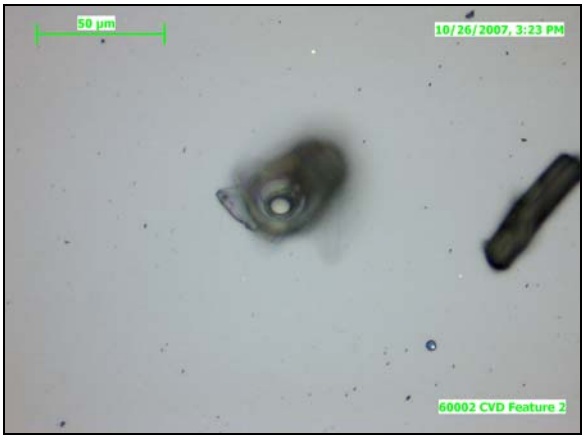
Feature 1.3: Branched or frayed dark fiber, possibly backshell/spacecraft material. Scale bar is 50µm (photo 60002B50X001_07).



Features 1.4: Concave, jagged, transparent bubble fragment. Scale bar is 50µm (photo 60002B50X001_09).

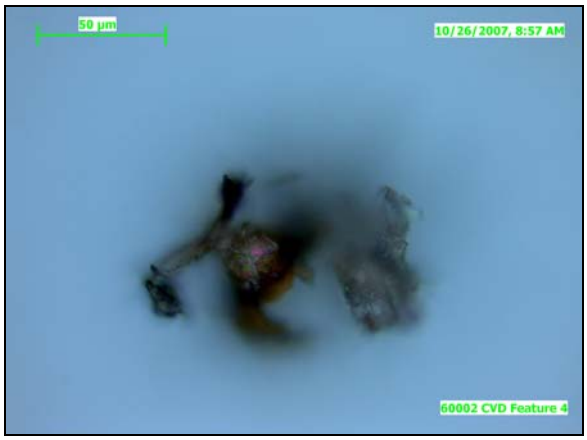


Feature 1.5: Two views of debris cluster extending approximately. 20µm above the surface of CVD target. Dark material is grouped with material lighter in color. In top view (left), a dark particle is in focus (photo 60002B50X001_12). At right, the same view is focused at the surface of CVD target. Surface debris consists of translucent particles, bubble fragments and one bright white circular area (photo 60002B50X001_14). Scale bars are 50µm.



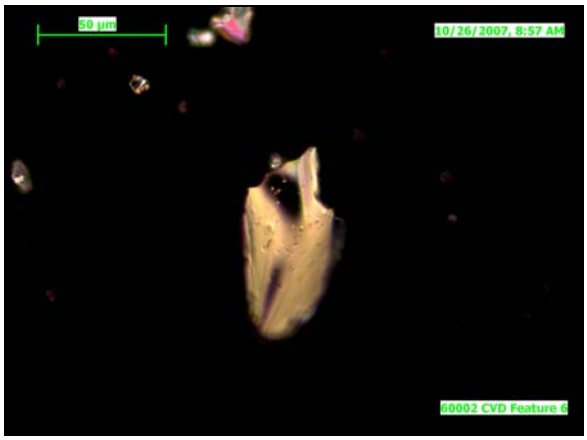
Feature 2: Two views of complex bubble fragment. At left, view of focused at target surface showing a concave bubble fragment with circular opening and a dark fiber approximately 12µm in width (photo 60002B50X002_01). At right, view focused approximately 20µm above CVD target surface shows small "budlike" protrusion attached to bubble fragment by a concentric darker material (photo 60002B50X002_03). Scale bars are 50µm.

Feature 3: Round feature surrounded by translucent bubble fragments and particulate contamination. Bright white circular area to the left of feature (photo 60002B50X003_01). Scale bar is 50µm.



Feature 4: Two views of debris cluster near target corner edge. At left, the color variance is due to gold cross shadowing. Scale bar is 500µm (photo 60002B5X041_01). At right, close-up view of top of debris cluster, extending approximately 50 µm above CVD target surface. Amber-colored bubble-like feature surrounded by a thin mesh of dark fibers and smaller transparent bubbles. Scale bar is 50µm (photo 60002B50X004_03).

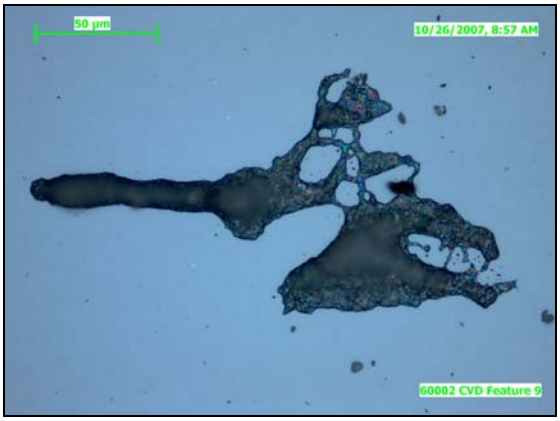
Feature 5: Two views of corner cross-holder shadow mark on CVD target. At left, scale bar is 500µm (photo 60002B5X005_01). At right, close-up view from near center of previous image. Scale bar is 50µm (photo 60002B50X005_02).



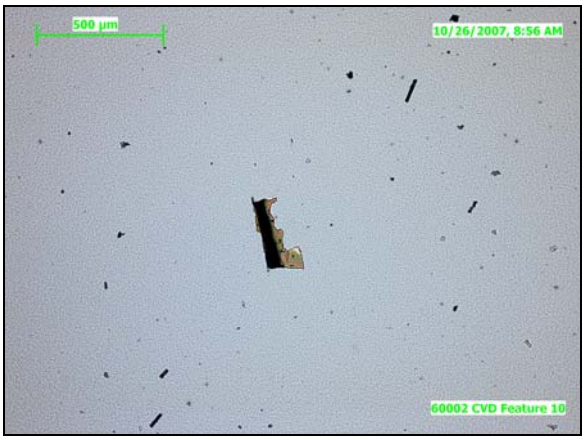
Feature 6: Bright and dark field views (photos 60002B50X006_01 and 60002D50X006_02) of an opaque, reflective, dark colored feature exhibiting micron-sized circular particles, bumps or pits on its surface. Feature appearance implies malleable material. Scale bars are 50µm.

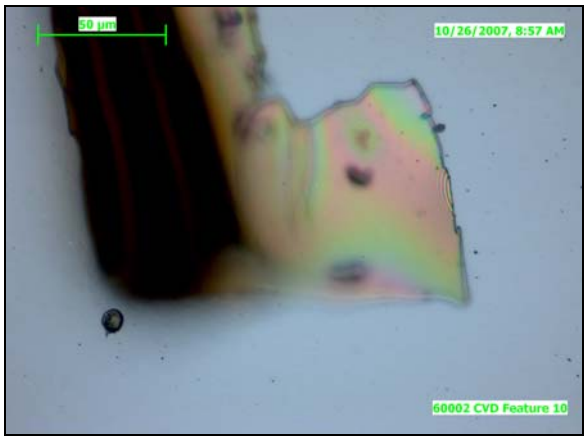
Feature 7: Thin, transparent, folded, reflective film-like feature. Dark, particulate contamination can be seen under film. Scale bar is 50µm (photo 60002B50X007_01).

Feature 8: Evaporation rings visible surrounding particle at 50X. Rings consist of fine brown-colored droplets, one ring is markedly darker, surrounded by smaller dispersion of droplets. Scale bar is 50µm (photo 60002B50X008_01).

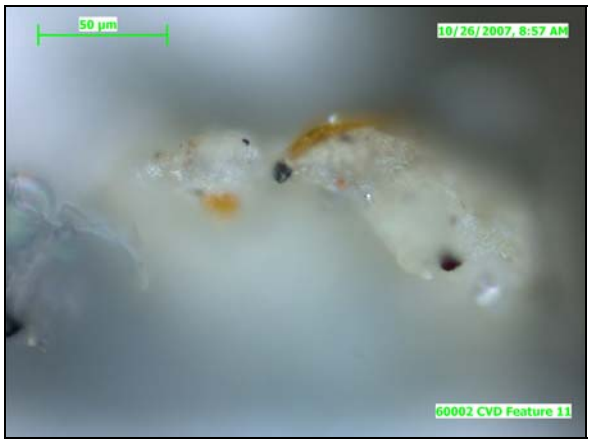


Feature 9: Salt/mud/debris clump. Hydrophobic tension with the target surface is observed at deposit edges. Scale bar is 50µm (photo 60002B50X009_01).

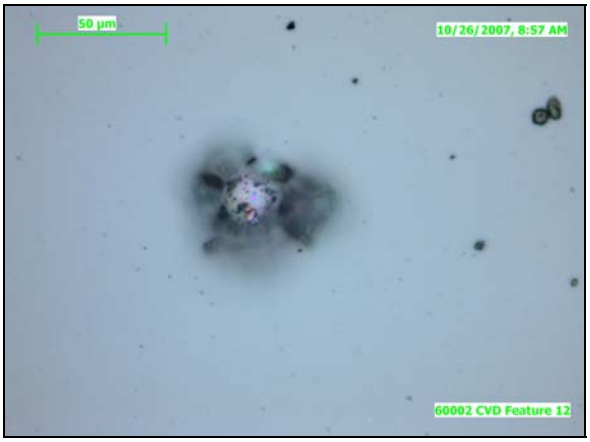




Feature 10: Two views of a thin, transparent film fragment on surface of CVD target (looks like Kapton tape) surrounded by dark fibers. Scale bars are 500µm and 50µm (photos 60002B5X010_01 and 60002B50X010_02).

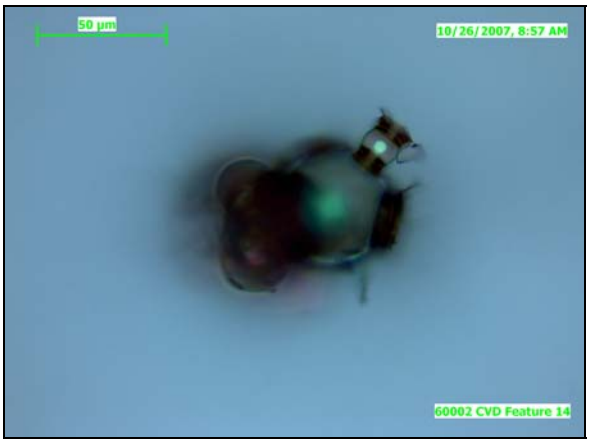


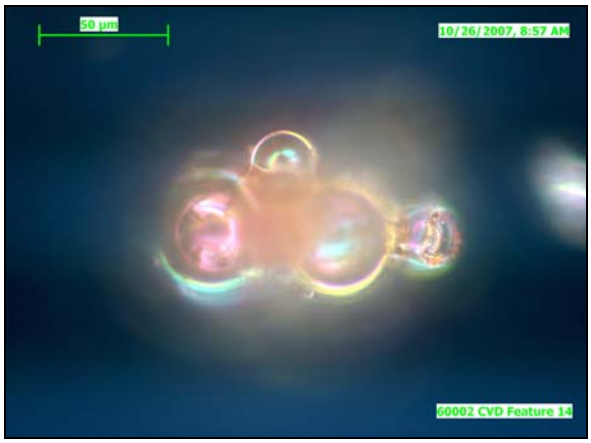
Feature 11: Two views of salt/mud/debris smear on quadrant outer radius curve. At left, view with scale bar equal 500µm, shows overview of smear and uneven, rough quadrant edge (photo 60002D5X011_01). At right, close-up view shows amber-colored matter and dark fibers within salt/mud/debris mass (photo 60002B50X011_04). Scale bas is 50µm.



Feature 12: Broken bubble fragment surrounded by particulate contamination (photo 60002B50X012_01). Scale bar is 50µm.

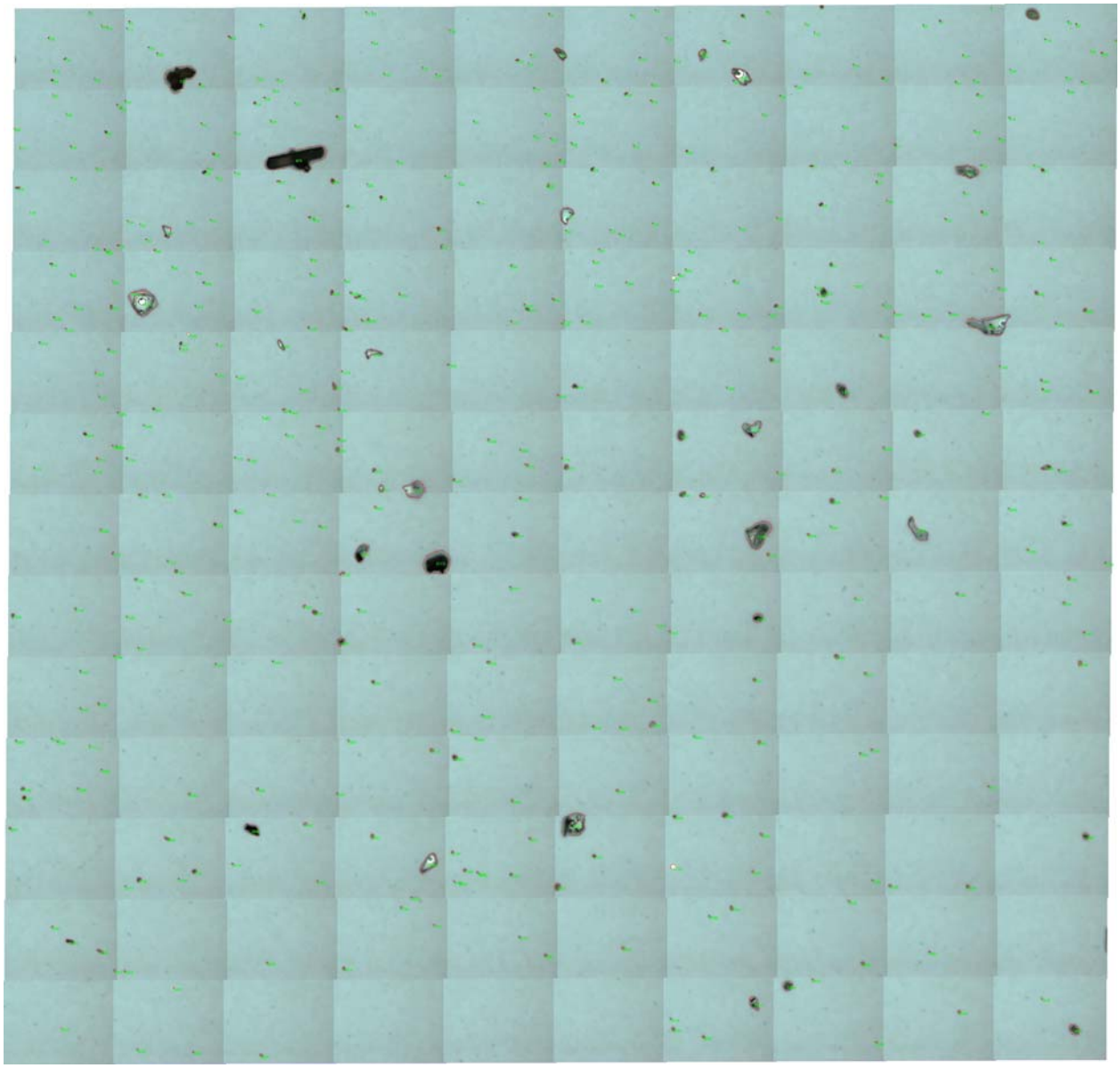
Feature 13: Two transparent, bluish-colored layers with blurred outlines (photo 60002B50X013_01). Scale bar is 50µm.



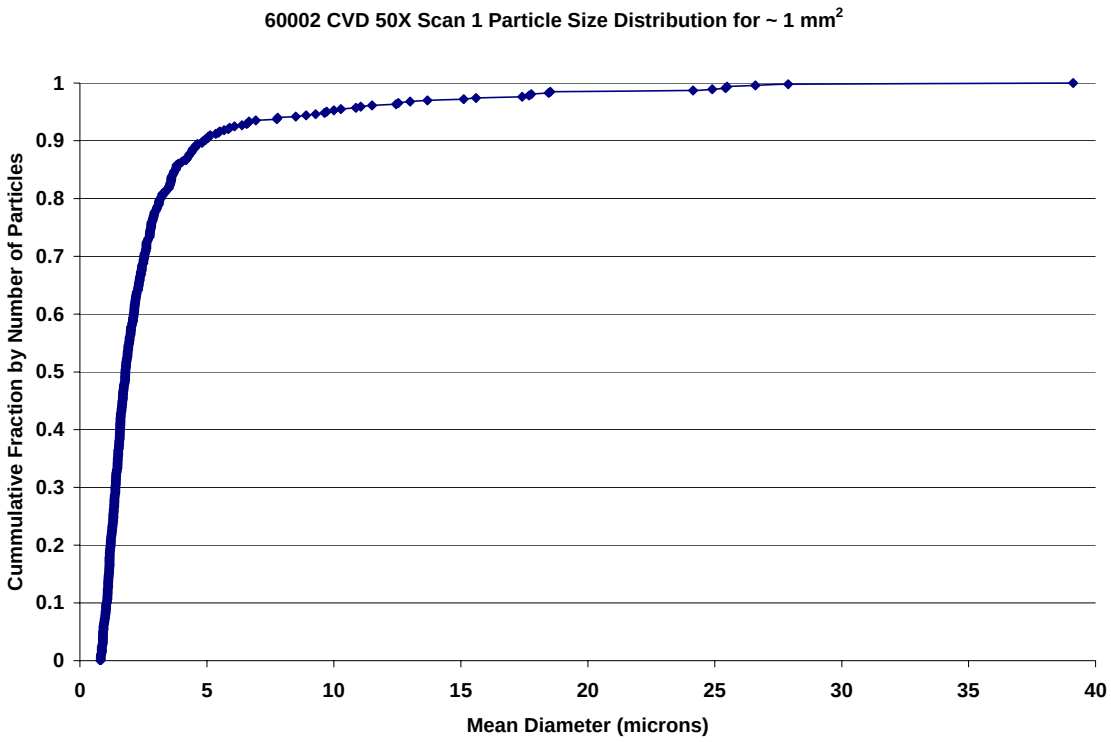


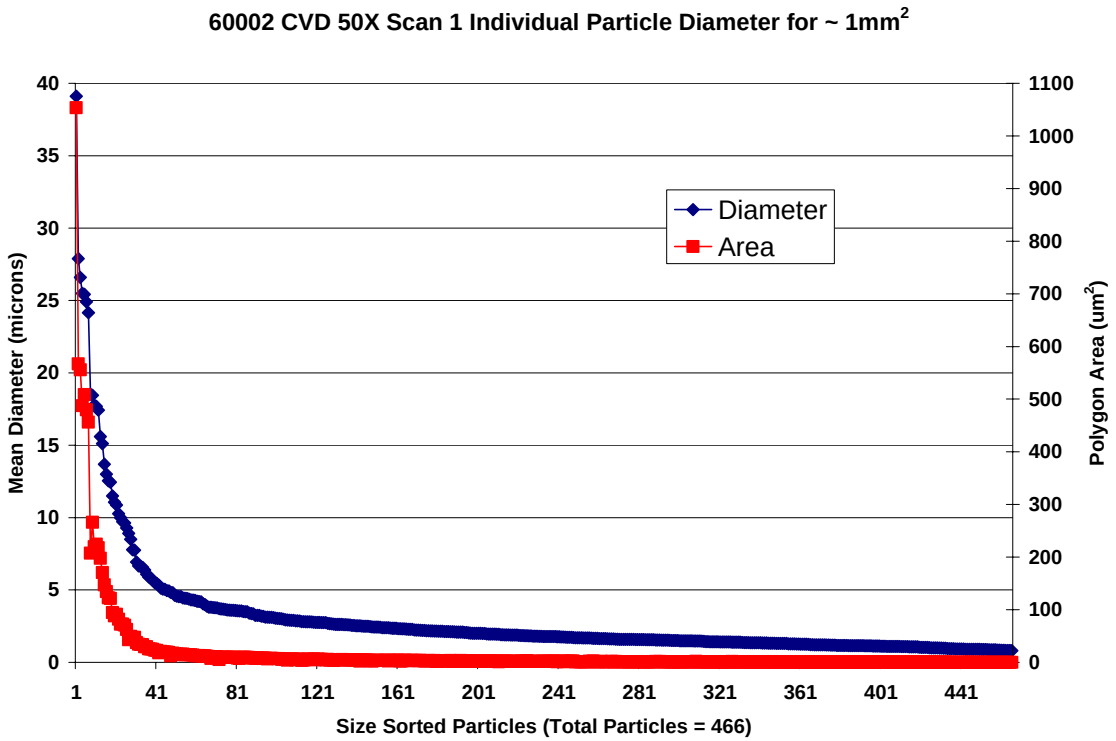
Feature 14: Two views of a complex bubble cluster taken at different focus depths, one bright field and one dark field illumination. At left, dark concentric material appears to adhere bubbles of various sizes (photo 60002B50X014_02). At right, dark field image shows a thin rope like feature around smallest bubble (photo 60002D50X014_04). Scale bars are 50µm.

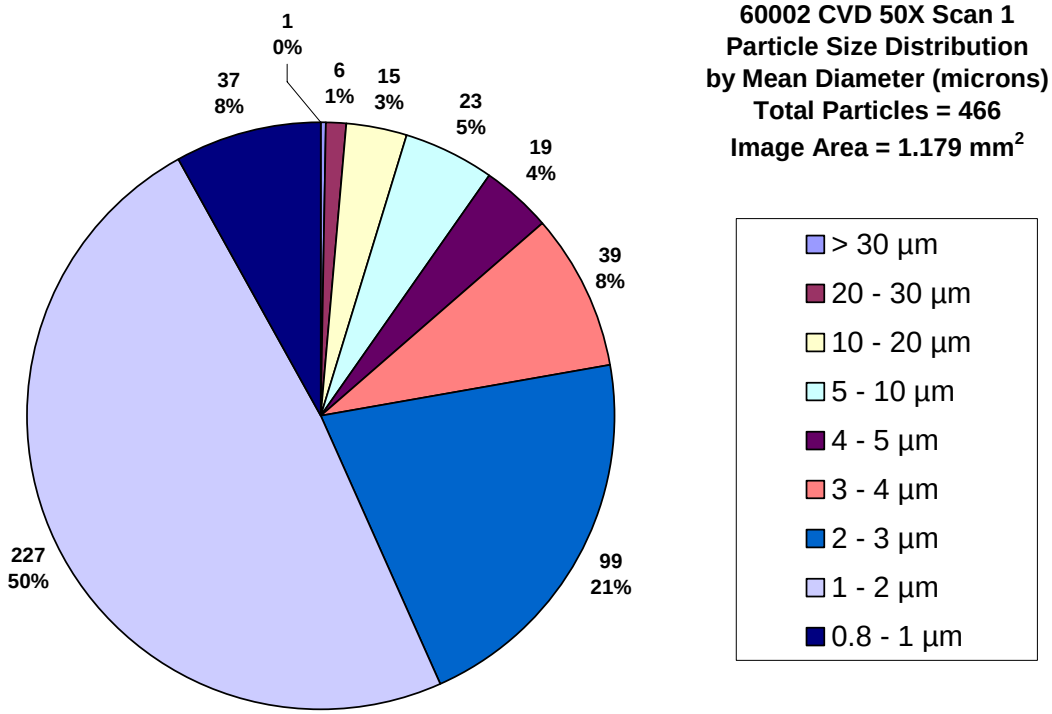
60002 CVD 13C-diamond 50X Mosaic and Particle Statistics: SCAN 1



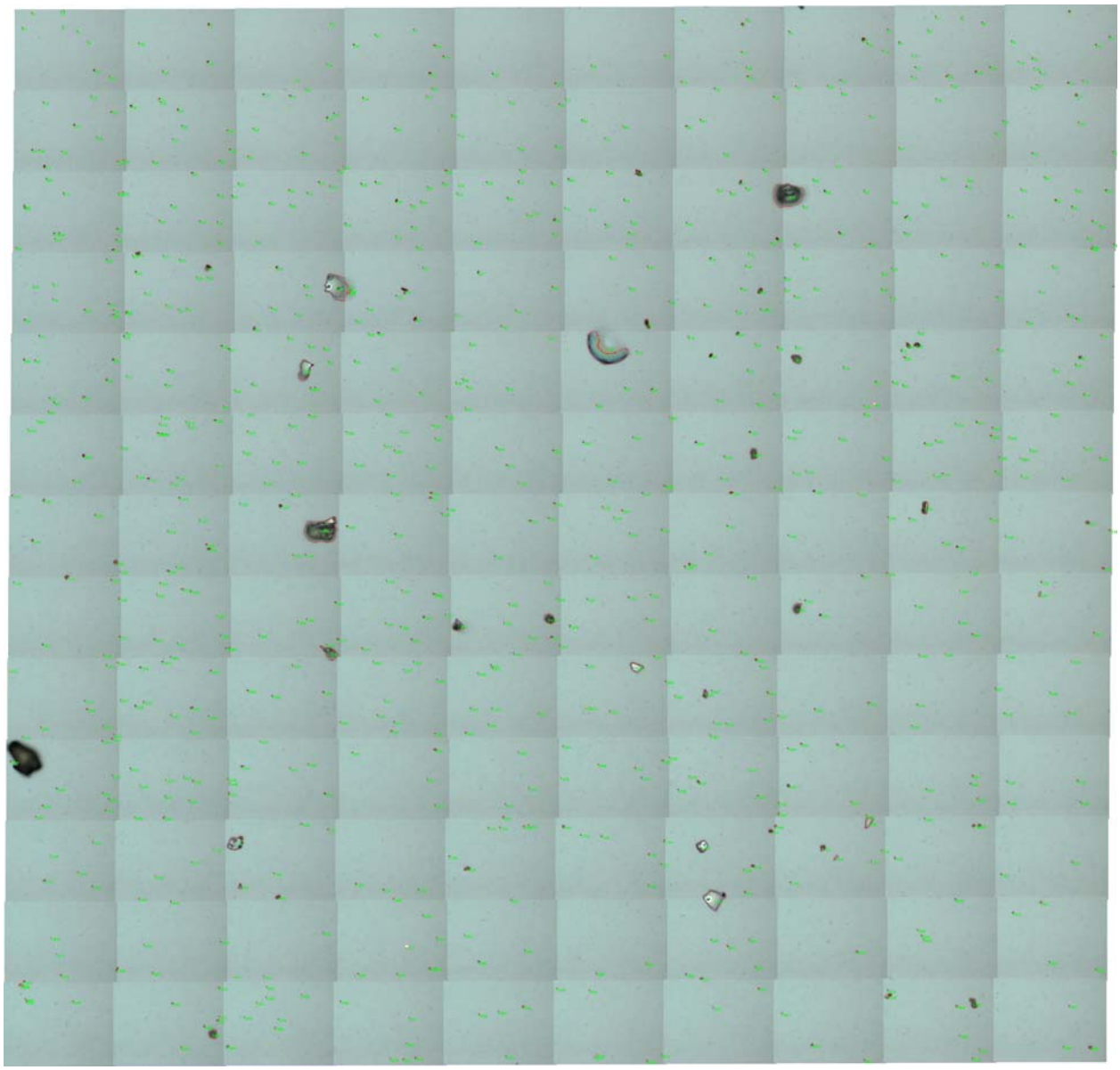
60002 50X objective scan area 1 particle map. Image area is 1.179 mm^2 with 466 particles identified > 0.3 microns. The red outline around an object is to mark where ImageProPlus has identified a particle and given the location a green number besides the object. The following three plots are the results of scan area 1 particle count using a filter to obtain all particles > 0.3 µm in diameter, length, and width.


60002 CVD 50X Scan 1 Particle Size Distribution for ~ 1 mm2
Cummulative Fraction by Number of Particles
Mean Diameter (microns)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
5
10
15
20
25
30
35
40

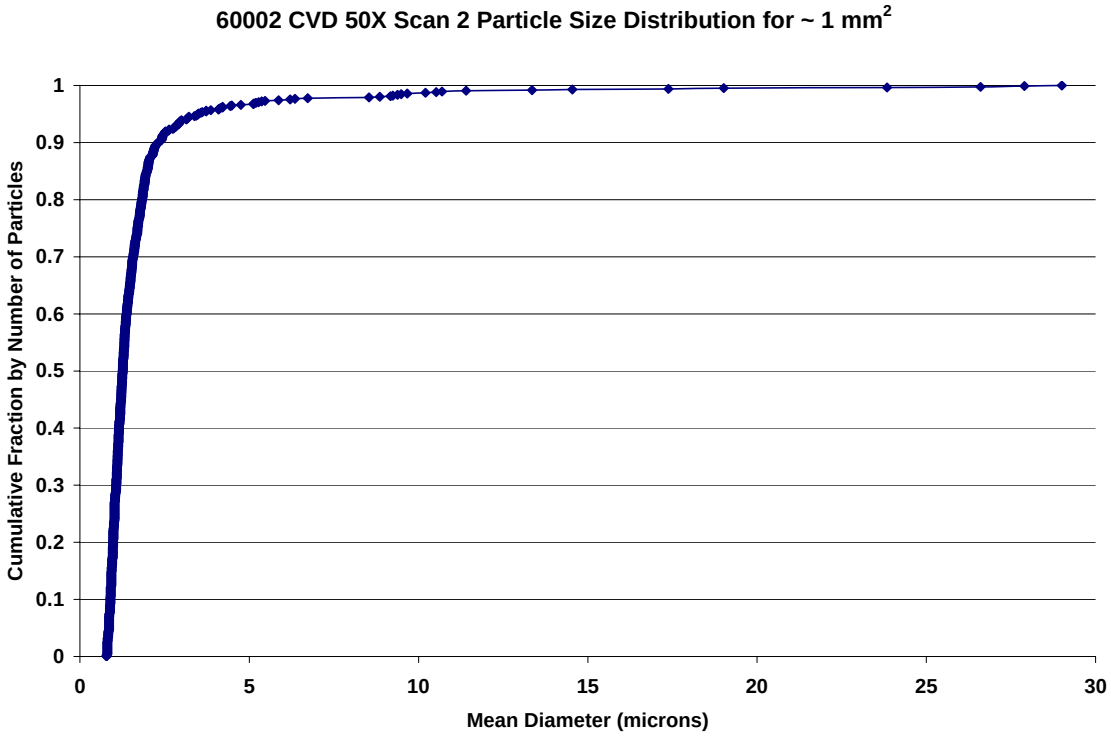

60002 CVD 50X Scan 1 Individual Particle Diameter for ~ 1mm2
Mean Diameter (microns)
Polygon Area (um2)
Size Sorted Particles (Total Particles = 466)
Diameter
Area
0
5
10
15
20
25
30
35
40
0
100
200
300
400
500
600
700
800
900
1000
1100
1
41
81
121
161
201
241
281
321
361
401
441

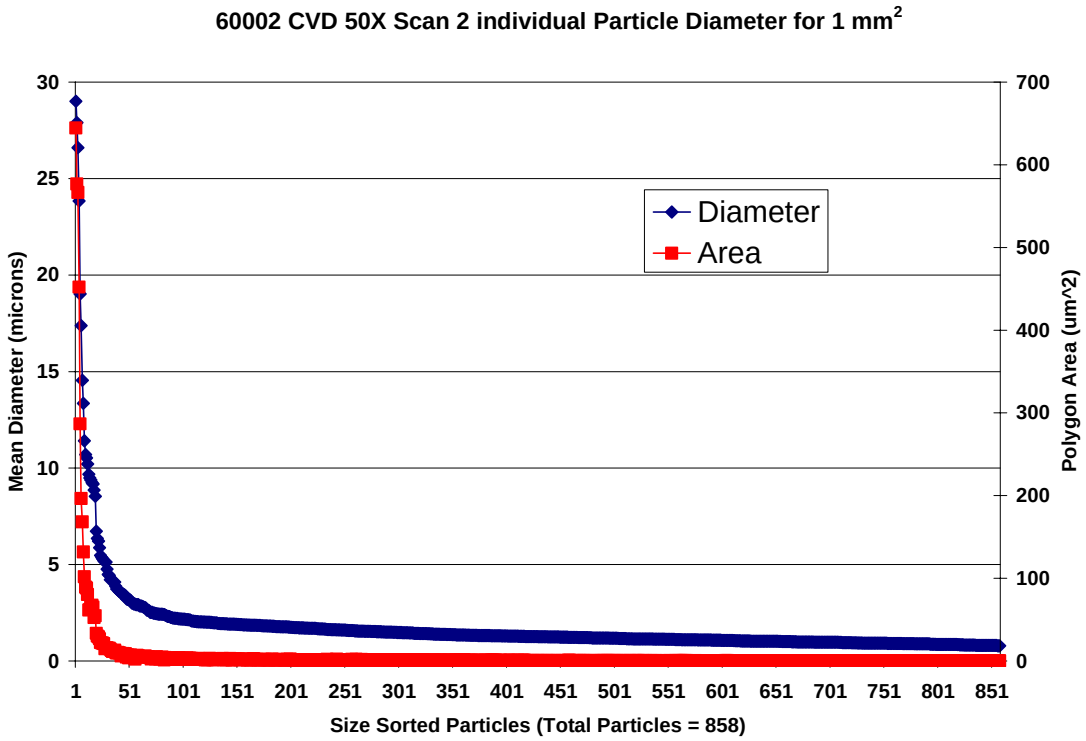

60002 CVD 50X Scan 1
Particle Size Distribution
by Mean Diameter (microns)
Total Particles = 466
Image Area = 1.179 mm2
1
0%
6
1%
15
3%
23
5%
19
4%
39
8%
99
21%
227
50%
37
8%
> 30 µm
20 - 30 µm
10 - 20 µm
5 - 10 µm
4 - 5 µm
3 - 4 µm
2 - 3 µm
1 - 2 µm
0.8 - 1 µm

SCAN 2



60002 50X objective Scan Area 2 particle map. Image area is 1.179 mm^2 with 858 particles identified > 0.3 microns. The red outline around an object is to mark where ImageProPlus has identified a particle and given the location a green number besides the object. The following three plots are the results of scan area 2 particle count using a filter all particles > 0.3 µm in diameter, length, and width.


60002 CVD 50X Scan 2 Particle Size Distribution for ~ 1 mm2
Cumulative Fraction by Number of Particles
Mean Diameter (microns)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1
0
5
10
15
20
25
30


60002 CVD 50X Scan 2 individual Particle Diameter for 1 mm2
Mean Diameter (microns)
Polygon Area (um^2)
Size Sorted Particles (Total Particles = 858)
Diameter
Area
0
5
10
15
20
25
30
0
100
200
300
400
500
600
700
1
51
101
151
201
251
301
351
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451
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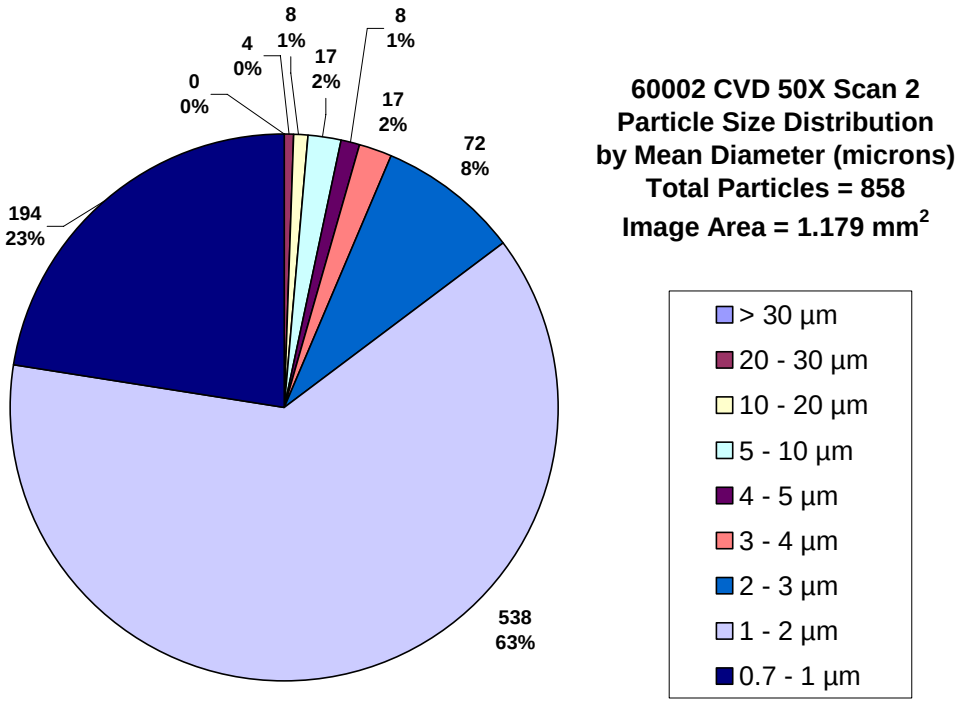
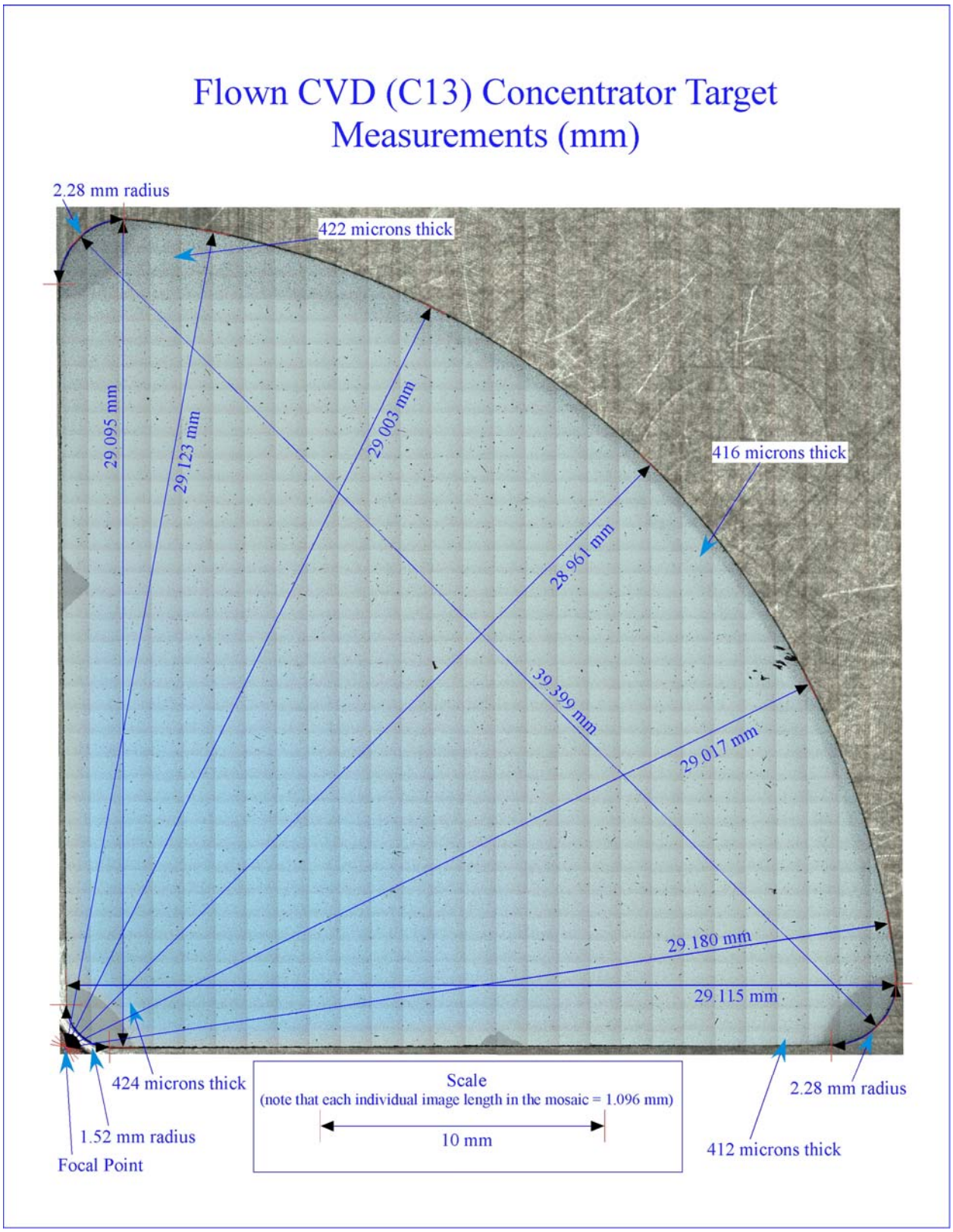
551
601
651
701
751
801
851


8
1%
8
1%
4
0%
17
2%
0
0%
17
2%
72
8%
194
23%
538
63%
60002 CVD 50X Scan 2
Particle Size Distribution
by Mean Diameter (microns)
Total Particles = 858
Image Area = 1.179 mm2
> 30 µm
20 - 30 µm
10 - 20 µm
5 - 10 µm
4 - 5 µm
3 - 4 µm
2 - 3 µm
1 - 2 µm
0.7 - 1 µm

Appendix 1: Quadrant Dimensions



Contamination on Genesis Flown Concentrator Acceleration Grid:
SEM/EDS Analysis Analog of Particle Contamination on Genesis Targets
Appendix 2
12/1/2007

SEM analysis has not been considered a non-destructive analytical technique on the flown concentrator targets. In general, the uses of routine use vacuum chambers have the potential for further contaminating the targets. However, particle contamination found on the flown concentrator's acceleration grid is a close analog for particle contamination on the flown concentrator targets. SEM analysis was performed on an unknown contamination glob from the acceleration grid. The ~ 1-cm triangular glob in Fig.1 and 2 is an extremely pliable, stretchable, colorless, transparent ("gooey") material in which microspheres and black fibers were trapped. The glob was removed from the concentrator stainless steel mesh acceleration grid by cutting away the mesh portion containing the glob. The glob was too elastic to subdivide and therefore required analysis of the entire specimen. EDS spectrums show that this material is carbon with no other trace elements. The following figures show typical contamination found in the concentrator acceleration grid.

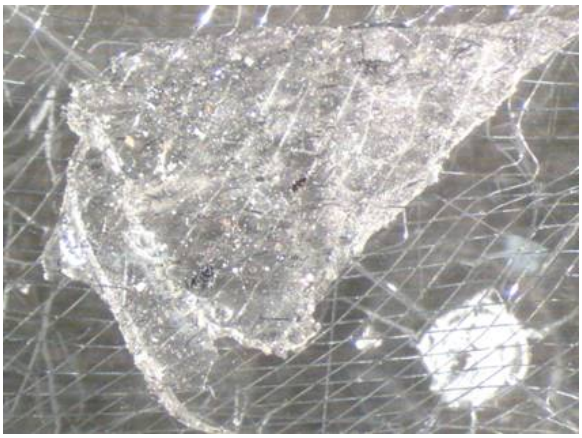


Fig. 1: This is a stereoscope image of the unknown transparent contamination glob before cutting. Field of View Length = ~ 1.0 cm.

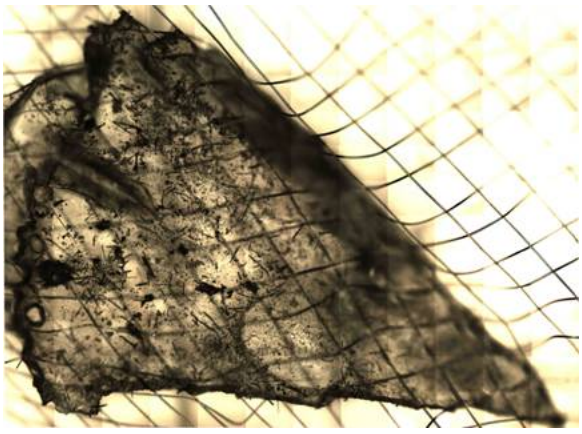


Fig. 2: This is a mosaic image with the 5X objective of the unknown transparent contamination glob before cutting. Field of View length = ~ 1.3 cm.

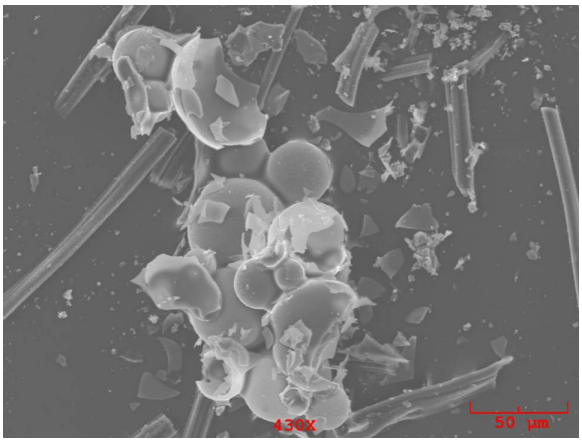


Fig. 3: Image of microspheres surrounded by carbon-carbon fibers. Microsphere fragments are a common contaminate found on the concentrator targets. The composition of the microspheres is consistent with soda-lime glass coated with a carbon-rich binding agent (possibly a type of silicone adhesive). The most likely source is the foam filler used in the super-light ablator (SLA) on the SRC.

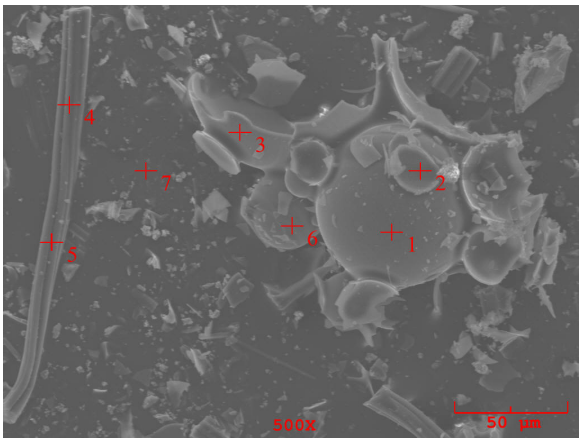


Fig. 4: Spot 1-3 & 6 are Si-rich microsphere fragments. Spot 4 and 5 are possible carbon-carbon heat-shield fiber. Spot 7 is the unknown carbon background transparent substance adhering to the acceleration grid in Fig. 1 and 2.

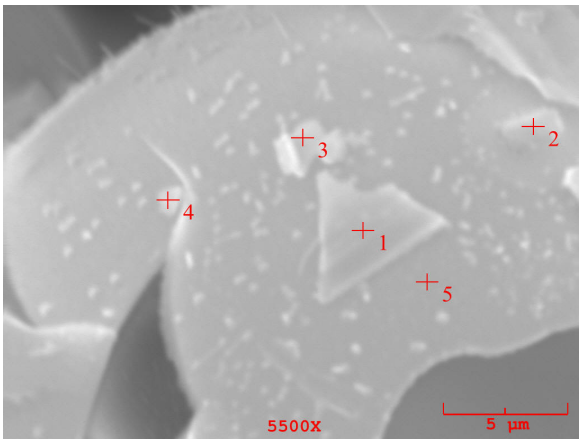


Fig. 5: This is a magnified view of a microsphere. The wall thickness can be measure to a thickness of ~ 300-400 nm.

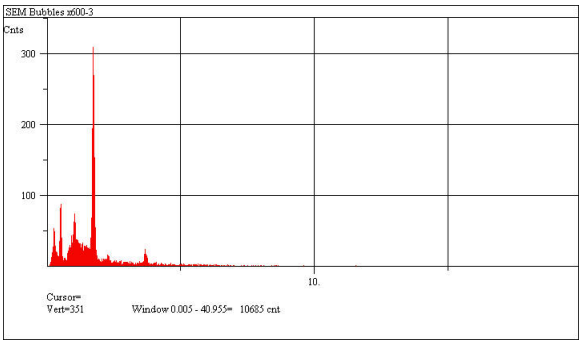


Fig. 6: EDS spectra of Fig. 5 microsphere. This spectra show a material enriched in Si with C, O, Na, and Ca. The composition of this individual microsphere is consistent with soda-lime glass coated with a carbon-rich binding agent (possibly a type of silicone adhesive).

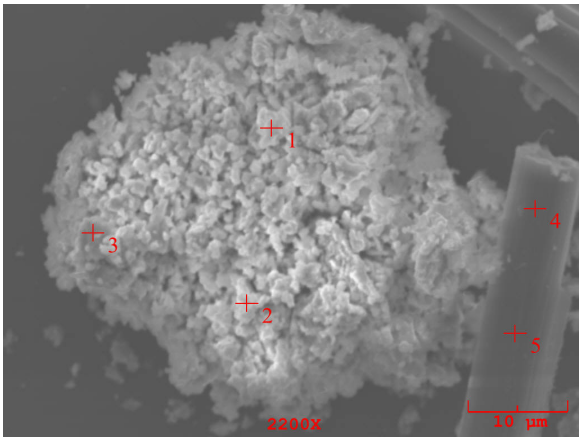


Fig. 7: The large white object is possibly white paint particle from the spacecraft (spots 1-3). On the right is chemically pure carbon (spot 4 and 5); possibly carbon-carbon fiber from heat shield.

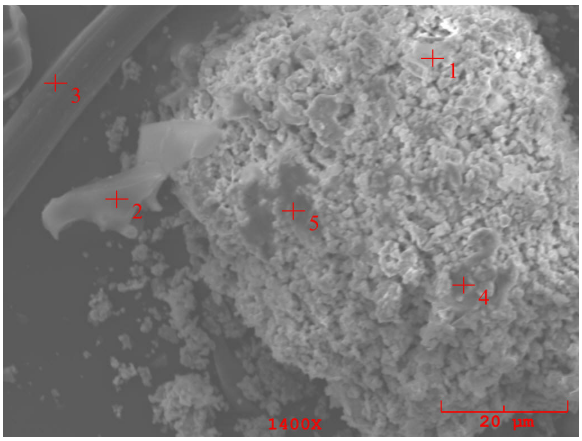


Fig. 8: The large white object is possibly white paint (spot 1, 4 & 5). Spot 2 is pure carbon and has a morphology of possible Kapton from the SRC. Spot 3 is pure carbon and is a possible carbon fiber.

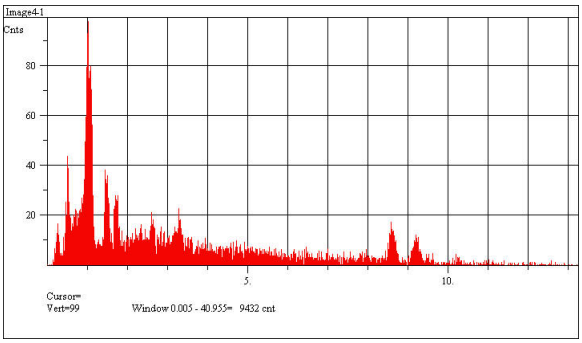


Fig. 9: This is a EDS spectrum of the possible white paint in Fig. 8 with elemental energy peaks for C, O, Na, Al, Si, Zn, Ga.

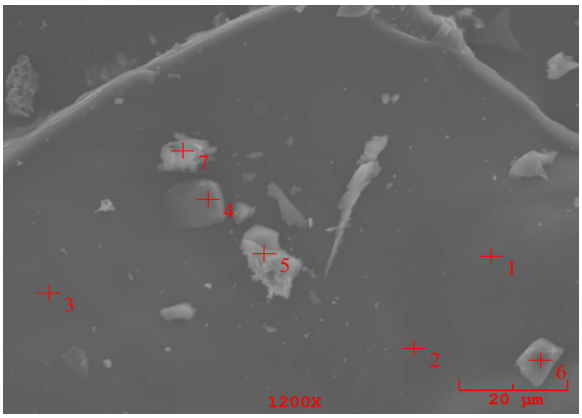


Fig. 10: This image shows the acceleration grid mesh in the upper right and left corners draped with the transparent contamination in Fig. 1 and 2. Spot 1-3 chemically show that this transparent material is pure carbon with no other trace elements. Spot 4 is possibly a Si array fragment. Spot 5, 6, and 7 are unknown pure-carbon materials.

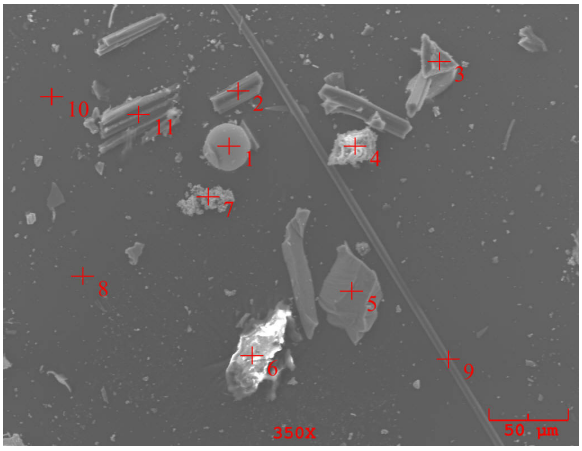


Fig. 11: Image of various contaminates. Spot 1 is a microsphere. Spot 2, 9, 11 are carbon fibers. Spot 3 is an unknown carbon fragment. Spot 4 is Si, Mg, O rich unknown substance with C, Ca, Al. Spot 7 is possible white paint. Spot 5 is a Si array fragment. Spot 6 is pure carbon (unknown origin). Spot 8 and 10 is the carbon transparent glob.

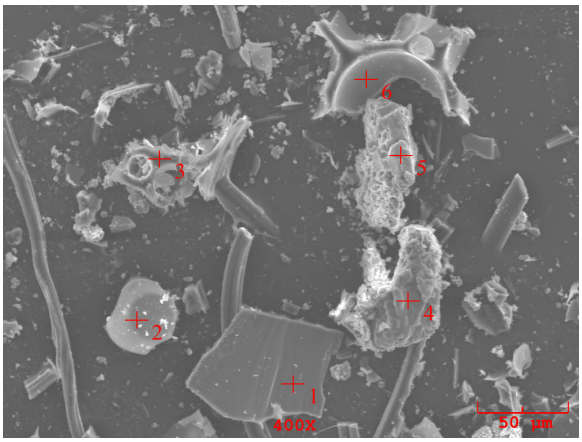


Fig. 12: Image of various contaminates. Spot 1 is a Si-array fragment. Spot 2, 3, 6 are Si-rich microspheres. Spot 4 and 5 are pure NaCl (halite).

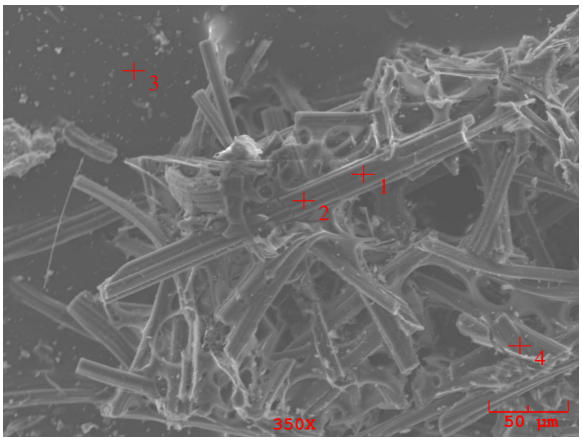


Fig. 13: Image of a pile of carbon-carbon fibers possibly from the SLA heat shield (spot 1, 2, 4). Spot 3 is the carbon transparent glob.

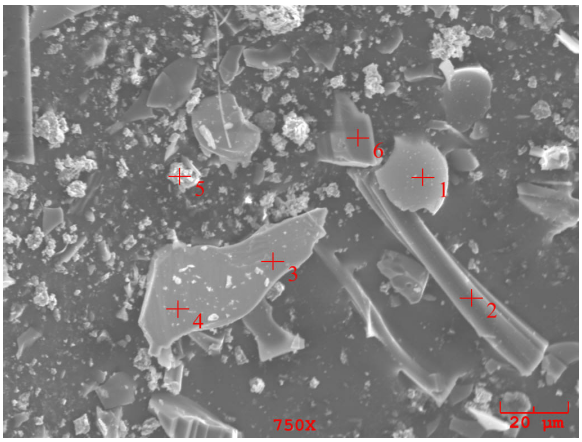


Fig. 14: Various shard fragments. Spot 1 is a Si-rich microsphere. Spot 2 is a carbon Fiber. Spot 3 and 4 are both Ge array fragments. Spot 5 is possibly white Paint. Spot 6 has no Data (too much noise in spectrum).

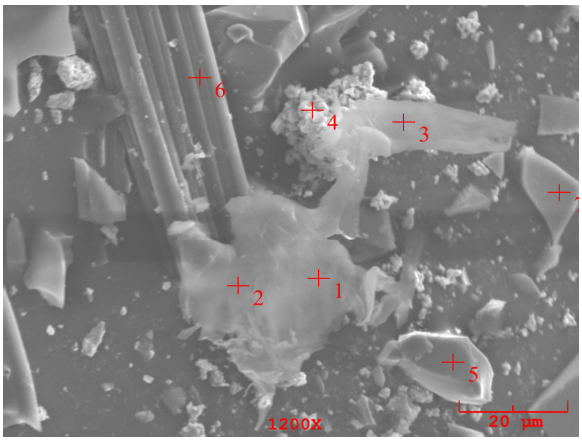


Fig. 15: The center material is pure carbon and has the morphology of Kapton (spot 1-3). Spot 4 is possibly white paint. Spot 5 is pure Al with some Al_2O_3. Spot 6 is a carbon fiber. Spot 7 is an unknown particle that is Si-rich with Ca and C.

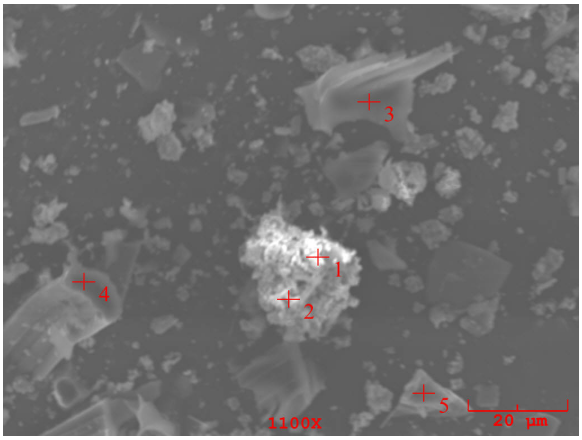


Fig. 16: Spot 1 and 2 is the possible white paint. Spot 3 is an unknown carbon substance. Spot 4 is a carbon fiber. Spot 5 is an Al fragment.

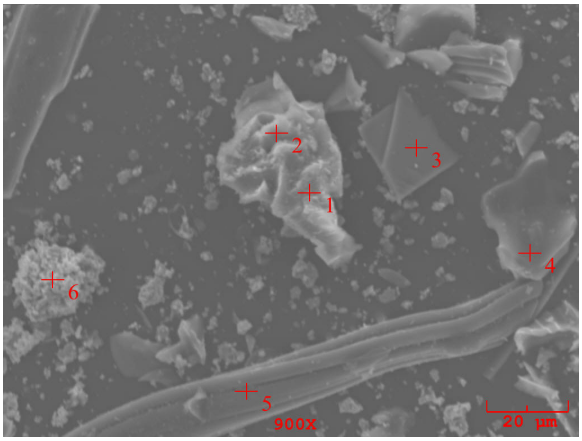


Fig. 17: Spot 1 and 2 is an unknown carbon substance. Spot 3 is a Si array fragment. Spot 4 is an unknown pure carbon substance. Spot 5 is a carbon fiber. Spot 6 is the possible white paint.