University of California
Lawrence Livermore National Laboratory

Stardust Preliminary Examination
Cratering Sub-Team

Microscopic Analysis of Aluminum Foils C2100N, C2054W, C2092W and C2027N from the Stardust Sample Tray Assembly.

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1. Samples Allocation

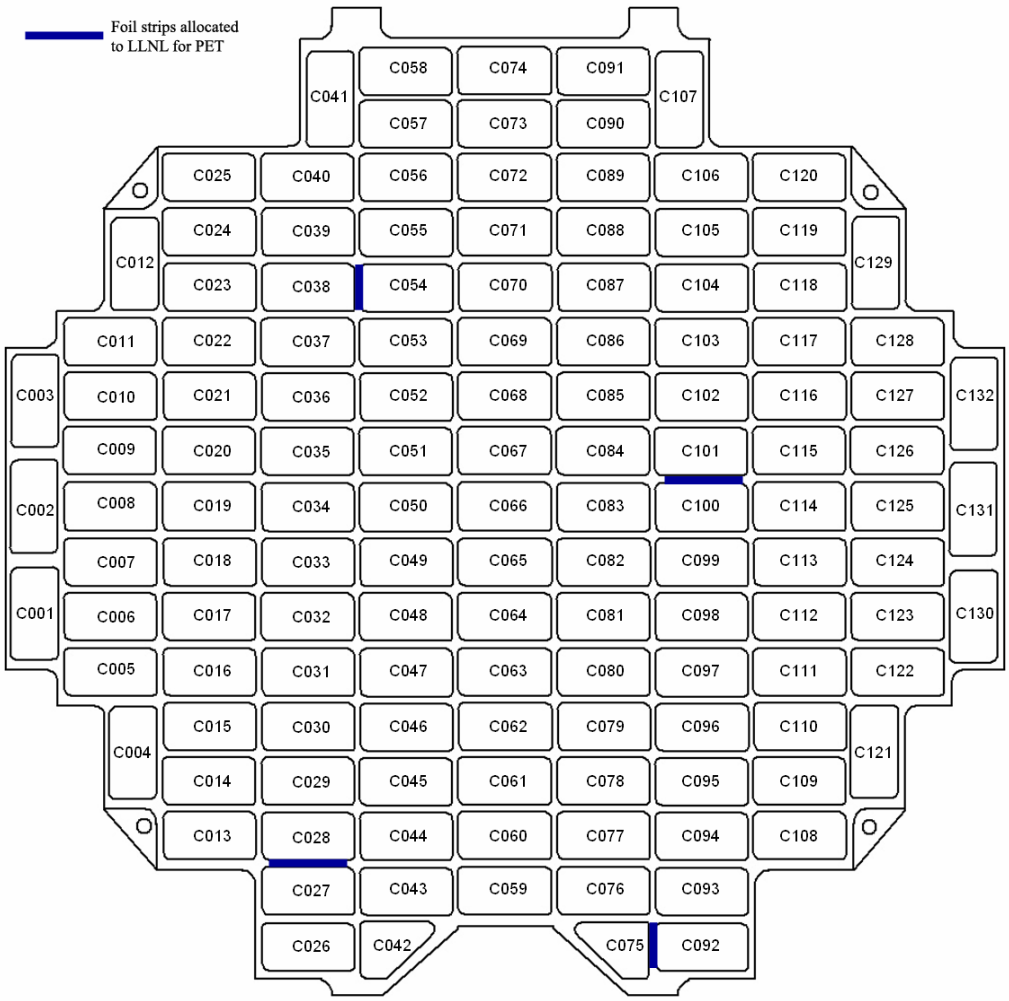


Figure 1. A schematic diagram of the Stardust Sample Tray Assembly (cometary side). The blue lines indicate the location of the aluminum foils (**C2027N**, **C2100N**, **C2092W** and **C2054W**) strips sent to Lawrence Livermore National Laboratory for SEM characterization (image courtesy of NASA).

2. Instrumentation & Methodology:

All foils analyzed were initially scanned optically at NASA-JSC Stardust curation facility by the NASA-JSC cratering team. At Lawrence Livermore National Laboratory the foils were mounted on a high-purity aluminum plate and held in place using aluminum foil such that the Stardust foil did not come into contact with carbon-based adhesive material.

All the foils were scanned using an FEI Nova 600 dual-beam focused ion beam/field emission scanning electron microscope operating at 5kV. Energy dispersive spectroscopy (EDS) was performed at 5 kV using an EDAX Genesis system. On selected craters extracted using focused ion beam milling detailed transmission electron microscopy was performed using a 200 kV FEI Tecnai G2 F20 UT (scanning) transmission electron microscope fitted with an EDAX EDX spectrometer and FEI TIA spectral processing software.

3. Analysis Overview

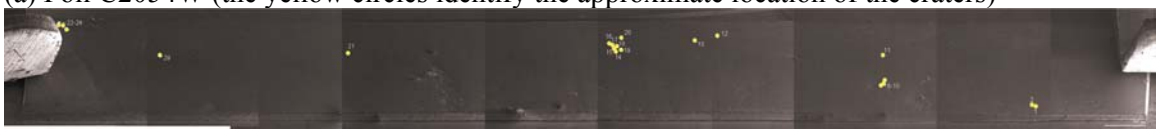
Table 1 summarizes the analysis on carried out on the craters located on the Stardust aluminum foils C2027N, C2100N, C2092W and C2054W. *Systematic crater search is defined as a series of scans at 2500x magnification over an area totaling 5mm^2.

Foil No.	Large crater (greater than 5μm)	Systematic crater search*	Craters located (less than 5μm)	Craters analyzed by EDS	craters analyzed by FIB/TEM
C2100N	-	yes	35	2	1
C2054W	1	-	29	9	3
C2092W	1	-	1	1	-
C2027N	-	-	3	2	-

4. Crater Search

All of the foil samples sent to Lawrence Livermore National Laboratory were scanned for the possible presence of impact craters generated during the encounter with comet Wild-2 using a field-emission scanning (FEI Nova 600 dual-beam microscope). The scanning was carried at initially 150x magnification at 52^{o} but was then revised to 120x magnification at 0^{o} tilt at these magnifications it is possible to locate craters down to approximately 5μm diameter (figure 2). The initial low magnification scan of foil C2100N failed to identify any impact features. The low magnification scan of foil C2054W identified one crater (Dc = 7.95μm, see figure 3 and section 6) however a random search at 800x identified a small sub-micron crater (figure 4). Further random scanning of foil C2054 at 1000x and 1500x magnifications identified a further 28 small craters with diameters ranging from approximately 3μm to 105nm (figure 5 and table 2). Similar observations were made by fellow cratering sub-team members (see reports by Borg and Hoppe) as a result the Stardust PE Cratering sub-team implemented a systematic survey strategy at higher magnification (2500x) of the Stardust foils. Foil C2100N was selected as part of this of this strategy and the detailed results are given in section 5. The low magnification scan of foil C2092W identified a single crater (figures 2b and 6) and was not subjected to a higher magnification scan. The low magnification scan of foil C2027N did not identify any micrometer-sized craters however a randomly selected 2500x magnification scan identified 3 craters below one micrometer diameter.

(a) Foil C2054W (the yellow circles identify the approximate location of the craters)



(b) Foil C2092W (the red circle identifies the location of the crater)

Figure 2. Montages of secondary electron images of (a) foil C2054W and (b) foil C2092W.

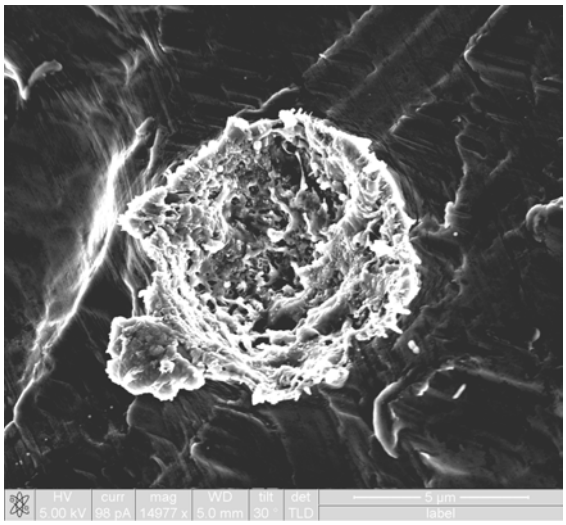


Figure 3. A secondary electron image of the shallow 7.95μm diameter crater preserved on the surface of foil C2054W.

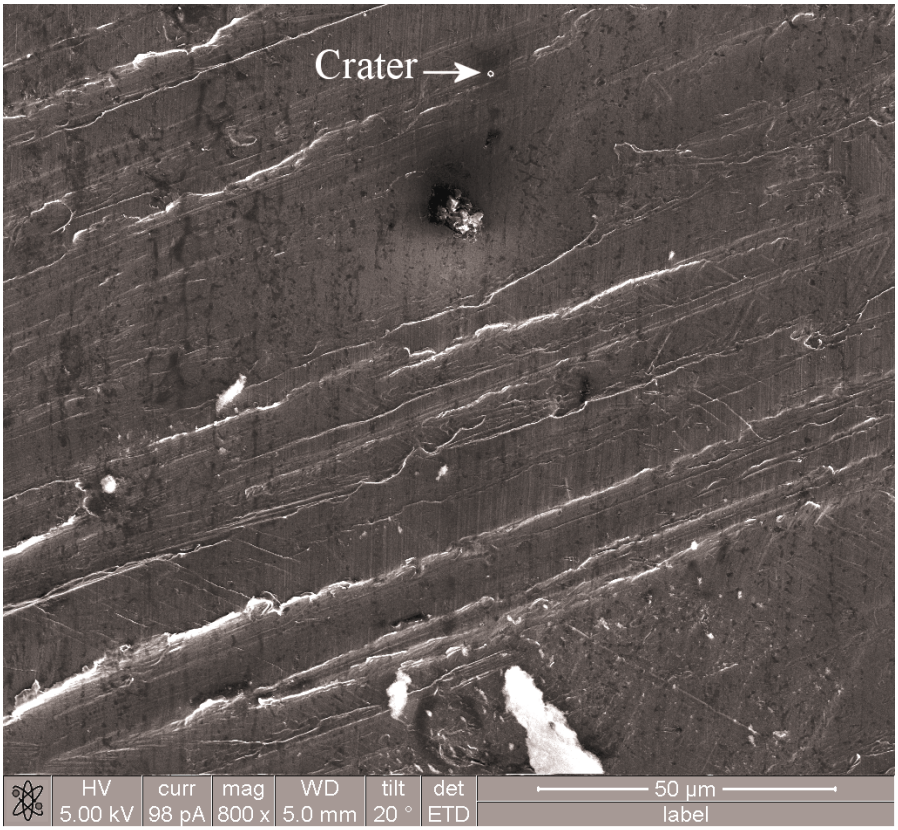


Figure 4. A secondary electron image at 800x magnification that shows a sub-micrometer crater.

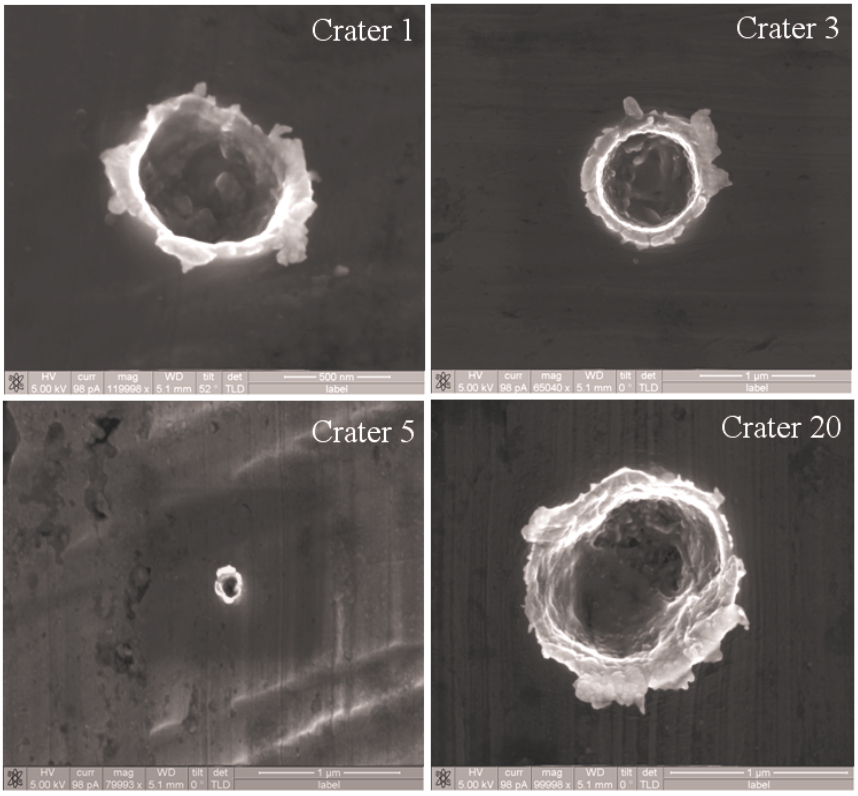


Figure 5. Secondary electron images of the typical craters identified on the surface of foil C2054W.

Crater No.	Crater Diameter (D_c) in nm	Crater No.	Crater Diameter (D_c) in nm
1	684.01	16	487.44
2	519.32	17	396.49
3	965.77	18	607.49
4	303.55	19	331.45
5	181.67	20	1170.00
6	422.49	21	404.15
7	311.84	22	412.69
8	839.11	23	1800.00
9	536.48	24	616.32
10	222.65	25	328.10
11	2950.00	26	350.69
12	7950.00	27	104.70
13	1050.00	28	550.92
14	1240.00	29	1740.00
15	430.70		

Table 2. The crater diameters (D_c) for the craters identified on the surface of foil C2054W (all diameters are in nanometers)

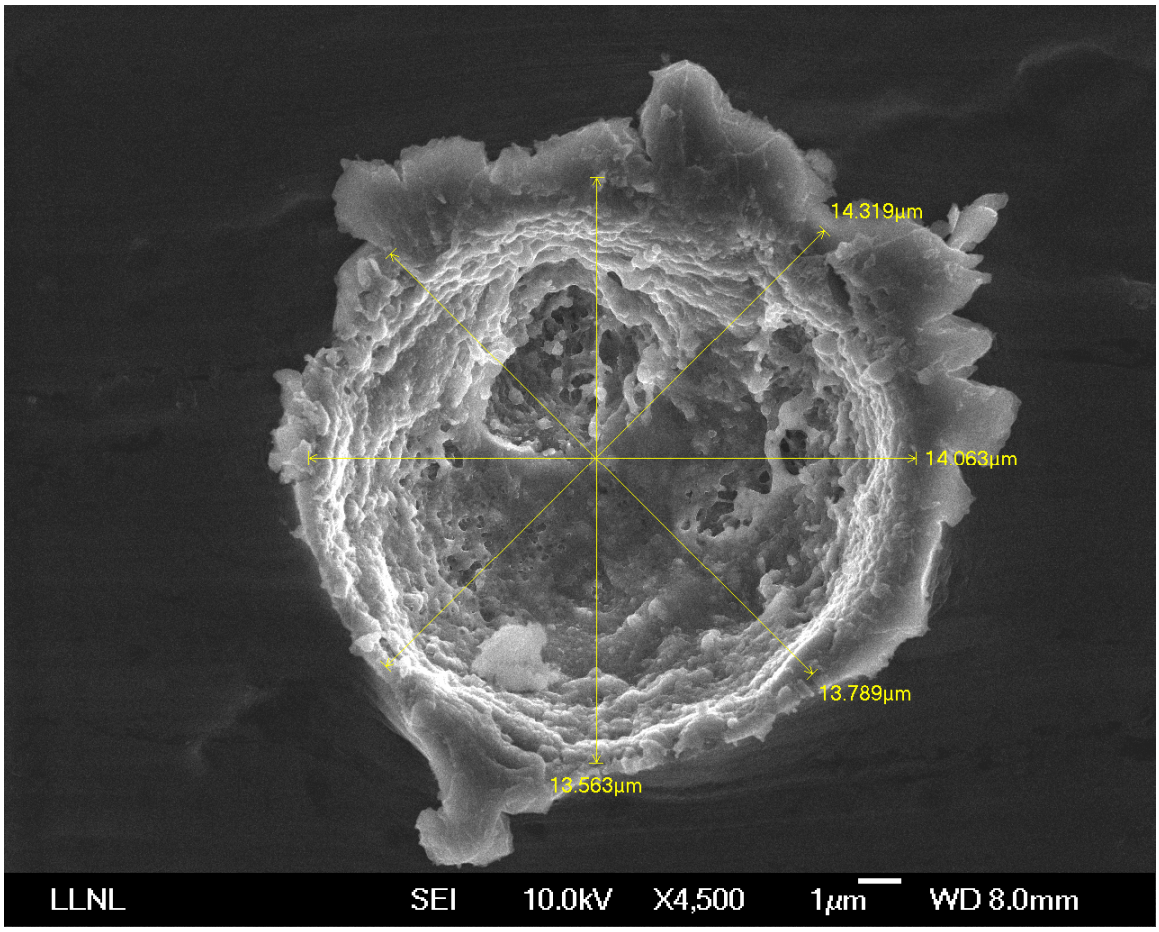


Figure 6. A secondary electron image of the crater identified in the low magnification scan of foil C2092W (image acquired using a JEOL JSM 7401F FESEM). The image shows distances used to measure the crater diameter (Dc).

5. Systematic Crater Survey

Foil C2100N was selected for a detailed survey scan at 2500x magnification to enable the identification of sub-micrometer craters preserved on the foil surface over an area totaling 5mm^2. This survey was divided into four randomly selected areas on the foil. Scan 1 was over a 1.36mm^2 area, Scan 2 was over a 1.50mm^2 area, Scan 3 was over a 1.29mm^2 area and Scan 4 was over a 0.854mm^2. The survey was performed using an FEI Nova 600 dual-beam FIB/FESEM microscope operating at 5kV. A summary of the survey is given in figures 7 to 10 and table 3.

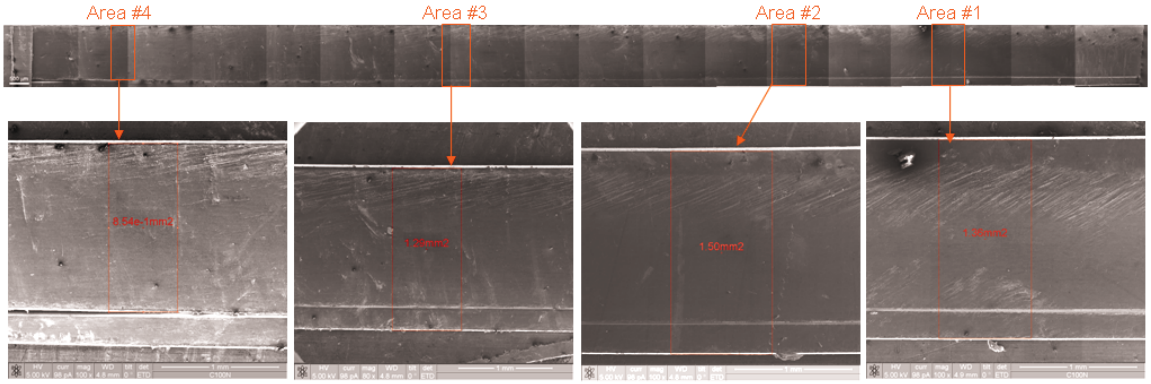


Figure 7. A montage secondary electron image of foil C2100N, identifying the location of the scanned areas used for the systematic survey. Secondary electron images of the Scan Areas 1-4.

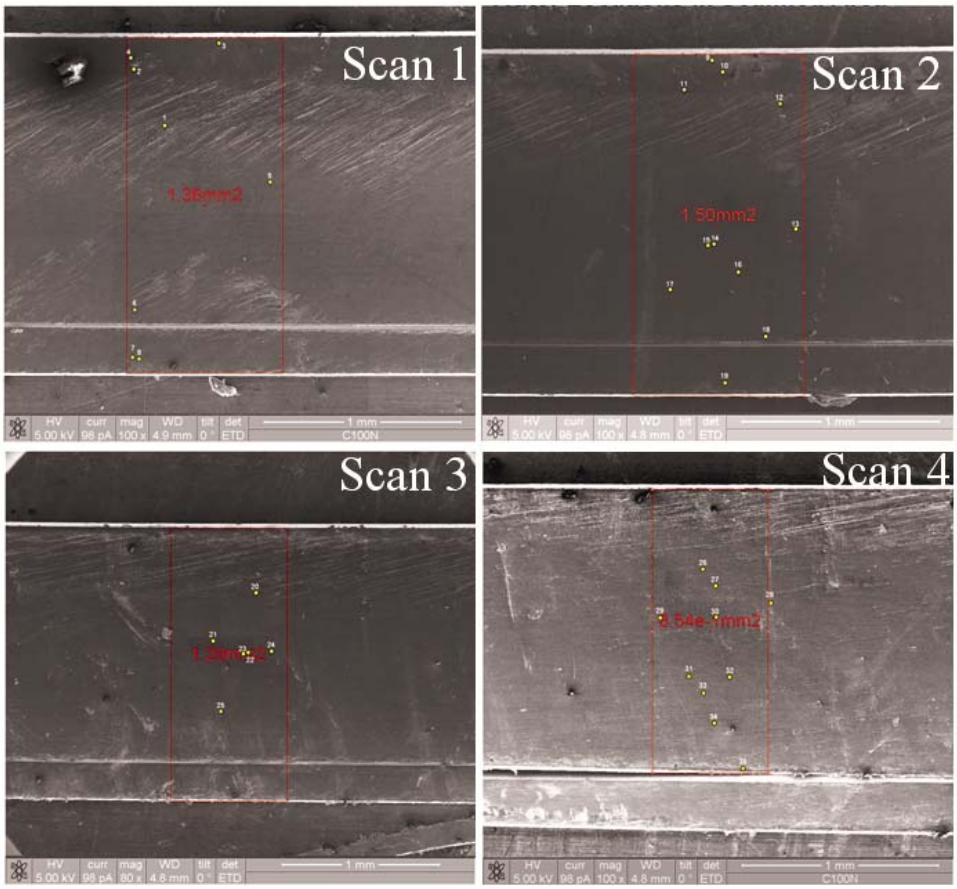


Figure 8. Secondary electron images of Scan Areas 1-4 with the location on the craters identified during the individual surveys.

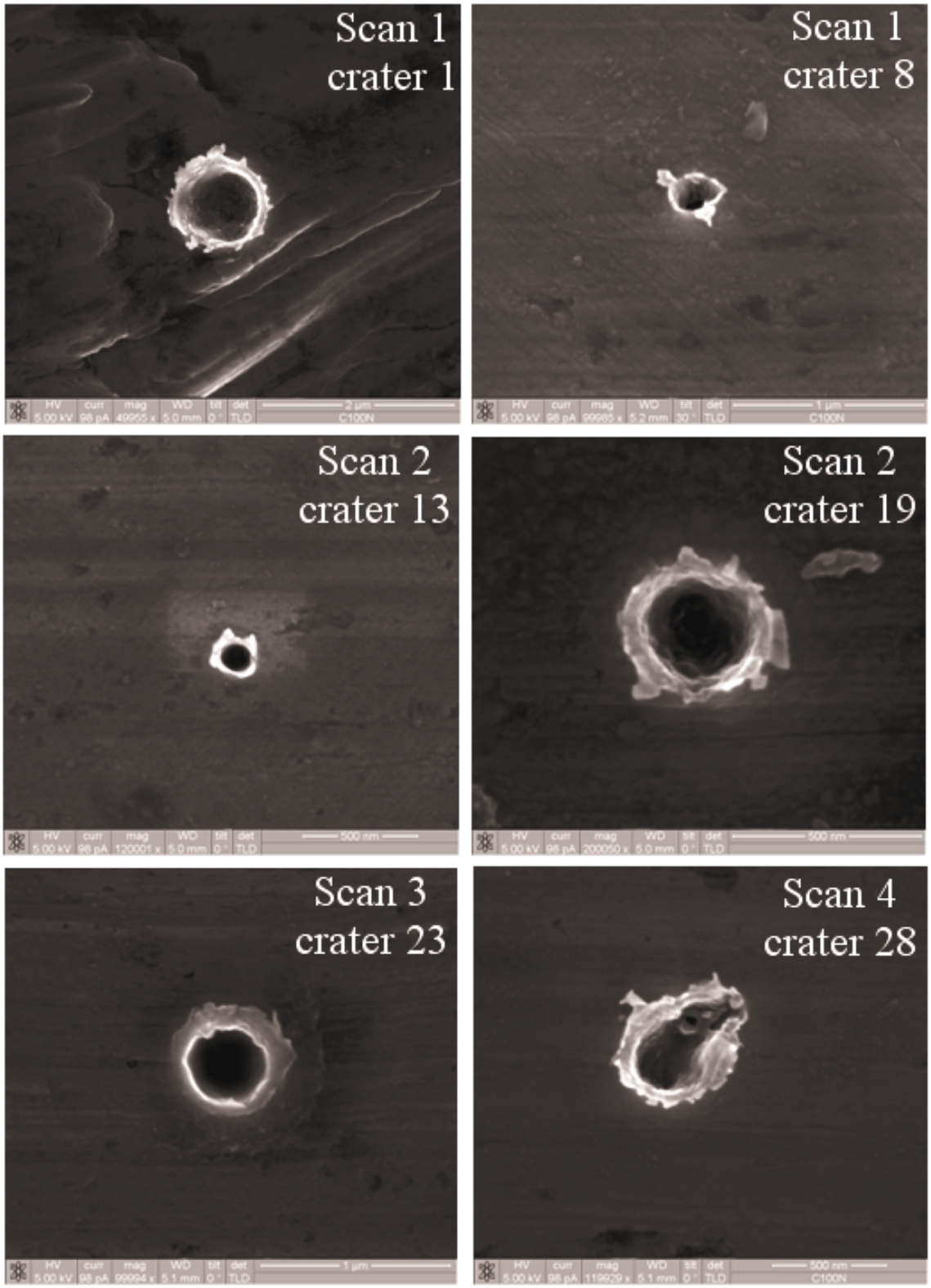


Figure 9. Secondary electron images of the typical crater morphologies observed in the systematic survey (See table 2 for the crater diameters).

Crater No.	Crater Diameter (D_c) in nm	Crater No.	Crater Diameter (D_c) in nm
1	910.20	20	448.38
2	181.88	21	281.33
3	250.00	22	212.44
4	301.50	23	475.92
5	199.97	24	172.85
6	284.30	25	257.91
7	339.00	26	357.32
8	254.55	27	438.45
9	309.31	28	428.97
10	486.57	29	595.73
11	288.24	30	294.64
12	354.50	31	258.69
13	145.52	32	183.32
14	264.34	33	319.89
15	258.62	34	308.82
16	321.20	35	341.30
17	152.36		
18	184.01		
19	303.46		

Table 3. The crater diameters (D_c) for the craters identified in scans 1 (yellow), 2 (blue), 3 (green) and 4 (red) of the systematic survey of foil C2100N (all diameters are in nanometers). The locations of the craters are shown in figure 8 on page 6.

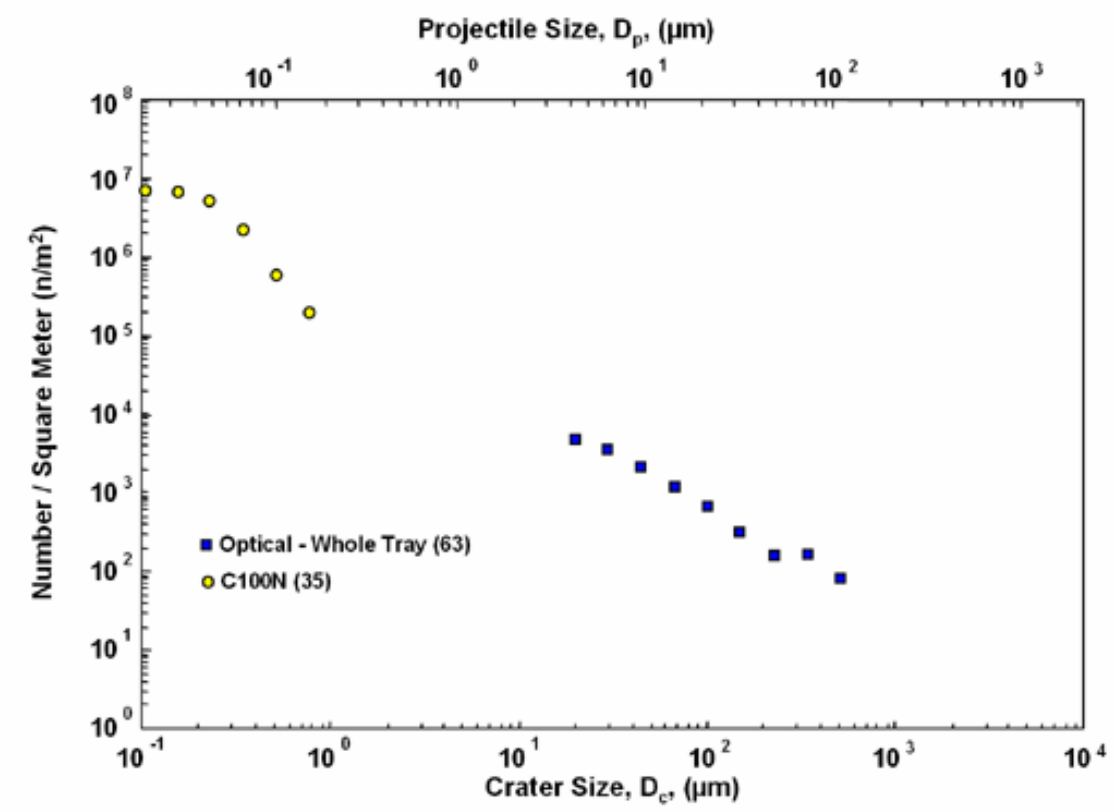


Figure 10. The crater size distribution plot for the craters identified in the systematic survey of foil C2100N along with optical data from the large crater diameter survey at NASA-JSC. Data courtesy of Fred Hörz (NASA JSC) Stardust Crater sub-team leader.

6. EDS Analysis of Craters

A total of 14 craters (2 from foil C2100N; 9 from foil C2054W; 1 from foil C2092W and 2 from foil C2027N) were analyzed by EDS (microscope operating conditions were an accelerating voltage of 5kV and integral collection time of 100s). Single spot analysis mode was used for all craters below 5µm diameter (figure 11). The 7.95µm and 13.93 µm diameter craters identified on the surface of foils C2054W and C2092W respectively were also subjected to EDS spectral mapping (figures 12 and 13). For every analysis acquired from a crater, a background spectrum was acquired for the substrate. All of the EDS (single spot and mapping) collection and post-data processing was performed using the Genesis software from EDAX. Table 4 gives a qualitative summary of the elemental composition of the craters.

Foil No.	Crater No.	EDS Elemental Data
C2054W	1	Mg, Si, Fe, S
C2054W	3	Fe, Ni, S
C2054W	11	Mg, Si, Fe, S
C2054W	12	Mg, Si, Fe, S
C2054W	13	Mg, Si, Fe, S
C2054W	14	Mg, Si, Fe, minor S
C2054W	20	Mg, Si, Fe, S
C2054W	23	Mg, Si, Fe, S
C2054W	29	Mg, Si, S, low Fe
C2100N	1	Mg, Si, Fe, S
C2100N	23	Mg, Si
C2092W	1	Mg, Si, Fe, S, minor Na
C2027N	2	Mg, Si, C
C2027N	3	Mg, Si, Fe, S

Table 4. The qualitative elemental composition of the craters analyzed by SEM/EDS.

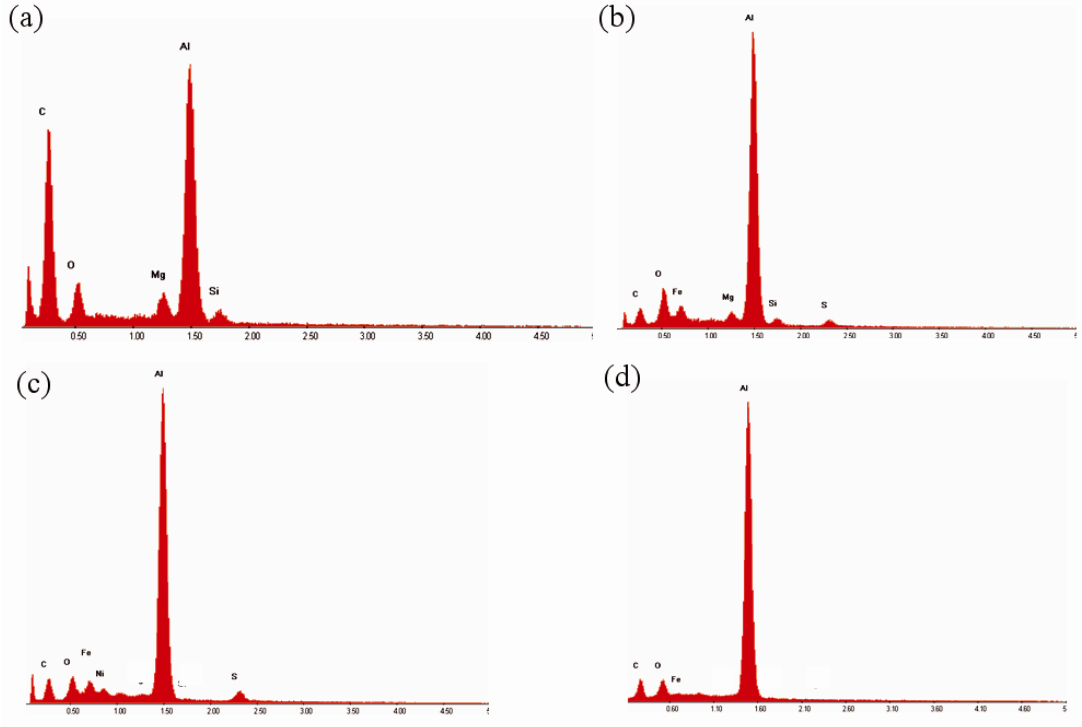


Figure 11. Examples of the EDS spectra acquire from the residue material in the foils analyzed. (a) Mg-Si, note high carbon; (b) Mg-Si-Fe-S; (c) Fe-Ni-S and (d) typical foil background.

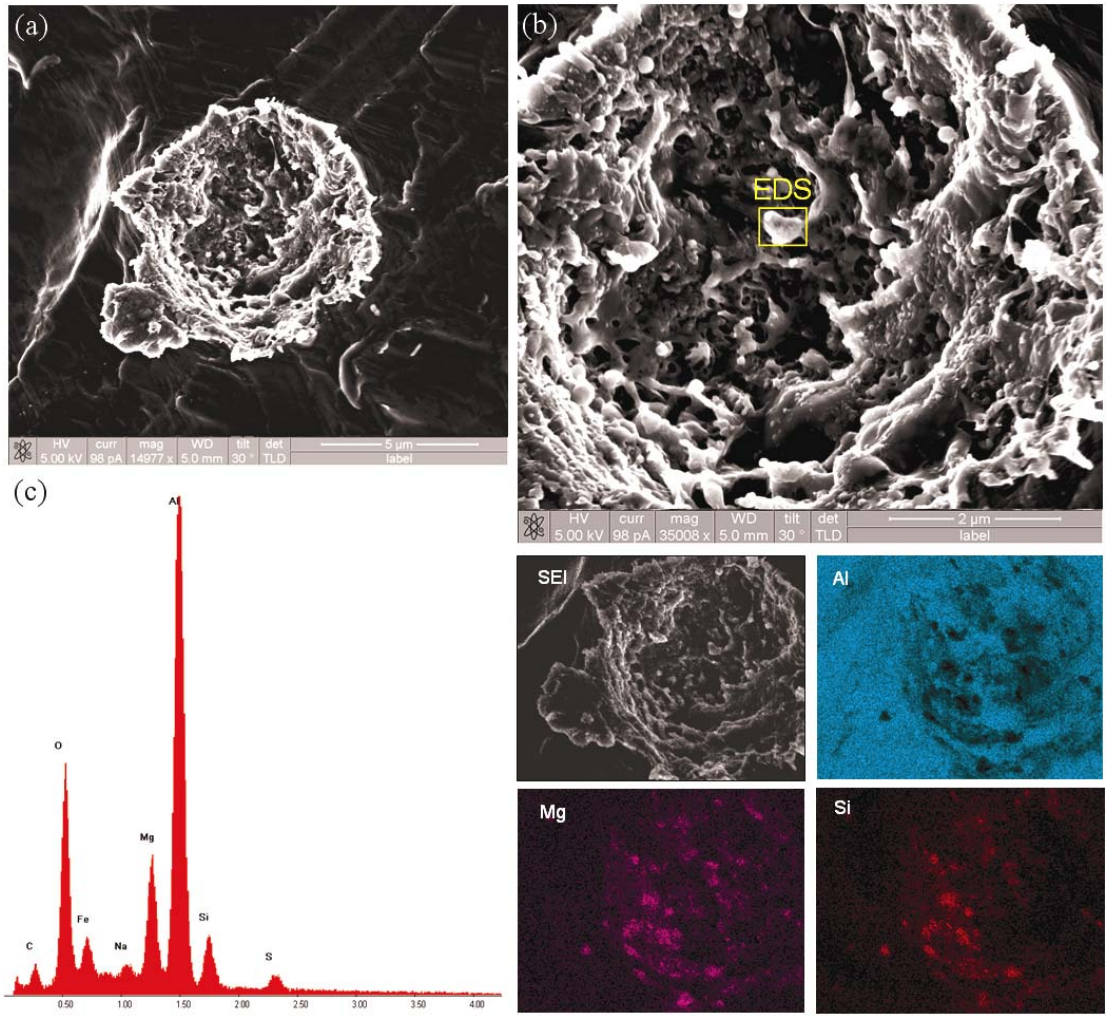


Figure 12. (a) Secondary electron image (SEI) of crater 12 from foil C2054W. (b) A higher magnification secondary electron image of the central area within the crater. (c) The 5kV EDS spectrum acquired for the impact residue melt fragment identified in image b. (d) The SEI and X-ray elemental maps acquired for Al, Mg and Si (mapping conditions: 1frame, 60ms).

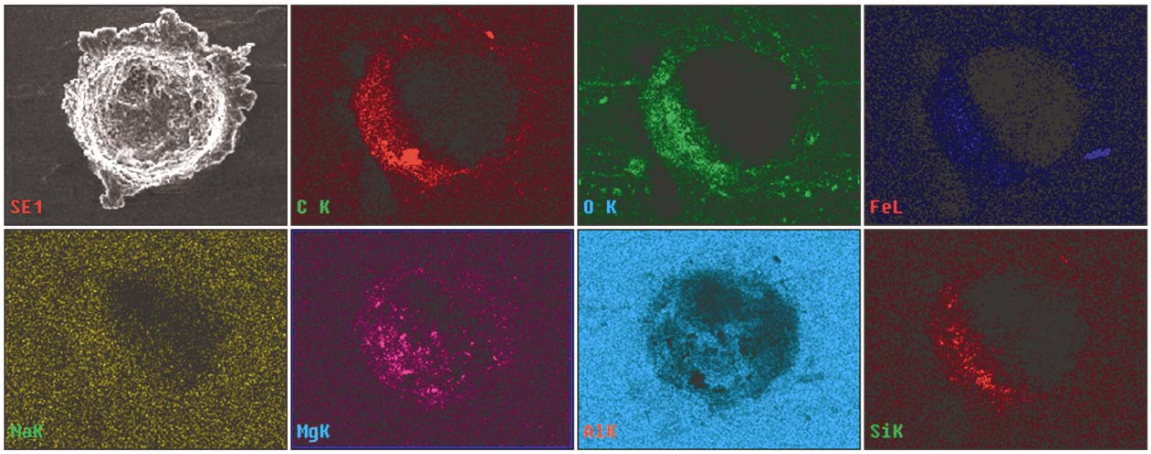


Figure 13. The SEI and X-ray elemental maps for crater 1 from foil C2092W. The Fe-map shows evidence of contamination from the foil substrate (bright area to the lower right of the crater). The Fe content within the foils analyzed has shown to be highly varied. The high carbon map is due to a fragment of aerogel.

7. FIB Sectioning and TEM Analysis

Craters 11, 13 and 23 from foil C2054W and crater 1 from C2100N have been subjected to precision focused ion beam milling to prepare electron transparent sections for detailed TEM analysis. Full description of the sample preparation techniques developed using focused ion beam microscopy for sectioning impact craters preserved in metallic foils is given in (Graham et al., 2006 and Leroux et al., 2006). The focused ion beam work at Lawrence Livermore National Laboratory was performed using an FEI Nova 600 dual-beam microscope. Figure14 gives a summary of the sample preparation.

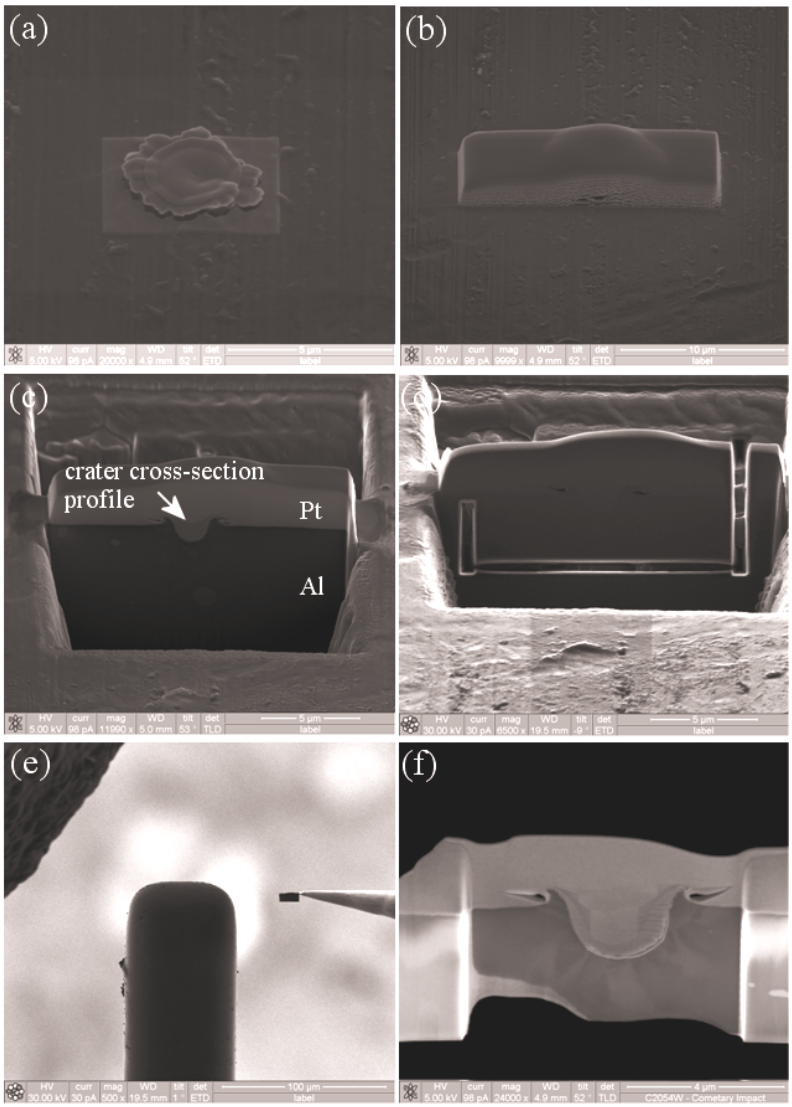


Figure 14. (a) A secondary electron image of crater 11 (foil C2054W) after a Pt "plug" has been deposited to protect the impact residue during the precision milling. (b) A Pt "strap" is then deposited over the entire crater. (c) The Focused ion beam is then used to trench either side of the Pt "strap" to produce a cross-sectional profile of the sample. (d) The section containing the cross-sectional profile us then thinned to approximately 1μm thickness after which the ion beam is used to make side wall and under cuts to enable the section to be extracted from the bulk foil. (e) The extracted section prior to being attached to the TEM grid. (f) After further low beam current milling the section is thinning to electron transparency (80-100nm thick).

The electron transparent sections containing the crater cross sections have been characterized using a 200kV Tecnai G2 (S)TEM microscope fitted with a EDAX EDS system. The post-spectral processing was performed using FEI's TIA spectral processing software.

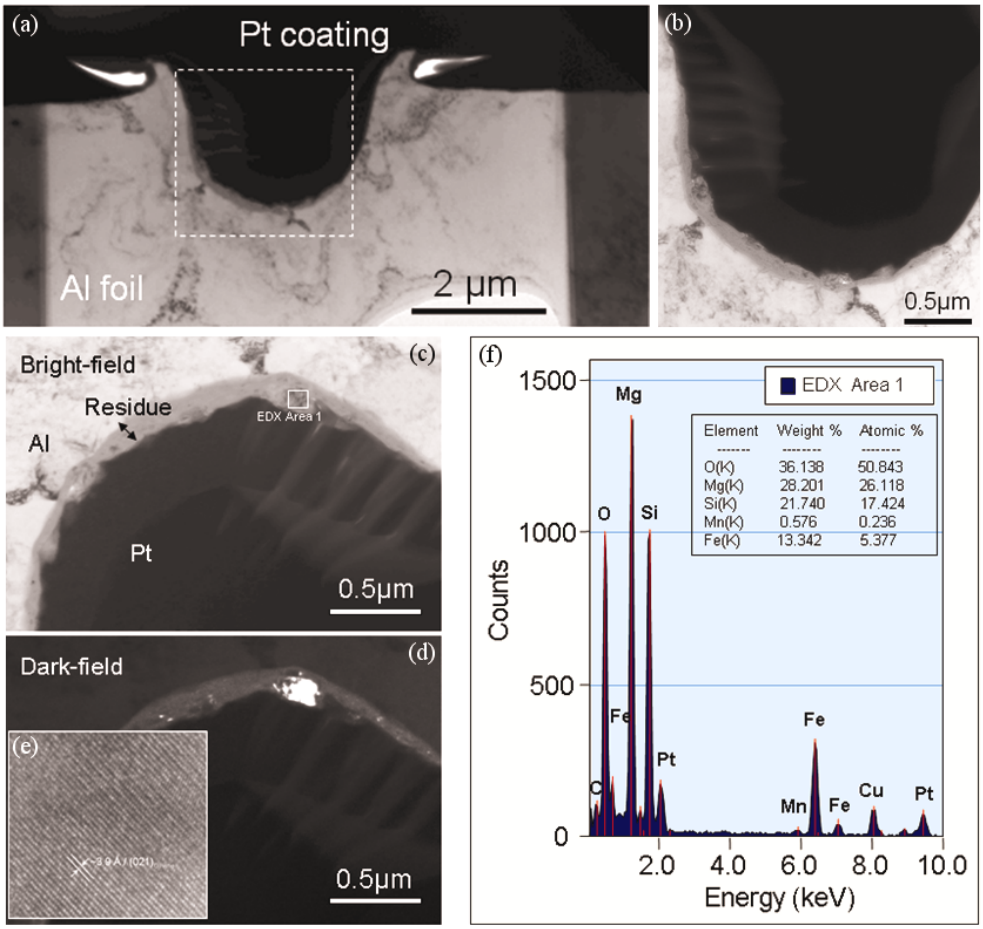


Figure 15. Imaging and EDS data acquire for crater 11 (foil C2054W). (a-c) Bright-field images of the crater and the impact residue layer. (d) A dark-field image that shows evidence of crystalline material preserved within the residue layer. (e) The lattice-fringe image acquired for the crystalline grain identified in the residue layer, which is identified as olivine. (f) The EDS spectrum acquired for the olivine grain.

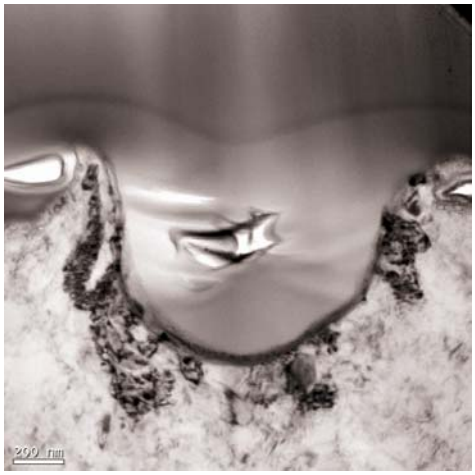


Figure 16. The bright-field image for crater 13 (foil C2054W)

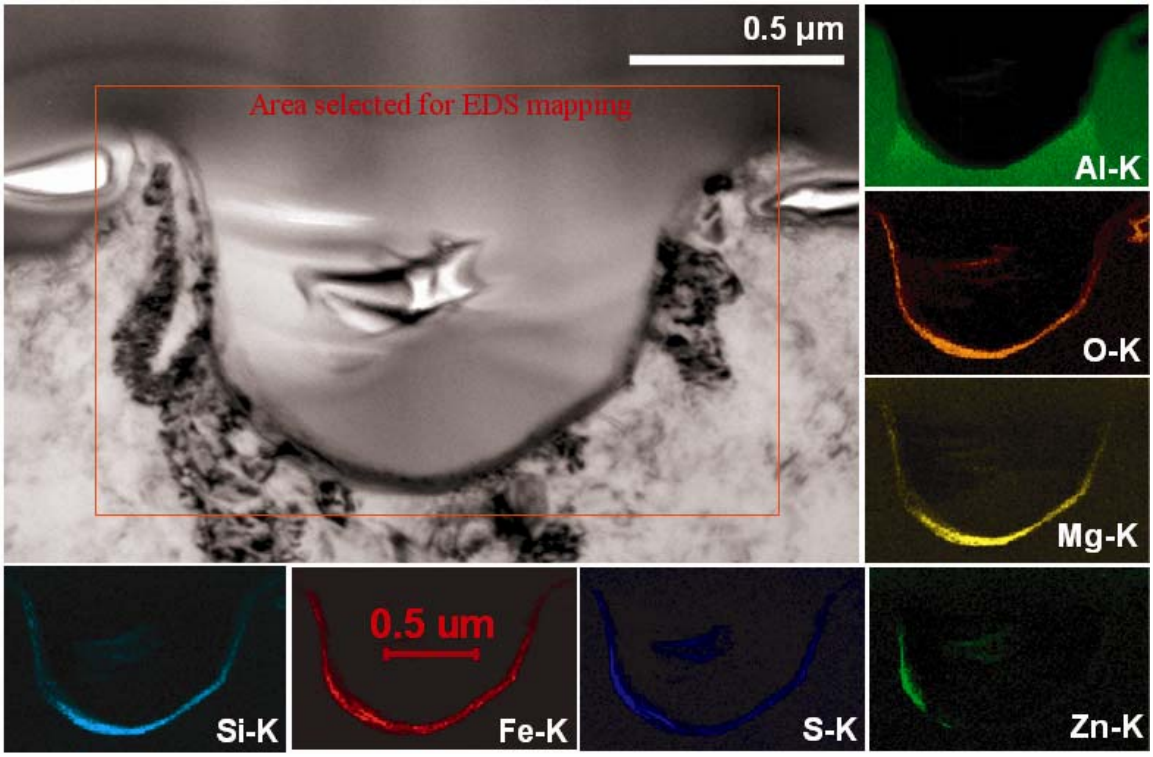


Figure 17. The bright-field image and the X-ray elemental maps acquired for the impact residue layer in crater 13 (foil C2054W).

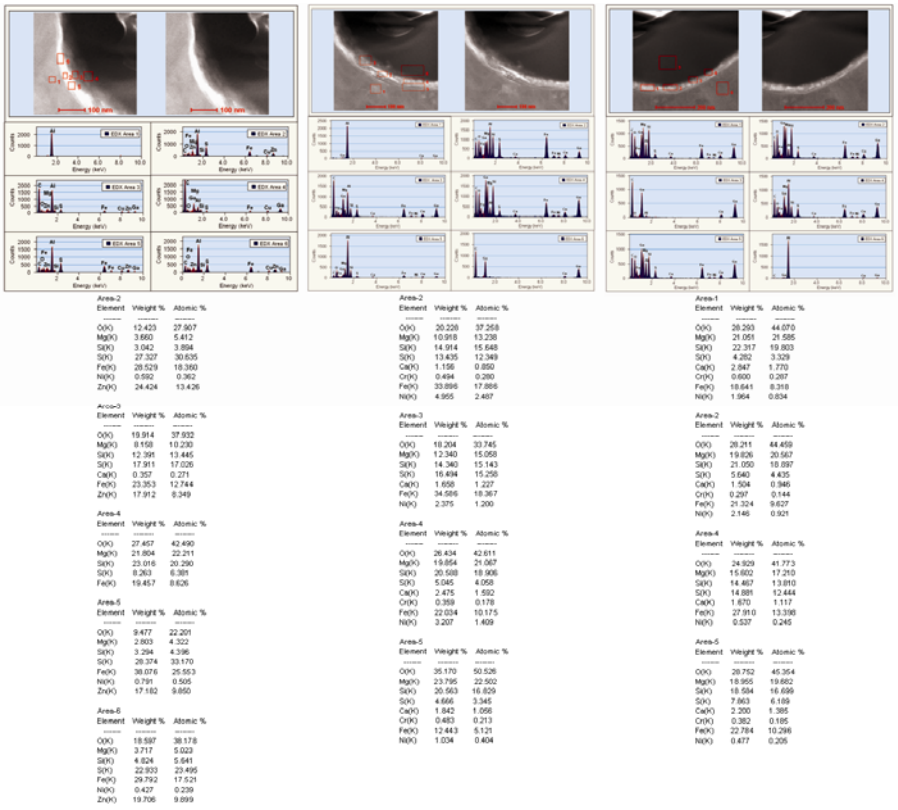
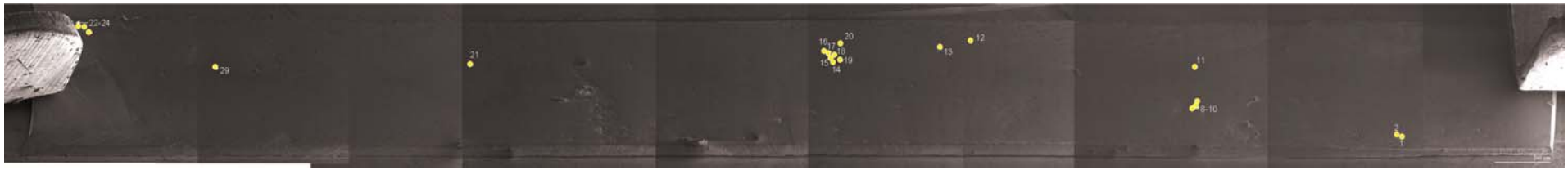


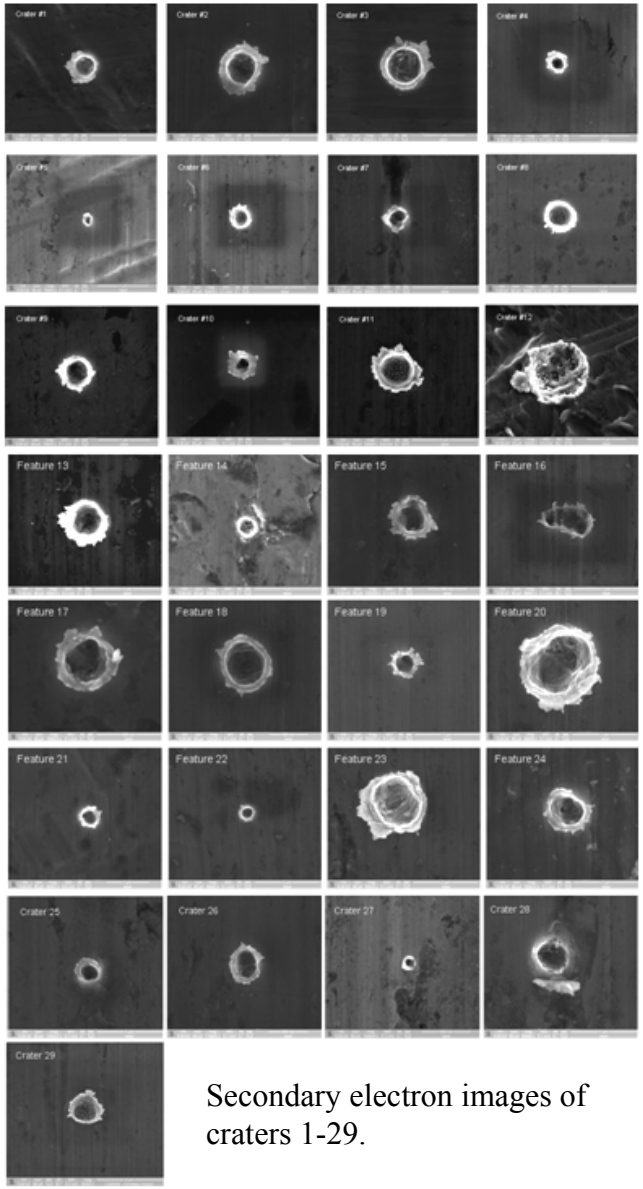
Figure 18. The EDS analysis of the residue impact layer in crater 13 (foil C2054W). The Ga peaks are associated with the focused ion beam preparation and the Cu peak is from the TEM grid.

Appendix 1: Foil C2054W, C2092W and C2027N

Summary data for foils C2054W, C2092W and C2027N.



Foil: C2054W

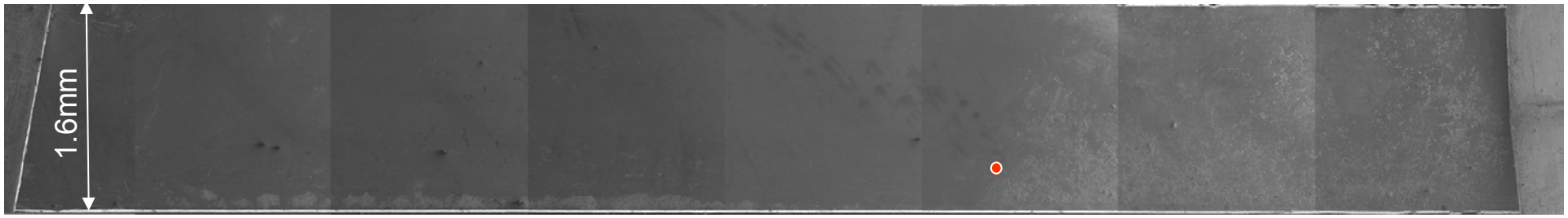


Secondary electron images of craters 1-29.

Crater No.	Crater Diameter (D_c) in nm	Crater No.	Crater Diameter (D_c) in nm
1	684.01	16	487.44
2	519.32	17	396.49
3	965.77	18	607.49
4	303.55	19	331.45
5	181.67	20	1170.00
6	422.49	21	404.15
7	311.84	22	412.69
8	839.11	23	1800.00
9	536.48	24	616.32
10	222.65	25	328.10
11	2950.00	26	350.69
12	7950.00	27	104.70
13	1050.00	28	550.92
14	1240.00	29	1740.00
15	430.70		

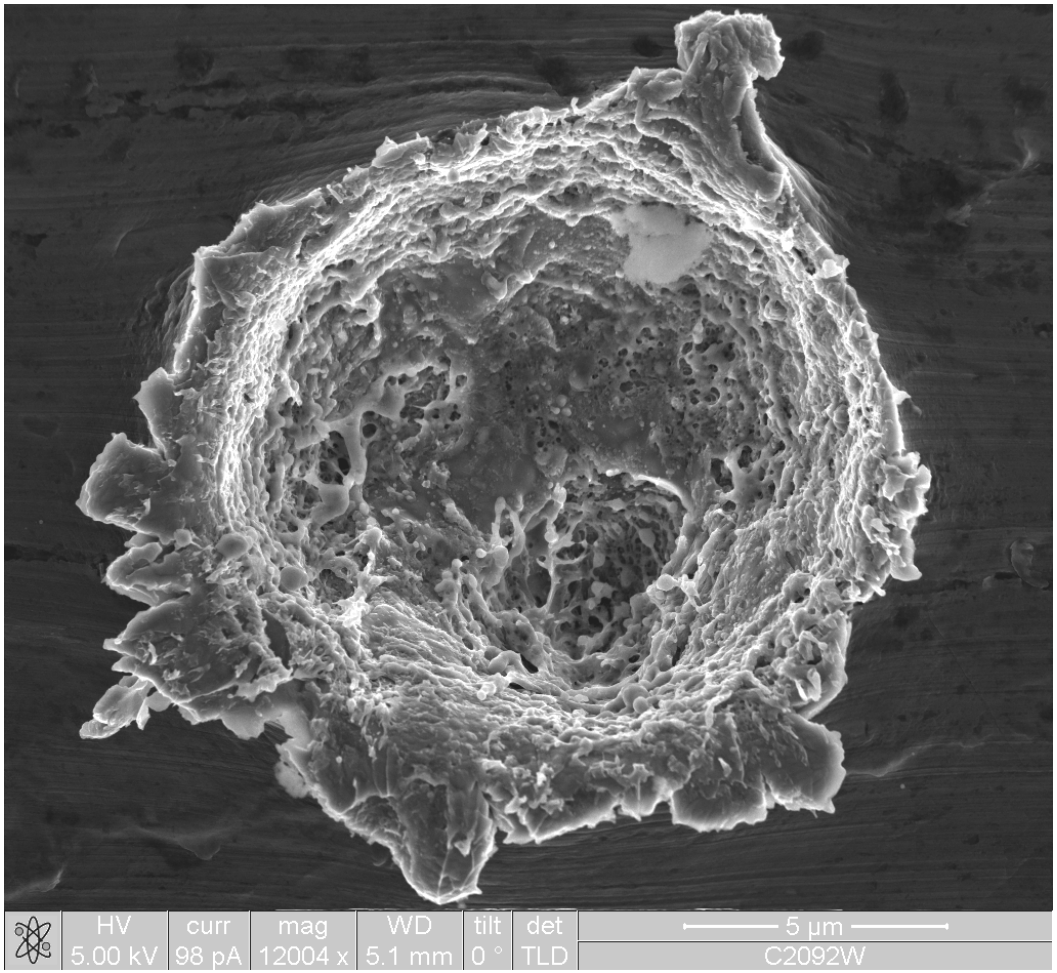
D_c and EDS data for craters 1-29

Foil No.	Crater No.	EDS Elemental Data	Foil No.	Crater No.	EDS Elemental Data
C2054W	1	Mg, Si, Fe, S	C2054W	13	Mg, Si, Fe, S
C2054W	3	Fe, Ni, S	C2054W	14	Mg, Si, Fe, minor S
C2054W	11	Mg, Si, Fe, S	C2054W	20	Mg, Si, Fe, S
C2054W	12	Mg, Si, Fe, S	C2054W	23	Mg, Si, Fe, S
			C2054W	29	Mg, Si, S, low Fe



Systematic low magnification scan identified one crater on the foil surface. System setup was 5 kV, 0 degrees tilt, 120x. Montage of 120x magnification scan of total foil surface. Position of the crater marked with a red circle.

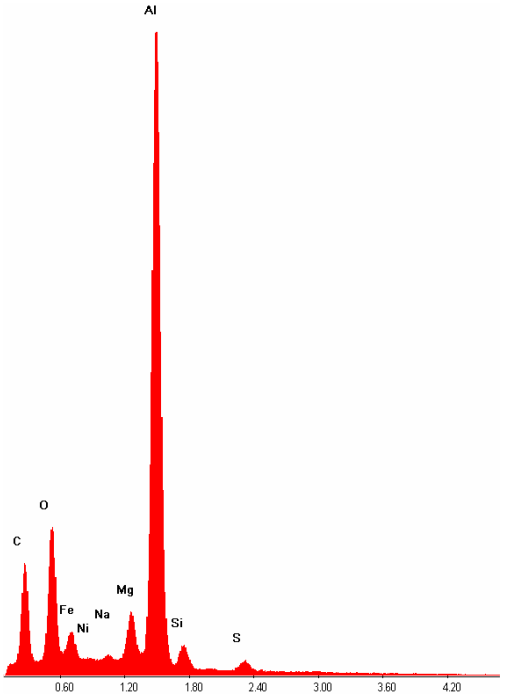
Foil: C2092W



5kV Secondary electron image of the impact crater. D_c = 13.93µm

Crater No.	Crater Diameter (D_c) in µm
1	13.93

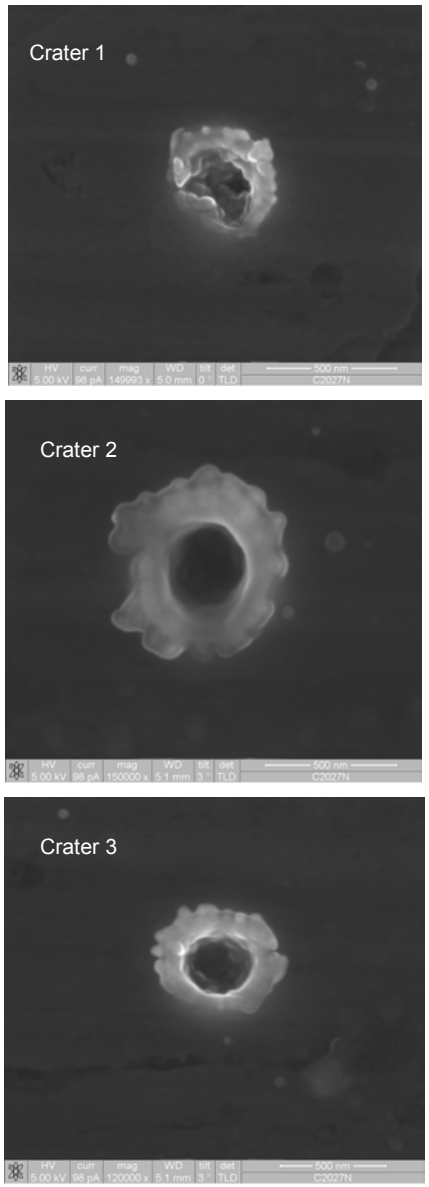
Foil No.	Crater No.	EDS Elemental Data
C2092W	1	Mg, Si, Fe, S, minor Na



5kV sum spectrum for the impact residue within the crater.

Systematic low magnification (120x) scan identified no craters (greater than 5µm) on the foil surface. A limited random search at 2500x magnification identified 3 sub-micrometer craters.

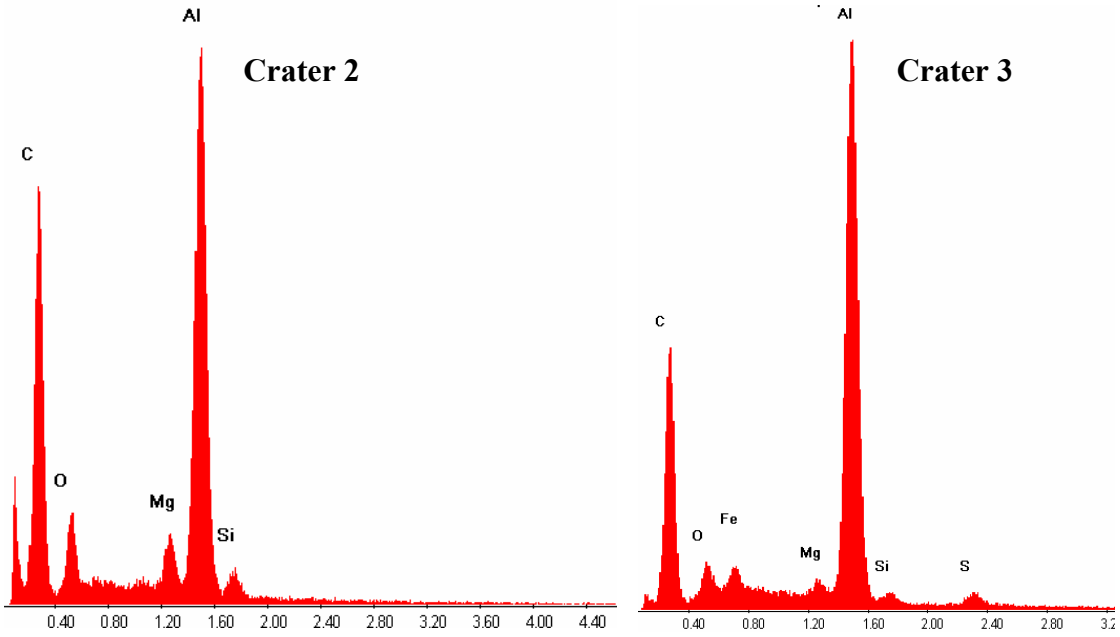
Foil: C2027N



Secondary electron images of the craters identified on surface of C2027N.

Crater No.	Crater Diameter (D_c) in nm
1	263.68
2	440.74
3	450.84

Foil No.	Crater No.	EDS Elemental Data
C2027N	2	Mg, Si, high C
C2027N	3	Mg, Si, Fe, S

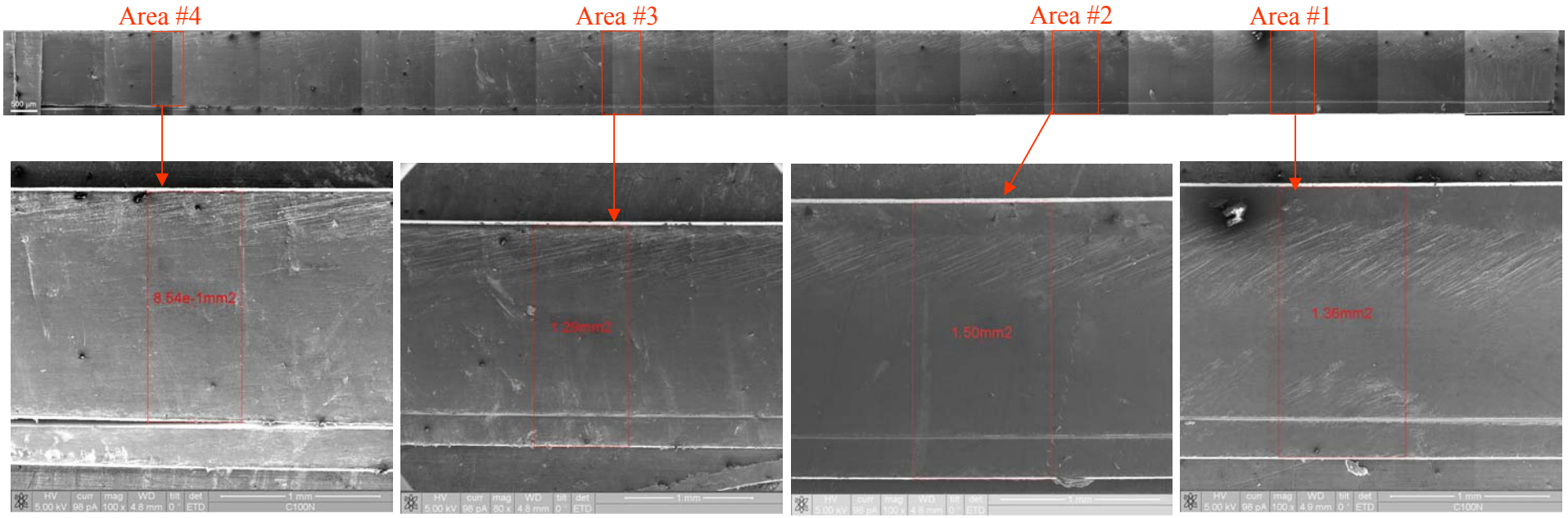


5kV sum spectra for the impact residues within the craters 2 & 3.

Appendix 2: Foil C2100N

Summary data for the systematic high magnification scans of C2100N.

Systematic low magnification (120x) scan identified no craters on the foil surface. The montage of 120x magnification scan of total foil surface identifies areas selected for 2500x high magnification scans in the red boxes.



Area #4 = 0.854 mm^2

Area #3 = 1.29 mm^2

Area #2 = 1.50 mm^2

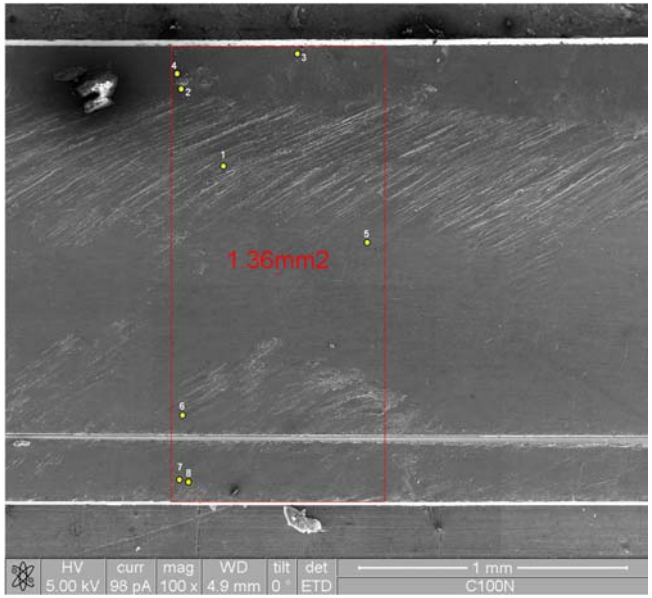
Area #1 = 1.36 mm^2

Foil: C2100N

Total area scanned at 2500x = 5.004 mm^2
Total number of craters = 35

Foil: C2100N

Area #1



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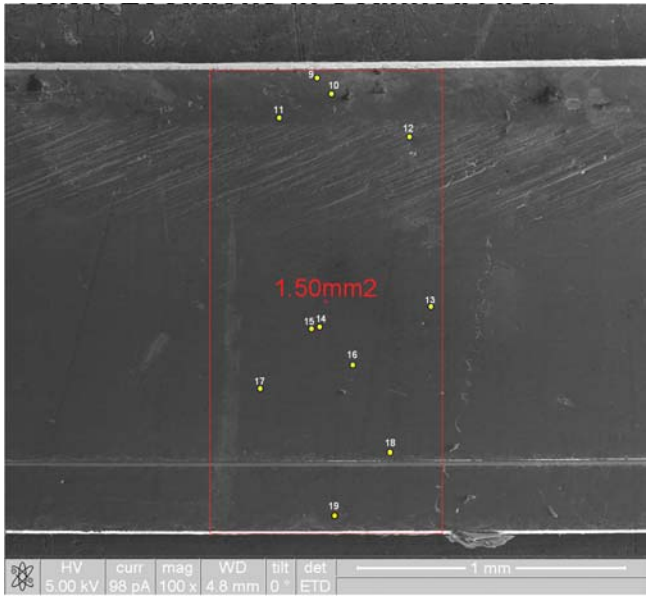
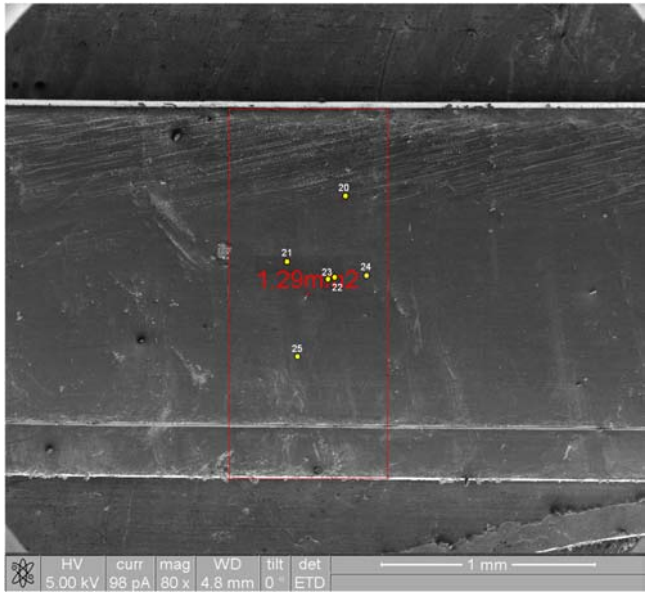


Area #3



Area #4

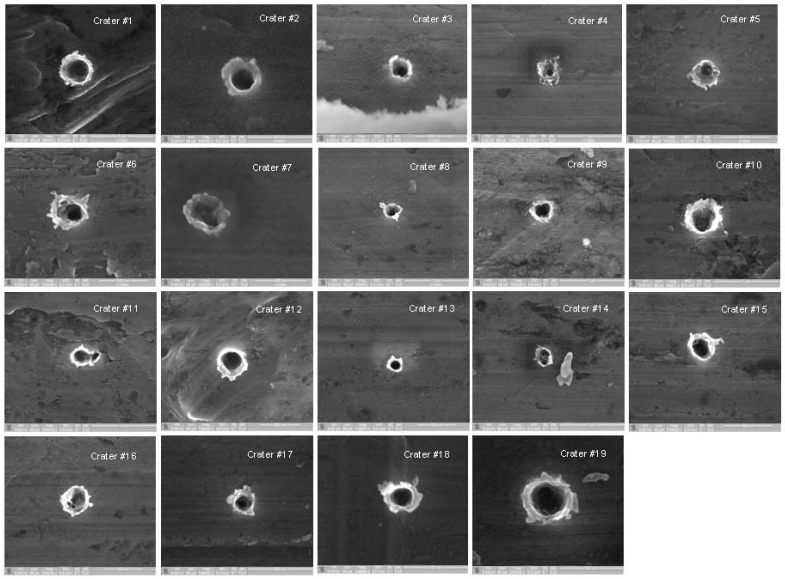
Secondary electron images at low magnification of the area scans identifying the position of the located craters

Foil: C2100N

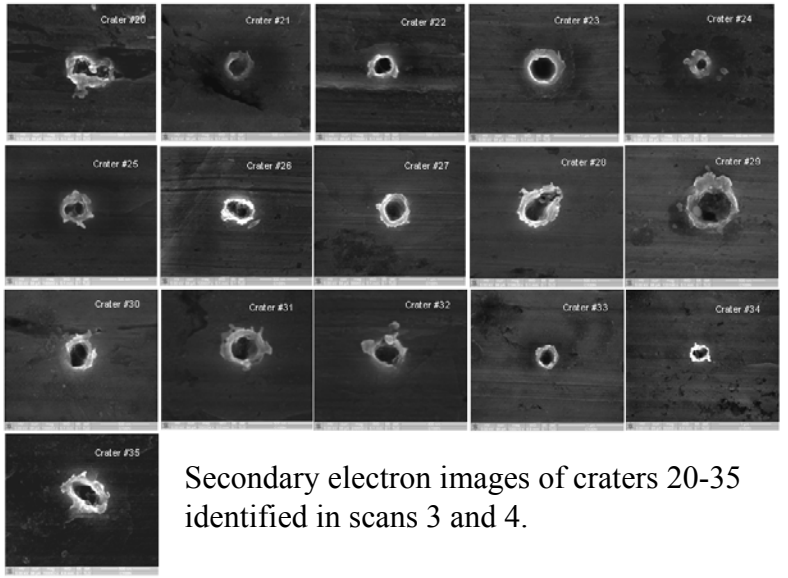
Scan No.	1
Crater	D_{co}
1	910.20 nm
2	181.88 nm
3	250.00 nm
4	301.50 nm
5	199.97 nm
6	284.30 nm
7	339.00 nm
8	254.55 nm
Scan No.	**2**
9	309.31 nm
10	486.57 nm
11	288.24 nm
12	354.50 nm
13	145.52 nm
14	264.34 nm
15	258.62 nm
16	321.20 nm
17	152.36 nm
18	184.01 nm
19	303.46 nm

Scan No.	3
Crater	D_{co}
20	448.38 nm
21	281.33 nm
22	212.44 nm
23	475.92 nm
24	172.85 nm
25	257.91 nm
Scan No.	**4**
26	357.32 nm
27	438.45 nm
28	428.97 nm
29	595.73 nm
30	294.64 nm
31	258.69 nm
32	183.32 nm
33	319.89 nm
34	308.82 nm
35	341.30 nm

Crater Diameters (D_C) for scan areas 1-4.



Secondary electron images of craters 1-19 identified in scans 1 and 2.



Secondary electron images of craters 20-35 identified in scans 3 and 4.

Foil No.	Crater No.	EDS Elemental Data
C2100N	1	Mg, Si, Fe, S
C2100N	23	Mg, Si

EDS Elemental Data for craters 1 and 23