CAL POLY   College of Engineering
           Mechanical Engineering Department

Summary Report

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SEM/EDS Operator: Wesley Powell

Procedure:

A steel sample with Mn and Si alloying elements was coated in Maxon CRS and allowed to sit in an open environment at room temperature for more than seven days (two weeks), allowing it to fully cure. Then the sample was prepared for SEM metallography according to ASTM E3-11 without a mount. An SEM image was produced at 100X magnification in a vacuum chamber with an accelerating voltage of 20.0 kV* and spot size of 4.0. The Maxon CRS coating was not washed off the sample so the EDS could be used to determine what elements seem to be present in the coating. A clean-up software protocol called "tidy-up" was performed on the EDS between this test and the previous, which should provide more accurate results and may explain why some of the results differ between this test and the last test performed.

Data:

Measurements were taken along even increments in one straight line normal to the surface (on the right side of the image) of the sample and starting at the Maxon CRS coating. Table I orders the results by the depth of point of interest from the surface of the sample.

Table I. Summary Table of Results

Depth (µm)	Carbon content (wt%)	Point of interest label
50	6.39	7 Day Spectrum 2(2)
120	20.69	7 Day Spectrum 2(3)
190	6.52	7 Day Spectrum 2(4)
260	11.63	7 Day Spectrum 2(5)
330	8.09	7 Day Spectrum 2(6)
400	3.38	7 Day Spectrum 2(7)
470	6.55	7 Day Spectrum 2(8)
540	9.13	7 Day Spectrum 2(9)
610	8.06	7 Day Spectrum 2(10)

Analysis/Discussion:

The results from this preliminary study show that phosphorus was no longer detected in any of the spectrums available. More investigation will need to be done to determine why it showed up in the previous test and not in this one. It is possible this occurred because of the clean-up protocol. More tests will be performed to see if phosphorus can be seen in other tests.

Carbon content in these tests, while more accurate than the previous test, still vary greatly between tests. This can make it difficult to perform a quantitative analysis because the difference in carbon percentage can be less than the level of error from the EDS. Dr. Harding suggests that with this level of error in EDS analysis, it may only be possible to do a quantitative analysis of carbon using auger electron spectroscopy (which would require equipment we don't have on campus, but UCSB may have).

It may be useful for these tests to know the elemental composition of the Maxon CRS so that I can pick a heavier element than carbon or oxygen to focus on. I could also try to do mapping instead of a line spectrum, which would take many measurements in an X-Y grid so that it could help reduce the significance of any individual error points (but lengthen the amount of time to perform EDS). I also could use a backscatter detector to try to do a qualitative analysis of the SEM image. With backscattered electrons the image may show discoloration where the Maxon CRS is present. Either way, I think a significant portion of this project will be including testing different methods of analysis and seeing which method provides the most reliable results. If phosphorus cannot be reliably used as an indicator of the depth and the amount of random error in the EDS detector makes it difficult to determine changes in carbon content, then this may take some time.

*The previous report mistakenly listed all the accelerating voltages in the units of keV instead of kV.
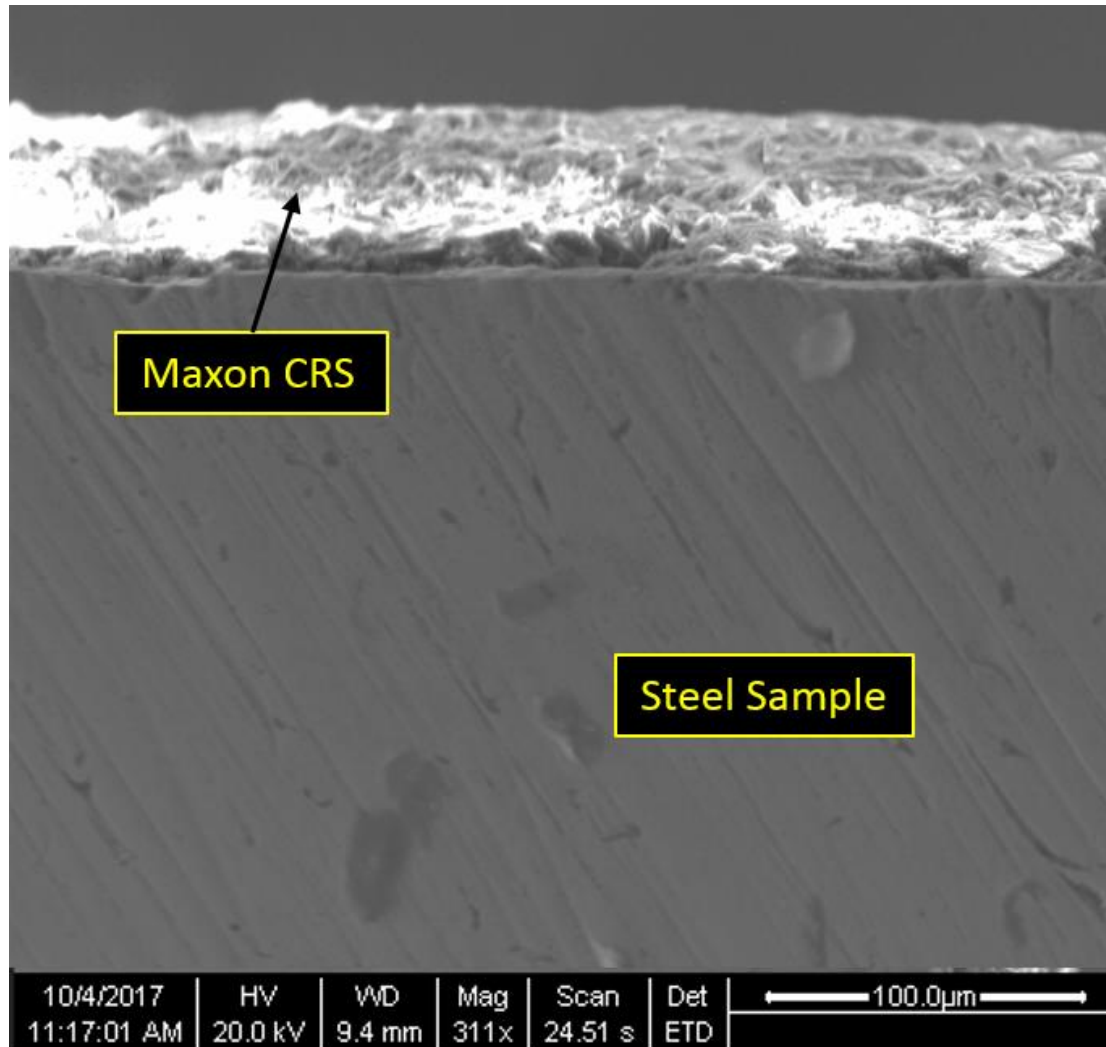
Figure 1. Image of Maxon CRS on steel sample after two weeks.

Spectrum processing :
Peak possibly omitted : 2.325 keV

Processing option : All elements analyzed (Normalised)
Number of iterations = 4

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
O   SiO2   1-Jun-1999 12:00 AM
Al   Al2O3   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	22.22	29.27
O K	62.75	62.05
Al K	14.59	8.55
Fe K	0.44	0.12
Totals	100.00	

600µm   Electron Image 1

This point of interest is located inside the coating (the bright white portion on the right side of the image). This was done to determine the most common elements inside the Maxon CRS. Fe is likely only present because of the proximity to the metal.

Note that there are some significant differences in this spectrum than the equivalent point of interest in the previous 5 day sample. This will be a significant point of this report, because it was expected that the same elements would be present in both readings. For comparison, the reading from the 5 day report detected all of the following: C, O, Na, Al, P, S, Cl, K, Ca, and Ba. This spectrum only suggest the presence of C, O, and Al in the coating.

Image Magnification: 100X
Spot Size: 4.0
Accelerating Voltage: 20kV (note the previous report accidently listed the accelerating voltage as 20 keV. This was a typo).
(These values remain constant for the entirety of this report)
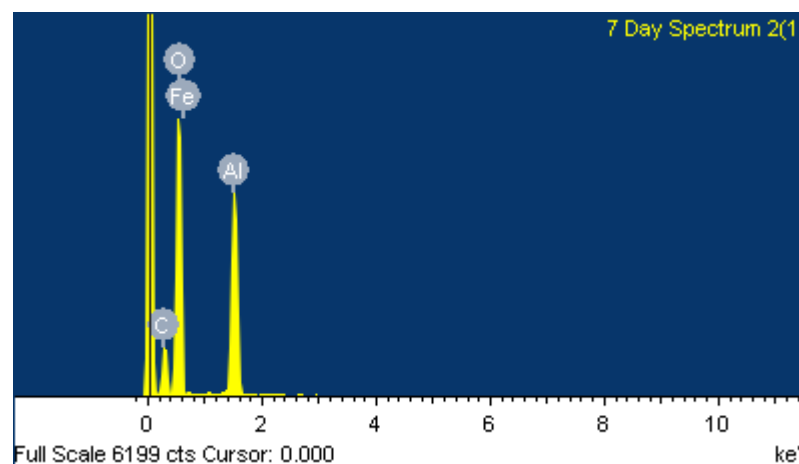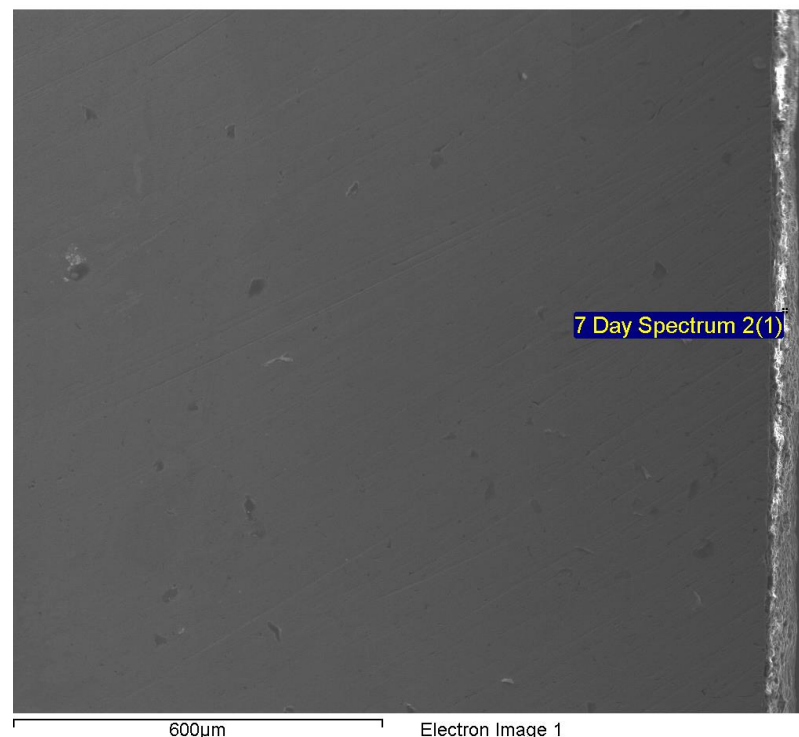
Full Scale 6199 cts Cursor: 0.000

INCA

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
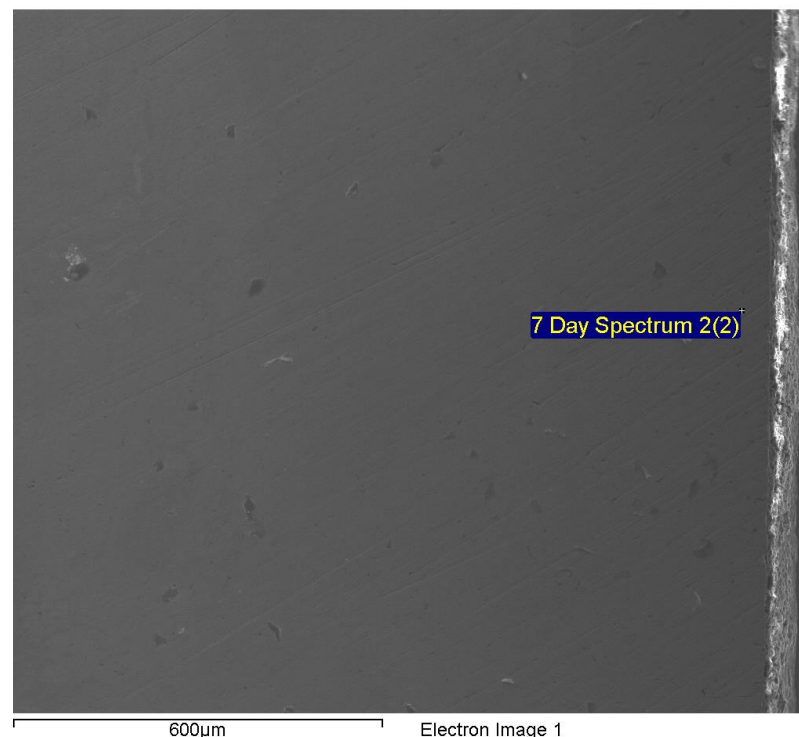Si   SiO2   1-Jun-1999 12:00 AM
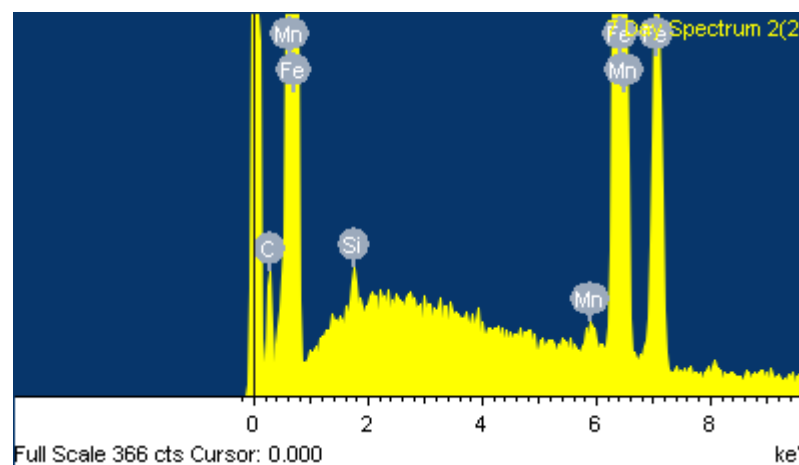Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	6.39	24.04
Si K	0.31	0.50
Mn K	0.57	0.46
Fe K	92.73	74.99
Totals	100.00	

600µm   Electron Image 1

Depth: ~50 µm

In this report one of the previous issues has seemed to be solved without adjusting to a smaller accelerating voltage (i.e. the carbon spectrum is distinguished enough from the background radiation that it is possible to see a unique peak. This is why there are no negative carbon values in this report. This might be attributed to a clean-up program that was done to increase the accuracy of the EDS.

One new issue though is that there is no longer a peak for phosphorus at any point in this test. (I do not currently have an explanation for this that involves setting with the EDS, so I wonder if there is something more chemically involved going on.)

5

Full Scale 366 cts Cursor: 0.000   keV

Spectrum processing :
Peak possibly omitted : 4.430 keV

Processing option : All elements analyzed (Normalised)
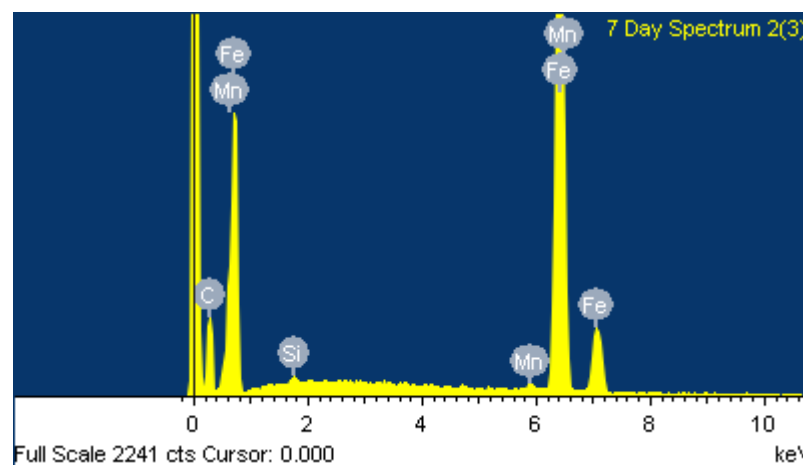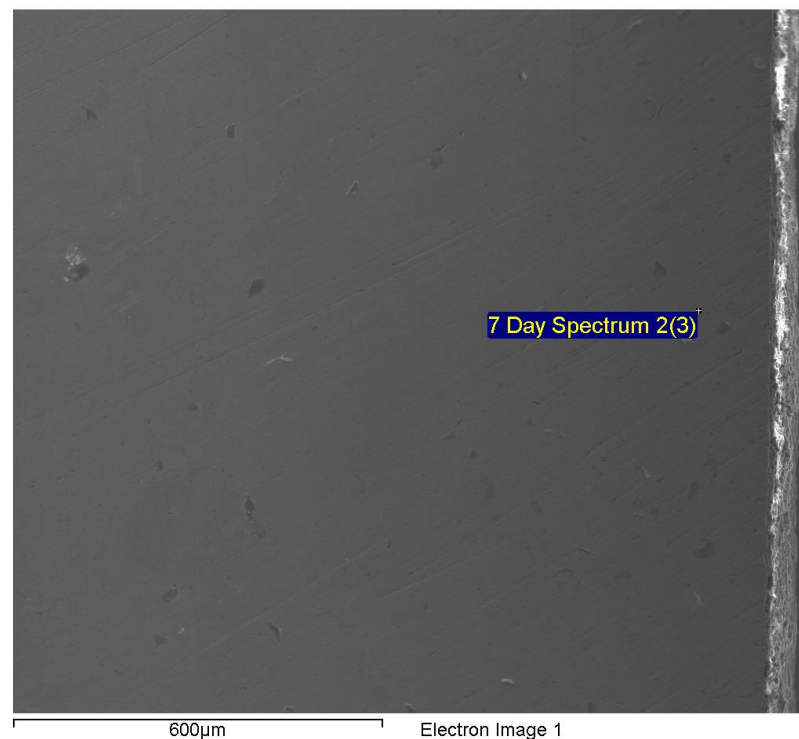Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	20.69	54.73
Si K	0.27	0.31
Mn K	0.90	0.52
Fe K	78.14	44.44
Totals	100.00	

7 Day Spectrum 2(3)

600µm   Electron Image 1

Depth: ~120 µm

6

Spectrum processing :
No peaks omitted

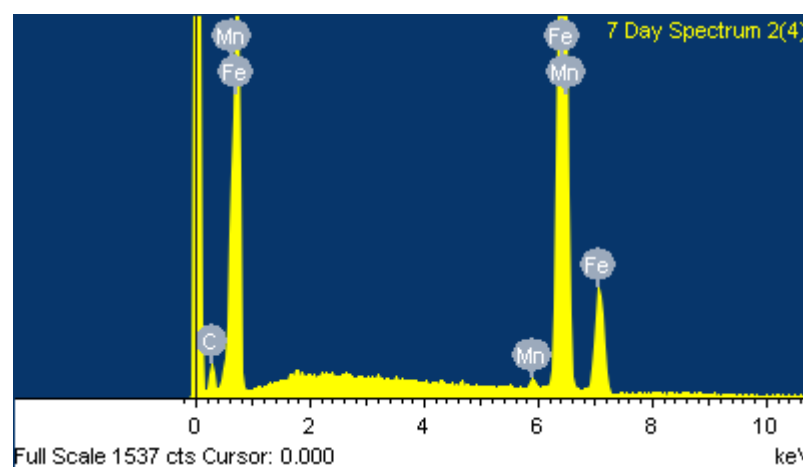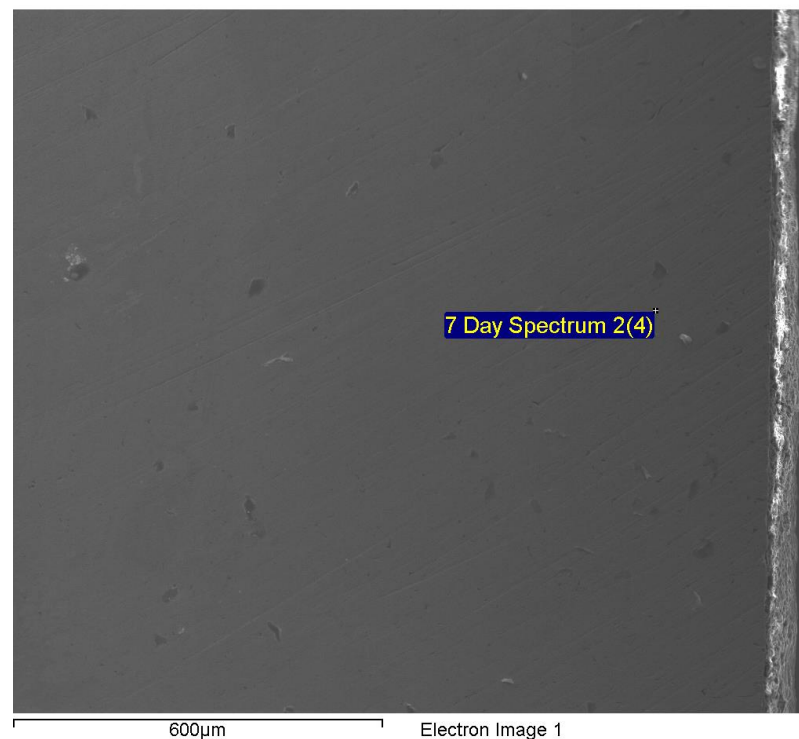Processing option : All elements analyzed (Normalised)
Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	6.52	24.49
Mn K	0.84	0.69
Fe K	92.64	74.82
Totals	100.00	

600μm   Electron Image 1

Depth: ~190 μm

7

7 Day Spectrum 2(4)

Full Scale 1537 cts Cursor: 0.000   keV

INCA

Spectrum processing :
Peak possibly omitted : 1.764 keV

Processing option : All elements analyzed (Normalised)
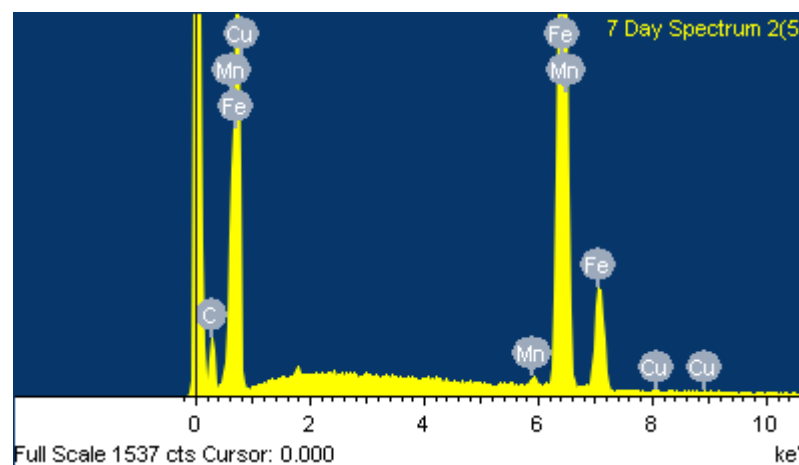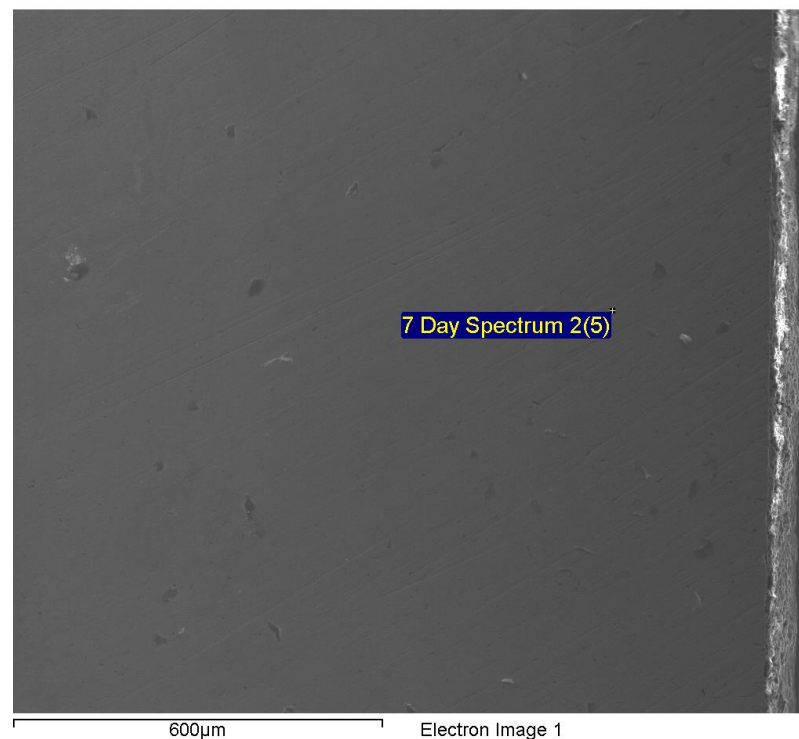Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM
Cu   Cu   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	11.63	37.97
Mn K	0.81	0.58
Fe K	86.99	61.10
Cu K	0.57	0.35
Totals	100.00	

7 Day Spectrum 2(5)

600µm   Electron Image 1

Depth: ~260 µm

8

Full Scale 1537 cts Cursor: 0.000

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 2

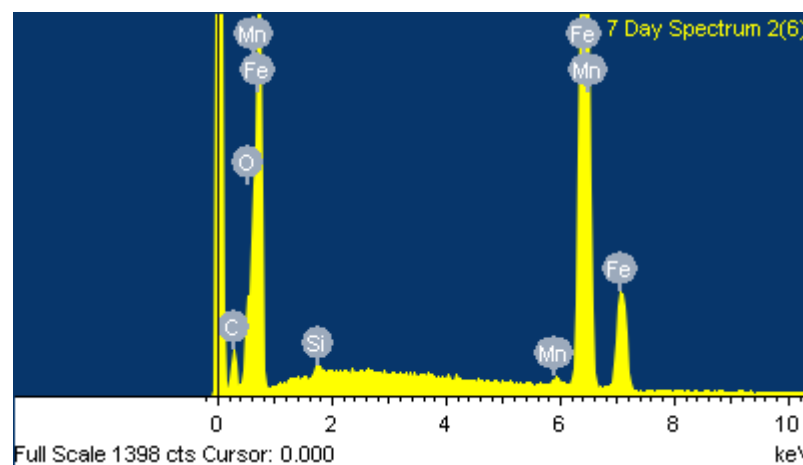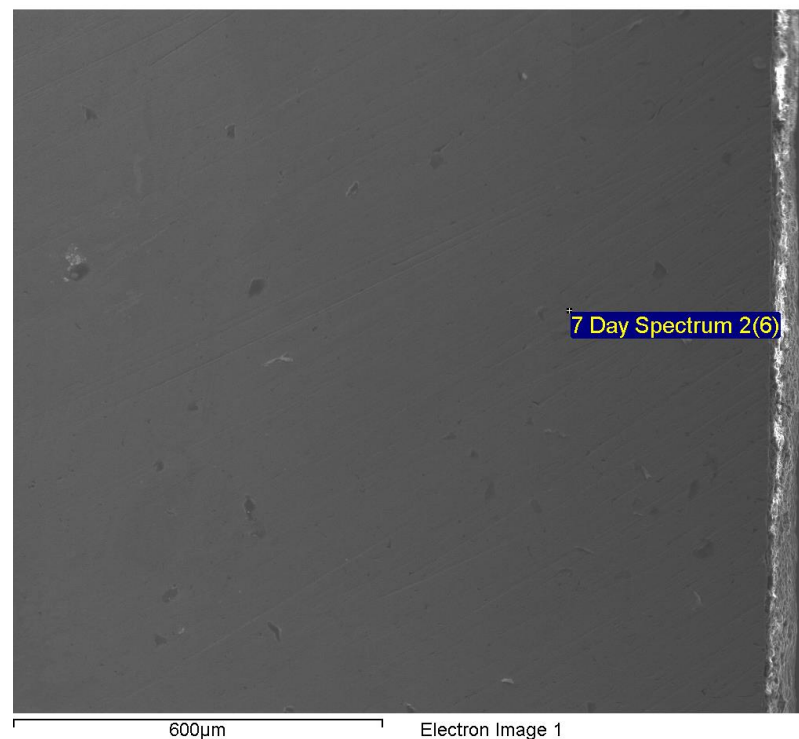Standard :
C   CaCO3   1-Jun-1999 12:00 AM
O   SiO2   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	8.09	26.64
O K	4.60	11.36
Si K	0.26	0.37
Mn K	0.79	0.57
Fe K	86.26	61.06
Totals	100.00	

7 Day Spectrum 2(6)

600µm   Electron Image 1

Depth: ~330 µm

9

7 Day Spectrum 2(6)

Full Scale 1398 cts Cursor: 0.000   keV

inca

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
Number of iterations = 1

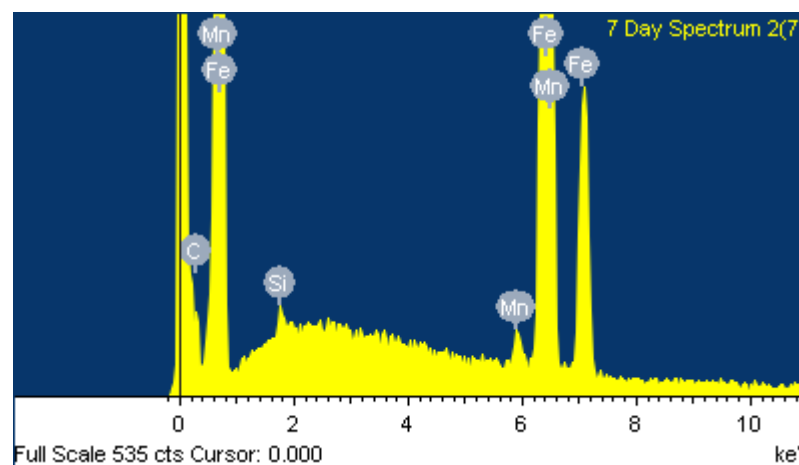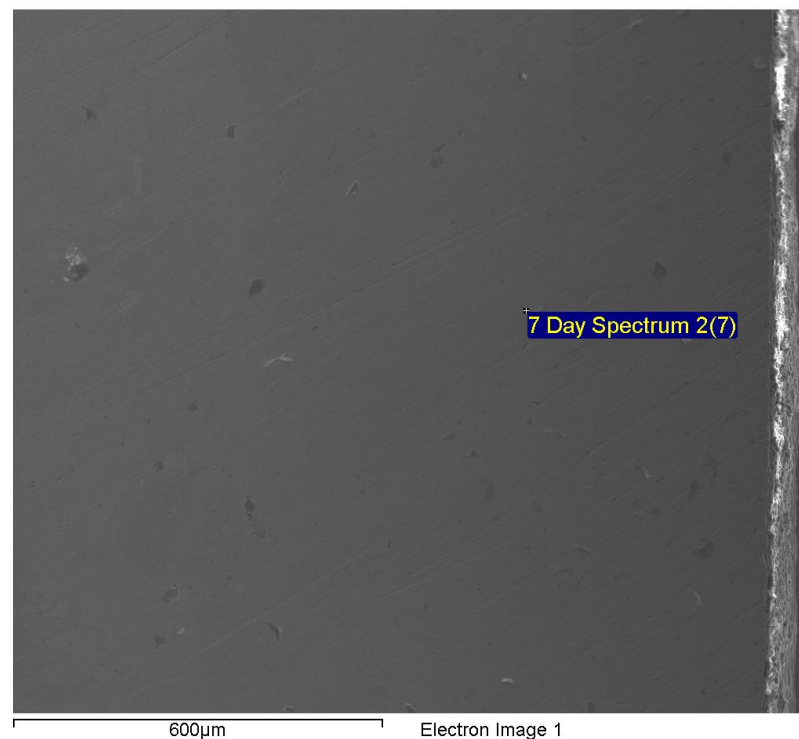Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	3.38	13.95
Si K	0.25	0.45
Mn K	1.03	0.93
Fe K	95.34	84.67
Totals	100.00	

600µm   Electron Image 1

Depth: ~400 µm

10

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
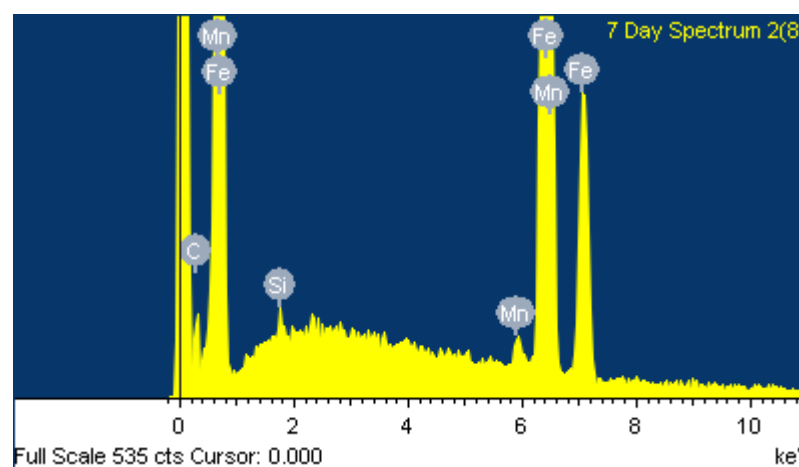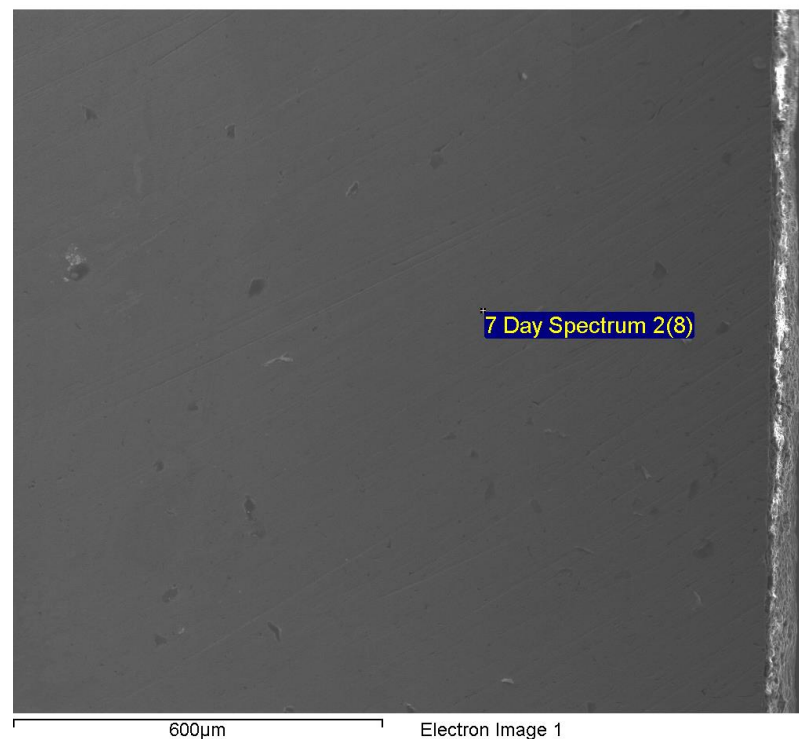Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	6.55	24.52
Si K	0.28	0.45
Mn K	0.95	0.78
Fe K	92.22	74.25
Totals	100.00	

7 Day Spectrum 2(8)

600µm   Electron Image 1

7 Day Spectrum 2(8)

Full Scale 535 cts Cursor: 0.000   keV

Depth: ~470 µm

INCA

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
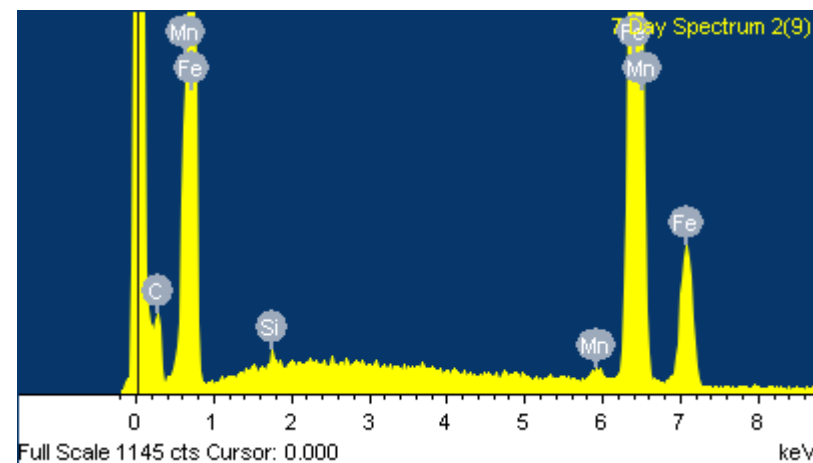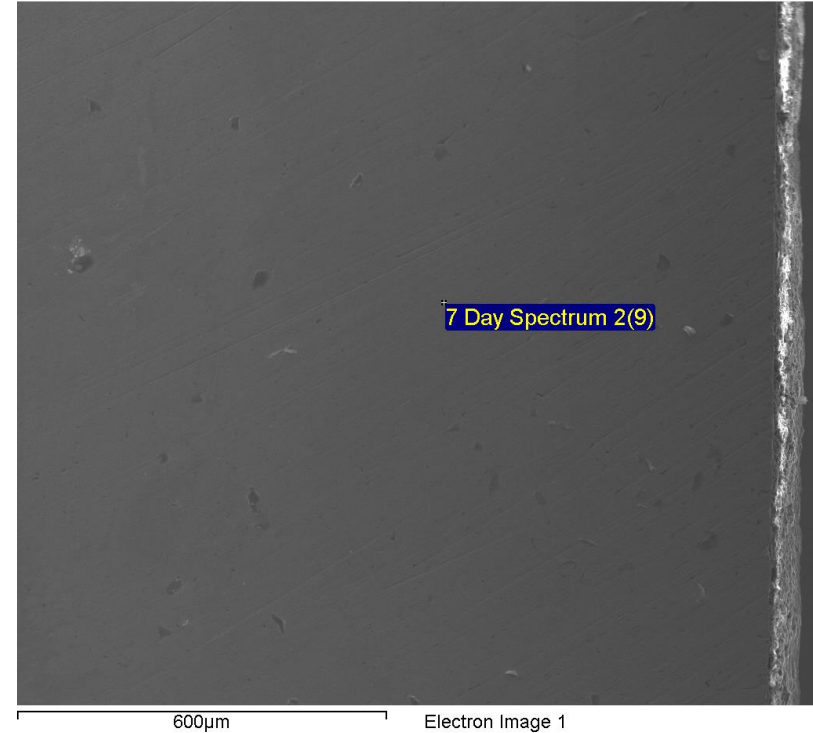Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	9.13	31.77
Si K	0.32	0.48
Mn K	0.76	0.58
Fe K	89.79	67.17
Totals	100.00	

600µm   Electron Image 1

Depth: ~540 µm

As there were more tests performed the amount of background radiation appears to have been growing, to the point it started to become more difficult to resolve the carbon peak.

12

Spectrum processing :
No peaks omitted

Processing option : All elements analyzed (Normalised)
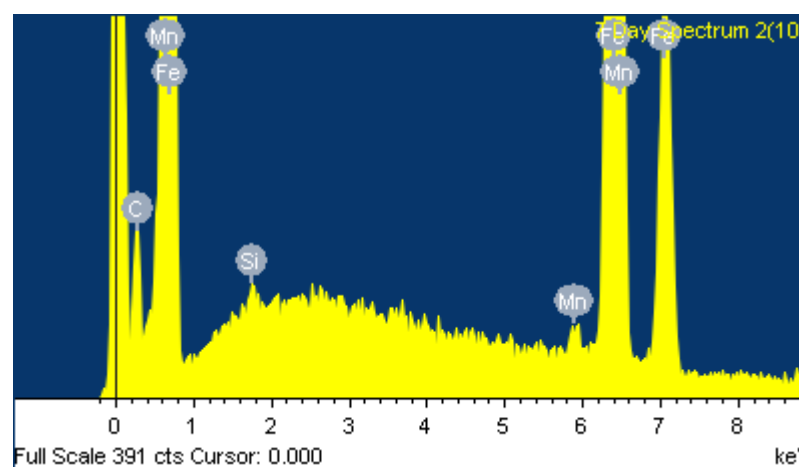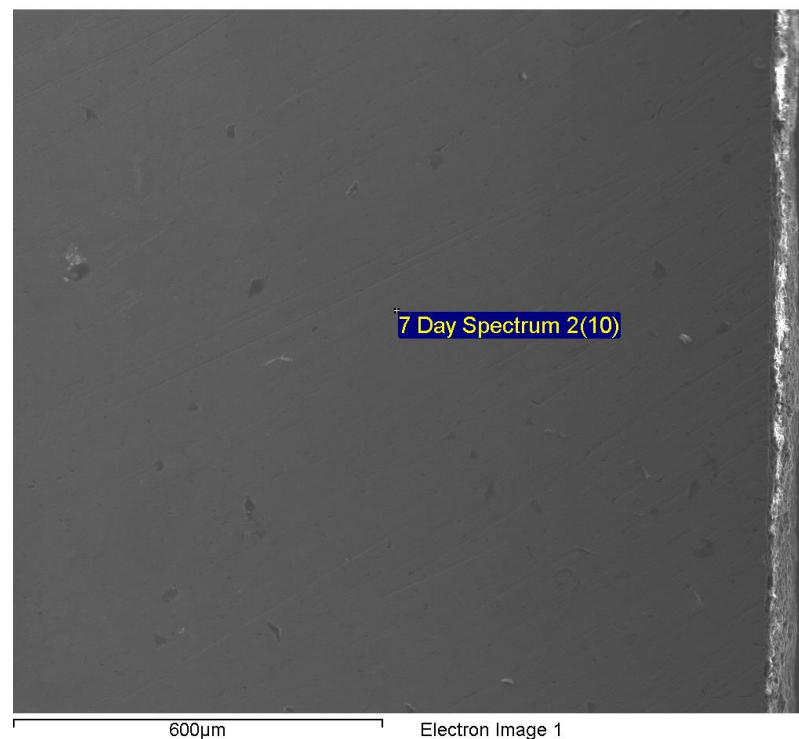Number of iterations = 2

Standard :
C   CaCO3   1-Jun-1999 12:00 AM
Si   SiO2   1-Jun-1999 12:00 AM
Mn   Mn   1-Jun-1999 12:00 AM
Fe   Fe   1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	8.06	28.91
Si K	0.25	0.38
Mn K	0.78	0.61
Fe K	90.91	70.09
Totals	100.00	

7 Day Spectrum 2(10)

600µm   Electron Image 1

Depth: ~610 µm

13

Full Scale 391 cts Cursor: 0.000   keV

INCA