

## Scanning Electron Microscope & Energy Dispersive X-Ray Spectrometer Analysis of Samples Cleaned with CleanWirx

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Spectroscopy Services Provided by Anastas Technical Services

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## **Overview:**

Two 2" square steel samples, were submitted to Anastas Technical Services for study by SEM / EDS. The purpose of this study is to satisfy the following objectives and evaluate the effectiveness of CLEANWIRX as a contaminant removal, surface preparation method by comparing / contrasting CLEANWIRX treated and untreated areas.

## **Objectives:**

1. Use CLEANWIRX System to establish a baseline "contaminant free" surface for contaminant deposition.
2. Intentionally and methodically deposit known elements and corrosive compounds in defined areas.
3. Verify the location and identity of elements (i.e. Sulfur, Chlorine) that are integral to known corrosive compounds (i.e. Iron Sulfides and Iron Chlorides).
4. Verify the absence of the above elements / compounds on areas treated with BMT System. Then compare and contrast (visually and quantitatively) the findings from #3 with areas on the same sample that have been treated with the CLEANWIRX System.

## **Background:**

SEM (Scanning Electron Microscope) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. To capture an image, a small coupon, in/on this particular instrument - no larger than 3"x3", is rigidly mounted on a specimen holder in a small chamber and then locked inside. The chamber is then pressurized and bombarded with electrons.

**SEM's can specifically identify and observe elements (such as Iron, Sulfur, Chlorine, etc...) but not compounds such as "Ferric Chlorides" or "Ferric Sulfides". Additionally, elements must first be located (i.e. "found" or "discovered") before identification can occur. This means that the potential for "false negatives" is very high in relation to corrosion control and contaminant (corrosive compound) detection in surface preparation methods.**

In other words, in order to positively identify the presence of an element, you first have to find it. In order to evaluate the efficacy of a method of cleaning, it must be compared against a control polluted surface.

## **Conclusion:**

All objectives were satisfied. The absence of contaminants, both visually and analytically, confirmed that BMT produced a reliably clean baseline surface to deposit and compare / contrast with contaminated areas. Likewise, CLEANWIRX effectively removed contaminants.

## **Samples Preparation Method:**

### **Sample #1, Coupon #2 (See Image #1):**

1. Cleaned / decontaminated with CLEANWIRX,
2. Saturated entire field with Ferric Chloride solution (Ferric Chloride powder dissolved in De-Ionized water),
3. Heated until dry over a propane gas stove,
4. Then a granule of Sulfur was deposited in the center of the coupon to develop a sulfide / sulfur field approximately the size of a pencil eraser.
5. Heated over propane gas stove until Sulfur granule melted and then evaporated
6. Blasted to White Metal with Aluminum Oxide abrasive media by an ISO 9001 coating application company,
7. Used magic marker pen to make four “dots” around the field of sulfur.

### **Sample #2, Coupon #15 (See Image #5):**

1. Cleaned / decontaminated with CLEANWIRX,
2. Saturated whole surface face with Sulfur,
3. Heated over propane gas stove until Sulfur melted and uniformly covered the field before evaporating,
4. Saturated with Ferric Chloride solution (Ferric Chloride powder dissolved in De-Ionized water),
5. Heated until dry over a propane gas stove,
6. Blasted to White Metal with Aluminum Oxide abrasive media by an ISO 9001 coating application company,
7. Less than 5 hours later,  $\frac{1}{2}$  of the coupon was decontaminated with CLEANWIRX in a diagonal field so as to divide the coupon into two “diamonds”.

## Summary:

### **Sample 1:**

Sample 1 was uniformly decontaminated with CLEANWIRX and then saturated with Ferric Chloride solution across the entire face and then a grain of Sulfur was deposited right in the center. The first image is a picture of the actual sample. Although the picture was taken after it had been vacuum sealed in a plastic bag for shipment, the Sulfur field is still evident through the plastic (see the slight / light colored circle about the size of a pencil eraser between all four dots and slightly larger than the white box).

The objective was to observe an area known to be populated with Sulfur particles. Four dots were placed around the Sulfur field to indicate where to inspect / image.

Mr. Anastas looked in a handful of regions in this field and everywhere he inspected he found deposits of Sulfur and Chlorine. The first magnified image (image 2) was taken at 250x and two additional inspection fields (A & B) were each imaged at 1000x (see images 3 & 4).

No measurements were recorded of this sample for two reasons:

1. The purpose of this sample was to demonstrate that when looking in a known, and intentionally contaminated, area deposited elements should be readily observable. **This was confirmed.**
2. The recordings taken from the contaminated field of Sample 2 are representative both visually and quantitatively of the contaminated field on this sample.

### **Sample 2:**

Sample 2 was uniformly decontaminated with CLEANWIRX and then saturated with Sulfur followed by Ferric Chloride solution. The sample was then cleaned in a diagonal cross section forming two triangular shaped fields – one contaminated, one decontaminated.

Multiple regions were analyzed (See Image 5) but only three (Fields A, B & C) were recorded as they were representative of all the unrecorded inspections on both samples.

Field A: A known contaminated area, High Sulfur and Chlorine were identified immediately with no searching.

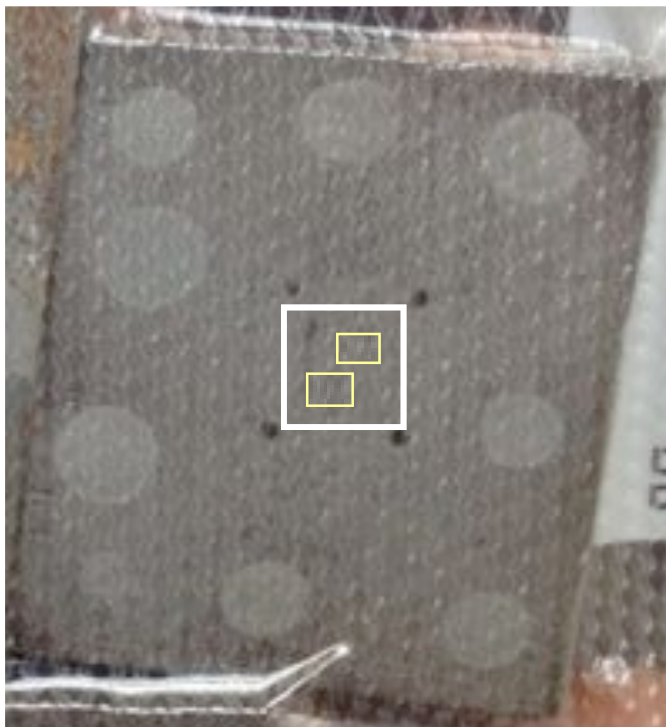
Fields B & C: Decontaminated Side, both fields were visibly different from Sample 1 and Sample 2, Field A. Except for embedded abrasive material (Aluminum Oxide), no Sulfur or Chlorine sites were identified.

**Sample #1**  
**Images**  
**Contaminated Area**

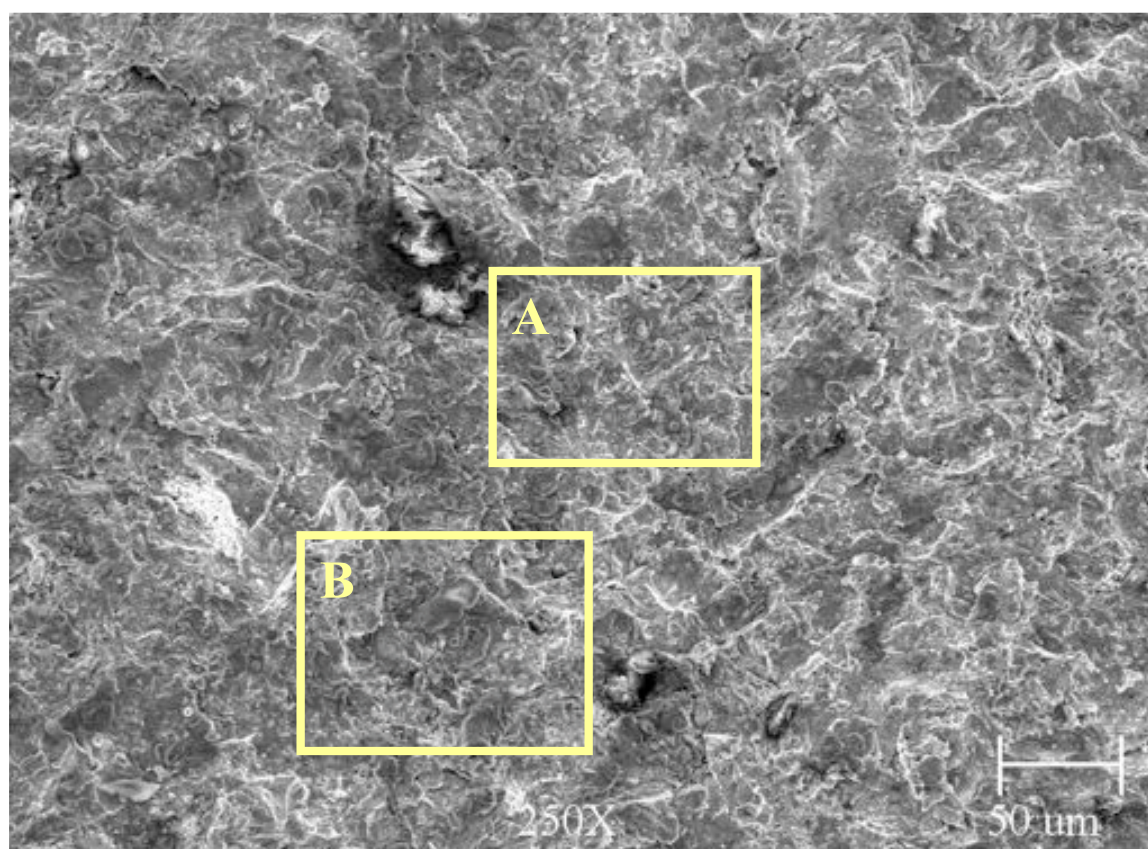
**(Sulfur Field Over Ferric Chloride Field)**

## Sample #1

#1

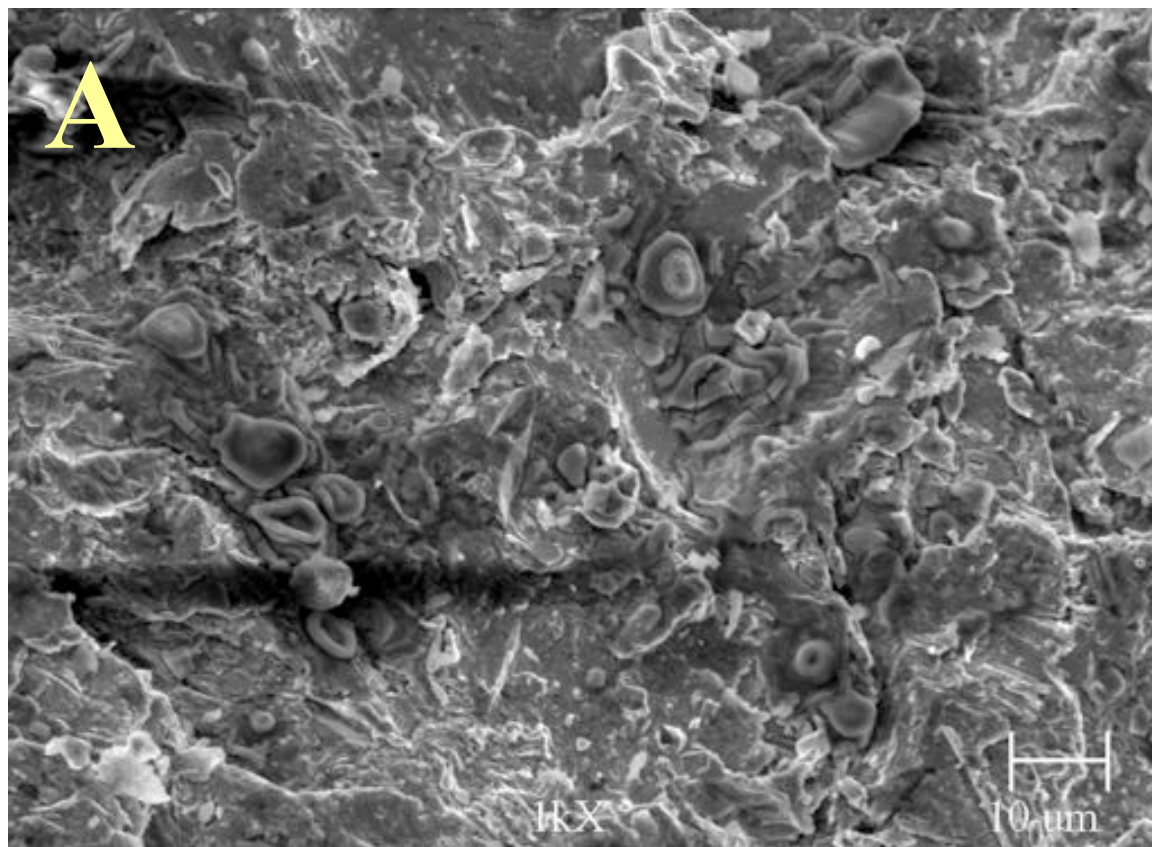


#2

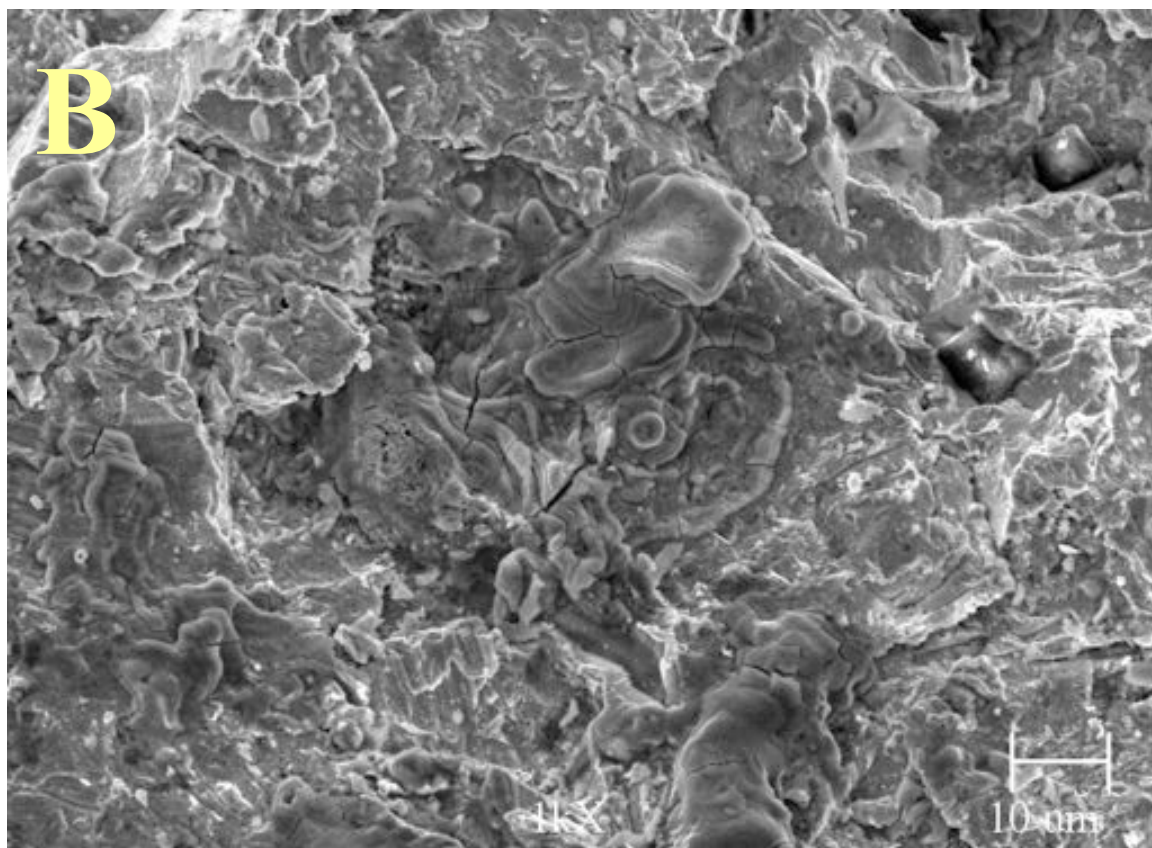




## Sample #1



#3



#4

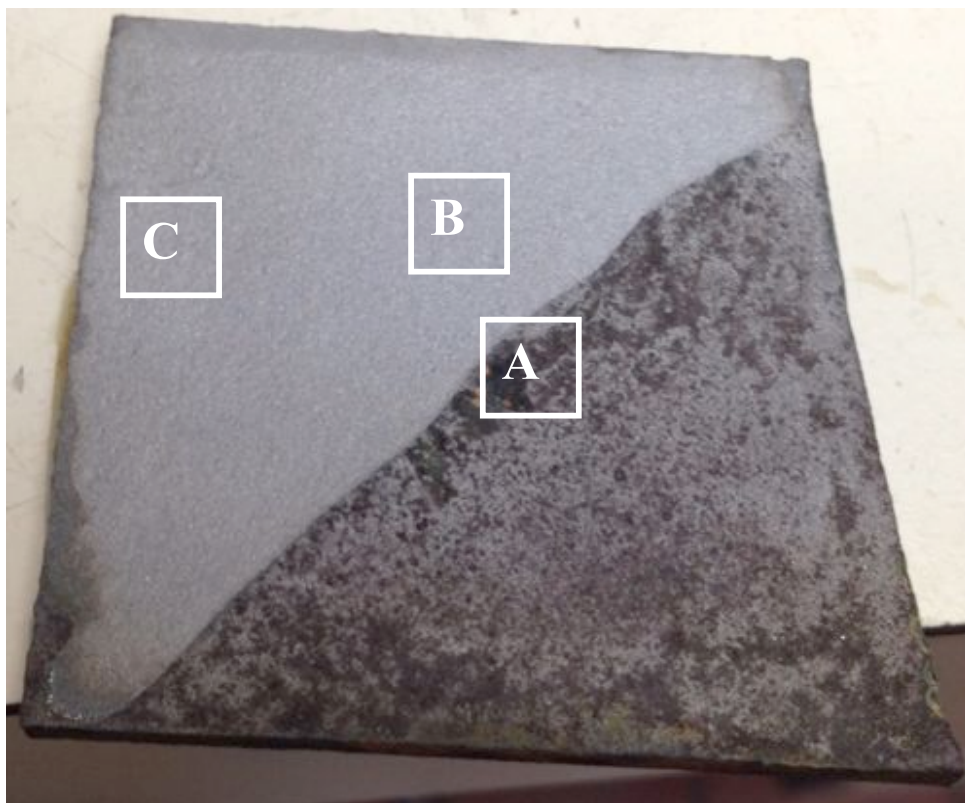


**Sample #2**

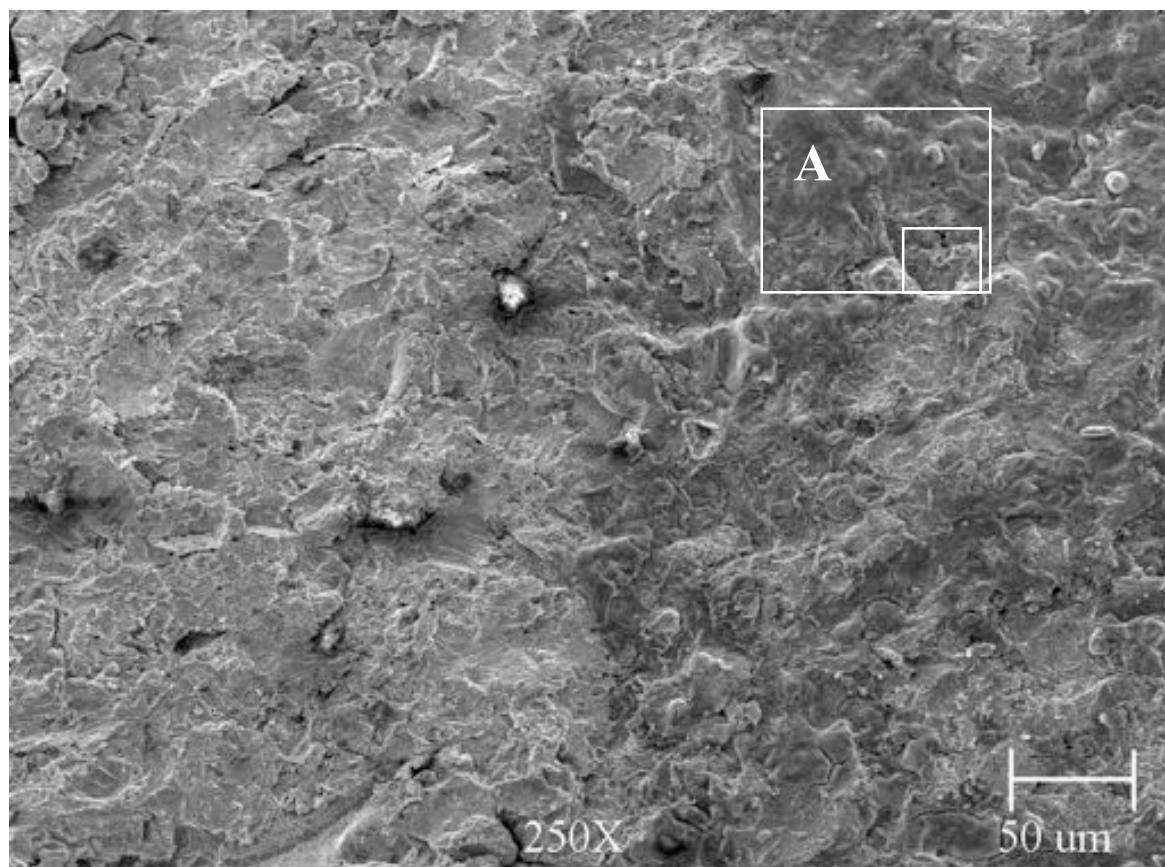
**EDS Reports &  
Images - Contaminated Side**

## Sample #2 – Contaminated Side, Field A:

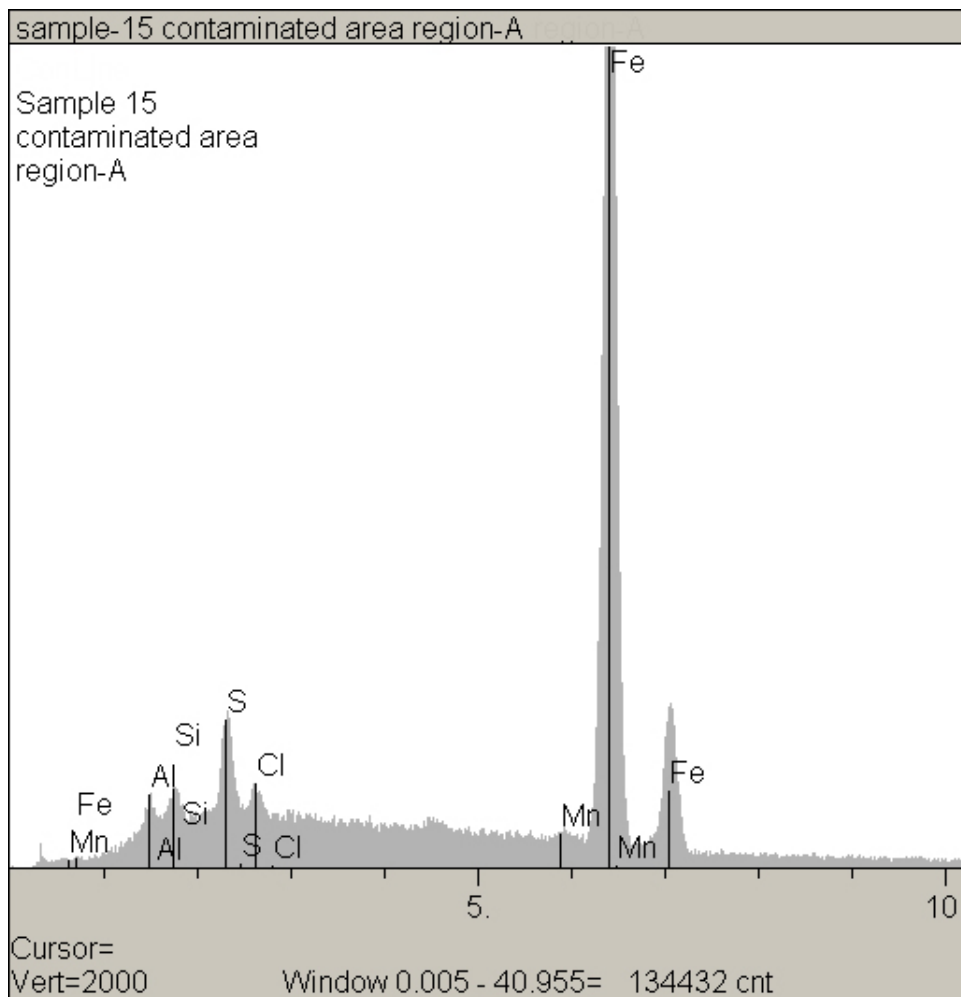
#5



#6



## Sample #2 – EDS Report, Field A, Region A:



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	14.57	0.986	1.620	wt.%	
Si	Ka	17.55	1.082	1.492	wt.%	
S	Ka	57.13	1.952	3.251	wt.%	
Cl	Ka	14.26	0.975	0.784	wt.%	
Mn	Ka	4.22	0.531	0.315	wt.%	
Fe	Ka	755.60	7.097	92.538	wt.%	
				100.000	wt.%	Total

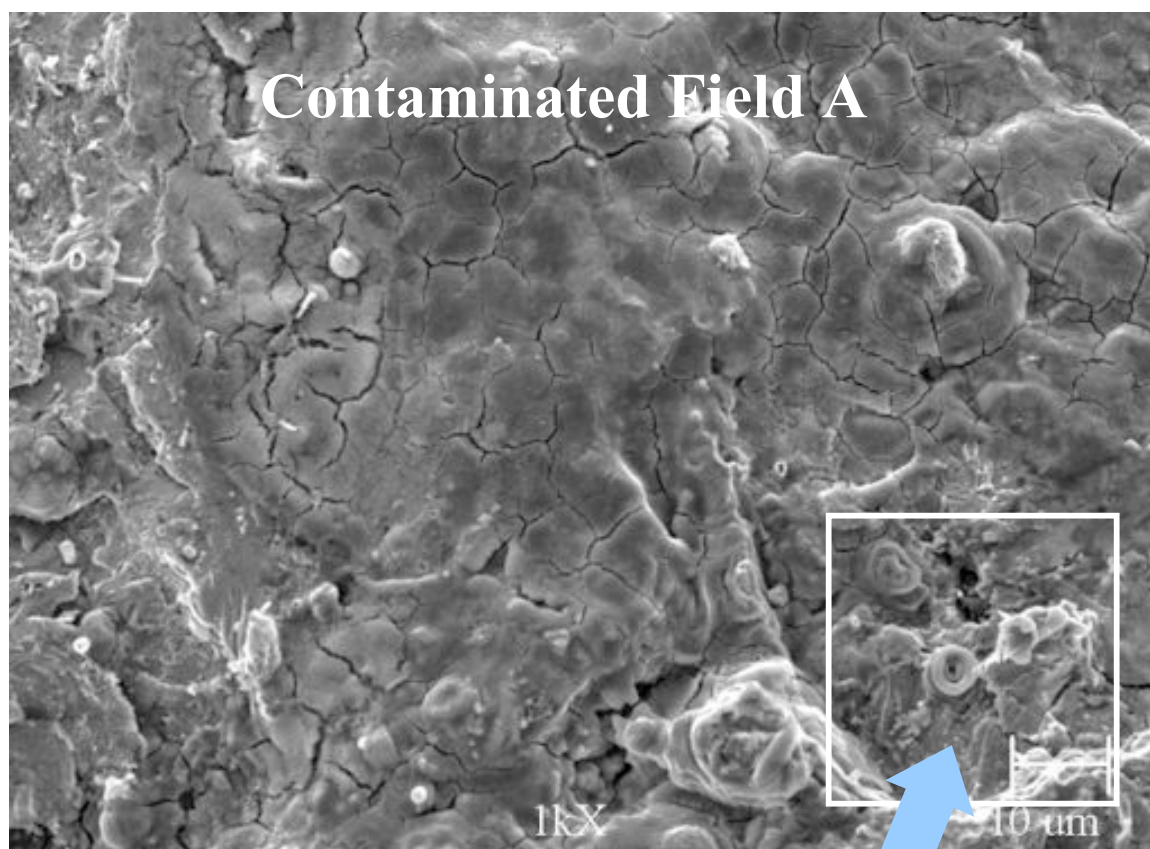
kV 15.0

Takeoff Angle 10.0°

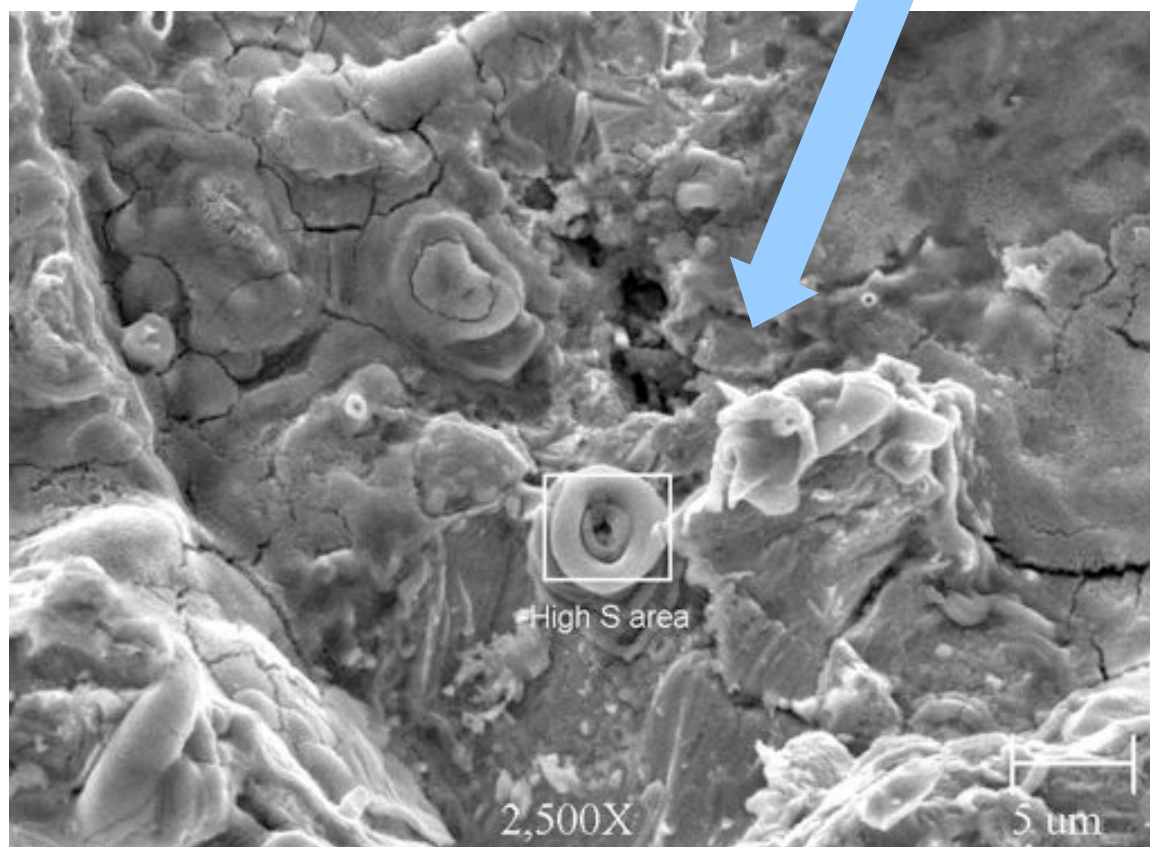
Elapsed Livetime 60.0

## Sample #2 – Contaminated Side, Field A, Cont'd:

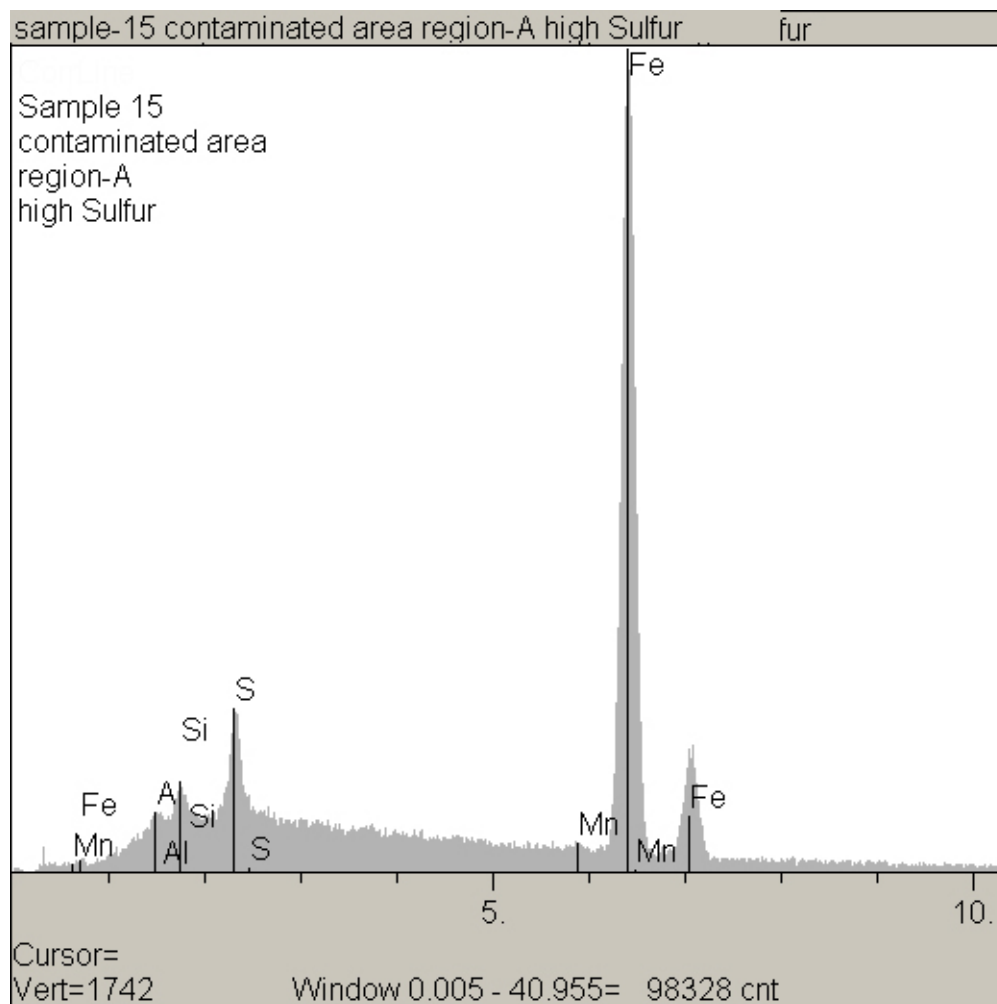
#7



#8



## Sample #2 – EDS Report, Field A, Region A:



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	5.09	0.583	0.871	wt.%	
Si	Ka	14.24	0.974	1.846	wt.%	
S	Ka	50.59	1.836	4.423	wt.%	
Mn	Ka	2.79	0.431	0.320	wt.%	
Fe	Ka	490.90	5.720	92.540	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

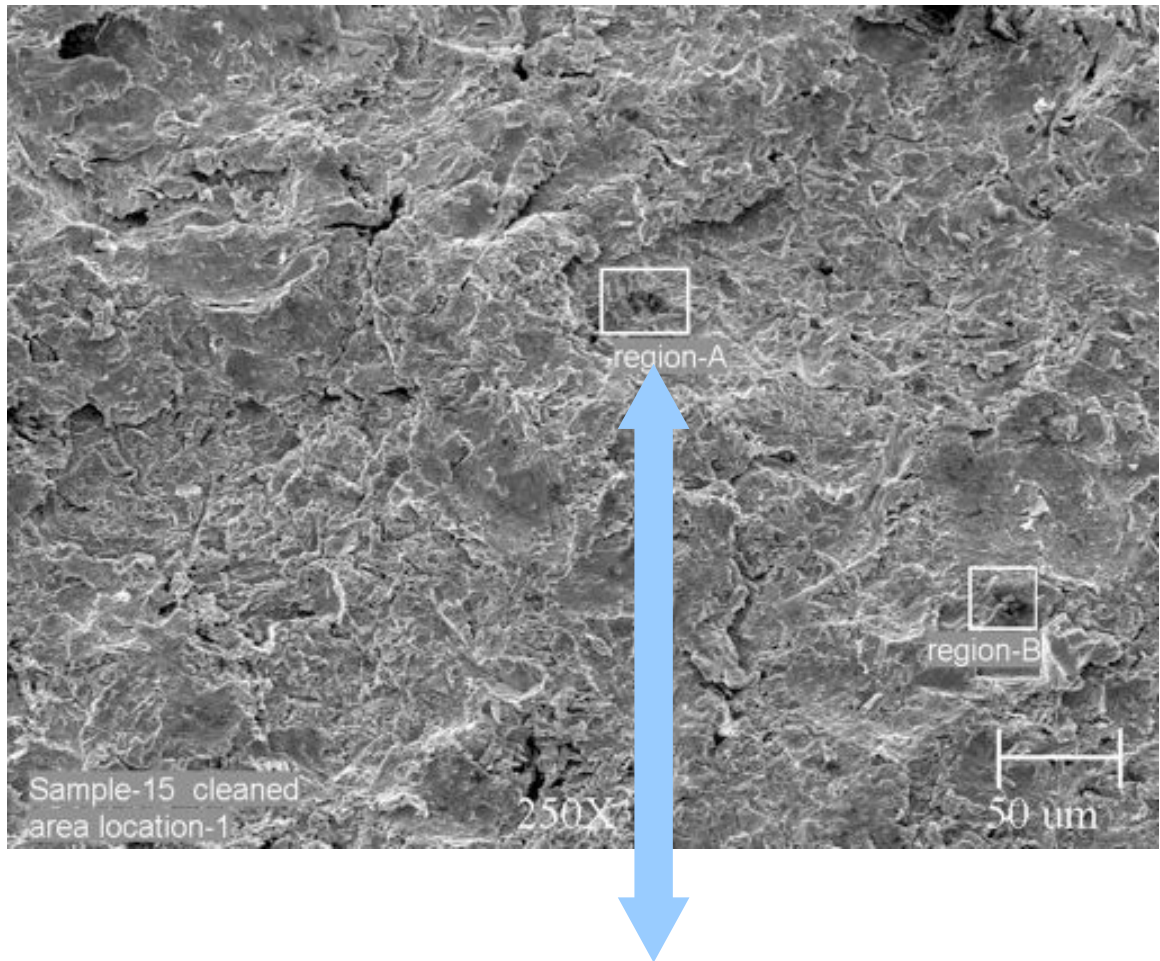
**Sample #2  
Field B,  
Regions A & B**

**EDS Reports &  
Images - Decontaminated (Clean) Side**



## Sample #2 - Clean Side, Field B, Region A:

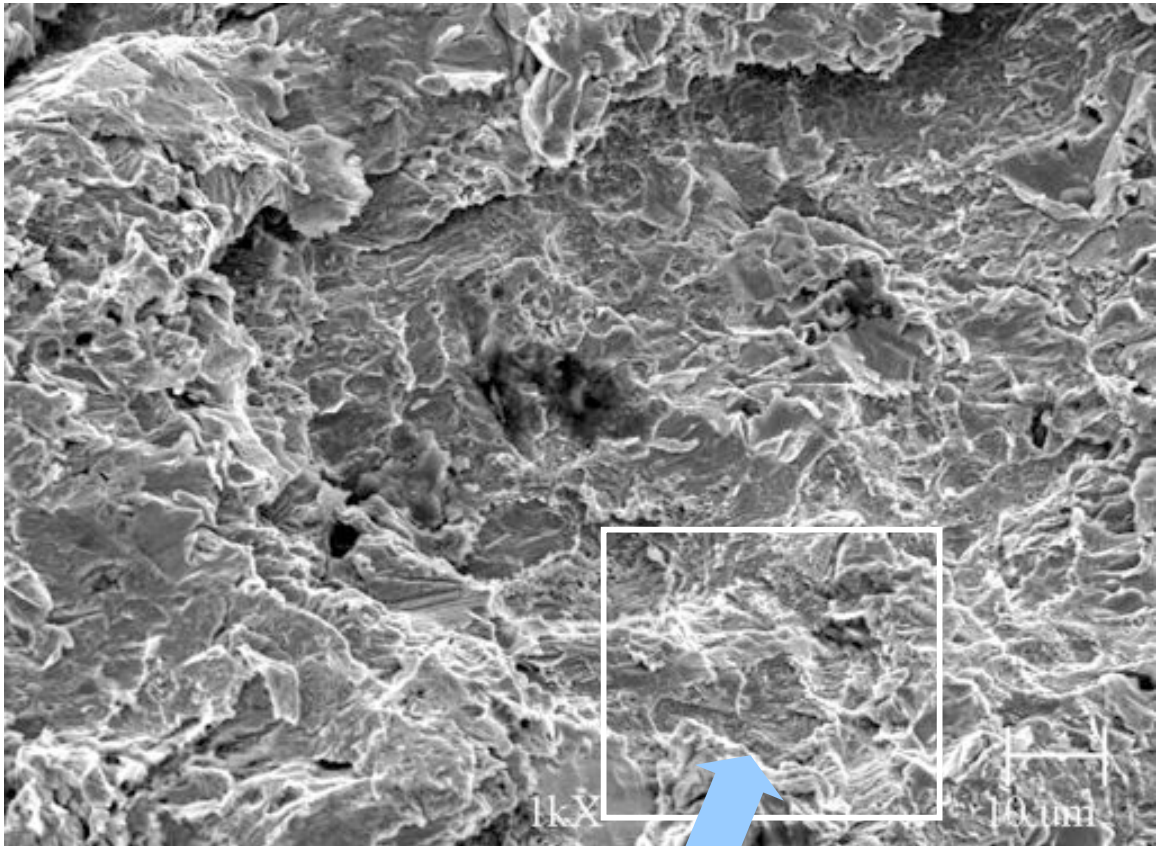
#9



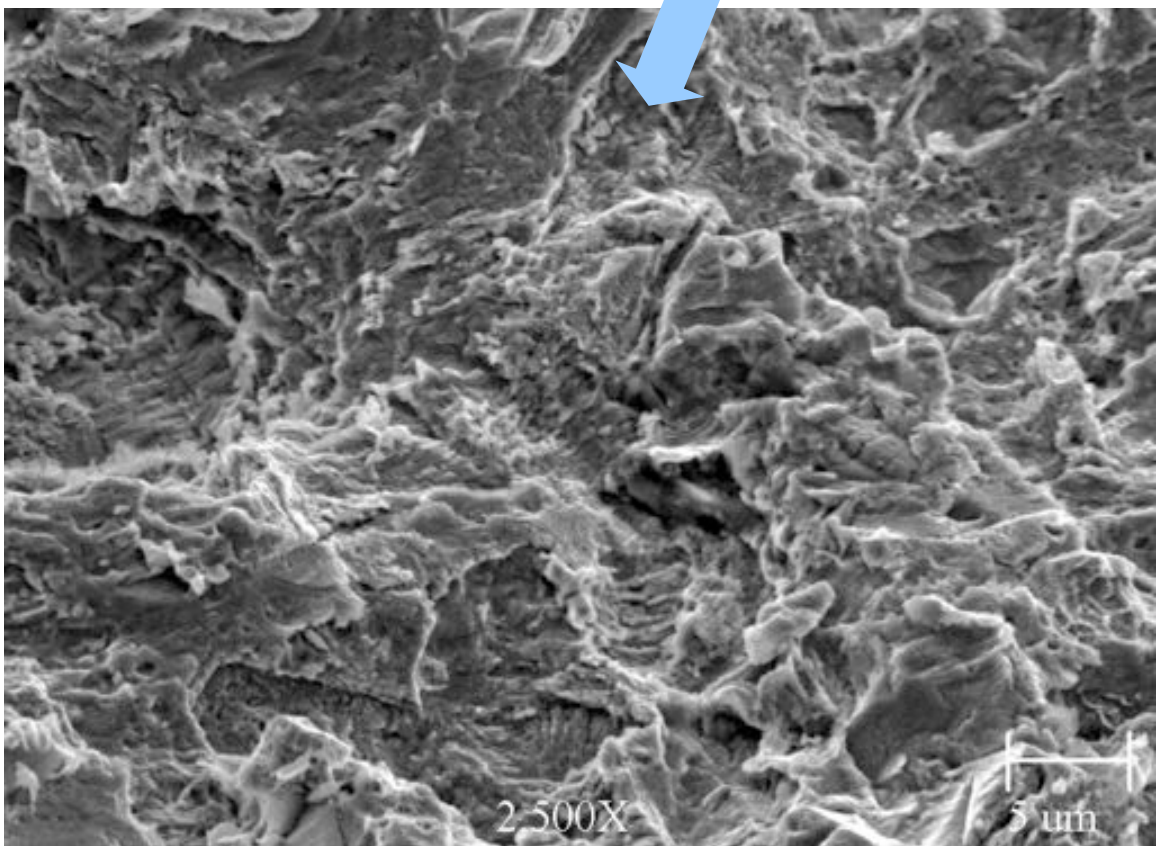
See Images #10 & #11

## Sample #2, Clean Side, Field B, Region A

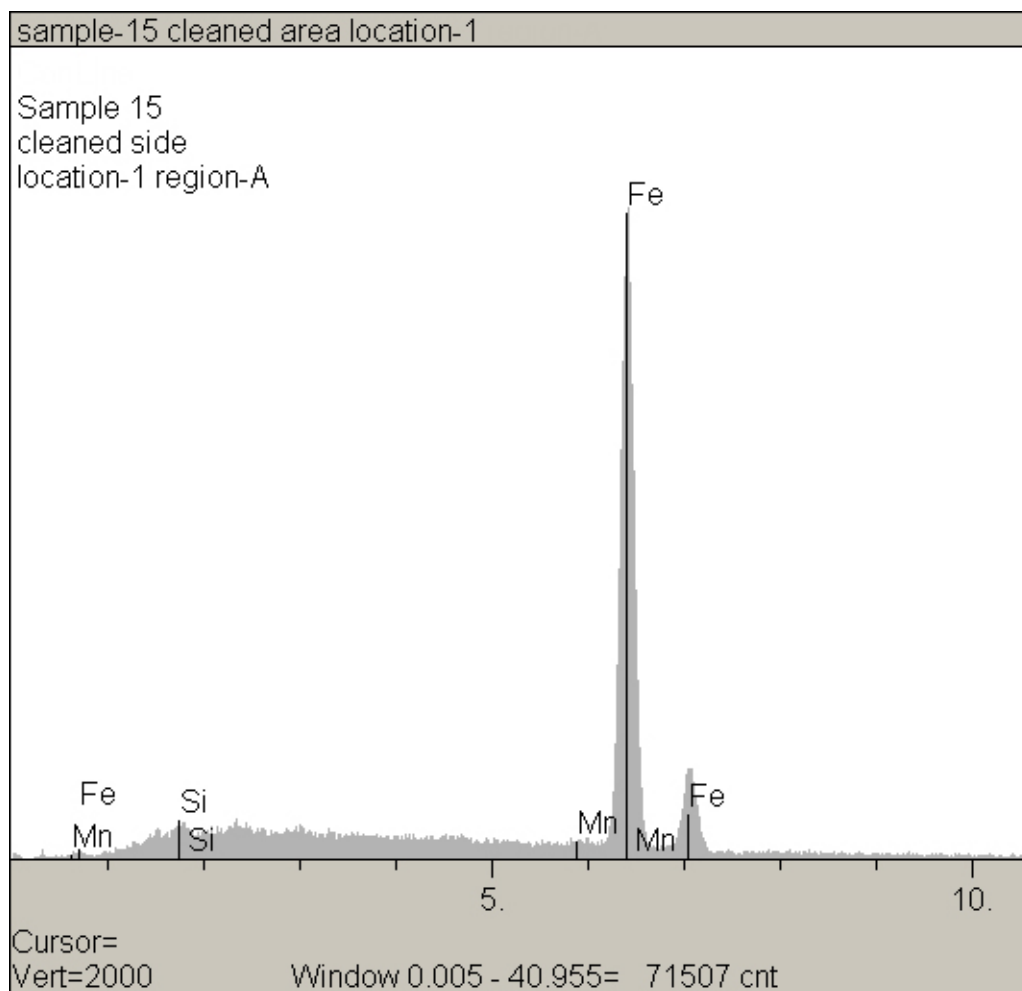
**#10**



**#11**



## Sample #2, EDS Report, Field B, Region A



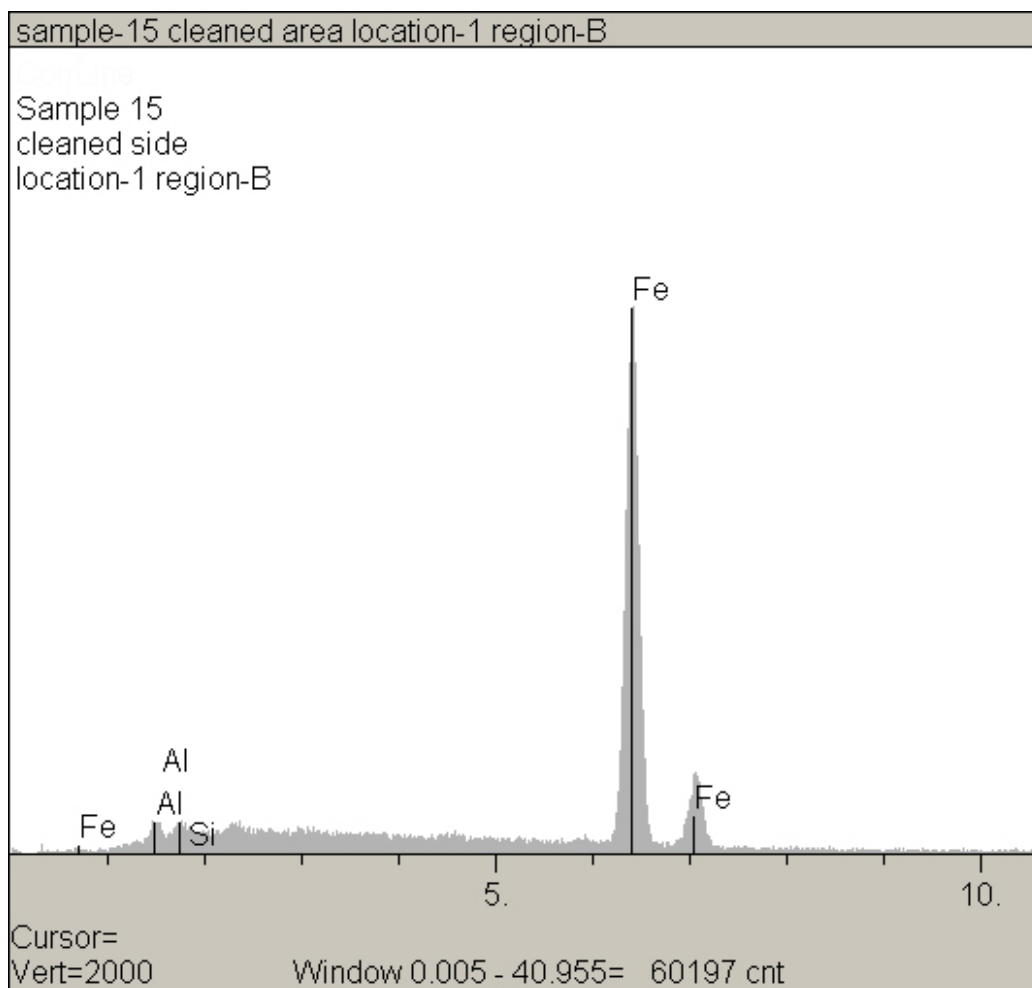
Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Si	Ka	5.46	0.603	0.869	wt.%	
Mn	Ka	1.92	0.357	0.221	wt.%	
Fe	Ka	444.66	5.445	98.910	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

## Sample #2, EDS Report, Field B, Region B



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	7.87	0.724	1.917	wt.%	
Si	Ka	6.18	0.642	1.152	wt.%	
Fe	Ka	374.48	4.996	96.931	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

**Sample #2**

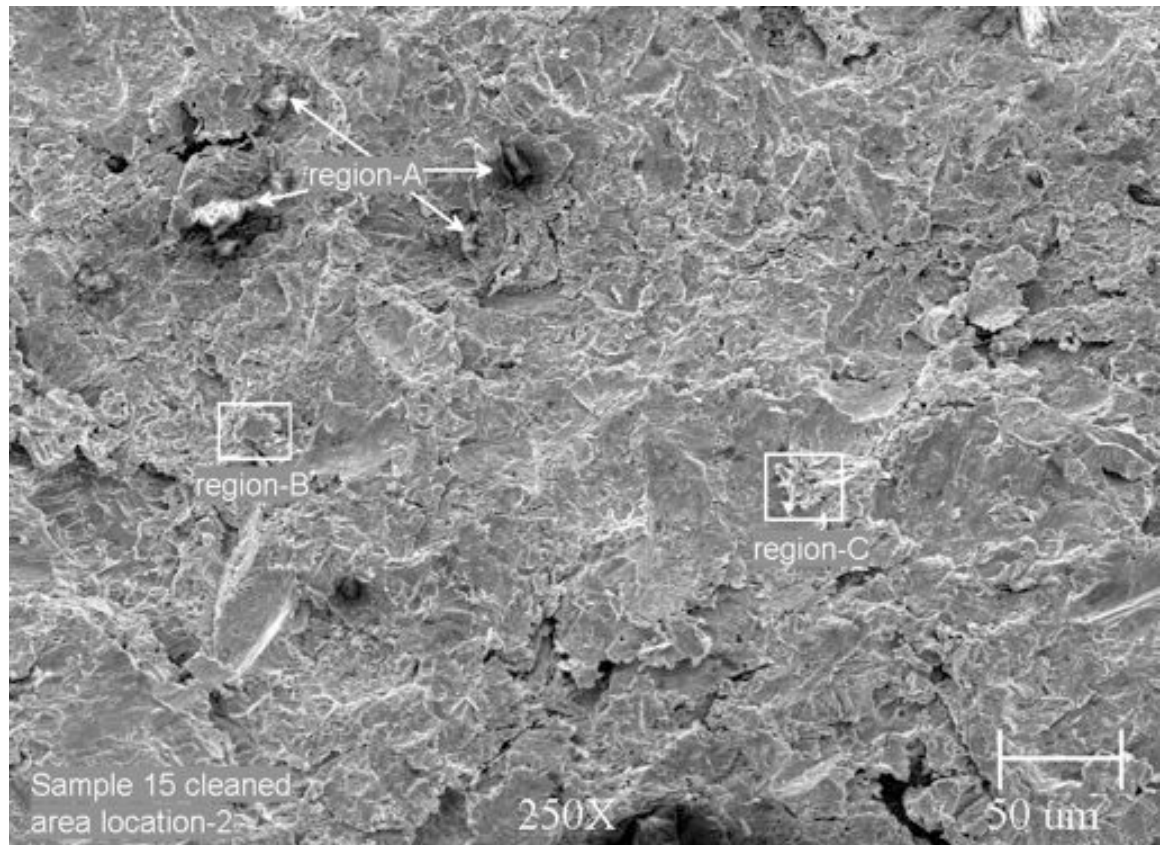
**Field C  
Regions A, B, C**

**EDS Reports &  
Images - Decontaminated (Clean) Side**



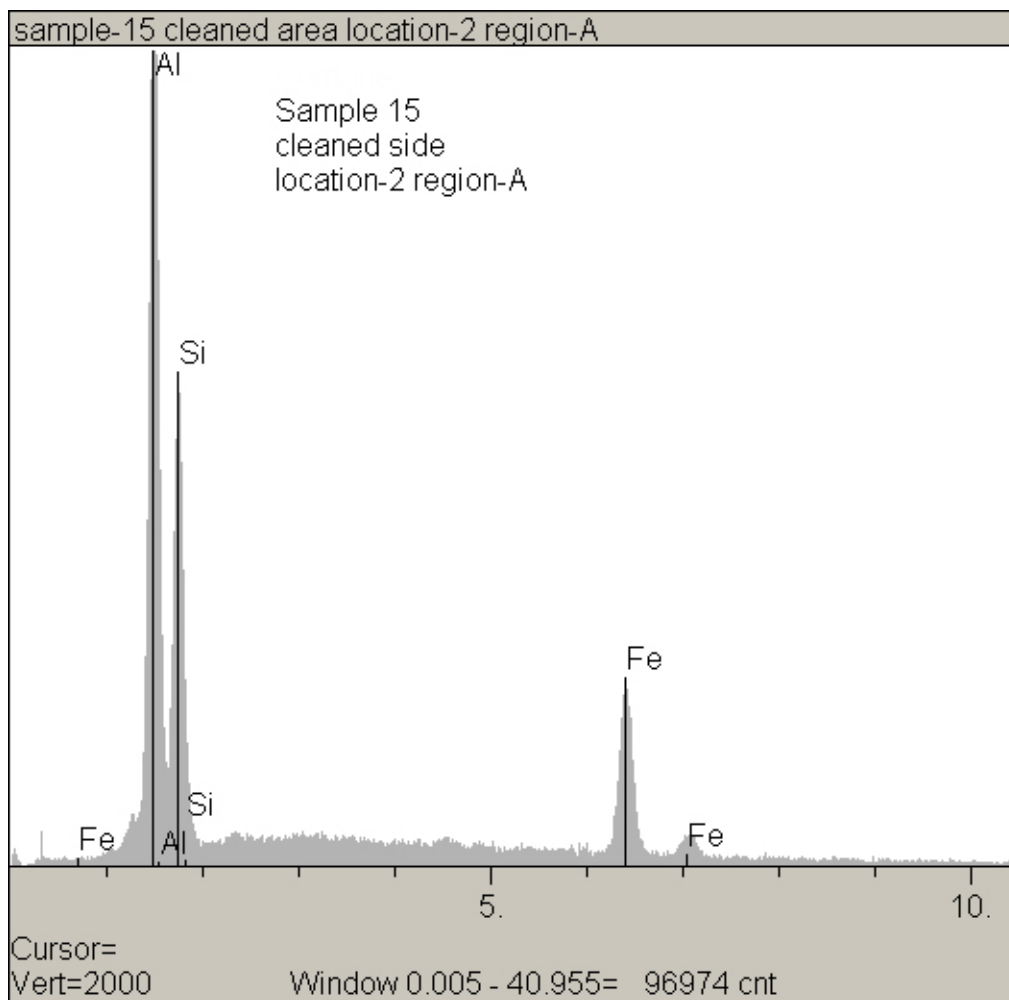
## Sample #2, Clean. Side, Field C, Regions A,B,C:

#12





## Sample #2, EDS Report, Field C, Region A



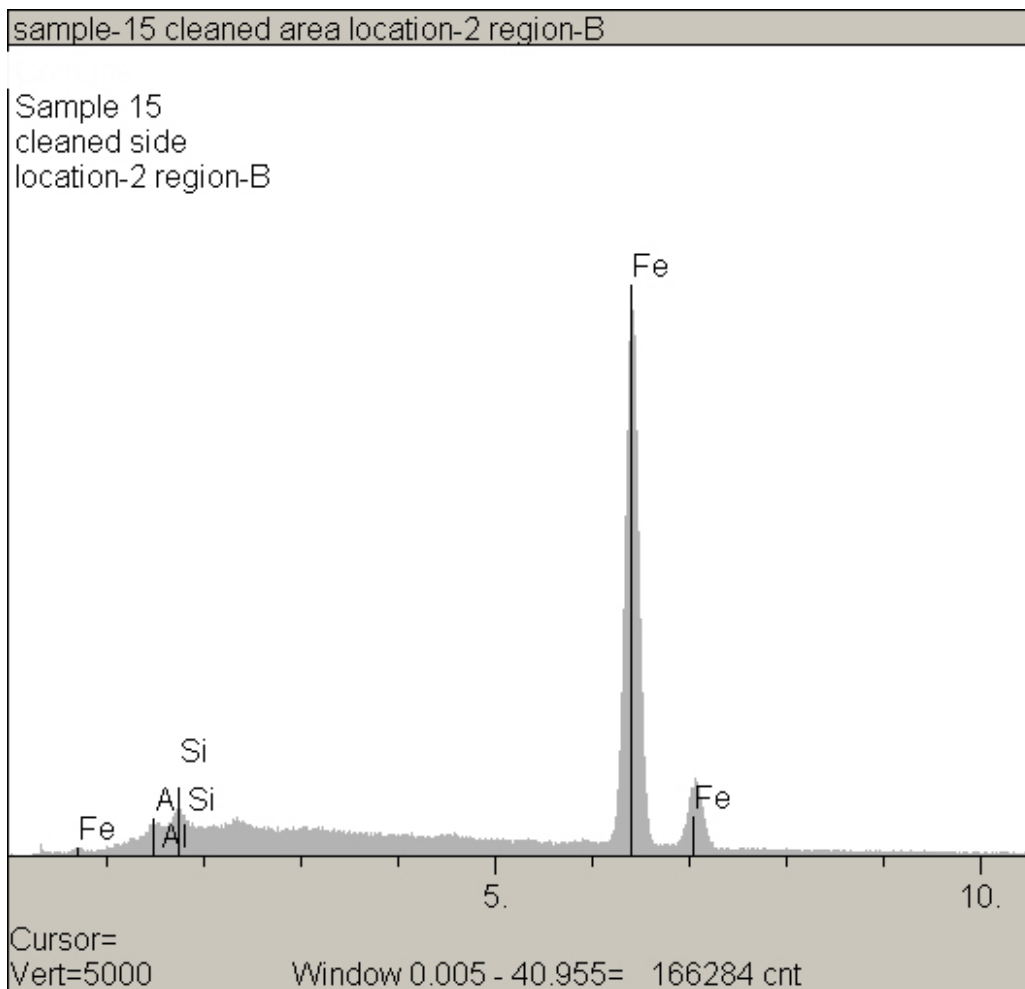
Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	461.70	5.548	39.720	wt.%	
Si	Ka	236.35	3.969	34.473	wt.%	
Fe	Ka	115.43	2.774	25.807	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

## Sample #2, EDS Report, Field C, Region B



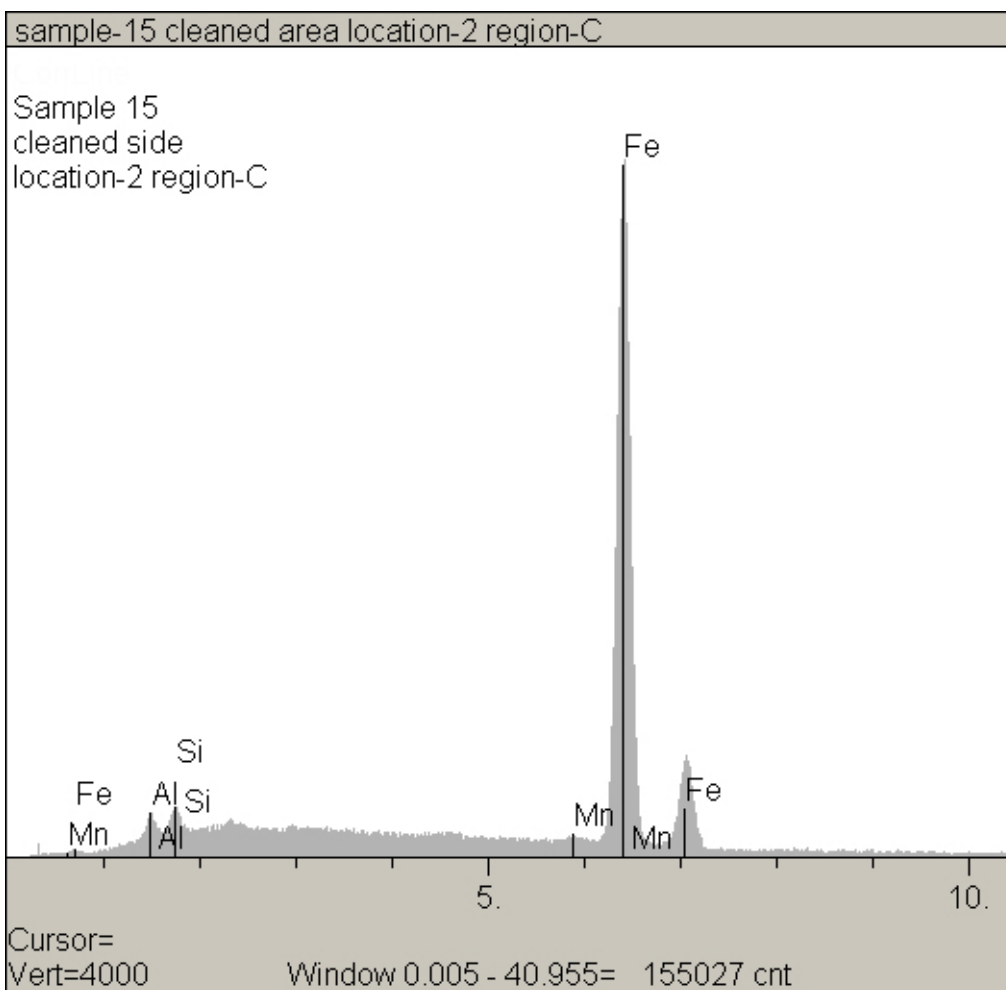
Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	14.59	0.986	1.359	wt.%	
Si	Ka	28.54	1.379	2.022	wt.%	
Fe	Ka	973.30	8.055	96.619	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

## Sample #2, EDS Report, Field C, Region C



Elt.	Line	Intensity (c/s)	Error 2-sig	Conc	Units	
Al	Ka	19.80	1.149	1.842	wt.%	
Si	Ka	23.96	1.264	1.705	wt.%	
Mn	Ka	5.86	0.625	0.327	wt.%	
Fe	Ka	967.93	8.033	96.126	wt.%	
				100.000	wt.%	Total

kV 15.0

Takeoff Angle 10.0°

Elapsed Livetime 60.0

**Anastas Technical Services**

17300 Mercury  
Houston, TX 77058  
(281) 488-9736

January 18, 2012

P.O. Box 6553  
Kingwood, TX 77325

Dear Mr. Hatle,

As requested I have examined and analyzed the corrosion coupon sample#15 that you submitted. The coupon sample was analyzed using a Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectrometer (EDS). It was desired to examine locations on the treated and untreated (corroded) sides of the coupon for the presence of corrosion products.

The sample was placed in the SEM and 15-20 locations were examined on the treated and untreated sides of the coupon. Each location was visually examined and the several regions of the surface deposits were checked using the EDS. Every location on the untreated side showed varying amounts of detectable Sulfur and Chlorine, in addition to Aluminum and Silicon along with Manganese and Iron that were components of the coupon base material. The deposits on the surface were typical for corrosion products and oxides. The examination of 15-20 locations on the treated side of the coupon showed no detectable Sulfur or Chlorine in any of the locations examined. There was evidence of embedded Aluminum and Silicon material that appears to be some type of abrasive blasting media. Several representative locations were documented and the SEM images and EDS spectra were submitted with the sample. If you have any questions or need any additional testing please let me know.

Thank you,

Tedd Anastas  
Anastas Technical Services

## Exhibits List:

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**\*\*NOTE:** Green areas indicate related images and EDS reports