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AUGER ELECTRON SPECTROSCOPY (AES)

SURFACE ANALYSIS REPORT
03 Mar 2017

JOB NUMBER Y0HVK289
PO NUMBER 7265

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AUGER ELECTRON SPECTROSCOPY (AES) ANALYSIS REPORT

Requester:	Gunner Puhl
Job Number:	Y0HVK289
Analysis Date:	03 Mar 2017

Purpose

The purpose of this analysis was to characterize the elemental surface composition and the oxide layer thickness of electropolished stainless steel using protocols based on SEMATECH SEMASPEC guidelines.

Sample ID

1. Control Sample
2. Sample 1

Summary

Please see the attached tables for the summary of the results.

Experimental

A summary of the instrumentation and analysis conditions is found at the end of this report.

Results and Interpretations

Data for the sample covered by this report are summarized in the attached tables and spectra. Sample identification is provided in each printout. For explanation of the terminology used in this report, please refer to the attached appendix. The etch rate for the instrument was calibrated with thermally grown SiO_2 and all thickness measurements are referenced to this material. As is specified in the SEMATECH procedure for stainless steel, we report here the as-received surface composition, the thickness of the surface oxide and carbon layers, the value and depth of the maximum Cr:Fe ratio and the composition of the surface at the end of the depth profile.

Carbon and oxygen detected at the end of profile may have been readsorbed during the depth profile.

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AUGER ELECTRON SPECTROSCOPY (AES) APPENDIX

This analysis was performed in accordance with SEMATECH Semaspec 91060573B-STD, Feb. 22, 1993 revision.

The Auger Electron Spectrometer used was a PHI 680. The following instrument operating parameters were used for the PHI analysis:

Beam energy	5kV
Beam current	200nA
Magnification	1000X
Angle between e-beam and surface normal	30°
Angle between spectrometer and surface normal	30°
Angle between argon beam and surface normal	33°
Analyzer Type	CMA
Argon beam energy	3kV
Etch rate, calibration standard	32Å/min, SiO_2
Date of last calibration	Jan 31, 2017

It should be noted that although the procedures used in these analyses follow SEMATECH methodology for characterization of electropolished cleanroom components, comparisons among different sets of samples can be accurately made only if analyzed under comparable conditions as listed above.

Survey scans (plots of the first derivative of the number of electrons detected as a function of energy) were used to characterize the elemental composition of the surfaces. Each survey provides information from the outermost few molecular layers of the surface. The composition values listed in Table A1 and used in the Atomic Concentration Profiles (ACP) were calculated using sensitivity factors based on pure elements or selected compounds and are expressed as atom percentages for the elements that were detected.

Depth profiles of Fe, Ni, Cr, O and C concentrations were acquired to a depth of 150Å. The etch depths are based on ion etch parameters calibrated with SiO_2 standards. Materials other than SiO_2 will etch at a rate faster or slower than SiO_2. Therefore, calculated etch depths are primarily useful in comparing information from similar materials.

GLOSSARY OF KEY PARAMETERS FOR 316L STAINLESS STEEL AS MEASURED FROM AES DEPTH PROFILES

The following is a glossary defining key values for evaluating electropolished 316L tubing for high purity gas delivery systems.

1. Oxide Thickness: The oxide thickness is defined using a full width half maximum (FWHM) measurement; it is the depth at which the oxide signal has fallen to half the maximum peak height, as described in SEMATECH methodology. Typical values for well electropolished material range from 20 to 50 Å.
2. Maximum Cr/Fe ratio: As determined from the Atomic Concentration Profile (ACP), the maximum ratio of Cr/Fe is the most direct measure of chromium enrichment in the oxide layer. In addition to the absolute value of the maximum Cr/Fe ratio, the depth at which it occurs is also recorded. Typical values for maximum Cr/Fe ratios range from ~1.5 to 2.0 or greater for well electropolished material. The depth at which the maximum Cr/Fe ratio is found varies, but is usually about one half the oxide thickness.
3. Surface iron oxide layer: The Fe signal usually does not intersect the Cr signal until at least 20 Å. When the Fe signal is initially higher than Cr (typically within the first 5 Å), a thin layer of surface iron oxide is present. AES Atomic Concentration Profiles (ACP) of well electropolished 316L stainless steel surfaces should exhibit a rise in the Fe signal from the outer surface to the maximum bulk concentration.
4. Carbon thickness: Measurement of the thickness of surface organic material uses a full width half maximum (FWHM) technique. Typical values for carbon thickness range from 5 to 10 Å, however, significantly contaminated samples can show surface carbon layer thickness of 20 Å or more.

Table A1. Surface elemental compositions estimated from the AES survey spectra (expressed as atomic percent for the detected elements)

Spectrum Number	Sample Description	Cl	S	P	C	N	O	Si	Cr	Fe	Ni	Mo
101	Control	---	0.4	---	44	---	32	---	---	10	3	---
201	#1	---	---	---	55	2	23	---	6	9	3	---

For those spectra that apply:
Values with a
< Indicates the detection limit with the acquisition parameters used. The element, if present, was below the stated limit.
--- Indicates no observation of a signal.

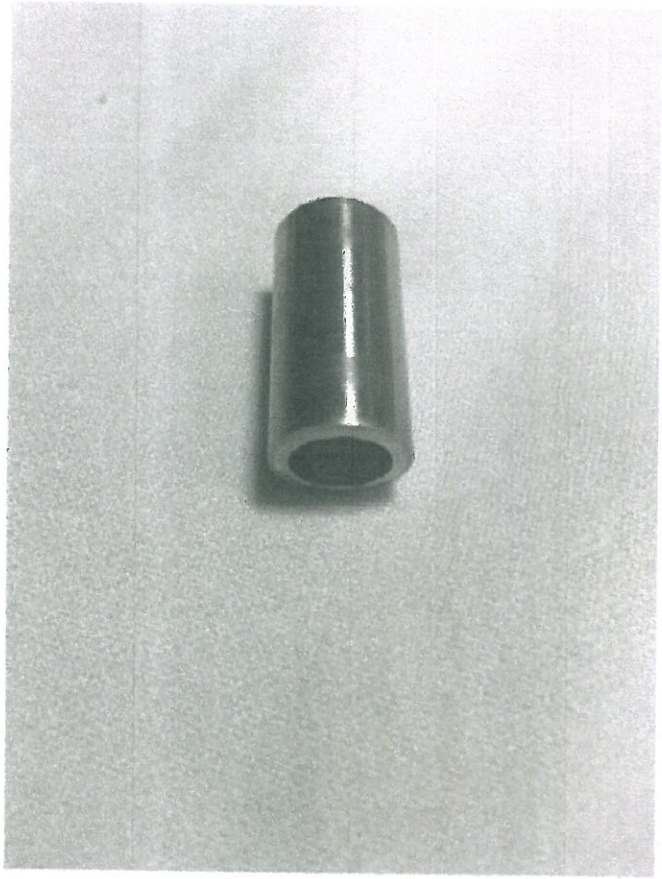
Table A2. Measurements from AES depth profile data. All depth and thickness values are expressed in SiO_2 equivalents.

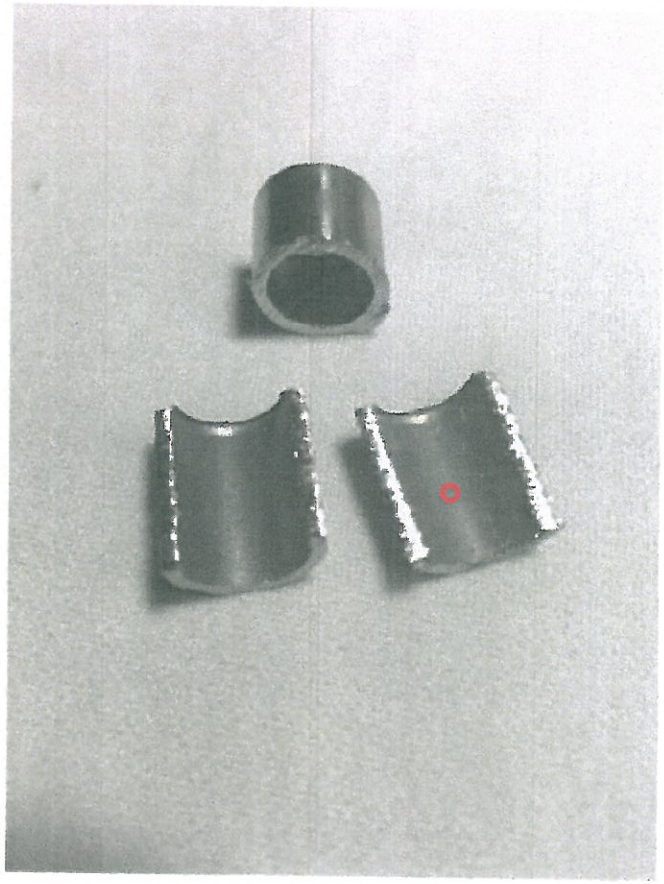
Sample Identification	Oxide Thickness (Å)[1]	Max. Cr/Fe Ratio (depth, Å)[2]	Surface Iron Oxide Thickness[3]	Carbon Layer Thickness (Å)[4]
Control	~900	0.65 @ 70	~900	<10
#1	~900	1.6 @ 9	~900 (below Cr)	<10

[1] 50% O signal (FWHM)
[2] from ACP profile
[3] point at which Cr/Fe is <1 before Cr enrichment
[4] 50% C signal ** (FWHM)

** An in-depth explanation of these terms is provided in the Glossary in item numbers corresponding to the superscripts above.

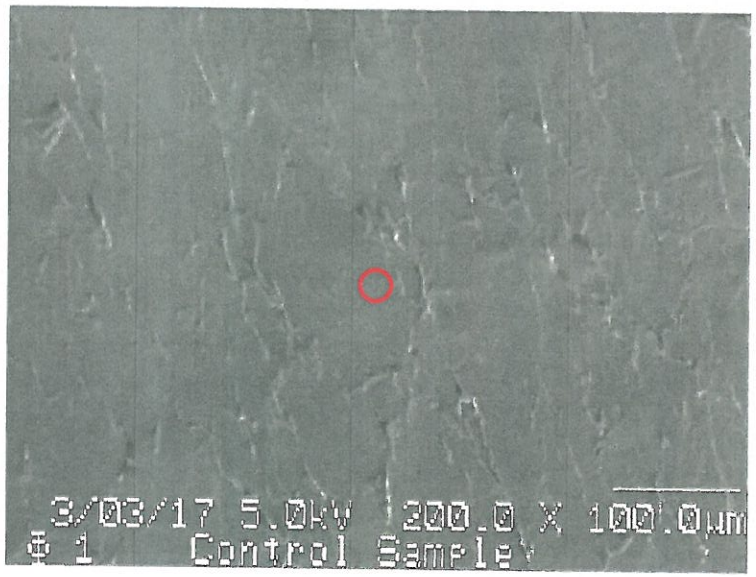
Digital Images Regarding Sample Preparation

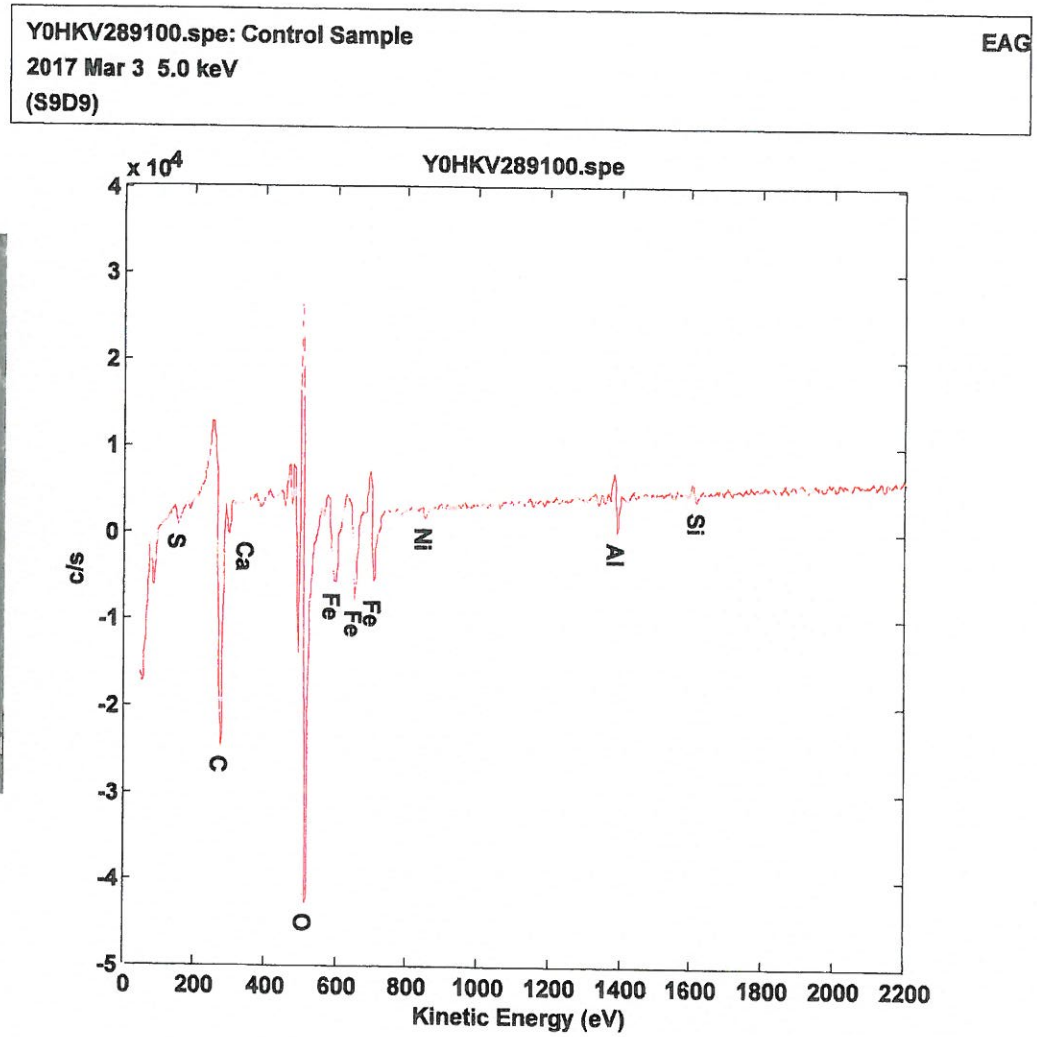




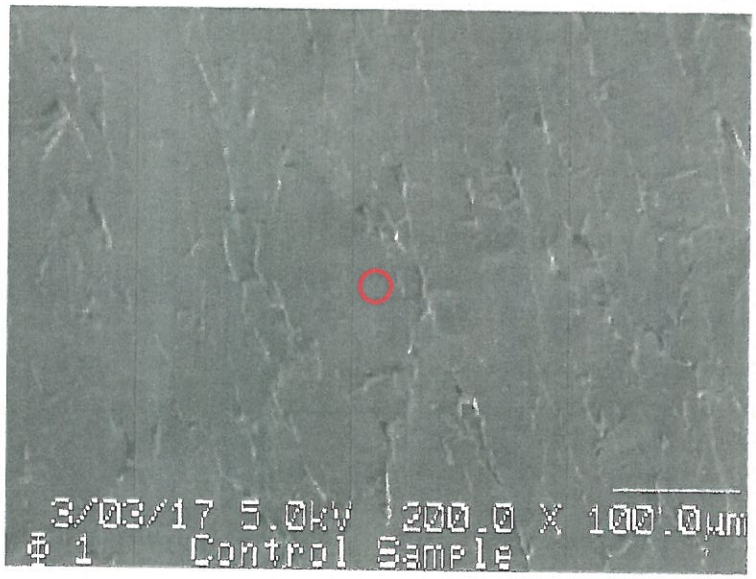
The surface of interest was the ID of the tubing sample. Therefore, each sample was cut to expose the surface of interest. The cuts were made without machining fluids and done slowly with cooling to minimize increased temperatures that might impact the surface oxide layer.

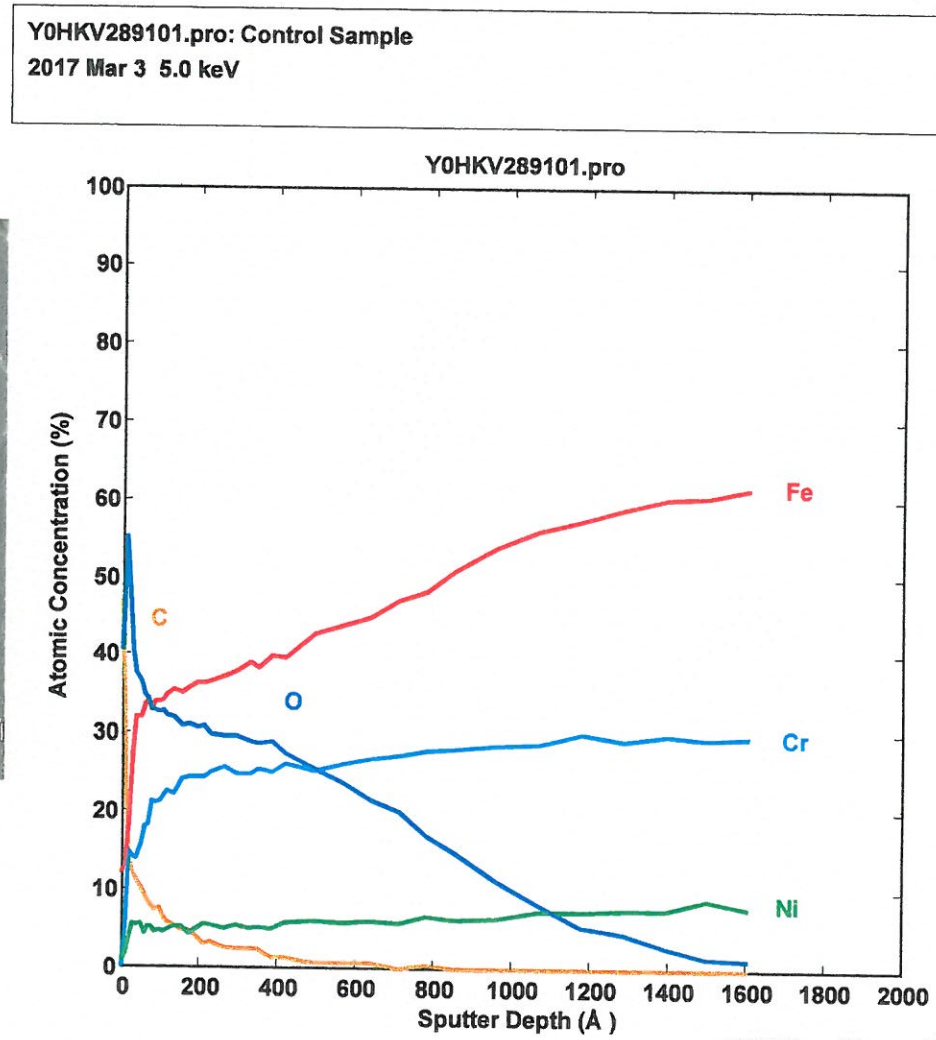
SEI and Auger Surface Survey Spectrum: Control Sample



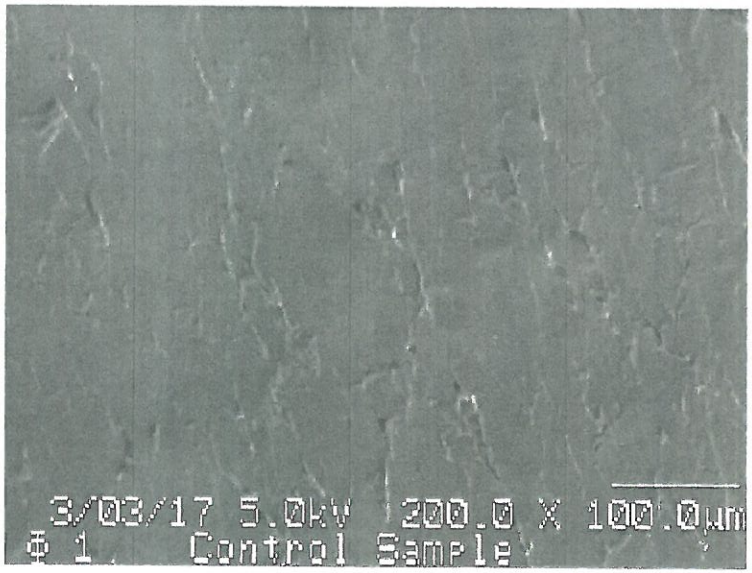


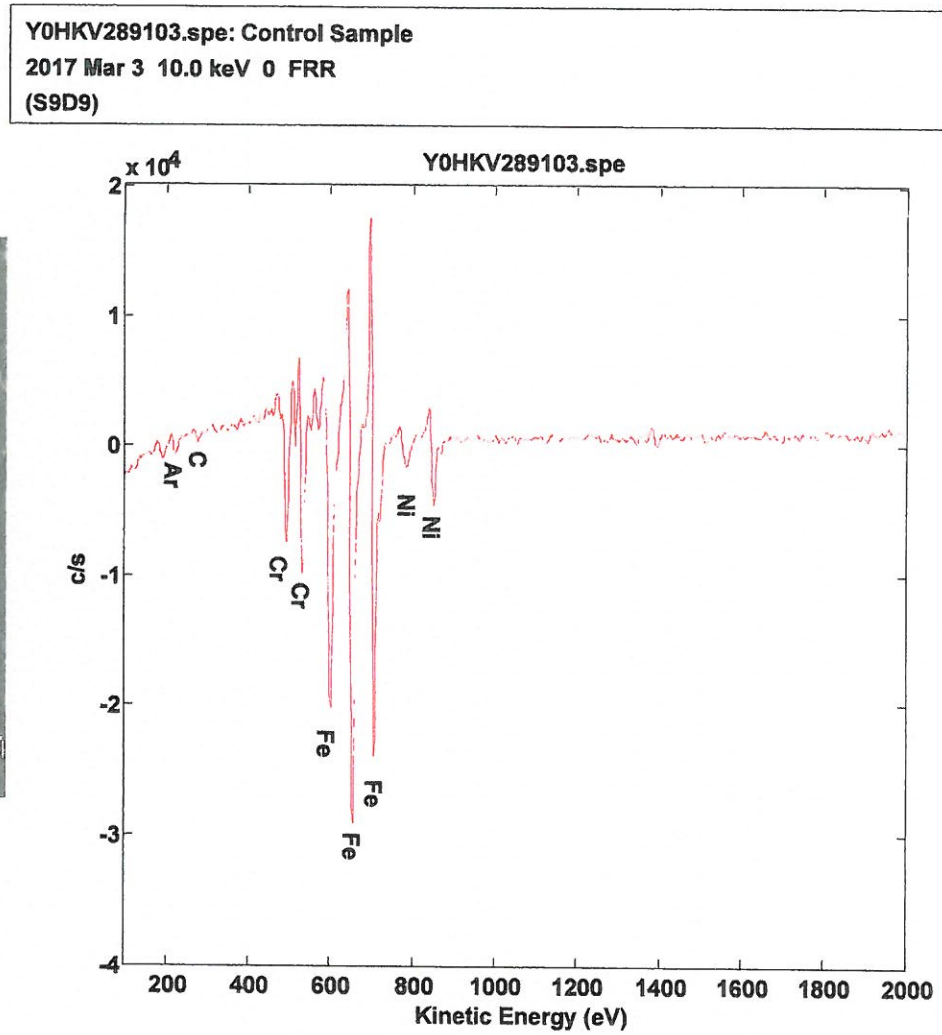
SEI and Auger Sputter Depth Profile: Control Sample



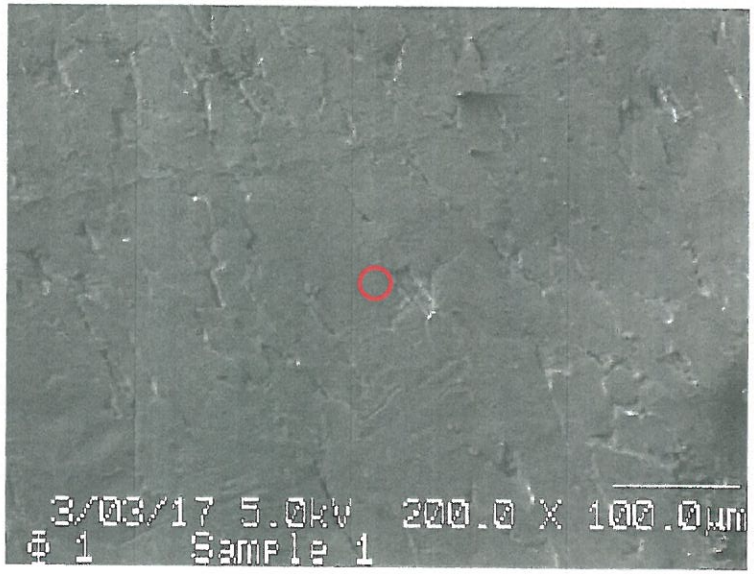


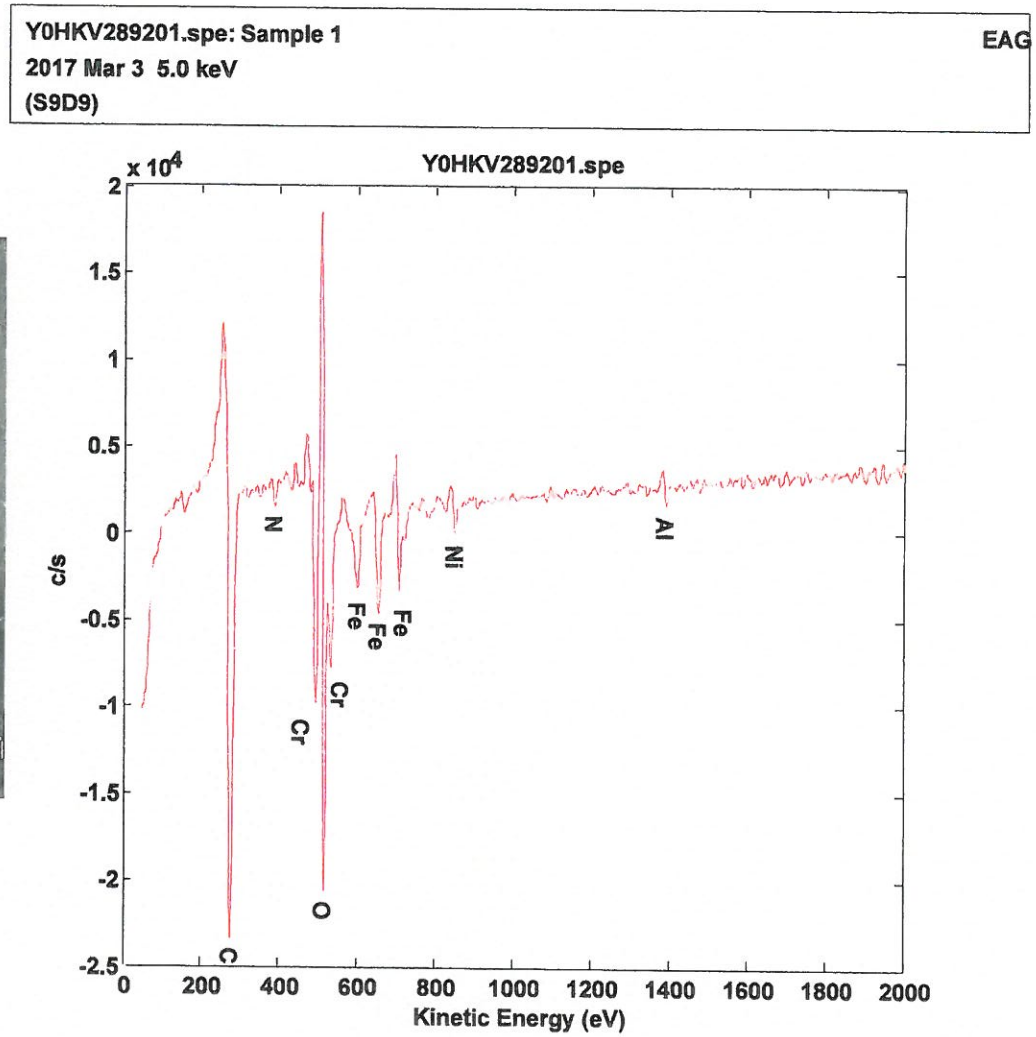
SEI and Post Profile Auger Spectrum: Control Sample



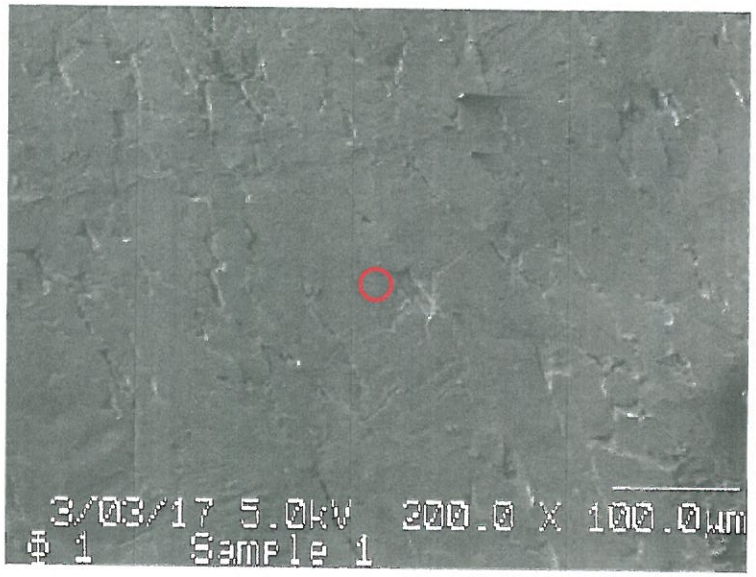


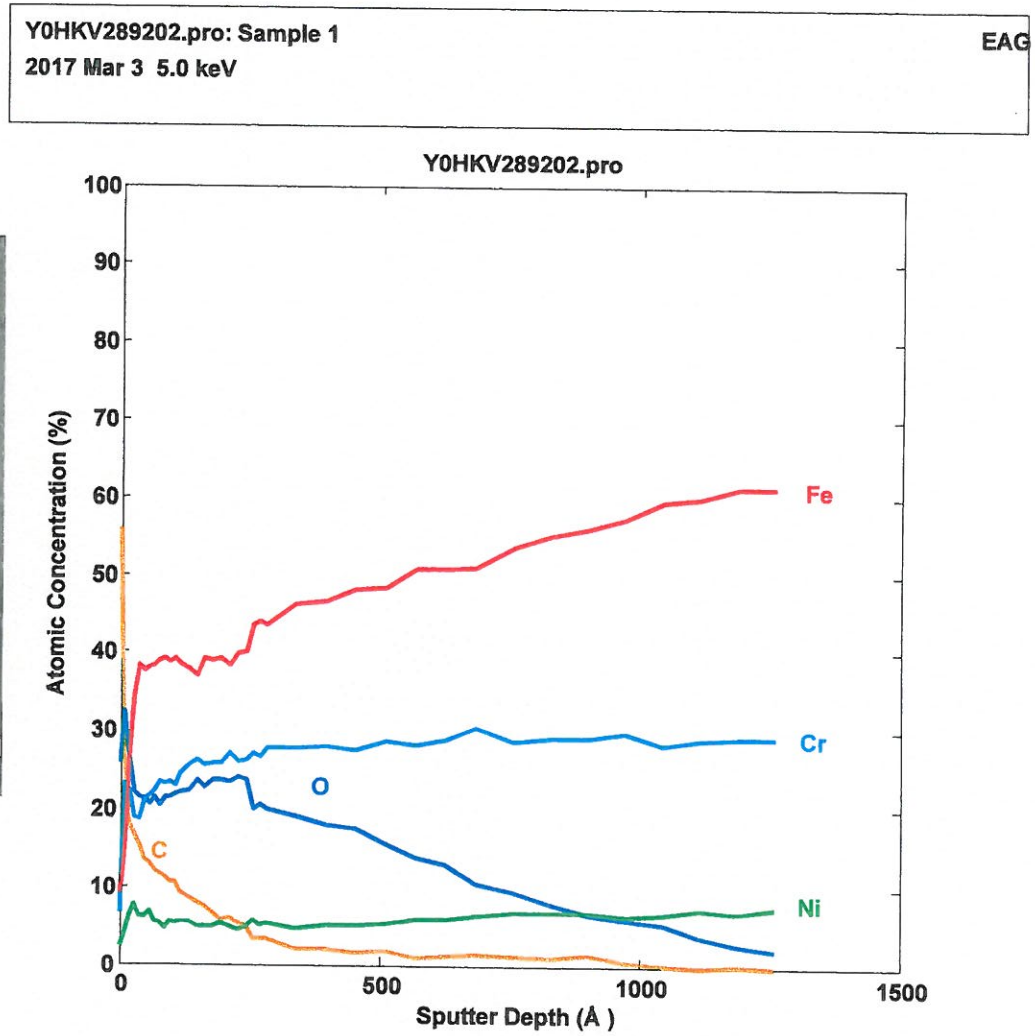
SEI and Auger Surface Survey Spectrum: Sample 1





SEI and Auger Sputter Depth Profile: Sample 1





SEI and Post Profile Auger Spectrum: Sample 1