LABORATORY NOTEBOOK

CNWRA/SwRI

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NOTEBOOK NO. 659
ISSUED TO Yiming Pan
ON 5/27/2004 **20**
DEPARTMENT 20
RETURNED **20**

Yiming Pan YP Pan YMP
Brian Dealy BKD

Continuation of Notebook 498

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TITLE Initial Scientific Notebook Entry

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Initial Entry for Phase Stability and Passive Film Study of Alloy 22

Title: Phase stability and passive film study of Alloy 22

Tests Performed by: Yi-Ming Pan and Darrell Dunn

Objectives: Evaluate the effect of fabrication processes on the microstructure of Alloy 22 and changes in the oxide composition and structure as a function of the applied potential, time, and temperature of electrochemical treatments

Equipments: High-temperature furnace, optical microscope, scanning electron microscope, X-ray photoelectron spectroscopy, and Auger electron spectroscopy

Materials: Alloy 22 specimens under mill-annealed, welded, and heat-treated conditions, and after various electrochemical treatments

Measurement Parameters: precipitate identification and volume fraction, temperature, and thickness, composition, and chemical state of the oxide films

Uncertainty and Sources of Error: Several precipitate phases with similar compositions and structures form in Alloy 22. The presence of oxide film that formed after air exposure may influence characterization of the passive film arising from electrochemical treatments.

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Book No.
TITLE Precipitate volume fraction measurements for GTAW and GMAW Alloy 22 welds

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Continuation of Notebook 498, p. 96.

Alloy 22 welds:

① GTAW material

Alloy 22 base metal heat 2277-1-3164

Filler heat WN 813 (Alloy 622)

② GMAW material

Alloy 22 base metal heat 2277-1-3164

Filler heats WN813 / XX19778 G11 (Alloy 622)

③ Heat treatments documented on p. 92, Notebook 498.

Results for GTAW weld samples:

T2(1125C/20min + 870C/60min)

image	% Precip.	% Matrix
M2080	2.0	98.0
M2081	3.6	96.4
M2082	4.1	95.9
M2083	4.1	95.9
M2084	5.3	94.7
M2085	2.8	97.2
M2086	2.6	97.4
M2087	3.0	97.0
M2088	3.1	96.9
M2089	2.5	97.5
M2090	3.6	96.4
M2091	4.8	95.2
M2092	1.9	98.1
M2093	4.5	95.5
M2094	2.3	97.7
M2095	4.2	95.8
M2096	3.2	96.8
M2097	5.8	94.2
M2098	6.0	94.0
M2099	3.9	96.1

Summary Statistics	
Mean	3.7
Standard Deviation	1.2
Minimum	1.9
Maximum	6.0
Sum	73.3
Count	20

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T3(870C/30min)

image	% Precip.	% Matrix
M2100	3.6	96.4
M2101	2.2	97.8
M2102	2.6	97.4
M2103	2.4	97.6
M2104	2.0	98.0
M2105	2.5	97.5
M2106	4.5	95.5
M2107	2.2	97.8
M2108	2.4	97.6
M2109	2.5	97.5
M2110	2.5	97.5
M2111	3.4	96.6
M2112	1.9	98.1
M2113	3.3	96.7
M2114	2.6	97.4
M2115	4.8	95.2
M2116	6.1	93.9
M2117	5.8	94.2
M2118	4.2	95.8
M2119	2.8	97.2

Summary Statistics	
Mean	3.2
Standard Deviation	1.2
Minimum	1.9
Maximum	6.1
Sum	64.3
Count	20

T5(870C/10hr)

image	% Precip.	% Matrix
M2120	15.1	84.9
M2121	22.1	77.9
M2122	15.7	84.3
M2123	12.3	87.7
M2124	21.4	78.6
M2125	20.8	79.2
M2126	20.8	79.2
M2127	16.1	83.9
M2128	15.2	84.8
M2129	11.1	88.9
M2130	17.3	82.7
M2131	18.2	81.8
M2132	14.9	85.1
M2133	22.3	77.7
M2134	17.3	82.7
M2135	22.9	77.1
M2136	28.4	71.6
M2137	19.3	80.7
M2138	20.7	79.3
M2139	20.6	79.4

Summary Statistics	
Mean	18.6
Standard Deviation	4.1
Minimum	11.1
Maximum	28.4
Sum	372.5
Count	20

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From Page No. Results for GMAW weld samples

M2(1125C/20min + 870C/60min)

image	% Precip.	% Matrix
M2005	2.5	97.5
M2006	4.4	95.6
M2007	3.1	96.9
M2008	8.2	91.8
M2009	7.5	92.5
M2010	8.0	92.0
M2011	3.0	97.0
M2012	3.5	96.5
M2013	3.6	96.4
M2014	4.1	95.9
M2015	3.6	96.4
M2016	1.3	98.7
M2017	3.3	96.7
M2018	3.8	96.2
M2019	5.1	94.9
M2020	4.0	96.0
M2021	4.6	95.4
M2022	5.4	94.6
M2023	6.1	93.9
M2024	4.9	95.1

Summary Statistics	
Mean	4.5
Standard Deviation	1.8
Minimum	1.3
Maximum	8.2
Sum	90.0
Count	20

M3(870C/30min)

image	% Precip.	% Matrix
M2025	3.2	96.8
M2026	5.7	94.3
M2027	5.9	94.1
M2028	5.6	94.4
M2029	4.2	95.8
M2030	6.5	93.5
M2031	9.4	90.6
M2032	3.5	96.5
M2033	3.5	96.5
M2034	5.4	94.6
M2035	4.3	95.7
M2036	3.2	96.8
M2037	3.7	96.3
M2038	3.1	96.9
M2039	4.7	95.3
M2040	3.4	96.6
M2041	4.1	95.9
M2042	4	96.0
M2043	2.9	97.1
M2044	3.3	96.7

Summary Statistics	
Mean	4.5
Standard Deviation	1.6
Minimum	2.9
Maximum	9.4
Sum	89.6
Count	20

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M5(870C/10hr)

image	% Precip.	% Matrix
M2060	22.3	77.7
M2061	18.4	81.6
M2062	20.0	80.0
M2063	19.8	80.2
M2064	19.4	80.6
M2065	20.0	80.0
M2066	18.3	81.7
M2067	17.1	82.9
M2068	19.2	80.8
M2069	18.9	81.1
M2070	17.0	83.0
M2071	19.3	80.7
M2072	17.0	83.0
M2073	19.4	80.6
M2074	17.6	82.4
M2075	19.0	81.0
M2076	11.4	88.6
M2077	13.6	86.4
M2078	17.7	82.3
M2079	17.4	82.6

Summary Statistics	
Mean	18.1
Standard Deviation	2.3
Minimum	11.4
Maximum	22.3
Sum	362.8
Count	20

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TITLE Precipitate volume fraction measurement for thermally aged Alloy 22

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Test material:

Alloy 22 base metal heat 2277-3-3266.

Mill-annealed Alloy 22 heat treated at 870°C for 30 minutes and 1, 10, and 100 hours. The detailed heat treatments are documented on p.10, Notebook 64.

Measurement results:

MA1(870C/30min)

image	% Precip.	% Matrix
M2140	1.6	98.4
M2141	1.2	98.8
M2142	0.9	99.1
M2143	1.3	98.7
M2144	1.7	98.3
M2145	1.8	98.2
M2146	1.7	98.3
M2147	1.8	98.2
M2148	1.4	98.6
M2149	1.8	98.2
M2150	1.7	98.3
M2151	1.4	98.6
M2152	1.5	98.5
M2153	1.6	98.4
M2154	2.9	97.1
M2155	1.3	98.7
M2156	1.7	98.3
M2157	1.4	98.6
M2158	1.5	98.5
M2159	1.7	98.3

Summary Statistics	
Mean	1.6
Standard Deviation	0.4
Minimum	0.9
Maximum	2.9
Sum	31.9
Count	20

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MA2(870C/60min)

image	% Precip.	% Matrix
M2160	1.6	98.4
M2161	1.9	98.1
M2162	1.7	98.3
M2163	2.2	97.8
M2164	1.9	98.1
M2165	2.5	97.5
M2166	1.8	98.2
M2167	2.5	97.5
M2168	2.2	97.8
M2169	2.6	97.4
M2170	2.4	97.6
M2171	1.8	98.2
M2172	1.8	98.2
M2173	2.9	97.1
M2174	2.2	97.8
M2175	2.3	97.7
M2176	2.8	97.2
M2177	3.5	96.5
M2178	3.4	96.6
M2179	2.9	97.1

Summary Statistics	
Mean	2.3
Standard Deviation	0.5
Minimum	1.6
Maximum	3.5
Sum	46.9
Count	20

MA3(870C/10hr)

image	% Precip.	% Matrix
M2180	5.1	94.9
M2181	5.4	94.6
M2182	6.7	93.3
M2183	6.9	93.1
M2184	6.1	93.9
M2185	7.3	92.7
M2186	7.3	92.7
M2187	6.5	93.5
M2188	6.9	93.1
M2189	6.1	93.9
M2190	6.2	93.8
M2191	6.2	93.8
M2192	7.0	93.0
M2193	5.6	94.4
M2194	6.6	93.4
M2195	6.6	93.4
M2196	5.7	94.3
M2197	5.7	94.3
M2198	6.7	93.3
M2199	6.3	93.7

Summary Statistics	
Mean	6.3
Standard Deviation	0.6
Minimum	5.1
Maximum	7.3
Sum	126.9
Count	20

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MA4(870C/100hr)

image	% Precip.	% Matrix
M2200	9.6	90.4
M2201	8.3	91.7
M2202	11.8	88.2
M2203	10.5	89.5
M2204	10.6	89.4
M2205	13.2	86.8
M2206	10.7	89.3
M2207	9.8	90.2
M2208	11.3	88.7
M2209	10.4	89.6
M2210	10.3	89.7
M2211	10.2	89.8
M2212	10.2	89.8
M2213	12.0	88.0
M2214	11.9	88.1
M2215	12.7	87.3
M2216	9.5	90.5
M2217	10.9	89.1
M2218	11.8	88.2
M2219	9.0	91.0

Summary Statistics	
Mean	10.7
Standard Deviation	1.2
Minimum	8.3
Maximum	13.2
Sum	214.7
Count	20

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TITLE Electrochemical treatment conditions for Alloy 22 XPS specimens 11, 12 & 13
Project No.
Book No.

From Page No.

Objective: Determine changes in passive surface film structure and composition formed on Alloy 22 specimens with three different electrochemical treatment conditions

Test specimens:

① XPS # 11
section of the long-term crevice specimen C22L14 exposed in a solution of 4M Cl^- + 1.4 mM HCO_3^- + 0.208 mM SO_4^{2-} + 0.162 mM NO_3^- + 0.105 mM F^- at 250 mV and 95°C (pH = 7.485). The detailed test conditions are documented on P. 89, Notebook 541.

② XPS # 12
Section of the long-term crevice specimen C22L3 exposed in a similar solution for XPS # 11 under open circuit conditions. The detailed test conditions are documented on P. 90, Notebook 541.

③ XPS # 13
section of the compact tension specimen C2204 exposed in Simulated Concentrated Water for about 8,000 hrs at 95°C. The detailed test conditions are documented on p.30, Notebook 599.

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CNWRA *A center of excellence in earth sciences and engineering*
6220 Culebra Road · San Antonio · Texas, U.S.A. 78228-5166

May 28, 2004

Mr. Carl Steinke
RBD Enterprises
563 SW 13th Street
Bend, OR 97702

Dear Carl:

Enclosed please find three types of Alloy 22 specimens in duplicate (XPS #11a&b, 12a&b, and 13a&b) submitted for surface analyses with x-ray photoelectron spectroscopy. Per our previous phone conversation, I need to have the specimens analyzed for determination of the thickness, composition, and oxidation state of the passive film. For each specimen, a wide-energy survey will be conducted on the as-received surface to determine surface elemental composition. The survey will be followed by high resolution scans combined with in-depth ion sputtering to determine concentration depth profile. The high-resolution spectra will be curve fitted to determine the oxidation state of nickel, chromium, and molybdenum, and the chemical-bonding environment for oxygen. Note that these specimens have been subjected to long-term immersion for several years. Formation of thicker oxide films is expected.

Please call me if you have any questions. Thank you for your assistance.

Sincerely,

Yiming Pan
Center for Nuclear Waste Regulatory Analyses
Southwest Research Institute
Tel: 210-522-6640
Fax: 210-522-5184
e-mail: ypan@swri.org

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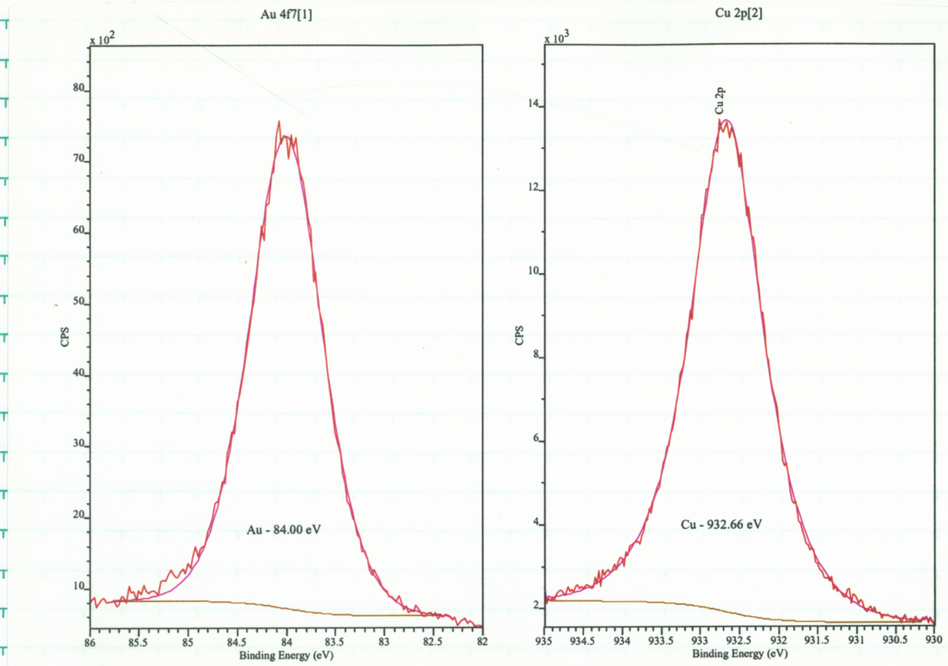
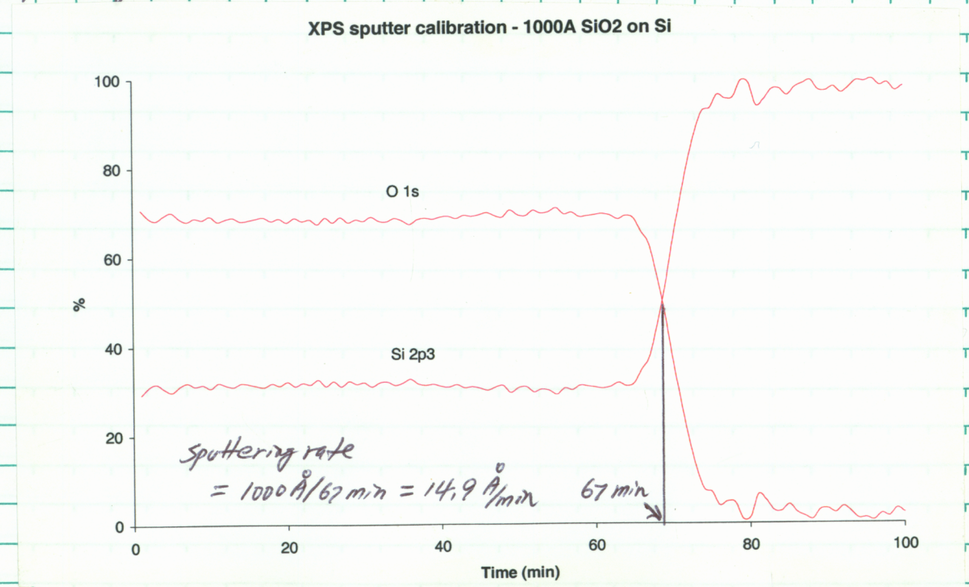
1. Binding energy



2. Sputtering rate



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TITLE XPS Analysis of Alloy 22 Specimens #11 and 12

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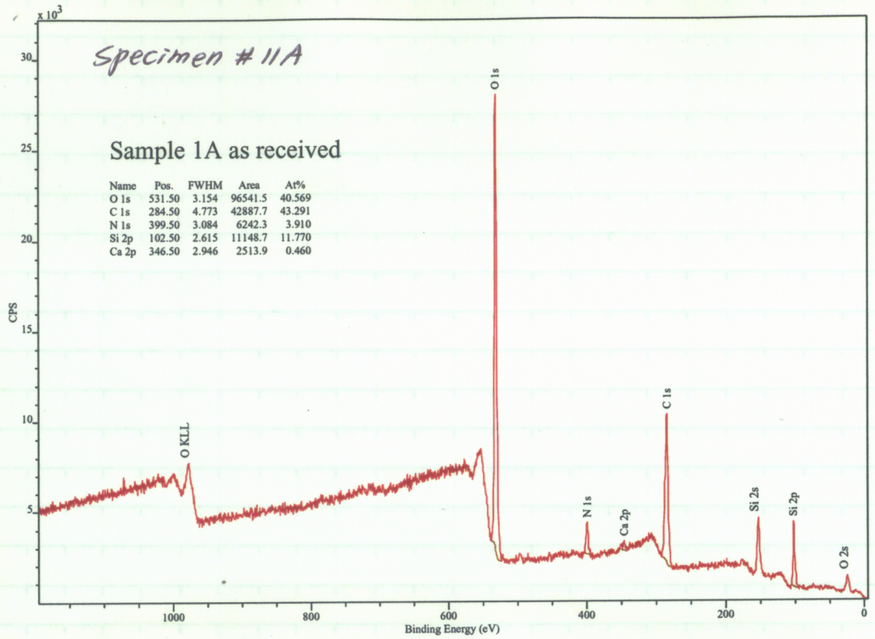
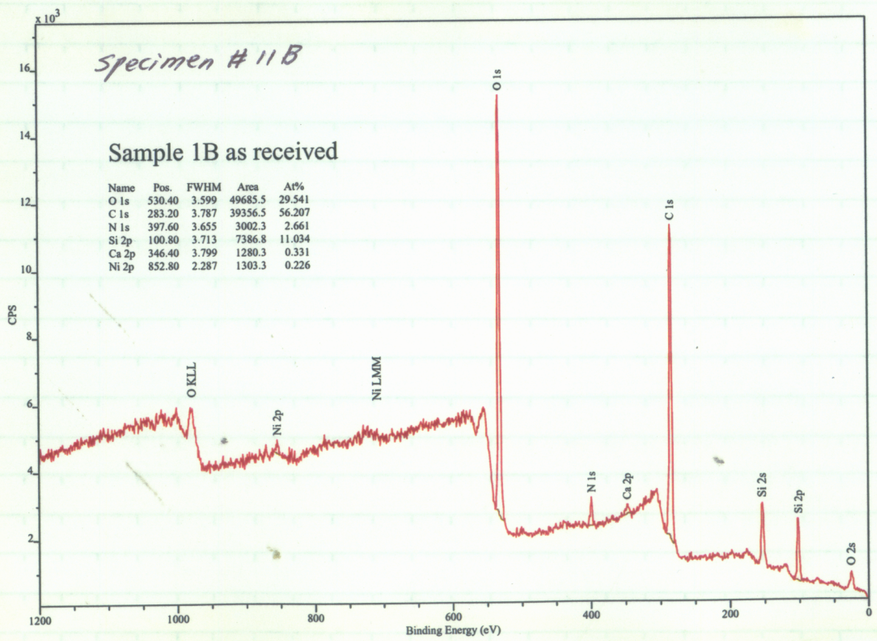
1. Wide-energy survey of the as-received surfaces





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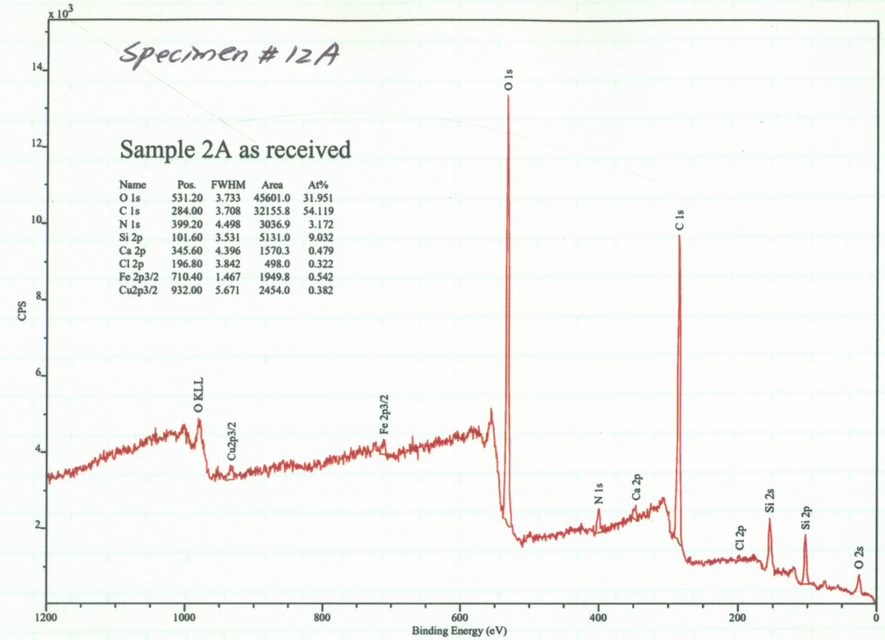

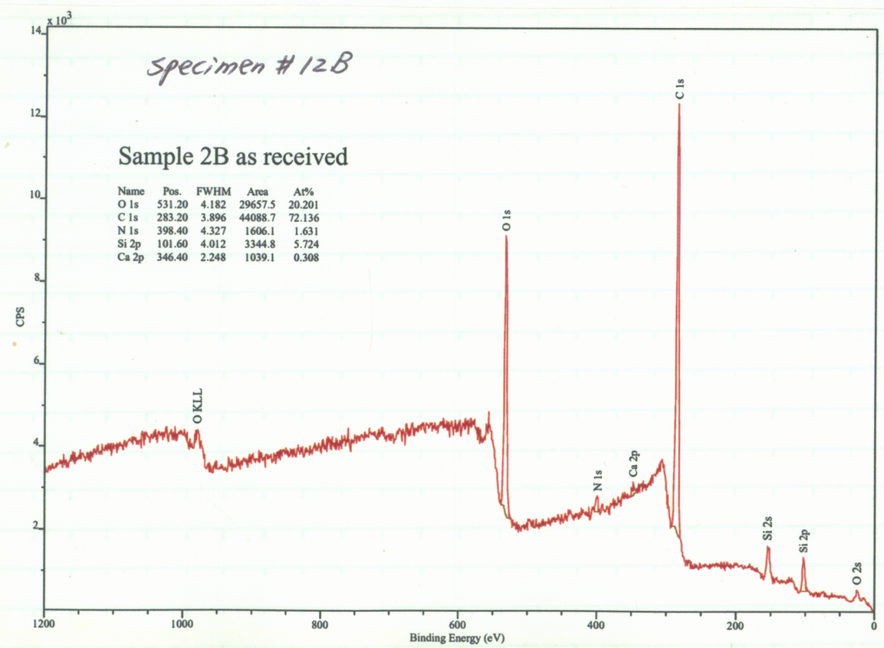

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