

Appendix F4

SEM/EDS Data for Test #4 Day-30 Steel Coupons

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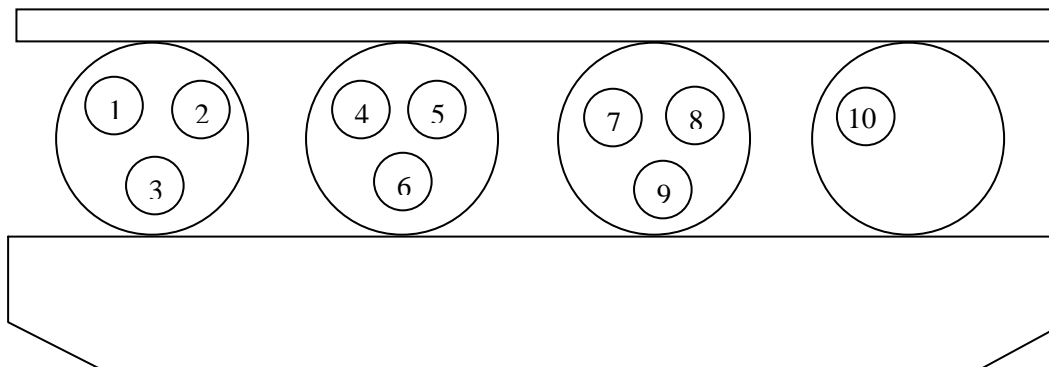
This appendix shows the SEM/EDS results for the metal steel coupons under two categories: (1) unsubmerged and (2) submerged. Unsubmerged refers to coupons held in the test tank gas space above the water level of the solution during ICET tests. Unsubmerged coupons were contacted by the solution only during the 4-hour spraying period at the initial date of the test. In addition, the surface of the unsubmerged coupons may also be affected by the moisture in the gas space during the test. Submerged refers to the coupons that were under the solution during the test.

The coupon samples were collected on June 23, 2005 (the date Test #4 was shut down) and examined by SEM/EDS on June 29, 2005. The steel coupon samples were dried in air before coating with Au/Pd for SEM examination. SEM results present the surface condition of the steel coupons. In addition, EDS results provide a semi-quantitative elemental analysis of the coupon surface and the corrosion products.

Transcribed Laboratory Log

Laboratory session from June 29, 2005.
SEM Test #4 Day-30 Steel Coupons.

- | | | | |
|-------------------|-------------------------|-------------------|---------------|
| 1. Unsubmerged Al | 3. Sus. Cu | 5. Sus. Gal-Steel | 7. Sus. Steel |
| 2. Submerged Al | 4. Sub. Cu | 6. Sub. Gal Steel | 8. Sub. Steel |
| 9. Sediment | 10. Powder on sub. Rack | | |



Unsubmerged Steel Coupons

Image:	T4D30SteelSusp020	100 ×	SEM image	Figure F4-1
	T4D30SteelSusp021	500 ×	SEM image	Figure F4-2
	T4D30SteelSusp022	1800 ×	SEM annotated image	Figure F4-3
EDS:	T4D30SteelSusp12		Particles shown in 022	Figure F4-4
	T4D30SteelSusp13		Surface shown in 022	Figure F4-5

Submerged Steel Coupons

Image:	T4D30SteelSubm023	100 ×	SEM image	Figure F4-6
	T4D30SteelSubm024	500 ×	SEM image	Figure F4-7
	T4D30SteelSubm025	1500 ×	SEM annotated image	Figure F4-8
EDS:	T4D30SteelSubm14		Particles shown in 025	Figure F4-9
	T4D30SteelSubm15		Surface shown in 025	Figure F4-10

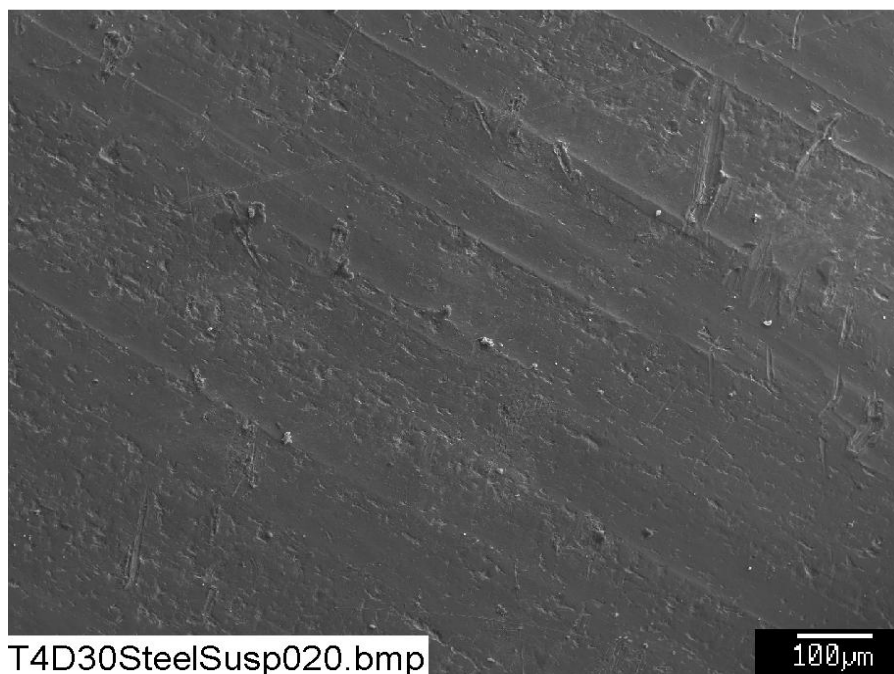


Figure F4-1: SEM image magnified 100 times for a Test #4 Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp020.bmp)

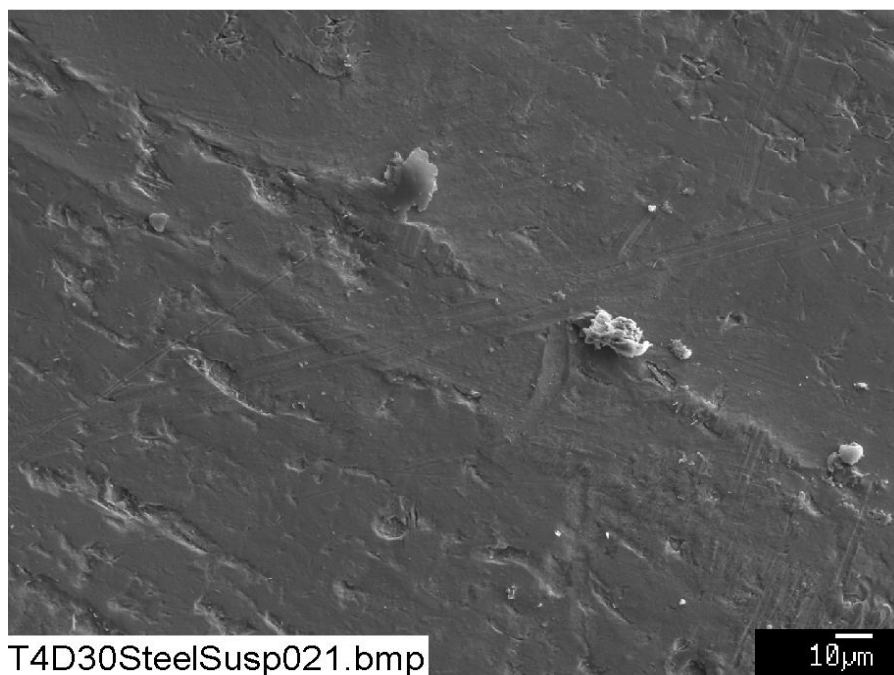


Figure F4-2: SEM image magnified 500 times for a Test #4 Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp021.bmp)

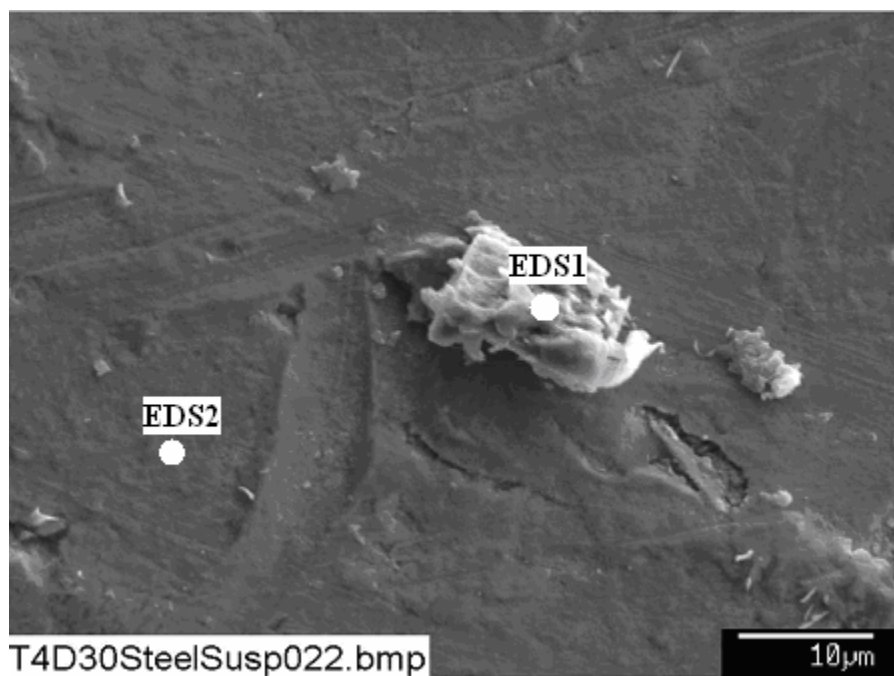


Figure F4-3: Annotated SEM image magnified 1800 times for a Test #4 Day-30 unsubmerged uncoated steel coupon sample. (T4D30SteelSusp022.bmp)

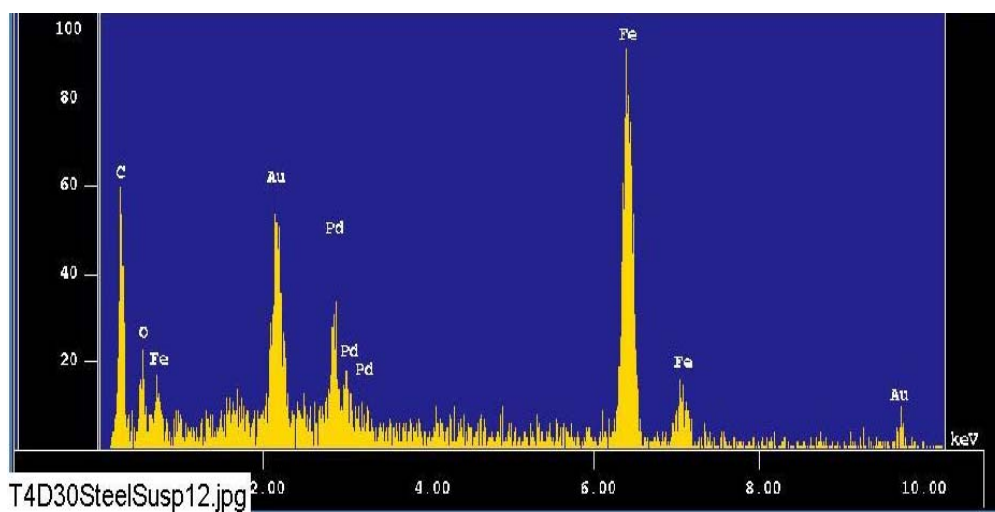


Figure F4-4: EDS counting spectrum for the white deposits (EDS1) on the coupon surface shown in Figure F4-3. (T4D30SteelSusp12.jpg)

The results from the chemical composition analysis for T4D30SteelSusp12.jpg are given in Table F4-1.

Table F4-1. Chemical Compositions for T4D30SteelSusp12.jpg, Figure F4-4.

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Group       : NRC
Sample      : T4D30 ID# : 12
Comment     : Particle on suspended steel
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
               Live Time  : 60.000 sec   Aperture #   : 2
               Acc. Volt   : 15.0 KV      Probe Current : 1.064E-09 A
               Stage Point : X=26.283 Y=57.998 Z=10.786
               Acq. Date   : Wed Jun 29 14:48:17 2005

```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
C K	Normal	0.09- 0.46	7.3229	0.0002	359 /	2
O K	Normal	0.25- 0.77	5.2438	0.0007	173 /	21
Fe K	Normal	6.00- 7.44	57.5951	0.0015	1453 /	1

Chi_square = 3.0082

Element	Mass%	Atomic%	ZAF	Z	A	F
C	23.723	54.7789	2.8221	0.9173	3.0766	1.0000
O	5.935	10.2882	0.9859	0.8744	1.1282	0.9995
Fe	70.342	34.9330	1.0639	1.0675	0.9967	1.0000

Total 100.000 100.0000
Normalization factor = 1.1480

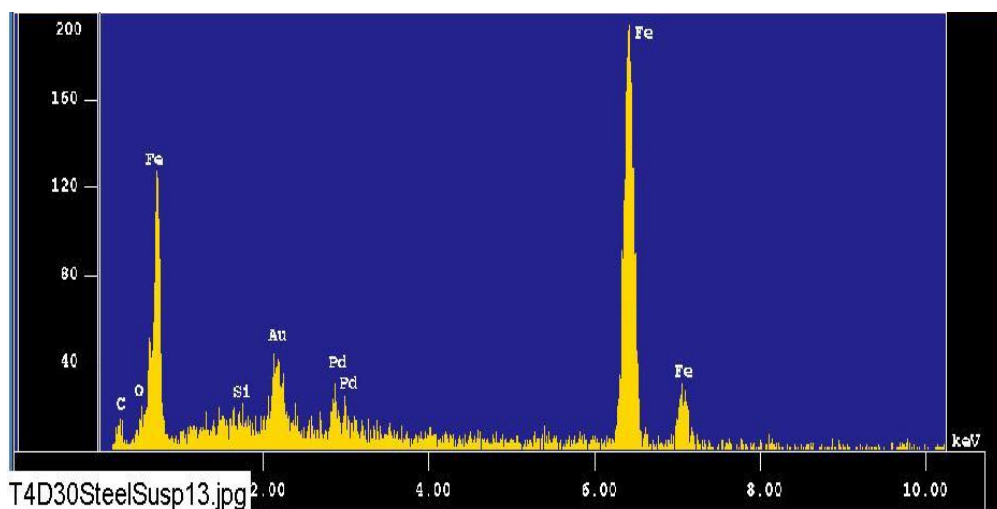


Figure F4-5: EDS counting spectrum for the flat coupon surface (EDS2) shown in Figure F4-3. (T4D30SteelSusp13.jpg)

The results from the chemical composition analysis for T4D30SteelSusp13.jpg are given in Table F4-2.

Table F4-2. Chemical Compositions for T4D30SteelSusp13.jpg, Figure F4-5.

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Group       : NRC
Sample      : T4D30 ID# : 13
Comment     : Surface of suspended steel
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
               Live Time  : 60.000 sec   Aperture # : 2
               Acc. Volt   : 15.0 KV      Probe Current : 1.065E-09 A
               Stage Point : X=26.283 Y=57.998 Z=10.786
               Acq. Date   : Wed Jun 29 14:52:52 2005

```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
Fe K	Normal	6.00- 7.44	131.0725	0.0022	3310 /	4
C K	Normal	0.09- 0.46	1.8367	0.0001	90 /	4
Si K	Normal	1.50- 2.07	0.0575	0.0003	7 /	14
O K	Normal	0.25- 0.77	2.0509	0.0010	68 /	17

Chi_square = 0.9378

Element	Mass%	Atomic%	ZAF	Z	A	F
Fe	94.919	80.9892	1.0113	1.0118	0.9995	1.0000
C	3.993	15.8420	3.0361	0.8799	3.4506	1.0000
Si	0.057	0.0961	1.3759	0.8380	1.6420	0.9999
O	1.032	3.0728	0.7025	0.8384	0.8387	0.9991

Total 100.000 100.0000

Normalization factor = 0.7161

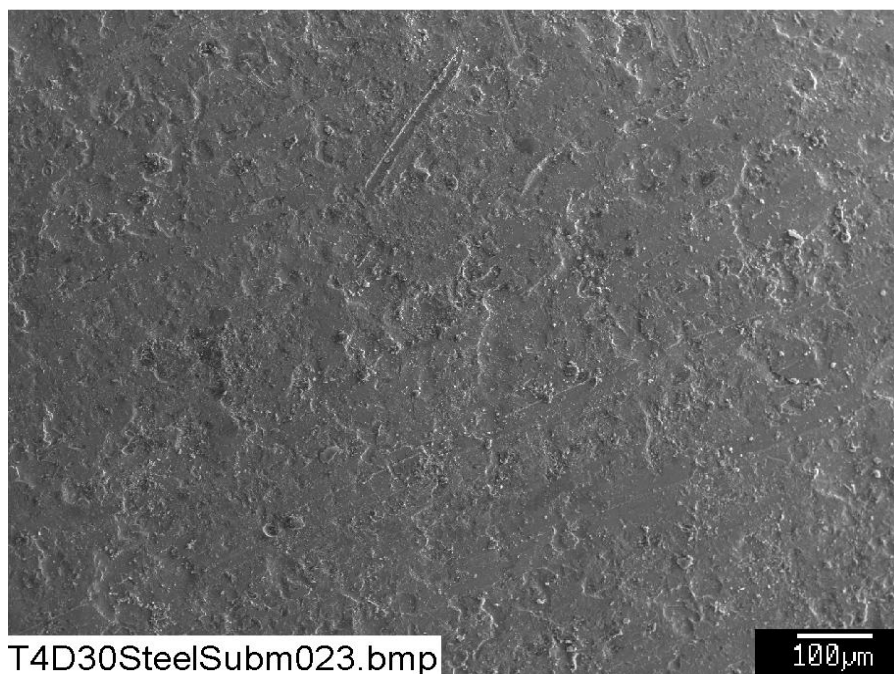


Figure F4-6: SEM image magnified 100 times for a Test #4 Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm023.bmp)

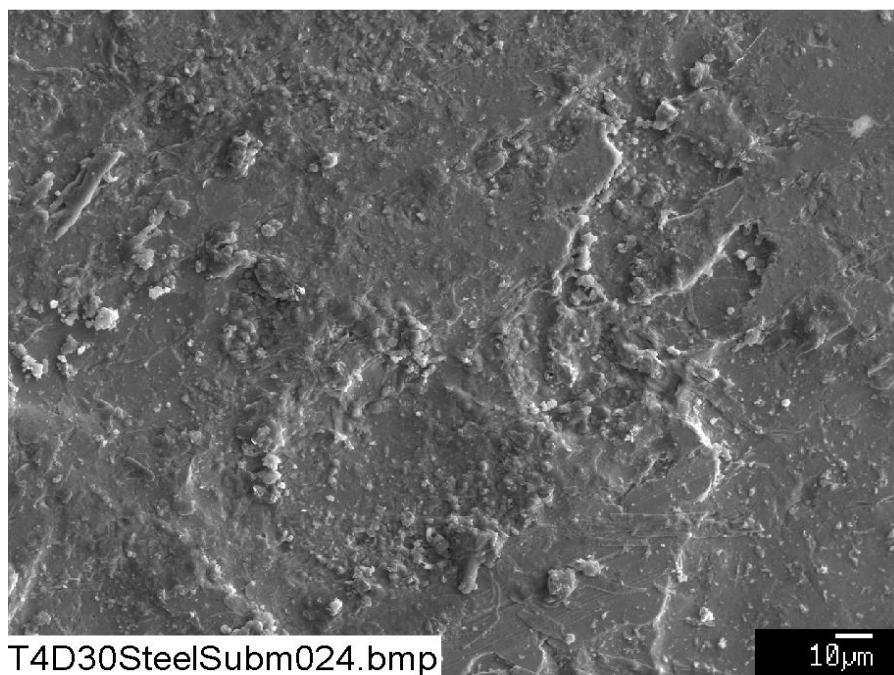


Figure F4-7: SEM image magnified 500 times for a Test #4 Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm024.bmp)

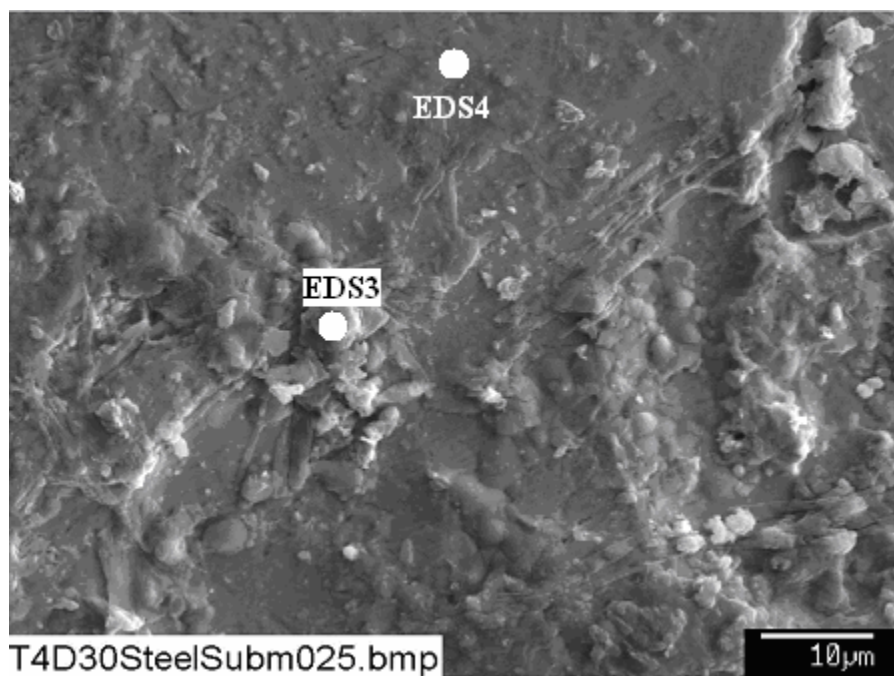


Figure F4-8: Annotated SEM image magnified 1500 times for a Test #4 Day-30 submerged uncoated steel coupon sample. (T4D30SteelSubm025.bmp)

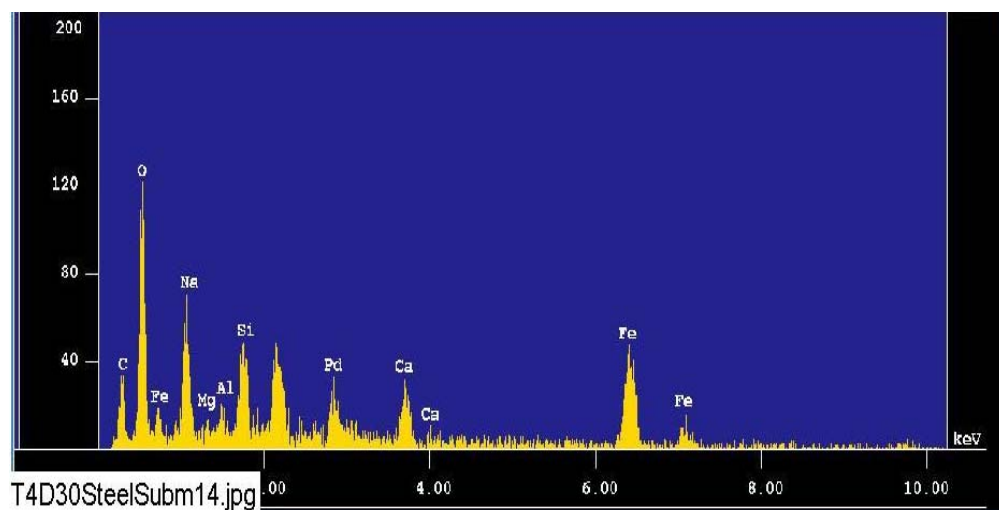


Figure F4-9: EDS counting spectrum for the deposit (EDS3) shown in Figure F4-8. (T4D30SteelSubm14.jpg)

The results from the chemical composition analysis for T4D30SteelSubm14.jpg are given in Table F4-3.

Table F4-3. Chemical Compositions for T4D30SteelSubm14.jpg, Figure F4-9.

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Group       : NRC
Sample      : T4D30 ID# : 14
Comment     : Particles on submerged steel
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
              Live Time  : 60.000 sec   Aperture #   : 2
              Acc. Volt   : 15.0 KV      Probe Current : 1.063E-09 A
              Stage Point : X=12.785 Y=58.790 Z=10.786
              Acq. Date   : Wed Jun 29 15:05:38 2005
  
```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
C K	Normal	0.09- 0.46	3.5591	0.0002	174 /	8
O K	Normal	0.25- 0.77	30.6742	0.0015	1013 /	13
Na K	Normal	0.81- 1.27	5.0811	0.0006	482 /	13
Al K	Normal	1.26- 1.78	0.7478	0.0003	102 /	22
Si K	Normal	1.50- 2.07	2.0211	0.0004	258 /	13
Ca K	Normal	3.40- 4.30	3.3136	0.0032	206 /	4
Fe K	Normal	6.00- 7.44	29.5118	0.0012	744 /	3
Mg K	Normal	0.97- 1.57	0.0852	0.0001	12 /	10

Chi_square = 2.0712

Element	Mass%	Atomic%	ZAF	Z	A	F
C	12.691	23.9254	3.2608	0.9777	3.3354	1.0000
O	31.600	44.7225	0.9420	0.9322	1.0108	0.9998
Na	11.110	10.9423	1.9994	0.9834	2.0328	1.0002
Al	1.177	0.9881	1.4397	0.9465	1.5219	0.9994
Si	2.951	2.3790	1.3351	0.9351	1.4279	0.9999
Ca	3.349	1.8922	0.9243	0.9416	0.9890	0.9926
Fe	36.931	14.9736	1.1443	1.1473	0.9974	1.0000
Mg	0.190	0.1769	2.0386	0.9256	2.2032	0.9997

Total 100.000 100.0000
Normalization factor = 1.0936

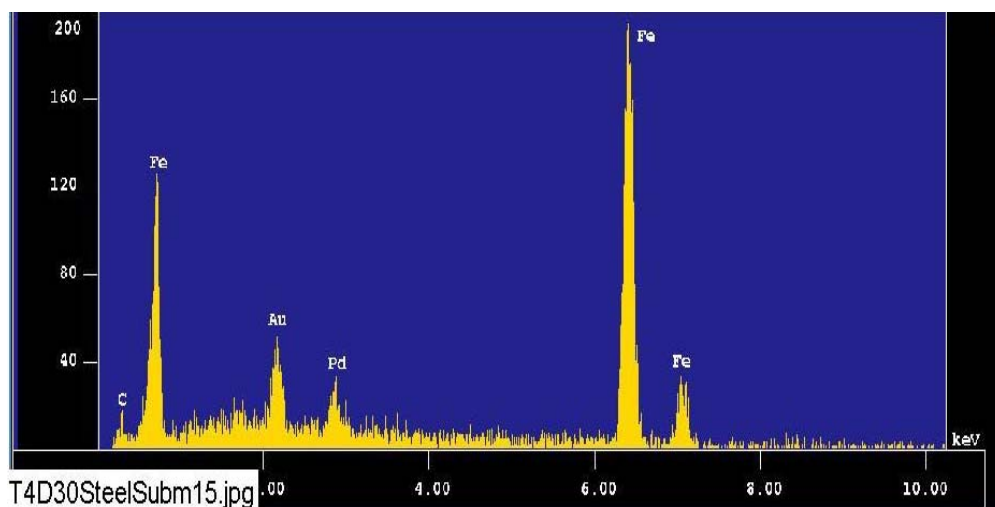


Figure F4-10: EDS counting spectrum for the flat coupon surface (EDS4) shown in Figure F4-8. (T4D30SteelSubm15.jpg)

The results from the chemical composition analysis for T4D30SteelSubm15.jpg are given in Table F4-4.

Table F4-4. Chemical Compositions for T4D30SteelSubm15.jpg, Figure F4-10.

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```

Group       : NRC
Sample      : T4D30 ID# : 15
Comment     : Surface of submerged steel
Condition   : Full Scale : 20KeV(10eV/ch,2Kch)
               Live Time  : 60.000 sec      Aperture #    : 2
               Acc. Volt   : 15.0 KV         Probe Current  : 1.064E-09 A
               Stage Point : X=12.785 Y=58.790 Z=10.786
               Acq. Date   : Wed Jun 29 15:10:11 2005

```

Element	Mode	ROI (KeV)	K-ratio(%)	+/-	Net/Background	
Fe K	Normal	6.00- 7.44	134.9460	0.0022	3404 /	3
C K	Normal	0.09- 0.46	1.2783	0.0001	63 /	4

Chi_square = 1.2118

Element	Mass%	Atomic%	ZAF	Z	A	F
Fe	97.224	88.2786	1.0059	1.0062	0.9997	1.0000
C	2.776	11.7214	3.0324	0.8759	3.4621	1.0000

Total 100.000 100.0000
Normalization factor = 0.7162