## Appendix C2

# SEM/EDS Data for Unused and Test-2 Day-30 Copper Coupons

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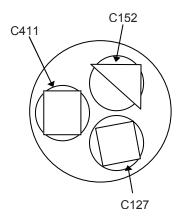
This appendix shows SEM/EDS results for the metal copper coupons in three different catagories: (1) unused; (2) suspended or unsubmerged; and (3) submerged. *Unused* refers to the coupon condition before being subjected to ICET test conditions. *Suspended* or *unsubmerged* refers to coupons located above the water level of the solution during ICET tests. Suspended coupons contacted the solution only during the 4-hour spray period at the start of the test. In addition, the surface of the suspended coupons may also be affected by moisture in the test chamber gas space. *Submerged* refers to the coupons that were immersed in the test solution for the entire test.

The coupon samples were collected on March 7, 2005 (the date Test #2 was shut down) and examined by SEM/EDS on April 4, 2005. The copper coupon samples were dried in air before being coated with carbon for SEM examination. SEM results present the surface condition of the copper coupon. In addition, EDS results provide a semiquantitative elemental analysis of the coupon surface and the corrosion products that are present.

#### **Transcribed Laboratory Log**

#### Laboratory session from April 4, 2005

#### Copper Samples



Conditions: 15-kV, 1-nA beam current, Aperture = 2

Metal	Sample	Test
Copper:	C411	Unused C411
	C152	Test #2 Unsubmerged C152
	C127	Test #2 Submerged C127

Note: All samples were carbon coated.

#### Sample C411 Copper Blank (unused)

Image:	C411052	$40 \times$	SEI	Figure C2-1
	C411053	150×	SEI	Figure C2-2
	C411054	1000×	SEI	Figure C2-3

#### Sample C152 Test 2 Unsubmerged Copper

Image:	C152055	$40 \times$	SEI	Figure C2-4
	C152056	150×	SEI	Figure C2-5
	C152057	1000×	SEI	Figure C2-6
	C152058	1000×	BEI	Figure C2-7
EDS:	CEDS30		Dark spot on T2D30 unsubmerged Cu	Figure C2-8

sample

### Sample C127 Test 2 Submerged Copper

Image:	C127059	$40 \times$	SEI	Figure C2-9
	C127060	150×	SEI	Figure C2-10
	C127061	150×	BEI	Figure C2-11
	C127062	1000×	SEI	Figure C2-12
EDS:	CEDS31		Dark spot on T2D30 submerged Cu	Figure C2-13
	CEDS32		Light spot on T2D30 submerged Cu	Figure C2-14
	CEDS33		Lathlike crystals on T2D30 submerged Cu	Figure C2-15

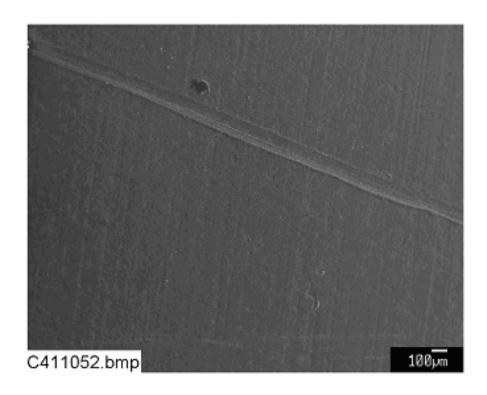


Figure C2-1. SEM image for an unused copper coupon (C411052).

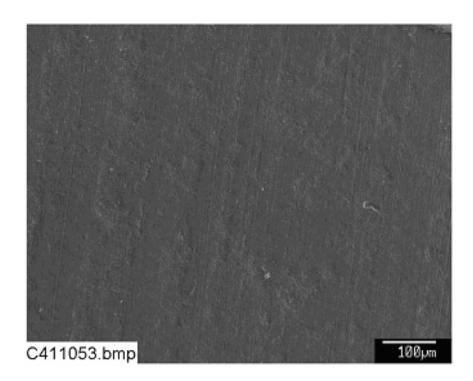


Figure C2-2. SEM image for an unused copper coupon (C411053).

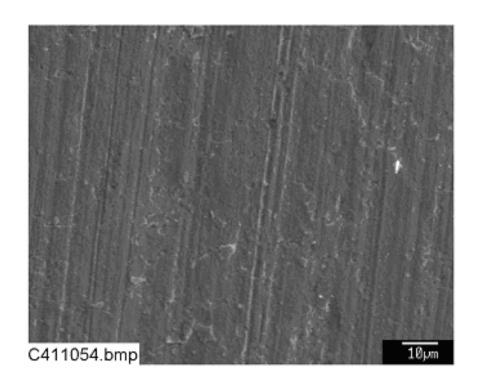


Figure C2-3. SEM image for an unused copper coupon (C411054).

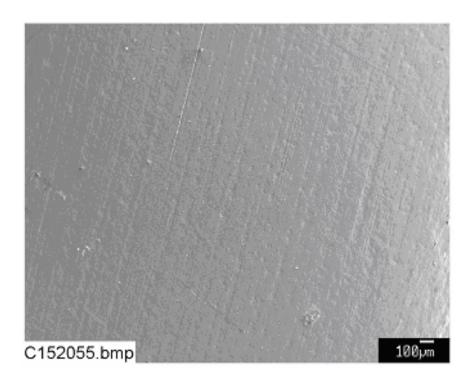


Figure C2-4. SEM image for a Test-2 Day-30 unsubmerged copper coupon (C152055).

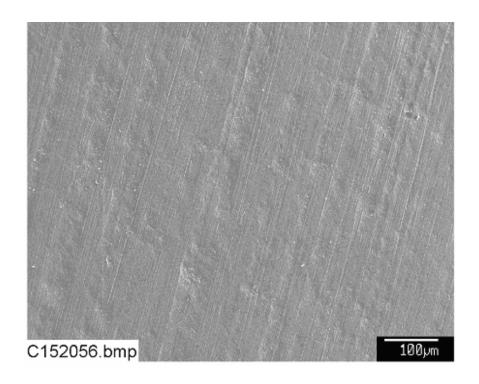


Figure C2-5. SEM image for a Test-2 Day-30 unsubmerged copper coupon (C152056).

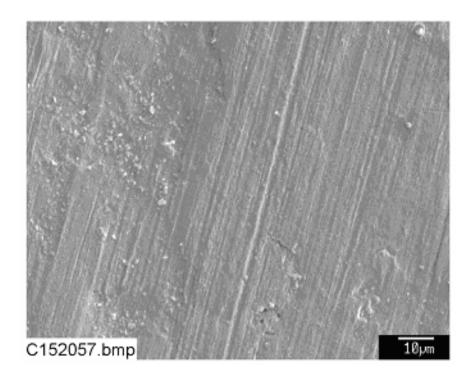


Figure C2-6. SEM image for a Test-2 Day-30 unsubmerged copper coupon (C152057).

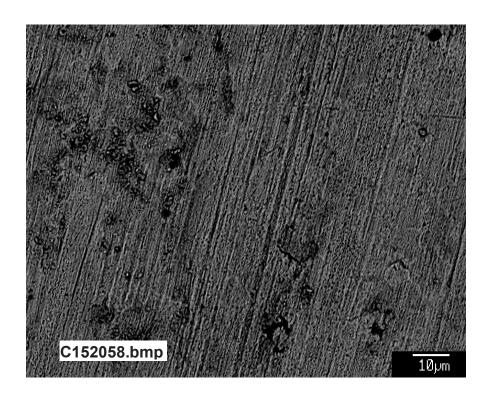


Figure C2-7. Backscattered SEM image for a Test-2 Day-30 unsubmerged copper coupon (C152058).

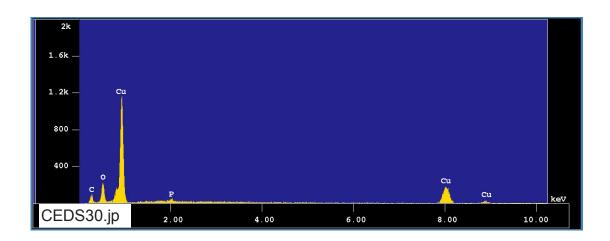


Figure C2-8. EDS counting spectrum for one of the dark spots shown in Figure C2-7 (CEDS30).

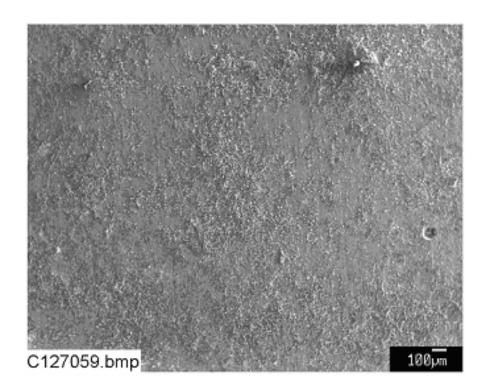


Figure C2-9. SEM image for a Test-2 Day-30 submerged copper coupon (C127059).

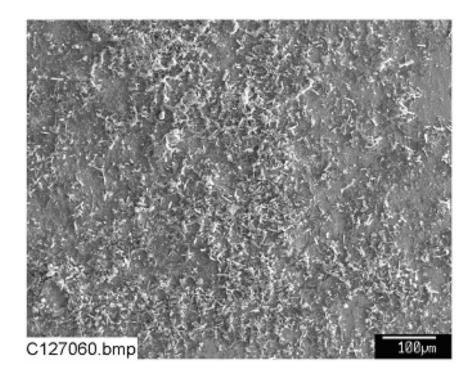


Figure C2-10. SEM image for a Test-2 Day-30 submerged copper coupon (C127060).

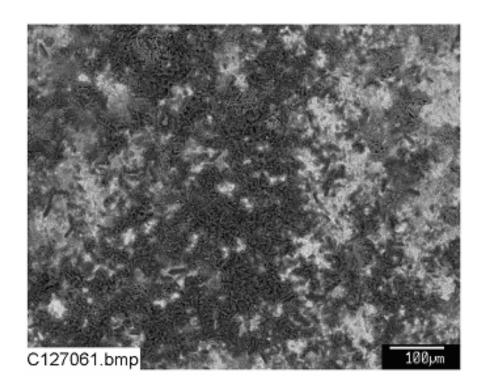


Figure C2-11. Backscattered SEM image for a Test-2 Day-30 submerged copper coupon (C127061).

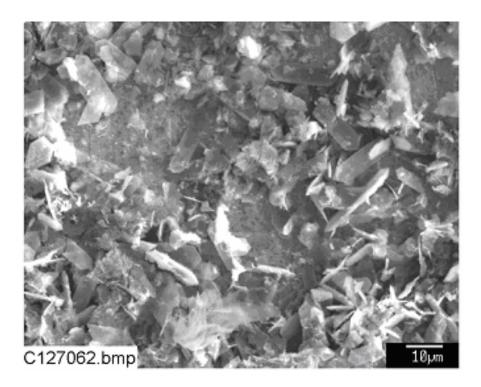


Figure C2-12. SEM image for a Test-2 Day-30 submerged copper coupon (C127062).

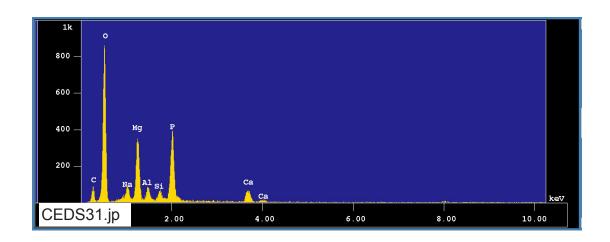


Figure C2-13. EDS counting spectrum for the dark patches shown in Figure C2-11 (CEDS31).

The results from the chemical composition analysis for CEDS31 are given in Table C2-1.

#### Table C2-1. The Chemical Composition for CEDS31 (Figure C2-13)

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```
Group
          : NRC
Sample
         : Metals ID# : 31
Comment : Dark spot on T2D30 submged Cu
Condition: Full Scale: 20KeV(10eV/ch, 2Kch)
            Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 5
                                       Probe Current : 5.546E-10 A
            Stage Point : X=42.970 Y=70.696 Z=11.000
            Acq. Date : Mon Apr 4 13:48:30 2005
Element
          Mode
                   ROI (KeV)
                              K-ratio(%) +/- Net/Background
  OK
         Normal 0.25-0.77
                               38.8311 0.0031
                               38.8311 0.0001
1.2974 0.0009 484 /
4.9621 0.0004 2689 /
                                                     5041 /
                                                            52
 Na K
         Normal 0.81- 1.27
                                                              136
 Mg K
         Normal 0.97-1.57
         Normal
 Al K
                  1.19- 1.83 0.7808 0.0003
1.50- 2.05 0.9519 0.0003
                                                    421 /
                                                               79
 Si K
          Normal
                                                    478 /
                                                              116
  PK
          Normal
                   1.75- 2.38 11.7319 0.0033
                                                    3637 /
                                                               37
      Normal
                  3.39-4.30 3.3210 0.0012 811 /
 Ca K
                                                                5
                           Chi square = 45.5460
Element Mass%
                Atomic% ZAF
                                   \mathbf{Z}
         59.978 72.8049 0.9785 0.9908 0.9877 1.0000
     0
    Na
         2.826 2.3873 1.3800 1.0457 1.3215 0.9986
         11.431 9.1315 1.4595 0.9845 1.4842 0.9988
    Mq
         1.789 1.2876 1.4515 1.0179 1.4298 0.9974
1.887 1.3046 1.2557 0.9952 1.2690 0.9943
    Al
    Si
         16.716 10.4809 0.9027 1.1946 0.7558 0.9997
    P
        5.372 2.6032 1.0249 1.0161 1.0086 1.0000
------
```

Total 100.000 100.0000

Normalization factor = 1.5785

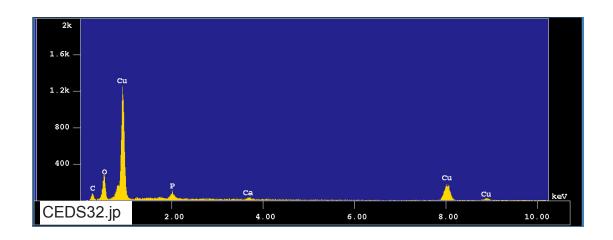


Figure C2-14. EDS counting spectrum for the light patches shown in Figure C2-11 (CEDS32).

The results from the chemical composition analysis for CEDS32 are given in Table C2-2.

#### Table C2-2. The Chemical Composition for CEDS32 (Figure C2-14).

Apr 4 14:01 2005 /tmp/eds\_pout.log Page 1

```
Group
            : NRC
Sample
            : Metals ID# : 32
Comment : Light spot on T2D30 submged Cu
Condition: Full Scale: 20KeV(10eV/ch, 2Kch)
                Live Time : 60.000 sec Aperture # : 1
Acc. Volt : 15.0 KV Probe Current : 5.518E-10 A
Stage Point : X=42.970 Y=70.696 Z=11.000
                Acq. Date : Mon Apr 4 13:58:47 2005
Element Mode ROI(KeV) K-ratio(%) +/- Net/Background
O K Normal 0.25- 0.77 15.4064 0.0023 1990 / 92
P K Normal 1.75- 2.38 2.0745 0.0020 640 / 26

      Normal
      1.75- 2.38
      2.0745
      0.0020
      640 / 26

      Normal
      3.39- 4.30
      1.1910
      0.0011
      289 / 14

      Normal
      7.63- 9.27
      61.8655
      0.0086
      3043 / 4

  Ca K
 Cu K
------
                                      Chi square = 7.3941
Element Mass%
                     Atomic%
                                    ZAF
                                               \mathbf{Z}
                                                         A
       0
          16.042 42.1700 0.8549 0.8436 1.0136 0.9998
       P
           2.528 3.4327 1.0005 1.0112 0.9894 1.0000
     Ca
            1.293 1.3565 0.8912 0.8542 1.0459 0.9975
      Cu 80.137 53.0408 1.0634 1.0641 0.9994 1.0000
Total 100.000 100.0000
Normalization factor = 1.2181
```

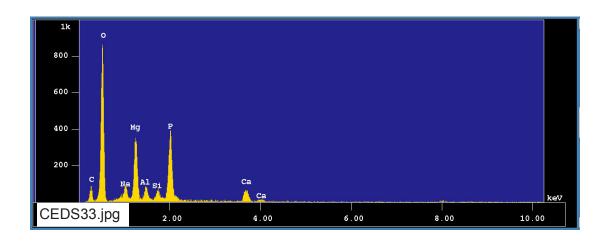


Figure C2-15. EDS counting spectrum for the lathlike crystals shown in Figure C2-12 (CEDS33).