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Scientific Notebook # 086

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CNWRA
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SOLUTION : EPR = 0.5 m H_2SO_4 + 0.01 m KSCN

ITIZED : 750/15 CPP = CPP

ION ANNEALED = SA EPR = SINGLE LOOP EPR

SOLUTION / TEMP	TEST / SCAN RATE	RESULTS	NOTEBOOK / PAGE
25% H_2SO_4 / 100°C	CPP / 1.67 mV/s	634 μA / 18.5 μA	086 / 10-11
"	CPP / 1.67 mV/s	1040 μA / 0 μA	086 / 12-13
20% H_2SO_4 / 100°C	EPR / 0.17 mV/s	-143 mV - 736 μA	086 / 16-17
"	EPR / 0.17 mV/s	-136 mV - 686 μA	086 / 19
20% H_2SO_4 / 100°C	EPR / 1.67 mV/s	-112 mV - 6.4 μA	086 / 20
"	EPR / 1.67 mV/s	-26 mV - 8.76 μA	086 / 23 ←
"	EPR / 1.67 mV/s	NO PEAK	086 / 24
"	EPR / 1.67 mV/s	NO PEAK	086 / 25
20% H_2SO_4 + 40% 0.01 KSCN	CPP / 1.67 mV/s	NO PEAK / ATTACK	086 / 30
"	CPP / 1.67 mV/s	"	086 / 31
30% H_2SO_4 / 100°C	EPR / 1.67	-120 mV 468.3 μA / GB STAIN ETCH	086 / 32 ←
30% H_2SO_4 / 100°C	EPR / 1.67	-130 mV 301.8 μA / STAIN	086 / 33 ←
"	CPP / 1.67	-59 mV 293 μA / GB ETCH	086 / 34
"	CPP / 1.67 mV/s	-59 mV 286 μA / NO CORROSION	086 / 35
"	EPR / 0.17	-105 mV 558.5 μA / GB + STAIN	086 / 36
"	EPR / 0.17	-106 mV 470.7 μA / STAIN	086 / 37
10% H_2SO_4 / 95	EPR / 0.17	-172 mV 1014 μA / GB + STAIN	086 / 38
"	"	-186 mV 169.7 μA / STAIN	086 / 39
5% H_2SO_4 / 95	EPR / 0.17	-172 mV 2 μA / GB + STAIN	086 / 40
"	"	-188 mV 8 μA / GB + STAIN	086 / 43
1% H_2SO_4 / 95	"	-198 mV 832 μA / GB	086 / 42
1% H_2SO_4 / 95	"	-210 mV 531 μA / ~~GB~~ STAIN	~~[illegible]~~ 086 / 41
10% H_2SO_4 / 95	EPR / 0.17	-172 mV 162 μA / GB ATTACK	086 / 52
"	"	-182 mV 358 μA / SLIGHT GB ATTACK	
"	EPR / 0.17	-154 1.3 μA	086 / 53
"	EPR / 0.17	-164 3.78 μA GB ATTACK	086 / 54
10% H_2SO_4 / 95	CPP / 0.17	-67 TO -145 mV .43 TO 152 μA	086 / 57-60
50% H_2SO_4 / 30	CPP 1.67	NO CORR	032 / 141
50% H_2SO_4 / 100	CPP 1.67	LIGHT ETCH	032
20% H_2SO_4 / 100°C	CPP 1.67	542 μA / 21.4 μA / GB	032
"	CPP 1.67	623 μA NO ATTACK	032
	SL EPR	SENS & NO SENS 304 G108 SOLN — SENS 825	
	DL EPR	SENS & NO SENS 304 SENS 825	
		~~CONC H_2SO_4~~ G108 CONC H_2SO_4	

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Page 1

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N. Sridhar
2/14/97

11/25/94

INITIAL ENTRY FOR THE SENSITIZATION STUDIES

Purpose: To measure the effects of intergranular precipitation on corrosion of austenitic alloys. This will involve evaluating existing test methods and develop new tests, if necessary.

Approach: Based on literature search, the electrochemical potentiokinetic reactivation (EPR) test method will be examined first. Such a test method already exists as ASTM G-108 test method for type 304L stainless steel. This test on sensitized type 304L SS has been performed to verify that the procedures used were adequate. The results have been documented in Laboratory Notebook 032, pages 70 through 77. The test method with an environment identical to the ASTM G-108 test (0.5 M Sulfuric acid + 0.01M KSCN at 30 C) was used for heat treated alloy 825 (750 C/200 hours). However no sensitization was detected unlike the case of Type 304L SS (Notebook 032, pages 83 - 84. The test environment and temperature were varied without any benefit (Notebook 032, pages 85 through 100). Then a pitting type environment was chosen (NaCl) and potentiostatic, and potentiodynamic experiments were conducted. following these tests, which were not discriminatory, higher sulfuric acid concentrations were tried with modifications to the EPR procedure. These are identified in Notebook 032, pages 134 through 160. The current series of tests reported in this notebook are continuations of these tests reported in notebook 032.

Procedure: The procedures are described in Notebook 032, pages 134 through 135. Other individual test procedures are given in appropriate sections in the current notebook.

Equipment: These are given in Notebook 032, page 134.

N. Sridhar
11/25/94

N. Sridhar
11/20/94

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N. Sridhar
11/30/94

7/28/93 PROCEEDURE FOR HEAT TREATING SAMPLES FOR EPR TEST

This proceedure is intended to produce sensitized samples for Electrochemical ~~Repa~~ fk 7/28 Potentiokinetic Reactivation (EPR) Tests.

~~2 samples will~~ fk 7/28
The samples are alloy 825, HT# HH4371FC. They have previously been heat treated at 750°F for 15 and 200 hours after being solution annealed. No sensitization, or at least very little, has occured so the samples are viewed to be as solution annealed that have been annealed to remove point defects that occur durring the quenching of solution annealed material. One sample that was used was solution annealed only.

The heat treatment is at 750°C for either 15, 20 or 210 hours. The extra time is needed for samples previously treated that lack point defects, carbide formation kinetics will be different.

The treatments are preformed in a Lindberg Model 51828 oven. A calibrated thermocouple, Omega Model HH22 calibrated 7 July 93 next due 7 Jan 94 (#ID T-94140), is used to check temperature. The tip of the thermocouple is placed inside the hole of one of the specimen.

Sample preperation schedual as follows:

~~In~~ fk 7/28

Sample	IN	Out	Result
CNWRA#1	7/21 5:00PM	7/22 8:00AM	750°C/15hrs
CNWRA#2	7/26 3:30 PM	7/27 11:30AM	750°C/20hrs
CNWRA#3	7/26 3:30 PM	7/27 11:30AM	"
CNWRA#4	7/26 3:30 PM	8/ ~~8-12~~ 7/28 9:30AM	750°C/210hrs
CNWRA#5	7/26 3:30 PM	8/ 9:30AM	"

Frank E. Kulik 7/28/93

7/28/93 Procedure for Cyclic Polarization (PCP)

The objective of this section is to find and alter environment that a double loop EPR technique will work for alloy 825.

The test will be controlled by M342C software. The data will also be saved using this software. The controlled potantiostat will be an EG&G M173, #62101 calibrated 19 May 93 Next due 19 May 94.

The samples that will be used will be alloy 825, HT# HH4371FC, of varing heat treatments.

The that will be used is a typical G-5 cell. A platinum ~~E~~ 7/28 counter electrode and a SCE reference electrode (Fisher 13-620-51) are used in both high and low temperature experiments. An aerater tube is used for systems that need to be deaerated. For low temperature tests a short lugen probe is used and a thermometer is used outside in the water bath. For high temperature experiments a long lugen probe and thermometer are used inside.

The sample is Not mounted and at least one side is polished to 1 micron. All remaining sides are polished to 600 grit. Specimen sides are first polished to 600 grit by grinding flat with 240 grit followed by 320 and 400 grit. The side or sides that are going to be polished to 1μ first have thier edges beveled to reduce grabing of sample and wear on cloth. Start off with 3μ diamond lapping film until all 600 grit scratches are gone. Rinse off specimen with D.I. water and polish with 3μ diamond paste. Rinse off and ultrasonicly clean for five minutes with detergent. ~~If~~ 7/28 The ultrasonic cleaning should be done before ~~the~~ 7/28 3μ film as well. Rinse and polish with 1μ diamond paste until 3μ scratches are gone.

7/28/93 PCP cont.

Solutions should be deaerated for 1 hour with Argon with solution at experiment temperature and sample not immersed. Deaeration should continue through the experiment. For high temperature applications a condenser tube should be used in conjunction.

After test is complete, sample is removed and observed.

Before using for another test repolish sample to specified finishes just peior to test. One micron side should be started at 3μ lapping film.

Frank E. Kalil 7/28/93

N. Sridhar
11/30/94

7/28/93 ~~825 CP12~~ pk 7/28 825 CP13

Sample: CNWRA #1 SEE page 7
1 side 1μ 4 sides 600 grit
Solution: 25% H_2SO_4 by weight

750 ml H_2O (18 MΩ)
T = 100°C Hg Therm 0323008
Calibrated Next due
Saved as: 825CP13
Reference: SN0165415
Technique: PCP page 8
Ecorr: -203

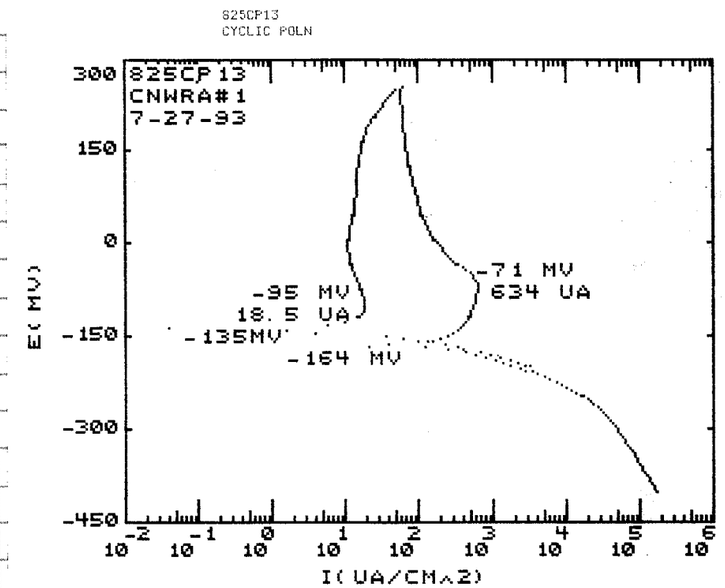
Appearance: grain boundary attack

Remarks: will try solutions annealed

Graph and Parameters on page 11

N. Sridhar
11/30/94

7/28/93 825CP13



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP13
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-400
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	5
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-203
MV/PT	4
DATA MAX	634
DATA MIN	-175600
ABS MIN	.04
ABS MAX	175600

Paul E. Kelly Jr.
7/28/93

LEGEND

825CP13
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: 25% H2SO4 BY WEIGHT
TEMP 100C
7/27/93

7/28/93 825CP14

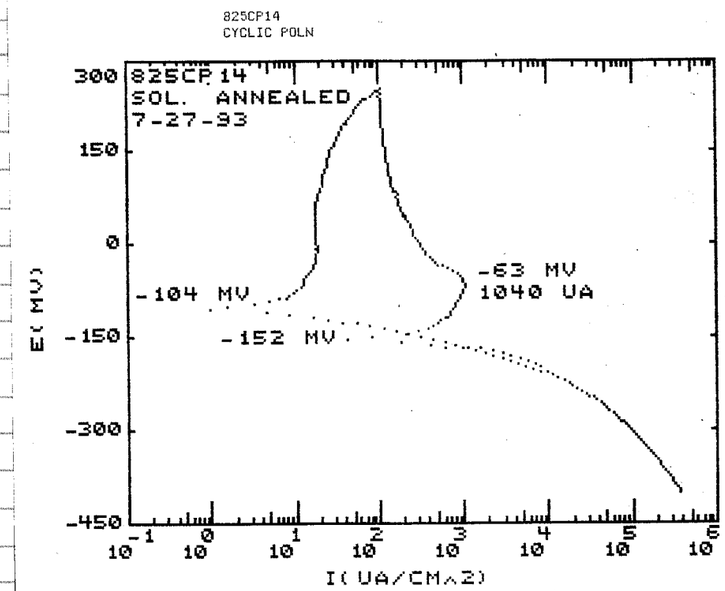
Sample: Solution Annealed
1 side / 1 micron 4 sides 600 grit
Solution: Reused from 825CP13 page 10
T = 100 °C Hg Therm 0323 008 same as 825CP13 pag 10
Saved as: 825CP14
Technique: PCP page 8
Reference: SN 0165 4/5
E_{corr} = -200 mV

Appearance: No attack.

Parameters and graph on page 13.

7/28/93 825CP14

Space reserved for graph and parameters of 825CP14. Printer down.



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP14
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-300
CONDITION T(S)	60
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	2.5
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-200
MV/PT	4
DATA MAX	1040
DATA MIN	-376800
ABS MIN	-968
ABS MAX	376800

LEGEND

825CP14
SOLUTION ANNEALED
1SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825CP13
TEMP 100C
7/27/93

Paul E. Huff 7/28/93

(PSEPR)

7/28/93 Procedure for Single Loop Electrochemical Potentio~~dynam~~ Kinetic Reactivation

The purpose of this test is to determine the degree of sensitization electrochemically using a proven method, but not necessarily the same environment used for 304 or 316 SS.

The samples will be various heat treatments of alloy 825, HT # HH4371FC. These samples are not mounted and have a threaded hole to convert a brass rod for electrical contact.

The EPR test will be controled by M342C software with the controled potentiostat being an EG&G M173, calibrated 19 May 93 next due 19 May 94 ID#62101. Data is saved by M342C software and placed on disk.

A typical G-5 cell is used. A platinum counter electrode, aerated tube (for deaerated solutions) with water trap, and ~~Re~~ PK 7/28 SCE Reference electrode (Fisher 13-620-51) are used for high and low temp. experiments. For high temperature experiments a long lugen probe and Hg therm are used in the cell. For low temp. a short lugen probe is used and a Hg therm is used in a water bath.

The sample has at least 1 side at a 1 micron finish and the remaining sides being 600 grit. All sides should be flat. This is obtained by starting with 240 ground planar. The following steps are 320, ~~400~~, and 600. 1 micron sides are started by ultrasonicly ~~cleaned~~ PK 7/28 cleaning in detergent for 5 minutes. The edges are then PK 7/28 beveled for the 1 micron side. The side is then lapped with 3μ diamond lapping film followed by 3μ paste. The sample is ultrasonicly cleaned. The sample is then polished with 1μ paste. The sample is then ultrasonicly cleaned.

7/28/93 PSEPR

The solution should be deaerated at experiment temp. for one hour. The deaeration should be continued through the test.

After the test the sample is removed and observed under the microscope to see microstructure to find the "G" value. The computer will calculate Q/A. The quantitative value of the sensitization is calculated by $P_A = (Q/A) / (5.1 \times 10^{-3} [\exp(.35G)])$

The above is probably not going to be used since this is a qualitative project.

For sample that are being reused: All non 1μ sides should be polished to 600 grit and 1μ sides start with 3μ lapping film.

Josh E. Kolb Jr. 7/28/93

7/28/93 825ER1

Sample: CNWRA #1 see page 7
1 side 1μ 4 - 600 grit

Solution: 20% H_2SO_4
108.5 ml 96% H_2SO_4 Lot # 925400
800 ml H_2O (18 MΩ)
T = 100 °C Hg Therm. 0323008 same as 825CP13 pk page 10

Saved as: 825ER1

Technique: PSEPR page 14.

Reference: SN 0165415

Ecorr: -97 mV

Q/A: N/A

Appearance: Didn't look.

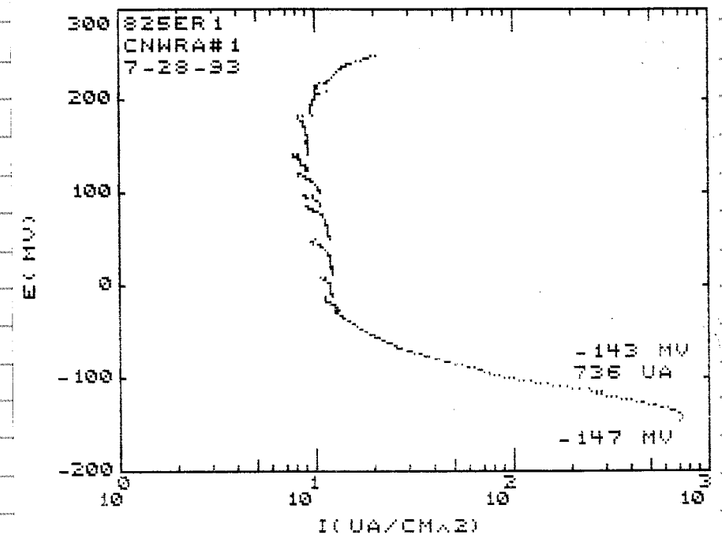
Remarks: Didn't go far enough to get Q/A will try again.

Parameters and graph on page 17

7/28/93 825ER1

This space reserved for graph and parameters for 825ER1. Printer down.

[signature] 7/28/93



RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER1
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

ECORR	-97
MV/PT	1
DATA MAX	738
DATA MIN	7.7
ABS MIN	7.7
ABS MAX	738

RESULTS

START E(MV)	-147
END E(MV)	250
MILLICOULOMBS/CM^2	146.65

LEGEND

825ER1
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: 20% H2SO4 BY WEIGHT
TEMP 100C
7/28/93

7/28/93 825 ER2

Sample: ~~CNWRA#2~~ CNWRA #1 (750°C/15 HOURS)
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Reference: SN 0165415
Saved as: 825ER2
Technique: PSEPR page 14

Ecorr: -169 mV

Q/A: ~~32338.5 mC/cm²~~ fk 7/28 329.63 mC/cm²

Appearance: Grain boundary corrosion

~~Part~~ fk 7/28
Printer out no graph or parameters [signature] 7/28/93

DISREGARD TEST DATA
8/6/93

RUN PARAMETERS

TECHNIQUE	REACTIVAT
ORIGINAL NAME	825ER2
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-250 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	2.5

DATA SCALE

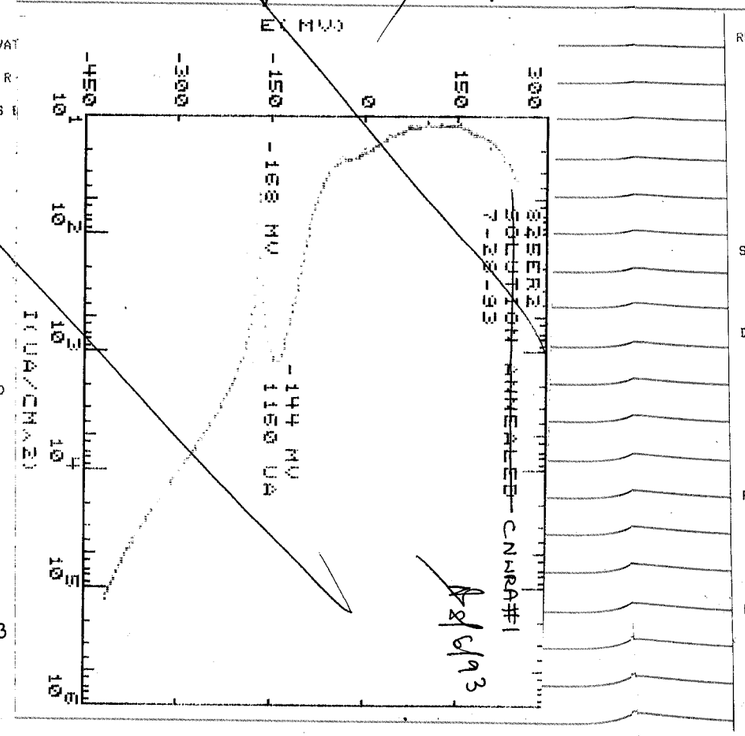
ECORR	-169
MV/PT	2
DATA MAX	1240
DATA MIN	-125600
ABS MIN	11
ABS MAX	125600

RESULTS

START E(MV)	-168
END E(MV)	250
MILLICOULOMBS/CM^2	329.63

LEGEND

825ER2
~~SOLUTION ANNEALED~~ CNWRA#1 8/6/93
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/28/93



7/28/93 825ER3

Sample: ~~Solution Annealed~~ CNWRA #1 [750°C/15 HOURS]
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Reference: SN 0165415
Technique: PSEPR page 14
Save as: 825 EP3

Ecorr: -204 mV

Q/A: ~~28.54 mC/cm²~~ fk 7/28 191.27 mC/cm²

Appearance: step structure

Remarks: rate too slow.

Printer out - no graph or parameters [signature] 7/28/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER3
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-250 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

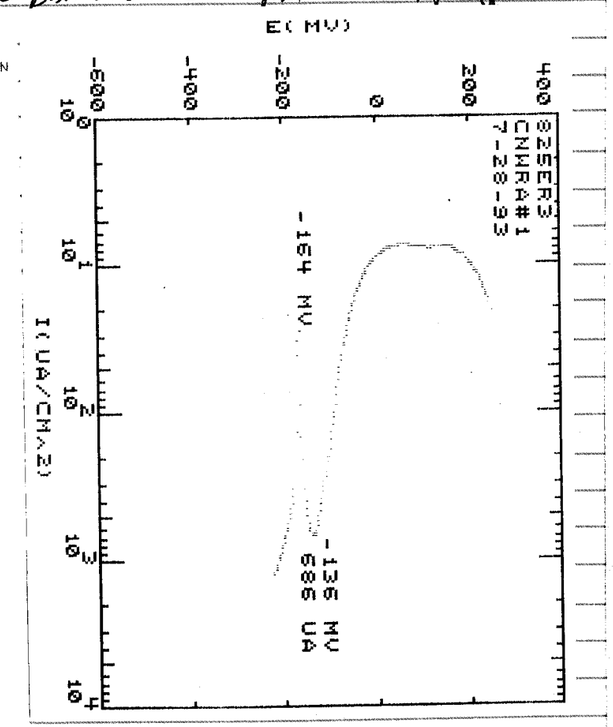
ECORR	-204
MV/PT	2
DATA MAX	686
DATA MIN	-1262
ABS MIN	7.26
ABS MAX	1262

RESULTS

START E(MV)	-164
END E(MV)	250
MILLICOULOMBS/CM^2	191.27

LEGEND

825ER2
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/28/93



7/28/93 ~~825ER2~~ fk 7/28 825ER4

Sample CNWRA#1 see page 7

1 side 1μ 4 sides 600 grit

Solution reused from 825ER1

T = 100°C Hg Therm 0323008 same as 825CP13 page 10

Technique: PSEPR page 14

Saved As: ~~825ER3~~ 825ER4

Reference: SN0165415

E_{corr} = -131 mV

Q/A = ~~-420.57 mC/cm²~~ fk 7/28/93 1.91 mC/cm²

Appearance: Very slight boundry attack

No parameters or graph. printer down

[signature] 7/28/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER4
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-150 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

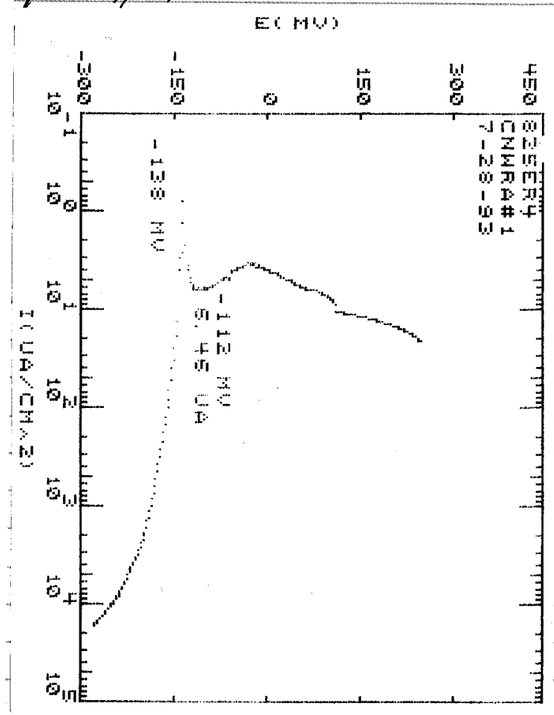
ECORR	-131
MV/PT	2
DATA MAX	21.48
DATA MIN	-16380
ABS MIN	.788
ABS MAX	16380

RESULTS

START E(MV)	-138
END E(MV)	250
MILLICOULOMBS/CM^2	1.91

LEGEND

825ER4
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: REUSED FROM 825ER1
TEMP 100C
7/28/93



7/28/93 825ER5

Sample: Solution Annealed

1 side 1μ 4 sides 600 grit

Solution: Reused from 825ER1

T = 100°C Hg Therm 0323008 same as ~~825ER1~~ 825CP13 fk 7/28 page 10

Technique: PSEPR page 14

Saved as 825ER5

Reference: SN0165415

E_{corr}: N/A

Q/A: N/A

Appearance: N/A

Remarks: one lead not hooked up - test invalid.

No parameters or graph; printer down

[signature] 7/28/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER5
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-150 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	2.5

DATA SCALE

ECORR	1006
MV/PT	2
DATA MAX	0
DATA MIN	0
ABS MIN	0
ABS MAX	0

LEGEND

825ER5
SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: REUSED FROM 825ER1
TEMP 100C
7/28/93

7/28/93 825ER6

Sample: Solution Annealed
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008 SAME AS 825CP13 page 10
Technique: PSEPR page 14
Reference: SN0165415
Saved As: 825ER6
Ecorr: 174 mV
Q/A: N/A
Appearance: slight facet corrosion

Remarks: I don't think test went far enough. didn't see a ~~rk~~ minimum open circuit potential.

~~Parameters and graph not available since printer out~~

[signature] 7/29/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER6
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-150 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

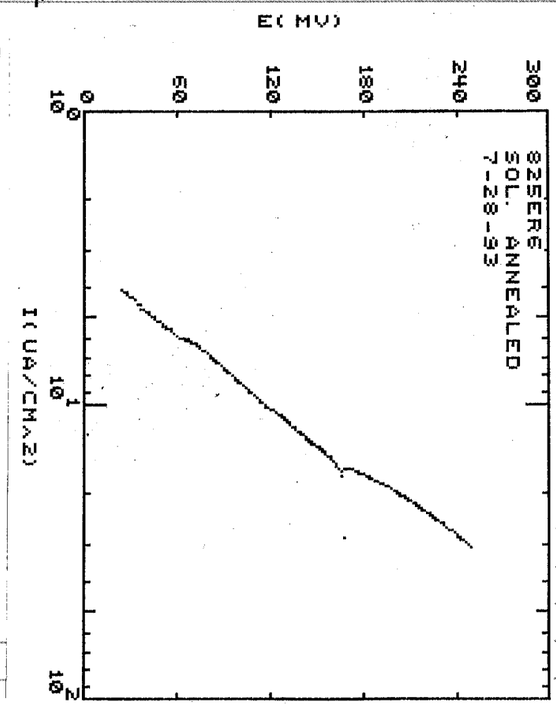
AREA(CMS^2)	2.5

DATA SCALE

ECORR	174
MV/PT	1
DATA MAX	30.48
DATA MIN	4.076
ABS MIN	4.076
ABS MAX	30.48

LEGEND

825ER6
SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/28/93



7/29/93 825ER7

Sample: Solution Annealed
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008 SAME AS 825CP13 page 10
Technique: PSEPR page 14
Saved As: 825ER7
Reference: SN0165415
Ecorr: -168 mV
Q/A: 1.79
Appearance: Did not look - left in and ran 825ER8 to get an idea of how curve looks for longer passivation time. It was not getting enough of a passive film in previous experiments.

~~Parameters and Graph not available; printer down.~~

[signature] 7/29/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER7
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-250 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	20 S

SAMPLE PARAMETERS

AREA(CMS^2)	2.5

DATA SCALE

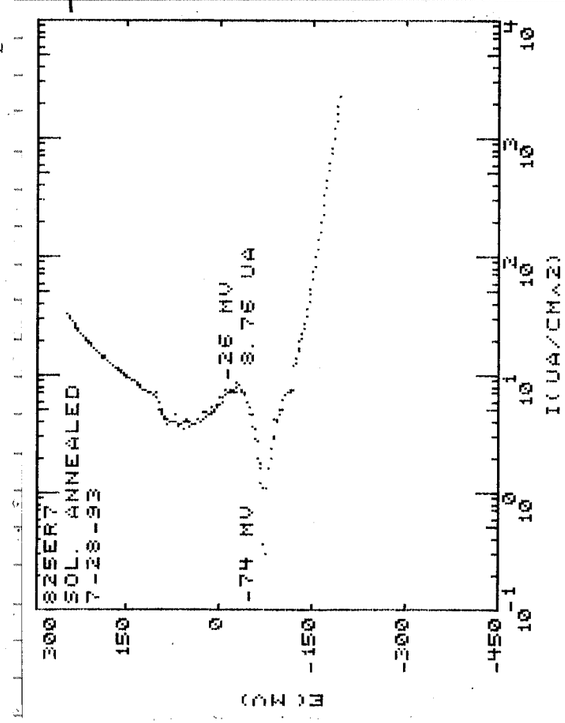
ECORR	-168
MV/PT	2
DATA MAX	33.92
DATA MIN	-2212
ABS MIN	.296
ABS MAX	2212

RESULTS

START E(MV)	-74
END E(MV)	250
MILLICOULOMBS/CM^2	1.79

LEGEND

825ER7
SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



7/29/93 825ER8

Sample: Solution annealed
~~ts~~ fk 7/29 Reused sample from 825ER7
Solution reused from 825ER1
T=100°C Hg Therm 0323008 same as 825CP13 pag 10
Technique: PSEPR page 14
Saved As: 825ER8
Reference: SN0165415
Ecorr: -125
Q/A: 0.78
Appearance: No attack

~~Parameters and Graph not available; No printer~~

[signature] 7/29/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER8
INITIAL E(MV)	300 VS R
PASSIVATION T(S)	300
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	2.5

DATA SCALE

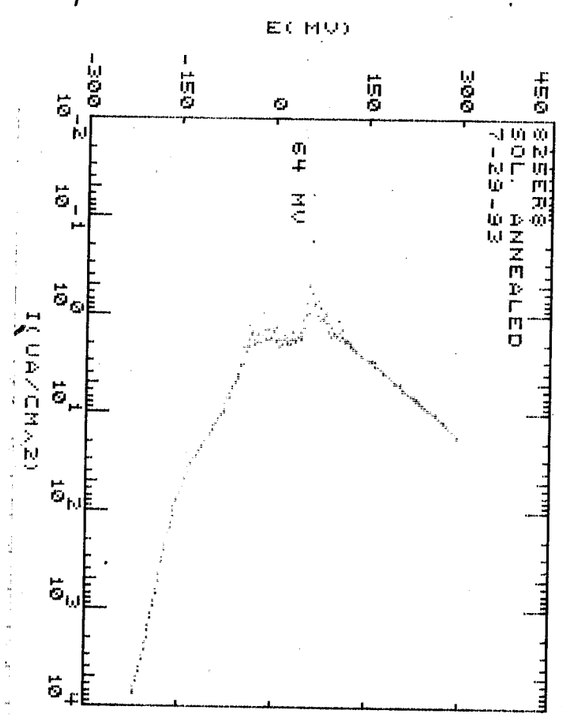
ECORR	-125
MV/PT	2
DATA MAX	17.48
DATA MIN	-7688
ABS MIN	.06
ABS MAX	7688

RESULTS

START E(MV)	64
END E(MV)	300
MILLICOULOMBS/CM^2	.78

LEGEND

825ER8
SOLUTION ANNEALED
REUSED SAMPLE FROM 825ER7
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



8 fk 7/29 7/29/93 825ER9

Sample: CNWRA#1 see page 7
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T=100°C Hg Therm 0323008
Technique: PSEPR page 14
Saved As 825ER9
Reference: SN0165415
Ecorr: -161 mV
Q/A: 0.87 mC/cm²
Appearance: Very slight grain boundry attack
Remarks: passive layer too thick

~~Parameters and Graph not available since printer out~~

[signature] 7/29/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER9
INITIAL E(MV)	300 VS R
PASSIVATION T(S)	300
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

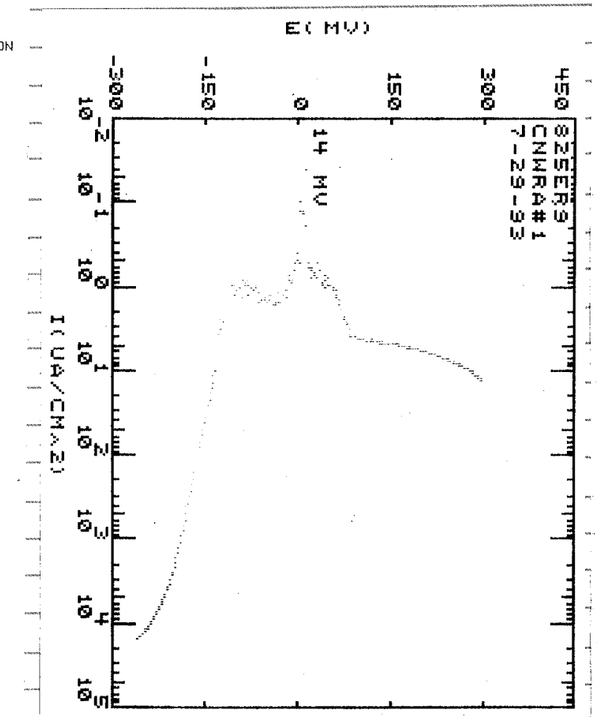
ECORR	-161
MV/PT	2
DATA MAX	13.08
DATA MIN	-15000
ABS MIN	.04
ABS MAX	15000

RESULTS

START E(MV)	14
END E(MV)	300
MILLICOULOMBS/CM^2	.87

LEGEND

825ER8
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



7/29/93 825ER10

Sample: CNWRA#1 see page 7
1 side 1μ 4 sides 600 grit
Solution: ~~Rat~~ fk 7/29 Reused 825ER1
T = 100°C Hg Therm 0323008 same as ~~825ER~~ fk 7/29
825CP13 page 10
Technique: PSEPR page 14
Saved As: 825ER10
Reference: SN0165415
Ecorr -165 mV
Q/A: 1.05 mC/cm²
Appearance: Didn't look

~~Parameters and Graph not available since printer out~~

[signature] 7/29/93 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER10
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	300
FINAL E(MV)	-150 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

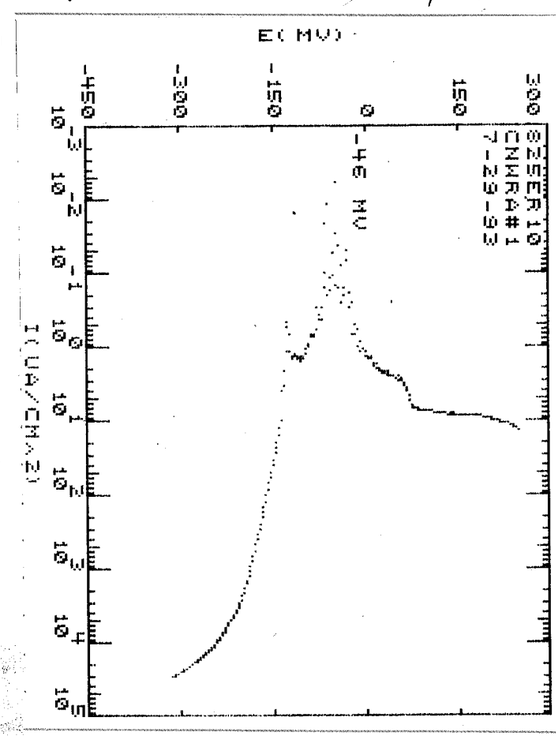
ECORR	-165
MV/PT	2
DATA MAX	13.42
DATA MIN	-30260
ABS MIN	.006
ABS MAX	30260

RESULTS

START E(MV)	-46
END E(MV)	250
MILLICOULOMBS/CM^2	1.05

LEGEND

825ER10
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



7/29/93 825ER11

Sample: CNWRA #1 see page 7
~~ts~~ fk 7/29 Reused sample from 825ER10
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Technique: PSEPR page 14
Saved As: 825ER11
Reference: SN0165415
Ecorr -154 mV
Q/A 1.07 mC/cm²
Appearance: very slight grainboundry corrosion

~~Parameters and graph not available since printer out~~

[signature] 7/29/93 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER11
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	210
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	20 S

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

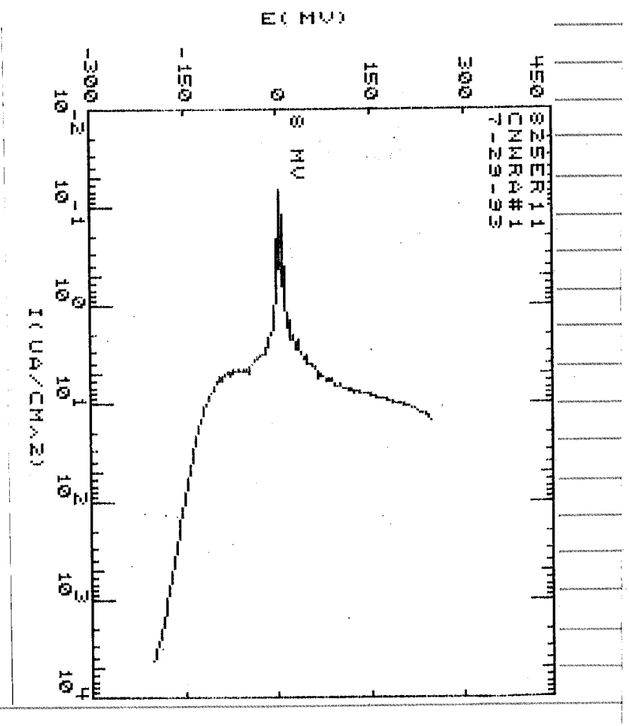
ECORR	-154
MV/PT	2
DATA MAX	14.82
DATA MIN	-4120
ABS MIN	.066
ABS MAX	4120

RESULTS

START E(MV)	8
END E(MV)	250
MILLICOULOMBS/CM^2	1.07

LEGEND

825ER11
CNWRA#1
REUSED FROM 825ER10
SOLUTION REUSED FROM 825ER1
TEMP 100
7/29/93



7/29/93 825ER12

Sample: CNWRA#1 see page 7
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008
Technique: PSEPR page 14
Saved As: 825ER12
Reference: SD0165415
Ecorr -171 mV
Q/A 1.65 mC/cm²
Appearance: No attack seen

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER12
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	165
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	20 S

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

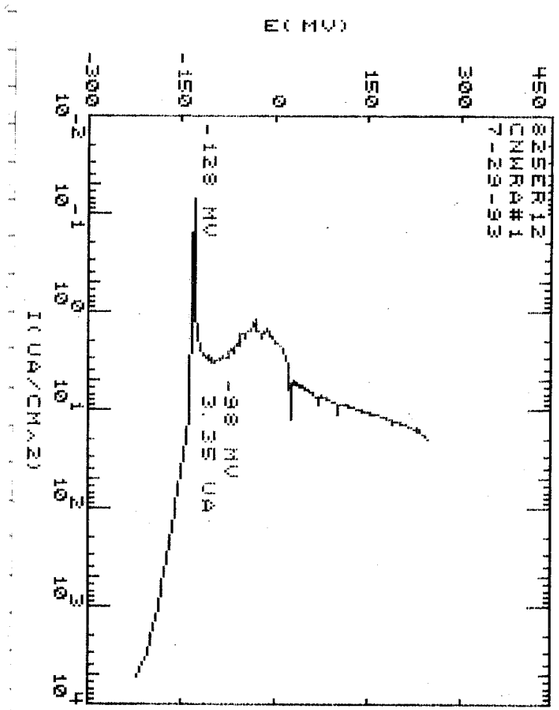
ECORR	-171
MV/PT	2
DATA MAX	20.2
DATA MIN	-5260
ABS MIN	.072
ABS MAX	5260

RESULTS

START E(MV)	-128
END E(MV)	250
MILLICOULOMBS/CM^2	1.65

LEGEND

825ER12
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



Paul E. Kilgo 7/29/93

7/29/93 825ER13

Sample: Solution Annealed
1 side 1μ 4 sides 600 grit
Solution: Reused from 825ER1
T = 100°C Hg Therm 0323008
Technique: PSEPR page 14
Saved As 825ER12
Reference: SD0165415
Ecorr -173 mV
Q/A 1.17
Appearance: No attack seen

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER13
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	165
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	20 S

SAMPLE PARAMETERS

AREA(CMS^2)	5

DATA SCALE

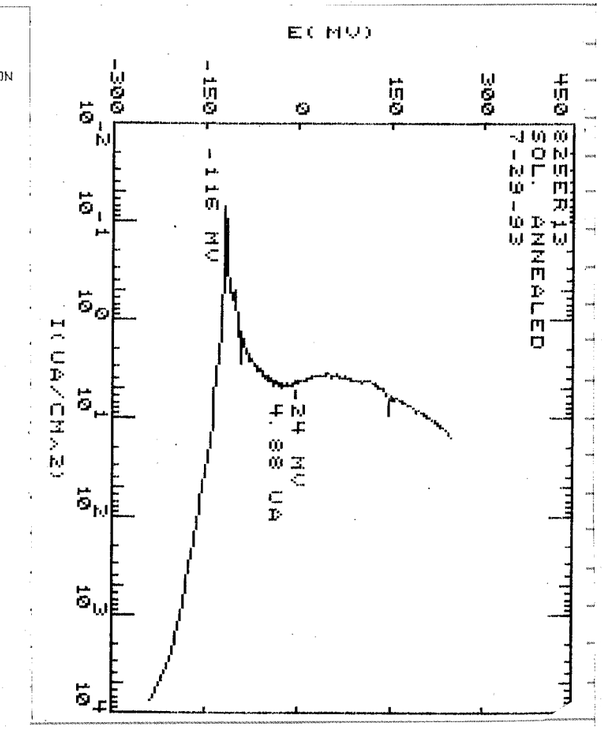
ECORR	-173
MV/PT	2
DATA MAX	15.4
DATA MIN	-7300
ABS MIN	.07
ABS MAX	7300

RESULTS

START E(MV)	-116
END E(MV)	250
MILLICOULOMBS/CM^2	1.17

LEGEND

825ER12
SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER1
TEMP 100C
7/29/93



Paul E. Kilgo 7/29/93

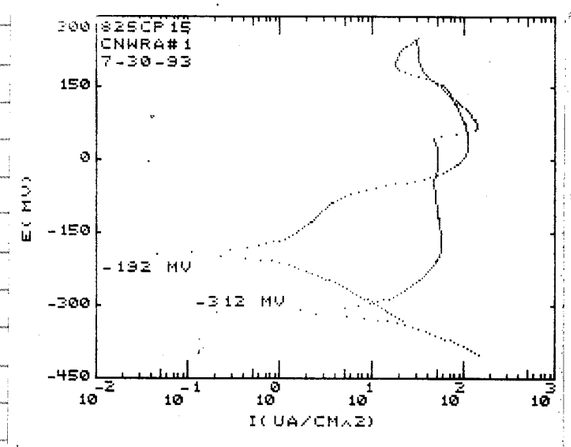
7/30/93 825CP15

Sample: CNWRA#1 see page 7
1 side 1μ 4 sides 600 grit
Solution: 20% by weight H_2SO_4 + .01M KSCN
119.5 ml ~~108.5 ml~~ fk 7/30 96% H_2SO_4 Lot # 925400
.9755 g KSCN Lot # 924916
~~800 ml~~ 7/30 880.5 ml H_2O
T = 40°C Hg Therm 0323004 Calibrated
2 Feb 93 Next due 2 Feb 94
Technique: PCP page 8
Saved as 825CP15
Reference: SN0165415
Ecorr: ~~302 mV~~ fk 7/30 -322 mV

Appearance: No attack

Parameters and Graph not available printer down

[signature] 7/30/93 8/13/93



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP15
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-400
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	11.8
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	[illegible]
MV/PT	[illegible]
DATA MAX	[illegible]9.7458
DATA MIN	-144.9153
ABS MIN	4.745763E-02
ABS MAX	144.9153

LEGEND

825CP15
CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: 20% H2SO4 + .01M KSCN
TEMP 40C
7/30/93

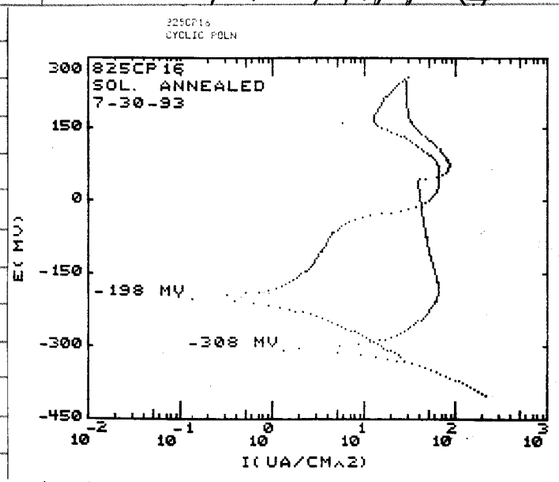
7/30/93 825CP16

Sample: Solution Annealed ~~so~~ fk 7/30/93
1 side 1μ 4 sides 600 grit
Solution: Reused from 825CP15
T = 40°C Hg Therm 0323004 same as 825CP15 page 30
Technique: PCP page 8
Saved as 825CP16
Reference SN0165415
Ecorr -326 mV

Appearance: No attack

Parameters and Graph not available, printer down

[signature] 7/30/93 8/13/93



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP16
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-400
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	10.668
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-326
MV/PT	4
DATA MAX	84.7394
DATA MIN	-220.285
ABS MIN	9.655043E-02
ABS MAX	220.285

LEGEND

825CP16
SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825CP15
TEMP 40C
7/30/93

7/30/93 825ER14

Sample: CNWRA #1 see page 7
1 side 1μ ~~4 sides~~ 5 sides fk 600 grit
Solution: 30% H_2SO_4
163 ml 96% H_2SO_4 Lot # 925400
700 ml 18MΩ H_2O
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Technique: PSEPR page 14
Saved As 825ER14
Reference: SN0165415
Ecorr:
Appearance: very visible grain boundry corrosion and slight grain etching.
Q/A = 21.16 mC/cm2
~~Parameters and Graphs not available; printer down.~~
Area = 11.809 cm²

[signature] 7/30/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER14
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	11.809

DATA SCALE

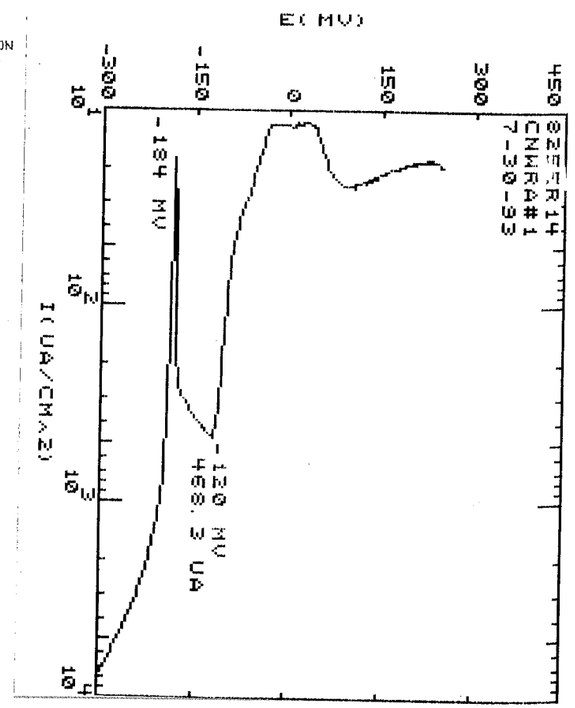
ECORR	-198
MV/PT	2
DATA MAX	468.2869
DATA MIN	-7392.667
ABS MIN	11.62673
ABS MAX	7392.667

RESULTS

START E(MV)	-184
END E(MV)	250
MILLICOULOMBS/CM^2	21.16

LEGEND

825ER14
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION: 30% H2SO4 BY WEIGHT
TEMP 100C
7/30/93



7/30/93 825ER15

Sample: Solution Annealed
1 side 1μ ~~5 sides~~ fk 7/30 5 sides 600 grit
Solution: Reused from 825ER14
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Technique: PSEPR page 14
Saved As 825ER15
Reference: SN0165415
Ecorr
Appearance: Stained.
Q/A = 8.78
~~Parameters and graph not available printer down~~
Area = 10.668 cm²

[signature] 7/30/93 [signature] 8/13/93

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER15
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	1.67
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	10.668

DATA SCALE

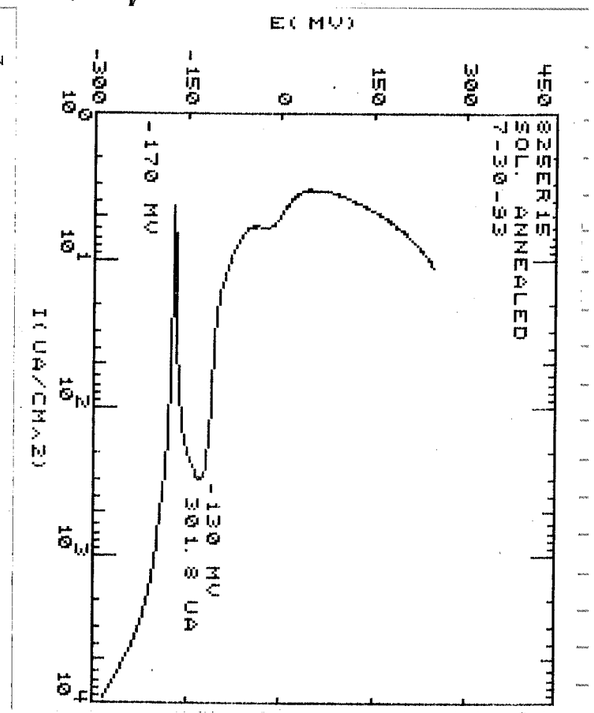
ECORR	-187
MV/PT	2
DATA MAX	301.8372
DATA MIN	-9289.464
ABS MIN	3.337083
ABS MAX	9289.464

RESULTS

START E(MV)	-170
END E(MV)	250
MILLICOULOMBS/CM^2	8.78

LEGEND

825ER15
SAMPLE: SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER14
TEMP 100C
7/30/93



7/30/93 825CP17

Sample: CNWRA#1 seepage 7
1 side 1μ 5 sides 600 grit
Solution Reused from 825ER14
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Technique: PCP page 8
Saved As 825CP17
Reference: SNO165415
Ecorr:
Appearance: Grain Boundary Corrosion.

Area = 11.809 cm²
~~Parameters and graph not available, printer down~~

7/30/93

7/30/93 825CP18

Sample: Solution annealed
1 side 1μ 5 sides 600 grit
Solution: Reused from 825ER14
T = 100°C Hg Therm 0323008 same as 825CP13 page 10
Technique: PCP page 8
Saved as 825CP18
Reference: SNO165415
Ecorr: -178 mV
Appearance: No large amount of corrosion visible.

Area = 10.668 cm²
~~Parameters and graph not available; printer out.~~

7/30/93

RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP17
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-400
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

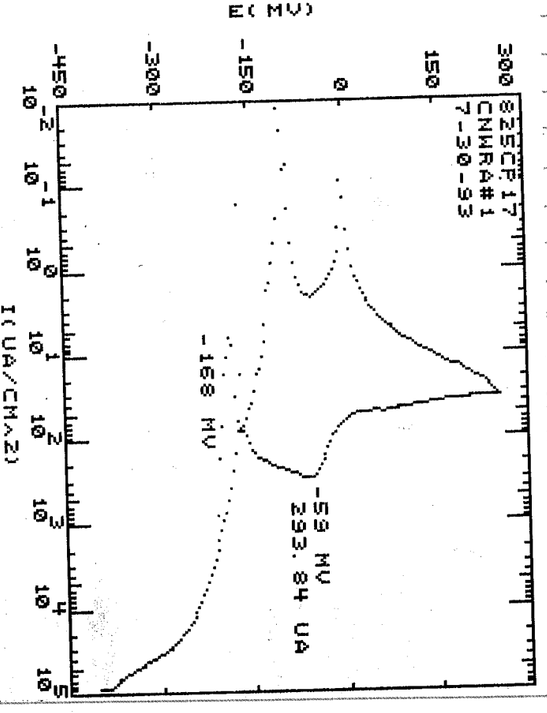
AREA(CMS^2)	11.809
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-208
MV/PT	4
DATA MAX	293.8437
DATA MIN	-88661.19
ABS MIN	1.185536E-02
ABS MAX	88661.19

LEGEND

825CP17
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER14
TEMP 100C
7/30/93



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP18
INITIAL E(MV)	-400 VS R
VERTEX E(MV)	250 VS R
FINAL E(MV)	0 VS E
SCAN RATE(MV/S)	1.67
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-350
CONDITION T(S)	250
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

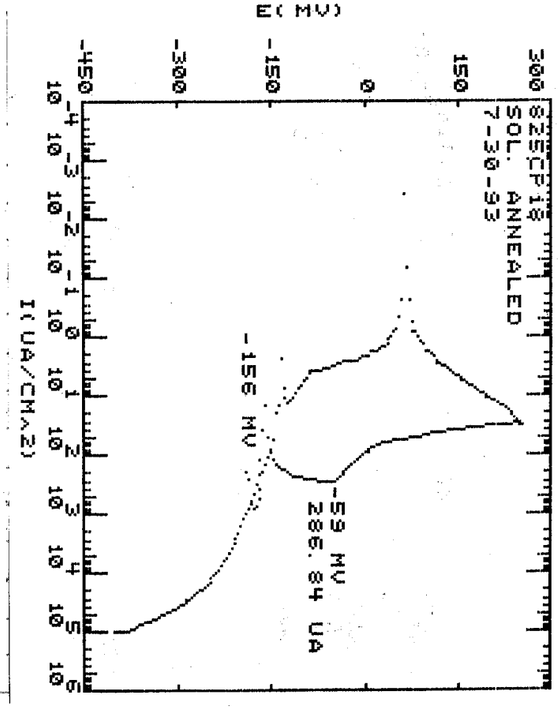
AREA(CMS^2)	10.668
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-193
MV/PT	4
DATA MAX	860.5174
DATA MIN	-98425.2
ABS MIN	3.749531E-03
ABS MAX	98425.2

LEGEND

825CP18
SAMPLE SOLUTION ANNEALED
1 SIDE 1 MICRON 4 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER14
TEMP 100C
7/30/93



8/2/93 825ER16

Sample: 825 CNWRA#1 SEE PAGE 7
1 side 1μ 5 sides 600 grit
Solution: Reused from 825ER14
T = 100°C Hg Therm 0323008 SAME AS 825CP13 PAGE 10
Technique: PSEPR PAGE 14
Saved as: 825ER16
Reference SN0165415
Ecorr: -35 mV
Scan Rate = .17 mV/s
Passivation 250 mV @ 120 s
Appearance: Grain Boundary corrosion and some staining

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER16
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	PASS

SAMPLE PARAMETERS

AREA(CMS^2)	11.8

DATA SCALE

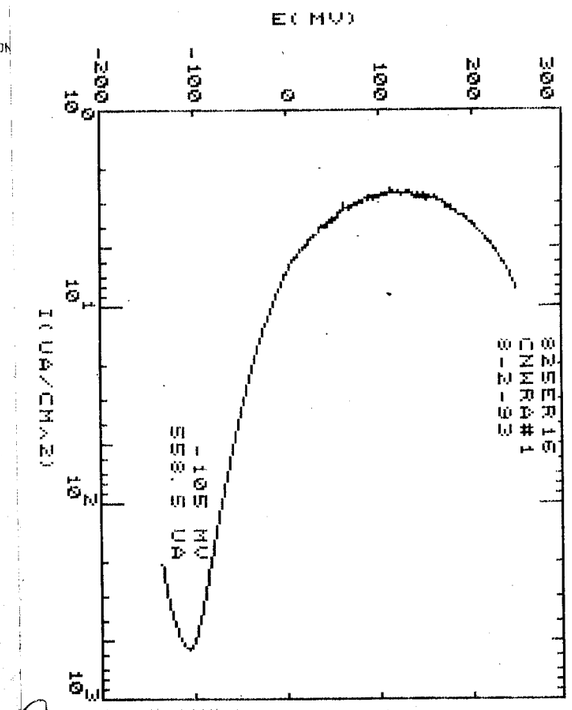
ECORR	-35
MV/PT	1
DATA MAX	558.4745
DATA MIN	2.508475
ABS MIN	2.508475
ABS MAX	558.4745

RESULTS

START E(MV)	-135
END E(MV)	250
MILLICOULOMBS/CM^2	151.78

LEGEND

825ER16
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER14
TEMP 100C
8/2/93



Robert E. Kelly 8/2/93

8/2/93 825ER17

Sample Solution Annealed
1 side 1μ 5 sides 600 grit
Solution: Reused from 825ER14
T = 100°C Hg Therm 0323008 SAME AS 825CP13 PAGE 10
Technique PSEPR PAGE 14
Saved As 825ER17
Ecorr = -169 mV
Scan Rate .17 mV/s
Passivation 250 mV @ 120 s
Appearance: Stained
Reference: SN0165415

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER17
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	20 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

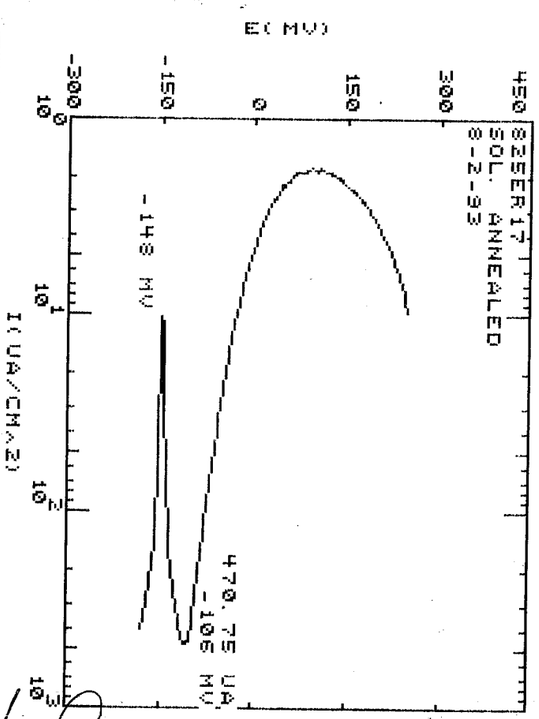
ECORR	-169
MV/PT	2
DATA MAX	470.7547
DATA MIN	-393.3962
ABS MIN	1.783019
ABS MAX	470.7547

RESULTS

START E(MV)	-148
END E(MV)	250
MILLICOULOMBS/CM^2	126.04

LEGEND

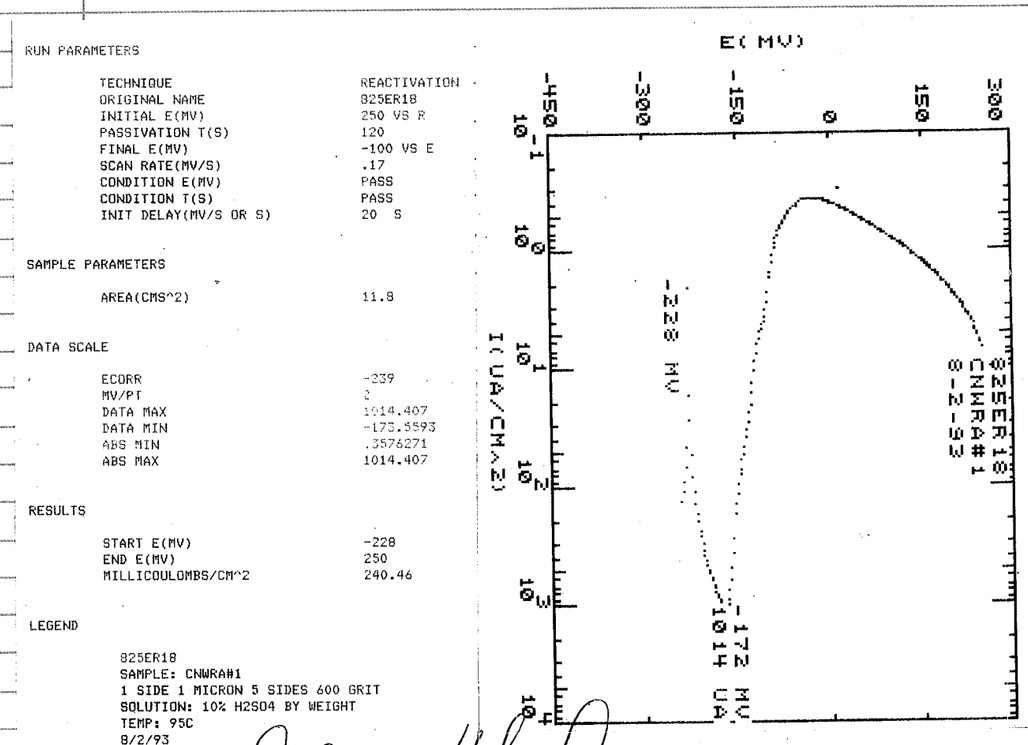
825ER16
SAMPLE: SOLUTION ANNEALED
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER14
TEMP 100C
8/2/93



Robert E. Kelly 8/2/93

8/2/93 ~~825ER17~~ 825ER18 fk 8/2

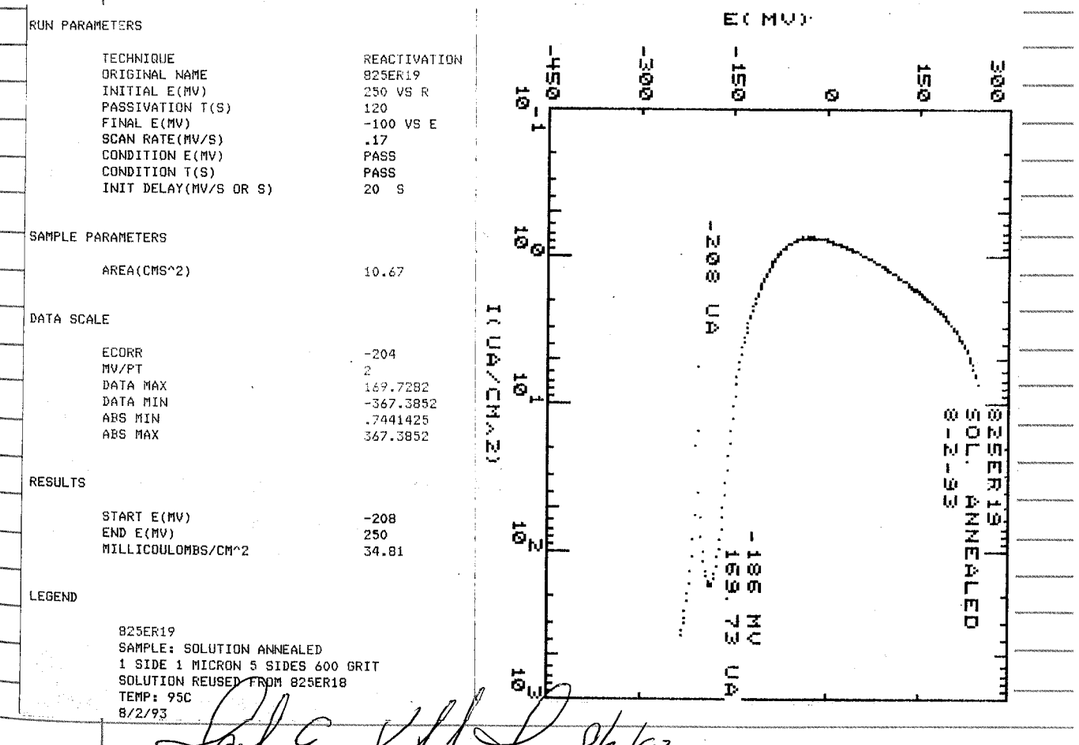
Sample CNWRA#1 see page 7
1 side 1μ 5 sides 600 grit
Solution: 10% H_2SO_4 by Weight
54.5 ml 96% H_2SO_4 Lot # 925400
900 ml H_2O (18MΩ)
T = 95°C Hg Therm 183303 calibrated
2 July 93 Next due 2 Jan 94
Technique: PSEPR page 14
Saved as 825ER18
Reference: SN0165415
Scan Rate .17 mV/s
Passivation 250 mV @ 120 s
Ecorr ~~-169 mV~~ fk 8/2 -239 mV
Appearance grain boundary corrosion and staining.



Paul E. Kalish 8/2/93

8/2/93 ~~825ER18~~ 825ER19 fk 8/2

Sample Solution Annealed
1 side 1μ 5 sides 600 grit
Solution Reused from 825ER18
T = 95°C Hg Therm 183303 same as 825ER18 page 38.
Technique PSEPR page 14
Saved as 825ER19
Reference SN0165415
Scan Rate .17 mV/s
Passivation 120 s @ 250 mV
Ecorr: ~~-16~~ fk 8/2 -204 mV
Appearance: Stained.



Paul E. Kalish 8/2/93

8/3/93 825ER20

Sample: CNWRA#1
1 side 1μ 5 sides 600 grit
Solution: 5% H_2SO_4 by weight
54.5 ml 96% H_2SO_4 Lot# 925400
1900 ml H_2O (18MΩ)
T = 95° Hg Therm 032300 SAME AS 825CP13 page 10
Technique: PSEPR page 14
Saved As 825ER20
E_{corr}: -198 mV
Passivation: 250 mV @ 120 s
Scan Rate: .17 mV/s
Reference: SN0165415
Appearance: Grain boundary corrosion and stained
Q/A = -184 mC 8/3

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER20
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.8

DATA SCALE

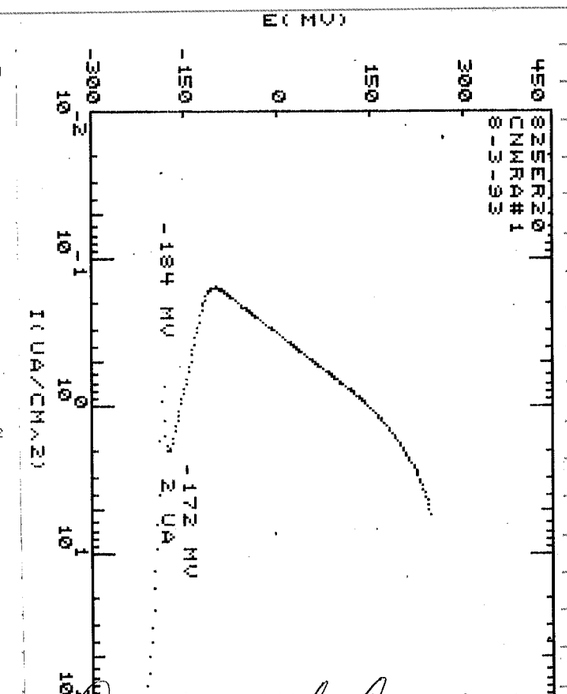
ECORR	-198
MV/PT	2
DATA MAX	5.415254
DATA MIN	-99.66102
ABS MIN	2.627119E-02
ABS MAX	99.66102

RESULTS

START E(MV)	-184
END E(MV)	250
MILLICOULOMBS/CM^2	2.32

LEGEND

825ER20
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION: 5% H2SO4 BY WEIGHT
TEMP: 95C
8/3/93



Frank E. Kelly Jr. 8/3/93

8/3/93 825ER21

Sample ~~CNWR~~ 8/3 Solution Annealed
1 side 1μ 5 sides 600 grit
Solution: 1% H_2SO_4 by weight
27.2 ml H_2SO_4 (96%) Lot # 925400
950 ml (18MΩ) H_2O
T = 95°C Hg Therm 183303 SAME AS 825ER18 page 38
Technique: PSEPR page 14
Saved As 825ER21
E_{corr}: -244 mV
Passivation: 250 mV @ 120 s
Scan Rate: .17 mV/s
Reference: SN0165415
Appearance: Stained slightly
Q/A = 118.59

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER21
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

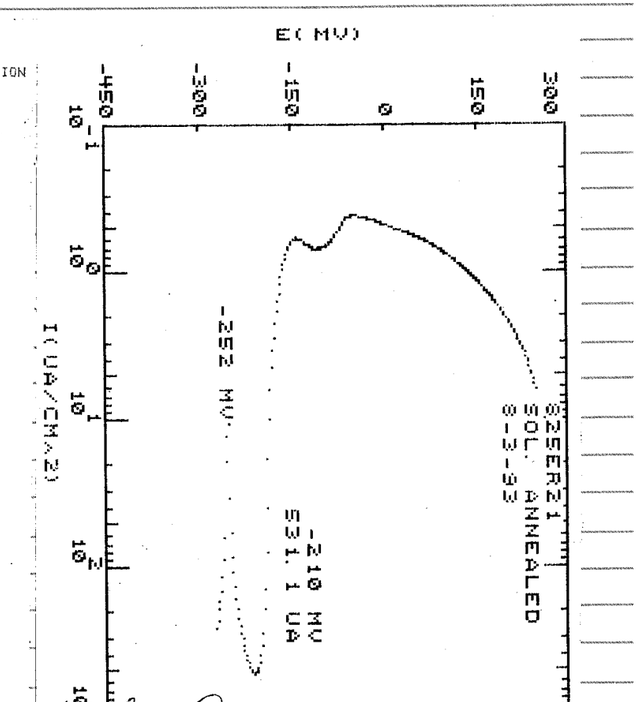
ECORR	-244
MV/PT	2
DATA MAX	531.1321
DATA MIN	-256.6038
ABS MIN	.4160377
ABS MAX	531.1321

RESULTS

START E(MV)	-252
END E(MV)	250
MILLICOULOMBS/CM^2	118.59

LEGEND

825ER21
SAMPLE: SOLUTION ANNEALED
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION: 1% H2SO4 BY WEIGHT
TEMP 95C
8/3/93



Frank E. Kelly Jr. 8/3/93

8/3/93 825ER22

Sample CNWRA#1

1 side 1μ 5 sides 600 grit

Solution Reused from 825ER21 (1% H_2SO_4)

T = 95°C Hg Therm 183303 same as 825ER18 page 38

Technique: PSEPR page 14

Saved as 825ER22

Ecorr -248 mV

Passivation: 250 mV @ 120 s

Scan Rate .17 mV/s

Reference: SN0165415

Appearance Grain boundry corrosion

Q/A 231.65

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER22
INITIAL E(MV)	250 VS. R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.8

DATA SCALE

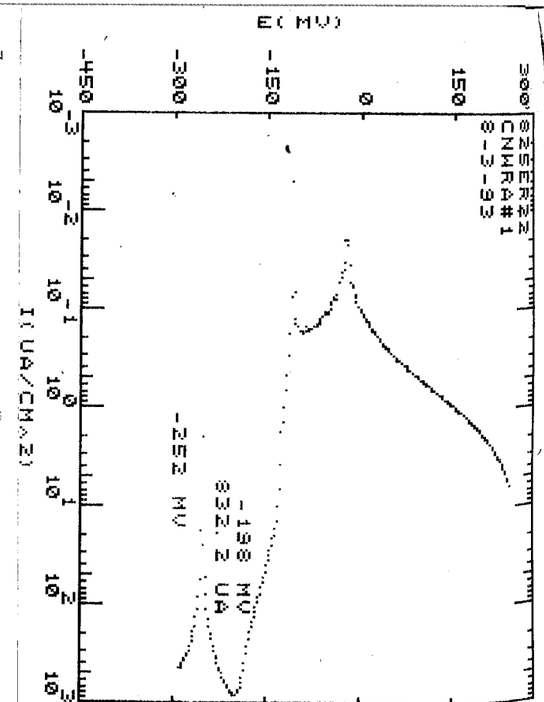
ECORR	-248
MV/PT	2
DATA MAX	832.2034
DATA MIN	-450
ABS MIN	5.084746E-03
ABS MAX	832.2034

RESULTS

START E(MV)	-252
END E(MV)	250
MILLICOULOMBS/CM^2	231.65

LEGEND

825ER22
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED 825ER21
TEMP 95C
8/3/93



Area changed @ approx -75 mV ~~(REMOVES~~ 8/3 made so sample not 100% submerged.

[signature] 8/3/93

8/3/93 825ER23

Sample: Solution Annealed

1 side 1μ 5 sides 600 grit

Solution: Reused from 825ER20 (5% H_2SO_4)

T = 95°C Hg Therm 0323008 same as 825CP13 page 10

Technique PSEPR page 14

Saved as 825ER23

Ecorr -196 mV

Passivation 250 mV @ 120 s

Scan = .17 mV/s

Reference: SN0165415

Appearance: Grain boundry corrosion

Q/A: 9.04

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER23
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

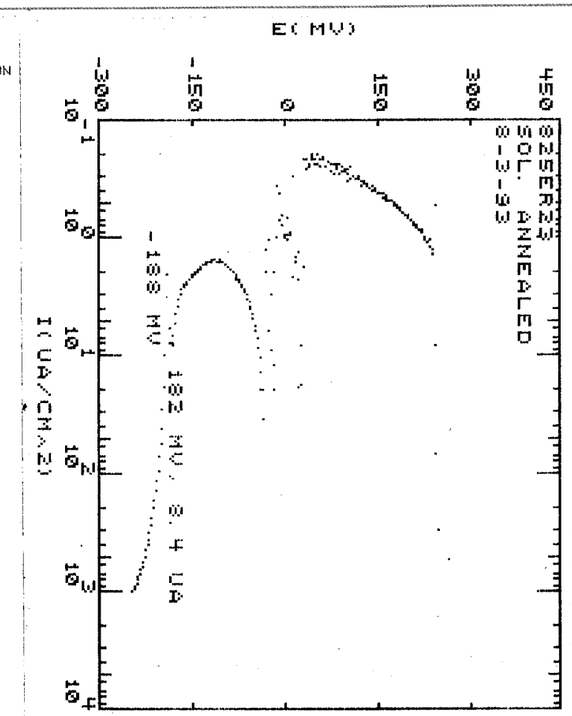
ECORR	-196
MV/PT	2
DATA MAX	701.2868
DATA MIN	-1008.491
ABS MIN	.1943396
ABS MAX	1008.491

RESULTS

START E(MV)	-188
END E(MV)	250
MILLICOULOMBS/CM^2	9.04

LEGEND

825ER23
SAMPLE: SOLUTION ANNEALED
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLTUION: REUSED FROM 825ER20
TEMP 95C
8/3/93



Bubble in salt bridge.

[signature] 8/3/93

8/3/93 825ER24

Sample: CNWRA#1 see page 7
1 side 1μ 5 sides 600 grit
Solution: Reused from 825ER20 (5% H_2SO_4)
T= Hg Therm 0323008 same as 825CP13 page 10
Technique: PSEPR page 14
Saved As 825ER24
Ecorr -196 mV
Scan .17 mV/s
Passivation 120s @ 250mV
Appearance Grain boundry corrosion (slight)
Q/A 2.96

UN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER24
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.8

DATA SCALE

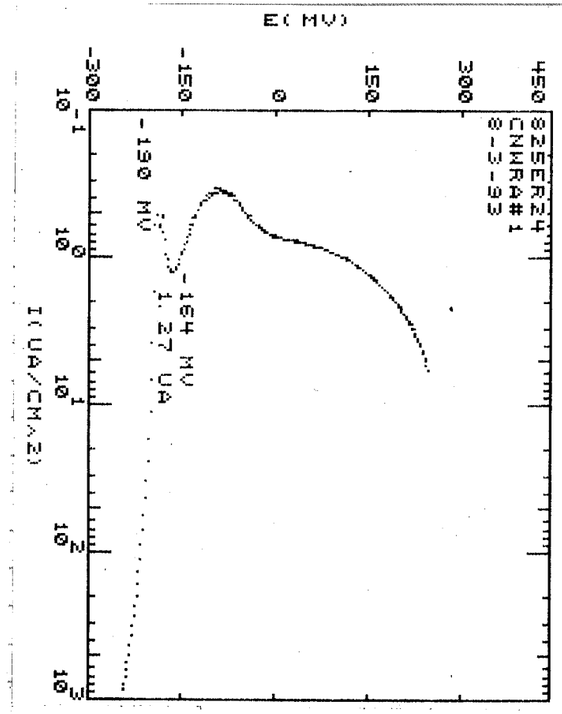
ECORR	-196
MV/PT	2
DATA MAX	5.940678
DATA MIN	-867.7966
ABS MIN	.1
ABS MAX	867.7966

RESULTS

START E(MV)	-190
END E(MV)	250
MILLICOULOMBS/CM^2	2.96

LEGEND

825ER24
SAMPLE; CNWRA#1
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER20
TEMP 95C
8/3/93



[signature] 8/3/93

8/4/93 825ER25

Sample: CNWRA#1
1 side 1μ 5 sides 600 grit
Solution Reused from 825ER21
T= 95°C Hg Therm 183303 same as 825ER18 page 38
Technique: PSEPR page 14
Saved As 825ER25
Ecorr: -233 mV
Scan .17 mV/s
Passivation 120s / 250mV
Appearance None seen
Q/A N/A

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER25
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

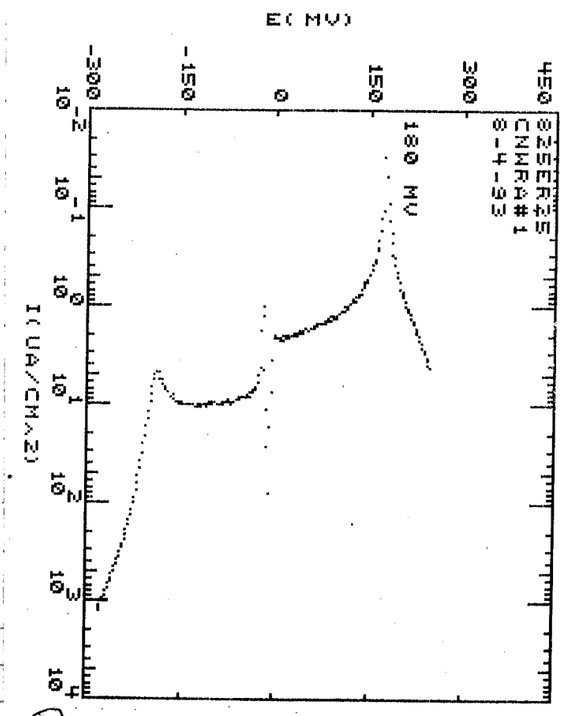
AREA(CMS^2)	11.758

DATA SCALE

ECORR	-233
MV/PT	2
DATA MAX	80.03062
DATA MIN	-1276.578
ABS MIN	1.020582E-02
ABS MAX	1276.578

LEGEND

825ER25
SAMPLE: CNWRA#1
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER21
TEMP 95C
8/4/93



[signature] ~~8/3/93~~ 8/4/93

8/4/93 825ER26

Sample Solution Annealed
1 side 1μ 5 sides 600 grit
Solution Reused 825ER20 (5%)
T = 95°C Hg Therm 0323008 SAME AS 825CP13 page 10
Technique: PSEPR page 14
Saved As 825ER26
Reference SN0165415
Ecorr -177 mV
Rate .17 mV/s
Passivation 120s / 250 mV
Appearance: No apparent corrosion
Q/A: 1.71

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER26
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

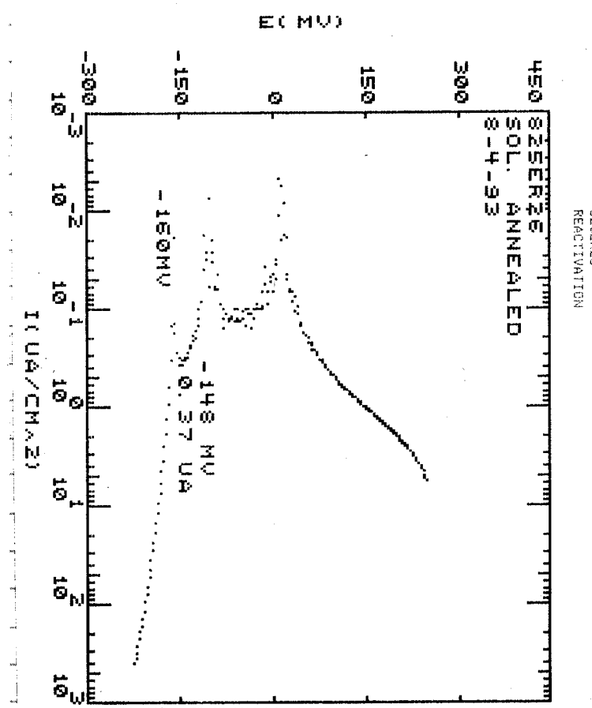
ECORR	-177
MV/PT	2
DATA MAX	5.679245
DATA MIN	-401.8868
ABS MIN	4.716981E-03
ABS MAX	401.8868

RESULTS

START E(MV)	-160
END E(MV)	250
MILLICOULOMBS/CM^2	1.71

LEGEND

825ER26
SAMPLE: SOLUTION ANNEALED
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER20
TEMP 95C
8/4/93



Joseph E. Kelly Jr. 8/4/93

8/4/93 825ER27

Sample: CNWRA#1 see page 7
1 side 1μ 5 sides 600 grit
Solution Reused from 825ER21 (1%)
T = 95°C Hg Therm ~~0~~ 4K 8/4 183303 SAME AS 825ER18 page 38
Technique: PSEPR page 14
Saved As: 825ER27
Reference: SN0165415
Ecorr: -205 mV
Rate: .17 mV/s
Passivation: 120s / 250 mV
Appearance: Didn't look - left in cell see 825ER28 page 48.
Q/A: -13.76

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER27
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.758

DATA SCALE

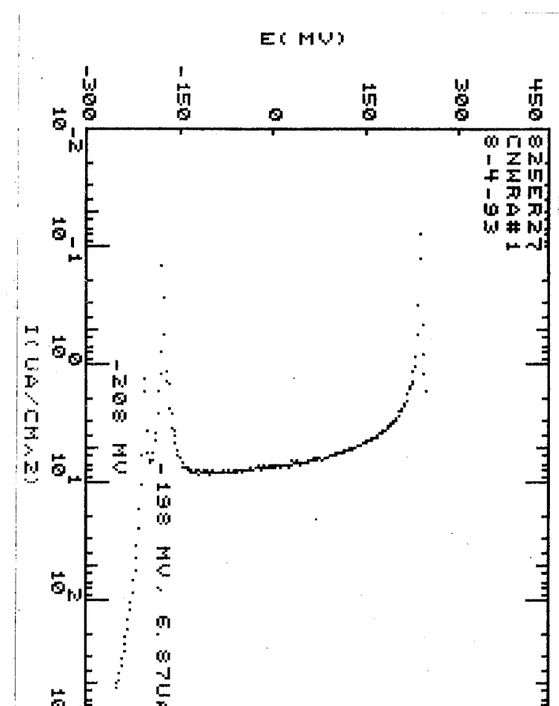
ECORR	-205
MV/PT	2
DATA MAX	6.871917
DATA MIN	-555.3665
ABS MIN	7.739411E-02
ABS MAX	555.3665

RESULTS

START E(MV)	-208
END E(MV)	250
MILLICOULOMBS/CM^2	-13.76

LEGEND

825ER27
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER2 (1%)
TEMP 95C
SAMPLE: CNWRA#1
8/4/93



Joseph E. Kelly Jr. 8/4/93

8/4/93 ~~825ER28~~ 4k 8/4 825ER28

Sample: CNWRA#1 SEE PAGE 7
Reused sample from 825ER27 w/out repolishing.
Cathodicly polarized to freq to remove oxide.

Solution Reused 825ER21 (10%)
T = 95°C Hg Therm 183303 SAME AS 825ER18 PAGE 38

Technique PSEPR PAGE 14

Saved As 825ER28

Reference SNO165415

Ecorr: -201 mV

Scan .17mV/s

Passivation 120s @ 250mV

Appearance: Very slight grain boundary attack

Q/A: N/A

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ER28
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	-450
CONDITION T(S)	600
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

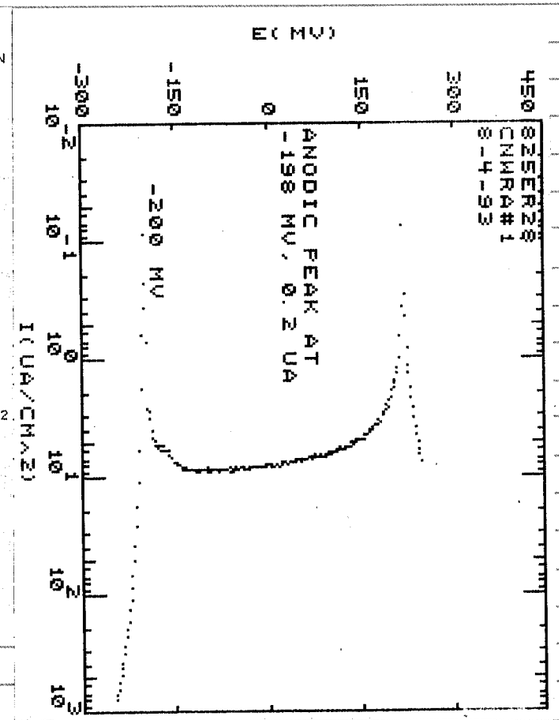
AREA(CMS^2)	11.758

DATA SCALE

ECORR	-201
MV/PT	2
DATA MAX	7.671372
DATA MIN	-797.7547
ABS MIN	7.654363E-02
ABS MAX	797.7547

LEGEND

825ER27
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SOLUTION REUSED FROM 825ER2 (1%)
TEMP 95C
SAMPLE: CNWRA#1
8/4/93



8/4/93

8/4/93 Process of Creating CNWRA#6 and CNWRA#7

The purpose of this is to create 2 heat treated samples; CNWRA#6 and CNWRA#7. These samples will primary be used for determining sensitization of alloy 825.

Both samples were sanded/ground to remove any pits and/or foreign oxides. Each sample was placed in a ceramic boat and had a calibrate thermocouple placed in the threaded hole of the sample. The thermocouple was an Omega Model HH22 #T-94140 calibrated 7 July 93 next due 7 Jan 94. The thermocouple was used to verify the temperature of the sample.

The samples were treated in a Lindberg oven Model 51828

The treatments of ~~bot~~ 8/4/93 the samples is as follows

Name	Previous treatment	This treatment	IN	Out
CNWRA#6	SA; 750°F/15hrs	800°C/1hr	1:30PM	2:30PM
CNWRA#7	SA	700°C/15hrs	6:50PM 8/4	9:50PM 8/5

The IN time represents when the sample reached the treatment temperature.

8/4/93

8/5/93 Procedure to test for Sensitization in Alloy 825 (PTS)

The purpose of this technique is to show quantitatively the degree of sensitization in Alloy 825 without using boiling Nitric acid.

Samples are Alloy 825, HT# HH4371FC, of various heat treatments. All samples should be at least 600 grit finished surfaces. For these tests 1 side will be 1μ or ~~1~~ PK 8/5/93 less finish. All sides are brought to a 600 grit finish. The side chosen for < 1 micron should first ~~be~~ PK 8/5 have all edges beveled with 600 grit. The sample should be ultrasonicly cleaned for 5 minutes in detergent, degreasing detergent. Rinse off with water. Polish with 3μ diamond film ~~for~~ PK 8/5/93 using degreasing detergant as lubricant. Rinse off with water. Polish with 3μ diamond paste and universal diamond extender. The sample is then ~~wash~~ PK 8/5 ultrasonicly cleaned for 5 minutes in degreasing detergent. The sample is then polished with Silica colloidal solution (Mastermet). The other sides are lightly polished with 600 grit. The polished side is swabed with water to remove foreign particles. The sample is then ultrasonicly cleaned with detergent. The sample is rinsed and the hole in the top for the brass rod is dried out and a brass rod is screwed into for electrical connection.

The solution used is 10% H_2SO_4 by weight made by adding 54.35 ml 96% H_2SO_4 to 900 ml 18MΩ water. The solution is in constant deaereation while in the cell and is held at 95°C.

The cell is a standard G-5 cell with a platinum counter electrode, Hg thermometer, long salt bridge, and aerator tube ~~cont~~ PK 8/5/93 connected to a condenser

8/5/93 ~~8/25~~ PK 8/5 PTS

tube and water trap. A SCE, Fisher 13-620-51, is used as Reference.

The potentiostat is an EG and G Model M173. ~~#0~~ PK 8/5/93 #62101 calibrated 19 May 93 Next due 19 May 94. The controlling software is M342C which also saves the collected data.

After the cell is constructed and up to temperature deaeration starts. The sample is in vapor space for 1 hour. The sample is immersed in solution after 1 hour. All connections are made from potentiostat to electrodes and salt bridge is positioned. The Reactivation option is selected. The parameters are 5 min @ -450 mV, .30 s delay, ~~start~~ PK 8/5/93 passivation 2 min @ 250 mV, Start at 250 mV, scan rate .17 mV/s, and stop at -50 mV vs E. When done disconnect all wires. Remove Reference electrode. Remove sample. Deaeration and heating can continue if another sample is to be done. The sample should be ultrasonicly cleaned to remove any oxides. View surface. Take picture if desired.

If sample reused Remove all signs of corrosion by doing as many steps in sample prep as necessary.

Paul E. Ruff Jr. 8/5/93

8/5/93 825ST1

Sample : CNWRA#1

Solution : 10% H_2SO_4 by weight

109.0 ml 96% H_2SO_4 Lot# 925400

1800 ml H_2O

T = 95° Therm # 0323008 same as 825CP13 page 10

Technique PTS page 50

Reference : SN0165415

Saved As 825ST1

Ecorr : -196 mV

Q/A : 28.96

Scan .17 mV/s

Appearance : GB attack

Remarks : peak is very small ; will try again

Ept = 70 mV

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ST1
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.75

DATA SCALE

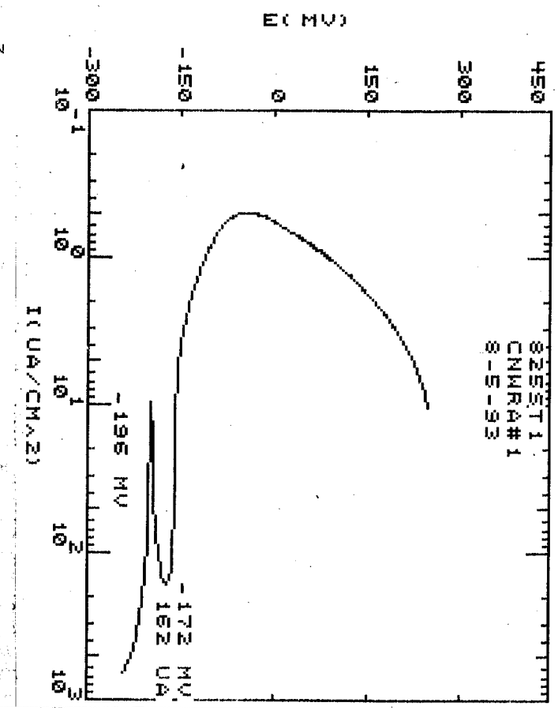
ECORR	-196
MV/PT	2
DATA MAX	162.5532
DATA MIN	-663.8298
ABS MIN	.4961702
ABS MAX	663.8298

RESULTS

START E(MV)	-196
END E(MV)	250
MILLICOULOMBS/CM^2	28.96

LEGEND

825ST1
SAMPLE: CNWRA#1
SOLUTION: 10% H2SO4
TEMP 95C
8/5/93



[signature] 8/5/93

8/5/93 825ST2

Sample : Solution Annealed

Solution : 10% H_2SO_4 by weight

used from stock solution made for 825ST1 page 52

Therm # 183303 same as 825ER18 page 38

Technique : PTS ~~TPK~~ 8/5 page 50

Reference : SN0165415

Saved As : 825ST2

Ecorr : -215

Q/A : 75.47

Appearance : very slight grain boundry attack

Remarks : dont know why peak larger than 825ST1 page 52

Ept = 705 mV

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ST2
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

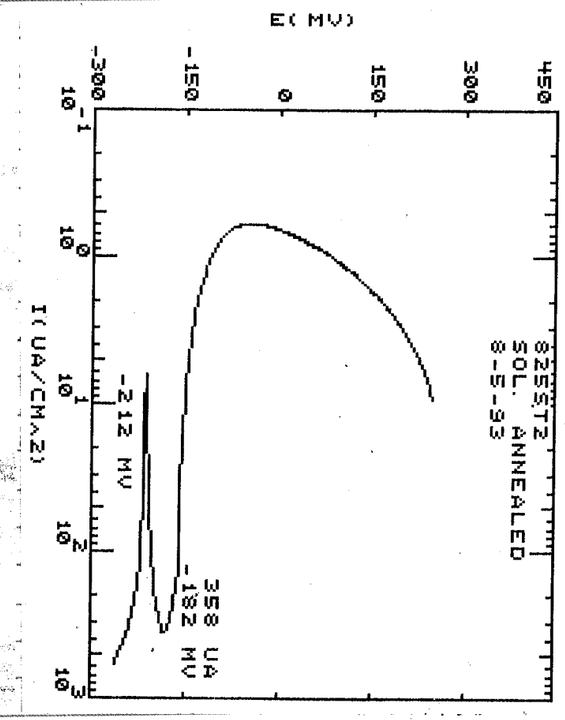
ECORR	-215
MV/PT	2
DATA MAX	358.4905
DATA MIN	-576.4151
ABS MIN	.6
ABS MAX	576.4151

RESULTS

START E(MV)	-212
END E(MV)	250
MILLICOULOMBS/CM^2	75.47

LEGEND

825ST2
SOLUTION ANNEALED
SOLUTION: 10% H2SO4
TEMP 95C
8/5/93



[signature] 8/5/93

8/5/93 825ST3

Sample CNWRA#1 see page 7

Solution: Reused from 825ST2 page 53 (10%)

Therm # 18303 ~~pa~~ pk 8/5 same as 825ER18 page 38

Technique: PTS page 50 except used 1μ diamond instead of Mastermet and no cathodic hold.

Saved As: 825ST3

Reference SN0165415

Ecorr: -189mV

Ept: 287mV

Scan: 17 mV/s

Appearance: grain boundry attack

Remarks: peak very small; there must be some large variability that's not apparent.

Q/A: 3.69

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ST3
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.75

DATA SCALE

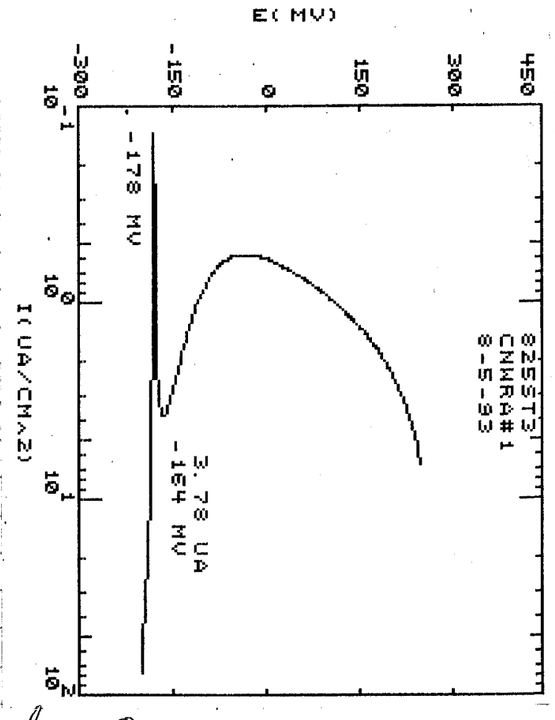
ECORR	-189
MV/PT	2
DATA MAX	6.638298
DATA MIN	-78.29787
ABS MIN	.1361702
ABS MAX	78.29787

RESULTS

START E(MV)	-178
END E(MV)	250
MILLICOULOMBS/CM^2	3.69

LEGEND

825ST3
SAMPLE; CNWRA#1
SOLUTION REUSED FROM 825ST2
TEMP 95C
8/5/93



Frank E. Kiff Jr. 8/5/93

8/5/93 825ST4

Sample: Solution Annealed

Solution: Reused from 825ST1 page 52 (10% H_2SO_4)

Therm 0323008 ~~pa~~ pk 8/5 same as 825CP13 page 10

Technique: PTS page 50 except used 1μ diamond insted of Master Met and no cathodic pretreatment.

Saved As 825ST4

Reference SN0165415

Ecorr -187mV

Ept: 326

Appearance: didn't look -

Remarks: At least this peak smaller than 825ST3 page 54

Q/A: 2.65

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ST4
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	10.6

DATA SCALE

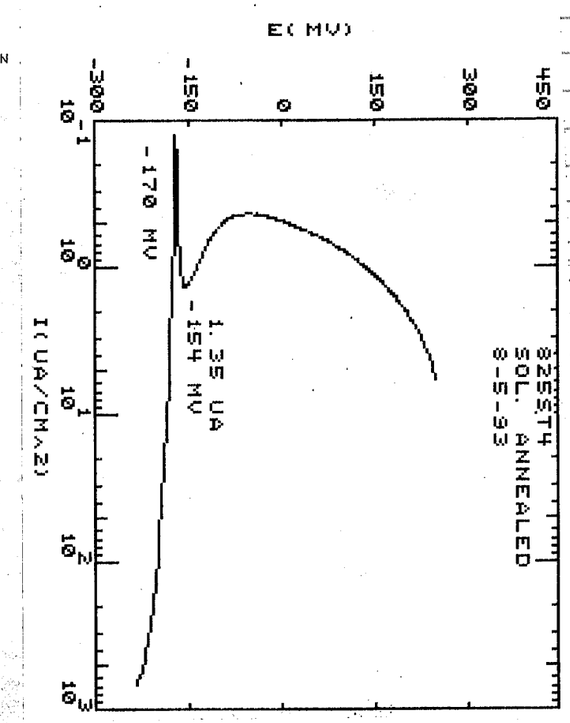
ECORR	-187
MV/PT	2
DATA MAX	5.754717
DATA MIN	-680.1887
ABS MIN	.1245283
ABS MAX	680.1887

RESULTS

START E(MV)	-170
END E(MV)	250
MILLICOULOMBS/CM^2	2.65

LEGEND

825ST4
SAMPLE: SOLUTION ANNEALED
SOLUTION REUSED FROM 825ST1
TEMP 95C
8/5/93



Frank E. Kiff Jr. 8/5/93

8/6/93 ~~8/6/93~~ PK 8/6 825ST5

Sample 825 · CNWRA#1 ~~SE~~ PK 8/6 15hrs/750°C

Solution Reused from 825ST1 (10% H_2SO_4)

T=95; Therm 0323008 same as 825CP13 page 10

Technique: PTS page 50 except no cathodic hold, polished to 1μ, and sample put in just before test, still deaerated for 1 hour.

Saved As 825ST5

Reference SN0165415

Ecorr: -182

Scan .17

Ept: +460 mV

Appearance: light grain boundry attack

Q/A ~~3.01~~ PK 8/6 3.01

RUN PARAMETERS

TECHNIQUE	REACTIVATION
ORIGINAL NAME	825ST5
INITIAL E(MV)	250 VS R
PASSIVATION T(S)	120
FINAL E(MV)	-100 VS E
SCAN RATE(MV/S)	.17
CONDITION E(MV)	PASS
CONDITION T(S)	PASS
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

AREA(CMS^2)	11.75

DATA SCALE

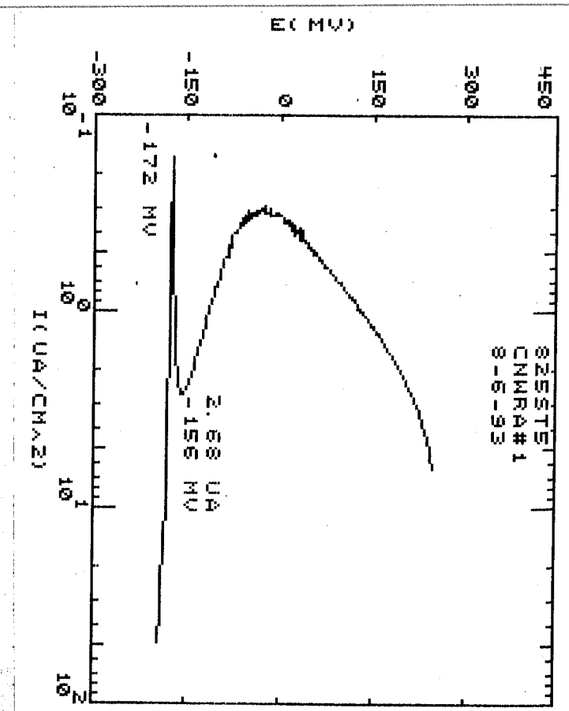
ECORR	-182
MV/PT	2
DATA MAX	6.289361
DATA MIN	-48.17021
ABS MIN	.1608511
ABS MAX	48.17021

RESULTS

START E(MV)	-172
END E(MV)	250
MILLICOULOMBS/CM^2	3.01

LEGEND

825ST5
SAMPLE: CNWRA#1
SOLUTION REUSED FROM 825ST1
TEMP 95C
8/6/93



Frank E. Kiff Jr. 8/6/93

8/6/93 825CP19

Sample CNWRA#1 (15hr/750°C)

1 side 1μ 5 sides 600 grit

Solution 10% H_2SO_4 Reused from 825ST1 page 52

T= ~~95°C~~ 90°C PK 8/6 Hg Therm 0323008 same as 825CP13 page 10

Technique: PCP page 8

Saved As 825CP19

Reference SN0165415

Ecorr -179 mV

Ept: 340 mV

Appearance: left in cell for 825CP20

Ie/If = N/A

Remarks. Repassivation peak higher than wanted.

RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP19
INITIAL E(MV)	-20 VS E
VERTEX E(MV)	200 VS R
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	.17
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

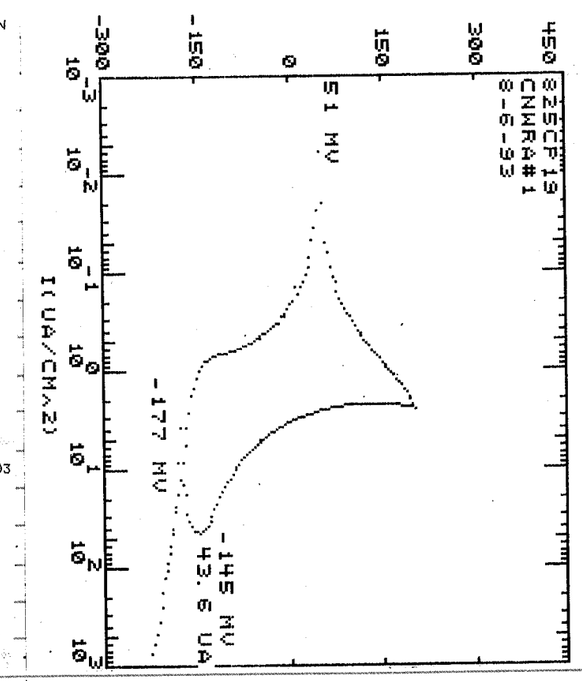
AREA(CMS^2)	11.75
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-179
MV/PT	4
DATA MAX	43.57447
DATA MIN	-754.0425
ABS MIN	5.957447E-03
ABS MAX	754.0425

LEGEND

825CP19
1 SIDE 1 MICRON 5 SIDES 600 GRIT
SAMPLE; CNWRA#1
SOLUTION: REUSED FROM 825ST1
TEMP 95C
8/6/93



Frank E. Kiff Jr. 8/6/93

8/6/93 825CP20

Sample CNWRA#1 750°C /15hrs

~~S~~ Yk 8/6 ~~to~~ Yk 8/6/93 Sample Reused from 825CP19 page 57

Solution Reused from 825ST1 page 52 (10% H_2SO_4)

T = ~~95°C~~ 90°C Yk 8/6 Hg Therm 0323008 same as 825CP13 page 10

Technique PCP page ~~10~~ Yk 8/6 page 8

Saved as 825CP20

Reference SN0165415

Ecorr = -170 mV

Scan 1mV/s

Ept = Didn't look

Appearance = Didn't look

RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP20
INITIAL E(MV)	-10 VS E
VERTEX E(MV)	200 VS R
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	1
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

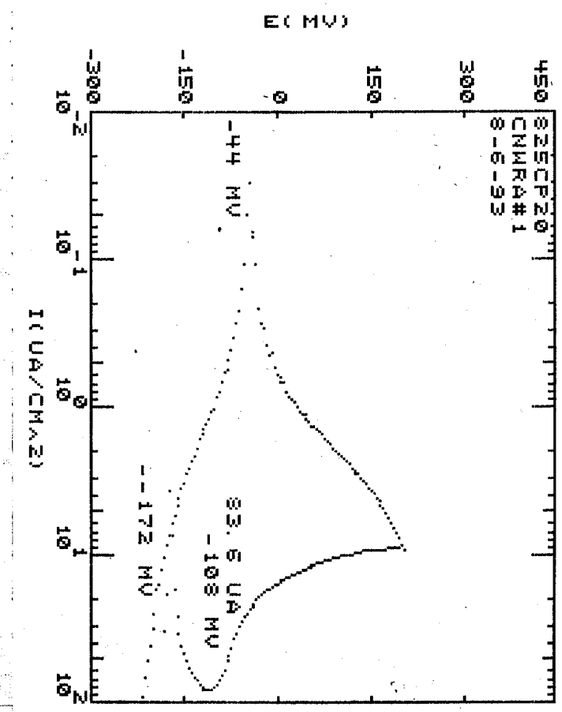
AREA(CMS^2)	11.75
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-170
MV/PT	4
DATA MAX	83.57447
DATA MIN	-91.48936
ABS MIN	.0306383
ABS MAX	91.48936

LEGEND

825CP20
SAMPLE; CNWRA#1
SAMPLE LEFT IN CELL FROM 825CP19
SOLUTION: REUSED FROM 825ST1 (10%)
TEMP 90C
8/6/93



Josh E. Kuhl Jr. 8/6/93

8/6/93 825CP21

Sample CNWRA#1 (750° /15hrs)

Not Repolished after 825CP20

Solution Reused from 825ST1 page 52 (10% H_2SO_4)

T = ~~95°C~~ 90°C Yk 8/6 Hg Therm 0323008 same as 825CP13 page 10

Technique PCP page 8

Reference SN0165415

Ecorr -172 mV

Ept = didn't look

Scan 1.5 mV/s

Saved as 825CP21

Appearance didn't look.

RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP21
INITIAL E(MV)	-10 VS E
VERTEX E(MV)	200 VS R
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	1.5
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

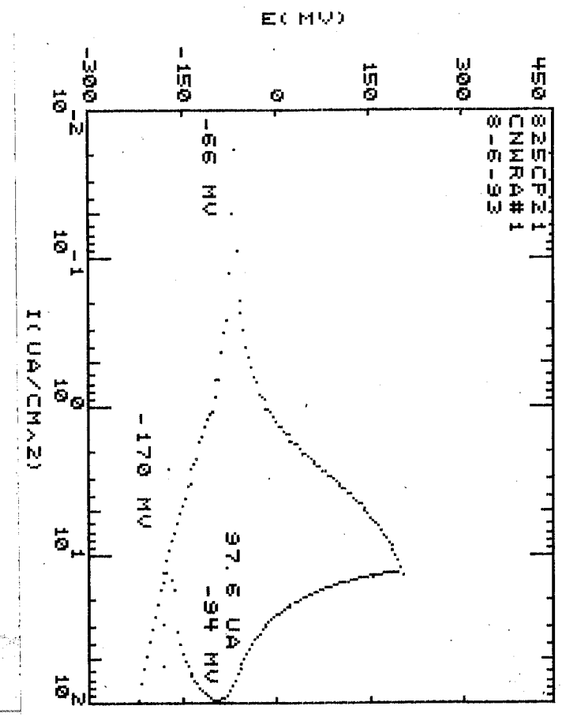
AREA(CMS^2)	11.75
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-172
MV/PT	4
DATA MAX	97.61702
DATA MIN	-94.55319
ABS MIN	.0187234
ABS MAX	97.61702

LEGEND

825CP21
SAMPLE: CNWRA#1
SAMPLE LEFT IN FROM 825CP20
SOLUTION REUSED FROM 825ST1 (10%)
TEMP 90C
8/6/93



Josh E. Kuhl Jr. 8/6/93

8/6/93 825CP22

Sample CNWRA#1 15 hours / 750°C

Not repolished from ~~fro~~ 4K8/6 825CP21

Solution Reused 825ST1 page ~~51~~ 4K8/6 52 (10% H_2SO_4)

T = ~~95°C~~ 4K8/6 90°C Hg Therm 0323008 same as 825CP13 page 10

Technique PCP page 8

Saved As 825CP22

Reference : SN0165415

Ecorr = -173 mV

Ept = didnt look

Scan 3mV/s

Appearance :

RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825CP22
INITIAL E(MV)	-10 VS E
VERTEX E(MV)	200 VS R
FINAL E(MV)	-50 VS E
SCAN RATE(MV/S)	3
THRESHOLD I(UA/CM^2)	PASS
CONDITION E(MV)	-450
CONDITION T(S)	300
INIT DELAY(MV/S OR S)	30 S

SAMPLE PARAMETERS

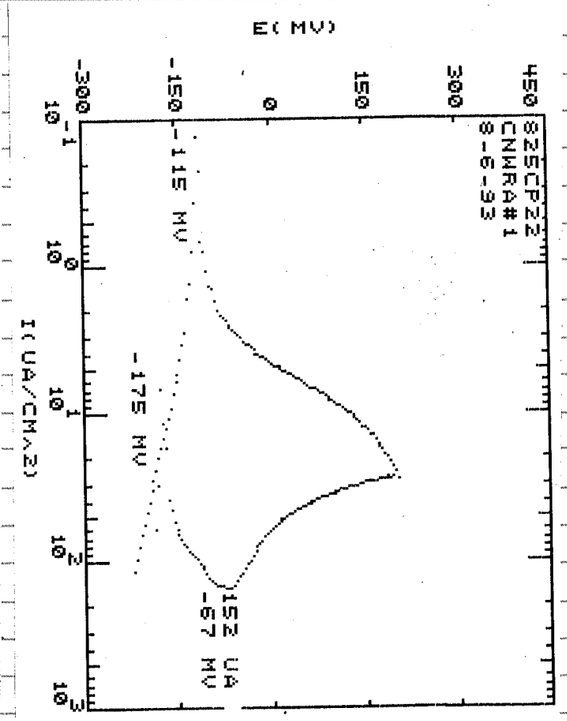
AREA(CMS^2)	11.75
EQ WT(GM)	PASS
DENSITY(GM/CM^3)	PASS
CATHODIC TAFEL(MV)	PASS
ANODIC TAFEL(MV)	PASS

DATA SCALE

ECORR	-173
MV/PT	4
DATA MAX	152.5957
DATA MIN	-115.9149
ABS MIN	.1285106
ABS MAX	152.5957

LEGEND

825CP22
SAMPLE: CNWRA#1
SAMPLE LEFT IN FROM 825CP21
SOLUTION REUSED FROM 825ST1 (10%)
TEMP 90C
8/6/93



[signature] 8/6/93

Pages 1 through 60 of this Scientific Notebook were reviewed for compliance with QAP-001 in response to Corrective Action Request 94-02. Corrections and clarifications were made as appropriate. In some cases, the date of a change will reflect the date of this review rather than the date of the original Scientific Notebook entry.

Randy Folch
SwRI-QA
12/01/94

I have reviewed this scientific notebook and find it in compliance with QAP-001. There is sufficient information regarding procedures used for conducting tests, acquiring and analyzing data so that another qualifiied individual could repeat the activity.

N. Sridhar
2/14/97

Initial scientific notebook entry for galvanic corrosion tests with A 516 grade 60 carbon steel and Ni base alloys.

Title: Galvanic corrosion.

Tests Preformed by: Darrell S. Dunn

Objectives: Determine the effect of parameters such as solution temperature, chloride concentration, pH, and area ratio on the galvanic corrosion of A516 grade 60 carbon steel coupled to other proposed Ni base container materials such as alloy 825.

Equipment: EG&G Versastat Serial Number 20104. EG&G model 352 corrosion software. Data acquisition computer. ESC 440 multi-channel potentiostat. Workbench software. Keithley Electrometer model 614 SN 555368 or equivalent. ASTM G-5 polarization cell, Teflon/glass reaction vessels or vessel meeting the requirements of TOP-008. Electrochemical impedance spectroscopy system consisting of a Solartron 1260 frequency response analyzer, an EG&G model 273 potentiostat and Zplot/Zview for windows data acquisition and analysis software.

Materials: A516 grade 60, Alloy 825, additional materials to be identified at time of test.

Specimen specifications: A516 Grade 60: 0.250"diameter x 1.915" long (+/- 0.005") specimens polished to a 600 grit finish. Specimens to be weighed before and after exposure. Additional specimen dimensions to be described in tests..

Measurement Parameters: As described in TOP-008

Required level of accuracy: Potentials $\pm$ 5mV. Current density $\pm$ 1 $\mu A/cm^2$.
Uncertainty and Sources of Error: Current density calculated as current divided by sample area. Actual current density of corroding areas is not determined.

4/16/97

4/16/97

GALV1.DAT

Specimen: Alloy 825 HH4371FG
start wt: 182.73865 g
end wt: 182.74675 g

A516
start wt: 10.80480 g
end wt. 10.01724 g
immersed at 5:1 ratio
Solution: 1000 ppm Cl^- 85 ppm HCO_3^- 20 ppm $SO_4^=$
10 ppm NO_3^- 2 ppm F^-

3.29850 g NaCl lot 960780
0.23649 g $NaHCO_3$ lot 923337A
40 ml $SO_4^=$ - 4/97 stock solution NB 157 p161
20 ml NO_3^- - 4/97 stock solution NB 157 p161
4 ml F^- - 4/97 stock solution NB 157 p161
+ DI water to 2000 ml
T = 95°C Hg thermometer 1238002
start pH 7.726 aerated w/ zero air
end pH 9.258
Potentiostat: ESC 440 #2 channel 3
data saved as GALV1.DAT

Reference electrode: Fisher SCE 13-620-51 SN 3106321

E_{corr} 825 -249 mV
E_{corr} 516 -644 mV

E applied: used as ZRA

Test started 11:15 Am 4/17/97
Test stopped 1:15 PM 5/1/97
825 repolished + reused

5/5/97
Walter J Mochowski

GALV2.DAT

Specimen: alloy 825 HH4371FG
start wt: 193.63790 g
end wt. 193.64680 g

A516
start wt. 10.91935 g
end wt. 10.08147 g
immersed at 10:1 ratio
Solution: 1000 ppm Cl^- 85 ppm HCO_3^- 20 ppm $SO_4^=$
10 ppm NO_3^- 2 ppm F^-

3.29743 g NaCl lot 960780
0.24268 g $NaHCO_3$ lot 923337A
40 ml $SO_4^=$ - 4/97 stock solution NB 157 p161
20 ml NO_3^- - 4/97 stock solution NB 157 p161
4 ml F^- - 4/97 stock solution NB157 p161
+ DI water to 2000 ml
T = 95°C Hg thermometer 1238002
start pH 7.695 aerated w/ zero air
end pH 9.253
Potentiostat: ESC 440 #2 channel 4
data saved as GALV2.DAT

Reference electrode: Fisher SCE 13-620-51 S/N 106345

E_{corr} 825 -223 mV
E_{corr} 516 -561 mV

set up as ZRA

Test started 11:15 Am 4/17/97
Test stopped 1:15 PM 5/1/97
825 repolished + reused

5/5/97
Walter J Mochowski

GALV3.DAT

Specimen: Alloy 825 HH 4331FG
start wt: 11.58062 g
end wt: 11.56483 g
A516
start wt: 11.00288 g
end wt: 10.13482 g

Solution: 1000 ppm Cl^- 85 ppm HCO_3^- 20 ppm $SO_4^=$
10 ppm NO_3^- 2 ppm F^-

3.29926 g NaCl lot 960780
0.24477 g $NaHCO_3$ lot 923337A
40 ml $SO_4^=$ - 4/97 stock solution NB157 p161
20 ml NO_3 - 4/97 stock solution NB157 p161
4 ml F^- - 4/97 stock solution NB157 p161
+ DI water to 2000 ml
T = 95° Hg thermometer 1238002
start pH 7.618 aerated w/ zero air
end pH 9.333
Potentiostat: ESC 440 #2 channel 6
data saved as GALV3.DAT
Reference electrode: Fisher SCE 13-620-51 S/N 0165415

E_{corr} 825 -252 mV
E_{corr} 516 -639 mV

set up as ZRA

Test started 11:15 Am 4/17/97
Test stopped 1:15 PM 5/1/97

5/5/97
Walter J Mackowski

Thermal conditioning of 825 and A516

Four specimens were put in ovens to be thermally conditioned.

A516, 825 } 100°C for 7 days

A516, 825 } 300°C for 7 days

825 #1 11.35653 g
825 #2 11.37865 "
516 #1 10.86171 "
516 #2 10.76313 "
} wts. before heat

Used Lindberg oven 51828 for 300°C
Used Fisher oven model 838F for 100°C

Oven's temperature checked vs. TC meter Omega DP465
cal lab S/N 3130900 cal 21 Nov 96 due 21 Nov 97

Specimens in oven at 4:40PM 4/17/97
Specimens out at 4:40 PM 4/24/97

Placed in desiccator under N_2.

The two 300°C specimen were darkened, after exposure. 825 was lighter than the A516.

Walter J Mackowski
4/25/97

Experiment conducted to obtain EIS spectra on alloy 825 at different potentials

Alloy 825 heat HH4371FC
start wt. 11.22470 g

Solution: 1000 ppm Cl^- 85 ppm NO_3^- 20 ppm $SO_4^=$
10 ppm NO_3^- 2 ppm F^-

3.29780 g NaCl lot 960780
0.24050 g $NaHCO_3$ lot 923337P
40 ml $SO_4^=$ - 4/97 stock solution NB157 p161
20 ml NO_3^- - 4/97 stock solution NB157 p161
4 ml F^- - 4/97 stock solution NB157 p161
room temperature aerated w/ zero air
start pH 8.021 end pH: not taken
Potential of 825 @ 40 min -188 mV
Platinum potential " +227 mV

Reference electrode: Fisher SCE 13-620-51
S/N 5144349

EIS recorded at O.C., -100 mV, 0 mV, +100 mV, +200 mV and + 300 mV with 10 minute hold at open circuit between runs

data recorded as:

825OC.Z	open circuit
825m1.Z	-100
825p0.Z	zero
825p1.Z	+100
825p2.Z	+200
825p3.Z	+300

on EIS data disk #1

Walter J. Mochowski 4/23/97

Experiment conducted to obtain EIS spectra on A516 gr 60 at different potentials.

A516 gr.60
start wt. 10.73090 g
end wt. 9.80676 g

Solution: same batch as used on p.68, room temp, aerated
start pH: 8.021 end pH: 9.975
potential of A516 -575 mV
potential of platinum +281 mV
same reference electrode as p.68

set up batch file to run EI6 at various potentials

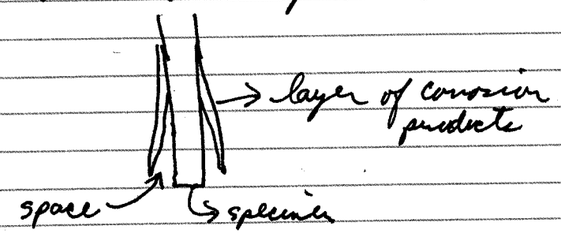
Pretreat Time	Pre-Treat Pot (V)	Setup File	Data File
10	open circuit	open circ 2. zpw	516Z1
60	" "	" "	516Z2
60	" "	" "	516Z3
60	" "	" "	516Z4
60	-.40	m400mVcs. zpw	516Z5
60	-.4	"	516Z6
60	-.4	"	516Z7
60	-.4	"	516Z8
60	-.3	m300mVcs. zpw	516Z9
60	"	"	516Z10
60	"	"	516Z11
60	"	"	516Z12
60	-0.2	m200mVcs. zpw	516Z13
60	"	"	516Z14
60	"	"	516Z15
60	"	"	516Z16
60	-0.1	m100mVcs. zpw	516Z17
60	"	"	516Z18
60	"	"	516Z19
60	"	"	516Z20

Walter J. Mochowski
4/23/97

60	0.0	100mV vs. zpw	516Z21
60	0.0		516Z22
60	0.0		516Z23
60	0.0		516Z24
60	+0.1	100mV vs. zpw	516Z25
60	+0.1		516Z26
60	+0.1		516Z27
60	+0.1		516Z28

Data stored on EIS data disk #1

Solution was a rusty brown w/ corrosion products
Specimen had a heavy layer of corrosion products all over it. Corrosion products formed a "cylinder" around the specimen



Corrosion appeared to be uniform.

Walter J. Mackowski
4/23/97

Experiment conducted to obtain EIS data on A516 gr 60 at different potentials at high pH.

A516 gr 60
start wt. 10.69044 g end wt. 10.21770 g.

Solution: 1000 ppm Cl^- 0.01M Na_2CO_3 20 ppm $SO_4^=$
10 ppm NO_3^- 2 ppm F^-

3.29980 g NaCl lot 960780
2.10660 g Na_2CO_3 lot 960685
40 ml $SO_4^=$ - 4/97 stock solution NB 157 p.161
20 ml NO_3^- - 4/97 " " " " "
4 ml F^- - 4/97 stock solution NB 157 p.161
dil. to 2000 ml with DI water

room temperature aerated w/ gen air
start pH 10.877 end pH 11.600

Reference electrode: Fisher SCE 13-620-51 S/N 5144349

E_{corr} 516 = -476 mV E_{Pt} = +156 mV

Same batch file protocol as on p.69 except data filed as 516Z29 → 516Z56.

Data stored on EIS data disk #1

Solution a rusty brown w/ dark corrosion products on bottom. Specimen had a medium layer of corrosion products (dark blue-green color). Attack seemed fairly uniform.

Walter J. Mackowski
4/25/97

GALV4.DAT

Specimen: Alloy 825 HH4371FG
start wt. 182.63840 g
end wt. 182.64690 g
A516
start wt. 10.63805 g
end wt. 10.49843 g
immersed at 5:1 ratio

Solution: 1000 ppm Cl^- 0.01M Na_2CO_3
20 ppm $SO_4^=$ 10 ppm NO_3^- 2 ppm F^-

3.29762 g NaCl lot 960780
2.12190 g Na_2CO_3 lot 960685
40 ml $SO_4^=$ 4/97 stock solution NB 157 p.161
20 ml NO_3^- 4/97 " " " " "
4 ml F^- 4/97 " " " " "
+ DI water to 2000 ml.

T = 95°C Hg thermometer #0323007
start pH 10.883 aerated w/zero air
end pH 10.324

Potentiostat: ESC 440 #2 channel 3
data saved as GALV4.DAT

Reference electrode: Fisher SCE 13-620-51 ~~SN# 5082405~~
SN# 3106321
5/6/97 WJ Machowski

Ecorr 825 -128 mV
Ecorr 516 -490 mV

Test started 3:05 PM 5/6/97
Test stopped 3:15 PM 5/20/97
825 repolished + reused

Walter J Machowski
5/21/97

GALV5.DAT

Specimen: Alloy 825 HH4371FG
start wt. 193.54590
end wt. 193.57002
A516
start wt. 11.19146 g
end wt. 11.11950 g
immersed at 10:1 ratio

Solution: 1000 ppm Cl^- 0.01M Na_2CO_3
20 ppm $SO_4^=$ 10 ppm NO_3^- 2 ppm F^-

3.29775 g NaCl lot 960780
2.12132 g Na_2CO_3 lot 960685
40 ml $SO_4^=$ 4/97 stock solution NB 157 p.161
20 ml NO_3^- 4/97 " " " " "
4 ml F^- 4/97 " " " " "
+ DI water to 2000 ml

T = 95°C Hg thermometer #0323005
start pH 10.872 aerated w/zero air
end pH 10.247

Potentiostat: ESC 440 #3 channel 4
data saved as GALV5.DAT

Reference electrode: Fisher SCE 13-620-51 ~~SN# 5087324~~
SN# 5129169
5/6/97
WJ Machowski

Ecorr 825 -243 mV
Ecorr 516 -356 mV

Test started 3:05 PM 5/6/97
Test stopped 3:15 PM 5/20/97
825 repolished + reused

Walter J. Machowski
5/21/97

GALV6.DAT

Specimen: Alloy 825 HH 4371FC
start wt. 11.22818 g
end wt. 11.23077 g
A516
start wt. 11.06695 g
end wt. 11.02458 g

Solution: 1000 ppm Cl^- 0.01M Na_2CO_3
20 ppm $SO_4^=$ 10 ppm ~~NO_3^-~~ 2 ppm F^-
NO_3^- wgm 5/5/97

3.29969 g NaCl lot 960780
2.11987 g Na_2CO_3 lot 960685
40 ml $SO_4^=$ 4/97 stock solution NB157 p.161
20 ml NO_3^- 4/97 " " " " "
4 ml F^- 4/97 " " " " "
+ DI water to 2000 ml.

T = 95°C Hg thermometer #0323007
start pH 10.833 aerated w/zero air
end pH 10.337

Potentiostat: ESC 440 #2 channel 6
data saved as GALV6.DAT

Reference electrod: Fisher SCE 13-620-51
SN# 0165415

Ecorr 825 -237 mV
Ecorr 516 -420 mV

Test started 3:05 PM 5/6/97
Test stopped 3:15 PM 5/20/97

5/21/97
Walter J. Markowski

Thermal oxidation of specimens for EIS studies.

Two specimens each of Alloy 825 (HH4371FC) and A516 gr.60 were placed in the Fisher oven model 838F for 7 days, starting at 3:30PM on 5/6/97. The temp was 100°C.

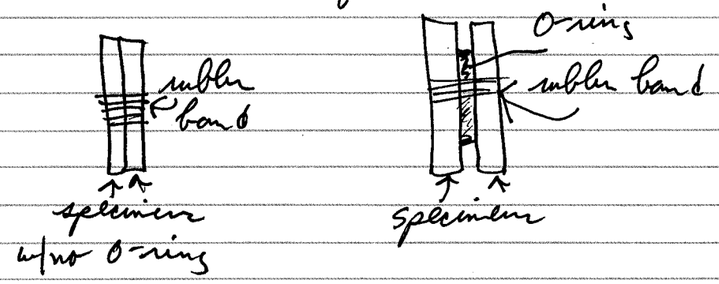
Start 3:30 pm 5/6/97
Temp. 100°C

Specimens removed @ 3:30PM 5/13/97
and put in dessicator under nitrogen

Walter J. Markowski
5/16/97

The purpose of the following tests is to develop EIS data on A516 gr 60 and alloy 825 as the surface varies with deposition of various oxides.

The tests were run by coupling two specimens against each other and holding them tightly together (face to face) w/a rubber band. In some tests an O-ring was inserted between the faces. This was sometimes left empty, filled w/test solution, or filled with wetted oxides of iron.



Walter J. Macknowski
5/8/97

~~516CPL-01~~ A516C-01 WJ Macknowski 5/8/97

Specimen: A516 gr. 60 w/mill scale

Condition: dry

Parameters: FRA Sweep 20,000 → 0.1 Hz
10 steps / decade
2 sec delay
2 sec integration time
P-Stat DC vs. open circuit
10 mV amplitude
Hi-F auto
lo-F auto
auto min 10 μA Trans.F $1E^{-5}$
Attenuator: ON

Stored as ~~516CPL-01.Z~~ A516C-01.Z on EIS data disk 1 of 4/18/97
WJ Macknowski 5/8/97

Walter J. Macknowski
5/8/97

~~516CPL-02~~ A516C-02 wJMacharski 5/8/97

Specimen: A516 gr.60 w/mill scale

Condition: dry with O-ring

Parameter: FRA: Sweep 20,000 → 0.1 Hz
10 steps/decade
2 sec delay
2 sec integration time
P-Stat: DC vs. open circuit
10 mV amplitude
Hi-F auto
Lo-F auto
auto min 10 μA
Trans F 1E-5
Attenuator: ON

Stored as ~~516CPL-02.Z~~ on EIS data disk 1
A516C-02.Z 4/18/97
wJMacharski
5/8/97

Walter J. Macharski
5/8/97

~~516CPL-03~~ A516C-03 wJMacharski 5/8/97

Specimen: A516 gr.60, 600 grit polished

Condition: dry

Parameter: FRA: Sweep 20,000 → 0.1 Hz
10 steps/decade
2 sec delay
2 sec integration time
P-Stat: DC vs. open circuit
10 mV amplitude
Hi F - auto
lo F - auto
auto min 10 μA
Trans. F 1E-5
Attenuator: ON

Stored as ~~516CPL-03.Z~~ on EIS data disk 1
A516C-03.Z 4/18/97
wJMacharski
5/8/97

Walter J. Macharski
5/8/97

A516C-04

Specimen: A516, 600 grit polished

Condition: dry with O-ring

Parameters: as on p.79

Stored as: A516C-04.Z on EIS data disk 1
4/18/97

Walter J. Mochowski
5/8/97

WJ Mochowski
5/8/97 ~~A516-05~~ A516C-05

Specimen: A516 gr.60, 600 grit polished

Condition: dry, w/O-ring, dry Fe_3O_4

Parameters: as on p.79

Stored as: A516C-05.Z on EIS data disk 1
4/18/97

Fe_3O_4 - Johnson-Matthey lot 593424AR

Walter J. Mochowski
5/8/97

~~A516-06~~ A516C-06 wjmachwski 5/8/97

Specimen: A516 gr.60, mill scale

Conditions: O-ring filled w/1000 ppm Cl^- solution

Parameter: as on p.79

Solution: 1000 ppm Cl^- 85 ppm HCO_3^-
20 ppm $SO_4^=$ 10 ppm NO_3^- 2 ppm F^-

3.30290 g NaCl lot 960780
0.24159 g $NaHCO_3$ lot 923337A
40 ml $SO_4^=$ 5/97 stock solution NB157 p. 82
20 ml NO_3^- 5/97 " " "
4 ml F^- 5/97 " " "
diluted w/ DI water to 2000 ml
pH 8.112

Stored as: ~~A516-06.Z~~ on EIS data disk 1
A516C-06.Z 4/18/97
wjmachwski
5/8/97

Walter J. Machwski
5/8/97

~~A516-07~~ A516C-07 wjmachwski 5/8/97

Specimen: A516 gr.60, 600 grit polished

Conditions: O-ring w/ Fe_3O_4 wetted w/1000 ppm Cl^- solution

Parameter: as on p.79

Solution: as on p.82

Stored as: ~~A516-07.Z~~ on EIS data disk 1
A516C-07.Z 4/18/97
wjmachwski
5/8/97

Fe_3O_4 – Johnson-Matthey lot S93434AR

Walter J. Machwski
5/8/97

wj Machowski 5/8/97 ~~A51608~~ A516C-08

Specimen: A516 gr.60, 600 grit polished

Condition: O-ring, dry Fe_2O_3

Parameter: as on p.79

Stored as: A516C-08.Z on EIS data disk 1 4/18/97

Fe_2O_3 – Johnson-Matthey lot 597184

Walter J. Machowski 5/8/97

A516C-09

Specimen: A516 gr.60, 600 grit polished

Condition: O-ring w/ Fe_2O_3 wetted w/ 1000 ppm Cl^- solution

Parameter: as on p.79

Solution: as on p.82

Stored as: A516C-09.Z on EIS data disk 1 4/18/97

Fe_2O_3 Johnson-Matthey lot 597184

Walter J. Machowski 5/8/97

A516C-10

Specimen: A516 gr. 60, 600 grit polished

Condition: O-ring, filled w/1000 ppm Cl^- solution

Parameter: as on p. 79

Solution: as on p. 82

Stored as: A516C-10.Z on EFS data disk 1
4/18/97

Walter J. Machowski
5/8/97

~~A516C11~~ A516C-11
WJ Machowski
5/8/97

Specimen: A516 gr. 60, 600 grit polished

Condition: O-ring, dry corrosion product from CALV 3

Parameter: as on p. 79

Stored as: A516C-11.Z on EFS data disk 1
4/18/97

Walter J. Machowski
5/8/97

A516C-12

Specimen: A516 gr.60, 600 grit polished

Condition: O-ring filled w/ corrosion product from GALV 3
wetted w/ 1000 ppm Cl^- solution

Parameter: as on p. 79

Solution: as on p. 82

Stored as: A516C-12.Z on EIS data disk 1
4/18/97

Walter J Machowski
5/8/97

A516C-13

Specimen: A516 gr.60, 600 grit polished

Condition: O-ring, with dry FeO(OH)

Parameter: as on p. 79

Stored as: A516C-13.Z on EIS data disk 1
4/18/97

FeO(OH) Johnson-Matthey lot I06G10

Walter J. Machowski
5/8/97

A516C-14

Specimen: A516 gr. 60, 600 grit polished

Condition: O-ring, w/wetted FeO(OH); wetted w/ 100ppm Cl^-

Parameters: as on p. 79

Solution: as on p. 82

Stored as: A516C-14.Z on EIS data disk 1
4/18/97

FeO(OH) – Johnson Matthey lot I06G10

Walter J. Machowski
5/8/97

825C-15

Specimen: 825 (HH4371FC), 600 grit polished

Condition: dry

Parameters: as on p. 79

Stored as: 825C-15.Z on EIS data disk 1
4/18/97

Walter J. Machowski
5/8/97

825C-16

Specimen: 825 (HH 4371FG), 600 grit polished

Condition: O-ring, dry

Parameter: as on p. 79

Stored as: 825C-16.Z on EIS data disk 1
4/18/97

Walter J. Machowski
5/8/97

825C-17

Specimen: 825 (HH 4371FG), 600 grit polished

Condition: O-ring filled w/ 1000 ppm Cl^- solution

Parameter: as on p. 79

Solution: as on p. 82

Stored as: 825C-17.Z on EIS data disk 1
4/18/97

Walter J. Machowski
5/8/97