

308 --- Q200311210004
Scientific Notebook # 025: Crevice Corrosion
Experiments of Alloy 825 (12/01/1993 through
02/14/1997)

12/01/93
02/14/97
21
300
REC.

**CNWRA
CONTROLLED
COPY 025**

Pages 1 through 304 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
SWRT - QA
11/28/94

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EQUIPMENT

ELECTRODES -

- 1) ORION MODEL 94-17 Chloride electrode
- 2) ORION MODEL 95-02 CO₂ electrode
- 3) ORION MODEL ROSS pH Comb. electrode

- 1) ORION electrode meter model 720A 003368
- 2) ESC 440 multichannel potentiostat 9029127
- 3) AUSTIN 386SX
- 4) WORK BENCH SOFTWARE - Strawberry tree INC.

(See equipment list on page 849)

N. J. Smith
 12/1/93

12/6/93

EQUIPMENT 10/92 - 7/93

PH METERS

ORION EA 920 SN 3001A

ORION EA 940 ~~SN 230~~ SN 2330

ORION 720A SN 005885

FISHER ACCUMET 950 SN 3340

ELECTRODES - TSE / pH / SCE / Ag/AgCl /

FISHER 13-620-96 PN ELECTRODE SN 0260250

FISHER 13-620-~~900~~ 16 ATC PROBEORION 94-17 B Cl⁻ ION SELECTIVE ELECTRODE

ORION 90-02-00 REFERENCE ELECTRODE

FISHER SCE 13-620-51 SN's 9214083, 821163

0165415, 0169033, 3106337, 2134032, 3106340,
3106321, 0165403

FISHER Ag/AgCl REFERENCE 13-620-53 SN 8118182

MICROELECTRODES INC

MI506 FLEXABLE PN ELECTRODES SERIAL #'S 44817,
45147, 43715, 44060, 49445MI200 Cl⁻ ION SELECTIVE ELECTRODES SERIAL #'S

44199, 44447, 47226, 47239, 47233

47228, 44176

MI402 / MI403 Ag/AgCl REFERENCE ELECTRODES SERIAL #'S

49467, 46024, 43647, 43636, 44671, 45860,

44628, 44629, 41432, 41436, 45867, 46104

MERCURY THERMOMETERS FISHER SCIENTIFIC

SERIAL #'S ASSIGNED BY CALIBRATION LABORATORY

AT INITIAL CALIBRATION SERIAL #'S

1238001, 1238002, 1238004, 183305, 183301, 183303,

183302, 183304, 183306, ~~183~~ ~~00~~ 0323002

0323003, 0323004, 0323005, 0323007, 0323008

COMPUTERS

COMPUADD 333E

COMPAQ 386/20

COMPUADD 212

12/6/93

EQUIPMENT 10/92 - 7/93

POTENTIOSTATS

ESC 440 #1 SN 9029127

ESC 440 #2 ~~SN 9138~~ ~~DO~~ SN 9209138

ESC 410 POWER SUPPLY / POTENTIOSTAT 9105504 / 9105556

ESC 410 POWER SUPPLY / POTENTIOSTAT 9029495 / 9105557

ESC 410 POWER SUPPLY / POTENTIOSTAT 9105503 / 9029548

ELECTRONIC BALANCE SARTORIUS MC1 RESEARCH

RC 210 P SN 10704379

METERS / ELECTROMETERS

KEITHLEY 614 SN 555368

KEITHLEY 614 SN 467374

KEITHLEY 617 SN 537418

KEITHLEY 485 SN 509163

FLUKE 8050A SN 5005078

FLUKE 8050A SN 5005110

CONDUCTIVITY METER YSI MODEL 35 SN 900014379

DIAPCELLS $K = 0.1 \text{ cm}^{-1}$ FISHER 3992 $K = 1.0 \text{ cm}^{-1}$ FISHER 4062

STRIP CHART RECORDERS ABB (ACSA BROWN BOUGRI)

SE120 SN 0515265

SE120 SN 0049616

THERMOCOUPLE METER OMEGA MODEL HH22

SN T 94140

OVENS: FISHER ISOTEMP MODEL 838F SN 00300001

BLUE-M STABIL-THERM MODEL-OV-510A-3

SN OV-5647

LINDBERG MODEL 51828 SN 909027

SHARING WATER BATH FISHER VERSABATH MODEL 236

SN 90100023

WORKBENCH PC SOFTWARE V 2.0.4 1990

QUATTRO PRO V 3.01 DA246H0021600

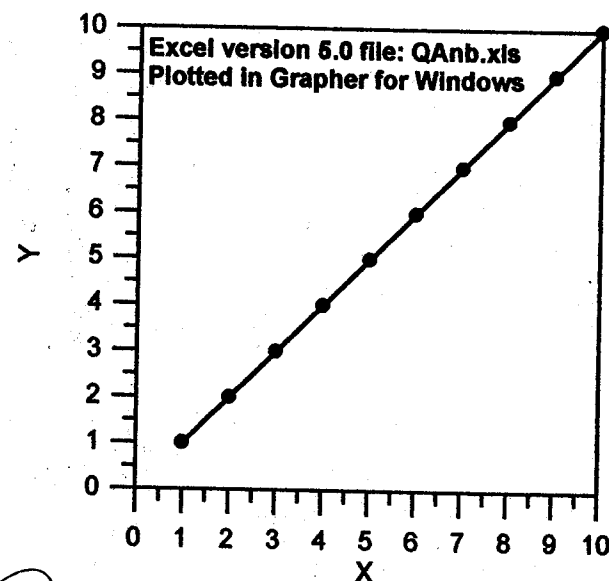
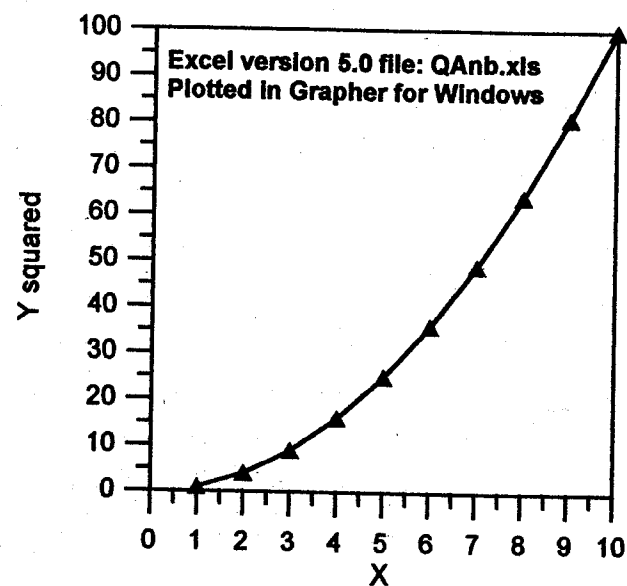
REACTION KETTLES: 2, 1-PIECE, 2-LITER REACTION KETTLES

w/GROUND GLASS JOINTS AND 4, 2-PIECE, 1-LITER

REACTION KETTLES WITH GROUND GLASS JOINTS

D. J. D. 12/6/93

INSTALLATION TEST DOCUMENTATION FOR MICROSOFT EXCEL VERSION 5.0 AND GRAPHER FOR WINDOWS VERSION 1.23



X	Y	Ysquared
1	1	1
2	2	4
3	3	9
4	4	16
5	5	25
6	6	36
7	7	49
8	8	64
9	9	81
10	10	100

David D 4/1/97

David D 4/1/97

David D 4/1/97

INTERFERENCE

Purpose: The purpose of this experiment is to determine whether several species can interfere with the CO_2 or Cl^- electrodes.

Several sample matrices were generated for different levels of HCO_3^- , Cl^- , SO_4^{2-} , F^- , NO_3^-

	HCO_3^-	Cl^-	SO_4^{2-}	NO_3^-	F^-	LABEL (PPM)
*	88	6	20	10	2	I 1 A-C
	88	10,000	20	10	2	I 2
	88	6	10,000	10	2	I 3
	88	6	20	10,000	2	I 4
	88	6	20	10	200	I 5
*	8800	6	20	10	2	II 1 A-C
	8800	10,000	20	10	2	II 2
	8800	6	10,000	10	2	II 3
	8800	6	20	10,000	2	II 4
	8800	6	20	10	200	II 5

* 3 solutions A, B, C

These solutions were prepared using stock solutions and dry chemicals (HCO_3^- & high conc)

F^-	1000 ppm	SN CNWRA	013-107	7/1/90	7/1/91
NO_3^-	1000 ppm	SN CNWRA	019-63	3/21/91	3/21/92
SO_4^{2-}	1000 ppm	SN CNWRA	019-97	4/10/91	4/10/92
Cl^-	600 ppm	SN CNWRA	013-10	7/1/90	7/1/91
NaHCO_3	dry	Lot no	897789		
NaNO_3	dry	Lot no	897674		
Na_2SO_4	dry	Lot no	901213		

The pH, ppm CO₂, ppm Cl⁻ were measured using electrode procedures.

The pH was measured using an Orion combination pH electrode.

CO₂ ppm was measured using ASTM D513-88 test method A and an Orion combination carbon dioxide electrode model 95-02.

Cl⁻ ppm was measured using an Orion model 94-17B Chloride electrode.

	CO ₂ ppm	Cl ⁻ ppm	pH
I 1-A	67.3	10.6	8.168
-B	67.8	9.3	8.345
-C	68.7	10.1	8.410
I 2	74.1	(1:100) 110	8.157
I 3	73.8	17.5 *	8.091
I 4	72.3	8.5	8.510
I 5	78.0	7.6	8.132
	1:20 dil	2.40 TAP	
II 1-A	298	7.48	8.209
-B	322	8.56	8.290
-C	338	8.60	8.275
II 2	334	(1:100) 125	8.050
II 3	327	7.80	8.129
II 4	339	8.10	8.144
II 5	365	8.31	8.245

* Recheck I 3 - new solution

I 3	73	8.6	8.177
	HCO ₃ ⁻ (ppm)	Cl ⁻ ppm	pH
I.	99.4 ± 5.5	9.12 ± 1.11	8.259 ± 0.161
II.	9200 ± 560	8.14 ± .44	8.192 ± 0.088

4/28/24

6/14/91

ASTM G61 - Multichannel Potentiometer w/WORK BENCH™

Purpose: The purpose of this experiment was to test work bench software and multichannel potentiometer. 9029127.
(Trial Run not verification)

ASTM TEST G61 procedures were done using work bench software and program ASTM G61. The measured potential and current density were logged using the same program into file G61.DAT.DAT. WORKSHEET ASTM G61 and data file G61.DAT.DAT are saved on work bench - disk 1 (F) (3 1/2" disk.)

A 386SX AST^{TOP} AUSTIN PC with DAC and math coprocessor were used to run the software.

JCP 6/14/91

LTPT - Long-term Pitting Test - Initial Entry

Experimenters: Thomas Page, Narasi Sridhar, Walter Machowski

Procedure: The following series of tests follow TOP-008. Specific stepping progression and testing procedures follow Memorandum included below

MEMORANDUM

To: Tom Page/Walt Machowski

From: N. Sridhar

Date: June 26, 1991

Subject: Long-term Pitting Tests on 304L stainless Steel

Please conduct the following tests using the Multi-channel potentiostat and Workbench PC:

1. Prepare two liters of solution per TOP-010:
1000 ppm Cl, 10 ppm NO₃, 20 ppm SO₄, 2 ppm F, 85 ppm HCO₃, all as sodium salts
2. All tests at 95°C.
3. Prepare samples by polishing in 600 grit paper, washing in distilled water and acetone and drying.
4. Deaerate solution by bubbling nitrogen for 1 hour before test and throughout the test at a slower rate (say about a bubble every 2 seconds)

Test 1

5. Introduce sample after solution reaches 95°C. Maintain at open-circuit for 1 hour. Monitor potential and record.
-50
6. Polarize sample to ~~10~~ mV SCE for 30 minutes. Record potential and current density (30 sec. interval).
7. Increase the potential to +200 mV vs. SCE, record current density and potential for 1 hour. (30 sec)
8. Decrease potential in steps of 20 mV, while monitoring current density at each potential. If the current starts increasing with time, decrease the potential to the next step. When the current starts decreasing with time, stop stepping potential down and maintain potential and record current and potential for 48 hours.

More

Test 2:

1. Use another 304L sample. Repeat steps 5 and 6.
2. increase potential to 200 mV vs. SCE and hold for 4 hours.
3. Repeat step 8.

After test, rinse sample and store with appropriate designation.

The concentration of Cl^- , NO_3^- , SO_4^{2-} , F^- , HCO_3^- will be varied from trial to trial

Equipment:

Workbench PC software on 386 SX PC

ESC 440 multichannel Potentiostat SW # 9029127

CALIB: 1/16/91 → 7/16/91

Standard Calomel Electrode - Check before use TOP-008-5.5

Platinum Counter Electrode TOP-008-5.84^{TRP}

Standard Test Cell TOP-008-5.1

Mettler AE 240 electronic balance S/N 101237

CALIB: 5/16/91 → 11/91

Micrometer: Calib: 4/19/91 → 10/19/91 SW 20-M-1

Orion pH meter - Calib before use

Specific Program for each test

Keithley 614 electrometer 12/31/90 → 7/1/91 SW 13897

Samples: Prepared as described in TOP-003-001

JCP
6/28/91

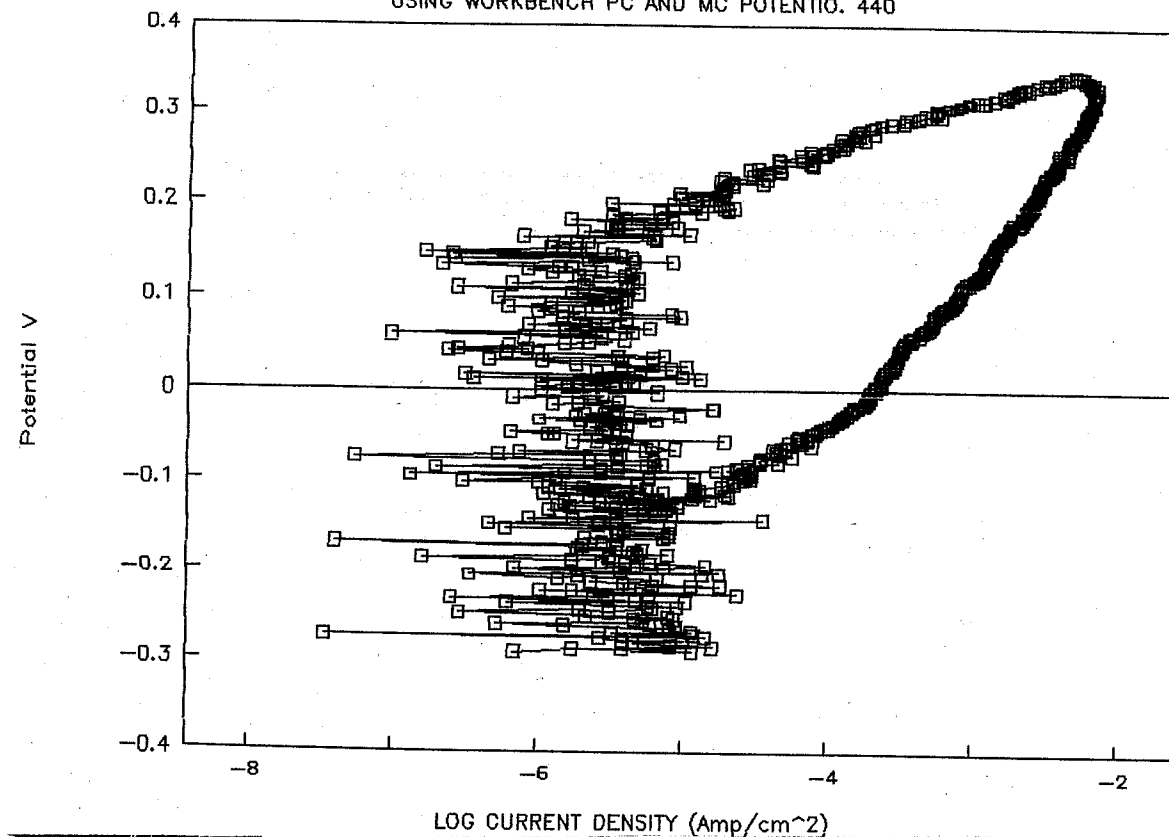
Tests terminated due to necessary changes in equipment. Data will not be used.

N.S. 8/21/91

N. Smith
4/28/94

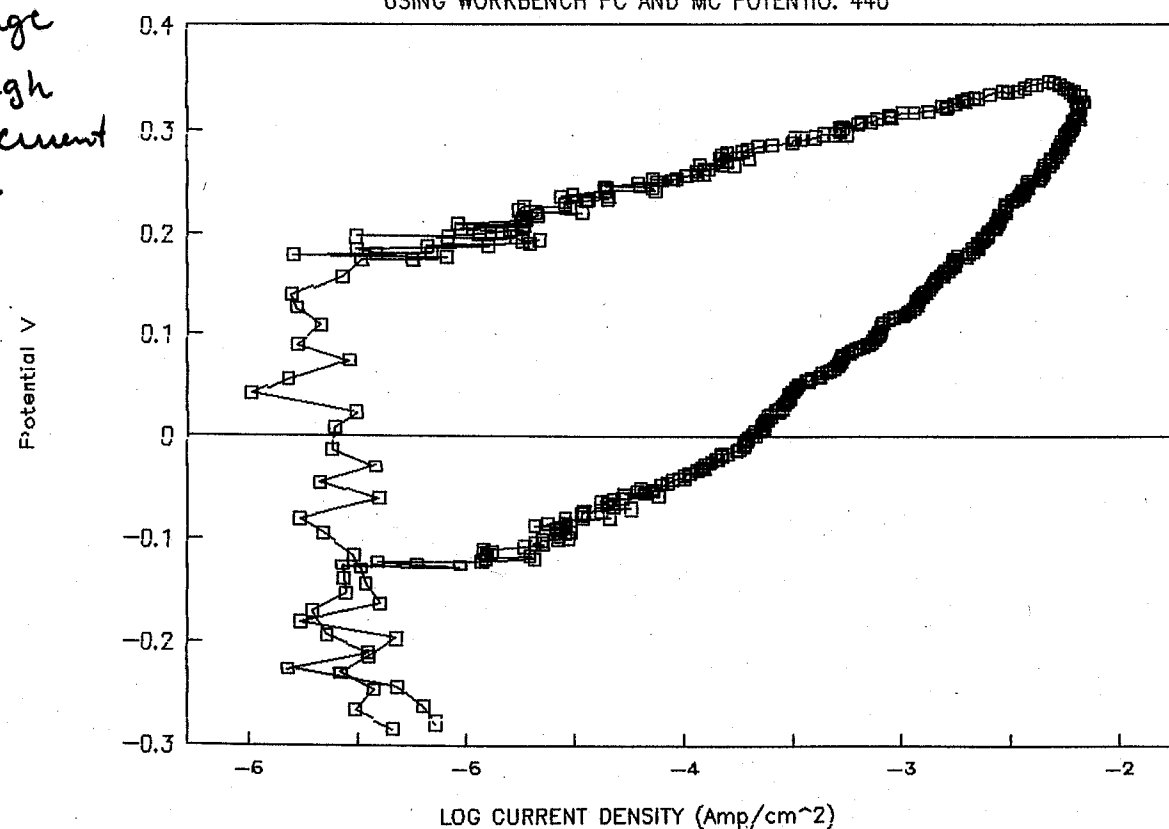
ASTM G61 VERIFICATION TEST

USING WORKBENCH PC AND MC POTENTIO. 440



ASTM G61 VERIFICATION TEST

USING WORKBENCH PC AND MC POTENTIO. 440



ten point
average
through
low-current
region.

LTPT - Verification G61

Purpose: To verify operation of equipment, software, and procedures for running cyclic potentiodynamic tests. The test serves as a check for the initiation of LTPT tests using ESC 440 multichannel potentiostat and workbench PC software.

The test was in accordance with TOP-008-6.1 and ASTM G-61. A cylindrical 304 LSS specimen from heat # T0954 was used. The NaCl solution was prepared from Fisher NaCl lot no. 885407. Argon (99.999%) was used for de-aeration.

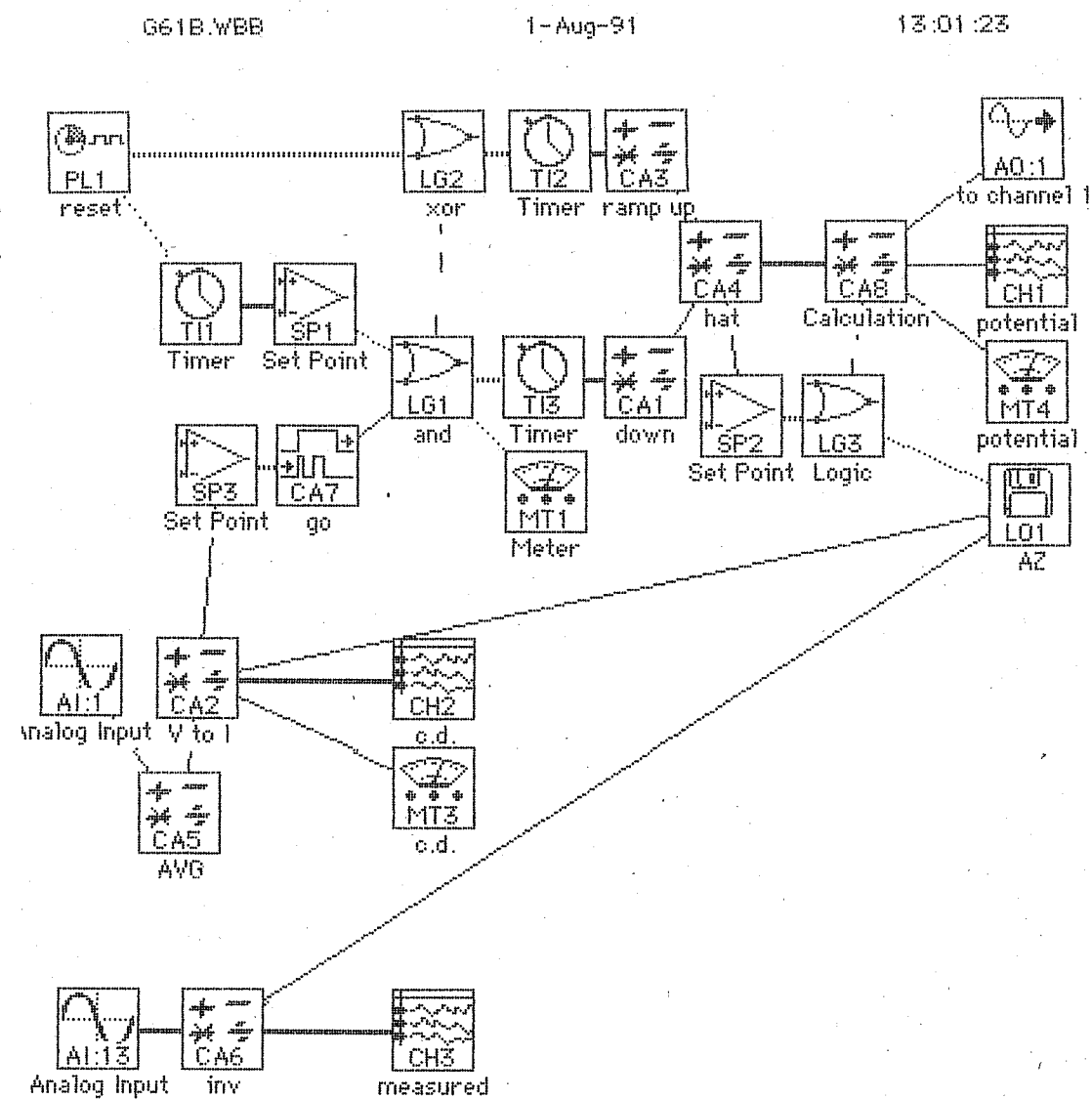
Spec. Dim.: ϕ 0.634 cm l = 1.909 cm A = 4.118 cm²

Pt potential 10 min before start +134 mV vs SCE
 E_{corr} at start -300 mV vs SCE

SCE was checked vs SCE - difference < 1.0 mV
Data saved on Disk - Workbench - Disk 1 (E)
in file ASTM G61.DAT

Test program is saved in ASTM G61.B.WBB on same disk.

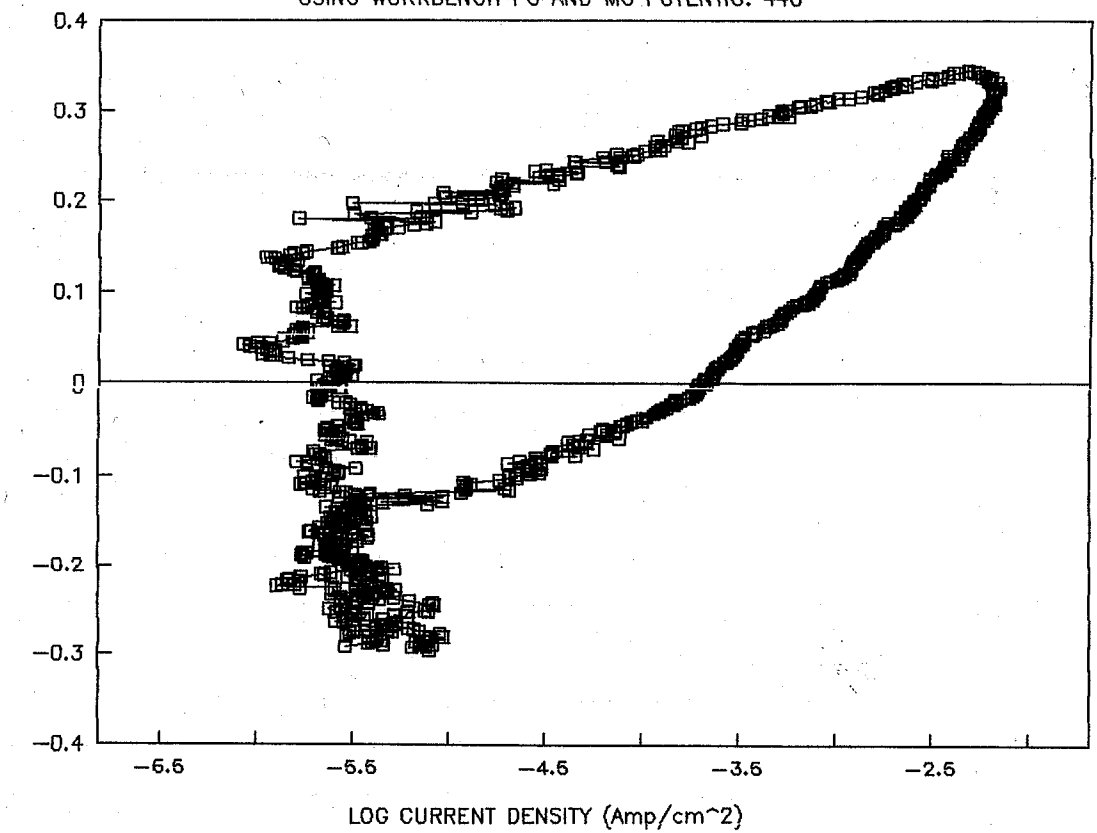
Thomas C Page
6/20/91



ASTM G61 VERIFICATION TEST

USING WORKBENCH PC AND MC POTENTIO. 440

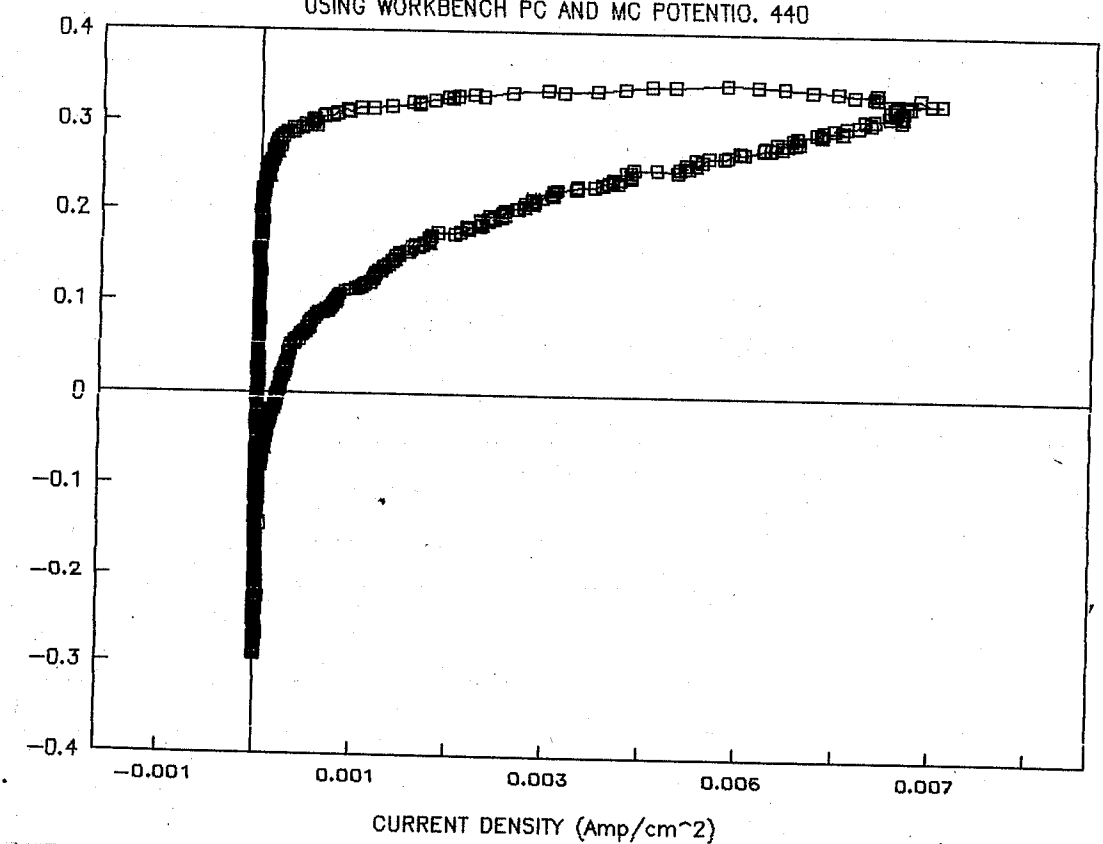
Potential V



ASTM G61 VERIFICATION TEST

USING WORKBENCH PC AND MC POTENTIO. 440

Potential V

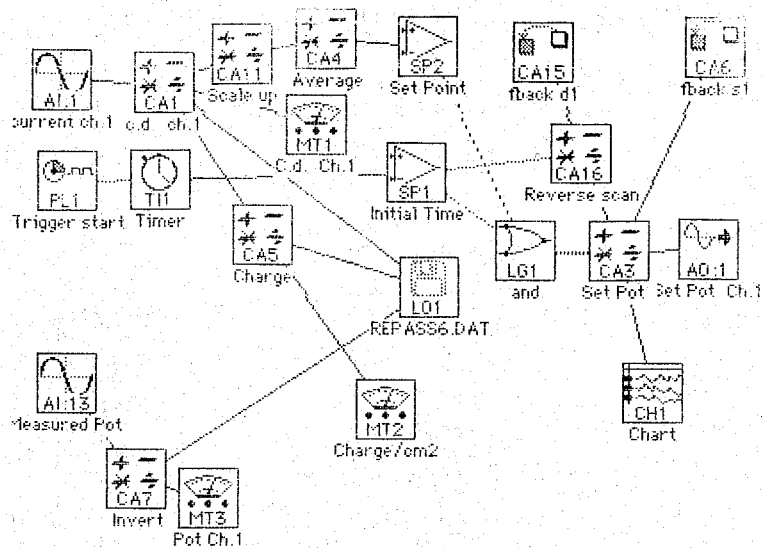


REPASS.WEE

12-Nov-91

09:15:45

Page 1



Worksheet Name: REPASS.WBB
 Hardware list:

Name	AI's	AO's	DIO's	CT's	DI's	DO's
STI ACPC-16	16	0	16	0	0	0
STI ACAO-12	0	6	8	0	0	0

Maximum icons: 464
 Grid size: 16
 Snap to grid: < disabled >
 Report Unsynch: < disabled >
 Fast Mode: < disabled >
 Fast Mode Samples: 1000
 Fast Mode Rate: 1.0 Kilohertz
 Com ports: 2

Port: COM 1
 Comment: Mouse connected

Port: COM 2
 Baud rate: 9600
 Data bits: 8
 Stop bits: 1
 Duplex: Half
 Parity: None
 XonXoff: < disabled >
 Echo wait: < disabled >
 Line delay: < disabled >

IEEE: < disabled >

Type: Analog Input

Name: AI:1 current ch.1
 Card Type: STI ACPC-16
 Channel Number: 1
 Range: +/-Auto V
 Resolution: Lo Noise (17ms)
 Output Type: Voltage
 Sample rate: 10.0 Hertz
 Fast Mode: < disabled >
 Inputs:
 < None >
 Outputs:
 CA1 c.d. ch.1

Name: AI:13 Measured Pot
 Card Type: STI ACPC-16
 Channel Number: 13
 Range: +/-Auto V
 Resolution: Lo Noise (17ms)
 Output Type: Voltage
 Sample rate: 10.0 Hertz
 Fast Mode: < disabled >
 Inputs:
 < None >
 Outputs:
 CA7 Invert

Type: Timer

Name: TI1 Timer
 Inputs:
 PL1 Trigger start
 Outputs:
 CP1 Initial Time

Type: Pulse

Name: PL1 Trigger start
 High Duration: 24.0 Hours
 Low Duration: 0.1 Seconds
 Start Value: Low
 Reset on exit: enabled
 Inputs: < None >
 Outputs: T11 Timer

Type: Calculation

Name: CA1 c.d. ch.1
 Function: aX / bY
 X input: AI:1 current ch.1
 Y input: 4.1
 "a" constant: -1.0
 "b" constant: 10.0
 "c" constant: 0.0
 Inputs: AI:1 current ch.1
 Outputs: MT1 C.d. Ch.1 CA5 Charge CA11 Scale up
 LO1 REPASS6.DAT

Name: CA3 Set Pot
 Function: $aX + bY + c$
 X input: LG1 and
 Y input: CA16 Reverse scan
 "a" constant: -0.02
 "b" constant: 1.0
 "c" constant: 0.6
 Inputs: LG1 and CA16 Reverse scan
 Outputs: AO:1 Set Pot Ch.1 CH1 Chart CA6 fback s1

Name: CA4 Average
 Function: Ave(X) for last (a) seconds
 X input: CA11 Scale up
 Y input: 0.0
 "a" constant: 2.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: CA11 Scale up
 Outputs: SP2 Set Point

Name: CA5 Charge
 Function: Integral X dt
 X input: CA1 c.d. ch.1
 Y input: 0.0
 "a" constant: 0.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: CA1 c.d. ch.1
 Outputs: MT2 Charge/cm2 LO1 REPASS6.DAT

Name: CA6 fback s1
 Function: Global Feedback(a:address)

X input: CA3 Set Pot
 Y input: 0.0
 "a" constant: 1.0
 "b" constant: 0.0
 "c" constant: 0.0

Inputs:

CA3 Set Pot

Outputs:

< None >

Name: CA7 Invert
 Function: $aX + bY$
 X input: AI:13 Measured Pot
 Y input: 0.0
 "a" constant: -1.0
 "b" constant: 0.0
 "c" constant: 0.0

Inputs:

AI:13 Measured Pot

Outputs:

MT3 Pot Ch.1

LO1 REPASS6.DAT

Name: CA11 Scale up
 Function: $aX + bY$
 X input: CA1 c.d. ch.1
 Y input: 0.0
 "a" constant: 10000.0
 "b" constant: 0.0
 "c" constant: 0.0

Inputs:

CA1 c.d. ch.1

Outputs:

CA4 Average

Name: CA15 fback d1
 Function: Global Feedback(a:address)
 X input: 0.0
 Y input: 0.0
 "a" constant: 1.0
 "b" constant: 0.0
 "c" constant: 0.0

Inputs:

< None >

Outputs:

CA16 Reverse scan

Name: CA16 Reverse scan
 Function: $c(X + a)(Y + b)$
 X input: CA15 fback d1
 Y input: SP1 Initial Time
 "a" constant: -0.6
 "b" constant: 0.0
 "c" constant: 1.0

Inputs:

CA15 fback d1

SP1 Initial Time

Outputs:

CA3 Set Pot

Type: Set Point

Name: SP1 Initial Time
 Function: $X > Y$
 X input: TI1 Timer
 Y input: 120.0
 Dead Band: 0.0
 Inputs:

T11 Timer

Outputs: LG1 and CA16 Reverse scan
 Name: SP2 Set Point
 Function: X > Y
 X input: CA4 Average
 Y input: 0.5
 Dead Band: 0.05
 Inputs: CA4 Average
 Outputs: LG1 and

Type: Logic

Name: LG1 and
 Function: X AND Y
 X input: SP1 Initial Time
 Y input: SP2 Set Point
 Inputs: SP1 Initial Time SP2 Set Point
 Outputs: CA3 Set Pot

Type: Log

Name: LO1 REPASS6.DAT
 Log Status: < disabled >
 Sample Rate: 30.0 Seconds
 Gate: < None >
 Data Format:
 Heading: Repassivation Pot, Ch.1
 File Path: C:\WB
 File Name: REPASS6.DAT
 Date Stamp: enabled
 Time Stamp: enabled
 Inputs: CA7 Invert CA1 c.d. ch.1 CA5 Charge
 Outputs: < None >

Type: Meter

Name: MT1 C.d. Ch.1
 Output Type: Exponential
 Units: A/cm2
 Integer: 8
 Decimal: 1
 Inputs: CA1 c.d. ch.1
 Outputs: < None >

Name: MT2 Charge/cm2
 Output Type: Exponential
 Units: Coul/cm2
 Integer: 8
 Decimal: 1
 Inputs: CA5 Charge
 Outputs: < None >

Name: MT3 Pot Ch.1
 Output Type: Fixed Point

Integer: 6
Decimal: 3
Inputs:

CA7 Invert

Outputs:
< None >

Type: Chart

Name: CH1 Chart
Chart Color: White
X Axis Label: Minutes
X Axis Min: 0.0
X Axis Max: 10.0
Y Axis Label: < None >
Y Axis Min: -10.0
Y Axis Max: 10.0

Inputs:
CA3 Set Pot
Outputs:
< None >

Type: Analog Output

Name: AO:1 Set Pot Ch.1
Card Type: STI ACAO-12
Channel Number: 1
Range: +/- 5 Volts
Resolution: 0.024%

Inputs:
CA3 Set Pot
Outputs:
< None >

Pitting Protection Potential Tests

Description: The pitting protection potential tests are run in accordance with the Workbench PC software using a program called REPASS.WBB. This program is documented on p. 22. The ESC 440 multichannel potentiostat and an Austin 386 SX computer are used to run the program and to control the potentials.

The program works in the following manner. A selected potential is applied to the specimen for an initial time period. If after the initial time period the current density is above a selected maximum value, the applied potential will decrement by a selected amount. If the current threshold is still exceeded, it will decrement another step, until the current threshold remains below the selected threshold value. This potential at which the current no longer continues to increase above the selected threshold, is the pitting protection potential.

Set-up: The cells are 1-L reaction flasks w/ covers that allow penetrations for a platinum counter electrode, a specimen holder, a reference salt bridge (about 6x6 type modified 6x6 type w/ h. a gas sparging tube.

The reference electrode is kept cooled w/ a small condenser, an Allihn condenser refluxes the entire test cell, and a water trap is used in the gas exit line to prevent back streaming of oxygen. The cell is heated in a heating mantle using a rheostat controller and reading internal temperature w/ a thermometer in a thermowell in the cover.

w/ Prochowski
9/11/91

P3TSS001.DAT

Purpose: To determine the pitting protection potential as it may vary with propagation time and initial applied potential.

Spec. Prep: A long cylindrical (10 cm² area) specimen was wet polished w/320 followed by 600 grit paper. It was then cleaned in an ultrasonic bath for 5 minutes, rinsed w/DI water, rinsed w/acetone and dried under a hot air gun.

Solution Prep: To 500 ml of 17 MΩ water in a 2-L volumetric add 0.2452 g of NaHCO₃ (Fisher lot # 897789) and 3.2922 g of NaCl (Fisher lot # 885407). Pipet 40.0 ml of S₀ stock, 20.0 ml of NO₃ stock, and 4.0 ml of F stock solutions (#019-137, #019-199, and #019-191). Dilute to the mark.

Set-Up: The cell described on p.23 was used. 900 ml of test solution was used, the salt bridge was filled w/test solution. The specimen was immersed approximately with 60-70% of the length immersed to leave the crevice out of the solution. Ref SCE #1 was used + checked vs another SCE, difference was less than 1.0 mV. Nitrogen (hi-purity) used for sparging.

- ① Wt initial 11.3503 g Wt final not taken
- ② de-aeration for one hour before spec immersion
- ③ Hold at open circuit one hour after immersion
- ④ E can before applying potential -516 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 3600 secs. (1 hr) +600 mV w/Marked 9/11/91
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.38 @25°C Final pH 9.23 @25°C
- ⑨ Imm. area Immersed area ≈ not taken
- ⑩ Final E -987 mV vs SCE

Data stored on disk PPPT #1

② specimen misplaced

Walter J. Machowski
9/11/91

P3TSS002.DAT

Purpose: Same as P3TSS001

Sample Prep: Same as p.25Solution Prep: Same solution used as prepared for P3TSS001 (p.25).Set-Up: Identical to P3TSS001 (p.25), except ref SCE #4 was used.

- ① Initial WT 11.3682 g Final WT: not taken*
- ② de-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min -725 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial Time 7200 secs (2 hrs) +600 mV vs SCE WJ Machenski 9/11/91
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.38 @ 25°C Final pH 9.32 @ 25°C
- ⑨ Immersed area \approx wt taken*
- ⑩ Final E -1106 mV vs SCE

Data stored on disk PPPT #1

⑩ specimen misplaced

Walter J. Machenski
9/11/91

Pitting Protection Potential Test on 316L SS
(# P80746)

P3TSS003.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described in p.25 from same lot # chemicals + solutions. Actual salt wts. used were 0.2451 g NaHCO_3 and 3.2930 g NaCl .

Set-Up was as described on p.25. Ref SCE #1 was used.

- ① Initial wt. 11.3880 g Final wt. 11.3444 g
- ② De-aerate for one hour before immersion.
- ③ Hold at open circuit for one hour after immersion.
- ④ E com after 60 min -560 mV vs SCE
- ⑤ Test temp 95°C w/machwshi 9/12/91
- ⑥ Initial time ~~14,400 secs (4 hrs)~~ 3600 secs
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.38 @ 25°C Final pH 9.21 @ 25°C
- ⑨ Immersed area $\cong 7.22 \text{ cm}^2$ $l=3.462 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E -165 mV vs SCE

Stored on disk PPPT #1

Walter J. Machwshi
9/12/91

Pitting Protection Potential Test on 316L SS (# P80746)

P3TSS004.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Same solution used as in P3TSS003.DAT.

Set-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt. 11.4129 g Final wt. 11.3330 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion.
- ④ E com after 60 min. -753 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 7200 secs (2 hrs) +300 mV w/machwshi 9/12/91
- ⑦ Applied initial potential +100 mV vs SCE
- ⑧ Initial pH 8.38 @ 25°C Final pH 9.33 @ 25°C
- ⑨ Immersed area $\cong 6.23 \text{ cm}^2$ $l=2.965 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E -548 mV vs SCE

Stored on disk PPPT #2

Walter J. Machwshi
9/12/91

Pitting Protection Potential Test on 316L SS (lot # P80746)

P3TSS005.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.25 from same lot # chemicals + solutions. Actual salt wts. were 0.2447 g NaHCO_3 and 3.2918 g NaCl .

Set-Up: As described on p.25. Ref SCE #1 used.

- ① Initial wt. 11.4265 g Final wt. 11.2915 g
- ② de-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E com after 60 min -717 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.41 @ 25°C Final pH 9.27 @ 25°C
- ⑨ Immersed area $\cong 7.17 \text{ cm}^2$ $l=3.441 \text{ cm}$ $\phi=0.634 \text{ cm}$
- ⑩ Final E -153 mV vs SCE

Stored on disk PPPT #1

Walter J. Machorsch
9/13/91

Pitting Protection Potential Test on 316L SS (lot # P80746) 31

P3TSS006.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: ^{aglaachumli 9/13/91} Two liters same solution as on p.30 was used.

Set-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt. 11.3968 g Final wt. 11.3818 g
- ② de-aerate for one hour before immersion
- ③ Hold at open circuit after immersion
- ④ E com after 60 min -483 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial time 7200 secs (2 hrs)
- ⑦ Applied initial potential +100 mV vs SCE
- ⑧ Initial pH 8.41 @ 25°C Final pH 9.56 @ 25°C
- ⑨ Immersed area $\cong 6.64 \text{ cm}^2$ $l=3.168 \text{ cm}$ $\phi=0.635$
- ⑩ Final E -1169 mV vs SCE

Stored on disk PPPT #1

Walter J. Machorsch
9/13/91

Pitting Protection Potential Test on 316L SS
(~~lot~~ # P80246)

P3TSS007.DAT

Purpose: Same as P3TSS001.DAT.

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.25 from the same lot # chemicals + solutions.
Actual salt wts. were 0.2448 g NaHCO_3 and 3.2931 g NaCl .

Set-Up: As described on p.25. Ref SCE #1 was used.

- ① Initial wt. 11.3656 g Final wt. 11.3683 g
- ② de-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min. not recorded
- ⑤ Test temp 95°C
- ⑥ Initial time 14,400 secs (4 hrs)
- ⑦ Applied initial voltage +100 mV vs SCE
- ⑧ Initial pH 8.38 @ 25°C Final pH 9.27 @ 25°C
- ⑨ Immersed area $\cong 5.78\text{ cm}^2$ $l=2.739\text{ cm}$ $\phi=0.635\text{ cm}$
- ⑩ Final E +96 mV vs SCE

Stored on disk PPPT #1

No Pitting

Walter J. Macchowski
9/16/91

Pitting Protection Potential Test on 316L SS (~~lot~~ # P80246)

P3TSS008.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Used same solution as P3TSS007.DAT.

Set-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt. 11.3803 g Final wt. 11.3798 g
- ② de-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min. not recorded
- ⑤ Test temp 70°F (room temp)
- ⑥ Initial time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +400 mV vs SCE
- ⑧ Initial pH 8.38 @ 25°C Final pH 9.29 @ 25°C
- ⑨ Immersed area $\cong 8.23\text{ cm}^2$ $l=3.971\text{ cm}$ $\phi=0.634\text{ cm}$
- ⑩ Final E +401 mV vs SCE

Stored on disk PPPT #1

No Pitting

Walter J. Macchowski
9/16/91

Pitting Protection Potential Tests on INC 825
(Rt # HH4371FC)

P3TIN009.DAT

Purpose Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.25 from the same lot # chemicals and solutions. Actual salt wts were 0.2480 g NaHCO_3 and 3.2864 g NaCl .

Set-Up: As described on p.25. Ref SCE #1 was used.

- ① Initial Wt. 11.7427 g Final Wt. 11.7368 g
- ② De-aerate for one hour before immersion.
- ③ Hold at open-circuit for one hour after immersion.
- ④ E corr after 60 min. -581 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 3600 secs (2 hr)
- ⑦ Applied initial potential +600 mV vs SCE
- ⑧ Initial pH 8.45 @ 25°C Final pH 9.38 @ 25°C
- ⑨ Immersed area $\approx 8.23 \text{ cm}^2$ $l=3.972 \text{ cm}$ $\phi=0.634 \text{ cm}$
- ⑩ Final E +16 mV vs SCE

Stored on disk PPPT #1

Walter J. Mockowski
9/17/91

Pitting Protection Potential of INC 825
(Rt # HH4371FC)

P3TIN010.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Used same solution as P3TIN009

Set-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial Wt. 11.6526 g Final Wt. 11.5820 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E corr after 60 min -382 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial time 7200 secs (2 hrs)
- ⑦ Applied initial potential +600 mV vs SCE
- ⑧ Initial pH 8.45 @ 25°C Final pH 9.22 @ 25°C
- ⑨ Immersed area $\approx 7.19 \text{ cm}^2$ $l=3.448 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E -63 mV vs SCE

Stored on disk PPPT #1

Walter J. Mockowski
9/17/91

P3TIN011.DAT

Purpose: Same as P3TSS001.DATSample Prep: Same as p. 25Solution Prep: Two liters were prepared as described on p. 25 from the same lot # chemicals & solutions. Actual salt wts were 0.2451 g NaHCO_3 and 3.2898 g NaCl .Set-Up: As described on p. 25. Ref SCE #1 was used.

- ① Initial wt. 11.6215 g Final wt. 11.6090 g
- ② De-aerate for one hour before immersion.
- ③ Hold at open circuit for one hour before immersion.
W. J. MacKowski
9/23/91
- ④ E cor after 60 min. -531 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +600 mV vs SCE
- ⑧ Initial pH 8.35 @ 25°C Final pH 9.36 @ 25°C
- ⑨ Immersed area $\approx 7.64 \text{ cm}^2$ $l=3.671 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E -13 mV

Forgot to Start Logs; log started after ~4 hrs.One step drop was observed to ⁻¹³-1.3 mV vs SCE

Stored on disk PPPT #1

Walter J. MacKowski
9/23/91

P3TIN012.DAT

Purpose: Same as P3TSS001.DATSample Prep: Same as p. 25Solution Prep: Used same solution as P3TIN011.DAT.Set-Up: As described on p. 25. Ref SCE #4 was used.

- ① Initial wt. 11.7257 g Final wt. 11.7369 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min. -546 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 7200 secs (2 hrs)
- ⑦ Applied initial potential +400 mV vs SCE
- ⑧ Initial pH 8.65 @ 25°C Final pH 9.54 @ 25°C
- ⑨ Immersed area $\approx 7.55 \text{ cm}^2$ $l=3.628 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E +147 mV vs SCE

Forgot to Start Logs; log started after ~4 hrs. Two step drops were observed; 1st down to +207 mV and then a second down to +147 mV.

Stored on disk PPPT #1

Walter J. MacKowski
9/23/91

P3TIN013.DAT

Purpose: Re-run of P3TIN011.DAT due to log errorSample prep: Same as p.25Solution Prep: Two liters were prepared as described on p.25 from the same lot # chemicals + solutions. Actual salt wts were 0.2432 g NaHCO_3 and 3.2930 g NaCl .Set-Up: As described on p.25. Ref SCE #1 was used.

- ① Initial wt. 11.6649 g Final wt. 11.6532 g
- ② De-aerate for one hour before start ^{9/26/91} immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E con at 60 min -529 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +600 mV vs SCE
- ⑧ Initial pH 8.42 @ 25°C Final pH 8.86 @ 25°C
- ⑨ Immersed area $\approx 7.58 \text{ cm}^2$ $l=3.645 \text{ cm}$ $\phi=0.634 \text{ cm}$
- ⑩ Final E -53 mV vs SCE

Stored on disk PPPT #1.

Note 1

Initial time was 3 hours as shown in log.
not 4 hours as recorded in step 6

N. Endo/Kas
3/20/92

Walter J. Mackowski
9/26/91

P3TIN014.DAT

Purpose: Re-run of P3TIN012.DAT due to log error.Sample Prep: Same as p.25Solution Prep: Used same solution as P3TIN013.DATSet-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt. 11.5702 g Final wt. 11.5274 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E con after 60 min. -497 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 7200 secs (2 hrs)
- ⑦ Applied initial potential +400 mV vs SCE
- ⑧ Initial pH 8.42 @ 25°C Final pH 9.74 @ 25°C
- ⑨ Immersed area $\approx 6.30 \text{ cm}^2$ $l=3.001 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E +399 mV vs SCE

Stored on disk PPPT #1.

Walter J. Mackowski
9/26/91

Pitting Protection Potential Test on Luc 825
(Lot # HH4371FC)

P3TIN015.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.25 using the same lot # chemicals & solutions. Actual salt wt. used were 0.2445 g NaHCO_3 + 3.2930 g NaCl .

Set-Up: As described on p.25. SCE #1 was used.

- ① Initial Wt. 11.7285 g Final Wt. 11.7116 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min. -548 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +400 mV vs SCE
- ⑧ Initial pH 8.12 @ 25°C Final pH 9.71 @ 25°C
- ⑨ Immersed area $\approx 6.45 \text{ cm}^2$ $L=2.072 \text{ cm}$ $\phi=0.635 \text{ cm}$
- ⑩ Final E -1697 mV vs SCE

Solution dropped overnight to a level below that of the salt bridge type. Potential drop is probably due to this. D.I. Water was added & experiment continued for about 4 more hours.

Stored on disk PPPT #1

Ignore Test.

N. Smith
3/20/92

Walter J. Macchowski
10/1/91

Pitting Protection Potential Test on Luc 825
(Lot # HH4371FC)

P3TIN016.DAT

Purpose: Same as P3TSS001.DAT

Sample Prep: Same as p.25

Solution Prep: Used same solution as P3TIN015

Set-Up: As described on p.25. SCE #4 was used.

- ① Initial Wt. 11.6747 g Final Wt. 11.6644 g
- ② De-aerate for one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E cor after 60 min -352 mV vs SCE
- ⑤ Test Temp: room temp (70°F)
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +600 mV vs SCE
- ⑧ Initial pH 8.12 @ 25°C Final pH 8.98 @ 25°C
- ⑨ Immersed area $\approx 7.5 \text{ cm}^2$ (no mark for measurement)
- ⑩ Final E +607 mV vs SCE

Stored on disk PPPT #1.

Walter J. Macchowski
10/1/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS017.DAT

Purpose: Re-run of ... 003 to check reproducibility

Sample Prep: Same as p.25.

Solution Prep: Two liters were prepared as described on p.25 from the same lot # chemicals + solutions.
Actual salt wts. 0.2461 g NaHCO_3 and 3.2928 g NaCl .

Set-Up: As described on p.25. Ref SCE #1 was used.

- ① Initial WT. 11.4048 g Final WT. 11.3962 g
- ② De-aerate one hour before immersion
- ③ Hold at open circuit for one hour after immersion.
- ④ E can after 60 min -665 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 3600 secs (1-hr)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.15 @ 25°C Final pH 9.39 @ 25°C
- ⑨ Immersed area $\approx 7.97 \text{ cm}^2$ $l = 3.857 \text{ cm}$ $\phi = 0.635 \text{ cm}$
- ⑩ Final E -42 mV vs SCE

Stored on disk PPPT #1

Walter J. Mocharahi
9/7/91 wjm
10/7/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS018.DAT

Purpose: re-run of ... 004 to check reproducibility

Sample Prep: Same as p.25.

Solution Prep: Used same solution as P3TSS017.DAT.

Set-Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt 11.3943 g Final wt. 11.3655 g
- ② De-aerate one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E can after 60 min. -756 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 7200 secs (2 hrs)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.15 @ 25°C Final pH 2.36 @ 25°C
- ⑨ Immersed area $\approx 8.34 \text{ cm}^2$ $l = 4.023 \text{ cm}$ $\phi = 0.635 \text{ cm}$
- ⑩ Final E -163 mV vs SCE

Stored on disk PPPT #1

Walter J. Mocharahi
10/7/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS019.DAT

Purpose: Same as P3TSS001.DAT Except this test was run manually. See below.

Method: Initial potential applied and after the initial time the potential was lowered by 100 mV. The current density was monitored + if it was greater than 50 μA for 5 minutes, the potential was lowered 100 mV again. Again CD monitored and potential lowered 100 mV. The next decrement was to be 50 mV if CD > 50 μA for 10 minutes. The experiment was never allowed to go past this point because CD was too low after the 3rd decrement.

Solution Prep: Two liters were prepared as on p. 25 using same lot # chemicals and solutions. Actual salt weights were 0.2444g NaHCO_3 and 3.2931g NaCl .

Set-Up: As described on p. 25. Ref SCE #1 used

Specimen Prep: Same as p. 25

- ① Initial WT. 11.4269g Final WT. 11.4012g
- ② de-aerate one hour before immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E corr after 60 min -446 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Initial time 3600 secs (1 hr)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.12 @ 25°C Final pH 9.34 @ 25°C
- ⑨ Immersed area $\approx 7.62 \text{ cm}^2$ $l = 3.668$ $\phi = 0.634$
- ⑩ Final E -3 mV vs SCE

Once set to 0 mV, the CD was greater than 50 μA for about 60-75 seconds, slowly decaying to low values (10^{-7} A).

Stored on disk PPPT #1

Walter J. Kachurak
10/8/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS020.DAT

Purpose: Same as P3TSS019.DAT

Method: Same as on p. 44.

Solution Prep: Same solution used as P3TSS019.DAT.

Set-Up: As described on p. 25. Ref SCE #4 used.

Specimen Prep: Same as p. 25.

- ① Initial WT. 11.3851g Final Wt. 11.2521g
 - ② de-aerate for one hour before immersion
 - ③ Hold at open circuit for one hour after immersion
 - ④ E corr after 60 min -513 mV vs SCE
 - ⑤ Test temp ~~85°C~~ 95°C with 11/6/91
 - ⑥ Initial time 10,800 secs (3 hrs)
 - ⑦ Applied initial potential +300 mV vs SCE
 - ⑧ Initial pH 8.12 @ 25°C Final pH 9.68 @ 25°C
 - ⑨ Immersed area $\approx 7.96 \text{ cm}^2$ $l = 3.824$ $\phi = 0.635$
 - ⑩ Final E ?
- Once set to 0 mV, the CD was greater than 50 μA for about 3-4 minutes, slowly decaying to low values.

Stored on disk PPPT #1

Walter J. Kachurak
10/8/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80246)

P3TSS021.DAT

Purpose: This test is a re-run of P3TSS003. The program was modified to slow down the "decrementation". This is to compare results of the old program w/ the new. This program - REPASS1.WAB is documented on p. 50.

Sample Prep: Same as on p. 25.

Solution Prep: Two liters were prepared as described on p. 25 using the same chemical lot #'s and solutions. Actual salt weights were 0.2451 g NaH_2PO_4 and 3.2929 g NaCl .

Set-Up: As described on p. 25. Ref SCE #1 was used.

- ① Initial Weight 11.3532 g Final Weight N/A[Ⓢ]
- ② De-aerate one hour before immersion.
- ③ Hold at open circuit for one hour after immersion.
- ④ E com after 60 min -742 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 3600 secs (1 hr)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.14 @ 25°C Final pH 9.08 @ 25°C
- ⑨ Immersed area *
- ⑩ Final E -372 mV vs SCE

* Test aborted to excessive evaporation of test solution, which left reference probe out of liquid as well as excessively exposing test specimen above liquid level.

Data after 15105 seconds after start of polarization appears off due to the solution evaporation.

Walter J. Macchowski
11/8/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80246)

P3TSS022.DAT

Purpose: Same as p. 46. This is re-run of P3TSS004.

Sample Prep: Same as on p. 25.

Solution: Used same solution as on p. 46 ^{upward line}

Set-Up: As described on p. 25. Ref SCE #4 was used.
Run w/ REPASS1.WAB program.

- ① Initial WT. 11.4111 Final WT. Did not Measure ^{id}
- ② De-aerate for one hour before starting immersion
- ③ Hold at open circuit for one hour after immersion
- ④ E com after 60 min. -768 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Initial Time 7200 secs (2 hrs)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.14 @ 25°C Final pH 9.12 @ 25°C
- ⑨ Immersed area $\approx 9.55 \text{ cm}^2$ $l = 4.036 \text{ cm}$ $\phi = 0.634 \text{ cm}$
- ⑩ Final E -1 mV vs SCE

Stored on disk PPT #1

C.d. did not drop below $3 \times 10^{-5} \text{ A/cm}^2$.

Specimen sent for
metallographic examination

Walter J. Macchowski
11/8/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS023.DAT

Purpose: Same as p. 46.

Sample Prep: Same as p. 25.

Solution Prep: Two liters were prepared as described on p. 25 from the same lot # Chemicals & solutions, except the SO₂ stock was #019-231. Salt wts were 0.249 g NaHCO₃ and 3.2968 g NaCl.

Set-Up: As described on p. 25, Ref. SCE #1 was used
Run w/REPASSZ. WBB program

- ① Initial WT. 11.3996 g Final WT. 11.3726 g
- ② de-aerate one hour before immersion
- ③ Hold @ open circuit for one hour before start, after immersion
- ④ E corr after 60 min. -761 mV vs SCE
- ⑤ Test Temp. 25°C
- ⑥ Initial Time 3600 secs (1 hr)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.08 @ 25°C Final pH 9.53 @ 25°C
- ⑨ Immersed area $\approx 8.32 \text{ cm}^2$ $l = 4.012 \text{ cm}$ $\phi = 0.635 \text{ cm}$
- ⑩ Final E -13 mV

Stored on dish PPPT #2

Walter J Maclewski
11/13/91

Pitting Protection Potential Tests on 316L SS
(Lot # P80746)

P3TSS024.DAT

Purpose: Same as p. 46.

Sample Prep: Same as p. 25.

Solution: Same solution used as on p. 48

Set-Up: As described on p. 25 Ref. SCE #4 was used.
Run w/REPASSZ. WBB program

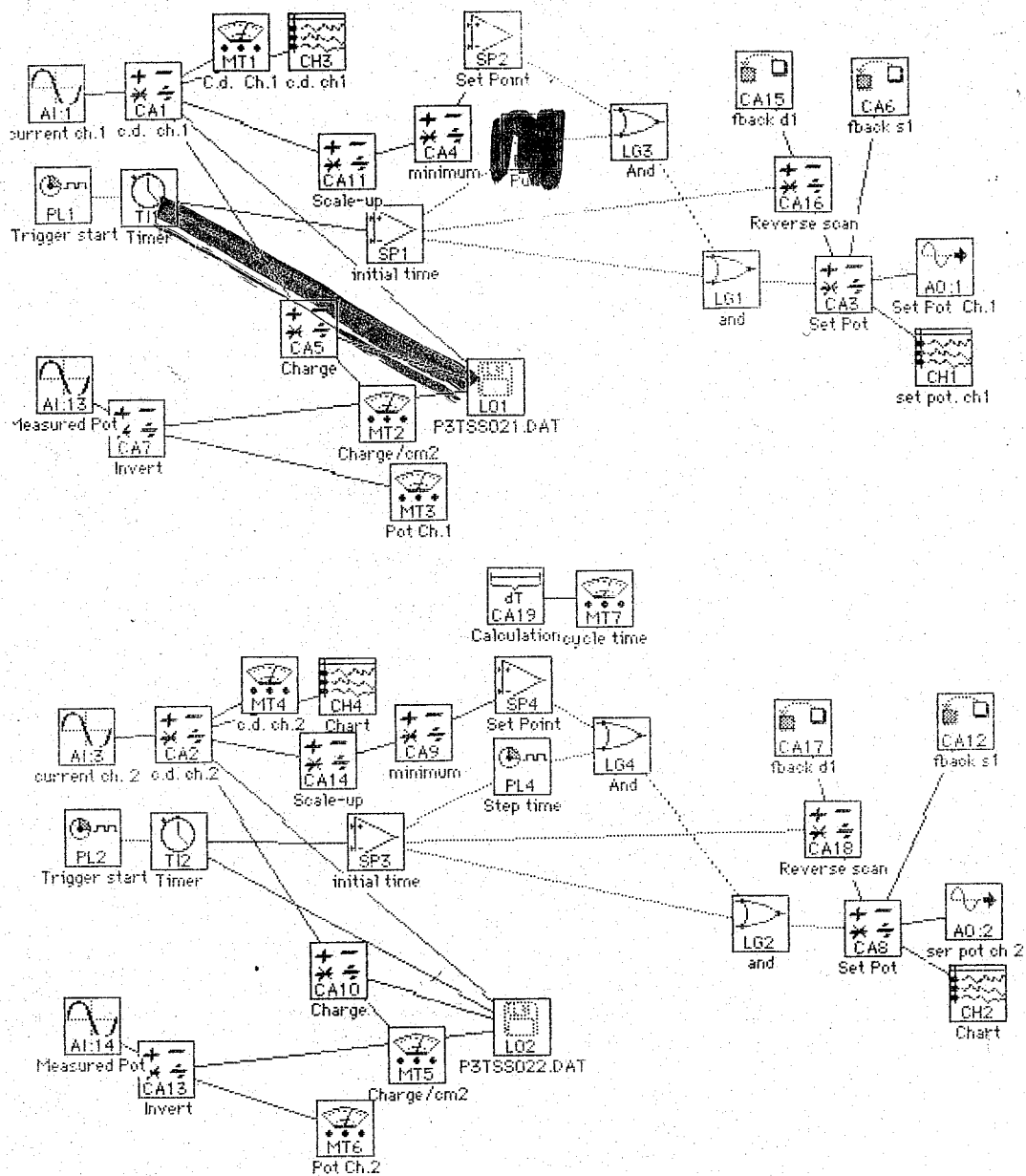
- ① Initial WT. 11.4075 g Final WT. 11.4445 g
- ② de-aerate one hour before immersion
- ③ Hold @ open circuit for one hour before immersion
- ④ E corr after 60 min. -743 mV vs SCE
- ⑤ Test Temp. 25°C
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +100 mV vs SCE
- ⑧ Initial pH 8.08 @ 25°C Final pH 9.51 @ 25°C
- ⑨ Immersed area $\approx 8.09 \text{ cm}^2$ $l = 3.903 \text{ cm}$ $\phi = 0.634$
- ⑩ Final E +98 mV

Stored on dish PPPT #7

Total Time at +100 mV sce was
76036 seconds. No change
in c.d. = $2 \times 10^{-6} \text{ A/cm}^2$.

N.S.

Walter J Maclewski
11/13/91



Worksheet Name: REPASS1.WBB
 Hardware list:
 Name
 STI ACPC-16
 STI ACAO-12
 Maximum icons: 448
 Grid size: 16
 Snap to grid: < disabled >
 Report Unsynch: < disabled >
 Fast Mode: enabled
 Fast Mode Samples: 1000
 Fast Mode Rate: 1.0 Kiloherztz
 Com ports: 2

Port: COM 1
 Comment: Mouse connected

Port: COM 2
 Baud rate: 9600
 Data bits: 8
 Stop bits: 1
 Duplex: Half
 Parity: None
 XonXoff: < disabled >
 Echo wait: < disabled >
 Line delay: < disabled >

IEEE: < disabled >

Type: Analog Input

Name: AI:1 current ch.1
 Card Type: STI ACPC-16
 Channel Number: 1
 Range: +/-Auto V
 Resolution: Lo Noise (17ms)
 Output Type: Voltage
 Sample rate: 10.0 Hertz
 Fast Mode: < disabled >
 Inputs:
 < None >
 Outputs:
 CA1 c.d. ch.1

Name: AI:3 current ch. 2
 Card Type: STI ACPC-16
 Channel Number: 3
 Range: +/-Auto V
 Resolution: Lo Noise (17ms)
 Output Type: Voltage
 Sample rate: 10.0 Hertz
 Fast Mode: < disabled >
 Inputs:
 < None >
 Outputs:
 CA2 c.d. ch.2

Name: AI:13 Measured Pot
 Card Type: STI ACPC-16
 Channel Number: 13
 Range: +/-Auto V
 Resolution: Lo Noise (17ms)
 Output Type: Voltage
 Sample rate: 10.0 Hertz

Fast Mode: < disabled >

Inputs:
< None >

Outputs:
CA7 Invert

Name: A1:14 Measured Pot
Card Type: ST1 ACPC-16
Channel Number: 14
Range: +/-Auto V
Resolution: Lc Noise (17ms)
Output Type: Voltage
Sample rate: 10.0 Hertz
Fast Mode: < disabled >
Inputs:

< None >

Outputs:
CA13 Invert

Type: Timer

Name: TI1 Timer

Inputs:
PL1 Trigger start

Outputs:
SP1 initial time LO1 P3TSS021.DAT

Name: TI2 Timer

Inputs:
PL2 Trigger start

Outputs:
SP3 initial time LO2 P3TSS022.DAT

Type: Pulse

Name: PL1 Trigger start

High Duration: 50.0 Hours

Low Duration: 0.1 Minutes

Start Value: Low

Reset on exit enabled

Inputs:
< None >

Outputs:
TI1 Timer

Name: PL2 Trigger start

High Duration: 50.0 Hours

Low Duration: 0.1 Seconds

Start Value: Low

Reset on exit enabled

Inputs:
< None >

Outputs:
TI2 Timer

Name: PL3 Pulse

High Duration: 1.0 Seconds

Low Duration: 180.0 Seconds

Start Value: Low

Reset on exit enabled

Inputs:
SP1 initial time

Outputs:
LG3 And

Name: PL4 Step time

High Duration: 0.1 Seconds
 Low Duration: 180.0 Seconds
 Start Value: Low
 Reset on exit enabled

Inputs:
 SP3 initial time
 Outputs:
 LG4 And

Type: Calculation

Name: CA1 c.d. ch.1
 Function: aX / bY
 X input: AI:1 current ch.1
 Y input: 7.5
 "a" constant: -1.0
 "b" constant: 10.0
 "c" constant: 0.0

Inputs:
 AI:1 current ch.1

Outputs:
 MT1 C.d. Ch.1
 CA11 Scale-up

CH3 c.d. chl
 LO1 P3TSS021.DAT

CA5 Charge

Name: CA2 c.d. ch.2
 Function: aX / bY
 X input: AI:3 current ch. 2
 Y input: 7.2
 "a" constant: -1.0
 "b" constant: 10.0
 "c" constant: 0.0

Inputs:
 AI:3 current ch. 2

Outputs:
 CH4 Chart
 CA14 Scale-up

MT4 c.d. ch.2
 LO2 P3TSS022.DAT

CA10 Charge

Name: CA3 Set Pot
 Function: aX + bY + c
 X input: LG1 and
 Y input: CA16 Reverse scan
 "a" constant: -0.01
 "b" constant: 1.0
 "c" constant: 0.3

Inputs:
 LG1 and

Outputs:
 AO:1 Set Pot Ch.1

CA16 Reverse scan

CH1 set pot. chl CA6 fback s1

Name: CA4 minimum
 Function: Min(X) for last (a) seconds
 X input: CA11 Scale-up
 Y input: 0.0
 "a" constant: 180.0
 "b" constant: 0.0
 "c" constant: 0.0

Inputs:
 CA11 Scale-up

Outputs:
 SP2 Set Point

Name: CA5 Charge
 Function: Integral X dt
 X input: CA1 c.d. ch.1
 Y input: 0.0
 "a" constant: 0.0

"b" constant: 0.0
 "c" constant: 0.0

Inputs:

CA1 c.d. ch.1

Outputs:

MT2 Charge/cm²

LO1 P3TSS021.DAT

Name:

CA6 fback s1

Function:

Global Feedback(a:address)

X input:

CA3 Set Pot

Y input:

0.0

"a" constant:

1.0

"b" constant:

0.0

"c" constant:

0.0

Inputs:

CA3 Set Pot

Outputs:

< None >

Name:

CA7 Invert

Function:

aX + bY

X input:

AI:13 Measured Pot

Y input:

0.0

"a" constant:

-1.0

"b" constant:

0.0

"c" constant:

0.0

Inputs:

AI:13 Measured Pot

Outputs:

MT3 Pot Ch.1

LO1 P3TSS021.DAT

Name:

CA8 Set Pot

Function:

aX + bY + c

X input:

LG2 and

Y input:

CA18 Reverse scan

"a" constant:

-0.01

"b" constant:

1.0

"c" constant:

0.3

Inputs:

LG2 and

CA18 Reverse scan

Outputs:

AO:2 ser pot ch 2

CH2 Chart

CA12 fback s1

Name:

CA9 minimum

Function:

Min(X) for last (a) seconds

X input:

CA14 Scale-up

Y input:

0.0

"a" constant:

180.0

"b" constant:

0.0

"c" constant:

0.0

Inputs:

CA14 Scale-up

Outputs:

SP4 Set Point

Name:

CA10 Charge

Function:

Integral X dt

X input:

CA2 c.d. ch.2

Y input:

0.0

"a" constant:

0.0

"b" constant:

0.0

"c" constant:

0.0

Inputs:

CA2 c.d. ch.2

Outputs:

MT5 Charge/cm²

LO2 P3TSS022.DAT


```

Name: CA11 Scale-up
Function: aX + bY
X input: CA1 c.d. ch.1
Y input: 0.0
"a" constant: 10000.0
"b" constant: 0.0
"c" constant: 0.0
Inputs:
  CA1 c.d. ch.1
Outputs:
  CA4 minimum

Name: CA12 fback s1
Function: Global Feedback(a:address)
X input: CA8 Set Pot
Y input: 0.0
"a" constant: 2.0
"b" constant: 0.0
"c" constant: 0.0
Inputs:
  CA8 Set Pot
Outputs:
  < None >

Name: CA13 Invert
Function: aX + bY
X input: AI:14 Measured Pot
Y input: 0.0
"a" constant: -1.0
"b" constant: 0.0
"c" constant: 0.0
Inputs:
  AI:14 Measured Pot
Outputs:
  MT6 Pot Ch.2

Name: CA14 Scale-up
Function: aX + bY
X input: CA2 c.d. ch.2
Y input: 0.0
"a" constant: 10000.0
"b" constant: 0.0
"c" constant: 0.0
Inputs:
  CA2 c.d. ch.2
Outputs:
  CA9 minimum

Name: CA15 fback d1
Function: Global Feedback(a:address)
X input: 0.0
Y input: 0.0
"a" constant: 1.0
"b" constant: 0.0
"c" constant: 0.0
Inputs:
  < None >
Outputs:
  CA16 Reverse scan

Name: CA16 Reverse scan
Function: c (X + a)(Y + b)
X input: CA15 fback d1
Y input: SP1 initial time
"a" constant: -0.3

```

LO2 P3TSS022.DAT

"b" constant: 0.0
 "c" constant: 1.0
 Inputs:
 CA15 fback d1 SP1 initial time

Outputs:
 CA3 Set Pot

Name: CA17 fback d1
 Function: Global Feedback(a:address)
 X input: 0.0
 Y input: 0.0
 "a" constant: 2.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs:
 < None >
 Outputs:
 CA18 Reverse scan

Name: CA18 Reverse scan
 Function: $c(X + a)(Y + b)$
 X input: CA17 fback d1
 Y input: SP3 initial time
 "a" constant: -0.3
 "b" constant: 0.0
 "c" constant: 1.0
 Inputs:
 CA17 fback d1 SP3 initial time
 Outputs:
 CA8 Set Pot

Name: CA19 Calculation
 Function: WB cycle time(msecs)
 X input: 0.0
 Y input: 0.0
 "a" constant: 0.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs:
 < None >
 Outputs:
 MT7 cycle time

Type: Set Point

Name: SP1 initial time
 Function: $X > Y$
 X input: TI1 Timer
 Y input: 3600.0
 Dead Band: 0.0
 Inputs:
 TI1 Timer
 Outputs:
 LG1 and PL3 Pulse CA16 Reverse scan

Name: SP2 Set Point
 Function: $X > Y$
 X input: CA4 minimum
 Y input: 0.5
 Dead Band: 0.0
 Inputs:
 CA4 minimum
 Outputs:
 LG3 And

Name: SP3 initial time

Function: X > Y
 X input: TI2 Timer
 Y input: 7200.0
 Dead Band: 0.0
 Inputs:
 TI2 Timer
 Outputs:
 LG2 and PL4 Step time CA18 Reverse scan

Name: SP4 Set Point
 Function: X > Y
 X input: CA9 minimum
 Y input: 0.5
 Dead Band: 0.01
 Inputs:
 CA9 minimum
 Outputs:
 LG4 And

Type: Logic

Name: LG1 and
 Function: X AND Y
 X input: SP1 initial time
 Y input: LG3 And
 Inputs:
 SP1 initial time LG3 And
 Outputs:
 CA3 Set Pot

Name: LG2 and
 Function: X AND Y
 X input: SP3 initial time
 Y input: LG4 And
 Inputs:
 SP3 initial time LG4 And
 Outputs:
 CA8 Set Pot

Name: LG3 And
 Function: X AND Y
 X input: PL3 Pulse
 Y input: SP2 Set Point
 Inputs:
 PL3 Pulse SP2 Set Point
 Outputs:
 LG1 and

Name: LG4 And
 Function: X AND Y
 X input: PL4 Step time
 Y input: SP4 Set Point
 Inputs:
 PL4 Step time SP4 Set Point
 Outputs:
 LG2 and

Type: Log

Name: LO1 P3TSS021.DAT
 Log Status: < disabled >
 Sample Rate: 2.0 Minutes
 Gate: < None >
 Data Format:
 Heading: pitting protection potential; ch 1
 File Path: C:\WB

File Name: P3TSS021.DAT
 Date Stamp: enabled
 Time Stamp: enabled
 Inputs:
 TI1 Timer CA7 Invert CA1 c.d. ch.1
 CA5 Charge
 Outputs:
 < None >

Name: LO2 P3TSS022.DAT
 Log Status: < disabled >
 Sample Rate: 2.0 Minutes
 Gate: < None >
 Data Format:
 Heading: pitting protection potential; ch 1
 File Path: C:\WB
 File Name: P3TSS022.DAT
 Date Stamp: enabled
 Time Stamp: enabled
 Inputs:
 TI2 Timer CA13 Invert CA2 c.d. ch.2
 CA10 Charge
 Outputs:
 < None >

Type: Meter

Name: MT1 C.d. Ch.1
 Output Type: Exponential
 Units: A/cm2
 Integer: 8
 Decimal: 1
 Inputs:
 CA1 c.d. ch.1
 Outputs:
 < None >

Name: MT2 Charge/cm2
 Output Type: Exponential
 Units: Coul/cm2
 Integer: 8
 Decimal: 1
 Inputs:
 CA5 Charge
 Outputs:
 < None >

Name: MT3 Pot Ch.1
 Output Type: Fixed Point
 Units: Volt
 Integer: 6
 Decimal: 3
 Inputs:
 CA7 Invert
 Outputs:
 < None >

Name: MT4 c.d. ch.2
 Output Type: Exponential
 Units: A/cm2
 Integer: 8
 Decimal: 1
 Inputs:
 CA2 c.d. ch.2
 Outputs:
 < None >

Name: MT5 Charge/cm2
 Output Type: Exponential
 Units: Coul/cm2
 Integer: 8
 Decimal: 1
 Inputs: CA10 Charge
 Outputs: < None >

Name: MT6 Pot Ch.2
 Output Type: Fixed Point
 Units: Volt
 Integer: 6
 Decimal: 3
 Inputs: CA13 Invert
 Outputs: < None >

Name: MT7 cycle time
 Output Type: Fixed Point
 Units: msec
 Integer: 6
 Decimal: 3
 Inputs: CA19 Calculation
 Outputs: < None >

Type: Chart

Name: CH1 set pot. ch1
 Chart Color: White
 X Axis Label: Hours
 X Axis Min: 0.0
 X Axis Max: 10.0
 Y Axis Label: < None >
 Y Axis Min: -0.6
 Y Axis Max: 0.4
 Inputs: CA3 Set Pot
 Outputs: < None >

Name: CH2 Chart
 Chart Color: White
 X Axis Label: Minutes
 X Axis Min: 0.0
 X Axis Max: 10.0
 Y Axis Label: < None >
 Y Axis Min: -0.6
 Y Axis Max: 0.4
 Inputs: CA8 Set Pot
 Outputs: < None >

Name: CH3 c.d. ch1
 Chart Color: White
 X Axis Label: Hours
 X Axis Min: 0.0
 X Axis Max: 10.0
 Y Axis Label: < None >
 Y Axis Min: 1.0e-07

Y Axis Max: 0.001

Inputs:
CA1 c.d. ch.1
Outputs:
< None >Name: CH4 Chart
Chart Color: White
X Axis Label: Minutes
X Axis Min: 0.0
X Axis Max: 5.0
Y Axis Label: < None >
Y Axis Min: -0.001
Y Axis Max: 0.05Inputs:
CA2 c.d. ch.2
Outputs:
< None >

Type: Analog Output

Name: AO:1 Set Pot Ch.1
Card Type: STI ACAO-12
Channel Number: 1
Range: +/- 5 Volts
Resolution: 0.024%
Inputs:
CA3 Set Pot
Outputs:
< None >Name: AO:2 ser pot ch 2
Card Type: STI ACAO-12
Channel Number: 2
Range: +/- 5 Volts
Resolution: 0.024%
Inputs:
CA8 Set Pot
Outputs:
< None >

P3TIN 025, DAT

Program: Same as p.46. Run w/ REPPST.NBB program.

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.25 from same chemical and solution lot #'s, except 50% stock solution was #019-231. Actual salt weights were 0.2441 g NaHCO_3 and 3.2932 g NaCl .

Set Up: As described on p.25. Ref SCE #4 was used.

- ① Initial wt. 11.6973g Final wt. 11.6879g
- ② de-aerate one hour before immersion
- ③ Hold @ open circuit for one hour before immersion
- ④ E com after 60 min -677 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 3600 sec (1 hr)
- ⑦ Applied Initial Potential +600 mV vs SCE
- ⑧ Initial Potential pH 8.07 @ 25°C Final pH 9.53 @ 25°C
upward trend
- ⑨ Immersed area $\approx 8.06 \text{ cm}^2$ $l = 3.889 \text{ cm}$ $\phi = 0.634 \text{ cm}$

Stored as PPPT #2 File lost.

3/26/92

Walter J. Moschinski

11/13/91

Pitting Protection Potential Tests on INC 825
(Lot # HH4371 FC)

P3TIN026.DAT

Purpose: Same as p. 46. Run w/REPASS1.WAB program.

Sample Prep: Same as p. 25

Solution: Used same solution as p. 51.

Set-Up: As described on p. 25. Ref SCE #4 was used.

- ① Initial WT. 11.6833 g Final WT. 11.1166 g
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion
- ④ E com after 60 min. -683 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied Initial Potential +600 mV vs SCE
- ⑧ Initial pH 8.07 @ 25°C Final pH 9.14 @ 25°C
- ⑨ Immersed area $\approx 7.73 \text{ cm}^2$ $l = 3.754 \text{ cm}$ $\phi = 0.634 \text{ cm}$

Stored on disk PPPT #7

Can not find file on this
disk or other companion disk.
The file is considered to be
lost

N. Smith

Walter J. Macchiusi
11/13/91

Pitting Protection Potential Tests on INC 825
(Lot # HH4371 FC)

P3TIN027.DAT

Purpose: Same as p. 46 Run w/REPASS1.WAB program.

Sample Prep: Same as on p. 25

Solution Prep: To 500 ml of 17M D₂O water in a 1-L volumetric flask add 0.1228 g of NaHCO₃ and 1.6468 g of NaCl (Fisher lot #'s 897289 + 885407 resp.). Pipet 20.0 ml of SO₄, 10.0 ml of NO₃, and 2.0 ml of F stock solutions (# 819-231, 019-199, + 019-191 resp.). Dilute to the mark.

Set-Up: As described on p. 25. Ref SCE #2 was used.

- ① Initial WT. 11.6941 g Final WT.
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion
- ④ E com after 60 min
- ⑤ Test Temp 95°C
- ⑥ Initial Time 7200 secs (2 hrs)
- ⑦ Applied Initial Potential +600 mV vs SCE
- ⑧ Initial pH 8.05 @ 25°C Final pH 9.39 @ 25°C

Data lost - not written to disk or logged for
unknown reasons.

Walter J. Macchiusi
11/14/91

Pitting Protection Potential Tests on 316L SS (Lot # P80246)

P3TSS028.DAT

Purpose: To re-run selected conditions to assess repeatability of PPPT experiments. Used REPP952. W.B.

Sample Prep: Same as p. 25.

Solution: Two liters were prepared as described on p. 25. All lot #'s and solution #'s were the same, except 504 stock which was 019-231. Actual salt weights were 0.249 g NaHCO₃ and 3.2928 g NaCl.

Set Up: As described on p. 25. Ref SCE #3 was used.

- ① Initial WT. 11.3018 g Final WT. 11.1155 g
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion.
- ④ E com after 60 min. -673 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +300 mV vs SCE
- ⑧ Initial pH 8.12 @ 25°C Final pH 9.18 @ 25°C
- ⑨ Immersed area $\approx 6.95 \text{ cm}^2$ $L = 3.3915 \text{ cm} = 0.623 \text{ cm}$
- ⑩ Final E +7 mV vs SCE (1 1/2 days)

Stored on dish PPPT #2.

Walter J. Macomber
12/4/91

Pitting Protection Potential Tests on 316L SS (Lot # P80246) 55

P3TSS029.DAT

Purpose: Same as p. 54.

Sample Prep: Same as p. 25.

Solution: Used same solution as on p. 54.

Set Up: As described on p. 25. Ref SCE #1 was used.

- ① Initial WT. 11.3015 g Final WT. 11.0947 g
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion
- ④ E com after 60 min. -721 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Initial Time 14,400 secs (4 hrs)
- ⑦ Applied initial potential +400 mV vs SCE
- ⑧ Initial pH 8.12 @ 25°C Final pH 9.20 @ 25°C
- ⑨ Immersed area $\approx 8.28 \text{ cm}^2$ $L = 4.092 \text{ cm} = 0.628 \text{ cm}$
- ⑩ Final E -21 mV vs SCE (2 days)

Stored on dish PPPT #2

Potential stayed at
+18 for upto

80,222 seconds (22.3 hrs.)

2 min had a brief increase in c.d.
which prompted a decrease
in potential. The potential
remained at -21 mV for
upto 179,420 seconds.

N. Sridhar
3/19/92

Walter J. Macomber
12/4/91

Pitting Protection Potential Tests on 316L SS
(Ref # P80746)

P3TSS030.DAT

Purpose: Same as p.54.

Sample Prep: Same as p.25.

Solution Prep: To 500 ml of 17 M seawater in a 1-L volumetric add 0.1231 g NaHCO_3 and 1.6463 g NaCl . Pipet 20.0 ml of SO_4 , 10.0 ml of SO_3 , and 20 ml of F stock solutions. Chemically solution lot #'s the same as on p.53

Set up: As described on p. 25. Ref SCE # 3 was used.

- ① Initial WT. 11.2933 g Final WT. 11.2065 g
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion.
- ④ E com after 60 min. -640 mV vs SCE
- ⑤ Test Temp. 95°C +300 mV 12/4/91
- ⑥ Applied initial potential +400 mV vs SCE
- ⑦ Initial Time 14,400 sec (4 hrs).
- ⑧ Initial pH 8.18 @ 25°C Final pH 9.12 @ 25°C
- ⑨ Immersed area $\approx 7.55 \text{ cm}^2$ $l = 3.671 \text{ cm}$ $\phi = 1.628 \text{ cm}$
- ⑩ Final E -3 mV vs SCE (after 2 days)

Stored on disk PPPT # 1

Walter J. Mochowski
12/6/91

Pitting Protection Potential Tests on INCO 825 (H44371FC) 57

P3TIN031.DAT

Purpose: Same as p.54.

Sample Prep: Same as p.25

Solution Prep: Two liters were prepared as described on p.54. Actual salt weights were 0.2451 g NaHCO_3 and 3.2927 g NaCl .

Set up: As described on p.25. Ref SCE # 1 was used.

- ① Initial WT. 11.7058 g Final WT. 11.6832
- ② De-aerate one hour before immersion
- ③ Hold @ open circuit for one hour after immersion
- ④ E com after 60 min. -535 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Applied Initial Potential +600 mV vs SCE
- ⑦ Initial Time 14,400 sec (4 hrs) 3/20/92
- ⑧ Initial pH 8.42 Final pH 9.25
- ⑨ Immersed area $\approx 6.942 \text{ cm}^2$ $l = 3.409 \text{ cm}$ $\phi = 1.620 \text{ cm}$
- ⑩ Final E +135 mV vs SCE (after ~6 days)
(stable for 142.7 hours) 3/20/92

Stored in PPPT # 1

Initial Time: 3600 seconds
as shown in log not
14,400 sec. as in step 7

- N. Sivaldas
3/20/92

N. Sivaldas
1/19/92

Pitting Protection Potential Tests on Luc 825 (HH4371FC)

P3TIN032.DAT

Purpose: Same as p.54Sample Prep: Same as p.25.Solution Prep: Used same solution as P3TIN031.Set-Up: As described on p.25. Ref SCE# 3 was used.

- ① Initial WT. 11.6871 g Final WT. 11.5917 g
- ② De-aerate one hour before immersion.
- ③ Hold @ open circuit for one hour after immersion.
- ④ E com after 60 min. -592 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Applied initial potential +600 mV vs SCE
- ⑦ Initial Time 7200 secs (2 hrs) ^{1/20/92}
- ⑧ Initial pH 8.42 Final pH 9.20
- ⑨ Immersed area $\approx 7.621 \text{ cm}^2$ $\ell = 3.738 \text{ cm}$ $\phi = .623 \text{ cm}$
- ⑩ Final E +98 mV vs SCE (after ~6 days)

Stored in PPPT #1

DIRECT TIMER LOG IS MISSING.

Potential varied between
604 and ~~583~~ ⁴⁷⁵ mV during
initial period. Actual Initial
Time logged was
11160 ^{AB} seconds (3 hrs + 6 min)

N. Suckas

1/19/92

N. Suckas

2/20/92

Pitting Protection Potential Tests on Luc 825 (HH4371FC) 59P3TIN033.DAT ^{p.25}Purpose: Same as p.54. Use REPASS2.WBB - see page 61. ^{WJ Macdonald 1/22/92}Sample Prep: Same as on p.25Solution Prep: To 1-liter of 17MR water in a 2-L volumetric add 3.2968g NaCl and 0.2445g NaHCO₃. Pipet 20.0 ml of SO₄, 10.0 ml of NO₃ and 2.0 ml of F stock solutions. Chemical + solution lot #'s are the same as on p.53.Set-up: Same as on p.25. Potentials and currents were verified using the Keithly 602 Electrometer and Keithly 485 Picoammeter respectively.

- ① Initial WT. 11.5750 g Final WT. 11.5664 g
- ② De-aerate one hour before immersion.
- ③ Hold @ open circuit for one hour after immersion.
- ④ E com after 60 min not recorded
- ⑤ Test Temp. 95°C
- ⑥ Applied initial potential +600 mV vs SCE
- ⑦ Initial Time 60 secs
- ⑧ Initial pH ~~8.00~~ 8.13 Final pH 9.22 ^{WJ Macdonald 1/22/92}
- ⑨ Immersed area $\approx 7.34 \text{ cm}^2$ $\phi = .634$ $A = 6.956 \text{ cm}^2$
- ⑩ Final E -572 mV ^{3.3341} (after ~5 days) ^{WJ Macdonald 1/22/92}

Stored in PPPT #1

Due to problems w/ hardware + software, this data is not valid.

Walter J Macdonald
1/24/92

Pitting Protection Potential Tests on Inc 825 (H4437)FQ

P3TIN034.DAT

Purpose: Same as p. 25. Use Repass 2 WBB.

Sample Prep: Same as p. 25.

Solution: Used same solution as P3TIN033.

Set-Up: Same as p. 25. Potentials + currents verified as described on p. 59.

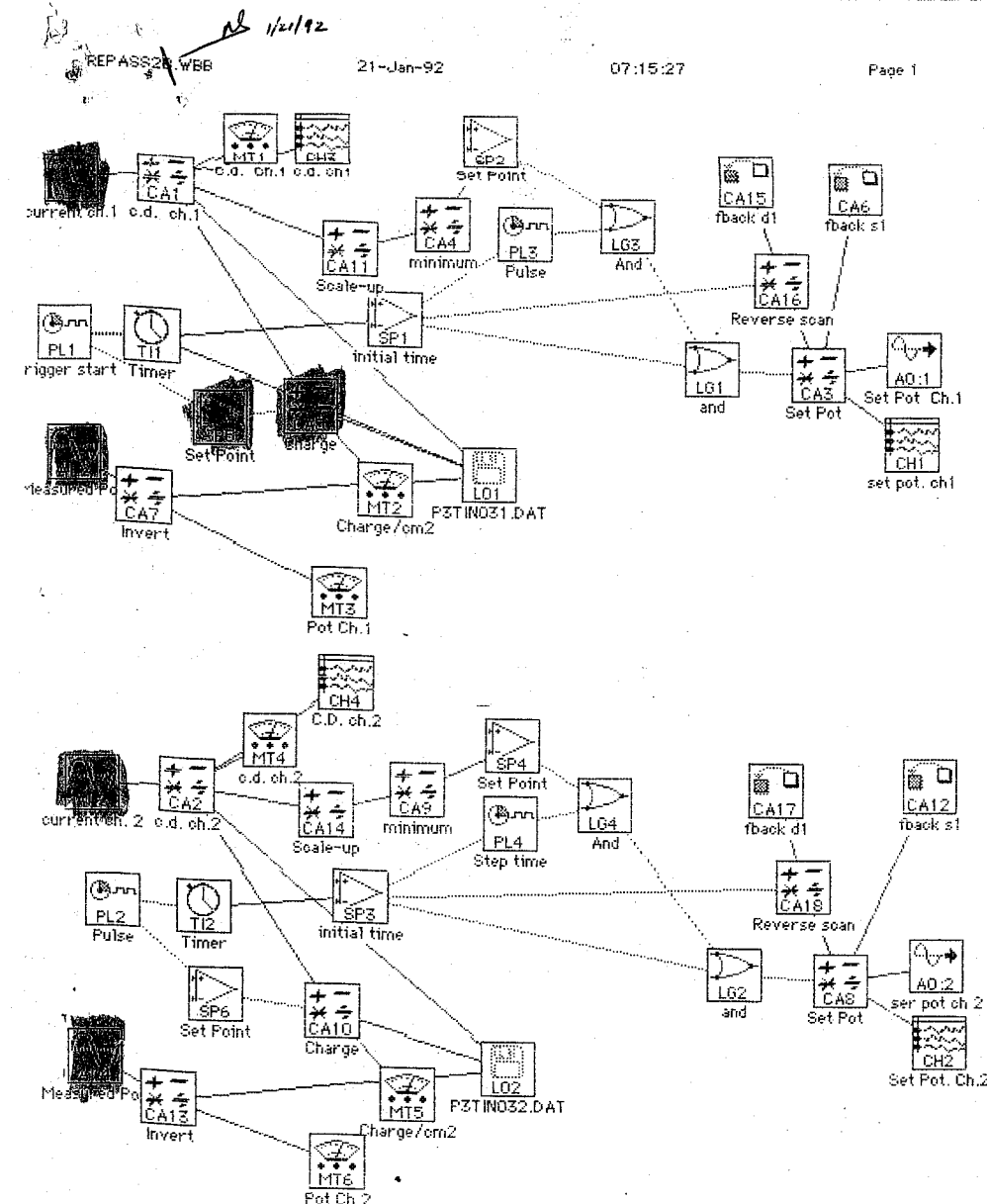
- ① Initial WT. 11.5757 g Final WT. 11.5718
- ② de-aerate one hour before immersion.
- ③ Hold at open circuit one hour after immersion.
- ④ E can after 60 min. -285 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Applied initial E +600 mV vs SCE
- ⑦ Initial Time 600 secs.
- ⑧ Initial pH 8.13 Final pH 9.16
- ⑨ Immersed area $\approx 7.308 \text{ cm}^2$ $\phi = .628 \text{ cm}$ $A = 6.394 \text{ cm}^2$
- ⑩ Final E +207 mV 3.084 $\mu\text{g}/\text{cm}^2$ 1/27/92 (after ~4 days)

Stored in PPPT #1

Walter J. Moench
1/24/92

Repass 2 WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.



N. J. Moench
1/25/92

Repas 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Worksheet Name:

REPAS2B.WBB

Hardware list:

Name

STI ACPC-16

STI ACAA-12

AI's AO's DIO's CT's DI's DO's

16 0 16 0 0 0

0 6 8 0 0 0

Maximum icons:

464

Grid size:

16

Snap to grid:

< disabled >

Report Unsynch:

< disabled >

Fast Mode:

enabled

Fast Mode Samples:

1000

Fast Mode Rate:

1.0 Kilohertz

Com ports:

2

Port:

COM 1

Comment:

Mouse connected

Port:

COM 2

Baud rate:

9600

Data bits:

8

Stop bits:

1

Duplex:

Half

Parity:

None

XonXoff:

< disabled >

Echo wait:

< disabled >

Line delay:

< disabled >

IEEE:

< disabled >

Type: Analog Input

Name:

AI:1 current ch.1

Card Type:

STI ACPC-16

Channel Number:

1

Range:

+/-Auto V

Resolution:

Lo Noise (17ms)

Output Type:

Voltage

Sample rate:

10.0 Hertz

Fast Mode:

< disabled >

Inputs:

< None >

Outputs:

CA1 c.d. ch.1

Name:

AI:2 Measured Pot

Card Type:

STI ACPC-16

Channel Number:

2

Range:

+/-Auto V

Resolution:

Lo Noise (17ms)

Output Type:

Voltage

Sample rate:

10.0 Hertz

Fast Mode:

< disabled >

Inputs:

< None >

Outputs:

CA7 Invert

Name:

AI:3 current ch. 2

Card Type:

STI ACPC-16

Channel Number:

3

Range:

+/-Auto V

Resolution:

Lo Noise (17ms)

Output Type:

Voltage

Sample rate:

10.0 Hertz

N. Smith
1/25/92

Repass 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

High Duration: 0.1 Seconds
 Low Duration: 180.0 Seconds
 Start Value: Low
 Reset on exit: enabled
 Inputs:
 SP3 initial time
 Outputs:
 LG4 And

Type: Calculation

Name: CA1 c.d. ch.1
 Function: aX / bY
 X input: AI:1 current ch.1
 Y input: 7.5
 "a" constant: -1.0
 "b" constant: 10.0
 "c" constant: 0.0
 Inputs:
 AI:1 current ch.1
 Outputs:
 MT1 C.d. Ch.1 CH3 c.d. ch1 CA5 Charge
 CA11 Scale-up LO1 P3TINO31.DAT

Name: CA2 c.d. ch.2
 Function: aX / bY
 X input: AI:3 current ch. 2
 Y input: 7.2
 "a" constant: -1.0
 "b" constant: 10.0
 "c" constant: 0.0
 Inputs:
 AI:3 current ch. 2
 Outputs:
 MT4 c.d. ch.2 CH4 C.D. ch.2 CA10 Charge
 CA14 Scale-up LO2 P3TINO32.DAT

Name: CA3 Set Pot
 Function: aX + bY + c
 X input: LG1 and
 Y input: CA16 Reverse scan
 "a" constant: -0.01
 "b" constant: 1.0
 "c" constant: 0.6
 Inputs:
 LG1 and CA16 Reverse scan
 Outputs:
 CH1 set pot. ch1 AO:1 Set Pot Ch.1 CA6 fback s1

Name: CA4 minimum
 Function: Min(X) for last (a) seconds
 X input: CA11 Scale-up
 Y input: 0.0
 "a" constant: 180.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs:
 CA11 Scale-up
 Outputs:
 SP2 Set Point

Name: CA5 Charge
 Function: Integral X dt
 X input: CA1 c.d. ch.1
 Y input: SP5 Set Point
 "a" constant: 0.0

N. Sridhar
 1/25/92

Repair 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

```

"b" constant:      0.0
"c" constant:      0.0
Inputs:
  CA1  c.d. ch.1    SP5  Set Point
Outputs:
  MT2  Charge/cm2   LO1  P3TIN031.DAT

Name:              CA6  fback s1
Function:          Global Feedback(a:address)
X input:          CA3  Set Pot
Y input:          0.0
"a" constant:     1.0
"b" constant:     0.0
"c" constant:     0.0
Inputs:
  CA3  Set Pot
Outputs:
  < None >

Name:              CA7  Invert
Function:          aX + bY
X input:          AI:2 Measured Pot
Y input:          0.0
"a" constant:     -1.0
"b" constant:     0.0
"c" constant:     0.0
Inputs:
  AI:2 Measured Pot
Outputs:
  MT3  Pot Ch.1     LO1  P3TIN031.DAT

Name:              CA8  Set Pot
Function:          aX + bY + c
X input:          LG2  and
Y input:          CA18 Reverse scan
"a" constant:     -0.01
"b" constant:     1.0
"c" constant:     0.6
Inputs:
  LG2  and          CA18 Reverse scan
Outputs:
  CH2  Set Pot. Ch.2  AO:2 ser pot ch 2  CA12 fback s1

Name:              CA9  minimum
Function:          Min(X) for last (a) seconds
X input:          CA14 Scale-up
Y input:          0.0
"a" constant:     180.0
"b" constant:     0.0
"c" constant:     0.0
Inputs:
  CA14 Scale-up
Outputs:
  SP4  Set Point

Name:              CA10 Charge
Function:          Integral X dt
X input:          CA2  c.d. ch.2
Y input:          SP6  Set Point
"a" constant:     0.0
"b" constant:     0.0
"c" constant:     0.0
Inputs:
  CA2  c.d. ch.2    SP6  Set Point
Outputs:
  MT5  Charge/cm2   LO2  P3TIN032.DAT

```

1/25/92

Repos 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Name: CA11 Scale-up
 Function: $aX + bY$
 X input: CA1 c.d. ch.1
 Y input: 0.0
 "a" constant: 10000.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: CA1 c.d. ch.1
 Outputs: CA4 minimum

Name: CA12 fback s1
 Function: Global Feedback(a:address)
 X input: CA8 Set Pot
 Y input: 0.0
 "a" constant: 2.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: CA8 Set Pot
 Outputs: < None >

Name: CA13 Invert
 Function: $aX + bY$
 X input: AI:4 Measured Pot
 Y input: 0.0
 "a" constant: -1.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: AI:4 Measured Pot
 Outputs: MT6 Pot Ch.2 LO2 P3TIN032.DAT

Name: CA14 Scale-up
 Function: $aX + bY$
 X input: CA2 c.d. ch.2
 Y input: 0.0
 "a" constant: 10000.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: CA2 c.d. ch.2
 Outputs: CA9 minimum

Name: CA15 fback d1
 Function: Global Feedback(a:address)
 X input: 0.0
 Y input: 0.0
 "a" constant: 1.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs: < None >
 Outputs: CA16 Reverse scan

Name: CA16 Reverse scan
 Function: $c(X + a)(Y + b)$
 X input: CA15 fback d1
 Y input: SP1 initial time
 "a" constant: -0.6

1/25/92

Repass 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

"b" constant: 0.0
 "a" constant: 1.0
 Inputs:
 CA15 fback d1 SP1 initial time
 Outputs:
 CA3 Set Pot

Name: CA17 fback d1
 Function: Global Feedback(a:address)
 X input: 0.0
 Y input: 0.0
 "a" constant: 2.0
 "b" constant: 0.0
 "c" constant: 0.0
 Inputs:
 < None >
 Outputs:
 CA18 Reverse scan

Name: CA18 Reverse scan
 Function: $c(X + a)(Y + b)$
 X input: CA17 fback d1
 Y input: SP3 initial time
 "a" constant: -0.6
 "b" constant: 0.0
 "c" constant: 1.0
 Inputs:
 CA17 fback d1 SP3 initial time
 Outputs:
 CA8 Set Pot

Type: Set Point

Name: SP1 initial time
 Function: $X > Y$
 X input: TI1 Timer
 Y input: 14400.0
 Dead Band: 0.0
 Inputs:
 TI1 Timer
 Outputs:
 LG1 and PL3 Pulse CA16 Reverse scan

Name: SP2 Set Point
 Function: $X > Y$
 X input: CA4 minimum
 Y input: 0.5
 Dead Band: 0.0
 Inputs:
 CA4 minimum
 Outputs:
 LG3 And

Name: SP3 initial time
 Function: $X > Y$
 X input: TI2 Timer
 Y input: 7200.0
 Dead Band: 0.0
 Inputs:
 TI2 Timer
 Outputs:
 LG2 and PL4 Step time CA18 Reverse scan

Name: SP4 Set Point
 Function: $X > Y$
 X input: CA9 minimum

1/25/92

Repass 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Y input: 0.5
Dead Band: 0.01

Inputs:
CA9 minimum

Outputs:
LG4 And

Name: SP5 Set Point
Function: X < Y
X input: PL1 Trigger start
Y input: 0.0
Dead Band: 0.0

Inputs:
PL1 Trigger start

Outputs:
CA5 Charge

Name: SP6 Set Point
Function: X < Y
X input: PL2 Pulse
Y input: 0.0
Dead Band: 0.0

Inputs:
PL2 Pulse

Outputs:
CA10 Charge

Type: Logic

Name: LG1 and
Function: X AND Y
X input: SP1 initial time
Y input: LG3 And
Inputs:
SP1 initial time LG3 And
Outputs:
CA3 Set Pot

Name: LG2 and
Function: X AND Y
X input: SP3 initial time
Y input: LG4 And
Inputs:
SP3 initial time LG4 And
Outputs:
CA8 Set Pot

Name: LG3 And
Function: X AND Y
X input: PL3 Pulse
Y input: SP2 Set Point
Inputs:
PL3 Pulse SP2 Set Point
Outputs:
LG1 and

Name: LG4 And
Function: X AND Y
X input: PL4 Step time
Y input: SP4 Set Point
Inputs:
PL4 Step time SP4 Set Point
Outputs:
LG2 and

Type: Log

1/25/92

Repass 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Name: LO1 P3TIN031.DAT
 Log Status: < disabled >
 Sample Rate: 15.0 Minutes
 Gate: < None >
 Data Format:
 Heading: pitting protection potential; ch 1
 File Path: C:\WB
 File Name: P3TIN031.DAT
 Date Stamp: enabled
 Time Stamp: enabled
 Inputs:
 TI1 Timer CA7 Invert CA1 c.d. ch.1
 CA5 Charge
 Outputs:
 < None >

Name: LO2 P3TIN032.DAT
 Log Status: < disabled >
 Sample Rate: 15.0 Minutes
 Gate: < None >
 Data Format:
 Heading: pitting protection potential; ch 1
 File Path: C:\WB
 File Name: P3TIN032.DAT
 Date Stamp: enabled
 Time Stamp: enabled
 Inputs:
 CA13 Invert CA2 c.d. ch.2 CA10 Charge
 Outputs:
 < None >

Type: Meter

Name: MT1 C.d. Ch.1
 Output Type: Exponential
 Units: A/cm2
 Integer: 8
 Decimal: 1
 Inputs:
 CA1 c.d. ch.1
 Outputs:
 < None >

Name: MT2 Charge/cm2
 Output Type: Exponential
 Units: Coul/cm2
 Integer: 8
 Decimal: 1
 Inputs:
 CA5 Charge
 Outputs:
 < None >

Name: MT3 Pot Ch.1
 Output Type: Fixed Point
 Units: Volt
 Integer: 6
 Decimal: 3
 Inputs:
 CA7 Invert
 Outputs:
 < None >

Name: MT4 c.d. ch.2
 Output Type: Exponential

1/25/92

Repair 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Units: A/cm2
 Integer: 8
 Decimal: 1
 Inputs: CA2 c.d. ch.2
 Outputs: < None >

Name: MT5 Charge/cm2
 Output Type: Exponential
 Units: Coul/cm2
 Integer: 8
 Decimal: 1
 Inputs: CA10 Charge
 Outputs: < None >

Name: MT6 Pot Ch.2
 Output Type: Fixed Point
 Units: Volt
 Integer: 6
 Decimal: 3
 Inputs: CA13 Invert
 Outputs: < None >

Type: Chart

Name: CH1 set pot. ch1
 Chart Color: White
 X Axis Label: Hours
 X Axis Min: 0.0
 X Axis Max: 24.0
 Y Axis Label: < None >
 Y Axis Min: -0.8
 Y Axis Max: 0.7
 Inputs: CA3 Set Pot
 Outputs: < None >

Name: CH2 Set Pot. Ch.2
 Chart Color: White
 X Axis Label: Hours
 X Axis Min: 0.0
 X Axis Max: 10.0
 Y Axis Label: < None >
 Y Axis Min: -1.2
 Y Axis Max: 0.8
 Inputs: CA8 Set Pot
 Outputs: < None >

Name: CH3 c.d. ch1
 Chart Color: White
 X Axis Label: Hours
 X Axis Min: 0.0
 X Axis Max: 10.0
 Y Axis Label: < None >
 Y Axis Min: 1.0e-07
 Y Axis Max: 0.001
 Inputs: CA1 c.d. ch.1

1/25/92

Repair 2. WBB Documentation

Purpose: Modification of Potentiostat Esc 440 & data acquisition system to release some channels.

Outputs:

< None >

Name: CH4 C.D. ch.2
 Chart Color: White
 X Axis Label: Minutes
 X Axis Min: 0.0
 X Axis Max: 5.0
 Y Axis Label: < None >
 Y Axis Min: -0.001
 Y Axis Max: 0.05

Inputs:
 CA2 c.d. ch.2

Outputs:
 < None >

Type: Analog Output

Name: AO:1 Set Pot Ch.1
 Card Type: STI ACAO-12
 Channel Number: 1
 Range: +/- 5 Volts
 Resolution: 0.024%
 Inputs:
 CA3 Set Pot
 Outputs:
 < None >

Name: AO:2 ser pot ch 2
 Card Type: STI ACAO-12
 Channel Number: 2
 Range: +/- 5 Volts
 Resolution: 0.024%
 Inputs:
 CA8 Set Pot
 Outputs:
 < None >

N. Sridhar
 1/25/92

Pitting Protection Potential Test on Inc 825 (HH4371FC)

P3TIN035.DAT

Purpose: Re-run of P3TIN033.DAT.Sample Prep: Same as p.25.Solution Prep: Same as p.59. Actual salt wts. were 3.2960g NaCl and 0.2436g NaHCO₃.Set-Up: Same as p.25. Currents and potentials verified as described on p.59. Used REPASS2.WBB (p.61)

- ① Initial WT. 11.5996g - Final WT. 11.4849g
- ② de-aerate one hour before immersion
- ③ Hold at open-circuit for one hour after immersion
- ④ E can after 60 min. -680 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Applied initial potential +600 mV vs SCE
- ⑦ Initial Time 60 secs
- ⑧ Initial pH 8.00 Final pH 9.09
- ⑨ Immersed area \approx undetermined $l =$ $\rho =$
- ⑩ Final E not meaningful

After running a few hours, re-set PL3 to 1 sec and 180 sec and re-started test (re-initiated pulse). The next day, changed specimen (called P3TIN035.DAT) and re-initiated using same solution. Anomalous behavior caused these changes to be made.

Specimen P3TIN035 was repolished in an attempt to reverse

Walter J. MacLachlan

1/29/92

Pitting Protection Potential Test on Inc 825 (HH4371FC)

P3TIN036.DAT

Purpose: Same as p.25.Sample Prep: Same as p.25.Solution Prep: Used same solution as P3TIN035.Set-Up: Same as p.25. Currents + potentials verified as on p.59. Use REPASS2.WBB (p.61)

- ① Initial WT. 11.5893g Final WT. 11.5476g
- ② de-aerate one hour before immersion
- ③ Hold at open-circuit for one hour before immersion
- ④ E can after 60 min. -511 mV
- ⑤ Test Temp 95°C
- ⑥ Applied initial potential +600 mV vs SCE
- ⑦ Initial time 3600 secs
- ⑧ Initial pH 8.00 Final pH 9.09
- ⑨ Immersed area \approx 6.951 cm² $l = 3.378$ cm $\phi = .626$ cm
- ⑩ Final E -703 mV (after ~ 2 days)

Data stored in PPPT#1 Diskette

The potential remained at +108 mV sce with a c.d. 5.1×10^{-7} to 4×10^{-7} A/cm for 29 hours. Then current suddenly increased prompting a decrease in potential. c.d. was high (10^{-4} A/cm²) even at -700 mV. This is most probably due to bubble in the salt bridge. 108 mV was assumed to be the repassivation potential.

N. Sridhar

3/20/92

Walter J. MacLachlan

1/31/92

Pitting Potential Test on 316L

P3TSS037.DAT

Purpose: To re-run a condution to try to analyze the anomalous behavior of REPASS 2. WBB

Test was aborted to flaws in the test WB program.

Stored in PPPT #1

Walter J. Moenchli
2/4/92

Pitting Repassivation Potential Test on 316L SS.

P3TSS038.DAT

2/4/92

Purpose: Determine repassivation potential of 316L stainless as a function of initial pit growth.

Sample Prep: Same as p. 25

Solution: Same solution composition as P3TINO35.DAT

Set up: Used REPASS 2. WBB Program, see p. 61. Used the WORKBENCH Ver 2.0.4 updated software (Has Password Protection)

at $t=0$, $E_{\text{corr}} = -818 \text{ mV/SCE}$ (Using Keithley 614 electrometer INST. # 11152)

Note: The E_{corr} was highly variable at first and also relatively high. A brief cathodic pulse of -1.5 V was given & then turned off. The specimen was allowed to stay at E_{corr} for 5 minutes.

Initial pH 8.02 @ 25°C Final pH 9.01 @ 25°C

Stored in PPPT #1

N. Smith
2/4/92

Preparation of NO_3 Stock Solution 1000 ppm
 Purpose: Solution to be used for preparation of test solutions.

Prep: To 500 ml of 17 Mr water in a 1-L volumetric add 1.3793 g of NaNO_3 (Fisher lot # 897183). Dilute to the mark.
 Label as SN CMCRA 025-66.

Walter J. Macdonald
 2/5/92

Pitting Repassivation Potential of 316L SS

39 WSM

PPTSS038.DAT

Purpose: Same as p. 65. This is a re-run of W. Macdonald's 2/5/92

Sample Prep: Same as p. 25.

Set-Up: Same as p. 25. Currents and potentials verified as described on p. 59. Use REPASS2.WBB (p. 61)

Solution Prep: Same as p. 59, except NO_3 stock solution was SN CMCRA 025-66. Actual salt weights were 3.2965 g NaCl and 0.2446 g NaHCO_3

- ① Initial WT. 11.3492 g Final WT. 11.3152 g
- ② All anode are born before immersion.
- ③ Hold at open circuit one hour after immersion
- ④ E can after 60 min. -448 mV vs SCE
- ⑤ Test Temp. 95°C
- ⑥ Applied initial potential +300 mV vs SCE
- ⑦ Initial Time 3600 secs
- ⑧ Initial pH 8.02 @ 25°C Final pH 9.09 @ 55°C
- ⑨ Immersed area $\approx 6.91 \text{ cm}^2$ $l = 3.365$ $\phi = .625$
- ⑩ Final E -41 mV vs SCE

Timer log missing

N. Swafford
 2/19/92

Stored in PPT # 1

N. Swafford
 2/18/92

Note for Experiments P3T XX033-038

Due to problems with the experimental hardware and software, data from these experiments are not valid.

Walter J. MacLuski
2/13/92

Pitting Protection Potential of 316L SS (Pt# P80746)

P3T SS040.DAT

Purpose: To determine repassivation potential of 316L SS as a function of pit growth (initial).

Set-Up: Used REPASS 2.WBB (p.61) Used Workbench version 2.0.4 updated software. Hardware, i.e. test cells, etc were as described on p. 23

Solution Prep: To one liter of 17M Ω water in a 2L volumetric add 3.2964 g of NaCl and 0.2447 g of NaHCO₃ (Fisher lot #'s 885407 and 897289 resp.). Pipet 20.0 ml of NO₃ stock, 40.0 ml of SO₄ stock and 4.0 ml of F stock solutions. (lot #'s 025-66, 019-231, and 019-191 resp.). Dilute to the mark.

- ① Initial WT. 11.3052 g Final WT. 11.2480 g
- ② All accurate w/ N₂ for one hour before immersion.
- ③ Hold at OCV for one hour after immersion.
- ④ E can after 60 min -471 mV vs SCE
- ⑤ Initial pH 7.91 @ 25°C Final pH 9.04 @ 25°C

Potential Measurement

ESC 440

Keithley 614

Data Acquisition
System

+0.035 V SCE

+0.0356 V SCE

+0.036 V SCE

① Initial applied potential +300 mV vs SCE

② Initial T_{im} 60 secs

③ Test Temp 95°C

④ Immersed area $\approx 7.15 \text{ cm}^2$ $l = 3.489 \text{ m}$ $d = 0.625$

⑤ Final E 0.036 upto 75297 seconds. Decreased beyond. Probably related to bubble formation in probe.

Stored on CPPT #1

Walter J. MacLuski
2/16/92

Pitting Protection Potential of 316L SS (P80746)

P3TSS041.DAT

Purpose: Same as P3TSS040Set-Up: Same as P3TSS040Solution: Used same solution as P3TSS040

- ① Initial WT. 11.3248g Final WT. 11.2865g
- ② de-aerate w/ N_2 for one hour before immersion
- ③ Hold at OCV for one hour after immersion
- ④ E_{con} after 60 min. -755 mV vs SCE
- ⑤ Initial pH 8.03 @ 25°C Final pH 9.08 @ 25°C

Potential Measurement

ESC 440

Keithley 619

Data Acquisition
System

+0.017V SCE +0.0178V SCE +0.018 V SCE

Final $E_{con} = -0.093 V$ SCE

- ① Initial applied E +300 mV vs SCE
- ② Initial T_{imm} 600 sec
- ③ Test Temp 95°C
- ④ Immersed area $\approx 7.63 \text{ cm}^2$ $l = 3.725 \text{ cm}$ $\phi = .626 \text{ cm}$
- ⑤ Final E -934 V vs SCE (For 120 hours)

Stored on PPPT #2

Timer not acc. logged.

The potential stayed at 18 mV for 96 hours. Then
 c.d. increased beyond threshold ^{but was near above 10^{-4} A/cm^2} , potential dropped in
 stages to -93 mV (final E) charge at 96 hours
 was 8.7 C/cm² Final charge: 27 C/cm².

N. Smith
3/19/92

Preparation of SO_4 Stock Solution 1000 ppm
Purpose: Solution to be used for preparation of
 test solutions.

Prep: To 500 ml of 17MR water in a 1-L
 volumetric add 1.4786g of Na_2SO_4 (Fisher
 lot # 901213), dilute to the mark.
 Label as SN CNWRA 025-71.

Walter J. MacKowski

2/18/95

P3TSS042.DAT

Purpose: Same as P3TSS040

Set-Up: Same as P3TSS040

Solution: To 1-Liter of 17 M Ω water in a 2-L volumetric add 3.2962g NaCl and 0.2451g NaHCO_3 (Fisher lot #'s 885407 + 897789 resp.). Pipet 20.0 ml of NO_3 stock, 40.0 ml of SO_4 stock, and 4.0 ml of F stock solutions. (#'s 025-66, 025-71 and 019-191 resp.).

① Initial WT. 11.2982g Final WT. 11.2625g

ESC 440	K. 614	DAC
+297 mV	+299 mV	+297 mV

- ② de-aerate w/ N_2 for one hour before immersion
- ③ Hold at open circuit for one hour
- ④ E corr after 60 min. -455 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Applied initial potential +300 mV vs SCE
- ⑦ Initial time 3600 secs.
- ⑧ Initial pH 8.04 Final pH 9.11
- ⑨ Immersed area $\pm 6.39 \text{ cm}^2$ $l = 4.034 \text{ cm}$ $\phi = .637 \text{ cm}$
- ⑩ Final E -2 mV vs SCE

Stored on PPPT #2.

Potential remained at -2 mV for 800,109 sec. (222 hrs) then decreased due to small increase in c.d. beyond threshold.

P3TSS043.DAT

Purpose: Same as P3TSS040

Set-Up: Same as P3TSS040

Solution: Used same solution as P3TSS042 (p. 72)

ESC 440	K. 614	DAC
+197 mV	+200 mV	+198 mV

- ① Initial WT. (not taken) Final WT. 11.2783
- ② de-aerate w/ N_2 for one hour before immersion
- ③ Hold at OCV for one hour
- ④ E corr after 60 min. -562 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Applied initial potential +200 mV vs SCE
- ⑦ Initial Time 3600 secs.
- ⑧ Initial pH 8.04 Final pH 9.48 @ 25°C
- ⑨ Immersed area $\pm 4.573 \text{ cm}^2$ $l = 2.173 \text{ cm}$ $\phi = .625 \text{ cm}$
- ⑩ Final E = +8 mV vs SCE

Test terminated due to excess evaporation; it appeared to have reached a stable E value prior to this.

Stored on PPPT #2

INITIAL part of i_c current; potential data not logged in.

Walter J. Machowski
2/27/92

P3TSS044. DAT

Purpose: Same as P3TSS040

Set-up: Same as P3TSS040

Solution: To 1-liter of 17MR water in a 2-L volumetric add 3.2962 g NaCl and 0.2451 g of NaHCO_3 (same lot #s as p. 72). Pipet 20.0 ml of NO_3 stock, 40.0 ml of SO_4 stock and 4.0 ml of F stock solutions (same lot #s as p. 72). Dilute to the mark.

- ① Initial WT. 11.2992 g
- ② De-aerate w/ N_2 for one hour before immersion
- ③ Hold at OCV for one hour.

Pot. Check:

Ketty 614	ESC 440	DAC
-541 mV	-542 mV	-547 mV
+97 "	+97 "	+98 "

- ④ E_{corr} at 60 min -541 mV vs SCE
- ⑤ Temp. 95°C
- ⑥ Applied potential (initial) +100 mV vs SCE
- ⑦ Initial Time 3600 sec.
- ⑧ Initial pH 7.98 @ 25°C
- ⑨ Immersed area $\geq 8.42 \text{ cm}^2$
- ⑩ Final pH not taken
- ⑪ $l = 4.042 \text{ cm}$ $\phi = .638 \text{ cm}$

Bubbles in Luggin probe caused problems w/ the current control. Test will be re-run.

Stored on PPPT #2

Walter J. Macchowski
3/2/92

P3TSS045. DAT

Purpose: Same as P3TSS040. This is a re-run of P3TSS044.

Set-up: Same as P3TSS040

Solution: To 1 liter of 17MR water in a 2-L volumetric add 3.2970 g of NaCl and 0.2437 g NaHCO_3 (same lot #'s as p. 72). Pipet 20.0 ml of NO_3 stock, 40.0 ml of SO_4 stock and 4.0 ml of F stock solutions (same lot #s as p. 72). Dilute to the mark.

- ① Initial wt. 11.2883 g
- ② Final wt. 11.2956 g
- ③ De-aerate one hour before immersion (N_2).
- ④ Hold at OCV for one hour.

Pot. Check:

Ketty	ESC 440	DAC
-365 mV	-366 mV	-365 mV
-673 mV	-676 mV	-671 mV

 → there are wrong channels

- ⑤ E_{corr} at 60 min. -731 mV vs SCE
- ⑥ Test Temp 95°C
- ⑦ Applied initial potential +100 mV vs SCE
- ⑧ Initial Time 3600 sec
- ⑨ Initial pH 8.01
- ⑩ Final pH 9.28
- ⑪ Immersed area $\geq 7.00 \text{ cm}^2$
- ⑫ $l = 3.368$ $\phi = .632 \text{ cm}$
- ⑬ Final E +59 mV vs SCE

Stored on PPPT #2

The current remained low at an applied potential of 100 mV for 54.15 hours. Then a sudden increase in current resulted in potential drop to 59 mV.

Walter J. Macchowski
3/15/92

P3TIN046.DAT

Purpose: Same as P3TSS040, except on INC 825.

Set-Up: Same as P3TSS040.

Solution: Used same solution as P3TSS045.

- ① Initial WT. 11.6352 g Final wt. 11.5997 g
 - ② Deaerate one hour before immersion (N_2).
 - ③ Hold at OCV for one hour.
- | | | |
|----------|---------|---------|
| Keithley | ESC 440 | DATC |
| -364 mV | -365 mV | -363 mV |

- ④ E can at 60 min. -416 mV vs SCE

Bubble in Luggin probe caused I to go cathodic.
Bubble was removed & test restarted.

New bubble formed - spec changed due to pit growth. New WT: = 11.6060

- ⑤ New OCV w/new spec. -445 mV
- ⑥ Test Temp 95°C
- ⑦ Applied initial Potential +500 mV vs SCE
- ⑧ Initial T_{irr} 3600 sec
- ⑨ Initial pH 8.01 Final pH 9.17
- ⑩ Immersed area $\cong 7.75 \text{ cm}^2$ $l = 3.732 \text{ cm}$ $\phi = .63 \text{ cm}$
- ⑪ Final E \rightarrow stayed at +500 mV then dropped rapidly to -1.6 V due to either bubble in probe or low electrolyte level in reference electrode. Test was terminated.

Stored on PPPT # 2

Potential stayed at 490-500 mV for 127.5 hours with c.d. of $1 \times 10^{-7} \text{ A/cm}^2$ approx. Then small increases in c.d. pushed potential down to very cathodic values.

Walter J. Macdonald
3/13/92

N. Sridhar
3/20/92

P3TIN047

Purpose: Same as P3TIN046.DAT

Set-Up: Same as P3TSS040.

Solution: To 1-liter of 17M Ω water in a 2-L volumetric add 3.2932 g of NaCl and 0.3429 g NaHCO_3 (same lot # as on p. 72). Pipet 20.0 ml of NO_3 stock, 40.0 ml of SO_4 stock and 4.0 ml of F stock solution (same lot # as on p. 72) dilute to the mark.

- ① Initial WT. 11.5683 g Final WT. 11.5476 g
- ② Deaerate w/ N_2 for one hour before immersion.
- ③ Hold at OCV for one hour.
- ④ E can at 60 min -662 mV vs SCE
- ⑤ Test Temp 95°C
- ⑥ Applied initial E +700 mV vs SCE
- ⑦ Initial T_{irr} 600 sec
- ⑧ Initial pH 8.05 Final pH 8.98 $\text{approximate } 3/12/92$
- ⑨ Immersed area $\cong 8.102 \text{ cm}^2$ $l = 4.5412 \text{ cm}$ $\phi = .633 \text{ cm}$
- ⑩ Final E = -2.0 Volts 3.916 cm

Test probably not valid due to bubbles in probe and erratic current control.

Stored on PPPT # 2

Potential stayed at 0.145 V for 47.8 hours with c.d. of about 10^{-7} A/cm^2 . Then c.d. increased forcing potential decrease.

N. Sridhar
3/20/92

Walter J. Macdonald
3/17/92

Pitting Potential of Inoc 825 (H# 4371 PC)

P3TIN048

Purpose: Same as P3TIN046.DAT.Set-Up: Same as P3TIN047Solution: Used same solution as P3TIN047.

- ① Initial WT. 11.6051 g Final WT. 11.6650
- ② Deaerate w/ H_2 for one hour before immersion.
- ③ Hold at OCV for one hour.
- ④ E com at 60 min -125 mV vs SCE
- ⑤ Test temp 95°C
- ⑥ Applied initial E +600 mV vs SCE
- ⑦ Initial Tim 3600 secs.
- ⑧ Initial pH 8.05 Final pH
- ⑨ Immersed area $\approx 9.301 \text{ cm}^2$ $l = 4.518 \text{ cm}$ $\phi = .635$
- ⑩ Final E ~~2.0 Volts~~ +358 mV vs SCE
w/ micrologger
3/16/92

Stored on PPPT #2

Walter J. Mochnowski
3/17/92

Pitting Potential of Inoc 825 (H# 4371 PC)

P3TIN049

Purpose: Re-run of P3TIN047Set-Up: Same as P3TIN047Solution: Prepared 2-liter as on p. 77 using same lot # chemicals and solutions. Actual salt weights were 3.2936 g NaCl and 0.2446 g $NaHCO_3$

- ① Initial WT. not taken
 - ② Deaerate w/ H_2 for one hour
 - ③ Hold at OCV for one hour
 - ④ E com at 60 min. -533 mV vs SCE
- | | | |
|----------------|---------------|------------|
| <u>Kithley</u> | <u>ESC440</u> | <u>DAC</u> |
| -483 mV | -484 | -483 |

- ⑤ Test temp 95°C
- ⑥ Applied initial E +700 mV vs SCE
- ⑦ Initial Tim 600 secs.
- ⑧ Initial pH 8.03 Final pH not taken

Test terminated early due to excessive fluid loss.

Stored in PPPT #2

Walter J. Mochnowski
3/19/92

P3TIN050

Purpose: Re-run of P3TIN048Set-up: Same as P3TIN047Solution: Used same solution as P3TIN049

- ① Initial WT. not taken
 - ② Deaerate w/ N_2 for one hour
 - ③ Hold at OCV for one hour
 - ④ E can at 60 min -445 mV vs SCE
- | | | |
|----------------|---------------|------------|
| <u>Kentley</u> | <u>ESC440</u> | <u>DAC</u> |
| -444 mV | -446 | -441 |

- ⑤ Test temp 95°C
- ⑥ Applied initial E +600 mV vs SCE
- ⑦ Initial time 3600 sec.
- ⑧ Initial pH 8.03 Final pH not taken

Test terminated early due to excessive fluid loss.

Stored in PPPT # 2

Walter J. MacKowski

P3TIN051

Purpose: Re-run of P3TIN047Set-up: Same as P3TIN047

Solution: Prepared 2-liters as on p. 77 using same lot # chemicals and solutions. Actual salt weights were 3.2906 g NaCl and 0.2450 g $NaHCO_3$.

- ① Initial WT. 11.6115 g Final WT. 11.5837 g
 - ② Deaerate w/ N_2 for one hour.
 - ③ Hold at OCV for one hour.
 - ④ E can at 60 min -467 mV vs SCE
- | | | |
|----------------|---------------|------------|
| <u>Kentley</u> | <u>ESC440</u> | <u>DAC</u> |
| -466 mV | -467 | -464 |

- ⑤ Test Temp 95°C
- ⑥ Applied initial E +700 mV
- ⑦ Initial time 600 sec
- ⑧ Initial pH 8.07 Final pH 9.18
- ⑨ Immersed area $\approx 8.06 \text{ cm}^2$ $L = 3.889 \text{ cm}$ $\phi = 1.634 \text{ cm}$

Test stopped early due to excessive noise in potential sensing circuit.

Stored on disk PPPT # 2.

Walter J. MacKowski
3/23/92

Pitting Potential of Luc 825 (HH4371 FC)

P3TIN052

Purpose: Return of P3TIN048

Set-Up: Same as P3TIN047

Solution: Used same solution as P3TIN051.

① Initial WT. 11.6093 g Final WT. 11.6045 g

② Deaerate w/ N_2 for one hour

③ Hold at OCV for one hour.

④ E can at 60 min -608 mV vs SCE

Keithley	ESC440	DAC
-608 mV	-608	-605

⑤ Test Temp. 95°C

⑥ Applied initial E +600 mV vs SCE

⑦ Initial time 3600 hrs.

⑧ Initial pH 8.07

Final pH 9.26

⑨ Immersed area $\approx 7.66 \text{ cm}^2$ $l = 7.455 \text{ cm}$ $\phi = .633 \text{ cm}$
3.696 cm w/ Macdonald
3/23/92

Test stopped overnight due to excessive fluid loss.

Stored on dish PPPT # 2

Walter J. Macdonald
3/23/92

Pitting Potential of Luc 825 (HH4371 FC)

P3TIN053

Purpose: Return of P3TIN048 w/ Macdonald 3/24/92 P3TIN047

Set-Up: Same as P3TIN047

Solution: To 500 ml of 17 M de water in a 1-liter volumetric add 1.6449 g NaCl and 0.1233 g of $NaHCO_3$. Pipet 2.0 ml of Fstoch, 10.0 ml of NO_3 stock and 20.0 ml of 50% stock solutions (chemicals & solutions are same lot #12 as p. 72). Dilute to the mark.

① Initial WT. 11.6115 g Final WT. 11.5713 g

② Deaerate w/ N_2 for one hour.

③ Hold at OCV for one hour

④ E can at 60 min

Keithley	ESC440	DAC
+697	+695	+704

⑤ Test Temp. 95°C

⑥ Initial applied E +700 mV vs SCE

⑦ Initial time 600 sec

⑧ Initial pH 8.00

Final pH 9.21

⑨ Immersed area $\approx 7.94 \text{ cm}^2$ $l = 3.848 \text{ cm}$ $\phi = .631 \text{ cm}$

Test was re-initiated on same specimen after removing or repolishing. Spec. had probably re-passivated by the time restart occurred.

Stored on dish PPPT # 2

Walter J. Macdonald
3/30/92

Pitting Potential of Luc 825 (HA4371FC)

P3TIN054

Purpose: Repetition of P3TIN047Set-Up: Same as P3TIN047Solution: Prepared as on p. 72 from same stock # chemicals + solutions. Actual salt weights were 1.6468 g NaCl and 0.1225 g NaHCO₃.

- ① Initial wt. 11.5889 g Final wt. 11.5516 g
- ② Deaerate w/N₂ for one hour.
- ③ Hold at OCV for one hour.
- ④ E can at 60 min.

Ketley	ESC	DAC
-168mV	-170	-175

- ⑤ Test temp 95°C
- ⑥ Initial applied E +700mV vs SCE
- ⑦ Initial time 600 sec
- ⑧ Initial pH 8.00 Final pH 9.28
- ⑨ Immersed area $\approx 8.32 \text{ cm}^2$ $l = 4.0374$, $d = .636 \text{ cm}$

Stored on disk 111T#2

Walter J. Macfarlane
3/30/92

Repasivation Potential on Crevice 316LSS (P80746)

CRREPO1

Purpose: To gather data on repasivation potential on a crevice specimen.Set-Up: Crevice specimens were machined at S.A.R.T. Crevices were obtained by mounting slotted discs against the flat surfaces of the specimen using C-276 nuts + bolts torqued to 50 lbs-ft in -16 w/torque wrench type Protus P-60; ser # A-90689, cal'd 2-14-92 due 18-14-92. The tightening bolt was electrically insulated from the specimen w/ polypropylene sleeve and TFE tape wrapping. The flat surfaces on which the crevices were made were wet polished w/ 600 grit paper. Used program CREVICE.WAB which is the same program as REPASS2.WAB.Solution: Prepared as on p. 72 from same stock # chemicals and solutions. Actual salt wts were 3.2430 g NaCl and 0.2444 g NaHCO₃.

- ① Initial wt. 35.0513 g Final wt. 35.0373 g
 - ② Deaerate w/N₂ for one hour
 - ③ Hold at O.C. for one hour E can @ 60 min -358mV
- | | | |
|--------|------|------|
| Ketley | ESC | DAC |
| -346mV | -347 | -338 |

- ④ Test temp 95°C
- ⑤ Initial applied E +100mV vs SCE
- ⑥ Initial time 3600 sec.
- ⑦ Initial pH 8.01 Final pH 6.67
- ⑧ Final E +97mV

Stored on disc "crevice #1"

Bulk of specimen not attached. 21 "sectors" showed localized attack. Most of it shallow and spread out. In about 5 sectors deeper pits were forming. Mounted area free of attack.

Walter J. Macfarlane
4/17/92

CRREPO2

Purpose: Same as CRREPO1

Set-Up: Same as on p. 85.

Solution: Used same solution as CRREPO1.

- ① Initial WT. 35.1634 g Final WT. 35.1585 g
- ② Deaerate w/ N_2 for one hour
- ③ Hold at O.C. for one hour E_{con} @ 60 min - 316 mV

Kalby	ESC	DAC
-339	-340	-337
- ④ Test temp. 95°C
- ⑤ Initial applied E +200 mV vs SCE
- ⑥ Initial time 3600 sec
- ⑦ Initial pH 8.01 Final pH 3.18
- ⑧ Final E +78 mV

Stored on Crevic #1

Faces of specimen were reddish-brown, but segments where ceramic formed crevices was still metallic. One sector had shallow, broad local attack. One other sector had a small but deeper pit. Mounted area free of attack.

Walter J. Macdonald
4/17/92

CRREPO3

Purpose: Same as CRREPO1

Set-Up: Same as p. 85. One difference is all faces of specimen polished to 600 grit and all non-crevice faces passivated w/nitric acid.

Solution: Prepared as on p. 27 from same lot # chemicals and solutions. Actual salt wts. 3.2922 g NaCl + 0.2424 g $NaHCO_3$.

- ① Initial WT. 34.8896 g Final WT. 34.8816 g
- ② Deaerate w/ N_2 for one hour
- ③ Hold at O.C. for 1 hour E_{con} at 60 min - 321 mV vs SCE

Kalby	ESC	DAC
-593	-594	-590
- ④ Test temp 95°C
- ⑤ Initial applied E +200 mV vs SCE
- ⑥ Initial time 7200 sec
- ⑦ Initial pH 8.03
- ⑧ Final E +58 mV

Stored on Crevic #1

Some mottled reddish-brown stains on surfaces. Some shallow, broad, localized attack in 17 sectors, most of it nearer the edges. Some areas of similar local attack (shallow) and some pits evident on non-creviced faces of specimen. Mounted area free of attack.

Walter J. Macdonald
4/23/92

CRREP 04

Purpose: Same as CRREP01Set-Up: Same as p. 85.Solution: Used same solution as CRREP03

- ① Initial WT. 35.0820g Final WT. 35.0602g
 ② Cleanse w/ N_2 for one hour.
 ③ Hold at O.C. for one hour E_{con} @ 60min -5mV
 Keithly ESC DAC
 -211mV -209 -210

- ④ Test Temp. 95°C 500 mV 10/15/92
 ⑤ Initial applied E +450mV vs SCE
 ⑥ Initial time ³⁶⁰⁰ 7200 sec. 10/15/92
 ⑦ Initial pH 8.01 Final pH 9.23
 ⑧ Final E +158mV

Stored on Crevice #1

Some brown-red stain around creviced areas and at mounting hole. 14 sectors show broad, shallow attack filling most of the sector area. Some crevice attack with one rather deep spot around mounting hole. Rest of specimen free of localized attack.

Walter J. Neckowski
4/23/92

CRREP05

Purpose: Same as CRREP01.

Set-Up: Same as p. 85. Also, the nut holding the specimen against the specimen holding rod was tightened to a controlled torque, which was 400g-in.

Solution: Prepared 2-liters as on p. 77 from same lot # chemicals and solutions. Actual salt weights were 3.2958g NaCl and 0.2432g NaHCO₃.

- ① Initial WT. 35.2385g Final WT. 35.19734g
 ② Cleanse w/ N_2 for one hour E_{con} at 60min -513mV
 ③ Hold at O.C. for one hour
 Keithly ESC DAC
 -503mV -504 -506

- ④ Test Temp 95°C
 ⑤ Initial applied E +500mV vs SCE
 ⑥ Initial time 7200 sec
 ⑦ Initial pH 8.00 Final pH 9.22
 ⑧ Final E +46mV

Stored on Crevice #1

Some reddish-brown stain around creviced areas and at mounting hole. 19 sectors show broad, shallow attack, in most cases filling the entire affected sector. Crevice attack around mount, one fairly large and deep spot. A few minor pits forming at other places on non-creviced surfaces.

Walter J. Neckowski
4/23/92

Repassivation Potential on Creviced Inc 825 (Lot # HH4371FC)

CRREP06

Purpose: Same as CRREP01.

Set-Up: As described on p. 89.

Solution: Used same solution as CRREP05.

- ① Initial WT. 35.4833 g Final WT. 35.4706 g
- ② Aerate w/ N_2 for one hour
- ③ Hold at O.C. one hour E_{can} @ 60 min -597 mV
- ④ Keithley ESC DAC
 -579 mV -581 -582
- ⑤ Test Temp 95°C
- ⑥ Initial applied E +450 mV vs SCE
- ⑦ Initial time 3600 sec
- ⑧ Initial pH 8.00 Final pH 9.27
- ⑨ Final E +217 mV

Stored on Crevice #1

Reddish-brown staining around crevice sectors. 14 sectors have broad, shallow attack filling the entire sector (in most cases). No attack around mount. Rest of specimen free from attack.

Walter J. Macdonald
4/23/92

Repassivation Potential on Creviced Inc 825 (Lot # HH4371FC)

CRREP07

Purpose: Same as CRREP01.

Set-Up: As described on p. 89.

Solution: Prepared 2-liters as on p. 77 from same lot # chemicals and solutions. Actual salt weights were 3.2957 g NaCl and 0.2460 g $NaHCO_3$.

- ① Initial WT. 35.3263 g Final WT. 35.1974 g
- ② Aerate w/ N_2 for one hour
- ③ Hold at O.C. for one hour E_{can} @ 60 min -555 mV
- ④ Keithley ESC DAC
 -469 mV -470 -469
- ⑤ Test Temp 95°C
- ⑥ Initial applied E +500 mV vs SCE
- ⑦ Initial time 600 sec
- ⑧ Initial pH 8.02 Final pH 8.98
- ⑨ Final E +36 mV

Stored on Crevice #1

(Potential stayed at 106 mV for a while for dipped possibly due to noise spike.)

Very little reddish-brown staining, most of it around the mounting hole. 21 sectors had broad, shallow attack. In most cases it filled all or most of the sector area. Moderate crevice attack around the mount. Rest of specimen free from attack.

Walter J. Macdonald
4/27/92

Repassivation Potential on Creviced Inc 825 (HH437/FC)

CRREP08

Purpose: Same as CRREP01.

Set-Up: As described on p. 89.

Solution: Used same solution as CRREP07.

- ① Initial WT. 35.1839 g Final WT. 35.1412 g
- ② degaerate w/ N_2 for one hour
- ③ Hold at O.C. one hour E con @ 60 min -511 mV
- Keithly E5C DAC
- 560 mV -561 -560
- ④ Test Temp 95°C
- ⑤ Initial applied E +450 mV vs SCE
- ⑥ Initial time 14,400 sec
- ⑦ Initial pH 8.02 Final pH 9.78
- ⑧ Final E +59 mV

Stored on Crevice #1

Small amount of reddish-brown stain around crevice sectors. 15 sectors had broad, shallow attack usually filling most of the sector area. No crevice attack or any attack at mounting hole. No attack on other specimen surfaces.

Walter J. Macdonald
4/27/92

Repassivation Potential on Creviced Inc 825 (HH437/FC)

CRREP09

Purpose: Same as CRREP01.

Set-Up: As described on p. 89.

Solution: Prepared 1-liter as described on p. 83 from same chemical + solution lot #'s. Actual salt weights were 1.6464 g NaCl and 0.1215 g $NaHCO_3$.

- ① Initial WT. 35.3318 g Final WT. 35.3312 g
- ② degaerate w/ N_2 for one hour.
- ③ Hold at O.C. one hour E con @ 60 min -294 mV
- Keithly E5C DAC
- 488 mV -490 -489
- ④ Test Temp. 95°C
- ⑤ Initial applied E +350 mV vs SCE
- ⑥ Initial time 3600 sec
- ⑦ Initial pH 7.99 Final pH 8.99
- ⑧ Final E +189 mV

Stored on Crevice #1

Cell had excessive evaporation.
again 4/27/92

Small amount of reddish-brown stain around crevice sectors. 3 sectors show broad, shallow attack. One sector full, other 2 sectors spotty. No attack at mounting hole. No attack on other surfaces.

Walter J. Macdonald
4/27/92

CRREP10

Purpose: Same as CRREP01Set-Up: As described on p. 89.Solution: Prepared 2-liters as on p. 77 from same lot # chemicals + solutions. Actual salt weights were 3.2930g NaCl and 0.2439g NaHCO₃.

- ① Initial WT. 34.7115g Final WT. 34.7047g | 28/92 WYM.
 ② deaerate w/H₂ for one hour -531mV
 ③ Hold at O.C. one hour E_{can} @ 60 min. -696mV
- | | | |
|---------------|------------|------------|
| <u>Kitchy</u> | <u>ESC</u> | <u>DAC</u> |
| -602mV | -603 | -602 |

④ Temp 95°C

⑤ Initial applied E +150mV vs SCE

⑥ Initial time 600 secs.

⑦ Initial pH 8.00

Final pH 9.00

⑧ Final E -33mV

Stored on Crevic #1

Some bluish-brown staining. Small and shallow local attack in 10 sectors. Moderately deep crevice attack at mounting hole.

Current was quite low at +0.036V.

Walter J. MacKowski
5/1/92

CRREP11

Purpose: Same as CRREP01.Set-Up: As described on p. 89.Solution: Used same solution as CRREP10.

- ① Initial wt. 34.8324g Final wt. 34.8279g
 ② deaerate w/H₂ for one hour
 ③ Hold at O.C. one hour E_{can} @ 60 min. -696mV
- | | | |
|---------------|------------|------------|
| <u>Kitchy</u> | <u>ESC</u> | <u>DAC</u> |
| -607mV | -608 | -607 |

④ Temp 95°C

⑤ Initial applied E +150mV vs SCE

⑥ Initial time 14,400 secs.

⑦ Initial pH 8.00

Final pH 9.20

⑧ Final E +18mV dropped to -51mV probably due to evap.
 Stored on Crevic #1

Some bluish-brown staining. Very small + shallow attack in 2 sectors. Moderately deep attack around the mounting hole.

Walter J. MacKowski
5/1/92

CRREP 12

Purpose: Same as CRREP 01Set-Up: As described on p. 89.Solution: Prepare 2 liters as on p. 77 from same lot # chemicals and solutions. Actual Salt weights were 3.2929 g NaCl and 0.2451 g NaHCO_3 .

① Initial WT. 34.7615g Final WT. 34.7501g

② Deaerate one hour w/ N_2 E con @ 60 min -732 mV

③ Hold at O.C. one hour

Keithly	ESC	DAC
-665 mV	-666	-666

④ Temp 95°C

⑤ Initial applied E +150 mV vs SCE

⑥ Initial time 3600 sec

⑦ Initial pH 7.92 Final pH 9.18

Stored on crevice #1.

Same staining of bluish-brown color. Very small spots of local attack in 7 sectors. Moderately deep attack around the mounting hole.

Walter J. MacKowski
5/15/92

CRREP 13

Purpose: Same as CRREP 01.Set-Up: As described on p. 89.Solution: Used same solution as CRREP 12.

① Initial WT. 34.9296g Final WT. 34.9249g

② Deaerate one hour w/ N_2 E con @ 60 min -516 mV

③ Hold at O.C. one hour

Keithly	ESC	DAC
-353 mV	-355	-347

④ Temp 95°C

⑤ Initial applied E +150 mV vs SCE

⑥ Initial time 7200 sec.

⑦ Initial pH 7.92 Final pH 9.24

Stored on Crevice #1

Very little staining. Very small spots of localized attack in 7 sectors. Some attack around mounting hole, but much less than previously seen, only about 30% of circumference is affected.

Walter J. MacKowski
5/15/92

Preparation of SO_4 Stock Solution

Purpose: Solution to be used for preparation of test solutions.

Prep: To 500 ml of 17 M Ω water in a 1-L volumetric flask add 1.4786 g of Na_2SO_4 (Fisher lot # 901213), dilute to the mark.

Labeled as SN CNARA 025-98

Walter J. Mochowski
5/4/92

CRRP 14

Purpose: Same as CRRP01, but using a Teflon crevice washer instead to see if crevice growth intensifies.

Set-Up: As described on p. 89, except that Teflon crevice washers were used and torqued to 11 inch-pounds.

Solution: One liter was prepared by adding 1.6463 g NaCl and 0.1236 g NaHCO_3 (Fisher lot # 885407 and 892289 resp.) Pipet 2.0 ml of F stock, 10.0 ml of NO_3 stock, and 20.0 ml of SO_4 stock solutions (lot # 019-191, 025-666 and 025-98). These were added to 500 ml of 17 M Ω water in a 1-L volumetric and diluted to volume.

① Initial Wt. 34.8608 g Final Wt. 34.8629 g ?

② Deaerate w/ N_2 for one hour E can @ 60 min

③ Hold at O.C. for one hour

Meas. ESC DAC
-485mV -487 -483

④ Temp 95°C

⑤ Initial applied E +150 mV w SCE

⑥ Initial time 3600 secs

⑦ Initial pH 7.96 Final pH 9.13

Stored on Crevice #1

Brown staining around circumference (outer) of TFE washer. Localized attack in 13 sectors, in many cases filling the entire sector area. 3 spots of shallow attack at mounting hole, again not affecting entire circumference + less severe than previously recorded.

Walter J. Mochowski
5/15/92

P3TIN055.DAT

Purpose: To assess growth rate of pits by initiation above pitting potential for a short time, and then holding at a secondary potential at which pits would grow but not initiate for a set time, and then allowing potential to decrease until the repassivation potential is passed.

Set-Up: All hardware was set up as described on p. 55. The ~~RASS~~ 2.WBO program (p. 61) was used, although manual input of the potential parameters was used to apply the initial and secondary potentials.

Solution: Prepared 2 liters as on p. 77, but using lot #s listed on p. 99. Actual salt weights were 32932g NaCl and 0.2440g NaHCO₃.

- ① Initial WT. 11.71643g Final WT. 11.71105g
 - ② Deaerate w/ N₂ for one hour E_{corr} @ 60min -389mV
 - ③ Hold at O.C. for one hour
- | Keithley | BSC | DAC |
|----------|------|------|
| -392 | -392 | -389 |

④ Temp. 95°C

- ⑤ Initial E +600mV vs SCE for 5 minutes
- Secondary E +400 " " for ~~one~~ ^{two} hours

⑥ Initial pH 8.02 Final pH 9.39

Stored on PPPT #2.

Walter J. MacKushin
5/21/92

P3TIN056.DAT

Purpose: Same as P3TIN055

Set-Up: Same as p. 100

Solution: Prepare 2 liters. To 1-liter of 17 M R water in a 2-L volumetric add 3.2279g NaCl and 0.2448g NaHCO₃ and 2.9578g Na₂SO₄ (Fisher lot #s 685402, 897289 + 901213 rep). Pipet 4.0 ml of E stock, 20.0 ml of NO₃ stock solution (lot #s on p. 99). Dilute to the mark.

- ① Initial WT. 11.7142g Final WT. 11.7019g
 - ② Deaerate w/ N₂ for one hour
 - ③ Hold at O.C. for one hour E_{corr} @ 60min -491mV
- | Keithley | BSC | DAC |
|----------|------|------|
| -494 | -493 | -491 |

④ Temp. 95°C

- ⑤ Initial E +800mV for ~~5~~ ⁷ minutes
 - Secondary E +400mV for ~~one~~ ^{two} hours
- w/ manual 5mV/sec

⑥ Initial pH 8.05 Final pH 9.27

Stored on PPPT #2.

Walter J. MacKushin
5/21/92

Pitting Protection Potential Test on Inoc 825 (HH4371FC)

P3TIN057.DAT

Purpose: Same as P3TIN055Set-Up: Same as p.100Solution: Used same solution as P3TIN055

- ① Initial WT. 11.6817 g Final WT. 11.6665 g
- ② Aerate w/Air for one hour
- ③ Hold at O.C. for one hour E_{con} @ 60 min -590 mV

Reading	PSC	DAC
-594	-594	-598
- ④ Temp 95°C
- ⑤ Initial E +600 mV for 5 min
Secondary E +400 mV for 6 hours
- ⑥ Initial pH 8.00 Final pH 9.62

Stored on PPPT #2

Walter J. Mackowski
5/22/92

Pitting Protection Potential Test on Inoc 825 (HH4371FC)

P3TIN058.DAT

Purpose: Same as P3TIN055Set-Up: Same as p.100Solution: Used same solution as P3TIN056.

- ① Initial WT. 11.6832 g Final WT. 11.6518 g
- ② Aerate w/Air for one hour
- ③ Hold at O.C. for one hour E_{con} @ -532 mV

Reading	PSC	DAC
-534	-534	-532
- ④ Temp 95°C
- ⑤ Initial E +800 mV for 5 minutes
Secondary E +400 mV for 6 hours
- ⑥ Initial pH 8.06 Final pH 9.27

Stored on PPPT #2

Walter J. Mackowski
5/22/92

Pitting Protection Potential Test on 316L SS (Lot # P80746)

P3T SS059. DAT

Purpose: Same as P3TIN055.

Set-Up: Same as p. 100

Solution: Prepared 2 liter by adding 3.2941 g NaCl and 0.2447 g NaHCO_3 (same lot #/s as p. 99) to 1 liter of 17 M R water in a 2-L volumetric. Pipet 4.0 ml of F stock, 40.0 ml of SO_4 stock, and 20.0 ml of NO_3 stock solution (same lot #/s as p. 99). Dilute to the mark.

- ① Initial WT. 11.3159 g Final WT. 11.2966 g
 ② Deaerate w/ N_2 for one hour
 ③ Hold at O.C. for one hour E_{corr} @ 60 min -624 mV
- | Keithley | ESC | DAC |
|----------|-----|-----|
| -20 mV | -22 | -21 |

- ④ Temp 95°C +300 mV $\text{O}_2/\text{H}_2\text{O}$ electrode
 ⑤ Initial E +200 mV vs SCE for 5 minutes
 Secondary E +200 mV for 1 hour

- ⑥ Initial pH 8.02 Final pH 9.08

Stored on TPT #2. Disc ran out of space - data did not transfer.
 Stored on Repass. Pot. Data Disk #3

After 5 min at 300 mV, potential was dropped to 100 mV. c.d. remained at $\approx 1 \times 10^{-6} \text{ A/cm}^2$. Even a brief increase to 200 mV did not cause renewed pitting. Then potential was dropped to 100 mV + no c.d. increase was observed for 63.9 hours.

Walter J. Macrowski
 6/8/92

Pitting Protection Potential Test on 316L SS (Lot # P80746)

P3T SS060. DAT

Purpose: Same as P3TIN055.

Set-Up: Same as p. 100.

Solution: To 1 liter of 17 M R water in a 2-L volumetric add 3.2948 g NaCl, 2.4560 g Na_2SO_4 and 0.2442 g NaHCO_3 (same lot #/s as p. 101). Pipet 4.0 ml of F stock and 20.0 ml of NO_3 stock solution (lot #/s as p. 99). Dilute to the mark.

- ① Initial wt. 11.3066 g Final WT. 11.2956 g
 ② Deaerate w/ N_2 for one hour.
 ③ Hold at O.C. for one hour E_{corr} @ 60 min -671 mV
- | Keithley | ESC | DAC |
|----------|------|------|
| -226 mV | -226 | -226 |

- ④ Temp 95°C
 ⑤ Initial E +300 mV for 5 minutes
 Secondary E +100 mV " 1 hour
 ⑥ Initial pH 8.01 Final pH 8.61

Stored on TPT #2. Disc ran out of space - data did not transfer.
 Stored on Repass. Pot. Data Disk #3

After 5 min at 300 mV, potential decreased to +100 mV. c.d. remained low at $\approx 1 \times 10^{-6} \text{ A/cm}^2$ for 2.57 hours. Then potential increased to +150 mV for 1 hour. The potential was then decreased in 30 min steps. The new repassivation potential was 69 mV for the threshold c.d. was set 18.53 hours. Then, for some reason, c.d. increased beyond threshold & the potential was decreased again to -1 mV for 5.5 hours, then decreased in steps to -74 mV. It stayed here for 39 hours.

Walter J. Macrowski
 6/8/92

Pitting Protection Potential Tests on 316L SS (Lot # P80746)

P3TSS061.DAT

Purpose: Same as P3TIN055

Set-Up: Same as p.100.

Solution: Used same solution as P3TSS059.

- ① Initial WT. 11.2793 g Final WT. 11.2534 g
 - ② Deaerate w/ N_2 for one hour
 - ③ Hold at O.C. for one hour E_{cor} @ 60 min -747 mV
- | Keithley | ESC | DAC |
|----------|------|------|
| -727 mV | -725 | -725 |

④ Temp 95°C

⑤ Initial E +300 mV for 5 minutes
Secondary E +200 mV for 6 hours

⑥ Initial pH 8.03 Final pH 9.08

Stored on PPT#2. Disc ran out of space - data did not transfer.

Stored on Repara. Pot. Data Disk #3

C.d. stayed low at +36 mV for 64.3 hours. At this time, a small increase in current (this must have been for less than 3 mV since it did not log in) resulted in reduction of potential to +26 mV at which it stayed for 15.6 hours.

Walter J. MacKowski
6/8/92

Pitting Protection Potential Tests on 316L SS (Lot # P80746)

P3TSS062.DAT

Purpose: Same as P3TIN055.

Set-Up: Same as p.100.

Solution: Used same solution as P3TIN060.

- ① Initial WT. 11.3318 g Final WT. 11.2807 g
 - ② Deaerate w/ N_2 for one hour
 - ③ Hold at O.C. for one hour E_{cor} @ 60 min -700 mV
- | Keithley | ESC | DAC |
|----------|------|------|
| -681 mV | -679 | -678 |

④ Temp 95°C

⑤ Initial E +300 mV for 5 minutes
Secondary E +100 mV for 6 hours

⑥ Initial pH 8.04 Final pH 9.19

Stored on Repara. Pot. Data Disk #3.

At 100 mV, the c.d. decreased over a period of time to very low & then to cathodic value. Time at 100 mV was Total of 22.5 hours. C.d. decreased below 1×10^{-6} A/cm² after 1.33 hours.

Walter J. MacKowski
6/8/92

Pitting Protection Potential Test on Hast C-22

P3T22063.DAT

Purpose: Same as P3TIN055.

Set-up: Same as p. 100

Solution: To 500 ml of 17MR water in a 1-L volumetric add 16.4610 g of NaCl (Fisher lot # 885407). Dilute to the mark.

- ① Initial WT. 12.2655 g Final WT. 12.1283 g
- ② Deaerate w/ N_2 for one hour.
- ③ Hold @ O.C. for one hour E_{com} @ 60 min -414 mV

<u>Rvblly</u>	<u>BSC</u>	<u>DAC</u>
-449 mV	-450	-447

④ Temp 95°C

⑤ Initial E +700 mV (see note)

⑥ Initial pH 5.72 Final pH 5.39

Note: Upon applying the initial potential of +700 mV, it was noted that the C.D. kept slowly decreasing. It was decided to let it stay at +700 mV for a few days and not go thru a potential decrementation since no localized attack was being initiated. The specimen was turning a golden yellow for the first day or two, which had become a very dark, almost black by the end of the experiment.

Stored on Repass. Pot. Lat. Dish #3.

Walter J. MacKowski
6/16/92

Pitting Protection Potential Test on 316L SS (Lot. P80746)

P3TSS064.DAT

Purpose: Re-run of P3TSS059.

Set-up: Same as p. 100.

Solution: Prepared 1-liter. To 500 ml of 17MR water in a 1-L volumetric add 16.460 g NaCl and 0.1226 g NaH_2PO_4 (same lot #/s as p 99). Pipet 2.0 ml of F stock, 20.0 ml of S₂ stock and 10.0 ml of NO₂ stock solution. (same lot #/s as p. 99). Dilute to the mark.

- ① Initial WT. 11.2967 g Final WT. 11.2603 g
- ② Deaerate w/ N_2 for one hour
- ③ Hold at O.C. for one hour E_{com} @ 60 min

<u>Rvblly</u>	<u>BSC</u>	<u>DAC</u>
-765 mV	-765	-764

④ Temp 95°C

⑤ Initial E +300 mV (see note)Secondary E +200 mV for 1 hour

⑥ Initial pH 8.03 Final pH 9.07

Note: Due to selection of improper parameters, the initial potential may have been at higher than +300 mV (maybe higher than 2.0V) for short periods of time. Also more than 5 minutes at initiating potential was incurred so that the pit initiation was more severe than planned.

Walter J. MacKowski
7/16/92

Repasivation Time experiments

Purpose: To determine the time and total charge taken by alloy P25 and 316LSS to repassivate after pits have been initiated and allowed to grow for a specified length of time. The Program: Reptime.WBB is documented below:

Repasivation Time on 316LSS (lot P 80746)

REPTIME 1

Purpose: To determine repassivation time.

Set-Up: As described on p. 25. Reptime.WBB was used.

Solution: 2-liters was prepared by adding 3.2422g of NaCl (Fisher lot # 915219) and 0.244g NaHCO₃ (Fisher lot # 897789) to 1 liter of 17 MΩ water in a two liter volumetric. Pipet 40.0 of 50% stock, 20.0 ml of 10% stock, and 2.4.0 ml of F stock solutions (#025-28, ^{when 8/7/92} 025-66 and 019-191 respectively), dilute to volume.

- ① Initial WT. 11.3611g Final WT. 11.2985g
- ② Deaerate w/ N₂ for one hour.
- ③ Hold at O.C. for one hr. E_{scan} @ 60min -600mV/rate

Reitels	ESC	DAC
-617mV	-618mV	-611mV
- ④ Temp. 95°C
- ⑤ Initial E +300mV for 600 secs.
- ⑥ Secondary E +5mV
- ⑦ Initial pH 8.03 Final pH 8.65

Stored on disc REPTIME #1

Walter J. Machowski
8/7/92

REPTIME 2

Purpose: Same as REPTIME 1.Set-Up: Same as p. 25. Used REPTIME-WBB.Solution: Used same solution as REPTIME 1.

- ① Initial WT, 11.3545g Final WT, 11.1850g
 ② Deaerate w/ N_2 for one hr
 ③ Hold at O.C. for one hr. E con @ 60 min -585 mV
- | Keithley | ESC | DAC |
|----------|------|------|
| -604 | -605 | -598 |

- ④ Temp. 95°C
 ⑤ Initial E +300 mV for 2,600 secs
 ⑥ Secondary E -5 mV
 ⑦ Initial pH 8.03 Final pH 9.22

Stored on REPTIME #1

Walter J. MacLuski
8/7/92

REPTIME 3

Purpose: Same as REPTIME 1.Set-Up: Same as p. 25. Used REPTIME-WBB.Solution: To 1 liter of 17MR water in a 1 liter volumetric add 3.2925g NaCl and 0.245g $NaHCO_3$ (same lot #1/2 as p 112) Pipet 40.0 ml of 50% stock, 20.0 ml of 10% stock and 4.0 ml of F stock solutions (same lot #'s as p 112) Dilute to mark.

- ① Initial WT 11.6687g Final WT. 11.3884g
 ② Deaerate w/ N_2 for one hr
 ③ Hold at O.C. for one hr E con @ 60 min -573 mV
- | Keithley | ESC | DAC |
|----------|------|------|
| -541 | -542 | -534 |

- ④ Temp. 95°C
 ⑤ Initial E +600 mV for 1800 secs. ①
 ⑥ Secondary E +400 mV

- ⑦ Initial pH 8.05 Final pH 9.15

① Due to high currents, potentials were stepped down manually according to the following schedule

Initial: +600 mV

Step 1: +400 mV (until 8/8/92)

" 2: +325 " (until 8/9/92, 7:45 AM)

" 3: +250 " (until 8/9/92, 9:30 AM)

" 4: +150 " (until 8/10/92, 7:30 AM)

" 5: 0 mV until finish

Test solution very murky.

Stored on REPTIME #1.

Walter J. MacLuski
8/10/92

Repassivation Time of INC 825 (HH 4371FC)

REPTIME 4

Purpose: Same as Reptime 1.Set-Up: Same as p. 25. Used REPTIME. WBB.Solution: Used same solution as REPTIME 3.

- ① Initial WT 11.6744 Final WT. 11.1817g
 ② deaerate w/ H_2 for one hr.
 ③ Hold at O.C. for one hr. E con @ 60 min -534 mV
- | Kethley | ESC | DAC |
|---------|------|------|
| -576 mV | -578 | -569 |

- ④ Temp. 95°C
 ⑤ Initial E +600 mV for 21,600 secs ①
 ⑥ Secondary E +350 mV
 ⑦ Initial pH 8.05 Final pH 9.02

① Due to high currents, potential was stepped down manually according to the following schedule:

Initial: +600 mV (1 hr)

Step 1: +350 " (until 8/9/92)

" 2: +200 " (until 8/10/92, 7:30 AM)

" 3: 0 " until finish.

Solution very murky w/ much corrosion product on specimen and at bottom of flask.

Stored on REPTIME #1

Walter J. MacKowski
8/10/92

Repassivation Time of INC 825 (HH 4371FC)

REPTIME 5

Purpose: Same as REPTIME 1. Used REPTIME. WBB w/ mod^②

② Modified to step down twice: First step down to an intermediate E to grow pits that have initiated and then step down again to an E where repassivation should occur. Vary the time of pit growth on first step down in different experiments.

Set-Up As on p. 25.Solution: Prepared 2 liter as on p. 111 from same chemical & solution lot #1s.

- ① Initial WT. 11.5917g Final WT. 11.5665g
 ② deaerate w/ H_2 for one hour
 ③ Hold at O.C. for one hour E con @ 60 min -620 mV
- | Kethley | ESC | DAC |
|---------|------|------|
| -509 mV | -511 | -503 |

- ④ Temp. 95°C
 ⑤ Initial E +600 mV for 1800 secs
 Second E +400 mV for 3600 secs ② 8/24 No polarization done at 400 mV.
 Third E +100 mV until finish

- ⑥ Initial pH 8.10 Final pH 9.25

Solution murky.

Stored on REPTIME #1

Walter J. MacKowski
8/19/92

REPTIME 6

Purpose: Same as REPTIME 1. Used mod'd REPTIME WAB.

Set-up: Same as p. 25

Solution: Used same solution as REPTIME 5.

- ① Initial WT. 11.5885 g Final WT. 11.5606 g
 ② Deaerate w/ N_2 for one hour
 ③ Hold at O.C. for one hour E con @ 60 min. -443 mV
- | Kalby | ESC | DAC |
|---------|------|------|
| -600 mV | -601 | -592 |

④ Temp. 95°C

- ⑤ Initial E +600 mV for 1800 secs
 Second E +400 mV for 21,600 secs
 Third E +100 mV until finish

⑥ Initial pH 8.10 Final pH 9.35

Solution was murky.

Stored on REPTIME #1

Walter J. Machowski
8/14/92

REPTIME 7

Purpose: Same as REPTIME 1. Used mod'd REPTIME WAB.

Set-up: Same as p. 25

Solution: Prepared two liters as on p. 111. Lot # of NaCl: Fisher 932649A. All other lot #'s the same. Actual salt weights 3.2930g NaCl and 0.2448g $NaHCO_3$.

- ① Initial WT. 11.5887 g Final WT. 11.5312 g
 ② Deaerate w/ N_2 for one hour
 ③ Hold at O.C. for one hr. E con @ 60 min. -456 mV vs SCE
- | Kalby | ESC | DAC |
|---------|------|------|
| -444 mV | -443 | -439 |

④ Temp 95°C

- ⑤ Initial E +600 mV for 1/2 hr.
 Second E +400 mV for 12 hrs
 Third E +150 mV until end.

⑥ Initial pH 8.03 Final pH 9.45

Solution was murky.

Stored on REPTIME #2

Walter J. Machowski
8/27/92

REPTIME 8

Purpose: Same as REPTIME 1. And mod'd REPTIME 4B/A.

Set-up: As on p. 35.

Solution: Used same solution as REPTIME 7.

- ① Initial wt. 11.5812g Final wt. 11.4820g
 ② Deaerate a/p₂ for one hr.
 ③ Hold at o.c. for one hr. E con @ 60min -658mV
- | Hubly | ESC | DAC |
|--------|------|------|
| -616mV | -618 | -611 |

④ Temp 95°C

- ⑤ Initial E +600mV for 1/2 hr.
 Second E +400mV for 18 hr.
 Third E +150mV until end.

- ⑥ Initial pH 8.03 Final pH 9.32

Solution was murky.

The current was quite high at 150 mV even after a long time (hrs). Large amount of corrosion product was present on the specimen. When

- ⑦ a glass the potential was reduced to 100 mV, no significant repassivation was seen for 24 hours - 2000 seconds. A glass rod was used to scrape the corrosion products gently. The current decreased immediately to 1.6×10^{-6} A/cm².

Stored on REPTIME # 2

Walter J. Mackowski
8/27/92

10/1/92

Preparation of stock solutions and Test solutions

Sulfate stock solution:

A 1000 ppm SO_4^{2-} stock solution was prepared by adding 1.4787 g of Na_2SO_4 to make 1 liter of solution in a volumetric flask. Actual weight was 1.4745 g. Lot No. 901213 (Fisher). stored in volumetric flask.

Nitrate stock solution

A 1000 ppm NO_3^- stock solution was prepared by adding 1.3763 g of NaNO_3 , lot No. 897183 (Fisher) to make 1 liter of solution. stored in volumetric flask.

Fluoride stock solution

A 1000 ppm F^- stock solution was prepared by adding 2.2102 g of NaF (lot No. 896405) to make 1 l of solution. This was stored in a polyethylene bottle.

Test solutions will be prepared using the above stock solutions.

N. Smith
11/1/92

10/1/92

Preparation of Test Solution of 1000 ppm Cl^-

0.2459

~~0.12198~~ g of NaHCO_3 (lot No. 897789) was added.3.2958 g of NaCl (lot No. 922649A) was addedThe salts were added to beaker, dissolved in nanopure water (17 M Ω), then poured in a 2-l volumetric flask.Ahs pipet 4 ml of F^- stock solution (p. 119)Ahs pipet 20 ml of NO_3^- stock solution (p. 119)Ahs pipet 40 ml of SO_4^{2-} stock solution (p. 119)

Make up total solution's volume to 2 l.

Label + date flask.

N. S. *[Signature]*
10/1/92

10/2/92

825 Surf1000 ppm Cl^- , 95°C

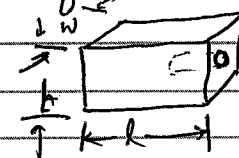
Purpose: To investigate the effect of surface Cr depletion on localized corrosion of alloy 825 using cyclic polarization test technique.

Material: Alloy 825, Heat HH4371FC, 0.5" plate

Solution: Prepared as shown on p. 120.

Test: Same procedure as TOP-008. The verification test (ASTM G-61) was conducted in a previous series by Donald Pile (depassivation tests). Hence it was not conducted again. The only difference was the surface preparation. Two surfaces were left in the as-mill finished condition. All the other surfaces were polished to 600-grit finish. Specimen was washed, degreased in acetone & dried before placing in cell. PAR 173/276 was used along with M342C software.

Parameters: Surface Area: length: 0.683 in



width: 0.528 in

Thickness: 0.483 in.

Area: 12.2 cm^2 (neglecting hole)

Temp: 93°C (Thermometer: 1238004)

Initial Measurements: Potential of Pt (before connecting to 173): -0.298

(used Keithley 614 electrometer, Ser. No. 467374) Potential of specimen: -0.589

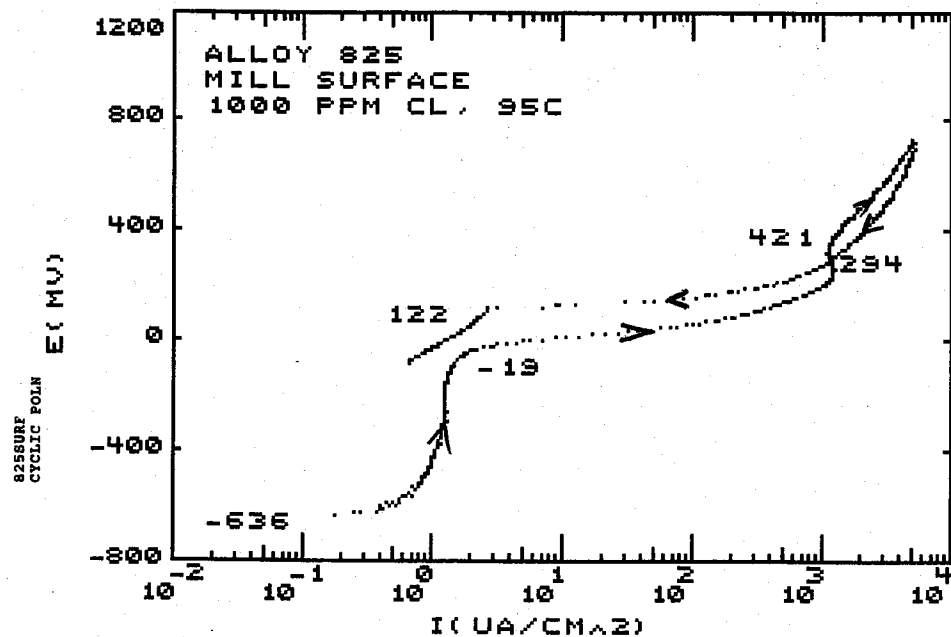
Ref. Electrode: Sat. Calomel, #1.

Initial pH (at 19°C): 8.30

Final pH (at 19°C): 9.92 (after 2 days)

Observation: The specimen showed uniform corrosion on the two mill-finished faces before onset of pitting. Pits were found on all faces. Data stored in file 2 (825surf).

N. S. *[Signature]*
10/2/92



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825SURF
INITIAL E (MV)	0 VS E
VERTEX E (MV)	100 VS E
FINAL E (MV)	2 VS E
SCAN RATE (MV/S)	.17
THRESHOLD I (UA/CM^2)	5000
CONDITION E (MV)	PASS
CONDITION T (S)	PASS
INIT DELAY (MV/S OR S)	3600 S

SAMPLE PARAMETERS

AREA (CMS^2)	12.2
EQ WT (GM)	PASS
DENSITY (GM/CM^3)	PASS
CATHODIC TAFEL (MV)	PASS
ANODIC TAFEL (MV)	PASS

DATA SCALE

ECORR	-636
HV/PT	4
DATA MAX	5221.312
DATA MIN	8.442623E-02
ABS MIN	8.442623E-02
ABS MAX	5221.312

RESULTS

E(I=0) (MV)	DEAERATE WITH ARGON
CATHODIC TAFEL (MV)	
ANODIC TAFEL (MV)	
I-CORR (UA/CM^2)	
CORR RATE (MPY)	
E(I=0) (MV)	
POL RES. (K-OHMS CM^2)	
I-CORR (UA/CM^2)	
CORR RATE (MPY)	

LEGEND

ALLOY 825 WITH MILL SURFACE
1000 CL
20 SO4
10 NO3
2 F
85 HCO3
95 C
DONE IN PAR 173/276
SCAN RATE 0.17 MV/SEC

10/6/92

Stock solution : 600 ppm Cl^-

0.988 g of NaCl to make 1 liter of solution
used lot No. 922649A (Fisher) of NaCl and Nanopure
water.

Test solution (6 ppm Cl^-):

Add 10 ml of 600 ppm Cl^- stock solution
Add 20 ml of SO_4^{2-} stock solution (p. 119)
Add 10 ml of NO_3^- stock solution (p. 119)
Add 2 ml of F^- stock solution (p. 119)
Add 0.122 g of NaHCO_3 (lot No. 897789, Fisher)
Make up solution to 1 l.

N. Snicker
10/6/92

N. Snicker
10/5/92

10/6/92 825 SUR2

Purpose: To test the effect of surface condition on cyclic polarization behavior of Inconel alloy 825 in 6 ppm Cl solution.

Material: Same as p. 121

Solution: Same as p. 123

Test: Same as p. 121.

Temperature: 95°C (Temp decreased to 90°C at start of test but was increased subsequently).

O.C. Potential of Pt: -0.29 V SCE } Keithly 614
 O.C. Potential of Specimen: } Sr. No. 467374
 O.C. Potential of Specimen at end of test: -0.512 V SCE .

Initial pH: 8.259 (at 19.8°C)

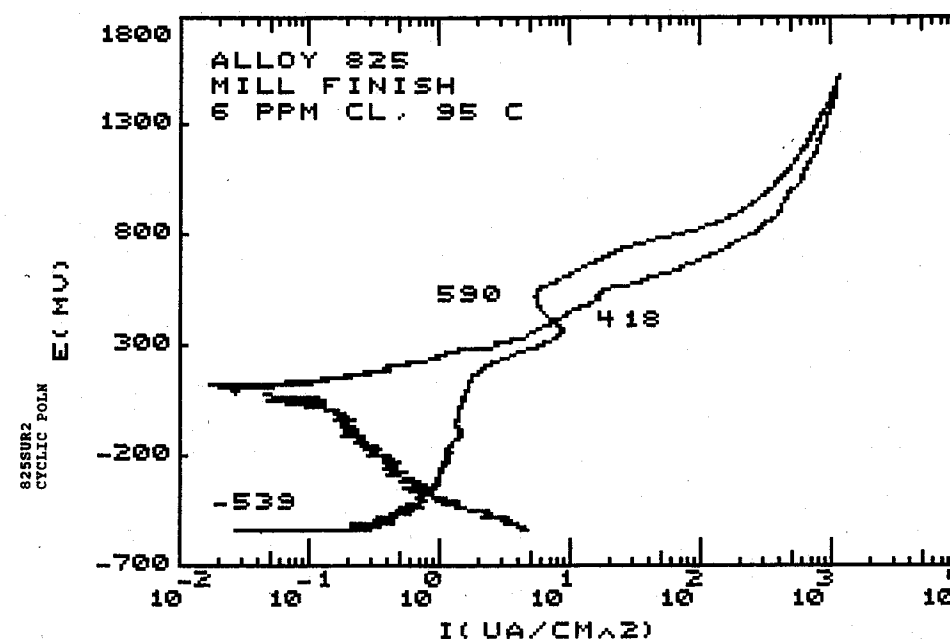
Final pH: 10.195 (at 19.8°C)

Observations: The specimen showed signs of pitting only on the mill-finish surface. Initially small pits were formed on both mill-finish faces after about 590 mV SCE. These then grew laterally to form greenish deposits over a wide area. The solution was slightly yellow. The green deposit washed off easily even with a gentle stream of water.

Data stored in File 2 disc.

N. Snidker
10/6/92

825 SUR2



RUN PARAMETERS

TECHNIQUE	CYCLIC POLN
ORIGINAL NAME	825SUR2
INITIAL E (mV)	0 VS E
VERTEX E (mV)	100 VS R
FINAL E (mV)	1 VS E
SCAN RATE (mV/s)	17
THRESHOLD I (uA/cm^2)	5000
CONDITION E (mV)	PASS
CONDITION T (s)	PASS
INIT DELAY (mV/s OR s)	3600 s

SAMPLE PARAMETERS

AREA (CMS^2)	12.2
EQ WT (GM)	PASS
DENSITY (GM/CM^3)	PASS
CATHODIC TAFEL (mV)	PASS
ANODIC TAFEL (mV)	PASS

DATA SCALE

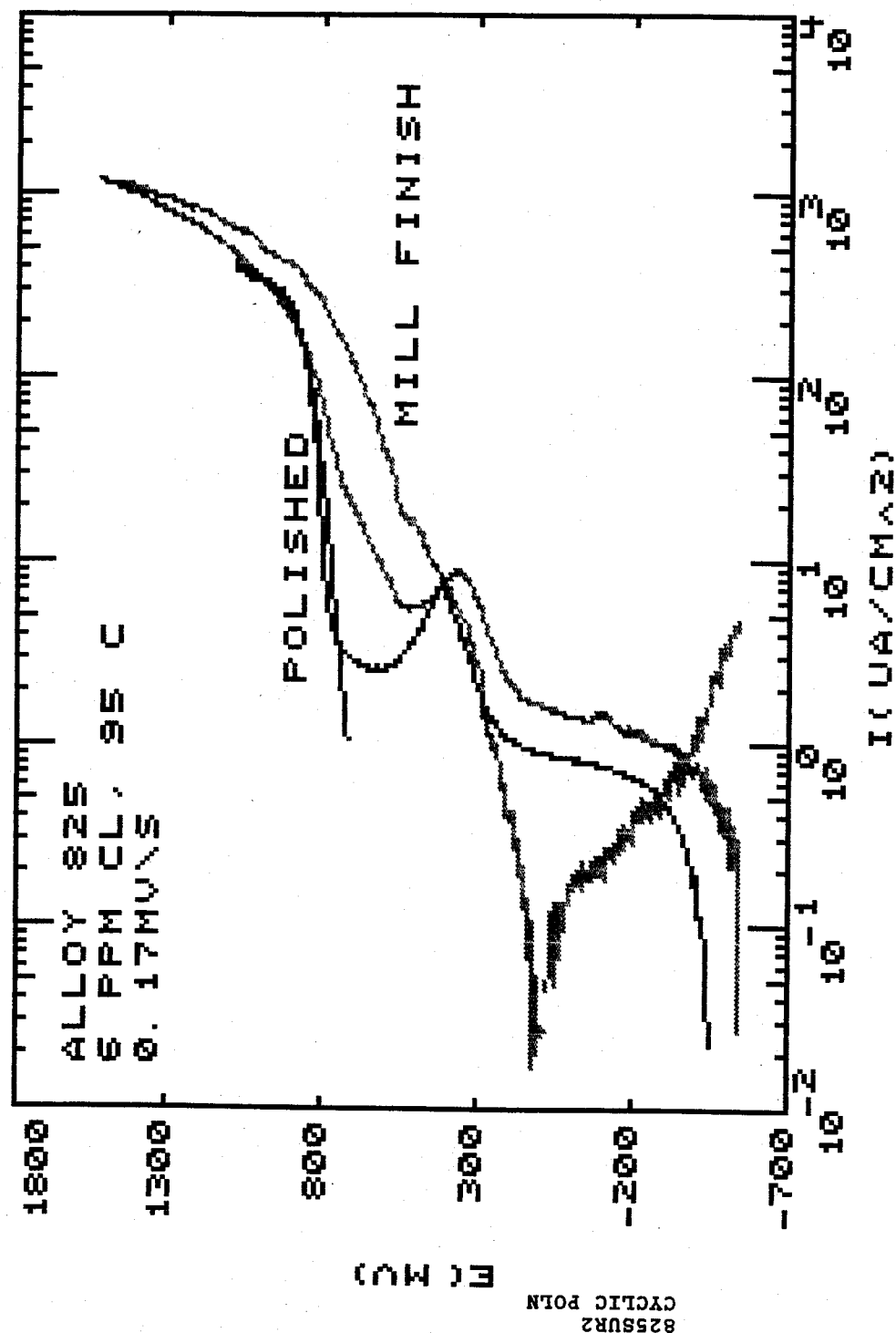
ECORR	-539
MV/PT	4
DATA MAX	1170.492
DATA MIN	-4.770492
ABS MIN	0
ABS MAX	1170.492

LEGEND

825 SUR2
6 PPM CL AT 95 C
MILL FINISH SURFACE
AR DEAERATED
0.17 M/SEC

N. Snidker
10/7/92

Polished vs. Mill Finished Alloy 825 in 6 ppm Cl⁻, 95°C



N. Sridhar
10/7/92

10/15/92

REFER TO PAGE 295 for Objectives and Justification
4/28/94 127
mV vs PPM Cl⁻ FOR MI 200/MI 403 ELECTRODES
USING ORION 720 A, SN 003368, CAL 10 FEB 92
NaCl LOT # 922649 A QA ACCEPTED 8/11/92
CONC Cl⁻ MI 200/MI 403 #1 MI 200/MI 403 #2

1 ppm	285 mV (DRIFTING)	282 mV (DRIFTING)
10 ppm	226 mV (DRIFTING)	244.2 mV (DRIFTING)
100 ppm	163.3 mV	173.8 mV
1000 ppm	111.0 mV	110.5 mV
10000 ppm	68.9 mV	63.6 mV
17725 ppm	58.9 mV	52.9 mV
35450 ppm	46.2 mV	40.7 mV

MI 200/MI 403 #1 $mV = -49.81 \log(\text{PPM Cl}^-) + 268.41$

MI 200/MI 403 #2 $mV = -56.87 \log(\text{PPM Cl}^-) + 292.49$

[Signature]

10/16/92

COMPARE FISHER A₁/A₉Cl ELECTRODE 13-620-53
SN 8118182 TO MICROELECTRODES INC
MI 402 A₁/A₉Cl

ELECTRODES CONNECTED TO KEITHLEY 614 ELECTROMETER
MI 402 TO RED (HIGH) AND FISHER 13-620-53
TO BLACK LEAD

CONC Cl ⁻	MI 402 #1 VS FISHER mV	MI 402 #2 VS FISHER mV	MI 402 #3 VS FISHER mV
10 ppm	0.9-4.0	4.0-8.0	5.5-7.0
100 ppm	1.1-3.7	7.0-9.0	4.1-4.5
1000 ppm	1.9-3.2	0.9-2.0	0.2-1.0
0.5 MCl ⁻	5.3-7.5	3.5-6.0	3.0-5.0
1.0 MCl ⁻	4.7-8.2	3.7-6.0	3.0-5.0

[Signature]
10/16/92

10/20/92

MI 506/MI 402 PH CALIBRATION

ORION 720A ORION EA 920 FISHER 950

SN 003368 SN 5001A SN 3340

MI 506 #1/MI 402 #1 MI 506 #2/MI 402 #2 MI 506 #3/MI 402 #3

PH	mV	mV	mV
1.00	339.3	336.4	333.6
4.00	170.4	166.8	162.3
7.00	-2.1	-5.4	-6.9
10.00	-179.6	-183.4	-184.6

MI 506 #1 SN # 43717 / MI 402 SN # ~~41402~~ ⁴¹⁴³² 7/27/94
 WITH ORION 720A SN 003368
 $mV = -57.64 (pH) + 399.02$

MI 506 #2 SN # ~~43717~~ ⁴⁴⁰⁶⁰ / MI 402 #2 SN # ~~41402~~ ⁴¹⁴³⁶ 7/27/94
 WITH ORION EA 920 SN 5001A
 $mV = -57.72 (pH) + 396.06$

MI 506 #3 SN # ~~44060~~ ⁴³⁷¹⁷ / MI 402 #3 SN # ~~41436~~ ⁴¹⁴³² 7/27/94
 WITH FISHER ACCUMET 950 SN 3340
 $mV = -57.46 (pH) + 392.13$

Paul D.
 10/20/92

10/22/92

ASSEMBLE CREVICE CELL

SAMPLE 304L HEAT # T0954 600 GRIT FINISH

PLACED IN CREVICE CELL. AFTER WASHING IN
 DI WATER AND ACETONE

ADJUSTED ELECTRODES TO JUST ABOVE LOWER EDGE
 OF TOP PLATE OF CELL 2 PH ELECTRODES
 1 AT CREVICE MOUTH 1 AT CREVICE TIP 1 CI ELECTRODE
 AT CENTER OF CELL

10/22/92

ASSEMBLE CREVICE CELL CONT'D

1 PH ELECTRODE PLACED IN BULK SOLUTION

1 CI ELECTRODE PLACED IN BULK SOLUTION

SOLUTION WAS 1000 PPM ? ~~CI~~ ^{AS} NaCl
 LOT 922649A NO HCO_3^- SO_4^{2-} NO_3^- OR F-

10/23/92

START CREVICE CORROSION EXPT.

NITROGEN HIGH PURITY GAS BUBBLED INTO SOLN
 THROUGHOUT ENTIRE TEST.

DATA COLLECTED W/ PC LAB FILE 304L600.ASC
 MANUAL LOG ON PAGES 130-131

INITIAL READINGS

PCLAB	PCLAB PH	METER	READING
PH #1	6.21	ORION 720A	6.253
PH #2	6.82	FISHER 950	6.895
PH #3	6.65	ORION 920	6.78

10/26/92

ENDED EXPERIMENT, DISASSEMBLED CELL
 CHECKED CALIBRATION OF PH REF AND CI ELECTRODES.
 ON PAGES 132

Paul D. 10/26/92

TIME	APPLIED V _{AG} CL BULK	APPLIED V _{AG} CL TIP	CURRENT	CI BULK	CI CENTER	CH 1 PH (720) BULK	CN 2 PH (F 950) MOUTH	CN 3 PH (0920) TIP	COMMENT
10/23/92 9:30 AM	-0.1548	-0.1620		89.8 uV	100.8 mV	6.16	6.89	6.69	Type 304 L 600 mV tip (T0954)
10:00 AM	-0.1575	-		89.4	101.0	6.24	7.035	6.88	
10:10 AM	+0.1012		24 uA	89.3	112.6	6.245	6.990	6.82	
10:55 AM	+0.1012		83.44	89.2	102.7	6.397	6.874	6.77	
11:25 AM	+0.1012		23.44 uA	89.2	102.3	6.530	6.846	6.75	
12:40 PM	+0.1012	+0.0952	49.50 uA	89.3	101.5	7.533	6.774	6.70	
1:33 PM	+0.1011	+0.0952	58.3 uA	89.2	101.2	7.921	6.734	6.66	1:38 PM INCREASE APPLIED V TO 200 uA
1:40 PM	+0.2009	+0.1975	103.3 uA	89.3	101.6	7.961	6.692	6.67	
2:25 PM	+0.2009	+0.2006	0.2002 mA	88.6	98.5	8.462	6.590	6.66	
3:07 PM	+0.2009	+0.2012	0.2214 mA	88.3	98.2	8.656	6.530	6.63	
3:11 PM	+0.2009		0.1978 mA	92.6	99.2	7.687	6.611	6.63	
3:17 PM	+0.3002		0.2612 mA	92.0	99.4	7.673	6.504	6.62	
3:31 PM	+0.3002	+0.2999	0.3122 mA	92.0	97.5	8.064	6.445	6.19	
4:53 PM	+0.3002	+0.0877	0.5400 mA	91.9	108.7	8.844	6.125	3.03	
6:00 PM	+0.3002	0.0504	0.67 mA	91.8	24.8	8.954	6.114	3.31	
7:00 PM	0.3002	0.0035	0.498 mA	91.5	9.4	9.217	3.625	3.58	
10/24/92 8:25 AM	0.3002	-0.0162	0.843 mA	91.5	36.8	9.714	2.715	3.49	
8:28 AM	-0.0002	-0.119	0.2460 mA	91.6	70.8	9.722	3.055	3.15	
7:31 AM	-0.0002	-	86.01 uA	91.9	106.0	9.747	3.126	3.15	
10:33 AM	-0.0002	-	54.78 uA	92.0	114.0	9.72	3.110	3.19	

N. Snidder
10/26/92

TIME	APPLIED V _{AG} BULK	APPLIED V _{AG} CL TIP	CURRENT	CL BULK (720A) (mV)	CL CENTER (EA 920) (mV)	CH 1 PH (720A) BULK	CH 2 PH (F 950) MOUTH	CH 3 PH (EA 920) TIP	Comments
10/24/92 10:47 AM	-0.0002	-0.059	47.03 uA	92.1	115.9	9.689	3.120	3.18	Type 304 L SS 600 mV tip
3:39 PM	-0.0002	-0.0393	24.58 uA	92.1	128.4	9.668	3.132	3.29	finish.
6:25 PM	-0.0002	-	21.59 uA	92.0	124.1	9.672	3.290	3.33	
6:25 PM	-0.0511	-0.086	20.61 uA	92.0	121.2	9.671	3.31	3.37-3.30	
3:05 PM	-0.0511	-0.0773	8.17 uA	91.1	109.0	9.52	3.409	3.56	
7:05 AM	-0.0511	-0.0785	7.72 uA	89.0	98.4	9.749	3.468	3.75	
7:10 AM	-0.1653	-0.1911	-	89.1	92.7	7.741	3.622	3.63	Disconnected Platinum

N. Snidder
10/26/92

10/26/92

PH CALIBRATION CHECK

ELECTRODES: MI 506 #1 MI 402 #1
 METER: ORION 720A, PC LAB PH #1

<u>BUFFER PH</u>	<u>ORION</u> <u>mV</u>	<u>PH</u>	<u>PC LAB PH</u>
1.00	334.7	2.151	1.17
4.00	171.4	4.51	4.00
7.00	-6.6	6.99	7.05
10.00	-187.6	9.57	10.20

ELECTRODES: MI 506 #2 MI 402 #2
 METER: FISHER 950 PC LAB PH #2

<u>BUFFER PH</u>	<u>ORION</u> <u>mV</u>	<u>PH</u>	<u>PC LAB</u> <u>PH</u>
1.00	326.6	1.183	4.67
4.00	159.5	4.09	5.48
7.00	-12.8	7.08	6.93
10.00	-189.0	10.135	8.41

ELECTRODES: MI 506 #3 MI 402 #3
 METER: ORION EA 920 PC LAB PH #3

<u>BUFFER PH</u>	<u>ORION</u> <u>mV</u>	<u>PH</u>	<u>PC LAB</u> <u>PH</u>
1.00	333.1	1.30	1.07
4.00	165.1	4.19	3.99
7.00	-8.4	7.15	7.00
10.00	-188.9	10.20	10.80

10/26/92

Cl⁻ CALIBRATION CHECK

ELECTRODES: MI 200 #1 / MI 403 #1, MI 200 #2 / MI 403 #2
 METERS: ORION EA 920, ORION 720A

	<u>ORION EA 920</u> <u>MI 200 #1 / MI 403 #1</u> <u>mV</u>	<u>ORION 720A</u> <u>MI 200 #2 / MI 403 #2</u> <u>mV</u>
PPM Cl ⁻		
10	207.1	211.7
100	160.5	150.8
1000	110.7	93.7
17725	54.2	34.2
35450	38.4	16.7

REFERENCE ELECTRODE CALIBRATION CHECK

ELECTRODES: MI 402 #'s 1, 2, 3 VS FISHER 13-620-53, 8118182
 METER: ORION EA 920, MI 200 TO REF JACK, FISHER TO BNC

	<u>MI 402 #1</u> <u>mV</u>	<u>MI 402 #2</u> <u>mV</u>	<u>MI 402 #3</u> <u>mV</u>
PPM Cl ⁻			
10	-7.6	-6.8	-10.6
100	-7.8	-6.5	-10.2
1000	-9.5	-8.2	-10.8
17725	-11.5	-11.0	-11.2
35450	-10.9	-10.6	-10.8

James A. Dineen
 10/26/92

10/29/92

CREVICE EXPT 825-1

SET UP CREVICE CELL AS ON PAGE 129
EXCEPT USING COMUADD 333T COMPUTER
AND WORKBENCH WITH ESC 440-2 POTENTIOSTAT

ALLOY 825 SAMPLE PLACED IN CELL BOLTS
TORQUED TO 20 IN OZ SOLUTION INJECTED INTO
CELL PRIOR TO TORQUE

2 PH ELECTRODES IN CELL 1 AT MOUTH 1 AT
CREVICE TIP 1 PH ELECTRODE IN BULK

2 Cl^- ELECTRODES 1 IN CENTER OF CREVICE
1 IN BULK

1 Ag/AgCl BULK ELECTRODE FISHER 13-620-53
SN 8118182 IN BRIDGE TUBE w/ 0.5M NaCl

CELL PLACED IN GLASS VESSEL WITH $\approx 500\text{ ml}$
SOLUTION: @ 6:20 PM

0.5M NaCl LOT 922649A

85 PPM HCO_3^- AS NaHCO_3 LOT 923337

20 PPM SO_4^{2-} AS Na_2SO_4 FROM STOCK SOLN

10 PPM NO_3^- AS NaNO_3 FROM STOCK SOLN

2 PPM F^- AS NaF FROM STOCK SOLN

HIGH PURITY N_2 GAS BUBBLED INTO SOLN
OVERNIGHT.

Daniel A. D.
10/29/92

10/30/92

CREVICE EXPT 825-1

CREVICE EXPERIMENT STARTED DATA STORED
IN C:\WB\DATA\825-1.DAT ON COMPUADD
333T MANUAL DATA LOG ALSO KEPT

Cl ELECTRODES NOT WORKING DURING TEST

TEST ENDED ~~10/30/92~~ ^{7/27/94} 11/3/92 PH
OF CREVICE 9.067 AT LOWEST VALUE
CURRENT VERY HIGH $2.6 \times 10^{-3}\text{ A}$ AT 200 mV
SAMPLE AREA 32.74 cm^2

SAMPLE EXAMINATION REVEALED SMALL
PITS IN CREVICE AREA AND LARGE
PITS UNDERNEATH MICROSTOP APPLICATION
WHEN 825 ROD MEETS FLAT SAMPLE

CROSS CHECK OF PH ELECTRODES INDICATES
THEY ARE READING CORRECTLY

DATA LOG ON PAGES ~~135~~ ^{7/27/94} 136-137

Daniel A. D.
10/30/92

PH AND Cl ISE CALIBRATION DATA STORED
IN 8251CAL.DAT ON IWPE-025 DISC

Daniel A. D. 4/28/94

[illegible][illegible]

11/11/92

CREVICE CORROSION ALLOY 825 #2

STARTED 11/6/92

ALLOY 825 SAMPLE 600 GRIT FINISH
 0.5 M NaCl, 85 PPM NaCO_3^- , 20 PPM SO_4^{2-} , 10 PPM NO_3^-
 2 PPM F^-

ELECTRICAL CONTACT MADE BY GRINDING
 0.091" 825 ROD FLAT AND THEN WELDING
 FLAT SIDE OF ROD TO EDGE OF THE SAMPLE
 0.032 STAINLESS ROD (WIRE) (304LSS) CONNECTED
 TO ALLOY 825 ROD ABOVE SOLUTION LEVEL
 NO MICROSTOP USED TO COAT CONTACTS.

SAMPLE PLACED INTO DELRIN CREVICE CELL
 SOLUTION INJECTED INTO CELL AND BOLTS
 TORQUED TO 20 IN. OZ ELECTRODE PLACEMENT
 AS LISTED ON PP. 128-129 ELECTRODE TIPS
 SURROUNDED BY GLASS WOOL. PH ELECTRODES
 CALIBRATED ON 11/5/92 SLOPE & INTERCEPT
 VALUES ENTERED INTO CREVICE.WAB ON WORKBENCH
 PROGRAM ON COMPAQ 333T COMPUTER

MI 506 #1 / MI 402 #1

$$mV = -57.416 (pH) + 390.517$$

$$pH = -17.416 (V) + 6.802$$

MI 506 #2 / MI 402 #2

$$mV = -57.253 (pH) + 383.433$$

$$pH = -17.467 (V) + 6.697$$

MI 506 #3 / MI 402 #3

$$mV = -57.48 (pH) + 391.44$$

$$pH = -17.397 (V) + 6.180$$

MI 200^{#2} / MI 403 #2

$$mV = -46.353 * \log(\text{PPM Cl}^-) + 239.794$$

Daniel A. D. 11/11/92

11/16/92

CREVICE CORROSION ALLOY 825 #2

STARTED 11/6/92 ENDED 11/16/92

DECREASE IN PH AT TIP AND MOUTH
 AFTER V_{APP} 0.400 V (11/13/92) Cl^- CENTER
 CONCENTRATION VARIED BUT WAS NEVER
 ABOVE ≈ 1.0 M

EXAMINATION OF SAMPLE INDICATES THAT
 CREVICE CORROSION OCCURRED ALONG THE ENTIRE
 UNDERNEATH SIDE OF SAMPLE. SOME GREEN
 CORROSION PRODUCTS NEAR TIP AND GLASS
 WOOL IN TIP WAS DISCOLORED. MUCH
 GREEN CORROSION PRODUCT AROUND MOUTH ON
 LOWER DELRIN BLOCK. NO CORROSION
 VISIBLE ON SAMPLE SURFACE OUTSIDE OF
 DELRIN CELL. NO CORROSION OBSERVED IN
 AREA OF WELD (SAMPLE TO 825 ROD).
 SMALL PITS VISIBLE ON UPPER SAMPLE SURFACE.
 SIMILAR FEATURES WERE OBSERVED PRIOR TO
 TEST.

OTHER NOTES BRIEF POWER BLACKOUT ON 11/10/92
 CAUSED COMPUTER AND POTENTIostat TO RESET
 SAMPLE. WAS CATHODIC FOR SHORT PERIOD.
 DATA COLLECTED ON 825-2.DAT & 825-2R.DAT.

MANUAL DATA LOC ON PAGES 140-143

Daniel A. D.

11/16/92

PH AND Cl^- ISG DATA STORED AS 8252CAL.DAT
 IN IWPE-025 DISK. DATA USED TO CALIBRATE
 PH AND Cl^- ISG ELECTRODES Daniel A. D. 11/28/94

Time	Applied External Potential (V)	Potential in the cavity (V)	Bulk pH	Mouth pH	Tip pH	Bulk Cl ⁻	Center Cl ⁻	Current Density	Comments
11/6/92 3:00 PM	0.0884	0.0841	9.012	7.176	8.157	139.0	48.97	2.00x10 ⁻⁵ A	START OF TEST
3:30 PM	0.0984	0.0842	9.033	7.192	8.171	140.1	47.1	5.5x10 ⁻⁶ A	
4:25 PM	0.0984	0.0845	9.066	7.262	8.264	140.4	47.4	1.9x10 ⁻⁶ A 3.0x10 ⁻⁶ A	
5:12 PM	0.0984	0.0845	9.094	7.298	8.325	142	46.4	2.7x10 ⁻⁶ A 3.5x10 ⁻⁶ A	0-920 ON CN 2: PA TIP VALUES ARE NOT CORRECT.
11/9/92 10:43 AM	0.0985	0.0842	9.406	7.534	9.312 5.923	109.0	48.1	1.0x10 ⁻⁶ A 1.5x10 ⁻⁶ A	
1:15 PM	0.0985	0.0842	9.427	7.551	9.259	110.9	46.0	1.0x10 ⁻⁶ A 2.8x10 ⁻⁶ A	
11/9/92 10:55 AM	0.0985	0.0870	9.585	7.694	9.404	97.1	42.1	2.4x10 ⁻⁶ A 3.6x10 ⁻⁶ A	
11/9/92 12:55 PM	0.0983	0.0853	9.603	7.688	9.45	106.2	42.4	3.2x10 ⁻⁶ A	Current is rising
7:57 AM	0.147	0.1333	9.600	7.752	9.473	51.7	39.7	6.4x10 ⁻⁶ A 7.5x10 ⁻⁶ A	
10:07 AM	0.147	0.1338	9.569	7.710	9.415	32.9	39.6	6.8x10 ⁻⁶ A 7.5x10 ⁻⁶ A	PURGED N ₂ FOR 1/2 10-11 AM
11:48 AM	0.147	0.1382	9.418	7.679	9.359	35.1	39.5	6.8x10 ⁻⁶ A 7.7x10 ⁻⁶ A	0-920 ON CN 2: PA
1:09 PM	0.147	0.1334	9.411	7.658	9.315	35.8	39.3	6.5-7.5x10 ⁻⁶ A	INCORRECT
3:07 PM	0.147	0.1330	9.368	7.634	9.244	31.4	39.2	6.5-7.5 x10 ⁻⁶ A	BULK CL ⁻ REF NAs BUBBLING

Quilley 10/16/92

Time	Applied External Potential (V)	Potential in the cavity (V)	Bulk pH	Mouth pH	Tip pH	Bulk Cl ⁻	Center Cl ⁻	Current Density	Comments
11/9/92 4:30 AM	0.147	0.1329	9.362	7.621	9.190	—	38.9	6.7-7.8 x10 ⁻⁶ A	
5:05 AM	0.147	0.1327	9.331	7.616	9.172	—	38.9	6.2-7.5 x10 ⁻⁶ A	0-920 ON CN 2: PA
11:43 PM	0.147	0.1317	9.320	7.608	8.790	—	38.7	6.0-7.2 x10 ⁻⁶ A	INCORRECT.
11/10/92 8:06 AM	0.147	0.1315	9.198	7.623	8.582	—	37.7	6.0-7.2 x10 ⁻⁶ A	
8:45 AM	—	—	—	—	—	—	—	—	POWER OUT Computer Restart
10:30 AM	—	—	UNMODIFIED	—	—	—	—	—	
11:30 AM	0.147	—	—	7.615	8.936	—	37.3	3.7-4.6 x10 ⁻⁶ A	
1:17 PM	0.147	—	—	7.618	8.892	—	37.3	3.5-4.2 x10 ⁻⁶ A	
11/11/92 7:46 AM	0.147	—	8.891	6.869	8.077	—	—	—	
11:14 AM	0.147	0.1820 0.1163	8.970	6.870	7.964	—	37.0	2.2-3.0 x10 ⁻⁶ A	
12:05 PM	0.197	0.182	8.943	6.870	7.861	—	37.0	1.7-3.0 x10 ⁻⁶ A	
12:58 PM	0.197	0.1819	8.931	6.871	7.870	—	36.4-37.4	1.5-2.4 x10 ⁻⁶ A	
1:47 PM	0.197	—	8.924	6.871	7.850	—	36.2-37.5	1.4-2.3 x10 ⁻⁶ A	

Quilley 11/16/92

Time	Applied External Potential (V)	Potential in the Corrosion	Bulk pH	Mouth pH	Tip pH	Bulk Δ	Center Δ	Current Density	Comments
11/10/92 3:27 PM	0.198		8.879	6.878	7.803		36.3	1.0×10^{-6}	
5:25 PM	0.198	0.1820	8.905	6.876	7.707		36-37mV	6.5×10^{-7} 1.2×10^{-6}	
10:25 PM	0.198	0.1817	8.850	6.881	7.556		36-37mV	10^{-7}	CURRENT $10 \times 10^{-7} - 2 \times 10^{-6}$
11/12/92 8:50 AM	0.2471 0.198	0.2307	8.691	6.901	7.330		35-36mV	-10^{-7} 10^{-7}	
11:23	0.298	0.2821	8.584	6.904	7.286		36-37mV	-10^{-7} -10^{-6}	
2:07 PM	0.298		8.560	6.924	7.200				
5:10 PM	0.298	0.2819	8.435	6.871	7.090		41-42mV	-10^{-7} -10^{-6}	
6:01 PM	0.298	0.2820	8.654	6.927	7.143		36-37mV	-1.2×10^{-6}	
11/13 7:20 AM	0.40	0.3830	9.12	6.95	7.03	—	36-37	-1.2×10^{-6}	On hanging on all night
11:30 AM	0.398 0.40	0.381	9.809	6.03	7.798		25-26	$3-4 \times 10^{-5}$	
1:25 PM	0.3986	0.381	9.909	5.647	7.912		29-30	1.7×10^{-4}	
2:59 PM	0.3985	0.384	9.817	5.350	7.926		36-37	8.6×10^{-4}	
4:35 PM	0.3985	0.3826	10.270	5.378	8.054 8.393		-13-14mV (NEGATIVE)	6.5×10^{-4}	

David R. 11/16/92

Time	Applied External Potential (V)	Potential in the Corrosion	Bulk pH	Mouth pH	Tip pH	Bulk Δ	Center Δ	Current Density	Comments
11/13/92 5:12 PM	0.3985	0.3826	10.070	5.062	8.094		36-37mV	6.7×10^{-4}	
11/14/92 9:23 AM	0.3985	0.3793	10.480	3.828	8.813		72-73	8.6×10^{-4}	
6:03 PM	0.3985	0.3739	10.576	3.102	8.580		138.2	6.2×10^{-4}	
11/15/92 11:00 AM	0.3985	0.3160	11.297	3.833	5.070		260.2	7.6×10^{-4}	POTENTIAL IN REFERENCE LOW
4:04 PM	0.3985	0.1707	11.788	4.102	2.001		237-238	9×10^{-4}	
11/16/92 7:53 AM	0.3985	0.1636	12.742	3.629	1.143		253-254	1.1×10^{-3}	SET POTENTIAL AT 0.00 V CITAT CORROSION
7:54 AM									94mV
9:28 AM	0.0009	-0.0277	9.887	5.021	1.951		88-89	7.1×10^{-5}	

David R. 11/16/92

11/11/92

REPASSIVATION EXPT ALLOY 825

EXPT NAME: REPTIM11, 11/11/92 4:15 PM
 SAMPLE: ALLOY 825 600 GRIT 15% HNO_3 CLEANED
 RINSED IN DI WATER.

SOLUTION: 1000 PPM Cl^- , 20 PPM SO_4^{2-} , 10 PPM NO_3^- ,
 85 PPM NaCO_3^- , 2 PPM F^- ,
 DEAIRATED W/ 99.999% N_2 AND
 MAINTAINED AT 95°C USING
 GLAS-COL TM 512 HEATERS
 TEMP CHECKED W/ N_2 THERM. # 1238004

POTENTIOSTAT: ESC 440 LABELED 440-1
 CHANNEL # 5 BOARD # 3
 CALIBRATED 13 JAN 92 DUE 13 JAN 93
 POTENTIOSTAT CONTROLLED W/ WORK BENCH
 REPTIMER.WBB DATA SAVED AS:
 C:\WB\DATA\REPTIM11.DAT AND:
 B:\REPTIM11.DAT ON REPTIME #2 3.5" DISK

PIT INITIATION: 600 mV SCE 30 min

PIT GROWTH 400 mV SCE 6 HOURS

PIT REPASSIVATION: 150 mV SCE

SCE ELECTRODE: FISHER 13-620-51 SN 0169033

11/12/92

LOGIC ERROR IN REPTIMER.WBB PROGRAM
 REPASSIVATION POTENTIAL WAS CATHODIC (-50 mV)
 FOR ≈ 5 min. SAMPLE REPASSIVATED VERY
 QUICKLY.

Daniel A. D.
 11/12/92

11/11/92

REPASSIVATION EXPT ALLOY 825

EXPT NAME: REPTIM12, 11/11/92 4:15 PM
 SAMPLE: ALLOY 825 600 GRIT 15% HNO_3 CLEANED
 RINSED IN DI WATER.

SOLUTION: 1000 PPM Cl^- , 85 PPM NaCO_3^- , 20 PPM SO_4^{2-} ,
 10 PPM NO_3^- , 2 PPM F^- ,
 DEAIRATED W/ 99.999% N_2 AND
 MAINTAINED AT 95°C USING GLAS-COL
 TM 512 HEATER. TEMP CHECKED W/
 N_2 THERM. # 1238001

POTENTIOSTAT: ESC 440 LABELED 440-1
 CHANNEL # 3 BOARD # 2
 CALIBRATED 13 JAN 92 DUE 13 JAN 93
 POTENTIOSTAT CONTROLLED W/ WORK BENCH
 REPTIMER.WBB DATA SAVED AS:
 C:\WB\DATA\REPTIM12.DAT AND AS:
 B:\REPTIM12.DAT ON REPTIME #2 3.5" DISK.

PIT INITIATION: 600 mV SCE 30 min

PIT GROWTH: 400 mV SCE 6 HOURS

PIT REPASSIVATION: 200 mV SCE

SCE ELECTRODE: FISHER 13-620-51 SN 8211163

11/12/92

LOGIC ERROR IN REPTIMER.WBB PROGRAM
 REPASSIVATION POTENTIAL WAS CATHODIC (-100 mV)
 FOR ≈ 5 min. SAMPLE REPASSIVATED VERY
 QUICKLY.

Daniel A. D.
 11/12/92

11/13/92

REPASSIVATION EXPT ALLOY 825 REPTIM 13

SAMPLE: ALLOY 825 600 GRIT FINISH CLEANED
IN 15% HNO_3 AND RINSED IN DI WATER
SOLUTION: SAME AS REPTIM 11 & REPTIM 12
1000 PPM Cl^- , 85 PPM NO_3^- , 20 PPM SO_4^{2-}
10 PPM NO_3^- , 2 PPM F^- DEAIRATED
WITH HIGH PURITY N_2 AND MAINTAINED
AT 95°C WITH GLAS-COL TM 512
TEMP CHECKED WITH Hg THERMO # 1238004
POTENTIOSTAT: SAME AS REPTIM 11 P 144
DATA STORED AS B: REPTIM13.DAT AND
C:\WB\DATA\REPTIM13.DAT
PIT INITIATION: 600 mV SCE FOR 30 min 11:50 AM
PIT GROWTH: 400 mV SCE FOR 6 HOURS
REPASSIVATION: 150 mV SCE

OPEN CIRCUIT POTENTIALS:


SAMPLE -53 mV SCE

PT -1.5 mV SCE

SCE ELECTRODE: FISHER 13-620-S1 SN 8211163

DATA ACQUISITION RATE TOO SLOW

SAMPLE DISCONNECTED EXPT STOPPED 11/16/92


11/16/92

11/13/92

REPASSIVATION EXPT ALLOY 825 REPTIM 14

SAMPLE: ALLOY 825 600 GRIT FINISH CLEANED
IN 15% HNO_3 AND RINSED IN DI WATER
SOLUTION: SAME AS REPTIM 11 & REPTIM 12
1000 PPM Cl^- , 85 PPM NO_3^- , 20 PPM SO_4^{2-}
10 PPM NO_3^- , 2 PPM F^- DEAIRATED
WITH HIGH PURITY N_2 AND MAINTAINED
AT 95°C WITH GLAS-COL TM 512
TEMP CHECKED WITH Hg THERMO # 1238001
POTENTIOSTAT: SAME AS REPTIM 12 P 145
DATA STORED AS B: REPTIM14.DAT
AND C:\WB\DATA\REPTIM14.DAT
PIT INITIATION: 600 mV SCE 30 min 11:50 AM
PIT GROWTH: 400 mV SCE FOR 6 HOURS
REPASSIVATION: 200 mV SCE

SPECIMEN DID NOT REPASSIVATE AFTER 254,000 SEC

OPEN CIRCUIT POTENTIALS:

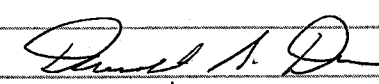
SAMPLE -432.9 mV SCE

PT -25.1 mV SCE

SCE ELECTRODE: FISHER 13-620-S1 SN 0165415

DATA ACQUISITION RATE TOO SLOW

EXPT STOPPED 11/16/92


11/16/92

148

11/19/92

REPASSIVATION EXPT ALLOY 825 REPTIM 15

SAMPLE ALLOY 825 600 GRIT FINISH RINSED
IN ACETONE, DI WATER, CLEANED IN 10% HNO_3
FOR 60 MIN. AND RINSED IN DI WATER

SOLUTION SAME AS REPTIM 11-14 1000 PPM Cl^-
85 PPM NCO_3^- 20 PPM SO_4^{2-} 10 PPM NO_3^-
2 PPM F^- SOLUTION MAINTAINED AT
95 °C AS BEFORE CHECKED WITH
 Hg THERMO. # 1238001 SOLUTION PURGED
WITH 99.999 % N_2

POTENTIOSTAT: SAME AS PAGE 144
DATA STORED AS B: REPTIM 15.DAT
AND AS C:\WB\DATA\REPTIM 15.DAT

PIT INITIATION: 600 mV SCE FOR 30 MIN STARTED 3:15 PM
PIT GROWTH: 400 mV SCE FOR ~~12 HOURS~~ 6 HOURS
REPASSIVATION: ~~150 mV SCE~~ FOR 175 mV SCE

SAMPLE START WEIGHT 11.56053
SCE ELECTRODE FISHER 1362051 SN 0169033
START Pt POTENTIAL: -54.3 mV SCE
START 825 POTENTIAL: -509.9 mV
START PH (25 °C NOT DEAERATED) 8.086

SAMPLES STOPPED AT 5:20 HELD AT -2.00 V
FOR 2 min AND RE STARTED AT 5:25 PM

SAMPLE STOPPED 11/20/92 TIME: 10:30 AM
RUNTIME: 61,400 SEC
REPASSIVATION TIME AT 175 mV SCE 275 SEC
END PH: 9.345
CHARGE DENSITY = 36 C/cm²

David A. [Signature]
11/20/92

149

11/19/92

REPASSIVATION EXPT ALLOY 825 REPTIM 16

SAMPLE: SAME AS PAGE 148.
SOLUTION: SAME AS PAGE 148 1000 PPM Cl^-
85 PPM NCO_3^- 20 PPM SO_4^{2-} 10 PPM NO_3^-
2 PPM F^- PURGED w/ 99.999% N_2 AND
MAINTAINED AT 95 °C. TEMP CHECKED
WITH Hg THERMO # 1238004
POTENTIOSTAT: SAME AS PAGE 145
DATA STORED AS B: REPTIM 16.DAT
AND AS C:\WB\DATA\REPTIM 16.DAT

PIT INITIATION 600 mV SCE FOR 30 MIN STARTED 3:15 PM
PIT GROWTH 400 mV SCE FOR 12 HOURS
REPASSIVATION 150 mV SCE

SAMPLE START WEIGHT 11.58224
SCE ELECTRODE FISHER 1362051 SN 8211163
START Pt POTENTIAL +12.8 mV SCE (OPEN CIRCUIT)
START 825 POTENTIAL -595 mV SCE. (OPEN CIRCUIT)
START PH (25 °C NOT DEAERATED) 8.086.

SAMPLE STOPPED AT 5:25 PM HELD AT -2.00 V
FOR 2.0 min AND RESTARTED AT 5:27 PM

SAMPLE STOPPED 11/20/92 TIME: 11:08 AM
RUN TIME: 63,100 SEC
REPASSIVATION TIME AT 150 mV SCE: 446 SEC
END PH: 9.302 ~~7/27/94~~
CHARGE DENSITY ~~77 C/cm²~~ 46 C/cm²

David A. [Signature]
11/20/92

11/24/92

REPASSIVATION EXPT ALLOY 825 REPTIM 17

SAMPLE. ALLOY 825 600 GRIT FINISH, RINSED IN ACETONE, DI WATER CLEANED IN 10% HNO_3 FOR 60 MIN, AND RINSED IN DI WATER

SOLUTION: SAME AS REPTIM 15. 95°C TEMP CHECKED WITH Hg THERMO 1238001 PURGED WITH 99.999% N_2

POTENTIAL STAT: SAME AS PAGE 144 DATA SAVED AS C:\WB\DATA\REPTIM17.DAT AND AS B:\REPTIM17.DAT.

CATHODIC STRIP: -2.00 V SCE FOR 2 MIN

PIT INITIATION: 600 mV SCE FOR 30 min

PIT GROWTH: 400 mV SCE FOR 6 HOURS

REPASSIVATION: 180 mV SCE

SCE ELECTRODE: FISHER 13-620-51 SN 8211163

SAMPLE START WEIGHT: 11.60060

SOLUTION PH AT 25°C 8.132

OPEN CIRCUIT POTENTIALS AT START. 11:20 AM

ALLOY 825 SAMPLE: -582 mV SCE

PT COUNTER ELECTRODE: ~~NO~~ -34 mV SCE

11/27/94

SAMPLES STOPPED 11/25/92 TIME: 2:52 PM

RUNTIME 97,800 SEC

REPASSIVATION TIME AT 180 mV SCE 43075 SEC

END PH (11/30/92): 8.873

CURRENT DENSITY LESS THAN $5 \times 10^{-5} \text{ A/cm}^2$ AT 525 SEC

AFTER SWITCHING TO 180 mV. AT 6535 SEC AFTER

180 mV CURRENT INCREASED ABOVE $5 \times 10^{-5} \text{ A/cm}^2$

CHARGE DENSITY - ~~356~~ C/cm^2 28 C/cm^2

[Signature]

11/30/92

11/24/92

REPASSIVATION EXPT ALLOY 825 REPTIM 18

SAMPLE ALLOY 825 600 GRIT FINISH, RINSED IN ACETONE, DI WATER, CLEANED IN 10% HNO_3 FOR 60 MIN, AND RINSED IN DI WATER.

SOLUTION: SAME AS REPTIM 15 P148 95°C TEMP CHECKED WITH Hg THERMO # 1238004 PURGED WITH 99.999% N_2

POTENTIAL STAT: SAME AS PAGE 145 DATA SAVED AS C:\WB\DATA\REPTIM18.DAT AND AS B:\REPTIM18.

CATHODIC STRIP: -2.00 V SCE FOR 2 MIN

PIT INITIATION: 600 mV SCE FOR 30 MIN.

PIT GROWTH: 400 mV SCE FOR 6 HOURS.

REPASSIVATION: 190 mV SCE

SCE ELECTRODE: FISHER 13-620-51 SN 0169033

SAMPLE START WT: 11.54103

SOLUTION PH AT 25°C 8.132.

OPEN CIRCUIT POTENTIALS AT START. 11:20 AM

ALLOY 825 SAMPLE: -523 mV SCE

PT COUNTER ELECTRODE: -46 mV SCE

SAMPLES STOPPED: 11/25/92 TIME: 2:42 PM

RUNTIME: 97,000 SEC

REPASSIVATION TIME AT 190 mV SCE: 49,867 SEC

END PH (11/30/92): 8.940

CHARGE DENSITY = ~~103~~ C/cm^2 34 C/cm^2

[Signature]

11/30/92

12/15/92

CREVICE CORROSION SAMPLE 825-3

STARTED 12/10/92 ENDED 12/11/92

SPECIMEN: ALLOY 825 600 GRIT FINISH

SURFACE AREA 20.87 cm²

SOLUTION 0.5M NaCl, 85 ppm HCO₃⁻, 20 ppm SO₄²⁻
 10 ppm NO₃⁻, 2 ppm F⁻ AT ROOM TEMP
 ≈ 64°F PURGED WITH N₂

ELECTRICAL CONTACT MADE WITH 1.13 mm 825
 WIRE TO TOP OF SPECIMEN NO MICRO STOP
 USED TO COVER CONTACT.

SPECIMEN PLACED IN PLEXIGLASS CELL WITH
 2 PORTS FOR ELECTRODES. TIP PORT PH ELECTRODES
 (PH #1) AND Cl⁻ ELECTRODE (Cl #2) MOUTH
 PORT PH ELECTRODES (PH #2 PAGE 128 FOR
 INFORMATION ON ELECTRODE #'S). PH #3 PLACED
 IN BULK ELECTROLYTE. Ag/AgCl REF FISHER
 13-620-53 SN 8118182 IN BULK SOLUTION
 WITH BRIDGE TUBE. Pt COUNTER ELECTRODE IN
 SIDE COMPARTMENT OF GLASS VESSEL.
 CELL WAS PLACED IN SOLUTION THEN BOLTS
 WERE TORQUED TO 20 IN·OZ.

DATA SAVED IN 825-3.DAT ON COMPAQ 333+
 COMPUTER USING WB\CREVICE.WAB. SLOPE
 AND INTERCEPT OF PH AND Cl ELECTRODES:

MI506 #1 / MI402 #1 (PH #1)

$$mV = -58.76 (pH) + 406.33$$

$$pH = -17.018(V) + 6.915$$

MI 506 #2 / MI402 #2 (PH #2)

$$mV = -58.17 (pH) + 402.57$$

$$pH = -17.192(V) + 6.921$$

12/15/92

CREVICE CORROSION 825-3

MI 506 #3 / MI 402 #3 (PH #3)

$$mV = -57.77 (pH) + 398.17$$

$$pH = -17.311(V) + 6.892$$

MI 200 #2 / MI 403 #2 (Cl #2)

$$mV = -19.102 \cdot \ln(\text{ppm Cl}^-) + 232.293$$

$$\text{ppm Cl}^- = \exp[50.755(V) + 11.790]$$

DURING EXPT SLOW DECREASE IN PH WAS
 OBSERVED AT MOUTH AND TIP POSITIONS.
 PH AT MOUTH WAS LOWER THAN THAT AT TIP
 SEVERE CORROSION OF SPECIMEN AT POINT OF
 ELECTRICAL CONTACT WAS ALSO OBSERVED. AFTER
 REMOVING SAMPLE SOME CREVICE CORROSION
 BETWEEN TIP & MOUTH ELECTRODE PORTS ON
 TOP SIDE OF SPECIMEN NO CREVICE CORROSION
 WAS OBSERVED ON BOTTOM SIDE OF SPECIMEN

INITIAL CONDITIONS

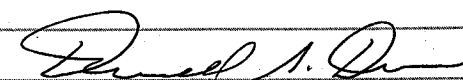
825-3 REST POTENTIAL = +0.083V vs Ag/AgCl

Pt REST POTENTIAL = +0.212V vs Ag/AgCl

PH TIP = 5.90 MOUTH = 4.90 BULK = 9.00

Cl ^{DO 712194} AT TIP = 13,000 ppm

POST EXPT CHECK OF ELECTRODES INDICATED PH ELECTRODES
 WERE OPERATING CORRECTLY BUT Cl ELECTRODE
 HAD BUBBLING IN OUTER BARRIL.



12/15/92

PH AND Cl⁻ ISC CALIBRATION DATA STORED IN
 8253CAL.DAT ON IOWE 025 DISK ② 4/28/94

12/16/92

REPASSIVATION EXPT ALLOY 825 REPTIM 19

SAMPLE: ALLOY 825 600 GRIT FINISH RINSED
IN ACETONE, DI WATER CLEANED IN 10% HNO_3
FOR 60 MIN, AND RINSED IN DI WATER.

SOLUTION: SAME AS REPTIM 15 95°C CHECKED
WITH N_2 THERMO 1238001 PURGED WITH
99.999% N_2

POTENTIOSTAT: SAME AS PAGE 145 DATA STORED
AS C:\WB\DATA\REPTIM19.DAT AND B: REPTIM19.DAT

CATHODIC STRIP -2.00 V FOR 2 MIN

PIT INITIATION 600 mV SCE FOR 30 MIN

PIT GROWTH 400 mV SCE FOR 6 hr

REPASSIVATION 175 mV SCE

SCE ELECTRODE FISHER 13-620-51 SN 0169033

SAMPLE START WEIGHT 11.47052

SOLUTION PH AT 25°C 8.054

OPEN CIRCUIT POTENTIALS AT START: 5:00 PM 12/15/92

ALLOY 825 SAMPLE -651 mV

PT COUNTER ELECTRODE NOT RECORDED

SAMPLE STOPPED 12/18/92 TIME 9:40 AM

RUNTIME 144,700 SEC

REPASSIVATION TIME AT 175 mV 173 SEC

END PH 9.196

CHARGE DENSITY 9 C/cm^2

Paul A. D.
12/18/92

REPASSIVATION EXPT ALLOY 825 REPTIM 20

SAMPLE ALLOY 825 600 GRIT FINISH RINSED
IN ACETONE, DI WATER, CLEANED IN 10% HNO_3
FOR 60 MIN AND RINSED IN DI WATER.

SOLUTION: SAME AS REPTIM 15 95°C CHECKED
WITH N_2 THERMO 1238004 PURGED WITH
99.999% N_2

POTENTIOSTAT: SAME AS PAGE 144 DATA STORED
AS C:\WB\DATA\REPTIM20.DAT AND B:\REPTIM20.DAT.

CATHODIC STRIP -2.00 V FOR 2 MIN

PIT INITIATION 600 mV SCE FOR 30 MIN

PIT GROWTH 400 mV SCE FOR 6 hr

REPASSIVATION 190 mV SCE

SCE ELECTRODE FISHER 13-620-51 SN 0165415

SAMPLE START WEIGHT 11.58026 g

SOLUTION PH AT 25°C 8.054

OPEN CIRCUIT POTENTIALS AT START 5:10 PM 12/15/92

ALLOY 825 SPECIMEN -735 mV

PT COUNTER ELECTRODE NOT RECORDED

SAMPLE STOPPED 12/21/92 TIME 2:00 PM

RUNTIME 506,000 SEC

REPASSIVATION TIME AT 190 mV 151,825

END PH 9.049

CHARGE DENSITY 54 C/cm^2

Paul A. D.
12/21/92

2/21/92 CR6VIC6 CORROSION EXPT 825-4 RUN 12/16/92

SPECIMEN ALLOY 825 600 GRIT FINISH
 SURFACE AREA 20.90 cm²
 START WEIGHT: 24.22537 END WEIGHT 24.223

SOLUTION 0.5M NaCl 85PPM HCO₃ 20PPM SO₄²⁻
 10PPM NO₃⁻ 2PPM F⁻ AT ROOM TEMP
 ≈ 64°F PURGED WITH N₂

ELECTRICAL CONTACT MADE WITH 2.35mm 825 ROD
 WITH GROUND FLATS TO SIDE OF
 825 SPECIMEN NO MICROSTOP USED
 TO COVER CONTACT

SPECIMEN PLACED IN PLEXIGLASS CELL, ELECTRODE
 PLACEMENT SAME AS 825-3 P152.

DATA SAVED AS 825-4.DAT ON COMPUAD
 333+ USING WORKBENCH CR6VIC6 SWBB

SLOPE AND INTERCEPT OF PN AND CI ELECTRODES

MISOG #1 / MI 402 #1 (PN #1)

MV = -57.75 (pH) + 397.30

pH = -17.31 (V) + 6.897

MISOG #2 / MI 402 #2 (PN #2)

MV = -57.58 (pH) + 394.70

pH = -17.368 (V) + 6.855

MISOG #3 / MI 402 #3 (PN #3)

MV = -57.521 (pH) + 396.847

pH = -17.383 (V) + 6.899

MI 200 #1 MI 403 #1

MV = -18.574 ln(ppm Cl⁻) + 223.952

MI 200 #2 MI 403 #2

MV = -21.127 ln(ppm Cl⁻) + 244.012

12/21/92

START CONDITIONS TIMER 73,700 SEC

E_c = +195 mV Ag/AgCl

E_{pt} = +0.424 V Ag/AgCl

REFERENCE ELECTRODE FISHER 13-600-53

PN TIP = 5.95 PN MOUNT = 4.45

PN BULK = 9.06 CI BULK = 15K-16K PPM

CI TIP = 15K-16K PPM

APPLIED POTENTIAL = 400 mV Ag/AgCl

TEST INTERRUPTED AFTER 2 HOURS DUE TO
 ELECTRODE INTERFERENCE PROBLEMS.

12/21/92

CR6VIC6 CORROSION EXPT 825-5 RUN 12/17/92

SPECIMEN ALLOY 825 600 GRIT FINISH

SURFACE AREA = 20.65 cm²

START WEIGHT 23.72003 g 12/17/92

END WEIGHT 23.70692 g 12/18/92

SOLUTION, ELECTRICAL CONTACT, PLEXIGLASS CELL

PN & CI ELECTRODE PLACEMENT SLOPES AND INTERCEPT
 SAME AS 825-4 P156.

DATA STORED AS 825-5 ON COMPUAD 333+

APPLIED POTENTIAL +400 mV Ag/AgCl

REFERENCE ELECTRODE = FISHER 13-620-53

UPON APPLYING POTENTIAL TO SPECIMEN INTERFERENCE
 WITH PN AND CI⁻ ELECTRODES OCCURS.
 PROBLEM TRACED TO WORKBENCH DATA CARD

PN AND CI ISG CALIBRATION DATA STORED AS 8254CAL.DAT
 ON IWRG-025 DISK

4/28/94

12/21/92

12/21/92

CREVICE CORROSION EXPT 825-6 12/21/92

SPECIMEN AZLOY 825 600 GRIT FINISH
SURFACE AREA

START WEIGHT = 23.42206 g 12/21/92
END WEIGHT : NOT RECORDED

SOLUTION 0.5M NaCl (29.22549 g NaCl LOT 922649A)
85 PPM HCO_3^- (0.11880 g NaHCO_3 LOT 897789)
20 PPM SO_4^{2-} (20 ml / 1000 ppm STOCK SOLUTION)
USING 1.4787 g Na_2SO_4 ~~1000~~ 1000 ml LOT 901213
10 PPM NO_3^- (10 ml / 1000 ppm STOCK SOLUTION)
USING 1.3706 g NaNO_3 IN 1000 ml LOT 897183
2 PPM F^- (2 ml / 1000 ppm STOCK SOLUTION)
USING 2.2100 g NaF LOT 896405 IN 1000 ml)

ELECTRICAL CONTACT MADE WITH 2.35 mm 825 ROD
WITH GROUND FLATS SPOT WELDED TO SINGS
OF SPECIMEN NO MICROSTOP USED TO COVER CONTACT
SOLUTION PURGED WITH N_2

SPECIMEN PLACED IN PLEXIGLASS CELL MI 506/MI 402
#1 (PN #1) PLACED IN TIP LOCATION
MI 506 #2 / MI 402 #2 (PN #2) PLACED IN MOUTH
MI 506 #3 / MI 402 #3 (PN #3) PLACED IN BULK
MI 200 #1 / MI 403 #1 (CI #1) PLACED IN TIP
MI 200 #2 / MI 403 #2 (CI #2) PLACED IN BULK

PN #1 CONNECTED TO ORION 720A SN005885 CN1

PN #2 CONNECTED TO ORION 720A SN003368 CH #1

PN #3 CONNECTED TO ORION 720A SN003368 CN2

CI #1 CONNECTED TO ORION EA920 SN5001/A CN1

CI #2 CONNECTED TO ORION EA920 SN5001/A CN2

PN #1 RECORDED BY RED PEN ABB SN 0515265 CHART

RECORDER PN #2 TO BLUE PEN

CI #1 RECORDED BY RED PEN ABB SG 120 SN

0049616 CURRENT TO BLUE PEN

CURRENT MEASURED WITH KEITNEY 485 PICOAMMETER

12/21/92

CREVICE CORROSION EXPT 825-6 12/21/92

POTENTIAL MEASURED WITH KEITNEY 614 ELECTROMETER
HIGH IMP LEAD TO REFERENCE ELECTRODE (FISHER)
BLACK LEAD TO SPECIMEN.

CREVICE POTENTIAL MEASURED WITH ORION 720A
SN 005885 CHANNEL #2 USING MI 402 #44671

POTENTIAL APPLIED WITH ESC 410 IA # 9105557
AND ESC 420A IA # 9029495
A₁ AgCl REF ELECTRODE FISHER 13-620-53
SN 8118182

SLOPE AND INTERCEPT OF PH AND CI ELECTRODES

MI 506 #1 / MI 402 #1 (PN #1) AT TIP

$mV = -57.733(PN) + 400.833$

$PN = -0.01732(mV) + 6.943$

MI 506 #2 / MI 402 #2 (PN #2) AT MOUTH

$mV = -57.578(PN) + 394.647$

$PN = -0.01737(mV) + 6.854$

MI 506 #3 / MI 402 #3 (PN #3) IN BULK

$mV = -57.753(PN) + 398.293$

$PN = -0.01732(mV) + 6.896$

7/27/94

MI 200 #1 / MI 403 #1 ~~CI #1~~ (CI #1) AT TIP.

$mV = -20.469 \cdot h \text{ PPM } \text{Cl}^- + 242.384$

$\text{PPM } \text{Cl}^- = \text{EXP} [-0.04885 \cdot mV + 11.8414]$

MI 200 #2 / MI 403 #2 (CI #2) AT BULK.

$mV = -18.3023 \cdot h (\text{PPM } \text{Cl}^-) + 226.473$

$\text{PPM } \text{Cl}^- = \text{EXP} [-0.05464 \cdot mV + 12.3740]$

PH AND Cl^- ISE CALIBRATION DATA SAVED AS

8256 CAL.DAT ON IWEPE-025 DISK *David D* 4/28/94

David D

12/21/92

12/31/92

REPASSIVATION EXPT ALLOY 825 REPTIM 21

SAMPLE: ALLOY 825 600 GRIT FINISH RINSED
IN ACETONE, DI WATER, CLEANED
IN 10% HNO_3 FOR 60 MIN AND RINSED
IN DI WATER.

SOLUTION: 1000 PPM Cl^- 85 PPM HCO_3^- 20 PPM SO_4^{2-}
10 PPM NO_3^- 2 PPM F^- AS Na SALTS
3.29554 g NaCl LOT 922649A
0.11572 g NaHCO_3 LOT 897789
40 ml / 1000 PPM SO_4^{2-} (1.4787 g Na_2SO_4 LOT 901203)
20 ml / 1000 PPM NO_3^- (1.3767 g NaNO_3 LOT 897183)
4 ml / 1000 PPM F^- (2.2100 g NaF LOT 896405)
+ DI H_2O TO A VOLUME OF 2000 ml
SOLUTION PURGED W/ N_2 AND TEMP
CHECKED WITH Hg THERMO #1238004
TEMP AT 95°C

POTENTIOSTAT ESC 440-1 CALIBRATION DUE
13 JAN 93 CHANNEL #2
DATA SAVED AS WA\DATA\REPTIM 21.DAT
AND AS B:\REPTIM 21.DAT.

CATHODIC STRIP: -2.00V FOR 2 MIN
PIT INITIATION: 600 mV SCE FOR 30 MIN 10:04 AM
PIT GROWTH: 400 mV SCE FOR 6 HOURS
REPASSIVATION: 160 mV SCE
SCE ELECTRODE FISHER 13-620-51 SNO169033
SAMPLE START WEIGHT 11.62603 g
SOLUTION PH AT 25°C : 8.144
OPEN CIRCUIT POTENTIALS AT START
ALLOY 825 SAMPLE: -592 mV -922 AFTER STRIP
PE COUNTER ELECTRODE: -341 mV

SAMPLE STOPPED 1/1/93 11:12 AM

RUNTIME 89249 SEC

REPASSIVATION TIME: 654.3 SEC TO $1 \times 10^{-6} \text{ A/cm}^2$

END PH 9.256 \ 199 SEC TO $5 \times 10^{-5} \text{ A/cm}^2$

CHARGE DENSITY 20 C/cm² *David A. Q* 1/1/93

12/31/92

REPASSIVATION EXPT ALLOY 825 REPTIM 22

SAMPLE: ALLOY 825 600 GRIT FINISH RINSED
IN ACETONE DI WATER CLEANED IN 10% HNO_3
FOR 60 MIN AND RINSED IN DI WATER.

SOLUTION SAME AS REPTIM 21 P 160
SOLUTION AT 95°C PURGED WITH N_2
TEMP CHECKED WITH Hg THERMO 1238001
POTENTIOSTAT ESC 440-1 CALIBRATION DUE
13 JAN 93 CHANNEL #3 DATA
SAVED AS WA\DATA\REPTIM 22.DAT AND
AS: B:\REPTIM 22.DAT

CATHODIC STRIP: -2.00V FOR 2 MIN
PIT INITIATION: 600 mV SCE FOR 30 MIN 10:22 AM
PIT GROWTH: 400 mV SCE FOR 6 HOURS
REPASSIVATION: 169 mV SCE ~~FOR~~ 7/27/94
SCE ELECTRODE FISHER 13-620-51 SNO165043
SAMPLE START WEIGHT: 11.57849 g
SOLUTION PH AT 25°C : 8.144
OPEN CIRCUIT POTENTIALS AT START
ALLOY 825 SAMPLE -524 mV -903 AFTER STRIP
PE COUNTER ELECTRODE: -345 mV

SAMPLE STOPPED 1/1/93 11:12 AM

RUNTIME 88921 SEC

REPASSIVATION TIME 45202 TO $1 \times 10^{-6} \text{ A}$ 25178 TO $5 \times 10^{-5} \text{ A}$

END PH: 9.504

CHARGE DENSITY 23 C/cm²

David A. Q
1/1/93

1/10/93

REPASSIVATION POTENTIAL ALLOY 825 RP825P1

SPECIMEN: ALLOY 825 1.915" LONG 0.249" DIA
 600 GRIT FINISH CLEANED IN ACETONE,
 10% HNO₃ FOR 60 MIN, RINSED IN DI
 WATER HH 4371 FC
 START WT: 11.65891 g
 END WT: 11.47200 g 1/22/93 DD

SOLUTION: 1000 PPM Cl⁻, 85 PPM HCO₃⁻, 20 PPM SO₄⁼
 10 PPM NO₃⁻, 2 PPM F⁻ MADE AS FOLLOWS
 - 3.29963 g NaCl LOT 922649A
 - 0.23303 g NaHCO₃ LOT 897789
 - 40 ml / 1000 PPM SO₄⁼ STOCK SOLUTION MADE USING
 1.4787 g Na₂SO₄ / 1000 ml DI H₂O LOT# 901213
 - 20 ml / 1000 PPM NO₃⁻ STOCK SOLUTION USING
 1.3767 g NaNO₃ LOT 897183 / 1000 ml w/ DI H₂O
 - 4 ml NaF 1000 PPM LOT 896405 MADE USING
 2.2100 g NaF + H₂O TO 1000 ml
 + DI H₂O TO 2000 ml

SOLUTION AT 95°C TEMP CHECKED WITH
 Hg THERMO 1238004 PURGED WITH N₂

POTENTIOSTAT ESC 440-1 CHANNEL #3 PROGRAM
 WORKBENCH WB\WB\REPASS2B.DAT IN RP825P1.DAT

ELECTRODE FISHER SCE 1362051 SN 0165415

CATHODE STRIP -2.00V SCE FOR 2 min
 PIT INITIATION +0.600V SCE FOR 30 min
 PIT GROWTH +0.400V SCE FOR 12 HRS.

SOLUTION PH AT 25°C 7.815

REST POTENTIALS AT START.

E_{corr} = -689 mV SCE KEITNEY 614 X -1
 E_{pt} = -225 mV SCE " " "

SAMPLE STARTED 1:40 PM 1/10/93
 SAMPLE STOPPED 10:00 AM 1/11/93
 RUNTIME 73,400 SEC
 REPASSIVATION POTENTIAL 105 mV
 CHARGE DENSITY 96 COUL / CM²
 END PH: 9.073

[Signature] 1/10/93

1/10/92

REPASSIVATION POTENTIAL ALLOY 825 RP825P2

SPECIMEN ALLOY 825 HH 4371 FC 1.914" LONG 0.249" DIA
 600 GRIT FINISH CLEANED IN ACETONE
 10% HNO₃ FOR 60 MIN RINSED IN DI
 H₂O
 START WT: 11.63461 g
 END WT: 11.63326 g 1/22/93 DD

SOLUTION: 100 PPM Cl⁻, 85 PPM HCO₃⁻, 20 PPM SO₄⁼
 10 PPM NO₃⁻, 2 PPM F⁻ MADE AS FOLLOWS.
 100 ml STOCK SOLUTION # 1000 Cl⁻ - 1/93 (1000 PPM Cl⁻)
 0.11614 g NaHCO₃ LOT 897789
 20 ml STOCK SOLUTION # SO₄ - 1/93 (1000 PPM SO₄⁼)
 10 ml STOCK SOLUTION # NO₃ - 1/93 (1000 PPM NO₃⁻)
 2 ml STOCK SOLUTION # F⁻ - 1/93 (1000 PPM F⁻)
 (STOCK SOLUTION DETAILS ON PAGE 164)
 IN 1000 ml FLASK + H₂O TO 1000 ml
 SOLUTION AT 95°C Hg THERMO
 PURGED WITH N₂

POTENTIOSTAT ESC 440-1 CHANNEL #2 WORKBENCH
 WB\WB\REPASS2D.DAT IN RP825P2.DAT

ELECTRODE FISHER SCE 1362051 SN 0169033

CATHODE STRIP -2.00V SCE FOR 2.00 min
 PIT INITIATION +0.600V SCE FOR 30 min
 PIT GROWTH +0.400V SCE FOR 12 HOURS.

SOLUTION PH AT 25°C 8.150

REST POTENTIALS AT START

E_{corr} -575 mV SCE KEITNEY 614 X -1
 E_{pt} -366 mV SCE " " "

SPECIMEN STARTED 1/10/93 1:45 PM
 SAMPLE STOPPED 10: AM 1/11/93
 RUNTIME 73,200 SEC
 REPASSIVATION POTENTIAL: NA PITTING NEVER STARTED
 CHARGE DENSITY 8.3 x 10⁻² COUL / CM²
 END PH 9.964

[Signature] 1/10/93

1/11/93

STOCK SOLUTIONS

1/93

SO₄-1/93

1000 ppm SO₄²⁻ AS Na₂SO₄
 1.48040 g Na₂SO₄ FISHER LOT # 901213
 + DI H₂O TO 1000 ml

NO₃-1/93

1000 ppm NO₃⁻ AS NaNO₃
 1.37258 g NaNO₃ FISHER LOT # 897183
 + DI H₂O TO 1000 ml

F-1/93

1000 ppm F⁻ AS NaF
 2.20977 g NaF FISHER LOT # 896405
 + DI H₂O TO 1000 ml

1M Cl-1/93

1MOLAR NaCl

58.44 g NaCl + H₂O TO 1000 ml
 FISHER LOT 922649A

1000 Cl-1/93

3.29721 g NaCl + H₂O TO 2000 ml
 FISHER LOT 922649A

100 Cl-1/93

100 ml 1000 Cl-1/93 + 900 ml H₂O

10 Cl-1/93

10 ml 1000 Cl-1/93 + 990 ml H₂O

David A. D.
 1/11/93

1/13/93

REPASSIVATION POTENTIAL ALLOY 825 RP825P3

*SPECIMEN ALLOY 825 600 GRIT FINISH

L = 1.915" d = 0.250"

START WT: 11.63209g HEAT # NH 4371 FC

END WT: ~~11.58956g~~ 1/22/93 Φ 8.91711gWET SURFACE AREA = 8.0 cm²

SOLUTION 10000 ppm Cl⁻ 85 ppm NaCO₃ 20 ppm SO₄²⁻
 10 ppm NO₃⁻ 2 ppm F⁻ MADE AS FOLLOWS

16.48255 g NaCl LOT 922649A

0.11648 g NaHCO₃ LOT 89778920 ml SS# SO₄-1/93 (1000 ppm SO₄²⁻ AS Na₂SO₄)10 ml SS# NO₃-1/93 (1000 ppm NO₃⁻ AS NaNO₃)2 ml SS# F-1/93 (1000 ppm F⁻ AS NaF)

STOCK SOLUTION DETAILS ON PAGE 164

+ DI H₂O TO 1000 ml T AT 95°C CNGCKEDWITH N₂ TNGRMD # 1238001 PURGEDWITH N₂ 99.999%

START PH = 7.960

END PH = 9.431

POTENTIOSTAT ESC 440-1 CHANNEL #3 DATA

SAVED AS WB\DATA\RP825P3.DAT USING

WB\WB\REPASS2B PROGRAM.

ELECTRODE: FISHER SCE 13-620-51 SN 0169033

E_{CORR} = -608 mV SCE KESTNLEY 614 SN 467374E_{PT} = -105 mV SCE " " " "

CATHODIC STRIP: -2.0V FOR 2 min

PIT INITIATION: 600 mV SCE FOR 30 min

PIT GROWTH 400 mV SCE FOR 12 HOURS

SPECIMEN STARTED 1/13/93 2:45 PM

SPECIMEN STOPPED 1/14/93 9:34 AM

RUNTIME 67500 SEC

REPASSIVATION POTENTIAL +5.4 mV KESTNLEY 614

CHARGE DENSITY 1.4 x 10³ COUL/cm²

* SPECIMEN CLEANED IN DI WATER, ACETONE, 10%
 HNO₃ FOR 60 MIN, RINSED IN DI WATER

David A. D. 1/13/93

1/13/93

R6 PASSIVATION POTENTIAL RP825 P4 ALLOY 825

SPECIMEN ALLOY 825 600 GRIT FINISH BULK COMPOSITION

 $l = 1.916''$ $d = 0.249''$ WET SURFACE AREA = 8.0 cm^2

START WT - 11.63803g HEAT # HH 4371FC

END WT: ~~8.9177g~~ DD 1/22/93 11.58956gSPECIMEN CLEANED IN DI H_2O , ACETONE,10% HNO_3 FOR 60 min RINSED IN DI H_2O SOLUTION 500 PPM Cl^- 85 PPM NO_3^- 20 PPM SO_4^{2-} 10 PPM NO_3^- 2 PPM F^- MARK AS FOLLOWS0.82285g NaCl LOT 922649A0.11534g NaNO_3 LOT 89778920 ml 55% SO_4^{2-} 1/93 (1000 PPM SO_4^{2-} AS Na_2SO_4)10 ml 55% NO_3^- 1/93 (1000 PPM NO_3^- AS NaNO_3)2 ml 55% F^- 1/93 (1000 PPM F^- AS NaF)+ DI H_2O TO 1000 ml 55% ON PAGE 164T = 95°C CHECKED W/ H_2 THERMO # 1238064PURGED W/ N_2 99.999%

START PH = 8.186

END PH = 9.470

POTENTIAL STAT ESC 440-1 CHANNEL 2 DATA

SAVED ON WORKBENCH WB\DATA\RP825P4.DAT

USING WB\WB\RPASS2B

ELECTRODE SCE FISHER 13-620-51 SNO165405

 $E_{\text{CORR}} = -456 \text{ mV SCE}$ KEITNELY 614 SN 467374 $E_{\text{PT}} = 1.1 \text{ mV SCE}$ " " " "

CATHODIC STRIP: -200V FOR 2m

PIT INITIATION 600mV SCE FOR 30min

PIT GROWTH 400mV SCE FOR 12 HOURS

SPECIMEN STARTED 1/13/93 2:50 pm

SPECIMEN STOPPED 1/14/93 9:35 am

RUNTIME 67400 SEC

REPASSIVATION POTENTIAL 147.7 mV KEITNELY 614

CHARGE DENSITY $2.6 \times 10^{-1} \text{ coul/cm}^2$

D. A. D. 1/14/93

1/14/93

CREVICE CORROSION EXPT 825-7 1/11/93-1/14/93

SPECIMEN ALLOY 825 600 GRIT FINISH CLEANED

IN ACETONE RINSED W/ DI WATER.

SURFACE AREA = 20.69 cm^2

START WT: 24.06859g

END WT: 24.04532g

HEAT # HH 4371FC

SOLUTION: 0.5M NaCl 85 PPM NO_3^- 20 PPM SO_4^{2-} 10 PPM NO_3^- 2 PPM F^- MARK AS FOLLOWS.29.22437g NaCl LOT 922649A0.11682g NaNO_3 LOT 89778920 ml 1000 PPM SO_4^{2-} STOCK SOLUTION # SO_4 -1/9310 ml 1000 PPM NO_3^- STOCK SOLUTION # NO_3 -1/932 ml 1000 PPM F^- STOCK SOLUTION # F^- 1/93

+ DI WATER TO 1000 ml (55% PAGE 164)

T AT ROOM TEMP $\approx 20^\circ\text{C}$ PURGED W/ N_2

ELECTRICAL CONTACT SAME AS 825-6 P158

SPECIMEN PLACED IN PLEXIGLASS CELL WITH

2 ELECTRODE PORTS, 5 ELECTRODES IN EACH PORT.

PORT #1 AT TIA LOCATION:

MI 506 #1 / MI 402 #1 (PN #1) TO ORION 720A SN 5885 CH #1

MI 200 #1 / MI 403 #1 (CI #1) TO ORION EA920 SN 5001A CH #1

MI 402 SN 44629 TO ORION EA940 SN 2330 CH #1

PORT #2 AT MOUTH LOCATION

MI 506 #2 / MI 402 #2 (PN #2) TO ORION 720A SN 003368 CH #1

MI 200 #2 / MI 403 #2 (CI #2) TO ORION EA920 SN 5001A CH #2

MI 402 SN 44671 TO ORION EA940 SN 2330 CH #2

BULK ELECTRODES

MI 200 (RIGID Si ELECTRODE) W/ MI 402 SN 44628TO ORION ~~EA920~~ 720A SN 5885 CH #2

MI 506 #3 / MI 402 #3 (PN #3) TO ORION 720A SN 003368

CHANNEL # 2 ELECTRODE IDENTIFICATION ON

PAGE 128.

CURRENT MEASURED WITH KEITNELY 485 PICO AMMETER

SN 13096 RECORDED W/ ABB CART RECORDER

SE 120 SN 0049616 BLUE PEN

1/14/93

Cl TIP RECORDED WITH ABB SG 120 SN 0049616
WITH RED PEN.

PH #1 RECORDED WITH ABB SG 120 SN 0515265
WITH RED PEN BLUE PEN OF SN 0515265
RECORDED PH #2

POTENTIAL (APPLIED) MEASURED WITH KEITHLEY 617
SN 15481 CAL DUE 12/23/93

CREVICE POTENTIAL MONITORED W/ ORION EA 940
SN 2380 AND REF ELECTRODES (MICROELECTRODES)

POTENTIAL APPLIED WITH ESC #410 ID# 9105557
AND ESC 420A ID# 9029495

Ag/AgCl REF ELECTRODE FISHER 13-620-53
SN 8118182

SLOPE AND INTERCEPT OF pH AND Cl⁻ ELECTRODES

MI 506 #1 / MI 402 #1 (PH #1) AT TIP

$$mV = -57.71 (pH) + 403.88$$

$$pH = -0.01733 (mV) + 6.993$$

MI 506 #2 / MI 402 #2 (PH #2) AT MOUTH

$$mV = -58.00 (pH) + 401.95$$

$$pH = -0.01724 (mV) + 6.930$$

MI 506 #3 / MI 402 #3 (PH #3) AT BULK

$$mV = -57.50 (pH) + 398.76$$

$$pH = -0.01739 (mV) + 6.935$$

Cl MI 200 #1 / MI 403 #1 (Cl #1) AT TIP

$$mV = -21.388 \cdot \ln (ppm Cl^-) + 236.806$$

$$ppm Cl^- = \frac{EXP [0.04676 \cdot mV + 11.072]}{EXP [0.04676 \cdot mV + 11.072]}$$

MI 200 #2 / MI 403 #2 (Cl #2) AT MOUTH

$$mV = -21.552 \cdot \ln (ppm Cl^-) + 251.659$$

$$ppm Cl^- = \frac{EXP [-0.04640 (mV) + 11.677]}{EXP [-0.04640 (mV) + 11.677]}$$

MI 200 RIGID Cl ELECTRODE SN 42978 & MI 402 # 44678

$$mV = -21.185 \cdot \ln (ppm Cl^-) + 257.92$$

$$ppm Cl^- = \frac{EXP [0.04720 \cdot mV + 12.174]}{EXP [0.04720 (mV) + 12.174]}$$

1/14/93

SOLUTION START pH = 7.838

SOLUTION END pH = 8.067

APPLIED POTENTIAL + 300 mV Ag/AgCl
CURRENT : 1.1 mA (HIGH) - 0.30 mA (LOW)

SPECIMEN HAD CREVICE CORROSION BETWEEN
MOUTH AND PORT CLOSEST TO MOUTH ON
TOP SIDE SOME CREVICE CORROSION BETWEEN
MOUTH AND TIP ELECTRODE PORTS. RUN
STOPPED DUE TO PROBLEM WITH TIP pH
ELECTRODE AND NOISY Cl⁻ ELECTRODE SIGNALS.
CAUSED BY BUBBLE IN Cl⁻ REFERENCE ELECTRODES
pH AT MOUTH DROPPED TO \approx pH 4.0
pH AT TIP TO \approx 6.5-7.0
Cl⁻ MEASUREMENTS NOT RELIABLE DUE TO REFERENCE
PROBLEMS.

BULK pH DID DECREASE SOMEWHAT
BULK Cl⁻ MOSTLY CONSTANT.

Paul A. De
1/14/93

pH AND Cl⁻ ISE CALIBRATION DATA SAVED
AS 8257.CAL.DAT ON TUBE-025 DISK