Continuation from Notebook No. 744

Projed # 20.06002.01.322

CONTROLLED COPY NO. 815

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Initial Scientific Notebook Entry for Corrosion of Dissimilar Metal Crevices as a Function of Solution, Torque, Area, and Dissimilar Metals

Title: Corrosion of Dissimilar Metal Crevices as a Function of Solution, Torque, Area, and Dissimilar Metals

Tests Performed by: Xihua He, Div. 20; Brian Derby, Div. 18

Objectives: Determine the corrosion potential, crevice corrosion repassivation potential, and crevice corrosion penetration depths of dissimilar metal crevices as a function of solution, torque, area, and dissimilar metals

Proposed approach or procedure for achieving the objectives: Tests are conducted in a glass test cell using a multiple crevice assembly consisting of a number of grooves and plateaus in accordance with ASTM G78. In this assembly, the serrated crevice former machined from base Alloy 22 or Titanium Grade-7 contacts with the crevice specimen to form an artificial crevice with 24 crevice sites. Then immerse the crevice assembly in solution to monitor the corrosion potential and perform cyclic polarization to obtain crevice corrosion repassivation potential (see reference 1 below). Penetration depths are obtained by galvanically coupling the crevice assembly to larger cathodes to initiate localized corrosion. The crevice specimens tested are mill-annealed base Alloy 22, welded + solution annealed Alloy 22, 316L stainless steel, zirconium. By varying the surface area of the crevice former, torque between dissimilar metal contact, and combination of dissimilar metal contacts, the corrosion of dissimilar metal crevices can be studied as a function of torque, area, and dissimilar metals.

Equipment: Electrochemical test cell with multiple crevice assembly, Solartron 1287 and 1480 multi-channel potentiostats, temperature controller, thermometer, pH mater, balance, torque screwdriver, caliper, microscope.
Calibration and due dates are provided in data sheet for each test.

Materials: Mill-annealed base Alloy 22, welded Alloy 22, 316L stainless steel, titanium Grade-7, zirconium. Material heats to be added prior to testing

Specimen Specifications: crevice specimens CNWRA drawing #20.06002.01.322.002 and crevice former CNWRA drawing #20.06002.01.322.006, #20.06002.01.322.008.A, #20.06002.01.322.011

Measurement Parameters: Temperature, open circuit potential, potential and current of specimen during polarization, penetration depth per ASTM standard G46.

Required Level of Accuracy: Temperature ±2°C, Potentials ±1 mV, Current ±1 microamp.

Uncertainty and Sources of Error: Open circuit potential changes with time. Current measurement error can occur for localized corrosion processes because the actively corroding area is not the same as the surface area of the test specimen.

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1. D.S. Dunn, Y.-M. Pan, K. Chiang, L. Yang, G.A. Cragnolino, X. He, "The localized corrosion resistance and mechanical properties of alloy 22 waste package outer containers", *Journal of Metals*, 57 (2005) 49-55.

Xihua He 8/1/06

Galvanic Corrosion Test

Objective: SEE PAGE #1

SPECIMENS: Crevice Specimen drawing # 20.06002.01.322.002 polished to 600 Grit finish then cleaned in acetone

C-22 HT# 2277-3-3266 Crevice Specimen

Ti 7 Plate HT# CN2775

Ti 7 Crevice Washers & Hardware HT# CN2775

Torque Screwdriver: True Craft 75 In-lbs — **SN:** 694000691 — **Cal:** 7/25/06 — **Due:** 7/25/07

Initial Weight: 23.18155 g
Final Weight: 23.18147 g
Model: Sartorius Genius — **SN:** 12809099 — **Cal:** 5/9/06 — **Due** 11/9/06

SOLUTION: 4 M $MgCl_2 \cdot 6H_2O$
1626.53 g $MgCl_2 \cdot 6H_2O$ Lot# 050439
+ DI To 2000 mls

Reagents measured with **Model:** OHAUS — **SN:** 2883
Cal: ~~4/5/06~~ Date Rec'd 7/5/06 — **Due:** 1/5/07

Initial pH: 3.02
Final pH: Not Taken
Model: Orion EA 940 — **SN:** 2330
Cal: 7/6/06 — **Due:** 7/6/07
pH Probe: #13-620-296 — **SN:** 5003095

TEST TEMPERATURE: 95°C **Thermometer:** Fisher — **SN:** H98-182
Cal: 5/11/06 — **Due:** 5/11/07

Reference Electrode: Fisher SCE **# 13-620-52** — **SN:** 3329075

GAS: Zero Air

(CREVICE)
Ecorr: ~372 **mV vs SCE**

(PLATE)
Ecorr: -220 **mV vs SCE**
Model: Keithley 614 — **SN#:** 467374
Cal: 1/12/06 — **Due:** 1/12/07

Potentiostat:Solartron 1480 **sn#** 00240551 cal: 5/3/06 Due: 11/3/06

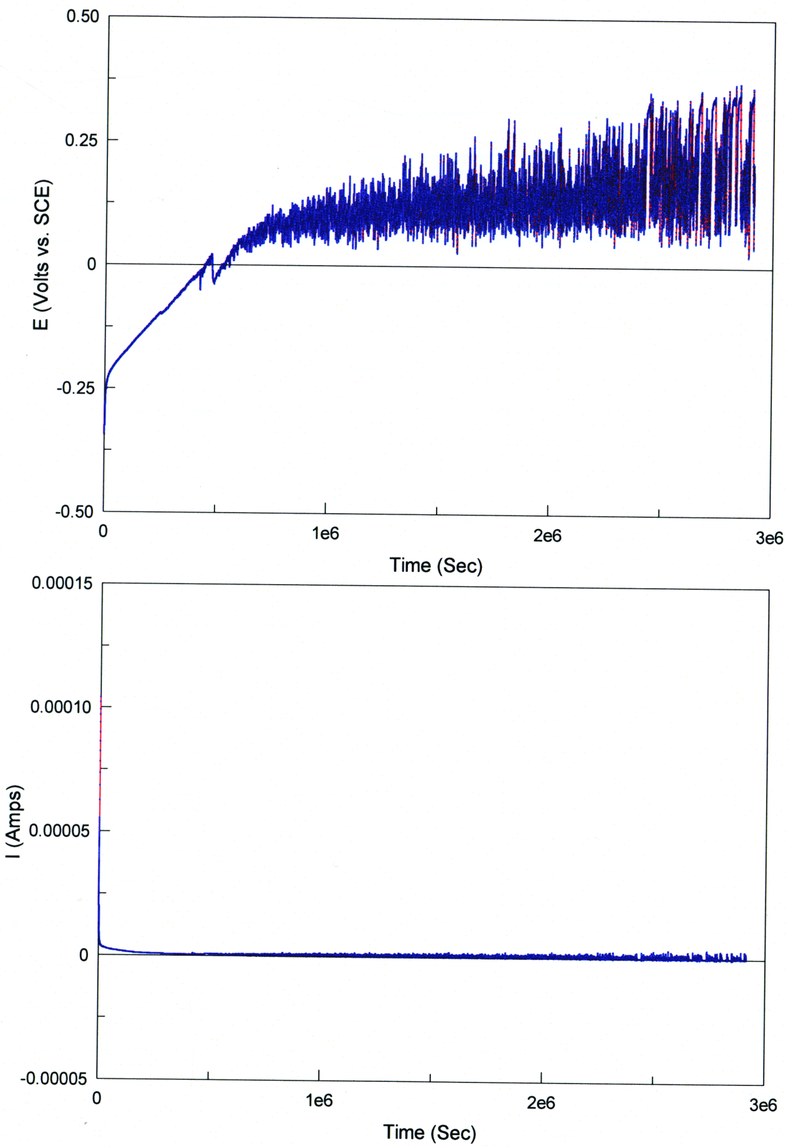
TEST ID: MA22Ti7Ti7g
DATA FILES: C22Ti7I0728a - Unich1, C22Ti7E0728a - Unich2, 0728, 0804, 0811, 0818, 0825

Specimen Examination: No Corrosion Very Mild staining

* Note: Repolished Specimen And Restarted Test.

Test Ended 8/31/06

8/2/06



No indication of crevice corrosion initiation. Xihua He 9/12/06

X.H 9/12/06

Galvanic Corrosion Test

Objective: SEE PAGE #1

SPECIMENS: Crevice Specimen drawing # 20.06002.01.322.002 polished to 600 Grit finish then cleaned in acetone

C-22 HT#2277-3-3266 Specimen

C-22 HT#2277-3-3266 Plate

C-22 HT#2277-3-3266 Crevice Specimen & Hardware (regular size)

Torque Screwdriver: True Craft 75 In.lbs **Cal:** 7/25/06 **SN:** 69400691 **Due:** 7/25/07

Initial Weight: 23.32787g
Final Weight: ~~23.34615g~~ 23.31782g 1/18/07

Model: Sartorius Genius **Cal:** 5/9/06 **SN:** 12809099 **Due** 11/9/06

SOLUTION: 4.0 m $MgCl_2 \cdot 6H_2O$
1626.67g $MgCl_2 \cdot 6H_2O$ lot # 054378
+ DI To 2000 mls

Reagents measured with **Model:** OHAUS **Cal:** 7/5/06 **SN:** 2883 **Due:** 1/5/07

Initial pH: 3.13
Final pH: 2.42

Model: Orion EA 940 **Cal:** 7/6/06 **pH Probe:** #13-620-296 **SN:**2330 **Due:** 7/6/07 **SN:** 5003085

TEST TEMPERATURE: 95°C **Thermometer:** Fisher **Cal:** 9/9/05 **SN:** C98-132 **Due:** 9/8/06

Reference Electrode: Fisher SCE **# 13-620-52** **SN:** 9252105

GAS: Zero Air

(CREVICE)
Ecorr: -199 **mV vs SCE**

(PLATE)
Ecorr: -145 **mV vs SCE**

Model: Keithley 614 **Cal:** 1/12/06 **SN#:** 467374 **Due:** 1/12/07

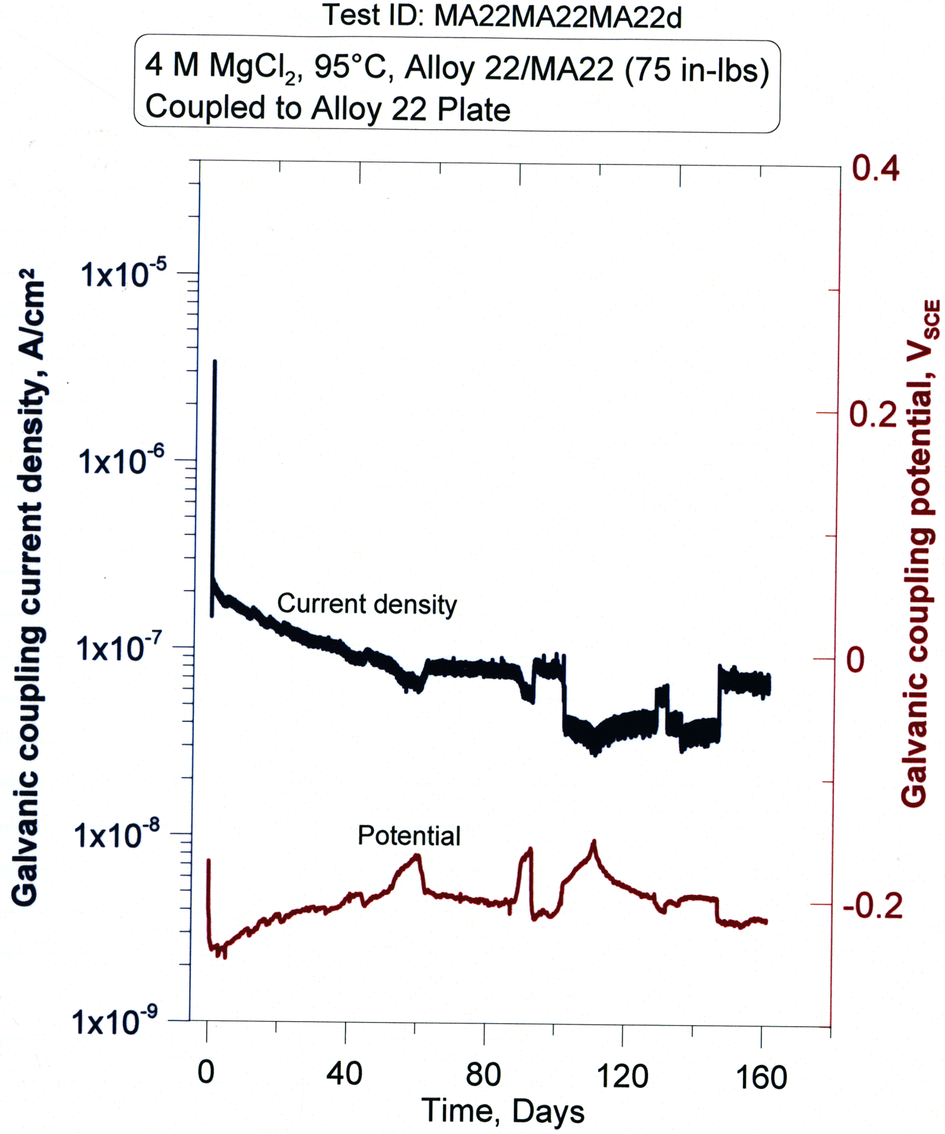
Potentiostat:Solartron 1480 **sn#** 00240551 cal: 5/3/06 Due: 11/3/06

TEST ID: MA22MA22MA22d
DATA FILES: C22C22I0728a-unICh7, C22C22E0728a-unICh8, 0728, 0804, 0811, 0818, 0825, 0901, 0908, 0915, 0922, 0929, 1006, 1013, 1020, 1027, 1103, 1110, 1117, 1124, 1201, 1208, 1215, 1222, 1229,

Specimen Examination: Crevice Corrosion on 23/24 feet of C-22 Crevice washers. Gold tint staining on specimen - Also Corrosion on 23/24 feet of the Crevice Washers they Also Have Gold tint staining C-22 Plate Ok Mild staining XH 1/11/07

Test duration: 7/28/06 – 1/18/07 161 days

8/2/06



penetration depth: 127 μm, 124 μm, 86 μm (3 deepest sites. others are too shallow to measure)
measured with Olympus metallurgical microscope BMX3
Cal: 12/21/06 Due: 6/21/07

XHuerta 1/11/07

XHuerta 1/18/07

Galvanic Corrosion Test

Objective: SEE PAGE #1

SPECIMENS: Crevice Specimen drawing # 20.06002.01.322.002 polished to 600 Grit finish then cleaned in acetone

C-22 HT# 2277-3-3266 Crevice Specimens — Ti 7 Plate HT# CN 2775 — Ti 7 Crevice Washers & Hardware HT# CN 2775

Torque Screwdriver: Tekcraft 75 In-lbs — Cal: 7/25/06 — SN: 694009691 — Due: 7/25/07

Initial Weight: 23.14442g
Final Weight: 23.14403g
Model: Sartorius Genius — Cal: 5/5/06 — SN: 12809099 — Due 11/9/06

SOLUTION: Same Solution As ON pg #2
4 M $MgCl_2 \cdot 6H_2O$
1626.53g $MgCl_2 \cdot 6H_2O$ Lot # 050439
+DI to 2000mL

Reagents measured with — Model: OHAUS — Cal: 7/5/06 — SN: 2883 — Due: 1/5/07

Initial pH: 3.02 As Previous
Final pH: 3.06
Model: Orion EA 940 — Cal: 7/6/06 — SN:2330 — Due: 7/6/07
pH Probe: #13-620-296 — SN: 5003095

TEST TEMPERATURE: 95°C — Thermometer: Fisher — Cal: 5/11/06 — SN: H98-182 — Due: 5/11/07

Reference Electrode: Fisher SCE — # 13-620-52 — SN: 3329075

GAS: Zero Air

(CREVICE)
Ecorr: -250 mV vs SCE

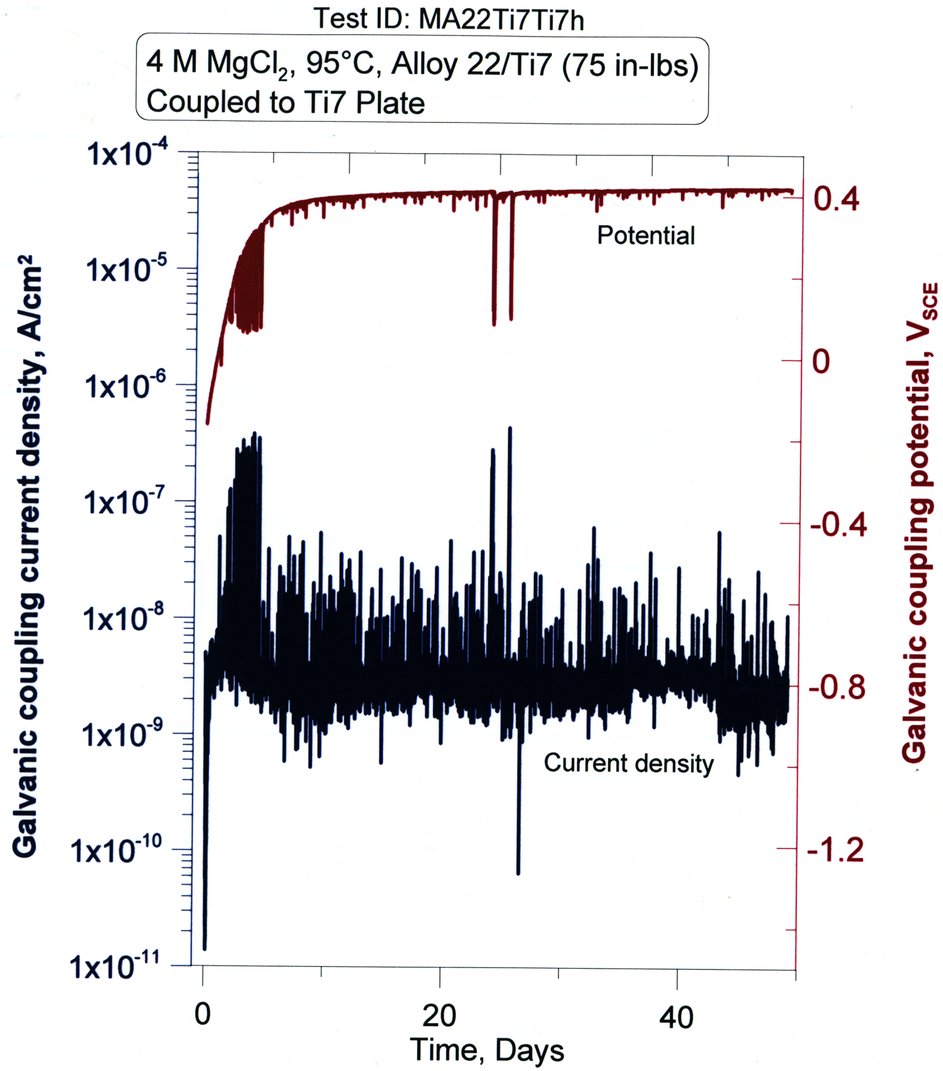
(PLATE)
Ecorr: -259 mV vs SCE
Model: Keithley 614 — Cal: 1/12/06 — SN#: 467374 — Due: 1/12/07

Potentiostat: Solartron 1480 — sn# 00240551 — cal: 5/3/06 — Due: 11/3/06

TEST ID: MA22Ti7Ti7h
DATA FILES: C22Ti7E0831a-Un1Ch1, C22Ti7E0831a-Un1Ch2, 0831, 0907, 0914, 0921, 0928, 1005, 1012, 1019

Specimen Examination: No Corrosion on specimen on Ti 7 Plate or Hardware. Very mild Surface Staining
* Note: will Replish for Further Testing 10/23/06

5/31/06



Xihua He 10/31/06

X-H 10/31/06

OCP Tests

Objective: to measure open circuit potentials

Specimen: C22 specimen Heat# 2277-3-3266 CNWRA Drawing 20.06002.01.322.002 polished to a 600 grit finish cleaned with Acetone

Crevice washers: Ti 7 Crevice Washers And Hardware (Large) HT# CN2775

Torque Screwdriver: TeveCraft 75 In-lbs — SN: 694000691 — Cal: 7/25/06 — Due: 7/25/07

Initial Weight: 23.93682g
Final Weight: 23.93701g
Model: Sartorius Genius — SN: 12809099 — Cal: 5/9/06 — Due: 11/9/06

SOLUTION: 4 m NaCl
467.5g NaCl Lot#052761
+ DI to 2000mls

Reagents measured with — **Model:** OHAUS — SN: 2883 — Cal: 7/5/06 — Due: 1/5/07

Initial pH: 4.89
Final pH: 7.73
Model: Orion — SN: 2330 — CAL: 7/6/06 — DUE: 7/6/07 — **pH Probe:** #13-620-296 — SN: 5003095

TEST TEMPERATURE: 95°C — **Measured with Hg Thermometer SN:** C96-637 — Cal: 8/19/06 — Due: 2/19/07

Counter Electrode: Platinum Flag

Reference Electrode: Fisher SCE — SN: 0066119

Gas: ~~99.999% Nitrogen~~ Zero Air 8to 14/06

Ecorr: -216 mV Vs SE
Ept: +293 mV Vs. SCE
Model: Keithley 614 — SN: 0704934 — Cal: 6/6/06 — Due: 6/6/07

Potentiostat: Solartron 1480 — SN# 00238265 — Cal: 9/11/06 — DUE: 3/11/07

Test ID: C22Ti7OCP01

Specimen Examination: No Visual Signs of Corrosion 0/24 feet of Ti 7 crevice washers - C-22 Specimen Appears To Have some Etching Below one of the Ti 7 Crevice washer feet - Color staining on Both C-22 And Ti 7 Hardware No Corrosion on Any of Ti 7 Hardware — No observable corrosion under microscope

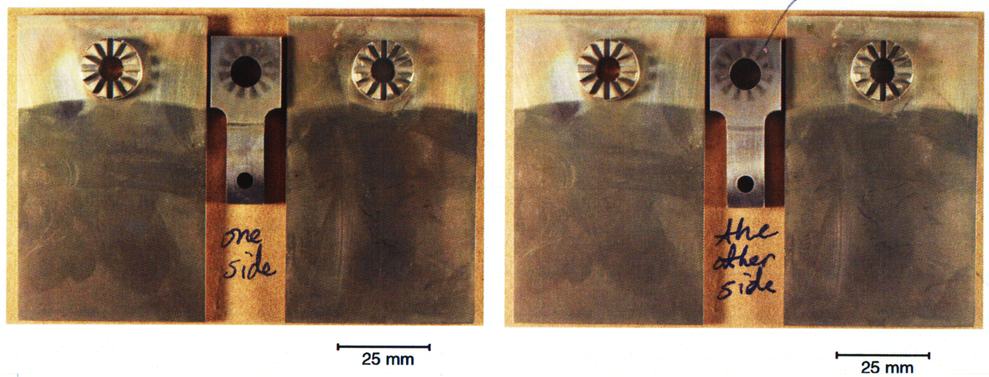
Data files: C22Ti7OCP-1003-Ua(Ch7), 1010, 1017, 1024, 1031, 1107, 1114, 1121, 1128, 1201, 1205, 1212, 1219, 1226

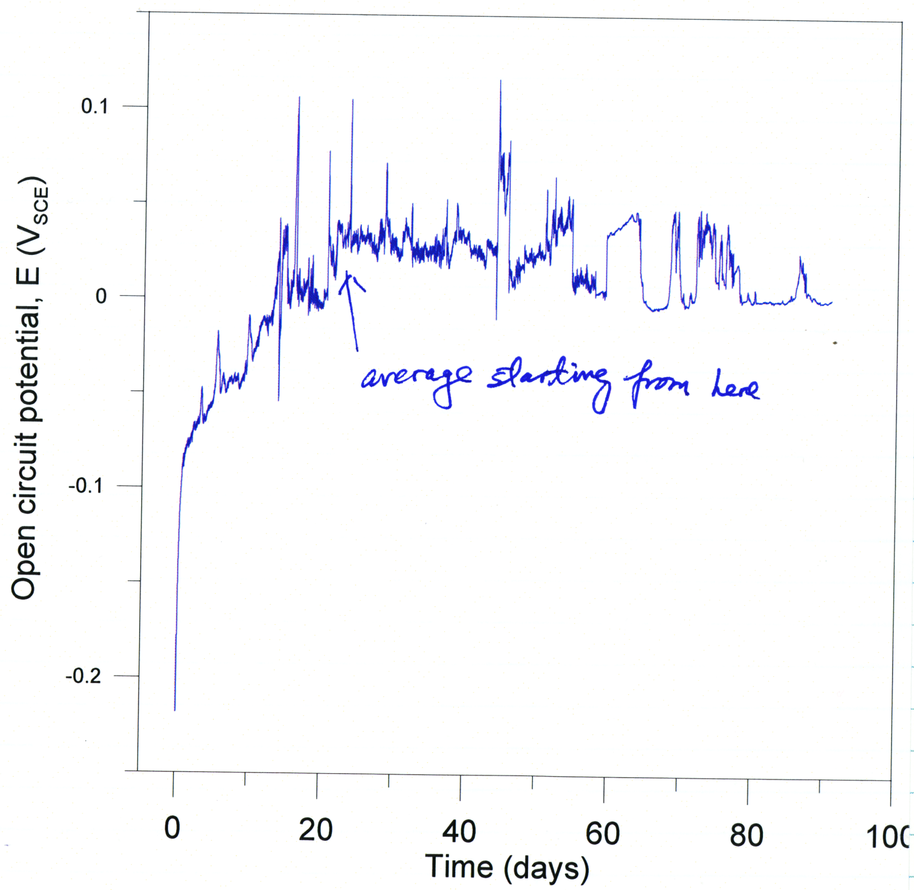
Test duration: 10/3/06 — 1/2/07 Total days: 92 days

10/3/06

specimen & washers after test

examined under microscope not corroded spot deposits build up





$\bar{E}_{average} = -0.0235\ V_{SCE}$

Xihua He 1/9/07

OCP Tests

Objective: to measure open circuit potentials

Specimen: C22 specimen Heat# 2277-3-3266 CNWRA Drawing 20.06002.01.322.002 polished to a 600 grit finish cleaned with Acetone

Crevice washers: C-22 HT#2277-3-3266 Crevice washers (Large) And Hardware

Torque Screwdriver: True Craft 75 In-lbs
Cal: 7/25/06 | SN: 694000 691 | Due: 7/25/07

Initial Weight: 23.89378g
Final Weight: 23.89343g
Model: Sartorius Genius | **Cal:** 5/9/06 | **SN:** 12809099 | **Due:** 11/9/06

SOLUTION: 4 m NaCl
467.58g NaCl Lot# 052761
+ DI To 2000 mls

Reagents measured with — **Model:** OHAUS | **Cal:** 7/5/06 | **SN:** 2883 | **Due:** 1/5/07

Initial pH: 4.93
Final pH: 5.44
Model: Orion | **CAL:** 7/6/06 | **SN:** 2330 | **DUE:** 7/6/07
pH Probe: #13-620-296 | **SN:** 5003095

TEST TEMPERATURE: 95°C — **Measured with Hg Thermometer SN:** 4279 | **Cal:** 6/1/06 | **Due:** 6/1/07

Counter Electrode: Platinum Flag

Reference Electrode: Fisher SCE | **SN:** 8131157

Gas: ~~99.999% Nitrogen~~ Zero Air (10/3/06 [initials])

Ecorr: -234 mV Vs SCE
Ept: +336 mV Vs SCE
Model: Keithley 614 | **Cal:** 6/6/06 | **SN:** 0764934 | **Due:** 6/6/07

Potentiostat: Solartron 1480 | SN# 00238265 | Cal: 9/11/06 | Due: 3/11/07

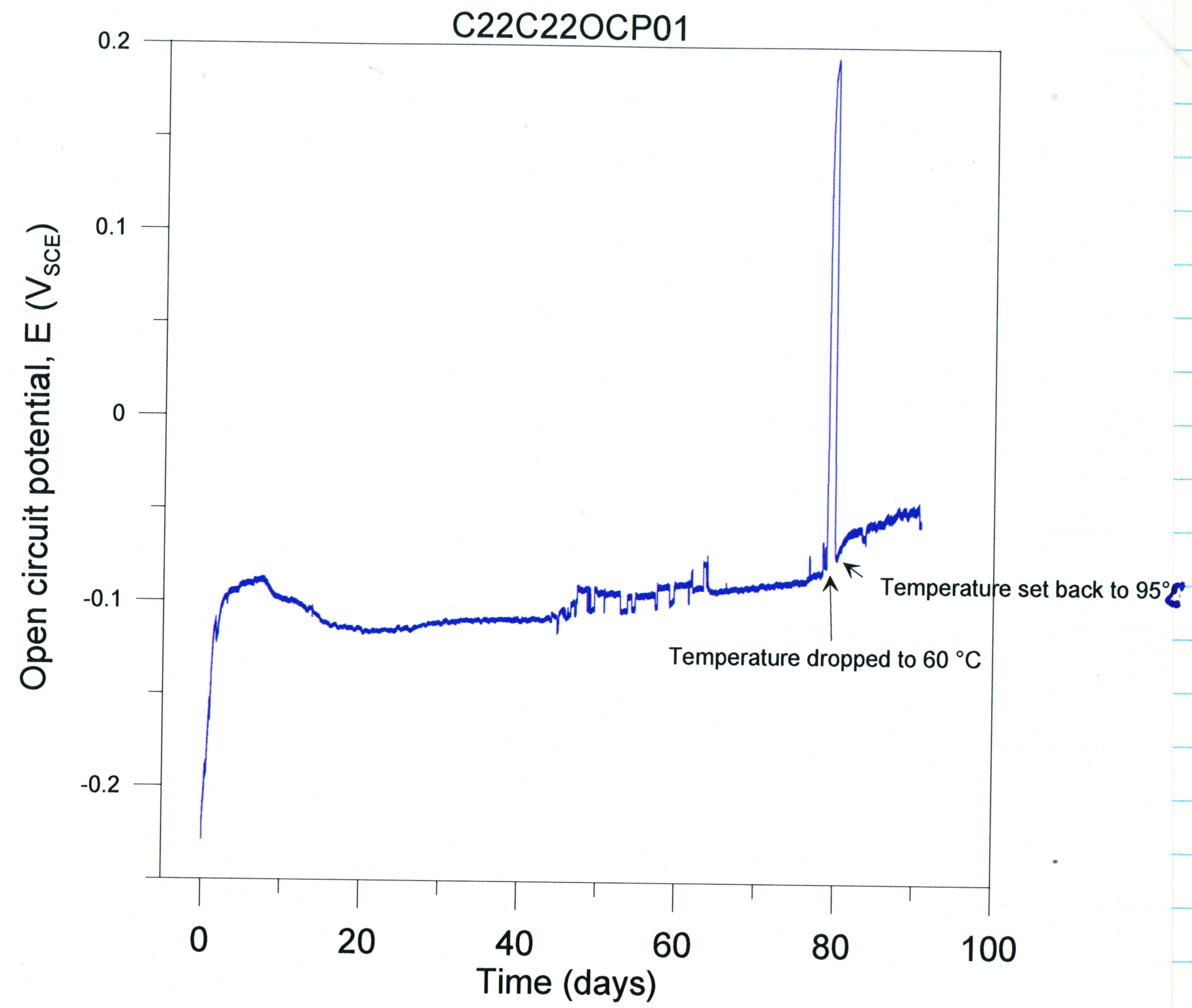
Test ID: C22C22OCP01

Specimen Examination: No Corrosion 9/24 feet of C-22 Specimen C-22 Hardware No Corrosion - Very mild surface staining on All C-22 material

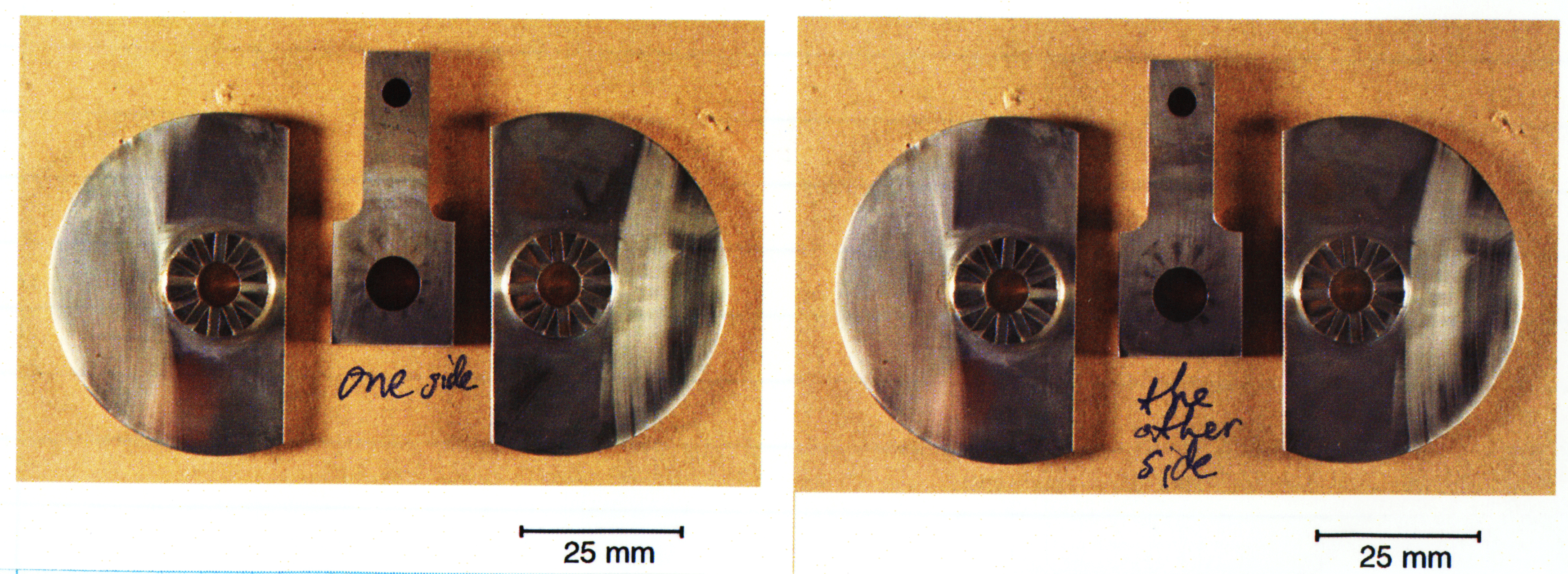
Data files: C22C22OCP-1003 thru 1018, C22C22OCP-1010, 1017, 1024, 1031, 1107, 1114, 1121, 1128, 1201, 1205, 1205a, 1212, 1219, 1226

Test time: 10/3/06 - 1/2/07

[signature] 10/3/06



Specimen & washers after test



Xihua He 1/9/07

OCP Tests

Objective: to measure open circuit potentials

Specimen: C22 specimen Heat# 2277-3-3266 CNWRA Drawing 20.06002.01.322.002 polished to a 600 grit finish cleaned with Acetone

Crevice washers: C-22 HT# 2277-3-3266 Crevice Specimen — Ti 7 HT# CN2775 (small) Crevice washers & Hardware

Torque Screwdriver: True Craft 75 In-lbs — Cal: 7/25/06 — SN: 694000691 — Due: 7/25/07

Initial Weight: 23.11403g
Final Weight: 23.11496g
Model: Sartorius Genius — Cal: 5/9/06 — SN: 12809099 — Due: 11/9/06

SOLUTION: 5 M NaCl
584.34g NaCl Lot# 054171
+ DI To 2000mls

Reagents measured with Model: OHAUS — Cal: 7/5/06 — SN: 2883 — Due: 1/5/07

Initial pH: 7.39
Final pH: 7.12
Model: Orion — CAL: 7/6/06 — DUE: 7/6/07
pH Probe: #13-620-296 — SN: 5003095

TEST TEMPERATURE: 95°C — Measured with Hg Thermometer SN: H98-182 — Cal: 5/11/06 — Due: 5/11/07

Counter Electrode: Platinum Flag

Reference Electrode: Fisher SCE — SN: 3329075

Gas: 99.999% Nitrogen

Ecorr: -214 mV Vs SCE
Ept: +256 mV Vs SCE
Model: Keithley 614 — Cal: 1/12/06 — SN: 467374 — Due: 1/12/07

Potentiostat: Solartron 1480 — SN# 00240551 — cal 5/3/06 — due 11/3/06

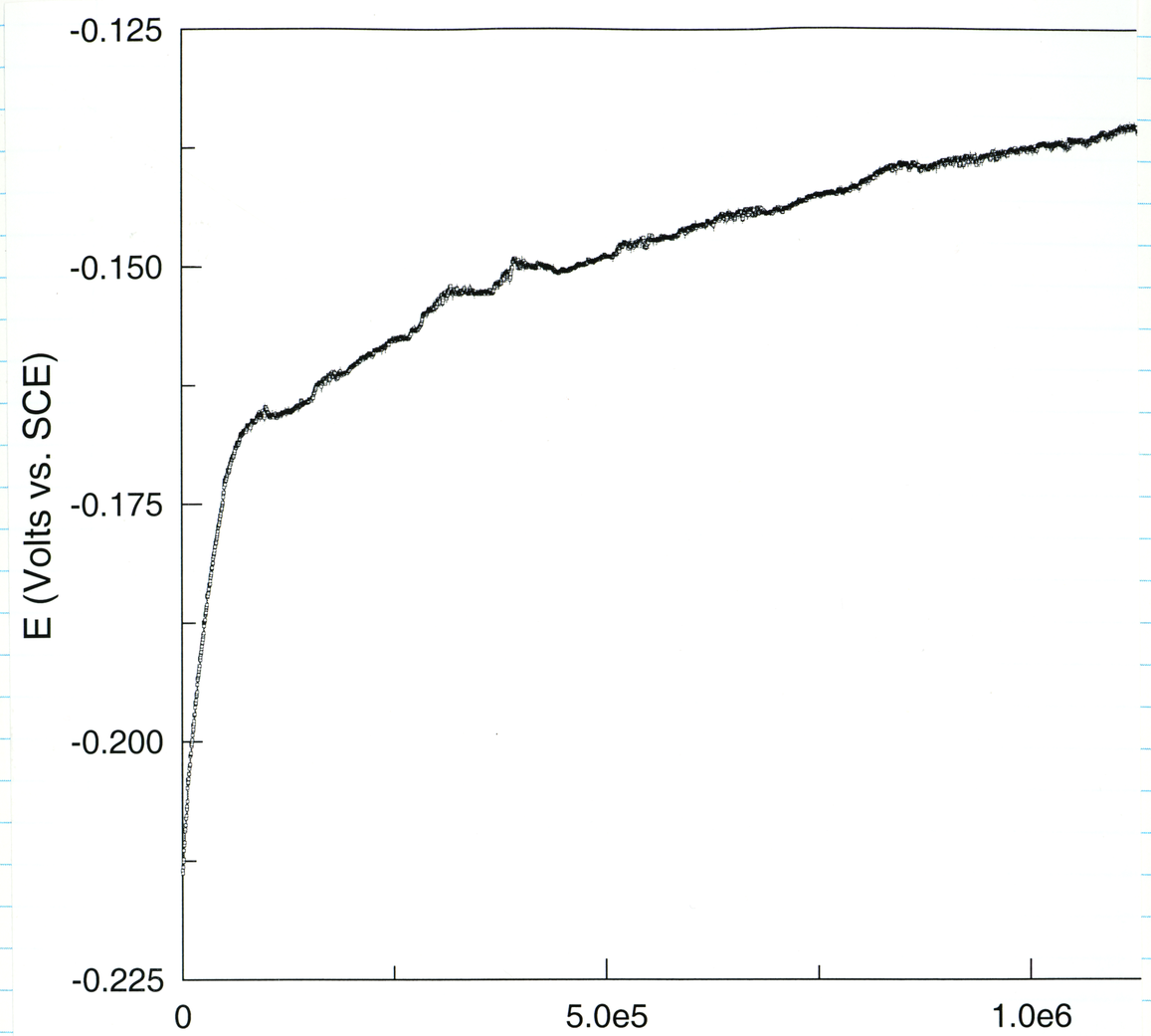
Test ID: C22Ti704P02 — Test time: 1.13x10^6 seconds = 13.1 days

Specimen Examination: Started 10/24/06 — shutdown 11/6/06

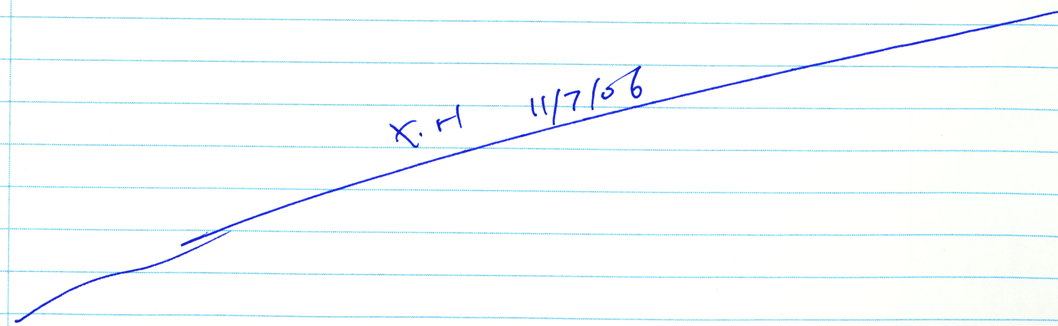
* No Corrosion on Specimen 9/24 feet of Ti 7 Crevice washers - No Corrosion or staining on Ti 7 Hardware or washers

* Specimen Repolished for Further Testing

10/24/06



Xuhua He 11/7/06



X.H 11/7/06

Galvanic Corrosion Test

Objective: SEE PAGE #1

SPECIMENS: Crevice Specimen drawing # 20.06002.01.322.002 polished to 600 Grit finish then cleaned in acetone

C-22 BA HT# 2277-3-3266 — Ti 7 Plate HT# CN 2775 — C276 Hardware PTFE Crevice washers

Torque Screwdriver: Proto 6104 50 In-Oz — **SN:** 139072 **Due:** 3/20/07
Cal: 9/20/06

Initial Weight: No weight
Final Weight: 23.45059 g
Model: Sartorius Genius — **SN:** 12809099 **Due** 11/9/06
Cal: BWP 11/7/06 5/9/06

SOLUTION: 4 m $MgCl_2$

Reagents measured with
Model: OHAUS — **SN:** 2883 **Due:** 1/5/07
Cal: 7/5/06

Initial pH: No pH — pH was not taken
Final pH: 1.66
Model: Orion EA 940 — **SN:**2330 **Due:** 7/6/07
Cal: 7/6/06
pH Probe: #13-620-296 — **SN:** 5003095

TEST TEMPERATURE: 95°C **Thermometer:** Fisher — **SN:** H 98-162 **Due:** 4/6/07
Cal: 4/7/06

Reference Electrode: Fisher SCE **# 13-620-52** — **SN:** 4028031

GAS: Zero Air

(CREVICE)
Ecorr: -327 **mV vs SCE**

(PLATE)
Ecorr: -288 **mV vs SCE**
Model: Keithley 614 — **SN#:** 0704934 **Due:** 6/6/07
Cal: 6/6/06

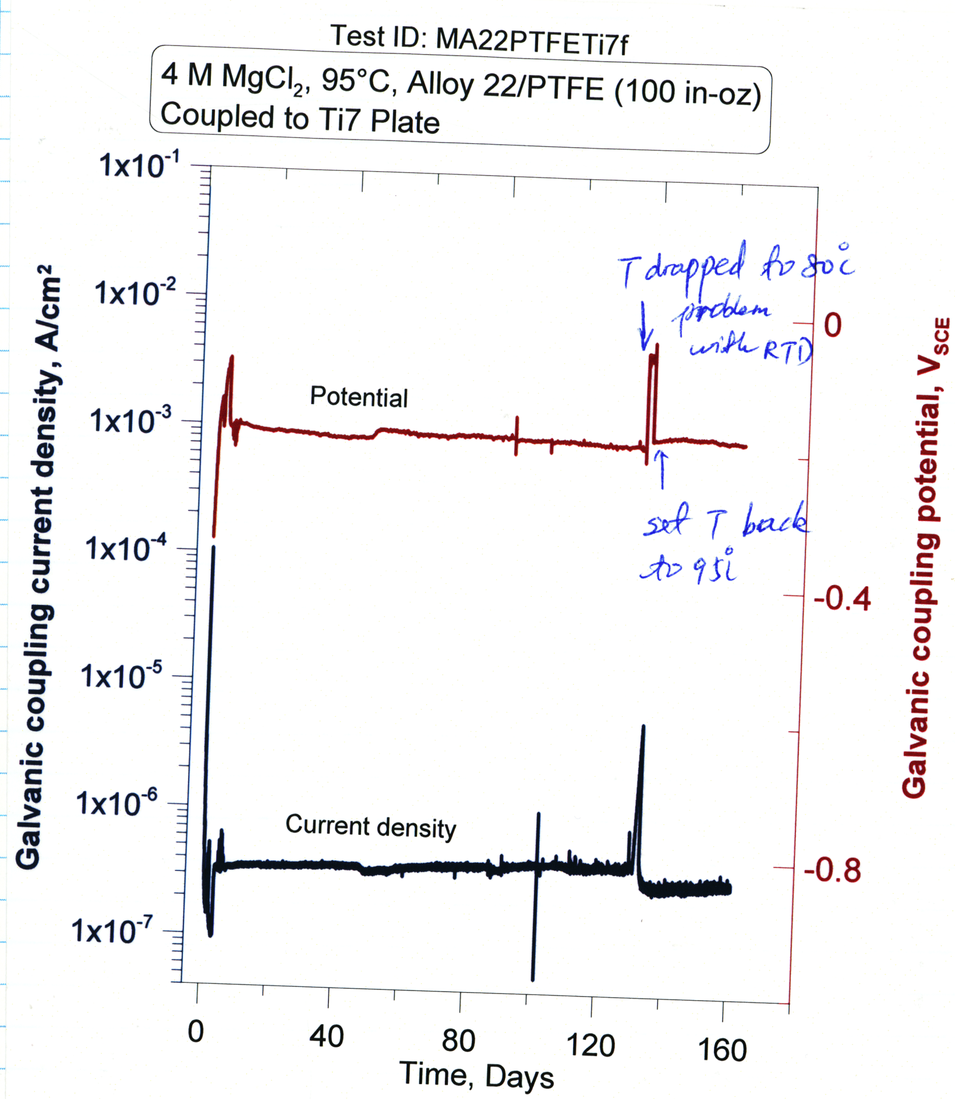
Test period: 7/28/06 – 1/5/07 total: 160.75 days
Potentiostat: Solartron 1480 — **sn#** 60240551 cal: 11/7/06 Due: 5/7/07

TEST ID: MA22PTFETi7f
DATA FILES: C22PTFETi7I0728a - Un1Ch3, C22PTFETi7I0728_Un1Ch3, 0804, 0811, 0818, 0825, 0901, 0908, 0915, 0922, 0929, 1006, 1013, 1020, 1027, 1103, 1110, 1117, 1124, 1201, 1205, 1208, 1215, 1222, E1229_Un1Ch4

Specimen Examination: Corrosion Developed on the Tip of PTFE Crevice Washer on the Specimen And It Has Creeped Into the Bolt hole Area Very Mild on the C-22 Specimen surface But Appears to Have More A Pit hole In the Bolt hole Location - Ti 7 Plate Dull tint Stain/y Corroded: 1/24 Sites

The depth is difficult to measure. X.H 11/18/07

11/7/06



Xihua He 11/1/07

OCP Tests

Objective: to measure open circuit potentials

Specimen: C22 specimen Heat# 2277-3-3266 CNWRA Drawing 20.06002.01.322.002 polished to a 600 grit finish cleaned with Acetone

Crevice washers: C-22 HT#2277-3-3266 washers & Hardware

Torque Screwdriver: True Craft (Small) 75 In-lbs SN: 694000691 Cal: 7/25/06 Due: 7/25/07

Initial Weight: 23.34086g **Final Weight:** 23.34029g Model: Sartorius Genius SN: 12809099 Cal: 5/9/06 Due: 11/9/06

SOLUTION: 4 m NaCl
467.55g NaCl #054171 Lot
+DI To 2000mls

Reagents measured with Model: OHAUS SN: 2883 Cal: 7/5/06 Due: 1/5/07

Initial pH: 7.21 **Final pH:** 7.52 Model: Orion CAL: 7/6/06 DUE: 7/6/07 pH Probe: #13-620-296 SN: 5003095

TEST TEMPERATURE: 95°C Measured with Hg Thermometer SN: C96-852 Cal: 6/27/06 Due: 12/27/06

Counter Electrode: Platinum Flag

Reference Electrode: Fisher SCE SN: 4028035

Gas: ~~99.999% Nitrogen~~ flow 11/7/06 Zero Air

Ecorr: -237 mV Vs SCE **Ept:** +223 mV Vs SCE Model: Keithley 614 SN: 0704934 Cal: 6/6/06 Due: 6/6/07

Potentiostat: Solartron 1480 SN# 00240551 cal 11/7/06 due: 5/7/07

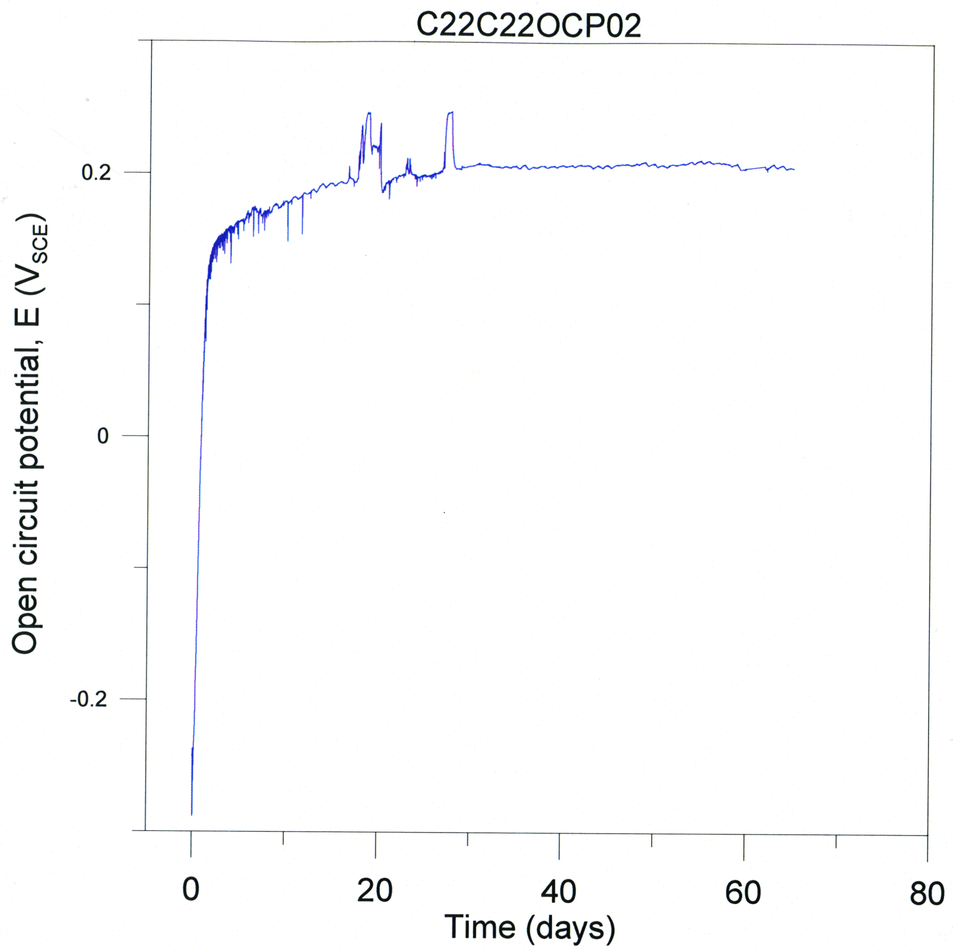
Test ID: C22C22OCP02

Specimen Examination: started 11/7/06 ended 1/11/07

No Corrosion on Specimen - Surface Staining on All Surfaces - Slight scratching from Crevice Washers - All C-22 Hardware & washers No Corrosion

Data files: ocpc22c221107_Unlch1, ocpc22c221114_Unlch1, ocpc22c221121, ocpc22c221128, 0105, 0109, 0116, 0123, 0130, 0108

11/7/06



X-Linear Fit 1/11/07

The data is invalid. please refer to NCR 2007-14.

X-Linear Fit 3/26/07