

308

Q200212020002

Scientific Notebook # 328

LABORATORY NOTEBOOK

CNWRA/SwRI

NOTEBOOK NO. _____

ISSUED TO Charles A. Greene

ON May 3, **19** 99

DEPARTMENT _____

RETURNED _____ **19** _____

Division - Project - Task: 20-1402-571

CNWRA
CONTROLLED
COPY 328

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5/22/99

Brian K. Deiby - B. Deiby BKB

INSTRUCTIONS

1. The primary purpose of this notebook is to protect your and the Company's Patent-Rights by keeping records of all original work in a form acceptable as evidence if any legal conflict arises.

2. • When starting a page, enter the title, project number, and book number.
• Use ink for permanence -- avoid pencil.
• Record your work as you progress, including any spur-of-the-moment ideas which may be developed later.
• Avoid making notes on loose paper to be recopied.
• Record your work in such a manner that a co-worker can continue from where you stop. You might be ill and to protect your priority it could be urgent that the work continue while you are absent.

3. • Give a complete account of your experiments and the results, both positive and negative, including your observations.
• Record all diagrams, layouts, plans, procedures, new ideas, or anything pertinent to your work including the details of any discussions with suppliers, or other people outside the Company.
• Do not try to erase any incorrect entries; draw lines deleting them, note the corrections, sign and date the changes. This extra care is worthwhile because of the necessity of original data to prove priority of new discoveries.

4. • After entering your data, sign and date the entries.
• Explain your work to at least two wit-

nesses who are not co-inventors, and have them sign and date the pages in the place provided.

- Record the names of operators and witnesses present during any demonstration and have at least two witnesses sign the page. If no witnesses are present during an experiment of importance, repeat it in the presence of two witnesses.

5. Since computer programs can be patented these instructions apply to the development of computer software. In this case a description of the structure and operation of the program should be recorded in the notebook, together with a basic flow diagram which illustrates the essential features of the program. In the course of developing the code, the number of lines of code written each day should be recorded in the notebook, together with a statement of the portion of the flow diagram to which the section of code is directed.

6. This notebook and its contents are the exclusive property of the Company. It is confidential and the contents are not to be disclosed to anyone unless authorized by the Company. You must return it when completed, upon request, or upon termination of employment. It should be kept in a protected place. If loss occurs, notify your supervisor immediately, and make a written report describing the circumstances of the loss.

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TITLE

From Pa

Initial Scientific notebook entry for Zircaloy-4 potentiodynamic polarization tests.

Title: Cyclic polarization tests, crevice repassivation tests.

Tests Performed by: Charles A. Greene

Charles A. Greene 5/5/99

Objectives: Measure crevice repassivation potential of Zircaloy-4.

Equipment: EG&G Versastat Serial Number 20104. EG&G model 352 corrosion software. NEC 586 computer. Keithley Electrometer model 614 SN 555368 or equivalent. ASTM G-5 polarization cell, Large 2 L glass cells with Teflon tops, Electrochemical Impedance Spectroscopy system including Solartron 1260 FRA and Solartron 1287 Potentiostat. ESC 440 multichannel potentiostats with National instruments Labview data acquisition software or Strawberry Tree data acquisition software.

Materials: Zircaloy-4

Specimen specifications: Crevice repassivation specimens with Teflon crevice washers attached to surface. Additional specimens to be identified at time of testing

Measurement Parameters: Current and Potential as described in TOP-008. Temperature of solution $\pm 2^{\circ}\text{C}$

Required level of accuracy: Potentials $\pm 5\text{mV}$. Current less than 0.1 microamp.

Uncertainty and Sources of Error: Current density calculated as current divided by sample area. Actual current density of corroding areas is not determined.

Div. Proj task
20-1402-571

Witness

BS 5/5/99

From Page No. _____

Cyclic Polarization Test EG&G VersaStat

Material: 316 L Heat No. p80746

Polished to 600 Grit - cleaned in Methanol

Weight: 29.3450 g Sartorius balance S/N 10704379 due: 25 Sept 99

Specimen dimensions:

Calipers: CAL: 03/16/99 DUE: 09/16/99

AN: 002185 SN: 20-8C-1

H - 0.744" W - 0.746" T - 0.372

0.746 0.746 0.373

0.742 0.750 0.371

H = 0.744" W = 0.748" T = 0.372"

D - 0.320"

0.321

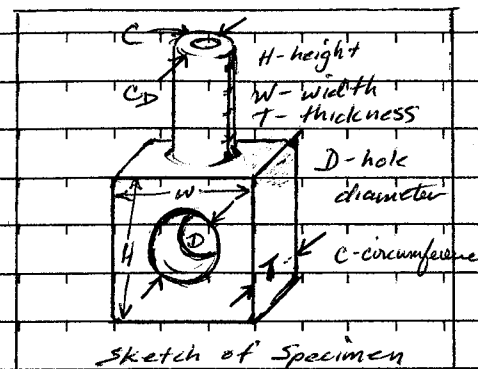
0.321

D = 0.321"

C_D - 0.298

0.300

0.298

C_D = 0.299"Surface Area: $2 \times T \times W + 2 \times T \times H + 2 \times W \times H - 2(\pi(\frac{D}{2})^2) + 2\pi(\frac{D}{2}) \times T$ Surface Area = 2.366145 in² = 2.37 in²
2.37 in² = 15.26 cm²

Solution

1000 ppm Cl⁻ 1.648 g # 97227485 ppm HCO₃⁻ 0.120 g # 89778920 ppm SO₄⁼10 ppm NO₃⁻2 ppm F⁻4/99 standard solutions 20 mL
10 mL
2 mL
1000 ppm
expires 5/13/99

Thermometer S/N C96816 T = 95°C

E_{corr} = -380 mV vs. SCE Keithley 614 S/N 0704936E_{graphite} = 160 mV vs SCE

To Page No. 3

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Charles A. Munn

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software setup:

Comment: 316 L SS 1000 ppm Cl 95 C deaerated w/N₂ crevice specimenCond. Time CT pass s Initial Pot. 1P -100.0E-3 V_{oc}Cond. Pot. CP pass V Vertex 1 Pot. V1 100.0E-3 V_{oc}Initial Delay ID 10 s I Threshold IT 5.000E-3 A/cm²

Final Pot. FP -100.5E-3

SI 2.000E-03

SR 1.669E-04

ST 1.198E+01

CR AUTO

NP 3999

IR None

FL 15.3 Hz

RT HIGH STABILITY

REF 0.24150 SCE

WRK SOLID

AR 1.526E+01

LS YES

IT 7.630E-02

ITA 1.526E+01

EN 0.000E+00

DEN 8.000E+00

AU NO

END

Weight after test: 29.3028

S/N 10704379

Red - counter electrode (CE)

green - working electrode (WE)

yellow (white) - reference electrode (RE)

Electrometer: red to RE and black to WE

Potentiostat noted in Performance Confirmation

Laboratory notebook #308, TOP 22 used to verify

To Page No. 8

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Book No. _____ TITLE _____

From Page No. _____

MATERIAL TEST REPORT

DATE : 05/04/99

PAGE : 1

ORDER: 35576

Metal Samples Company

P.O. Box 8

152 Metal Samples Road

Munford, AL 36268

Ph. (256)358-4202 Fx. (256)358-4515

Customer: 01482

SOUTHWEST RESEARCH INSTITUTE

Your PO#: 994062X

Lot No. E350

Mill: TELEDYNE WAH

Our Order Line No. 1

Description: ZR4

.588 X 3 X 69 7

Chemical Properties:

Cr:0.100

Fe:0.210

Sn:1.520

Physical Properties:

Condition:ANN

=====

We certify that the Material Test Report is correct to the best of our knowledge and that the material supplied meets your required P.O. specifications.

THANK YOU, Quality Control Dept.

Charles A. Greene 5/5/99

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04/02/1999 13:54 2563584515

ASPI

PAGE 06

Page 1 of 1

TO
ADDRESSMetal Samples
Route 1, Box 152
Munford, AL 36268

RECEIVED DEC 13 1985

TELEDYNE
WAH CHANG ALBANYP.O. BOX 480
ALBANY, OREGON 97321

(503) 926-4211 TWX (510) 595-0973

ATTENTION OF: Purchasing Agent

IN REGARD TO YOUR

PURCHASE ORDER NO. Sample

ITEM NO. 3200

DESCRIPTION Zr4 Plate

DIMENSIONS .575" x 3" x 69.69"

SPECIFICATIONS Per Purchase Order

ISSUED DATE: December 9, 1985

REVISED DATE:

DATE SHIPPED Ref. P.L.#

QUANTITY SHIPPED 1 pc.

WEIGHT SHIPPED 30.0 lbs.

SALES ORDER NO. 8100-467

HEAT NO. 220947Q Zr4

MACS NO. 43325

THE TEST REPORT FOLLOWS:

Material Condition: Annealed

INGOT ANALYSIS
COMPOSITION IN PERCENT

Element	1	2	3	4	5
Sn	1.52	1.53	1.52	1.54	1.45
Fe	0.21	0.21	0.20	0.22	0.18
Cr	0.10	0.11	0.10	0.11	0.10
Fe+Cr	0.31	0.31	0.30	0.32	0.28
Zr					BALANCE

MAXIMUM IMPURITIES IN PPM

Al	<33	31	26
B	<0.2	<0.2	<0.2
C	<100	100	170
Cd	<0.2	<0.2	<0.2
Co	<10	<10	<10
Cu	<25	<25	<25
Gd	<2.5	---	<2.5
H	<5	<5	<5
Hf	<39	40	37
Mn	<25	<25	<25
Mo	<10	---	<10
N	<16	19	10
Ni	<35	<35	<35
O	<1420	1500	1520
P	<8	---	7
Pb	<25	---	<25
Si	<94	92	81
Sm	<5	---	<5
Ta	<100	<100	<100
Ti	<3.5	---	<3.5
Ti	<25	<25	<25
U	<1.0	---	<1.0
U235	<0.010	---	<0.010
V	<25	---	<25
W	<50	<50	<50
Zn	<50	---	<50

MATERIAL
CERTIFIED BY

gf

Tony Nelson 129
Quality Assurance Dept.A1.04.25.02.06.01-01
C1.01.06.20*Charles A. Greene* 5/5/99

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Initial planning meeting 5/3/99 (Entered in notebook 5/6/99)
 Gustavo Cragolino, Darrell Dunn, Sean Brosna, Charles Greene.

Cyclic Polarization - Repassivation Potential Cyclic Potentiodynamic Tests.

Environment:

Chlorides, pH, Temperature (T)

T: three temperatures 25-95°C concentrate on higher T.

Test matrix: 30 tests - 30 crevice specimens (repolishable)

Cl⁻: three concentrations

J-13 simulated H₂O. with carbonate?
 Na⁺ as only cation.

Corrosion Potential verify & confirm (longer term)

oxidizing species with & without Fe³⁺

basket material in Waste Package: C-steel

galvanic coupling A-516

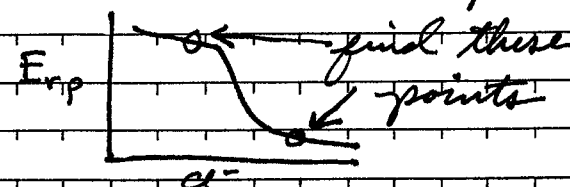
Equipment: EG&G cell,

rotation potentiostats

salt bridges heaters

pH → start around natural ~8.4 vary this parameter last.

lower pH. pH less than 1 to 14 for 2 different Cl⁻ concentrations. Check literature on zirconium E_{rep} dependence on pH.



0.001 M Chloride to 1 M Cl⁻

first: 1 M Cl⁻/25°C

2nd: 1 M Cl⁻/95°C

3: 95°C/decrease Cl⁻ conc.

later explore temperature effect or decide to go to 4 M

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Cyclic Polarization Test EG&G VersaStat

Material: Zircaloy-4 Heat No. 220947Q Zr4 (see pages 4&5)

Polished to 600 grit - cleaned in methanol

Weight: 29.97636 g

S/N 10704379

001: + 29.97636 g

002: + 29.97636 g

Room Temperature

T = 24°C S/N 096816

Charles A. Greene 5/7/99

Solution: pH

1 M Cl⁻ 58.441 g # 972274

Sodium Chloride

85 ppm HCO₃⁻ 0.117 g # 897789

Sodium Bicarbonate

20 ppm } 4/99 standard solutions 20 mL of 1000 ppm

10 ppm } expires 5/13/99 10 mL of 1000 ppm

2 ppm } 2 mL of 1000 ppm

pH S/N 3340 Fisher Scientific

standard pH 10 # 981233-24 exp. date 3/2000

made 9.976

solution: pH = 7.69, 7.69

H = 0.748" $2 \times (.493) \times (.739) - (0.070215) (= 0.658439)$

W = 0.739" $+ 2 \times (.493) \times (.748) - 2 \times (.162866) (= 0.737528)$

T = 0.493" $+ 2 \times (.739) \times (.748) - 2 \times \left[\pi \left(\frac{.322}{2} \right)^2 \right] (= 0.942678)$

D = 0.322" $+ 2 \times \left(\pi \left(\frac{.322}{2} \right)^2 \right) \times (0.493) (= 0.498715)$

C_D = 0.299" $\rightarrow C = \pi \left(\frac{.322}{2} \right)^2 = 0.070215$ Area = 2.83736 in²

Calipers S/N: 20-8C-1

x (2.54)²

Surface Area = 18.30 cm²

E_{corr} OC - 823 mV vs SCE Keithley 614 S/N 0704936

E_{graphite} 152 mV vs SCE

Torque: 50 in-oz

Final weight: 29.95585 g

S/N 314047

S/N 10704379

Setup file: Zrsetup

file name: Zr4cpp01.dat / Zr4cpp02.dat - test restarted

After test pH = 8.76

S/N 3340

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5/7/99

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Date

CA Greene
5/7/99

From Page No. _____

Model 352/252 Corrosion Analysis Software, v. 2.30
 Filename: a:\316cpp01.dat
 Pstat: VStat() Ver 2
 CP CYCLIC POLARIZATION
 Date Run: 04-12-99 File Status: NORMAL
 Time Run: 13:28:39

Cond. Time	CT	pass	s	Initial Pot.	IP	-100.0E-3	V oc
Cond. Pot.	CP	pass	V	Vertex 1 Pot.	VI	100.0E-3	V oc
Initial Delay	ID	10	s	I Threshold	IT	5.000E-3	A/cm ²
				Final Pot.	FP	-100.0E-3	V oc

Scan Rate	SR	166.9E-3	mV/s	Curr. Range	CR	Auto	
Scan Incr.	SI	2.000	mV	Step Time	ST	11.90	s
No. of Points	NP	1174					

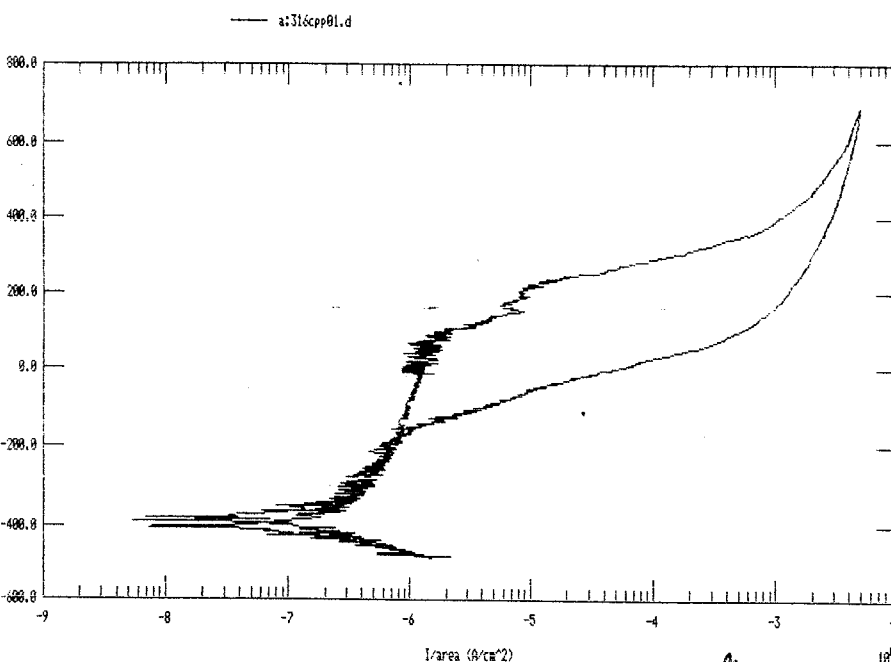
Line Sync.	LS	yes		GI Time Const.	TC	Off	
Rise Time	RT	high stability		IR Mode	IR	none	
Working Elec.	WE	Solid		Filter	FL	1 5.3Hz	
Sample Area	AR	15.26	cm ²	Ref. Elec.	RE	SCE 241.5E-3V	
Density	DE	0.500	g/ml	Equiv. Wt.	EW	27.50	g
Open Circuit	OC	-304.0E-3	V	AUX A/D	AU	no	

Comment: 316L SS 1000 ppm Cl 95 C deaerated w/42 Greive specimen

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Model 352/252 Corrosion Analysis Software, v. 2.30
 Filename: a:\316cpp01.dat
 Pstat: VStat() Ver 2
 CP CYCLIC POLARIZATION
 Date Run: 04-12-99 File Status: NORMAL
 Time Run: 13:28:39

CP PASS vs. R	CT	PASS	IP	-0.100 vs. OC	ID	10 S	VI	0.100 vs. OC	FP	-0.100 vs. OC	
SI	2.000E-03	SR	1.669E-04	ST	1.190E+01	CR	AUTO	NP	1174	IR	NONE
FL	1 5.3Hz	RT	HIGH STABILITY	REF	0.24150 SCE	WKS	SOLID	AR	1.526E+01	LS	YES
IT	7.630E-02	ITA	1.526E+01	EN	2.750E+01	DEN	0.000E+00	AU	NO	OC	-0.304



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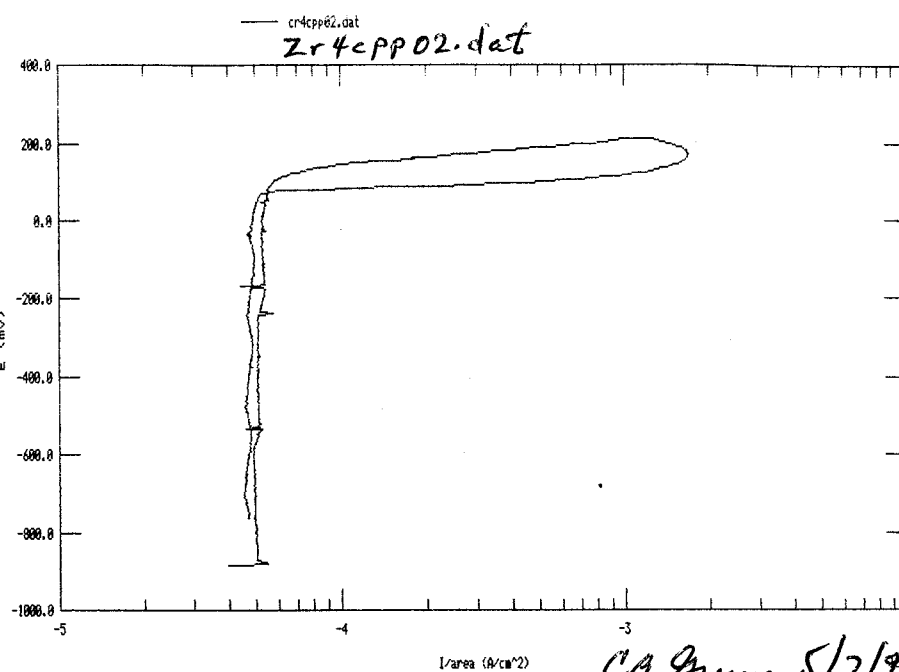
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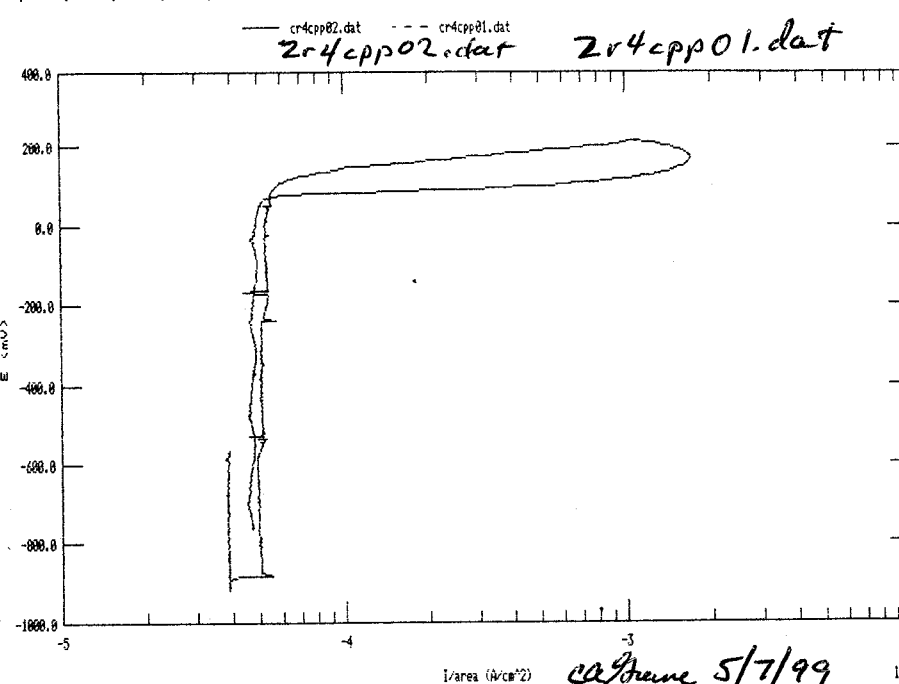
From Page No. _____

Model 352/252 Corrosion Analysis Software, v. 2.30
 Filename: a:\cr4cpp02.dat
 Pstat: VStat() Ver 2
 CP CYCLIC POLARIZATION
 Date Run: 05-07-99 File Status: NORMAL
 Time Run: 08:29:02

CP PASS vs. R	CT	PASS	IP	-0.100 vs. OC	ID	10 S	VI	0.101 vs. R	FP	-0.250 vs. OC	
SI	3.000E-03	SR	1.667E-04	ST	1.000E+01	CR	AUTO	NP	692	IR	NONE
FL	1 5.3Hz	RT	HIGH STABILITY	REF	0.00000 User	WKS	SOLID	AR	1.830E+01	LS	YES
IT	1.830E-02	ITA	1.830E+01	EN	0.000E+00	DEN	0.000E+00	AU	NO	OC	-0.780



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Test Matrix for Cyclic Polarization Tests on Zircaloy-4 Crevice Specimens
20 Specimens available plus 10 for confirmatory long term corrosion potential tests.

Test No.	Temp. (C)	Cl- Conc. (M)	pH	Comment
① → 1	25	1	7	Test underway - 05/07/99 (TEST 1) 5/17/99 ca ₂
2	25	1	2	
3	25	1	12	
⑦ → 4	65	1	7	5/17/99 ca ₂
5	65	1	2	
② → 6	65	1	12	
7	95	1	7	Next test planned - 05/10/99 (TEST 2) 5/10/99 ca ₂
8	95	1	2	
9	95	1	12	
⑩ → 10	25	0.1	7	5/18/99 ca ₂
11	25	0.1	2	
12	25	0.1	12	
⑪ → 13	65	0.1	7	5/18/99 ca ₂
14	65	0.1	2	
15	65	0.1	12	
⑤ → 16	95	0.1	7	5/14/99 ca ₂
17	95	0.1	2	- 5/20/99 ca ₂
18	95	0.1	12	- 5/21/99 ca ₂
⑧ → 19	25	0.01	7	5/17/99 ca ₂ 5/18/99 ca ₂
20	25	0.01	2	
21	25	0.01	12	
⑨ → 22	65	0.01	7	5/17/99 ca ₂
23	65	0.01	2	
24	65	0.01	12	
⑥ → 25	95	0.01	7	5/14/99 ca ₂
26	95	0.01	2	
27	95	0.01	12	
⑭ → 28	25	0.001	7	5/19/99 ca ₂
29	25	0.001	2	
30	25	0.001	12	
⑬ → 31	65	0.001	7	- 5/20/99 ca ₂
32	65	0.001	2	
33	65	0.001	12	
③ → 34	95	0.001	7	5/13/99 ca ₂
35	95	0.001	2	
36	95	0.001	12	
⑫ → 37	25	4	7	5/18/99 ca ₂
38	25	4	2	
39	25	4	12	
⑬ → 40	65	4	7	5/19/99 ca ₂
41	65	4	2	
42	65	4	12	
④ → 43	95	4	7	5/13/99 ca ₂ ca ₂ name 5/10/99
44	95	4	2	
45	95	4	12	

oxidizing species -
Fe3+
w & w/o peroxideAfter TESTS 1 & 2 - decrease Cl- Conc.,
later explore temperature effect
OR decide to go to 4 M Cl-

due to high current density in test on page 9, we will perform
Test 1 without the crevice.

Cyclic Polarization Test - Non creviced. EG&G Versa Stat

Material: Zircaloy-4 Heat No. 2209479 Zr4

Polished to 600 grit, center hole not polished, - cleaned in methanol
dried, weighed, weight: 29.82450g S/N 10704379

To Page No. 11

Witnessed & Understood by me,

Charles A. Purnell

Date

5/10/99

Invented by

Recorded by

Date

TITLE _____

From Page No. _____

Solution:

1 M Cl⁻ 58.442g #98530285ppm HCO₃⁻ 0.118g #89778920ppm SO₄²⁻ }10ppm NO₃⁻ } 4/992ppm F⁻ }

pH: 7.69 S/N 3340

E_{corr} OC = -953 mV vs SCE → -962 mV vs. SCEE_{platinum} = +287 mV vs SCE

Keithley 614 S/N 0704936

Save file as Zr4cpp03.dat

Checked current reading with Fluke 8050A Digital Multimeter
S/N 5005078

change salt bridge out for luggin probe - resolved problem.

file name Zr4cpp04.dat

Temperature: 20°C S/N C96816

pH after test: 8.56 S/N 3340

final weight: 29.80439g S/N 10704379

To Page No. 12

Witnessed & Understood by me,

Charles A. Purnell

Date

5/10/99

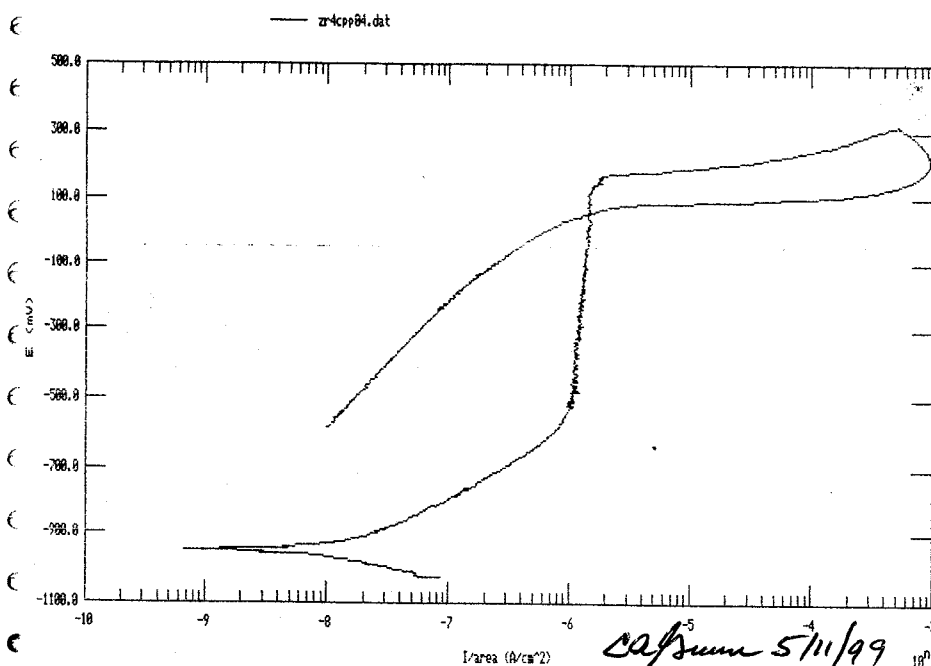
Invented by

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Date

From Page No. 11

Model 352/252 Corrosion Analysis Software, v. 2.30
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-09-99 Time Run: 10:47:46 Pstat: VStat() Ver 2
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI 0.899 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 749 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User WPK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.926
 Comment: Zr-4 IM Cl 25C pH 7.69 deaerated w/N2 Boldly exp. specimen



Solution of 1M Cl⁻ for salt bridge:
 58.44133g NaCl Lot # 985302 balance S/N 10704379

Cyclic Polarization test Crevised Specimen EG&G versaStat

Material Zr alloy-4 Heat No. 220947Q Zr-4

Polished to 600 grit cleaned in methanol

Weight: 29.79777g S/N 10704379

Solution: 1M Cl⁻ 58.44071g # 985302

85 ppm HCO₃⁻ 0.118g # 897789 (0.11800g)

20 ppm SO₄²⁻ } 4/99

10 ppm NO₃⁻ }

2 ppm F⁻ }

pH 7.714 - 7.698 S/N 3340 Fisher Scientific

pH electrode 13-620-296 S/N 5046380

To Page No. 13

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. 12

Temperature (S/N C96816) is 96°C to 95.5°C

E_{corr} OC = -998 mV to -962 mV

E_{platinum} = -49 mV to -47 mV

using salt bridge (no string, 1M Cl⁻ sol'n)

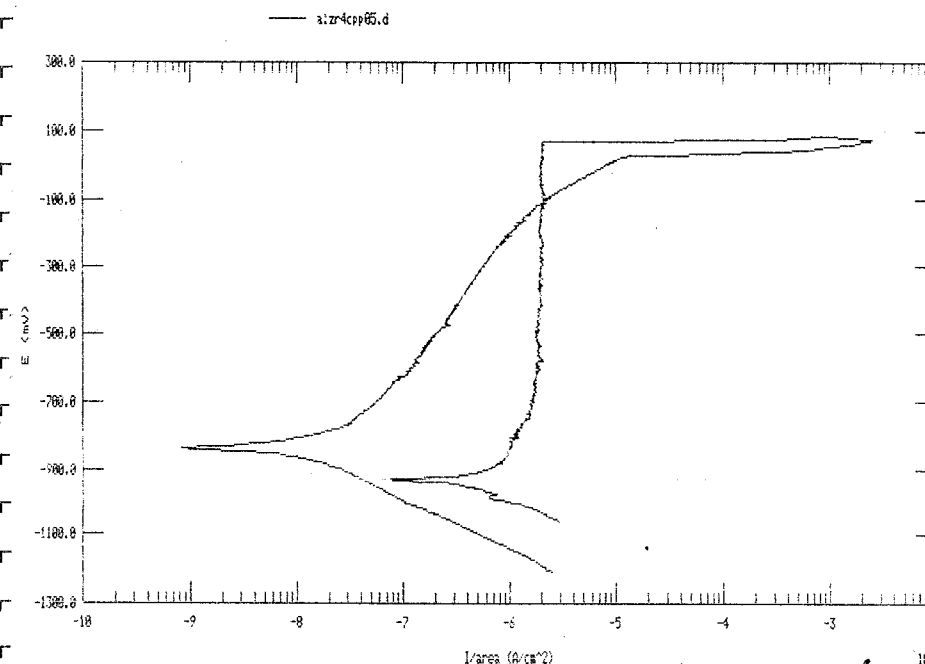
Torqued: 50 in. oz S/N 314047

Reference Electrode used to this point is Accumet 13-620-51
 S/N 8122010

zr4cpp05.dat
 final weight: 29.79477g

final pH: 9.28 I threshold = 500.0 E-6 A/cm²

Model 352/252 Corrosion Analysis Software, v. 2.30
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-10-99 Time Run: 11:10:25 Pstat: VStat() Ver 2
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.500 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 812 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User WPK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.961
 Comment: Zr-4 IM Cl 95C pH 7.69 deaerated w/N2 Crevised specimen



ca. 5/13/99

To Page No.

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. _____

discussed with Gustavo - next test (#34 in matrix on p.10)
0.001 M Cl^- neutral pH

Current $I_{REV} = I_{\text{density}} \times \text{Area} = 500 \text{ E-6 A/cm}^2 \times 18.3 \text{ cm}^2 = 0.009150$
0.05844 g Cl^- to make 0.001 M

Solution

0.05867 g #985302 0.001 M Cl^-
0.11670 g #897789 85 ppm HCO_3^-
4/99 { 20 ppm SO_3^{2-}
10 ppm NO_3^-
2 ppm F^-

Zr-4 CPP Crevise Specimen Torque: 50 in-oz S/N #2
Temperature: 95°C SN H98162

Solartron

Corrware Software

Specimen weight: 29.88700

pH: 8.085 = 8.08

100 mM salt bridge solution

Reference electrode 13-620-51 S/N 5144349

$E_{\text{corr OC}} = -837 \text{ mV vs SCE}$
 $E_p = -109 \text{ mV vs SCE}$ } Keithley 644 Electrometer

start test 3 pm

4 pm began to reverse current too soon due to
0.5 V E vertex setting.

restarted test T = 95°C SN H98162

Zr4cpp06.dat

Zr4cpp07.dat

To Page No. 16

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

TITLE _____

From Page No. _____

EG & G VersaStat

4 M Cl^- neutral pH crevice CPP test

Solution

233.762230 g #985302 4 M Cl^-
0.11893 g #897789 85 ppm HCO_3^-
4/99 { 20 ppm SO_3^{2-}
10 ppm NO_3^-
2 ppm F^-

pH = 7.360

Specimen weight: ^{CA915/13/99} 30.12160 g
30.11708 g

Zr4cpp08.dat Torque 50 in-oz SN #2

 $E_{\text{corr OC}} = -0.8082 \text{ V vs SCE}$ $E_p = 0.0241 \text{ V vs SCE}$

test began 6:24 pm 95°C SN C96816

SAVE AS Zr4cpp08.dat

To Page No. 17

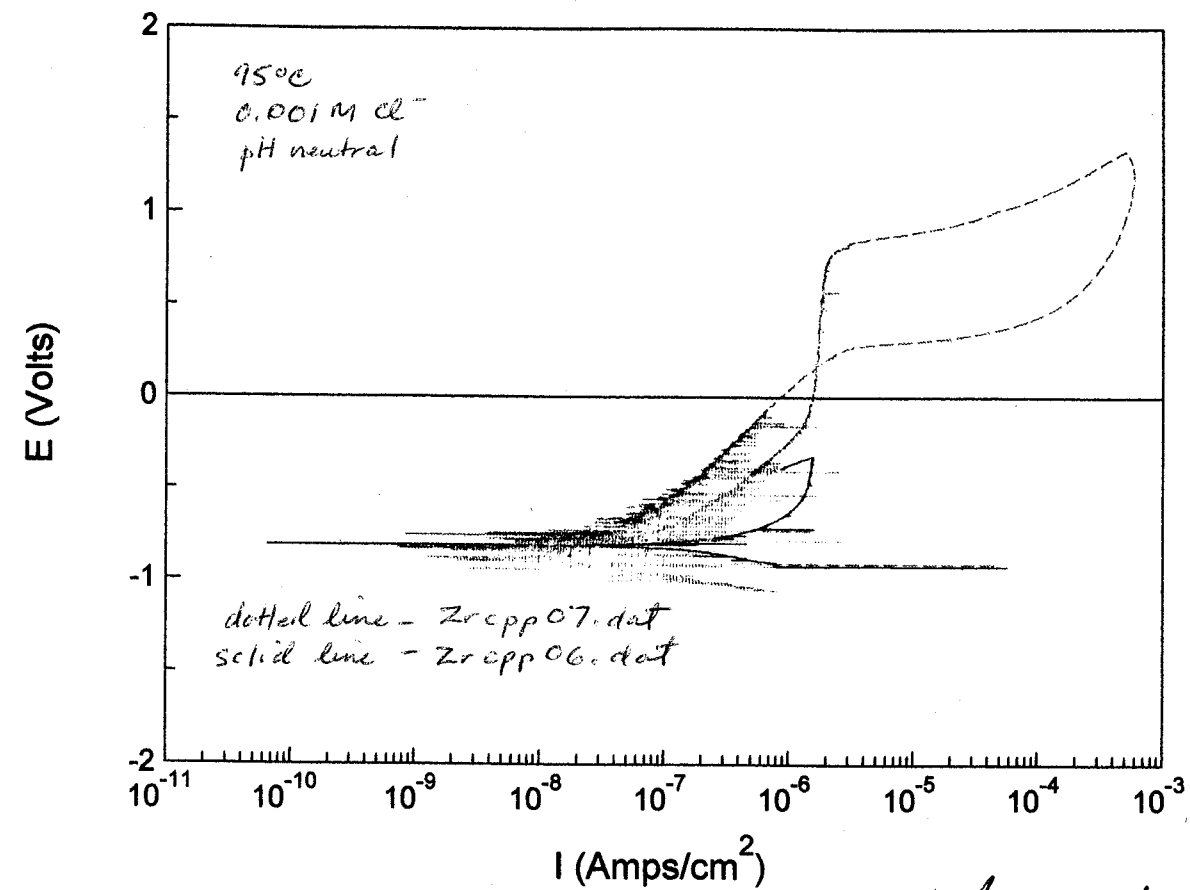
Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. 14

CA/Grum 5/14/99

Final pH = 10.438

Final specimen weight 29.88153g

To Page No. _____

Witnessed & Understood by me,

CA/Grum

Date

5/14/99

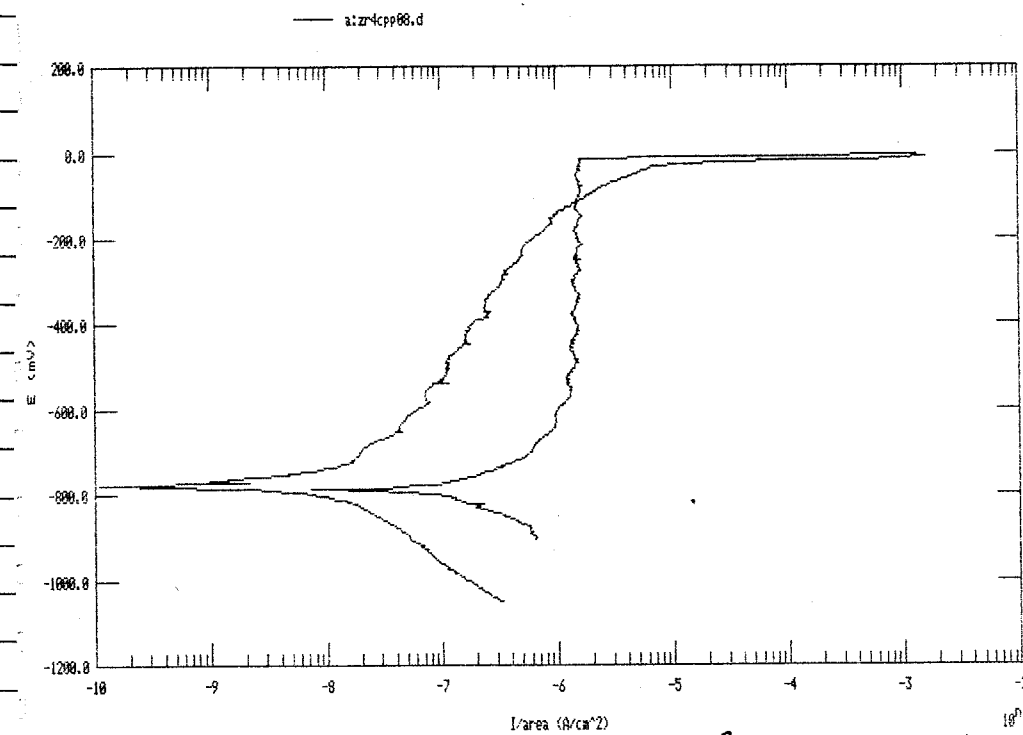
Invented by

Recorded by

Date

From Page No. 15

Model 352/252 Corrosion Analysis Software, v. 2.30
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-11-99 Time Run: 15:29:36 Pstat: VStat() Ver 2
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.499 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 640 TR NONE
 FL 1 S, 3Hz RT HIGH STABILITY REF 0.00000 User MK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.004
 Comment: Zr-4 4M Cl 95C pH 7.36 deaerated w/ N2 Crevice specimen



CA/Grum 5/14/99

Final pH = 8.498

Final specimen weight : 30.12945g

To Page No. _____

Witnessed & Understood by me,

CA/Grum

Date

5/14/99

Invented by

Recorded by

Date

From Page No. _____

Solatron Setup (p. 14)

Zr-4 crevice specimen CPP test

Polished to 600 grit cleaned in methanol

Weight: 30.17215 g

Solution

0.01 M Cl^- 0.58510 g # 98530285 ppm HCO_3^- 0.11871 g # 89778920 ppm SO_3^{2-} 10 ppm NO_3^- 2 ppm F^-

Standard Solution 5/99

pH: 8.197

T = 92 °C

 $E_{\text{corr OC}} = -880 \text{ mV vs SCE}$ $E_{\text{PE}} = -170 \text{ mV vs SCE}$

To Page No. 20

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Cafre

5/14/99

TITLE _____

From Page No. _____

EG&G VernaStat

Zr-4 crevice specimen CPP test

Polished to 600 grit cleaned in methanol

Weight: 30.22580 g

Solution

0.1 M Cl^- 5.84378 g # 98530285 ppm HCO_3^- 0.11872 g # 89778920 ppm SO_3^{2-} 10 ppm NO_3^- 2 ppm F^-

Standard Solution 5/99

pH: 7.962

T = 95 °C

 $E_{\text{corr OC}} = -940 \text{ mV vs SCE}$ $E_{\text{PE}} = -40 \text{ mV vs SCE}$

To Page No. 21

Witnessed & Understood by me,

Date

Invented by

Date

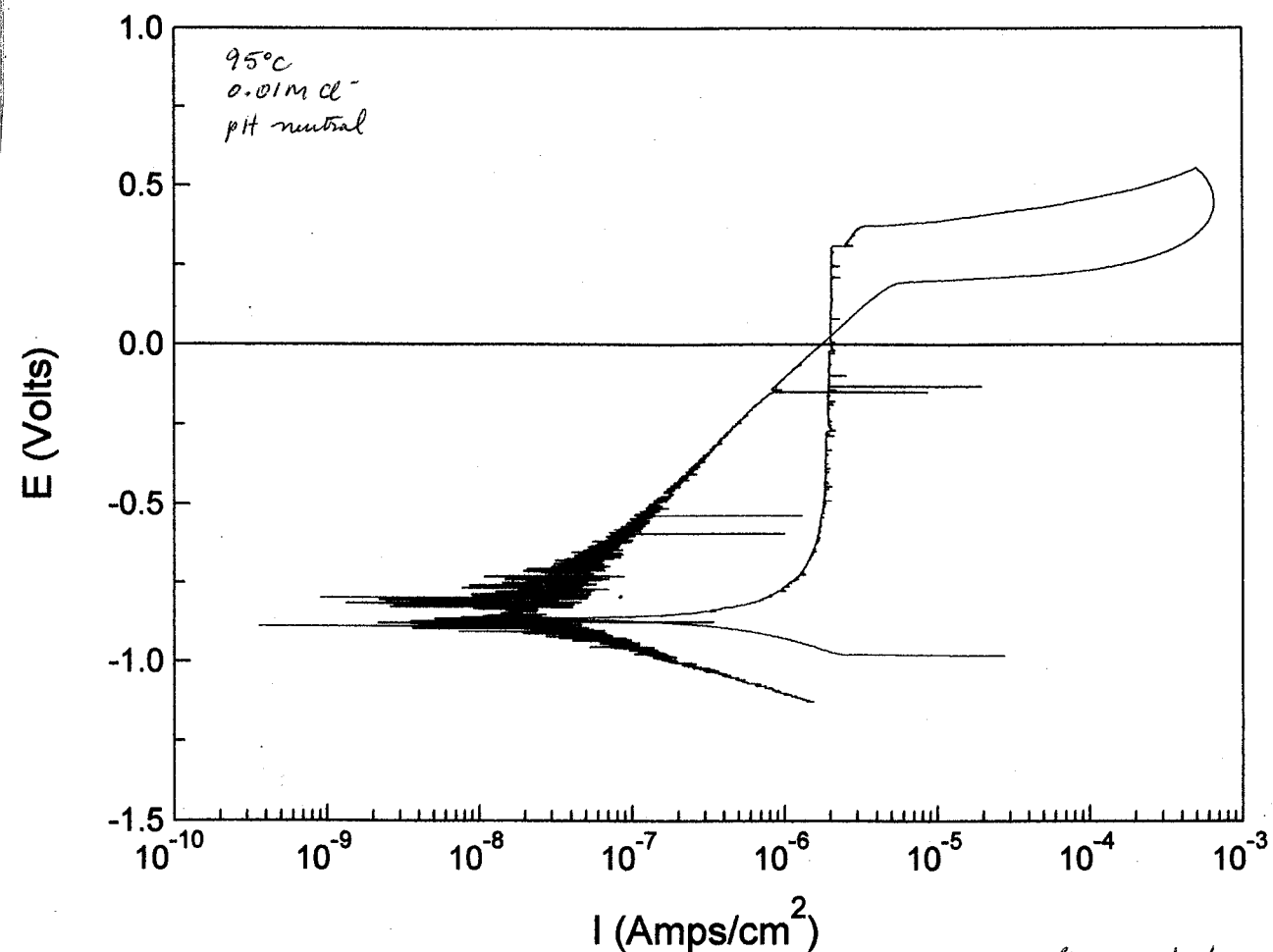
Recorded by

Cafre

5/14/99

From Page No. 18

file: Zr4cpp10.dat



final pH 10.016 final weight: 30.1602/g

To Page No. _____

Witnessed & Understood by me,

CA/Truene

Date

5/17/99

Invented by

Recorded by

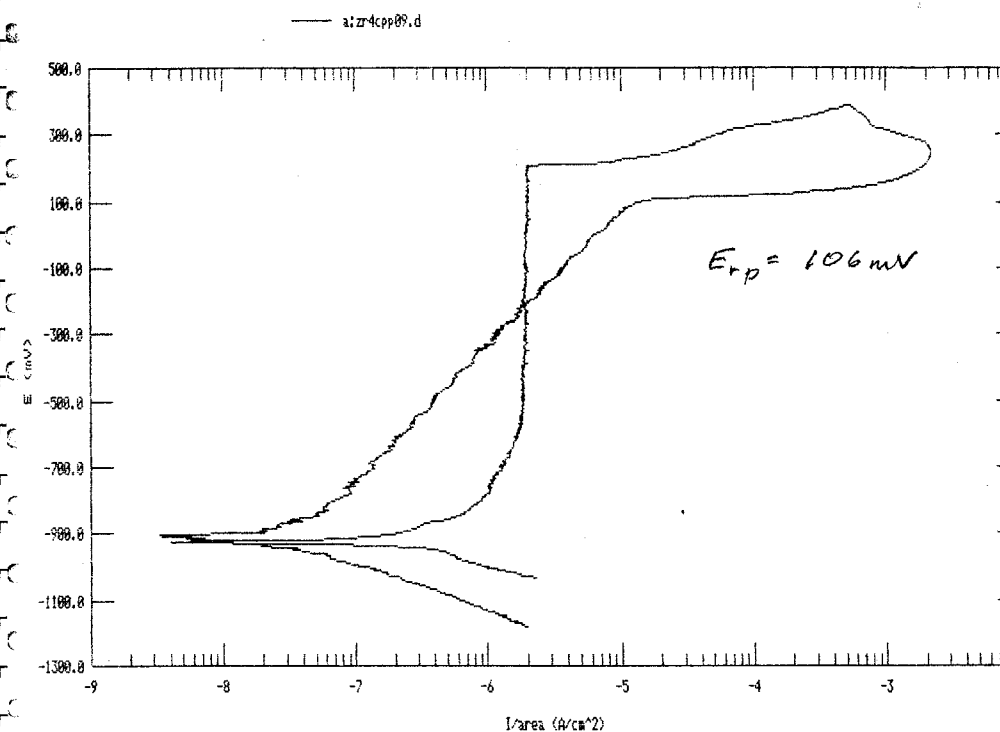
Date

From Page No. 19

Model 352/252 Corrosion Analysis Software, v. 2.38
CP CYCLIC POLARIZATION
CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.500 vs. R FP -0.250 vs. OC
SI 3.000E-03 SR 1.557E-04 ST 1.000E+01 CR AUTO MP 99% IR NONE
FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User NPK SOLID AR 1.830E+01 LS YES
IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.931

Comment: Zr-4 0.1M Cl 95C pH 7.6 deaerated w/N2 Device specimen

Filename: a:zr4cpp09.dat
File Status: NORMAL
Date Run: 05-12-99
Time Run: 10:46:38
Pstat: VStat() Ver 2



final pH = 9.387 final weight: 30.21173 g

Meeting with Gustavo, Sean Brossia & Darrell Dunn - in Gustavo's office.
To get a feel for open circuit potential: OPEN CIRCUIT POTENTIAL TEST,
1MCl, 95°C or 25°C (use 25°C initially) O₂ (air saturated) use
voltmeter & recorder, run 24 hrs then add H₂O₂ at 5mM see if
H₂O₂ has an effect on the potential, run additional 24 hrs add
Fe³⁺ as FeCl₃ in mM range 500 ppm or 1000 ppm.

To Page No. _____

Witnessed & Understood by me,

CA/Truene

Date

5/17/99

Invented by

Recorded by

Date

From Page No. _____

Solartron

2r-4 crevice specimen CPP test
polished 600 grit cleaned in methanol.

weight: 30.05607g

Solution:

0.01M Cl^- 0.58492g # 98530285ppm HCO_3^- 0.11690g # 89778920 ppm SO_3^{2-} 10 ppm NO_3^- 2 ppm F^-

} Standard Solution 5/99

pH = 8.176 T = 65°C S/N 0323004

 $E_{\text{corr OC}} = -0.6683 \text{ mV vs SCE}$ $E_{\text{Pt}} = 0.0801 \text{ V vs SCE}$

test began 6:12pm

To Page No. 26

Witnessed & Understood by me,

Date

5/17/99

Invented by

Date

Recorded by

Cafunum

TITLE _____

From Page No. _____

EG & G Versabstat

2r-4 crevice specimen CPP test
polished 600 grit cleaned in methanol

weight: 29.86520g

Solution:

1M Cl^- 58.44137g # 98530285 ppm HCO_3^- 0.11732g # 89778920 ppm SO_3^{2-} 10 ppm NO_3^- 2 ppm F^-

} Standard Solution 5/99

pH = 7.700 T = 65°C S/N 183302

 $E_{\text{corr OC}} = -792.0 \text{ mV vs SCE}$ $E_{\text{Pt}} = -137.2 \text{ mV vs SCE}$

test began: 6:18pm

To Page No. 27

Witnessed & Understood by me,

Date

5/17/99

Invented by

Date

Recorded by

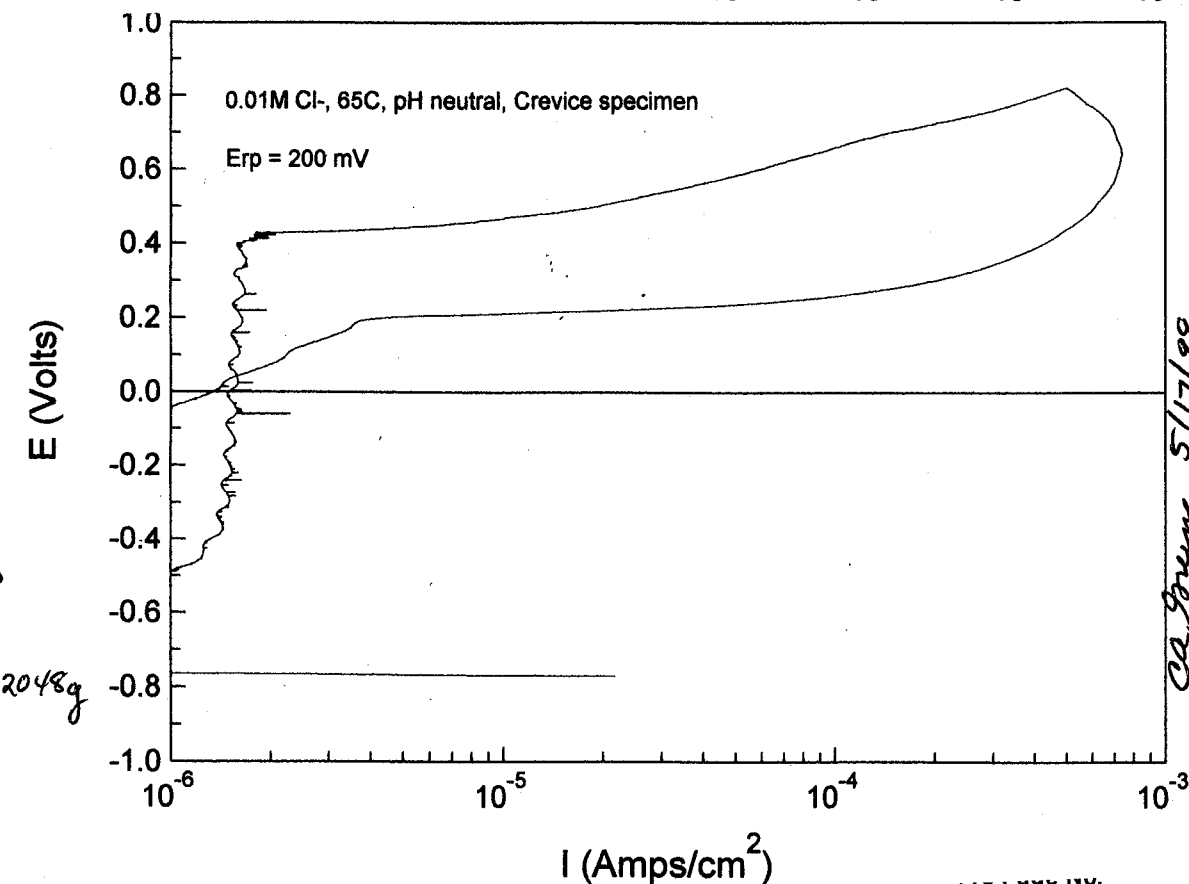
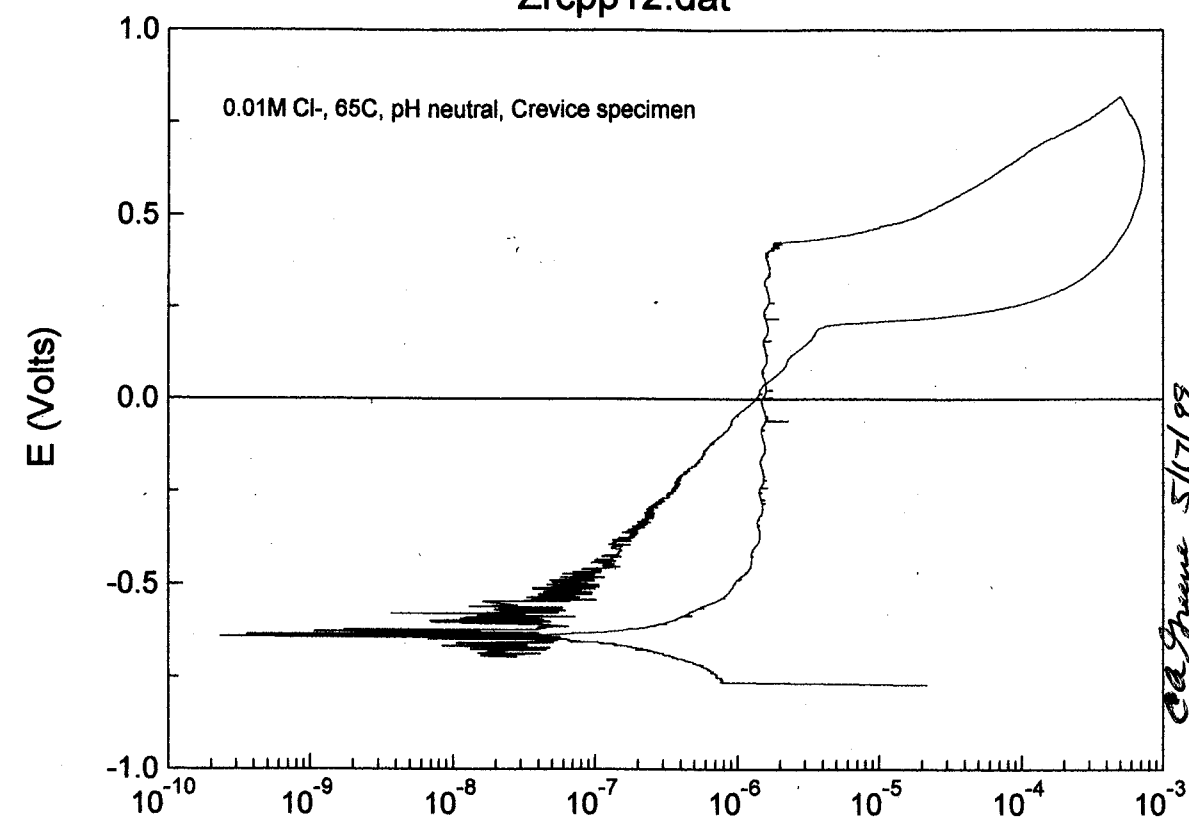
Cafunum

From Page No. _____	Solartron	25°C
Zr-4 crevice specimen CPP test		
polished 600 grit cleaned in methanol		
Solution:		
0.01M Cl^-	0.58430g	#985302
85 ppm HCO_3^-	0.11753g	#897789
20 ppm SO_3^{2-}	} Standard Solution 5/99	
10 ppm NO_3^-		
2 ppm F^-		
pH:	8.195	
Weight:	30.24167g	T = 21°C
E_{OC}	= -804 mV vs SCE	
E_{R}	= +197 mV vs SCE	
test start: 1:40 pm		
/		
To Page No. 30		
Witnessed & Understood by me, CA/Drum	Date 5/18/99	Invented by Recorded by

From Page No. _____	EG & G	25°C
Zr-4 crevice specimen CPP test		
polished 600 grit cleaned in methanol		
Solution:		
0.1M Cl^-	5.84462g ^{CO/Drum}	#985302
85 ppm HCO_3^-	0.11684g	#897789
20 ppm SO_3^{2-}	0.11723g ^{CO/Drum}	5/17/99
10 ppm NO_3^-	} Standard Solution 5/99	
2 ppm F^-		
pH:	8.002	
Weight:	30.00275g	T = 21°C
E_{OC}	= -682 mV vs SCE	
E_{R}	= +54 mV vs SCE	
test began 1:30 pm		
/		
To Page No. 31		
Witnessed & Understood by me, CA/Drum	Date 5/18/99	Invented by Recorded by

From Page No. 22

Zrcpp12.dat

pH_{final} = 9.733

Final Wt = 30.02048g

Witnessed & Understood by me,

CA Greene

Date

5/17/99

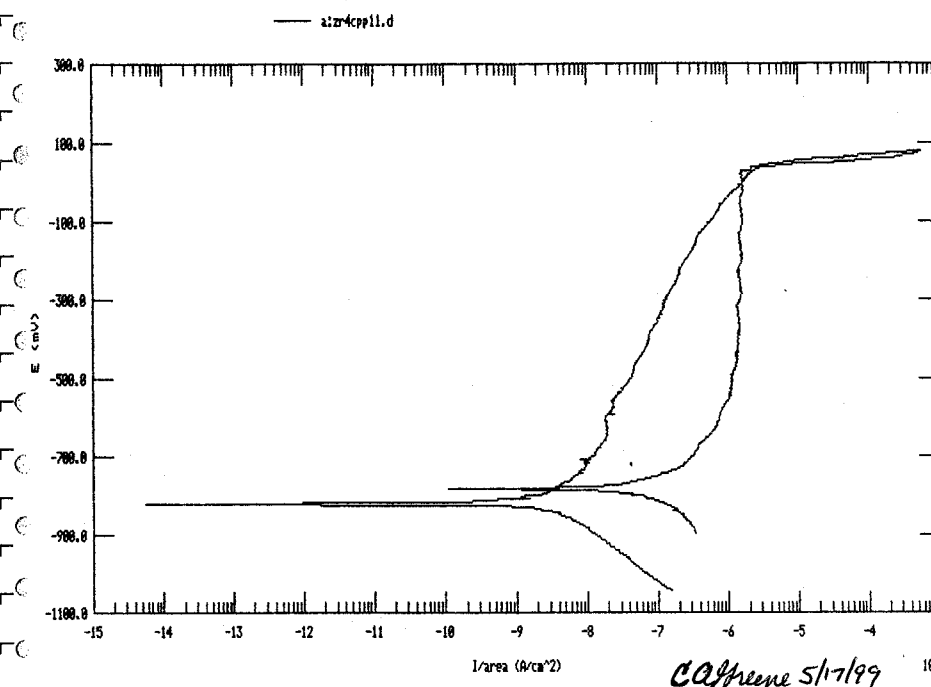
Invented by

Recorded by

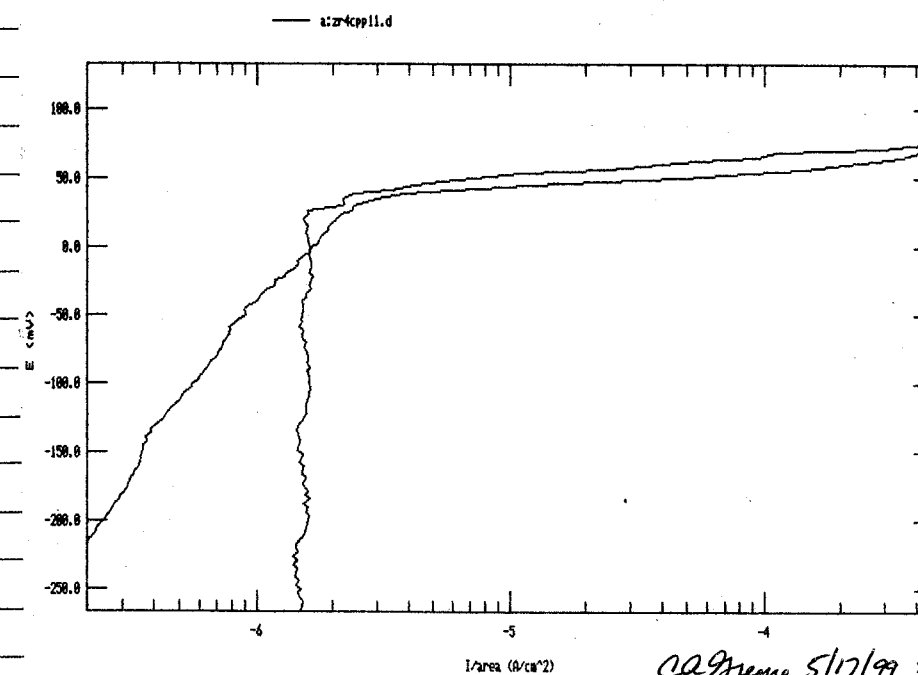
Date

From Page No. 23

Model 352/252 Corrosion Analysis Software, v. 2.38 File: a1zr4cpp11.dat Pstat: VStat() Ver 2
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-14-99 Time Run: 15:25:06
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.499 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO HP 690 IR NONE
 FL 1 5.3Hz RT HIGH STABILITY REF 0.00000 User MKK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EN 0.000E+00 DEN 0.000E+00 AU NO OC -0.795
 Comment: Zr-4 1M Cl⁻ 65C pH neutral deaerated w/12 Crevice specimen



Model 352/252 Corrosion Analysis Software, v. 2.38 File: a1zr4cpp11.dat Pstat: VStat() Ver 2
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-14-99 Time Run: 15:25:06
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.499 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO HP 690 IR NONE
 FL 1 5.3Hz RT HIGH STABILITY REF 0.00000 User MKK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EN 0.000E+00 DEN 0.000E+00 AU NO OC -0.795
 Comment: Zr-4 1M Cl⁻ 65C pH neutral deaerated w/12 Crevice specimen



Wt_{final} = 29.84438g
 pH_{final} = 8.97

Witnessed & Understood by me,

CA Greene

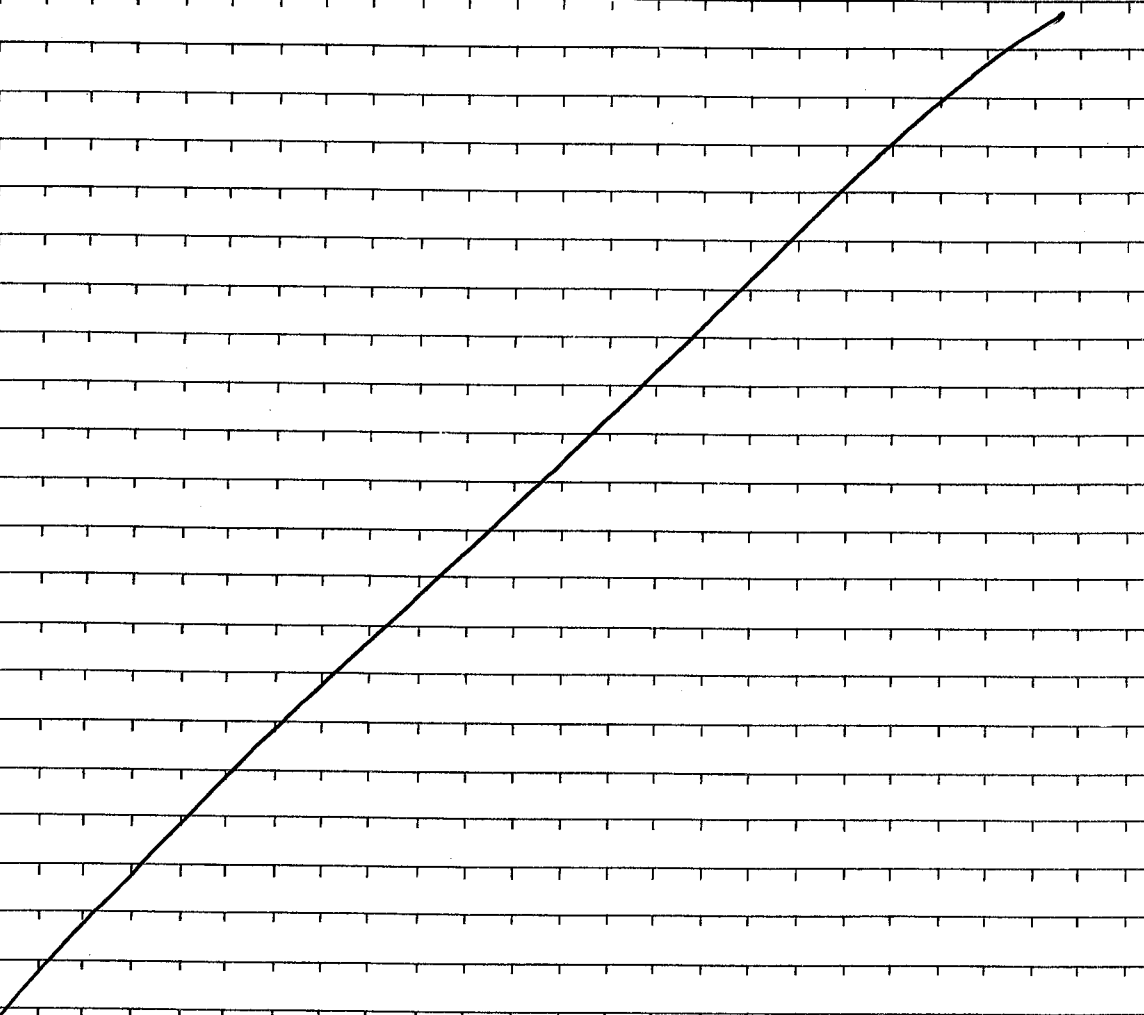
Date

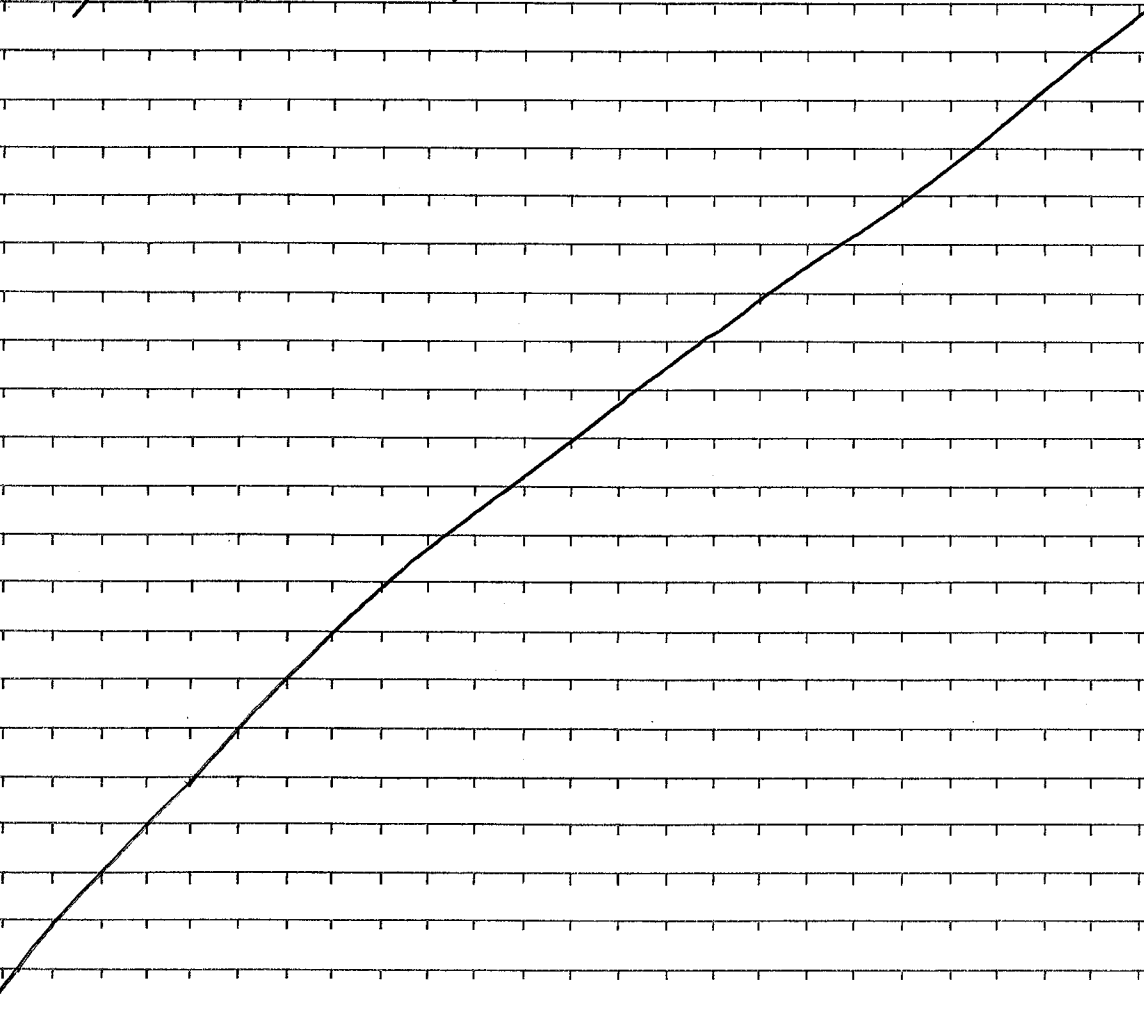
5/17/99

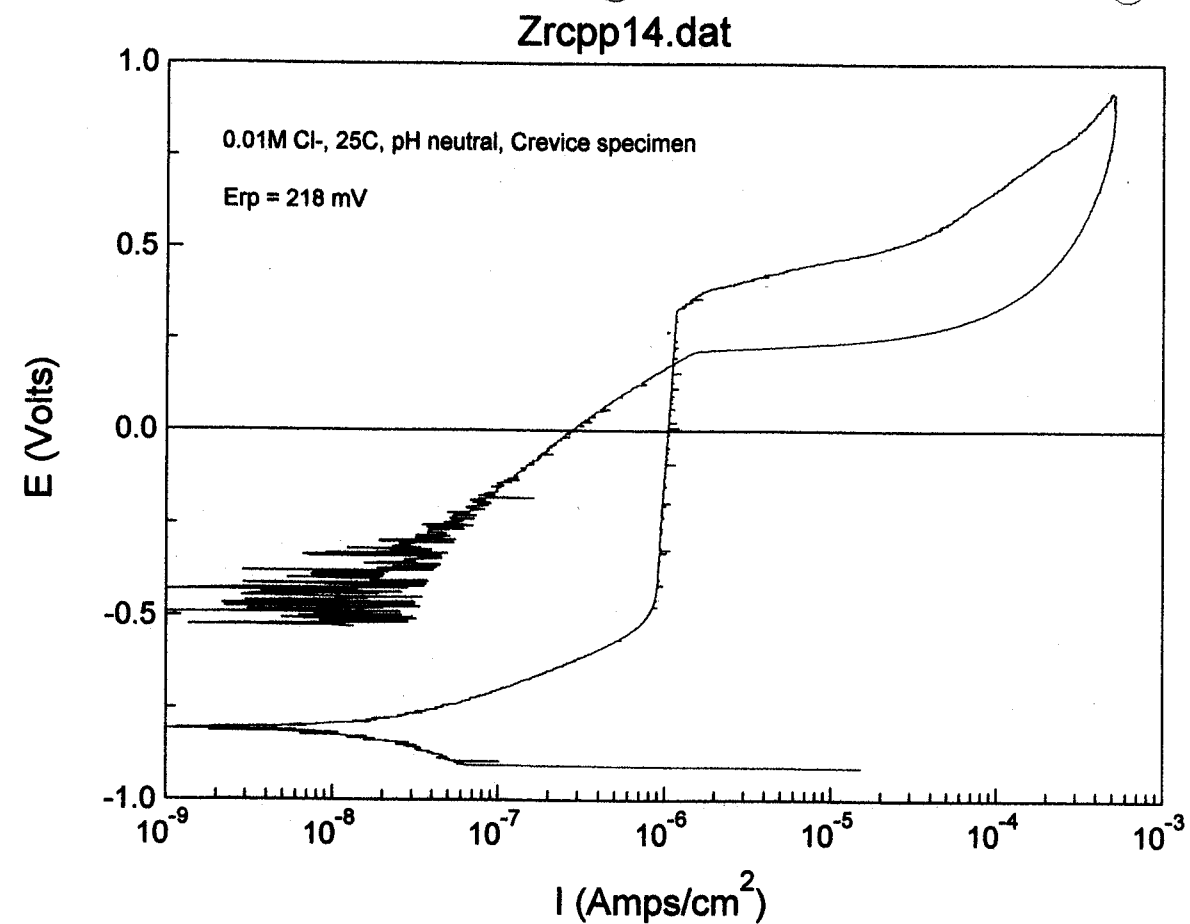
Invented by

Recorded by

Date

From Page No. _____	Solartron		
Zr-4 CPP test crevice specimen @ 65°C			
polished 600 grit cleaned in methanol			
Solution:	0.1 M Cl ⁻ <i>CAJme 5/18/99</i> 5.84406g # 985302 85 ppm HCO ₃ ⁻ 0.11748g # 897789 20 ppm SO ₃ ⁼ 10 ppm NO ₃ ⁻ } standard solutions 5/99 2 ppm F ⁻		
torque = 50 in-oz			
initial weight: 29.72995g		T = 65°C	
initial pH: 8.109			
E _{corr} OC vs SCE = -936 mV			
E _{ph} vs SCE = -17 mV			
			
To Page No. 32			
Witnessed & Understood by me,	Date	Invented by	Date
CAJme	5/18/99		
Recorded by			

From Page No. _____	EG&G		
Zr-4 CPP test crevice specimen @ 25°C			
polished 600 grit cleaned in methanol			
Solution	4 M Cl ⁻ 233.76030g # 985302 85 ppm HCO ₃ ⁻ 0.11784g # 897789 20 ppm SO ₃ ⁼ 10 ppm NO ₃ ⁻ } standard solution 5/99 2 ppm F ⁻		
torque = 50 in-oz			
initial weight: 29.99355g		58.44 g/M x 4 M 233.76 g 58.44/110 g 60.07998 g 60.41568 g + 54.82354 g 233.76030	
initial pH: 7.337			
E _{corr} OC = -716 mV vs SCE		T = 21°C	
E _{ph} = +116 mV vs SCE			
test start: 932 PM			
			
To Page No. 33			
Witnessed & Understood by me,	Date	Invented by	Date
CAJme	5/18/99		
Recorded by			

From Page No. 24

CA#me 5/18/99

Final pH: 9.372

Final weight: 30.20684g

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

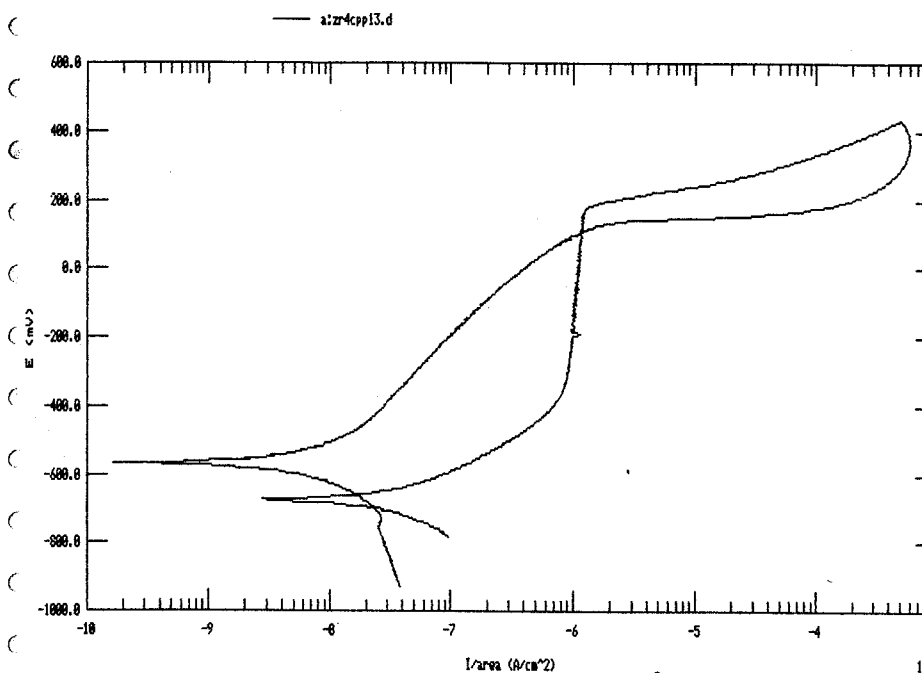
Recorded by

CA#me

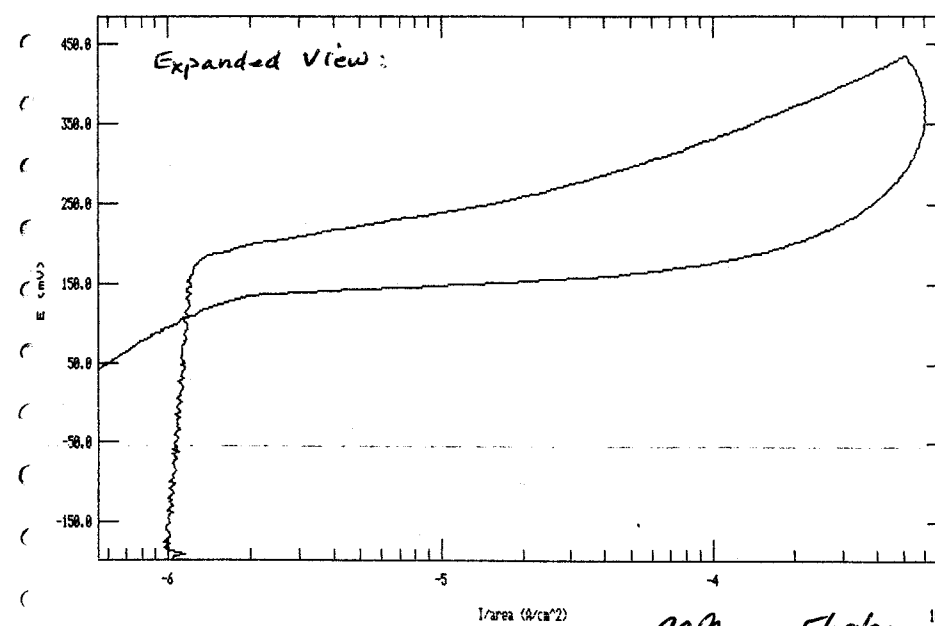
5/18/99

From Page No. 25

Model 352/252 Corrosion Analysis Software, v. 2.38
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-15-99 Time Run: 18:37:22 Pstat: VStat11 Ver 2
 CP PASS vs. R CT PASS IP -0.180 vs. OC ID 10 S VI -0.499 vs. R FP -0.258 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO MP 860 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User MK SOLID AR 1.839E+01 LS YES
 IT 9.150E-03 ITA 1.839E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.681
 Comment: 2-4 0.1M Cl⁻ 25C pH neutral desaturated w/12 Crevice specimen



CA#me 5/18/99



CA#me 5/18/99

Final pH: 9.062

Final weight: 29.97742g

Witnessed & Understood by me,

Date

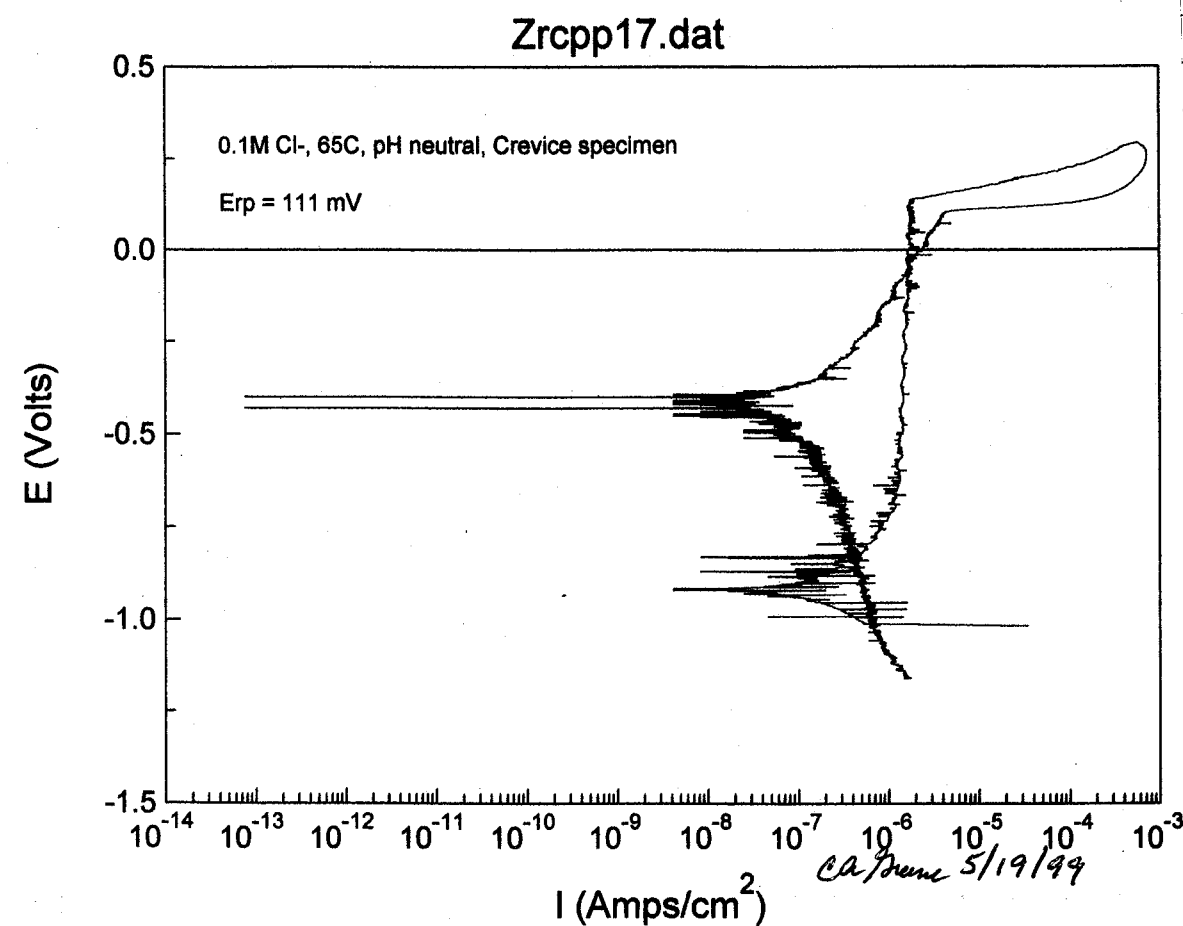
Invented by

Date

CA#me

5/18/99

Recorded by

From Page No. 28Final pH: 9.837Final wgt: 29.70766g

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

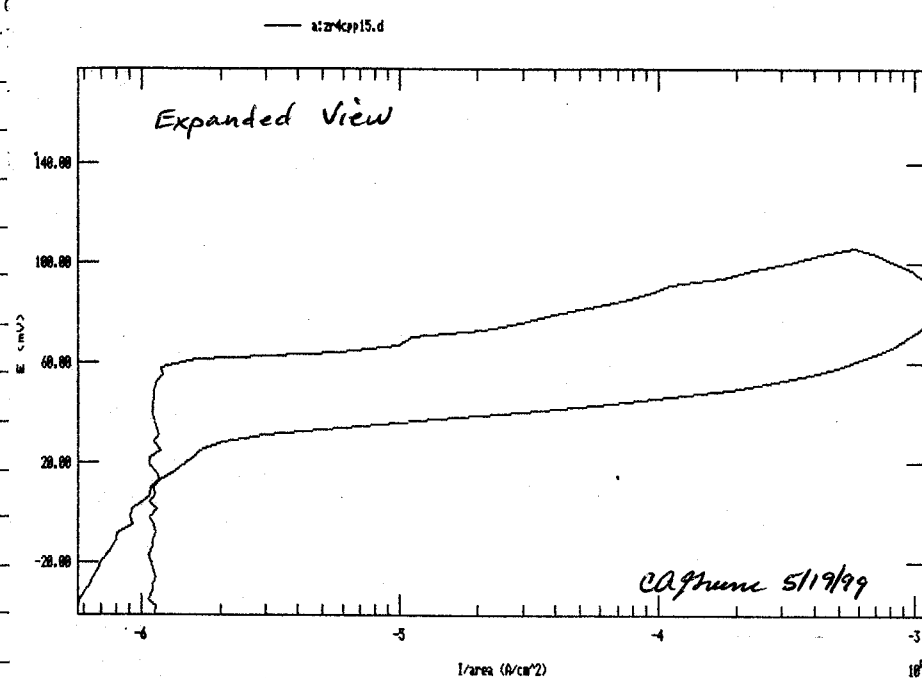
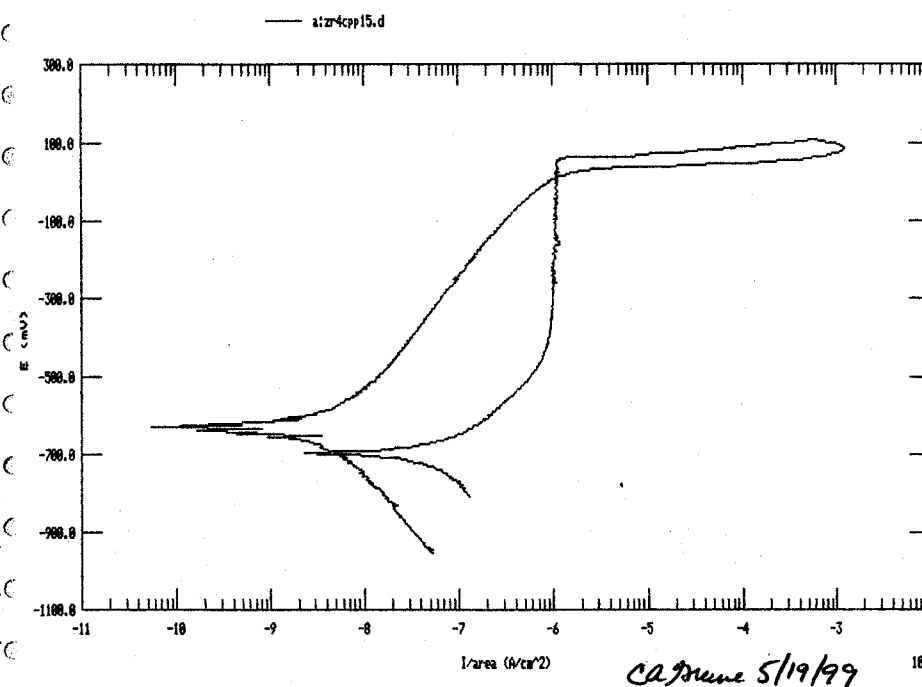
Recorded by

*CA/nume*5/19/99

From Page No. _____

4 M Cl⁻, 25°C Final pH: 8.347 Final wgt.: 29.99025g

Model 352/252 Corrosion Analysis Software, v. 2.30
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-15-99 Time Run: 10:39:04 Pstat: UStat() Ver 2
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.500 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 660 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User WRK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EN 0.000E+00 DEN 0.000E+00 AU NO OC -0.789
 Comment: 2-4 4M Cl 25C pH neutral deaerated w/2 Crevice specimen



To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

*CA/nume*5/19/99

From Page No. _____

- meeting with Dr. Bridha and Dr. Cragnolino: cajume 5/19/99
- I → Potentiostatic ~ 20 mV above E_{pp} at 1M Cl^- , 95°C (p.21)
- II → also run open circuit potential test at 1M Cl^- , 95°C (p.21)
- III → Finish temperature, chloride concentration tests:
28, 31 & 40 on page 10
- IV → Pick concentration and Temperature (0.1M, Cl^- , 95°C) and vary pH. This will be 2 tests pH 2 & pH 12.

EG&G

Zr-4 crucible specimen CPP test @ 65°C

polished, cleaned.

weight

SOLUTION: 4 M Cl^- 233.75958g # 985302 (233.76g)
 85 ppm HCO_3^- 0.11749g # 897789
 20 ppm SO_3^{2-} } 67.72676g
 10 ppm NO_3^- } Standard solution 5/99 65.52236
 2 ppm F^- } 69.66562
 + 30.84484
 233.75958g

pH : 7.448

initial Wgt: 29.88605g

(torque: 50 in. oz)

T = 65°C SIN 0323004

 $E_{corr OC} = -746$ mV vs SCE $E_{px} = +5$ mV vs SCE

Start time: 4:05 PM

To Page No. 36

Witnessed & Understood by me,

Date

5/19/99

Invented by

Recorded by

Date

TITLE _____

From Page No. _____

Solartron

Zr-4 crucible specimen CPP test @ 25°C
polished, cleaned

SOLUTION 0.001 M Cl^- 0.05846g # 985302
 85 ppm HCO_3^- 0.11730g # 897789
 20 ppm SO_3^{2-} }
 10 ppm NO_3^- } 5/99
 2 ppm F^- }

pH : 8.273

initial wgt: 29.95064g

T = 21°C

 $E_{corr OC} = -635$ mV vs. SCE $E_{px} = +284$ mV vs. SCE

Notes:

for pH 2 → 0.01 M HCl (12.1 M in bottle) } pH ~ 2
 0.09 M NaCl
 need to make 1 L of solution

pH 11 → 20 mM Na_2CO_3 pH 2 & pH 11 solutions in 0.1M Cl^- + simulated J-13 as per tests run up to this point.

To Page No. 37

Witnessed & Understood by me,

Date

5/19/99

Invented by

Recorded by

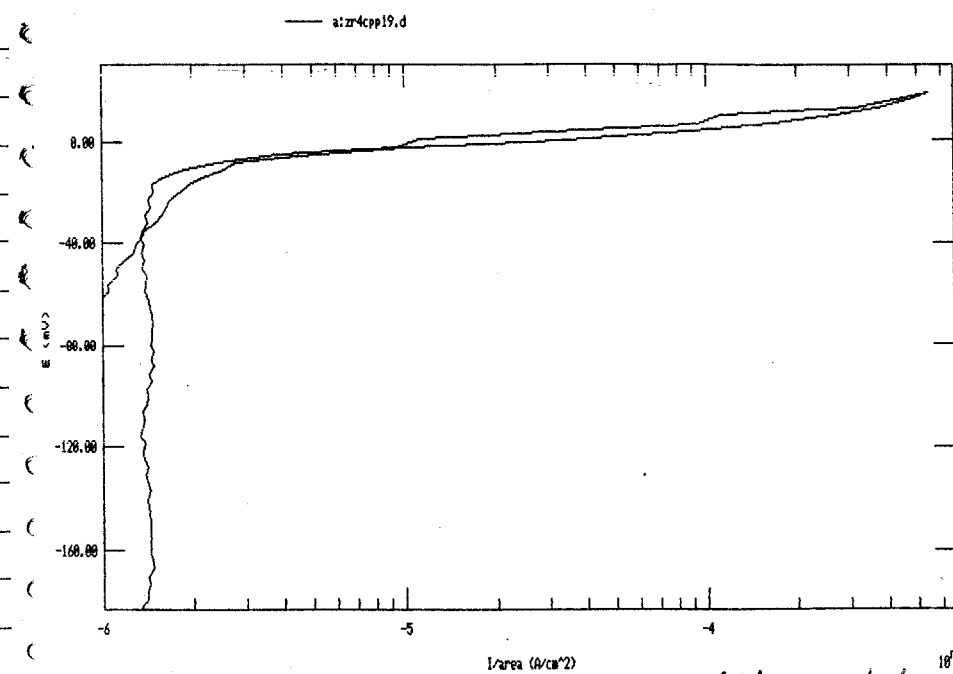
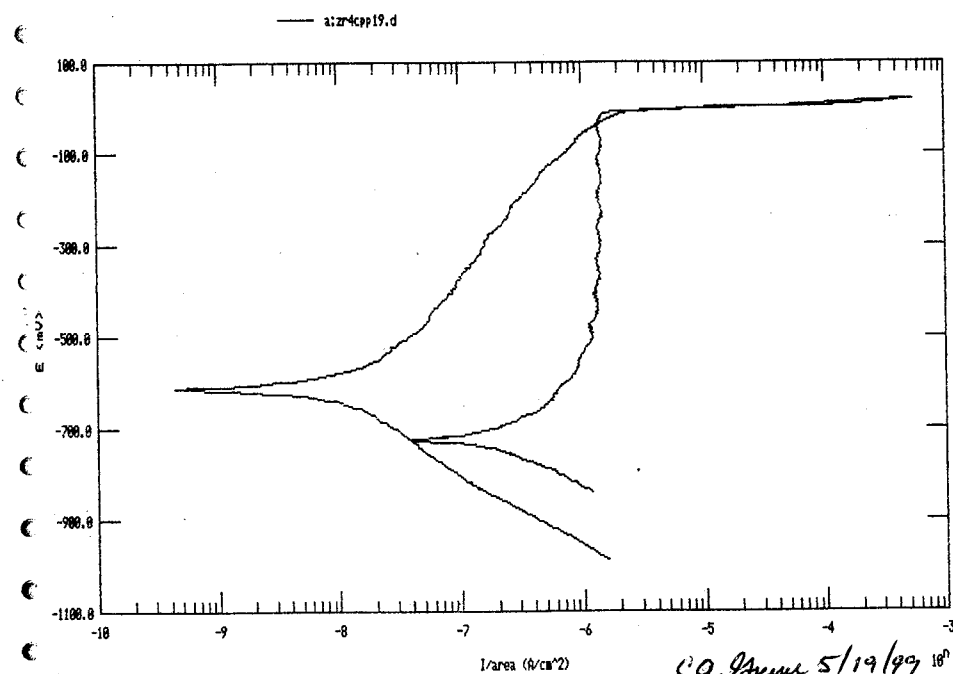
Date

From Page No. 34

Final pH: 8.708

Final weight: 29.89589g

Model 352/252 Corrosion Analysis Software, v. 2.38
 OP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-16-99 Time Run: 13:11:17
 CT PASS IP -0.100 vs. OC ID 10 S VI -0.500 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 622 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User WPK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EN 0.000E+00 DEN 0.000E+00 AU NO OC -0.739
 Comment: Zn-4 4M Cl 65C pH neutral deaerated w/12 Crevice specimen



Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

CAgnum

5/19/99

Page No. _____

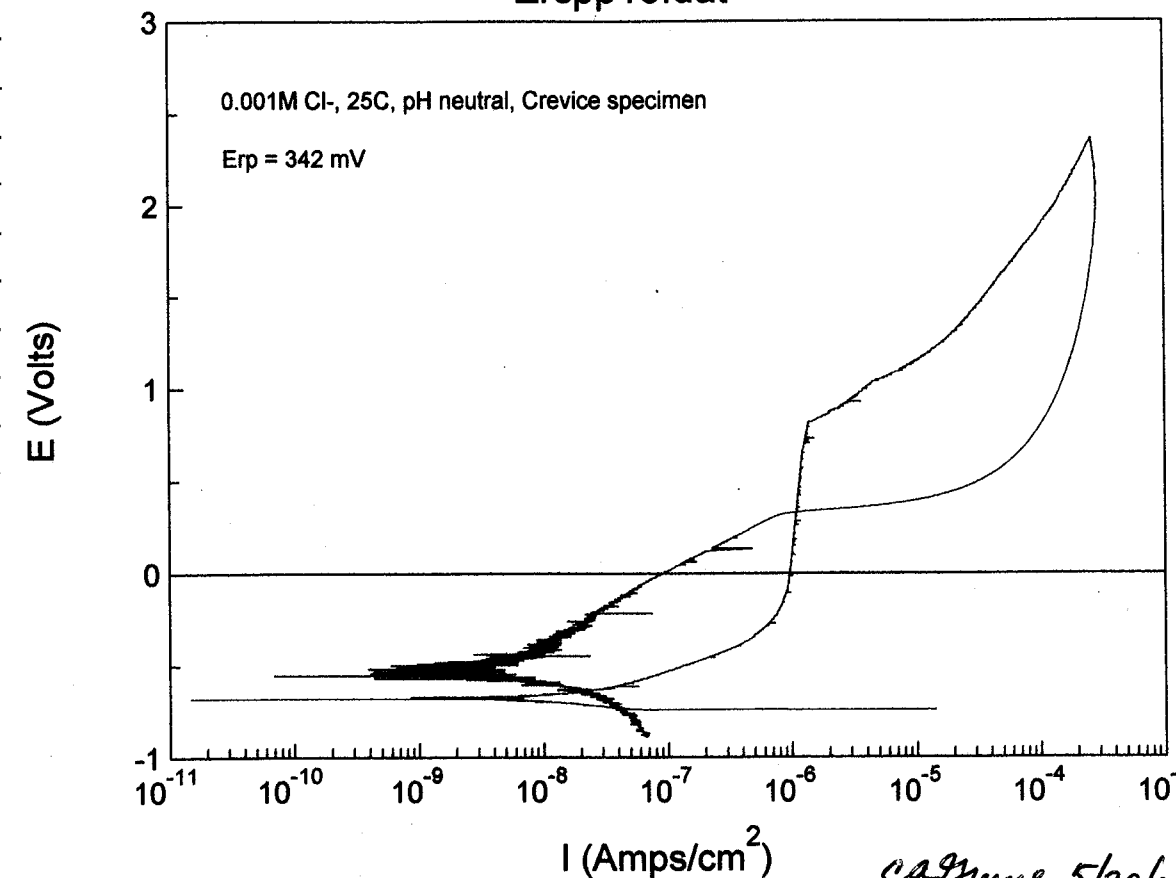
TITLE _____

From Page No. 35

Final pH: 9.652

Final wgt.: 29.93529g

Zrcpp18.dat



Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

CAgnum 5/20/99

5/20/99

To Page No. _____

From Page No. _____

EG&G

Zr-4 crevice specimen CPP test @ 95°C pH ~ 2

polished 600 grit, cleaned in methanol

Weight

SOLUTION: 0.1 M Cl^- 5.84751 g # 98651985 ppm HCO_3^- 0.11802 g # 89778920 ppm SO_4^{2-} } 5/9910 ppm NO_3^- }2 ppm F^- }

.01 M HCl 0.85 mL # 971828

Ca/gnum 5/19/99 pH .09 M NaCl 5.25682 g # 986519

initial weight: 29.79888 g pH: 2.088

T = 95°C -491 Ca/gnum 5/20/99

 $E_{\text{corr OC}} = -495 \text{ mV vs SCE}$ $E_{\text{pt}} = +558 \text{ mV vs SCE}$

final pH = 2.097

final spec. wt = 29.82489

.09 M NaCl = $58.44 \times .09 = 5.25960 \text{ g}$

.01 M HCl from 12.1 M → use 0.85 mL in 1 L

Test Start 2:23 pm

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Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

TITLE _____

From Page No. _____

Solartron

Zr-4 CPP test crevice specimen @ 65°C

polish 600 grit, clean in methanol

Weight

SOLUTION 0.001 M Cl^- 0.05841 g # 98530285 ppm HCO_3^- 0.11683 g # 89778920 ppm SO_4^{2-} } 5/99 Standard Solution10 ppm NO_3^- }2 ppm F^- }

pH: 8.258

initial weight: 29.75424 g

T = 65°C

 $E_{\text{corr OC}} = -782 \text{ mV vs SCE}$ $E_{\text{pt}} = +31 \text{ mV vs SCE}$

Test Start: 12:15 pm

final pH = 10.227

final wt = 29.67934 g

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Witnessed & Understood by me,

Date

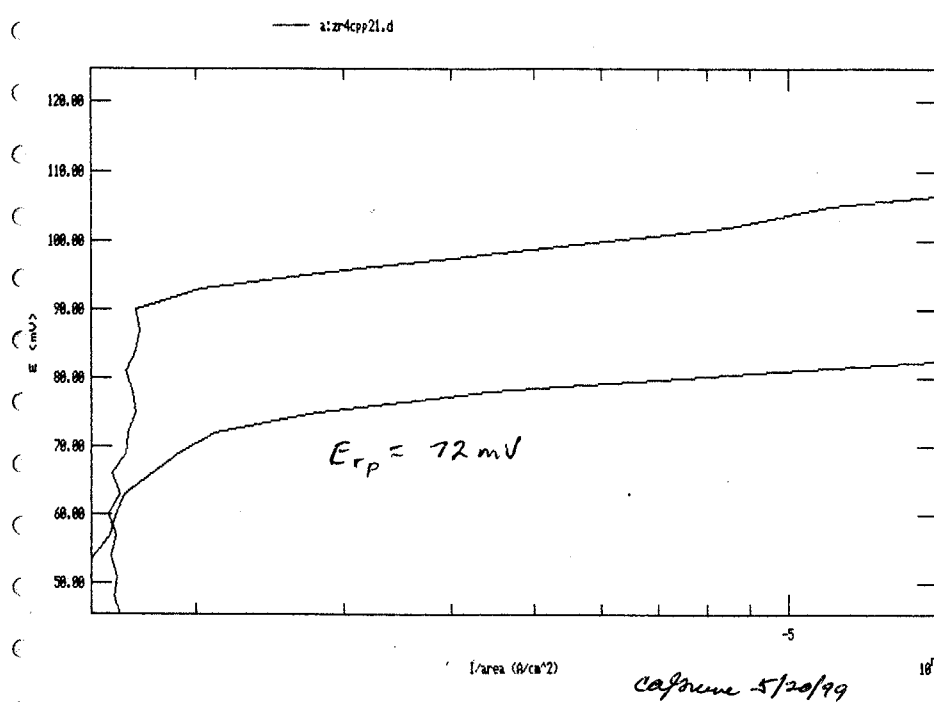
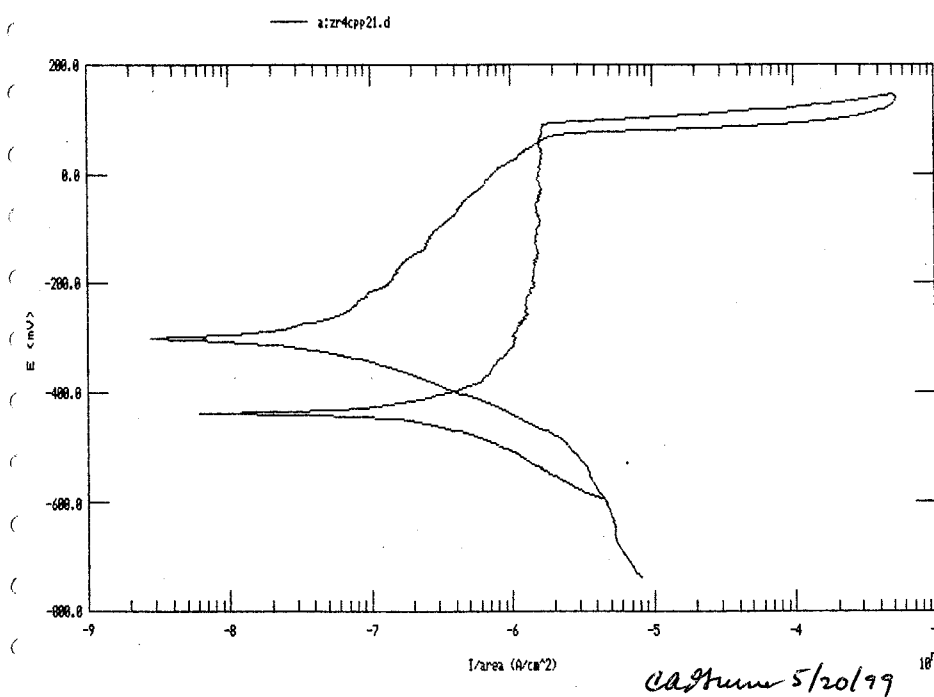
Invented by

Date

Recorded by

From Page No. 38

Model 352/252 Corrosion Analysis Software, v. 2.38
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-17-99 Time Run: 11:38:16 Pstat: VStat() Ver 2
 CP PASS vs. R CT PASS IP -0.100 vs. OC ID 10 S VI -0.501 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO HP 542 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User MK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -0.491
 Comment: Zr-4 0.1M Cl 95C pH 2.1 deaerated w/NO2 crevice specimen



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Witnessed & Understood by me,

Date

5/20/99

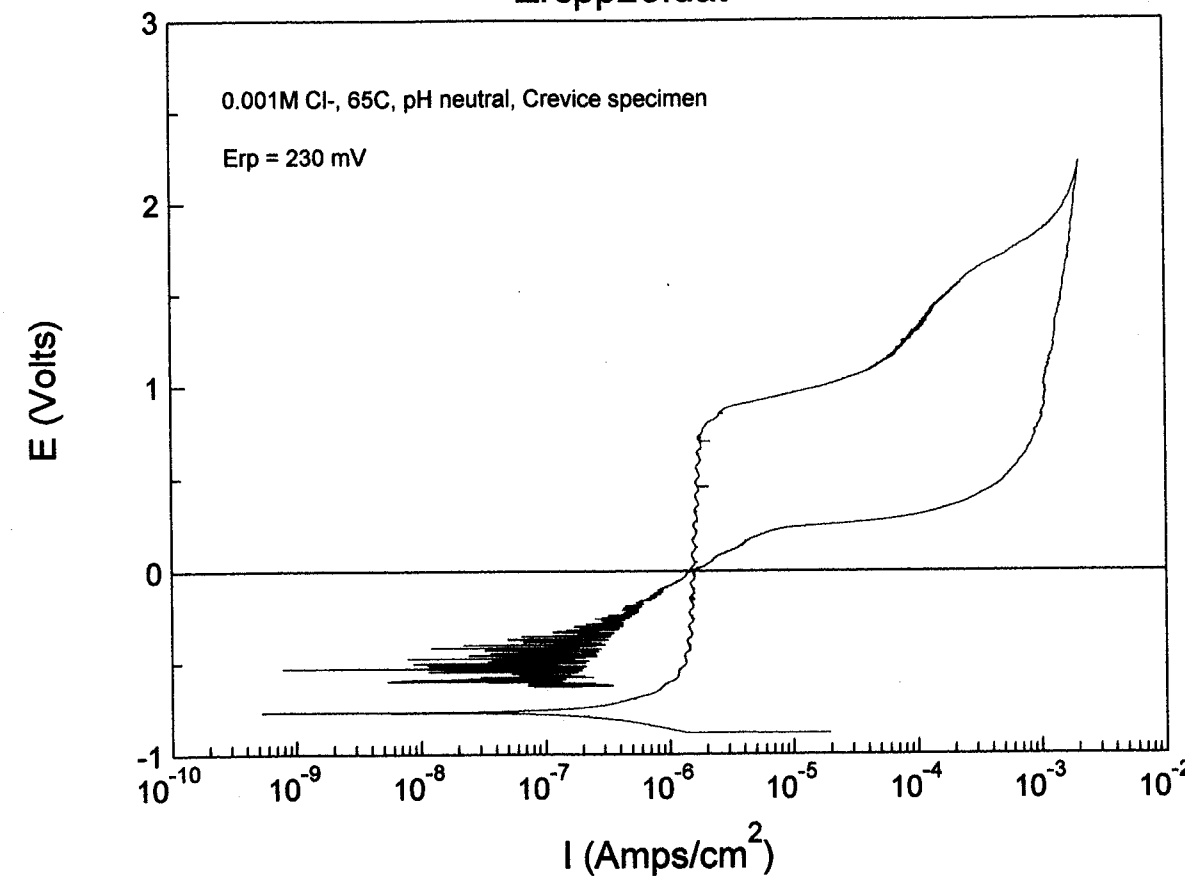
Invented by

Recorded by

Date

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Zrcpp20.dat



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Date

5/20/99

Invented by

Recorded by

Date

From Page No. _____

EG & G

Zr-4 crevice specimen CPP test @ 95°C pH ~ 11

polished 600 grit cleaned in methanol

weigh

SOLUTION 0.1 M Cl⁻ 5.84428 g # 98651985 ppm HCO₃⁻ 0.11818 g # 89778920 ppm SO₃⁼ } 5/9910 ppm NO₃⁻ }2 ppm F⁻ }Add 20 mM Na₂CO₃ 2.1153 g # 960685 S/Bron
5/21/99

Specimen = 29.8965 g

pH = 10.778 (initial)

T = 94 °C S/N 0323004 5/21/99

E_{open} OC = -1012 mVE_{PT} = -728 mV

S/Bron 5/21/99

final weight = 29.89197 g

final pH = 10.650

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Witnessed & Understood by me,

Date

Invented by

Date

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TITLE _____

From Page No. _____

Potentiostatic & open circuit potential tests -

1 M Cl⁻ (+ J-13), 95°COpen Circuit Potential Test on Solartron Potentiostat
material Zr-4 crevice specimen 600 grit, cleaned in methanol

SOLUTION: (2L)

1 M Cl⁻ 116.87827 g # 986519 ← 65.1959285 ppm HCO₃⁻ 0.23445 g # 897789 + 51.6823520 ppm NO₃⁻ SO₃⁼ } 40 mL 116.8782710 ppm NO₃⁻ } 5/99 20 mL2 ppm F⁻ } 4 mL

Specimen 29.69224 g

pH 7.770

T = 95 °C S/N H98-182

Reference electrode S/N 5144349

Torque wrench S/N 314047 50 in.oz

Aerated w/ compressed air

* Note Time when peroxide and ferric chloride
additions were made.

for peroxide at 5 mM in 1.75 L of solution in cell:

34.03 g/mol

5 mM / 1.75 L = 0.00875 mol

0.29776 g H₂O₂0.3 g H₂O₂ + 0.7 g H₂O = 1 g solution (30 wt% in bottle)0.3 g | 1 mL H₂O₂ + 0.7 g H₂O | 1 mL = 0.913 mL/g OR 1.095 g/mL0.29776 g H₂O₂ | 1 g soln | 1 mL = 0.906 mL (30 wt% H₂O₂)

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Witnessed & Understood by me,

Date

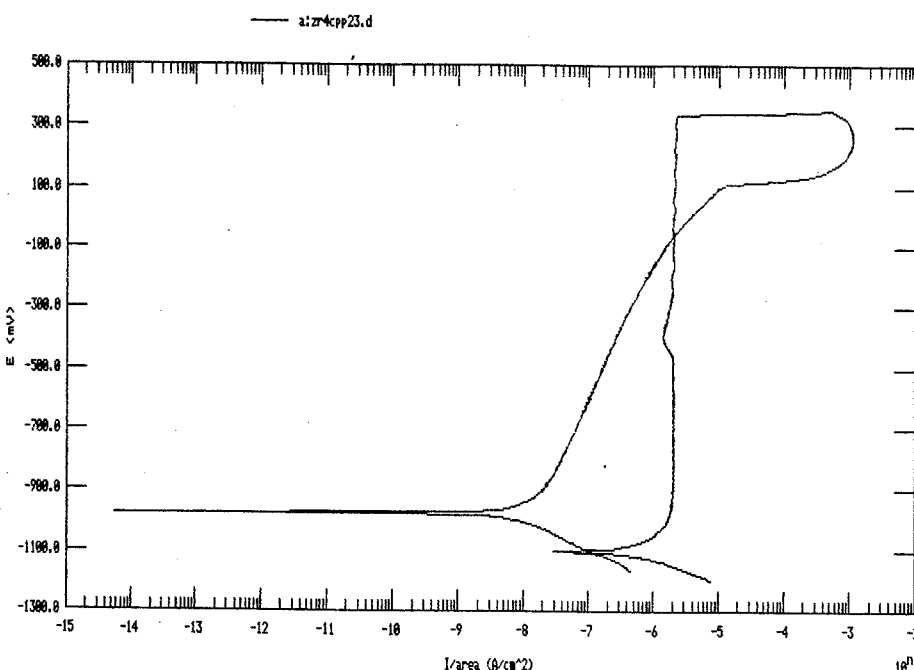
Invented by

Date

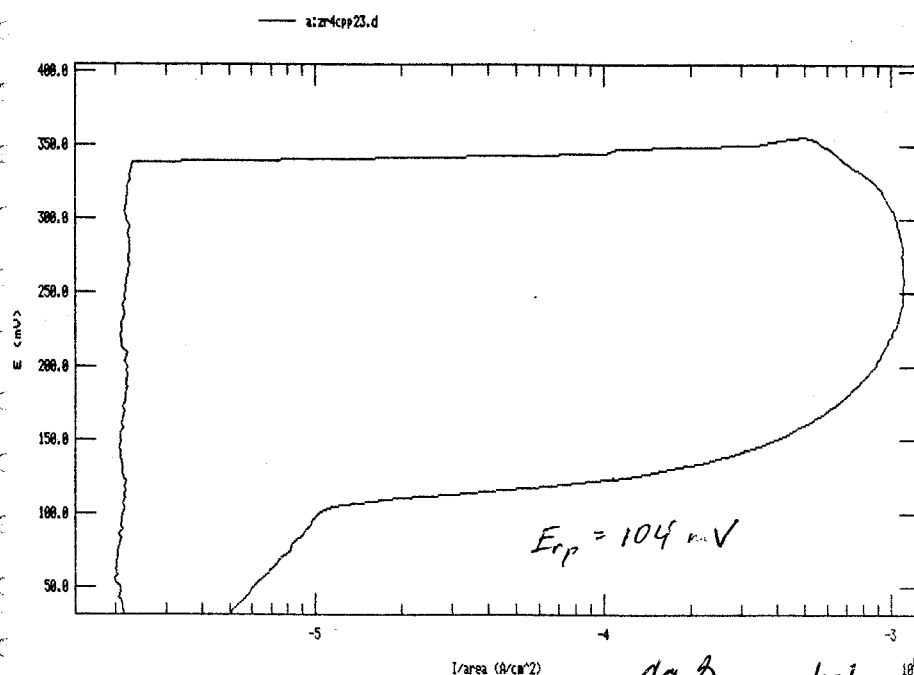
Recorded by

From Page No. 42

Model 352/252 Corrosion Analysis Software, v. 2.30
 CP CYCLIC POLARIZATION File Status: NORMAL Date Run: 05-18-99 Time Run: 04:28:43 Pstat: VStat[1] Ver 2
 CP PASS vs. R CT PASS IP -0.180 vs. OC ID 10 S VI -0.499 vs. R FP -0.250 vs. OC
 SI 3.000E-03 SR 1.667E-04 ST 1.000E+01 CR AUTO NP 1826 IR NONE
 FL 1.5.3Hz RT HIGH STABILITY REF 0.00000 User MRK SOLID AR 1.830E+01 LS YES
 IT 9.150E-03 ITA 1.830E+01 EM 0.000E+00 DEN 0.000E+00 AU NO OC -1.098
 Comment: Zr-4 0.1M Cl⁻ 95C pH 10.8 deaerated w/ N₂ Device specimen



ca/gnum 5/23/99

 $E_{rp} = 104 \text{ mV}$

ca/gnum 5/23/99

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Recorded by

From Page No. _____

EG & G
Potentiostatic Test- 1M Cl⁻ + J-13, 95°C

material Zr-4 crevice specimen, 600 grit, cleaned in methanol

SOLUTION (2L):

59.47393g } → 1 M Cl⁻ 116.87865g # 986519
 + 57.40472g } 85ppm HCO₃⁻ 0.11708g # 897789
 ✓ 20ppm SO₄²⁻
 ✓ 10ppm NO₃⁻ } 5/99
 ✓ 2ppm F⁻

pH: 7.621

initial weight: 29.49273g

 $E_{rr} \sim 35 \text{ mV vs. SCE (see p. 13) (1M Cl}^- + \text{J13 @ 95}^\circ\text{C)}$

Thermometer S/N C96-616 (11-12-99)

deaerated w/ N₂

initial potential = 55 mV

 $E_{corr, OC} = -843 \text{ mV vs. SCE}$ $E_{pk} = -136 \text{ mV vs. SCE}$ $T = 95^\circ\text{C}$ E_{rp} is more accurately about 28 mV∴ this test is ~ 28 mV above E_{rp}

file: a:\zr4pst01.dat ca/gnum 5/27/99

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Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

From Page No. 43

Solatron Open circuit Potential test

add peroxide #902183A (L+ #)

time: 321410.3 sec (point # 5311)

vol: 0.9 mL

SBR 5/25/99

> add peroxide - 0.9 mL #902183A

time: 405783 s

point: 6705

SBR 5/26/99

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Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

CA Green

5/26/99

TITLE _____

From Page No. _____

Zr-4 CPP Test Results - Effect of Temperature

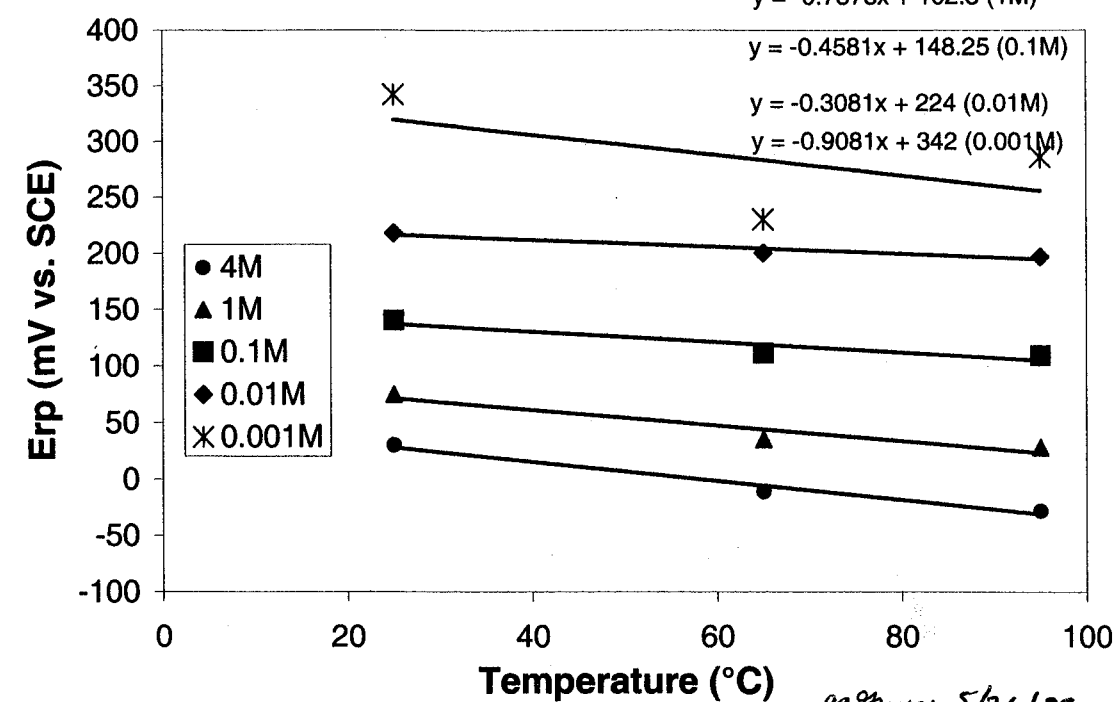
$$y = -0.8392x + 48.75 \text{ (4M)}$$

$$y = -0.7378x + 102.5 \text{ (1M)}$$

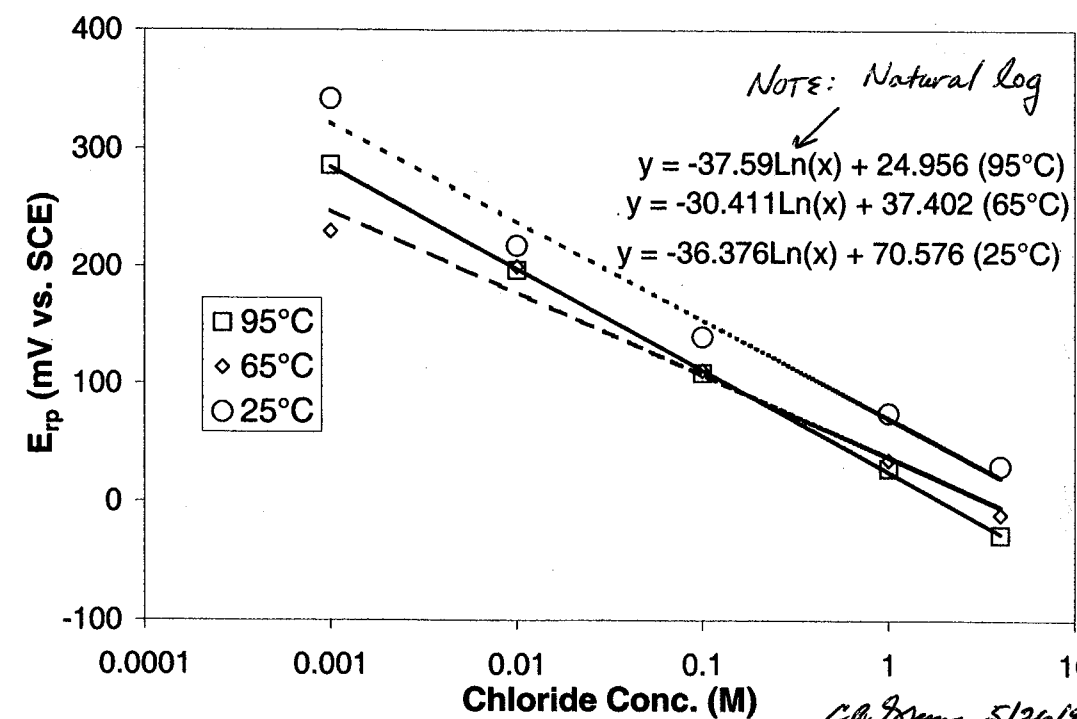
$$y = -0.4581x + 148.25 \text{ (0.1M)}$$

$$y = -0.3081x + 224 \text{ (0.01M)}$$

$$y = -0.9081x + 342 \text{ (0.001M)}$$



Zr-4 CPP Tests Results - Effect of Chloride Concentration



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Date

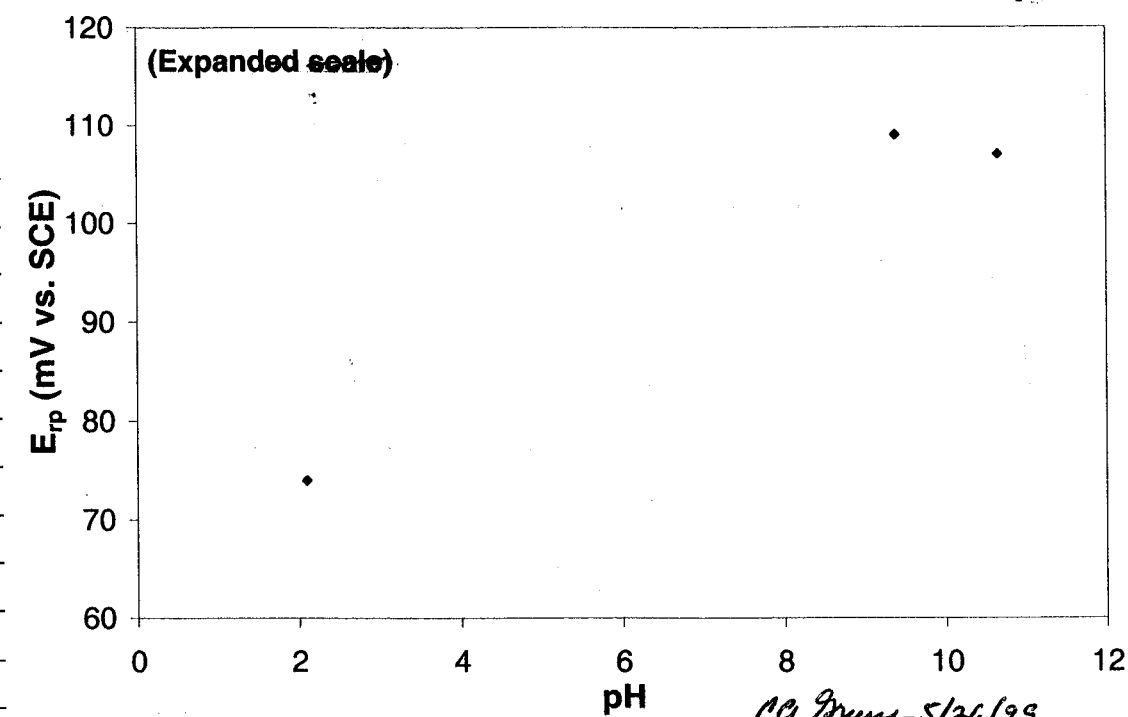
Recorded by

CA Green

5/26/99

From Page No. _____

Zr-4 CPP Tests Results - Effect of pH (0.1M, 95°C)



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Witnessed & Understood by me,

Date

5/26/99

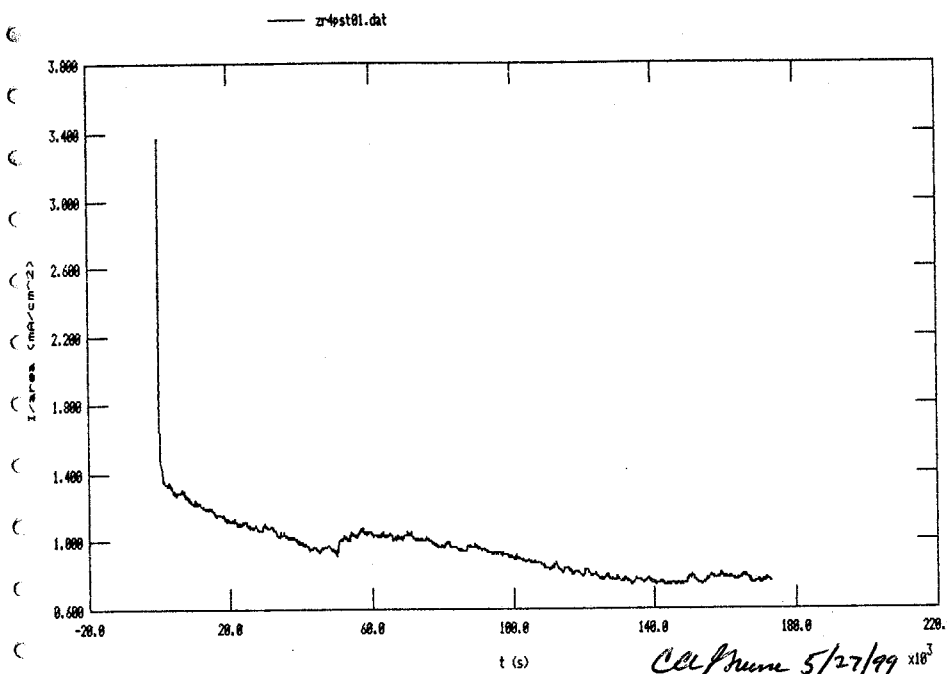
Invented by

Recorded by

Date

From Page No. 45

Model 332/252 Corrosion Analysis Software, v. 2.38
 PS POTENTIOSTATIC File Status: NORMAL Date Run: 05-20-99 Time Run: 13:48:32
 CP PASS vs. R CT PASS IP 0.055 vs. R ID 10 S TP 6.000E+01 T1 1.728E+05
 CR AUTO NP 2898 SO Pass IR NONE FL NONE RT HIGH STABILITY
 REF 0.00000 User WKK SOLID AR 1.830E+01 LS NO EM 0.000E+00 DEN 0.000E+00 AU NO
 OC -0.849
 Comment: Zr-4 IM Cl 95C pH 7.62 deaerated w/42 Crevice specimen



final pH = 7.905
 final wt = 28.9824 g

SB 5/28/99

→ extensive attack on exposed areas - crevice former protected; some corrosion products still attached after ultrasonic cleaning in MeOH.

- SB 5/28/99

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Witnessed & Understood by me,

Date

5/28/99

Invented by

Recorded by

Date

From Page No. 46

Zr-4 OCP Test - Experiment re-started Zr4ocp01b.dat

→ 10 mM FeCl_3 added Lot # 9915492.841 g FeCl_3 (anhydrous)

point #156

time ~ 9550 s

SBR 5/28/99

42 SBR 5/28/99
E mer to ~ 50 mV on addition

→ final file name = Zr4ocp01c.dat (1a & 1b merged)

final wt = 29.6883 g

final pH = 1.560

⇒ no attack under crevice former. Some corrosion observed though. Similar to previous tests.

SBR
6/2/99

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Witnessed & Understood by me,

Date

5/28/99

Invented by

Date

Recorded by

SBR

TITLE _____

From Page No. _____

Solution Zr4CAP24.DAT

Zr-4 crevice specimen CPP @ 25°C pH

polished 600 grit cleaned in methanol

weight =

Solution 1M NaCl → 58.44 g/Lt # 986519

Specimen weight = 29.61215 g

pH = 6.702

T = 25°C

Final weight = 29.59489 g

Final pH = 6.712

JL

6-15-99

No attack under crevice feet. Some local corrosion observed in multiple spots on flats and on one corner. Also fine of corrosion at vapor line. 6-15-99 JL

To Page No. _____

Witnessed & Understood by me,

Date

6/15/99

Invented by

Date

Recorded by

JL

From Page No. _____

Solution

Zr4CPP25.0AT

Zr4 crevice specimen CPP @ 25°C

Polished to 600 grit, cleaned in methanol

weight =

Solution = 10 mM NaCl → .5844g/Lt # 986519

Specimen weight = 29.31956g

PH = 6.520

T = 25°C

Final wt = 29.31357g

Final PH = 7.561

6-16-99

No effect under crevice feet. Some corrosion staining in multiple areas on flats and edges. Some located on cylindrical shaft in solution area.

To Page No. _____

Witnessed & Understood by me,

Date

6/16/99

Invented by

Recorded by

Date

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Solution

Zr4CPP26.0AT

Zr4 crevice specimen @ 25°C

polished to 600 grit + ultrasonically cleaned in methanol the heat treated @ 200°C for 8 days

Solution = 1M NaCl → 58.44g/Lt # 986519

Specimen weight = 29.47823g

PH = 6.440

T = 25°C

Final wt = 29.47746g

Final PH = 6.110

6-23-99

One small pit area under edge of foot, other small attack areas occurred randomly on flats, at on corner + two at vapor/water line.

6-23-99

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Witnessed & Understood by me,

Date

6-23-99

6-23-99

Invented by

Recorded by

Date

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Zr4 CPP27.DAT

Zr4 crevice specimen CPP@25°C

Polished to 600 grit + ultrasonically cleaned in methanol heat treated
for 9 days @ 200°CSolution = 10 mM \rightarrow NaCl \rightarrow .5844g/Lt #986519

Specimen weight = 29.81502g

pH = 5.730

T = 25°C

Final wt = 29.80194g

Final pH = 6.760

6-24-99

Some mild crevice pitting attack. Random pits found on flats and
edges 6/24/99

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Date

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Date

Recorded by

6-24-99

TITLE _____

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Zr4 CPP28.DAT

Zr4 crevice specimen CPP@25°C

Polished to 600 grit + ultrasonically cleaned in methanol. Heat treated
for 56 days @ 200°CSolution = 10 mM \rightarrow NaCl \rightarrow .5844g/Lt #985302

Specimen wt = 30.02438g

pH = 5.880

T = 25°C

Final wt = 30.02201g

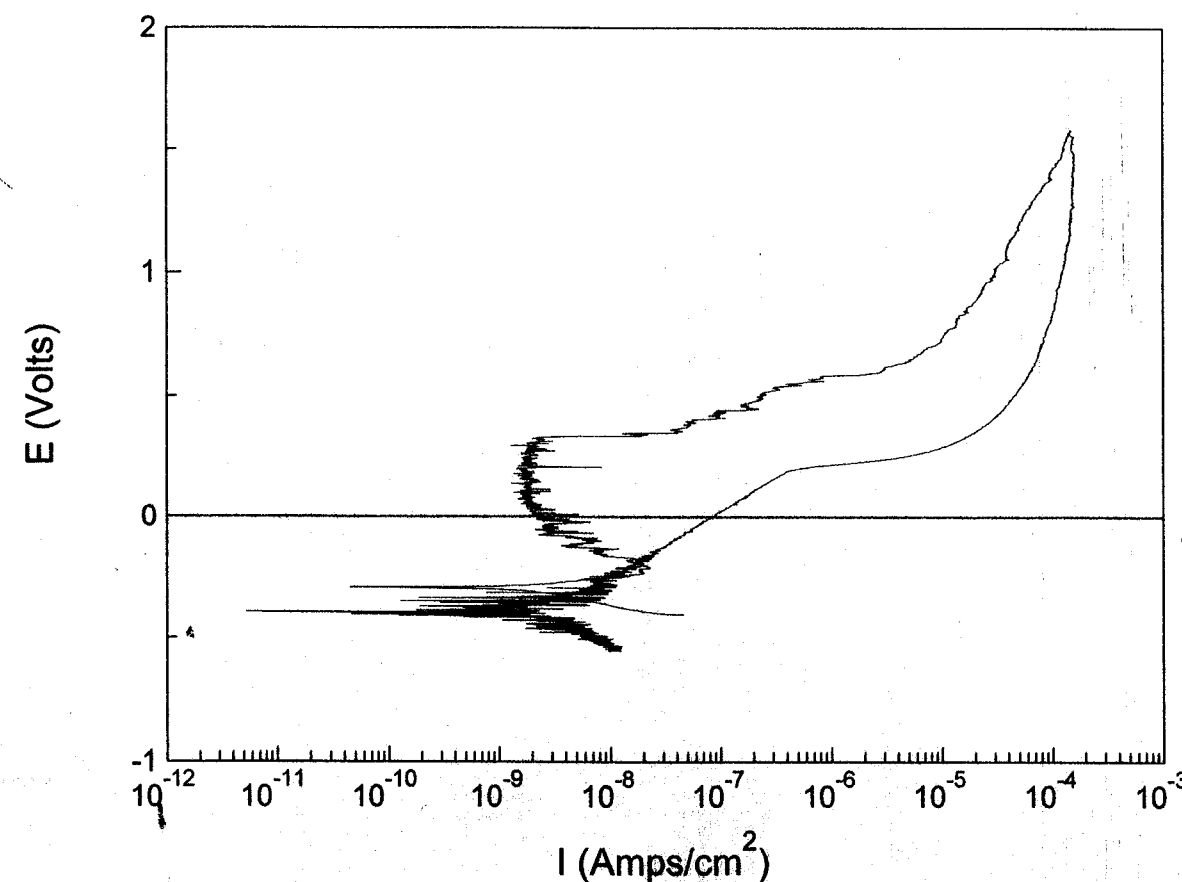
Final pH = 7.218

9-2-99

9/2/99

Some pitting noted on specimen but not under crevice area.

9-1-99



Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

9-1-99

From Page No. _____

Zr4 CPP29.DAT

Zr4 crevice specimen CPP25°C

Polished to 600 grit + ultrasonic cells cleaned in methanol heat treated
for 57 days @ 200°CSolution = 1M \rightarrow NaCl \rightarrow 58.44g/lit #985302

Specimen wt = 29.9171g

pH = 6.434

T = 25°C

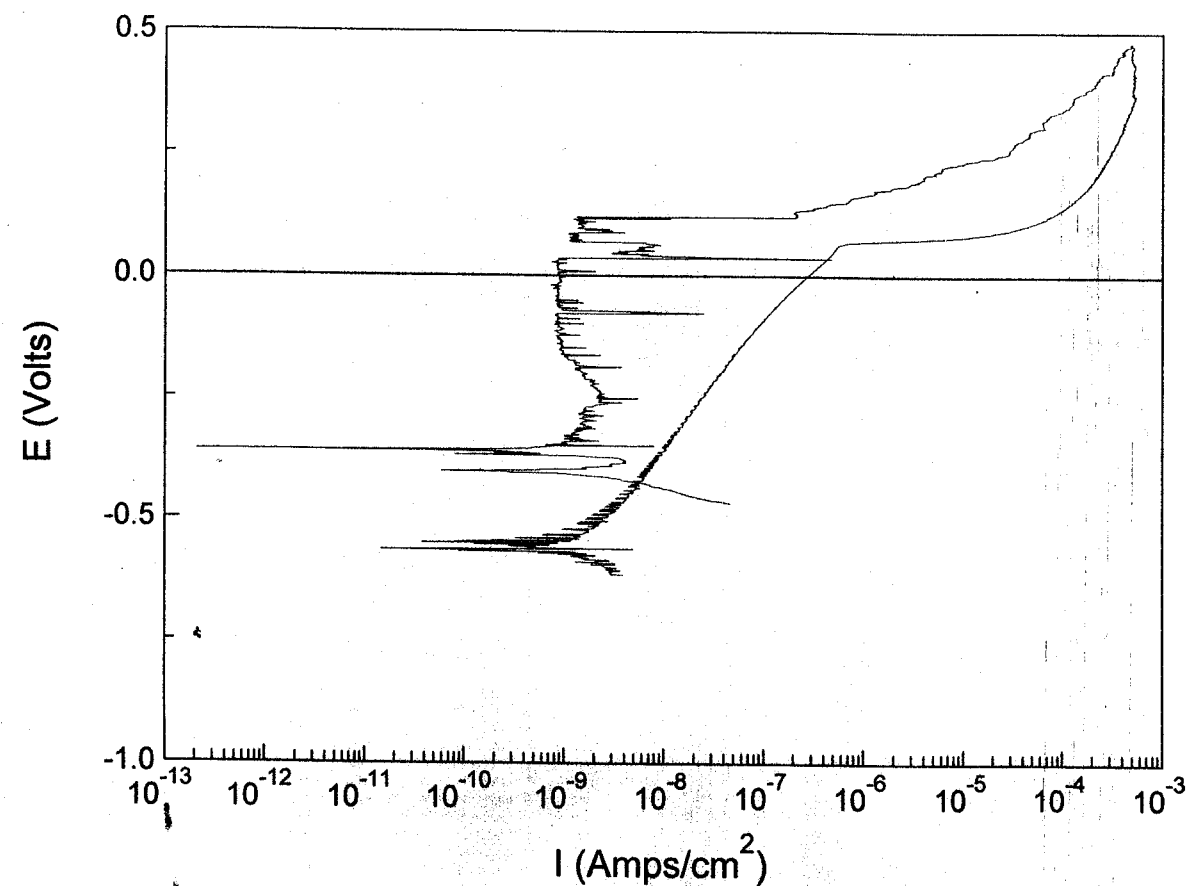
Final wt = 29.91494g

Final pH = 5.938

6-2-99

Some pitting noted but not under crevice foot area

9-2-99



Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

9-2-99

TITLE _____

From Page No. _____

Zr4 CPP30.DAT

Zr4 crevice specimen CPP25°C

Polished to 600 grit + ultrasonic cells cleaned in methanol, heat treated
for 63 daysSolution = 1M \rightarrow NaCl \rightarrow 58.44g/lit #985302

Specimen wt = 30.01630g

pH = 5.760

T = 25°C

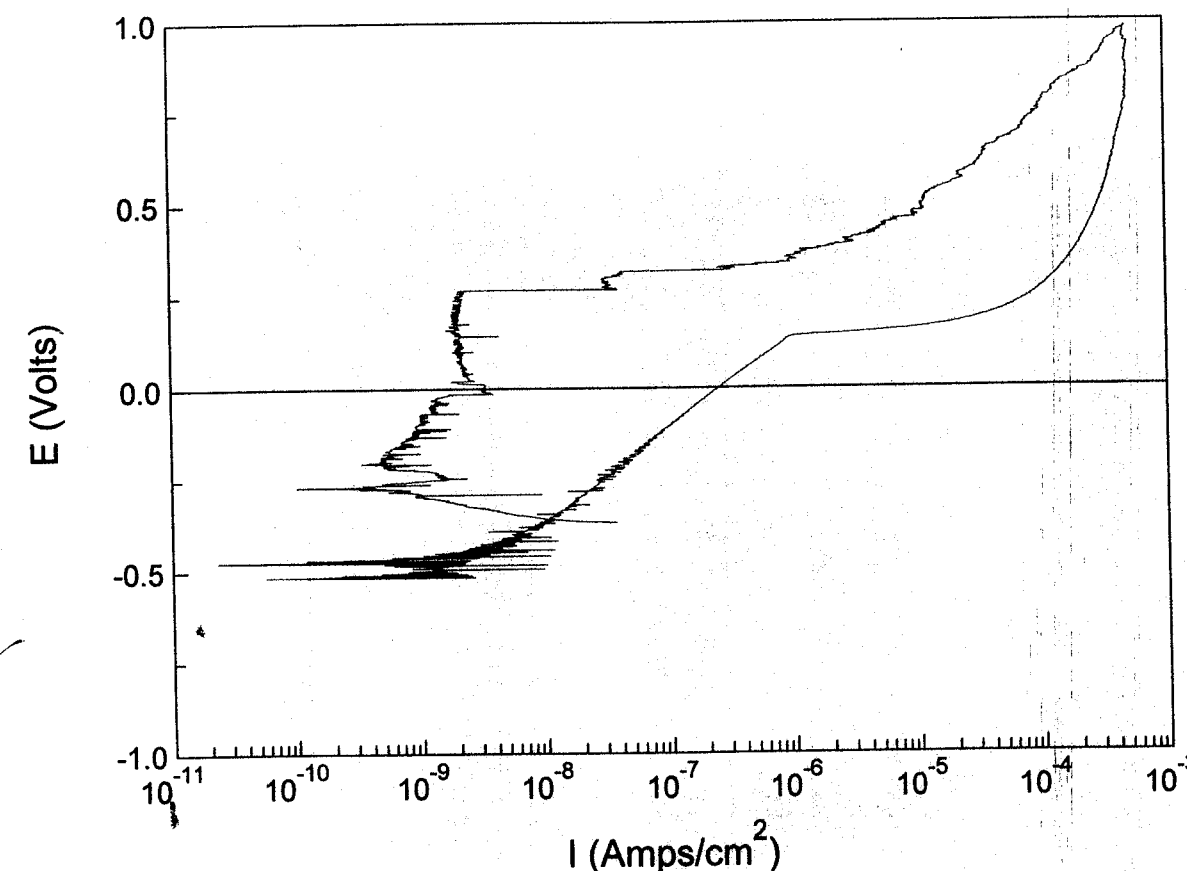
Final wt = 30.01056g

Final pH = 5.848

9-8-99

Some pitting noted but not under crevice area

9-8-99



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Invented by _____

Date _____

Recorded by _____

9-8-99

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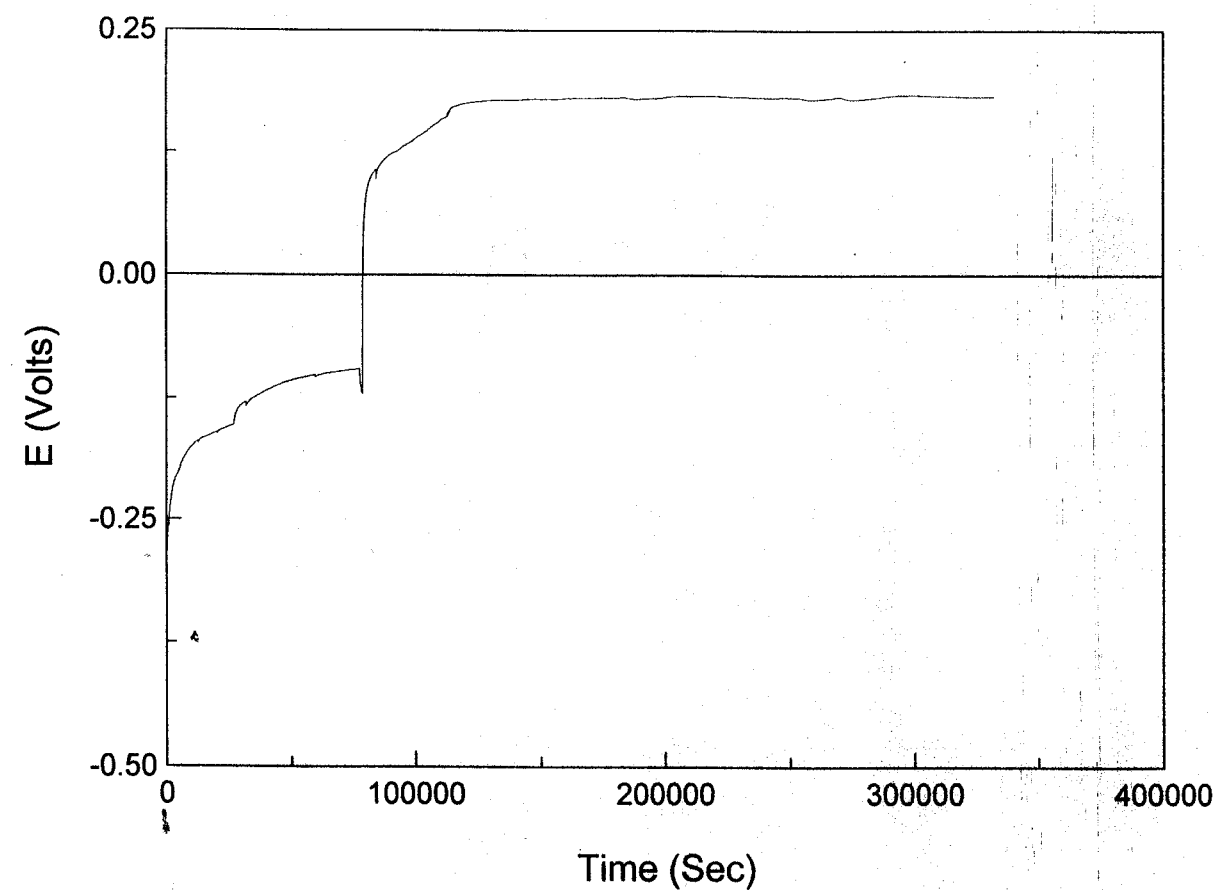
Zr heat treat @ 200°C for 71 days
OCP in 1-13 + 1 M Cl⁻ → 58.44g NaCl # 985302

↳ 25 mL Stock A

↳ 25 mL Stock B

pH_i = 8.800pH_f = 8.008 9/21/99- deaerated w/ N₂ ^{CSB} ~~SSB~~ 9/16/99- H₂O₂ (5 mM) → 0.45 mL added at p + 1820wt_i = N/Awt_f = N/A

SSB 9/21/99



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Witnessed & Understood by me,

SSB

Date

9/16/99

Invented by

Recorded by

Date

TITLE _____

From Page No. _____

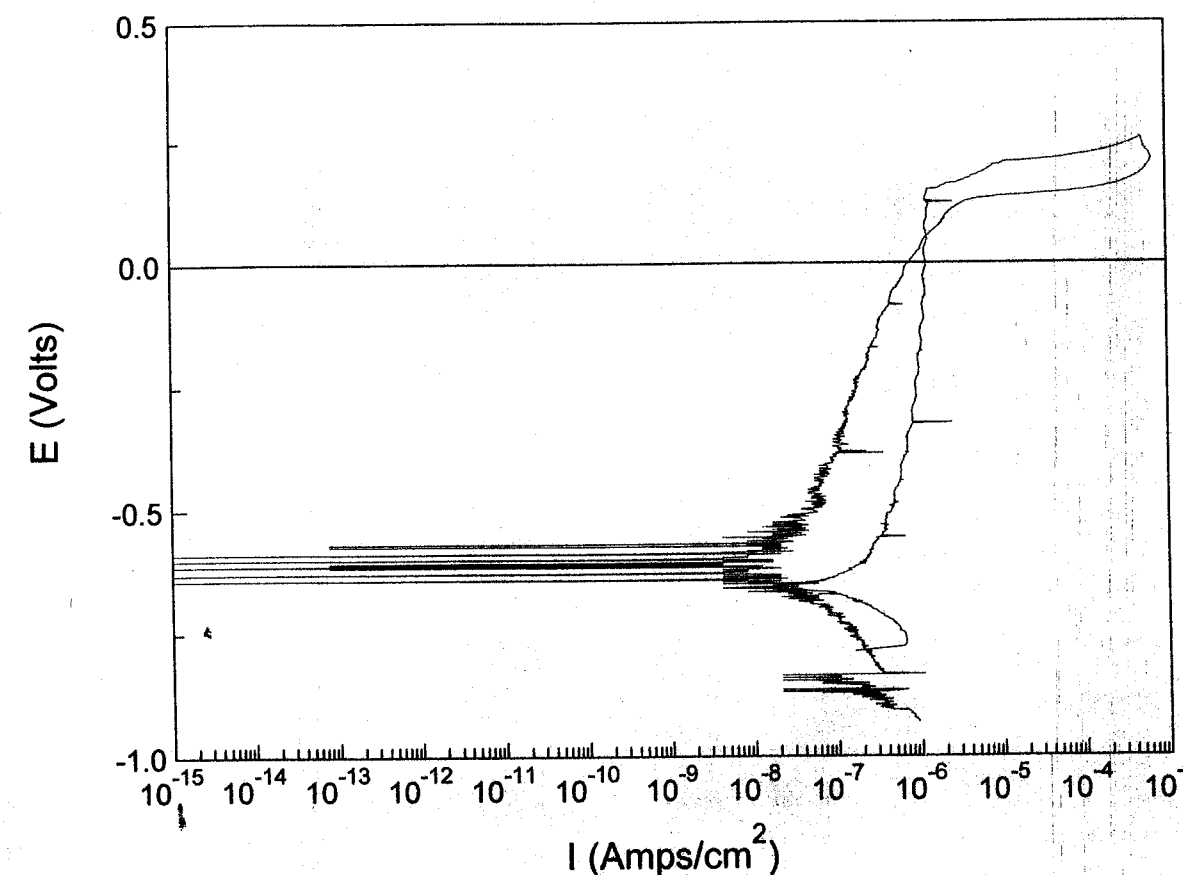
Zr-4 CPP in 1 mM NaCl @ 25°C

0.0584 g/L Lot # 985302

wt_i = 30.0527 gwt_f = 30.0517 gpH_i = 7.431pH_f = 7.604E_p = 456 mVE_r = 350 mV

SSB

ZRA Cpp 31.2at



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Witnessed & Understood by me,

CSB

Date

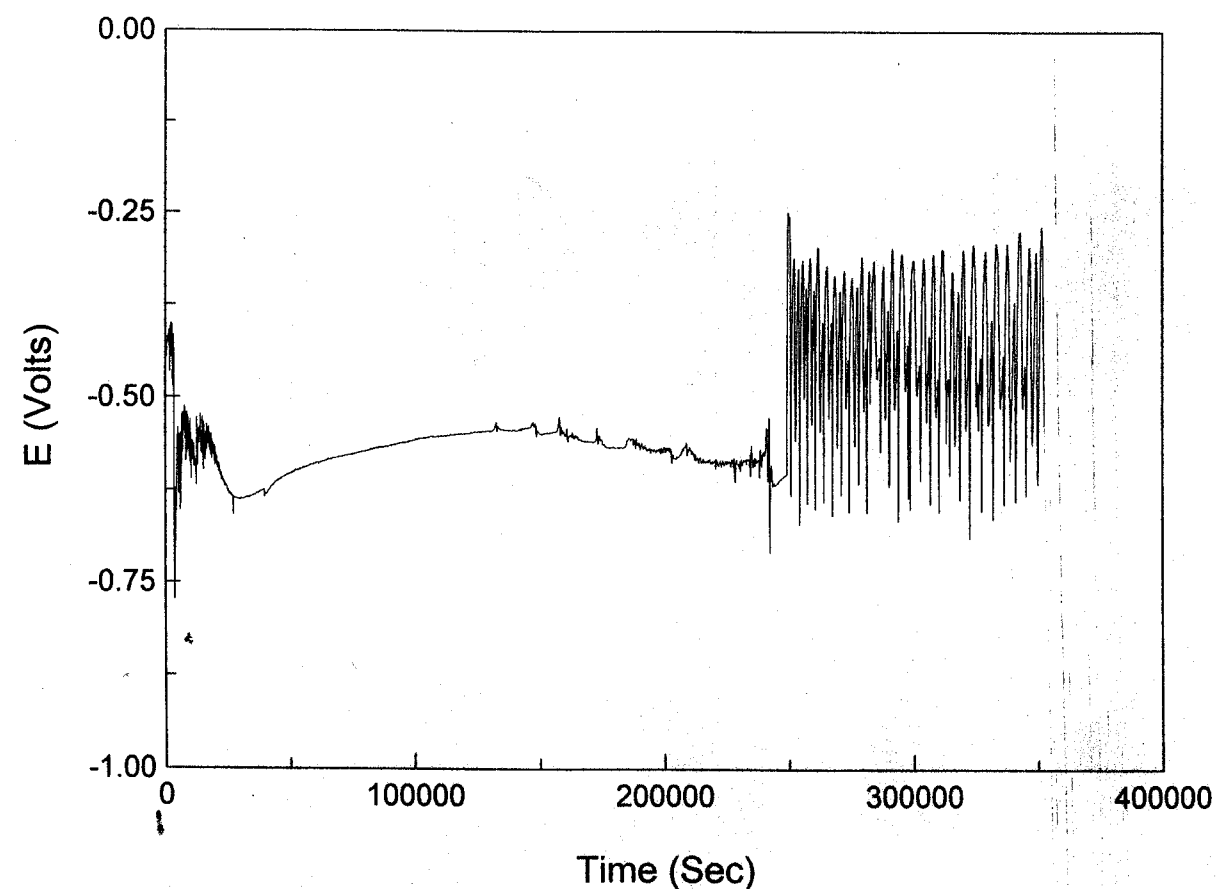
Date

9/28/99

Invented by

Recorded by

From Page No. _____

~~CSB~~ 90/1/99ocp of Zr-4 in 1M NaCl, 25°C SB 10/1/99
95°C + H₂O₂
2r4ocp03.dat - air sat'dpH_i = 6.477pH_f =wt_i = 30.07824wt_f =58.44g NaCl
972274H₂O₂ added @ 4120

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Witnessed & Understood by me,

CSB

Date

10/1/99

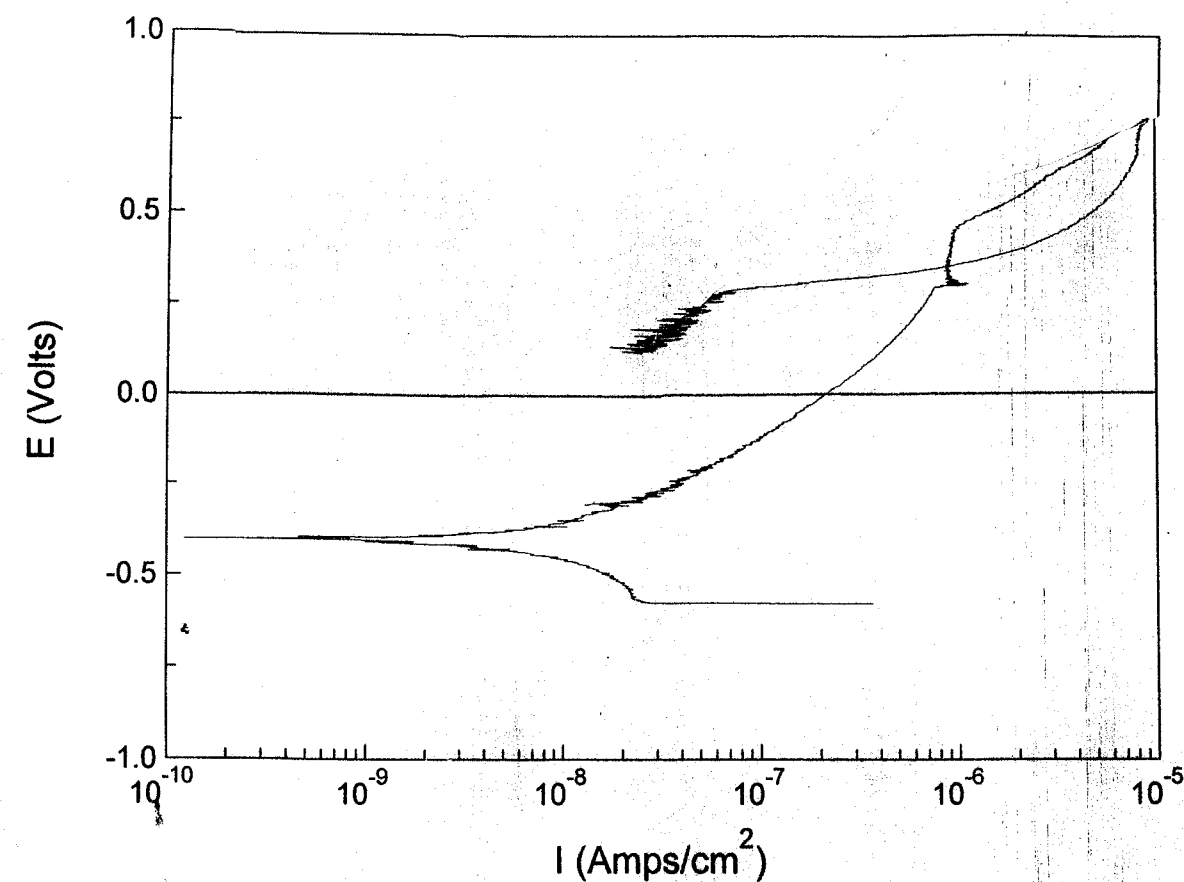
Invented by

Recorded by

Date

TITLE _____

From Page No. _____

Zr-4 CPl in 0.1 M HCl w/ pH adj. to 4.9 w/
HCl
2rCPl32.dat NaCl SB 4/18/99

To Page No. _____

Witnessed & Understood by me,

SB

Date

11/18/99

Invented by

Recorded by

Date

From Page No. _____

EIS to study oxide properties of Zr vs heat treatment

Zr-4 heat treated at 250 °C in air for 8 days

wt_i prior to heat treat = 28.77676 g

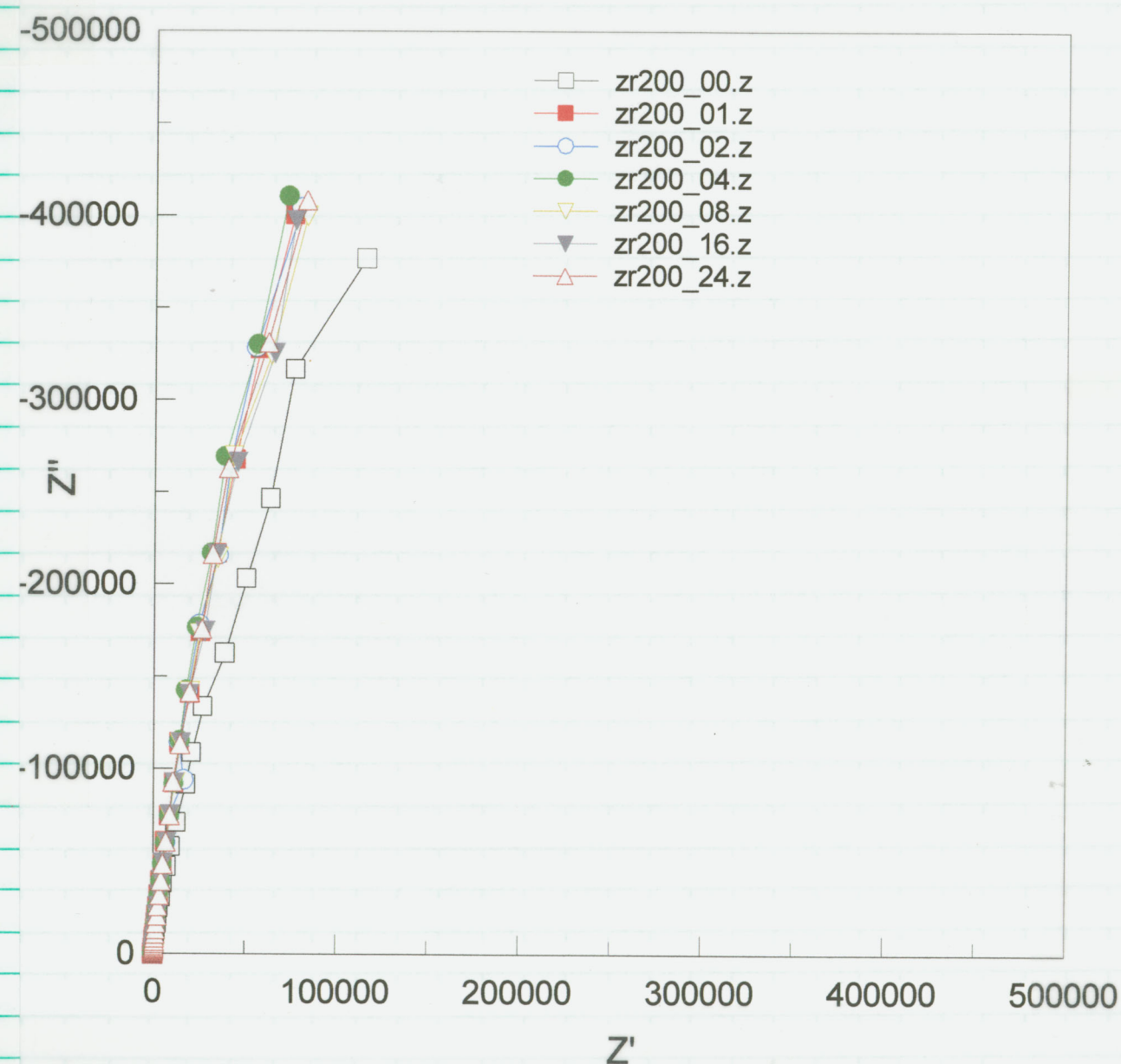
wt_f = wt after heat treat = 28.76864 g

wt_f = wt after EIS test = 28.76942 g

10mm NaCl

Lot 995718

EIS taken at -250 mV/sce at different times 0 - 24 h



Witnessed & Understood by me, _____

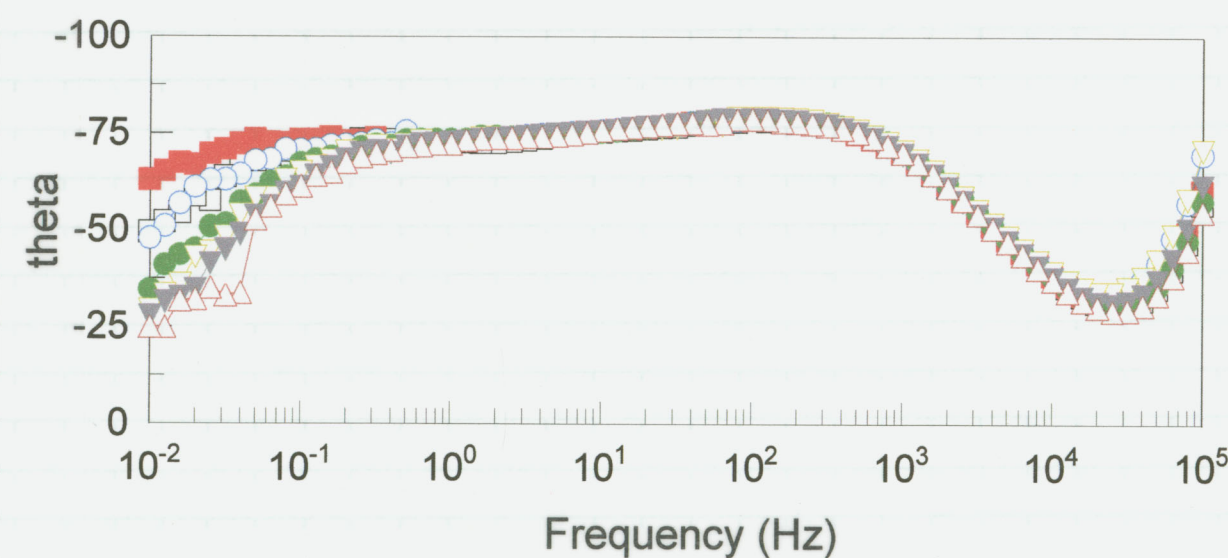
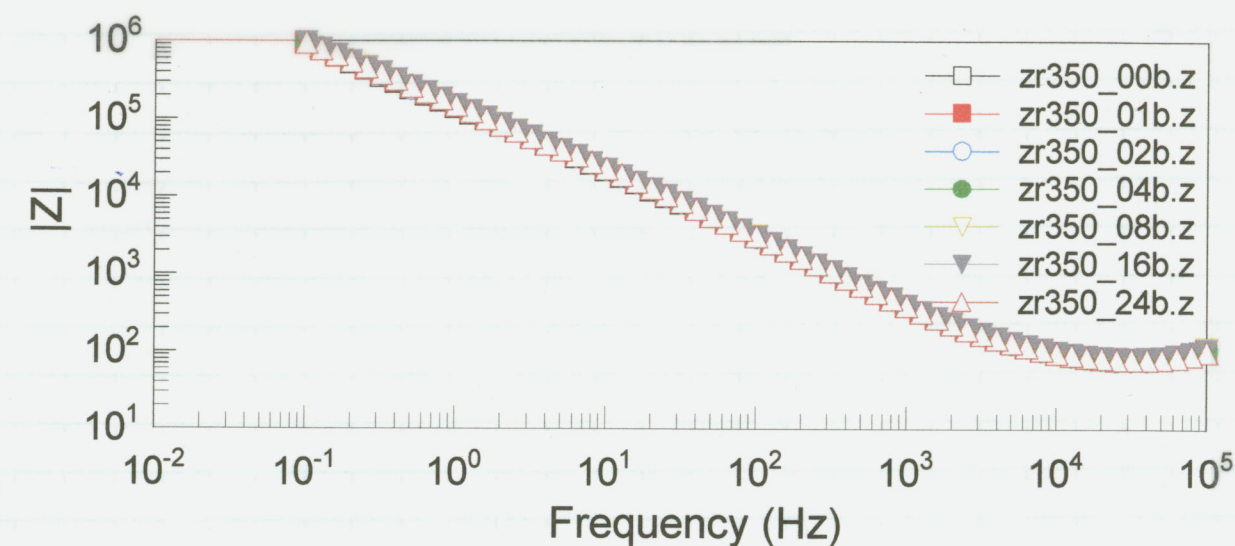
Date _____

Invented by _____

Date _____

Recorded by _____

From Page No. _____



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Witnessed & Understood by me, _____

Date _____

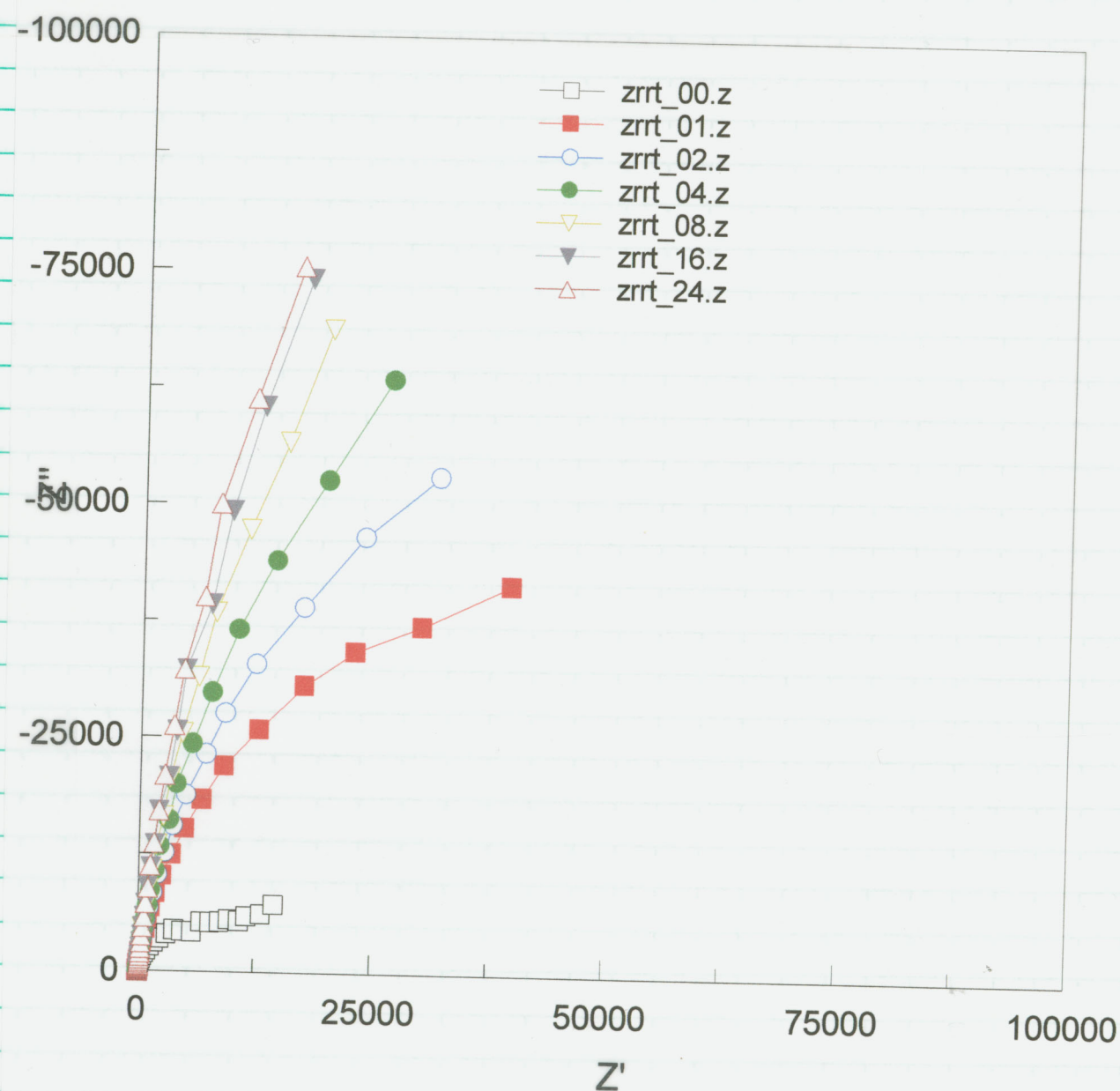
Invented by _____

Date _____

Recorded by _____

From Page No. _____

Zr-4 EIS of oxides
Room temp freshly polished, same setup & soln as
previous test listed on pg 62



Witnessed & Understood by me, _____

Date

12/30/99

Invented by _____

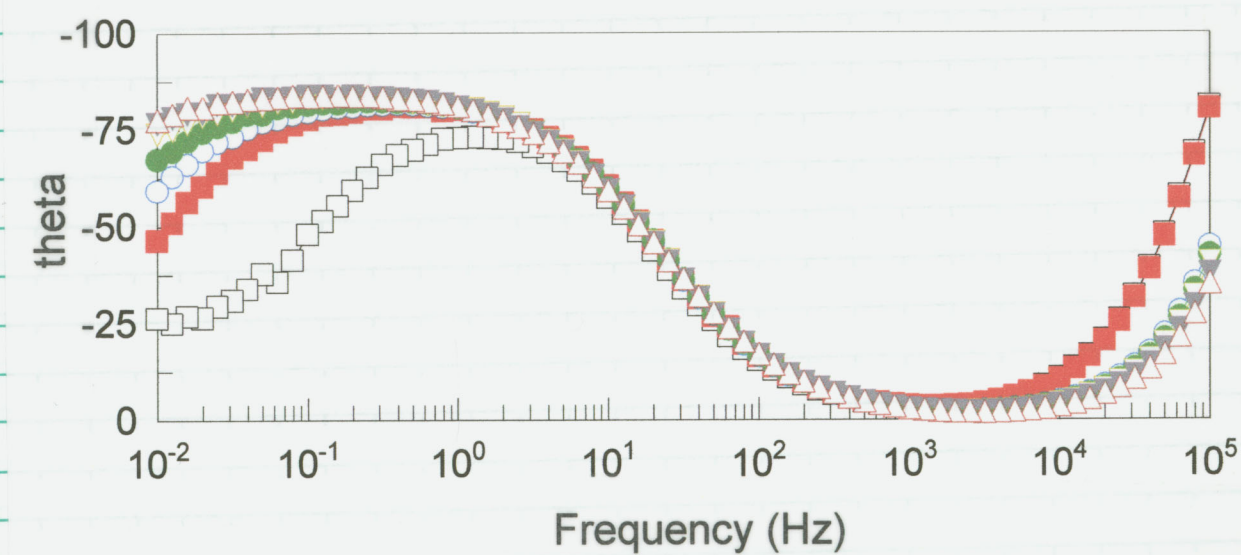
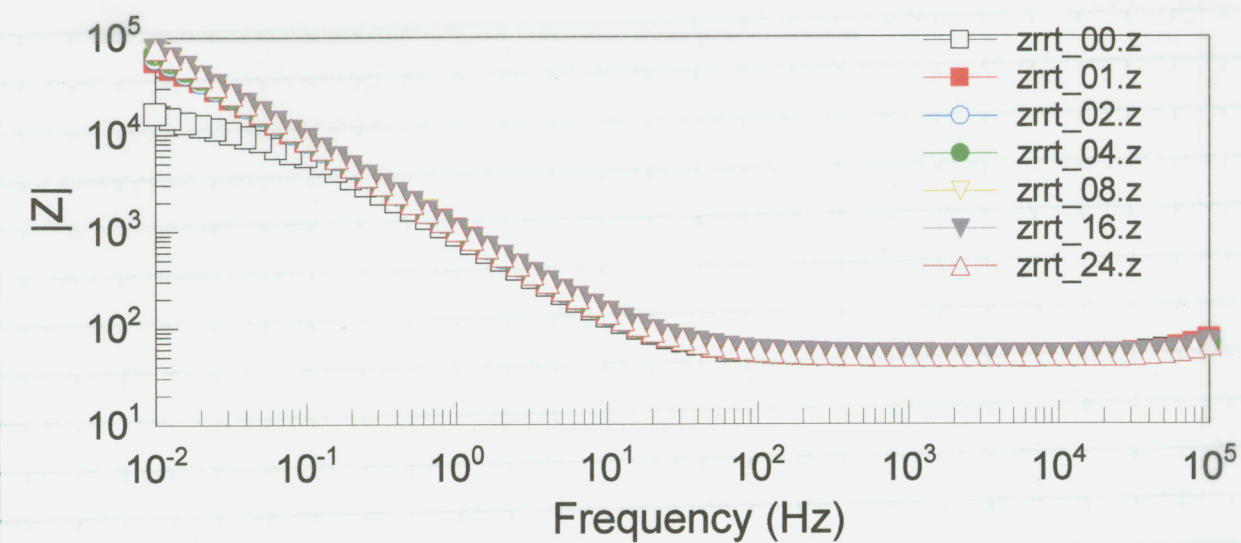
Recorded by _____

Date _____

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TITLE _____

From Page No. _____



Witnessed & Understood by me, _____

Date

12/30/99

Invented by _____

Recorded by _____

Date _____

To Page No. _____

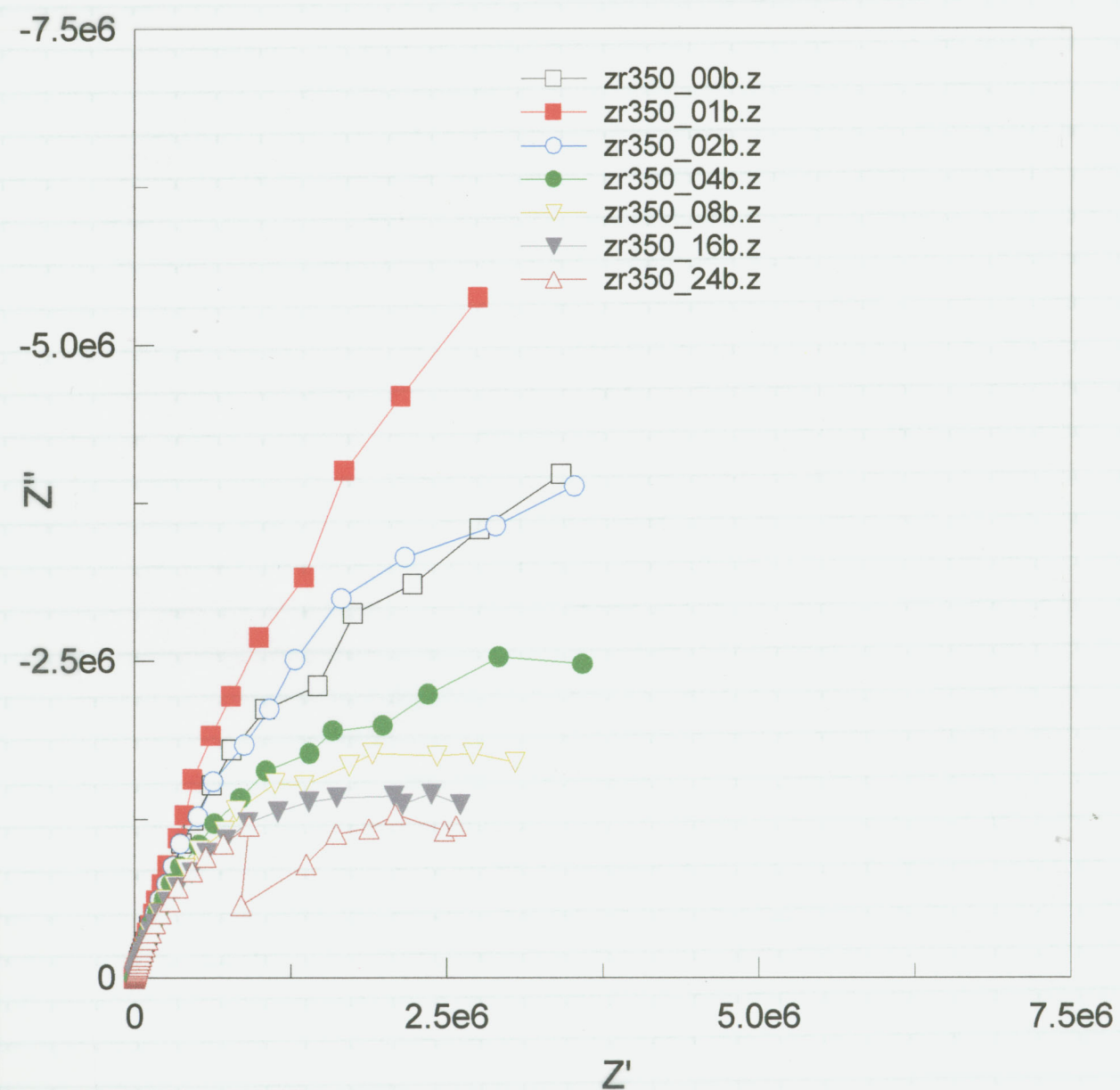
From Page No. _____

Zr -4 EIS of oxide formed at 350°C for 17d.

wti = 28.72184

wtg after heat treat = 29.14179

same set up as on pg 62



To Page No. _____

Witnessed & Understood by me,

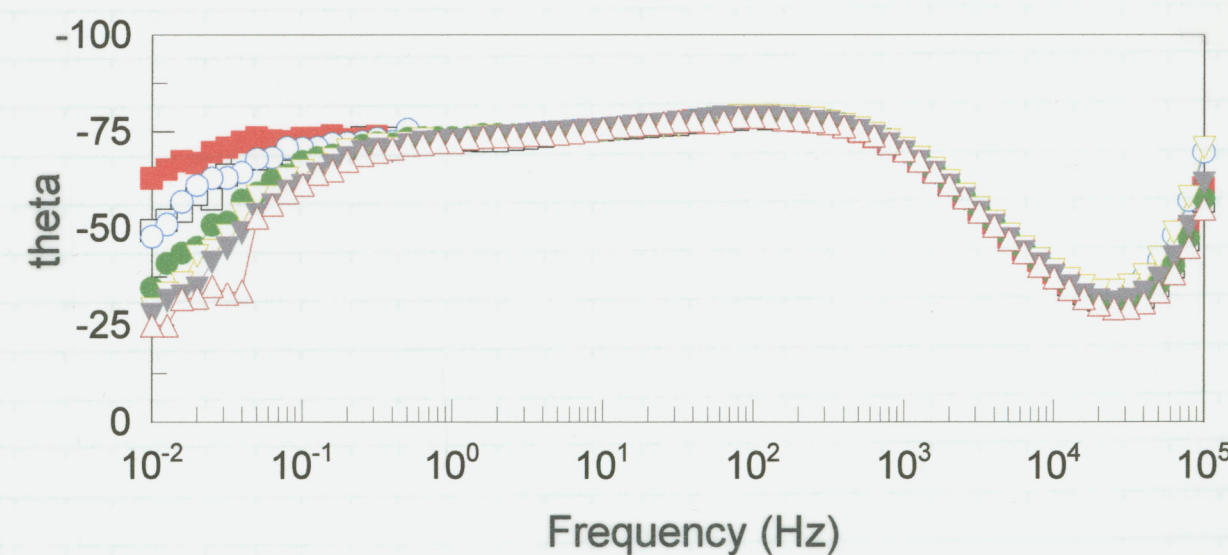
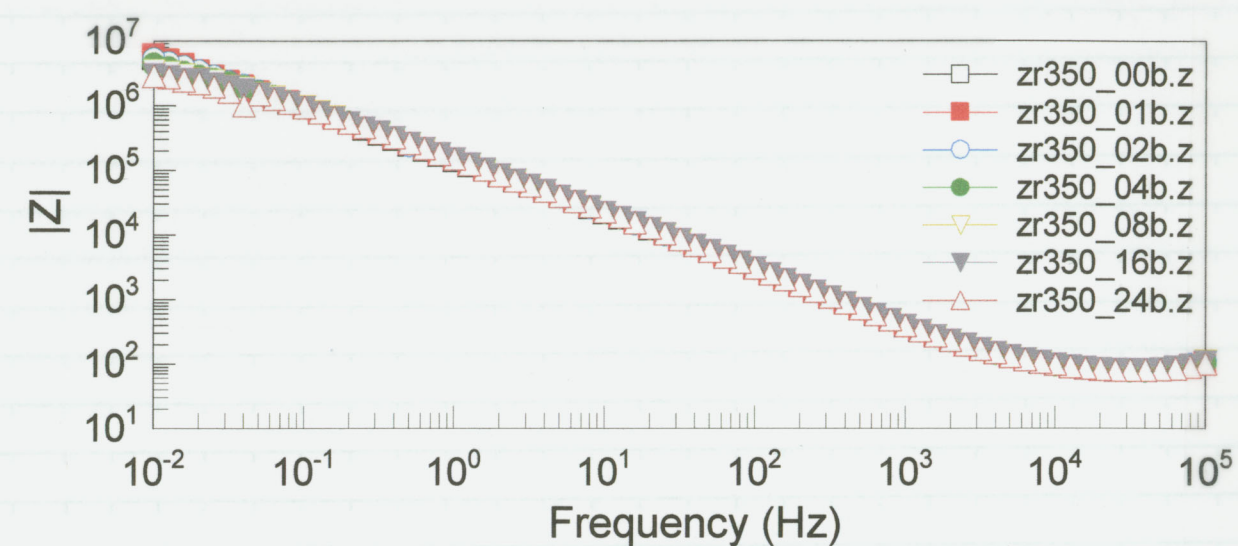
Date

Invented by

Date

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Witnessed & Understood by me,

Date

Invented by

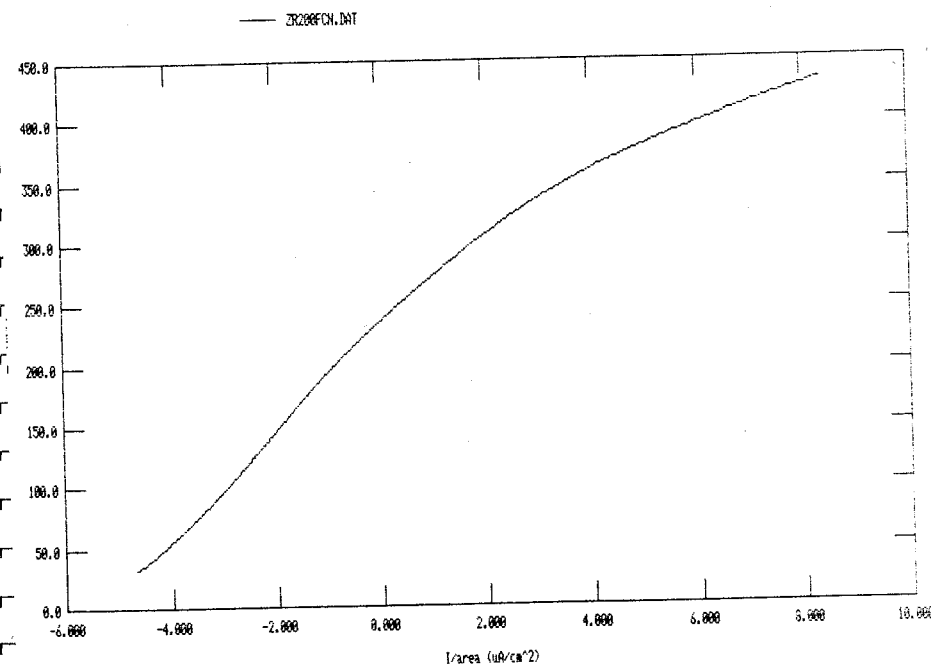
Date

Recorded by

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potentiodynamic polarization from -200 to +200 mV vs ocp
 were conducted in deaerated 0.1M Na_2SO_4 + 0.1M $\text{K}_3\text{Fe}(\text{CN})_6$
 + 0.1M $\text{K}_4\text{Fe}(\text{CN})_6$ to measure ionic & electrical conductivity of oxides
 - 200°C for 8 days

Model J52/552 Corrosion Analysis Software v. 2.38
 PP POTENTIODYNAMIC File Status: NORMAL Date Run: 12-18-99 Time Run: 09:19:38
 CP PASS vs. R CT PASS IP -0.200 vs. OC ID PASS PP 0.200 vs. OC SI 1.000E-03
 SR 1.667E-04 ST 6.000E+00 CR AUTO IR NONE FL 15.3Hz
 RT HIGH STABILITY REF 0.24150 SCE WKS SOLID AR 1.830E+01 LS YES EN 0.000E+00
 DEN 1.000E+00 AU NO OC 0.232



i_A = current density @ +200 mV ocp
 i_C = current density @ -200 mV ocp

$i_A = 8.372 \mu\text{A}/\text{cm}^2$
 $i_C = 4.67 \mu\text{A}/\text{cm}^2$

Na_2SO_4 -
 $\text{K}_3\text{Fe}(\text{CN})_6$ -
 $\text{K}_4\text{Fe}(\text{CN})_6$ -

To Page No. _____

Witnessed & Understood by me,

SR

Date

12/18/99

Invented by

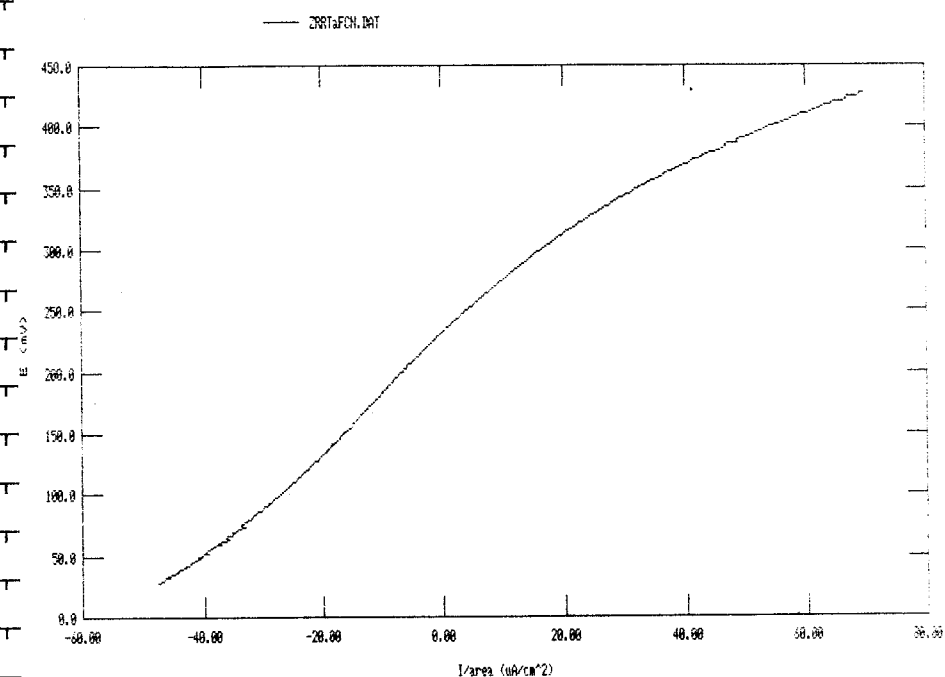
Recorded by

Date

From Page No. _____

Room temp, freshly polished oxide
 - same conditions as p 68

Model J52/552 Corrosion Analysis Software v. 2.38
 PP POTENTIODYNAMIC File Status: EDITED Date Run: 12-19-99 Time Run: 07:01:52
 CP PASS vs. R CT PASS IP -0.200 vs. OC ID PASS PP 0.200 vs. OC SI 1.000E-03
 SR 1.667E-04 ST 6.000E+00 CR AUTO IR NONE FL 15.3Hz
 RT HIGH STABILITY REF 0.24150 SCE WKS SOLID AR 1.830E+01 LS YES EN 0.000E+00
 DEN 1.000E+00 AU NO OC 0.232



$i_A = 69.73 \mu\text{A}/\text{cm}^2$
 $i_C = 47.65 \mu\text{A}/\text{cm}^2$

To Page No. _____

Witnessed & Understood by me,

SR

Date

12/19/99

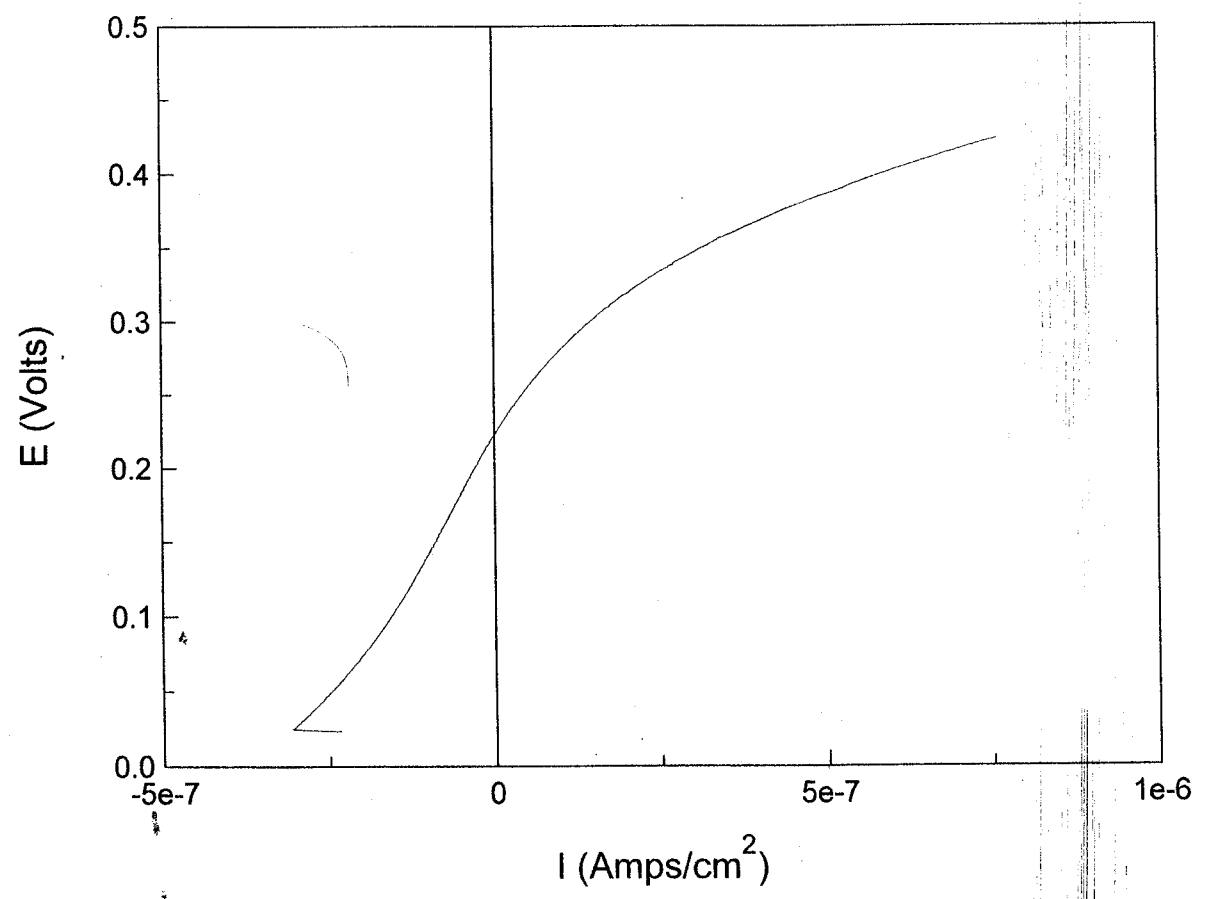
Invented by

Recorded by

Date

Project No. _____
Book No. _____

30 SB 1/16/00 300°C for 17d
Same condition as p 68



same i_A as 16 $\mu A/cm^2$
1/26/00 $i_c = 0.31 \mu A/cm^2$
 $\rightarrow i_A = 0.76 \mu A/cm^2$

oxide	relative i_A to RT	relative i_A to RT
RT	1.0	1.0
200°C	0.120 = 12%	0.098 = 9.8%
300°C	0.011 = 1.1%	0.007 = 0.7%

To Page No. _____

Witnessed & Understood by me, SB Date 1/26/00
Invented by _____ Date _____
Recorded by _____



CONAM KAWIN



194 Internationale Blvd., Glendale Heights, IL 60139
Telephone +1 630-681-0008
Facsimile +1 630-871-5520

SOUTHWEST RESEARCH INST. 7010
6220 CULEBRA RD
P.O. DRAWER 28510
SAN ANTONIO TX 78284
SEAN BROSSIA

P.O. # 50138

DESCR 01-25-00 P0# X94367X
REQ# 505956

REPORT DATE: 02/15/2000

LAB NO: 0128-005 / 05

JOB NO:

ASTM B350 WROUGHT ZIRCALOY-4 HEAT# ~~2209478~~ 2144502 CSB

CHEMICAL ANALYSIS

Cr	.12	Sn	1.59	Fe
Zr	BALANCE			
Cb	.005			

TEST METHODS: ASTM E 663 ; ASTM E 1019 ; CLASSIC WET ;

must type of Heat # \rightarrow matches
w/ pg 14 &
pg 32 in S/N 368

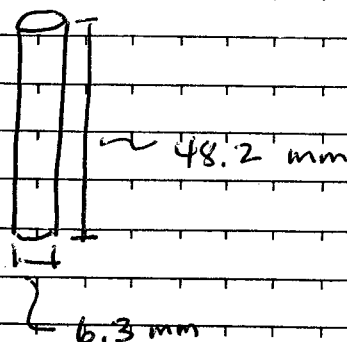
fh
QA INSPECTOR

ALL CHEMICAL TEST RESULTS ARE REPORTED IN WEIGHT PERCENT UNLESS OTHERWISE NOTED.

PAGE 5 OF 16

THIS REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL,
WITHOUT THE WRITTEN APPROVAL OF CONAM KAWIN, INC.

Witnessed & Understood by me, _____ Date _____
Invented by _____ Date _____
Recorded by SB Date 5/15/00

From Page No. New 2r-4 CPP Specimens AD X86114S

05/15/2000 18:41 2563584515

ALSP I

PAGE 01



Metal Samples Corrosion Monitoring Systems
a division of Alabama Specialty Products, Inc.
152 Metal Samples Rd. P.O. Box 8
Munford, AL 36268
Phone: (256) 358-4202 Fax: (256) 358-4515
e-mail: berl@alspi.com Internet: www.alspi.com

Fax Transmission from Benjamin Lackey

Recipient Sean Brosie
Company Southwest Research Institute
Fax Number 1-210-522-5184
Subject MTR's for your order# X86114S
Date 05/15/2000
Number of Pages (including cover) 5

Dear Sean,

Following are copies of the Metal Samples MTR sheet and the original manufacturer MTR's for the materials used on the above mentioned order.

Ti Gr2 - Metal Samples Lot# N843 - CPP Electrodes
Ti Gr2 - Metal Samples Lot# H801 - Crevice Repassivation Specimens
Zr4 - Metal Samples Lot# D830 - CPP Electrodes

Thank you for your order. If you have any questions, or need anything else, please let us know.

Best regards,
Benjamin Lackey

Page No. _____

Witnessed & _____

Recorded by

6/1/00

From Page No. _____

05/15/2000 18:41 2563584515

ALSP I

PAGE 02

MATERIAL TEST REPORT

DATE : 05/15/00
PAGE : 1
ORDER: 43363

Metal Samples Company
P.O. Box 8
152 Metal Samples Road
Munford, AL 36268
Ph. (256) 358-4202 Fx. (256) 358-4515

Customer: 01482 SOUTHWEST RESEARCH INSTITUTE
Your PO#: X86114S

Lot No. N843 Mill: .375"RD X 639.75" (50R/L) Our Order Line No. 1
Description: TIGR2
Chemical Properties:
C:0.011 Fe:0.080 H:0.0015 N:0.006
O:0.150 Ti: BALANCE
Physical Properties:
Tensile-PSI: 79,710 Elong-%: 26.56
Yield-PSI: 63,560 R/A-%: 47.39
Condition: ANLD

Lot No. H801 Mill: TICO TITANIUM, INC. Our Order Line No. 2
Description: TIGR2 .375"X25"X48"
Chemical Properties:
C:0.032 Fe:0.080 H:0.004 N:0.002
O:0.110 Ti: BALANCE
Physical Properties:
Tensile-PSI: 66,900 Elong-%: 25.6
Yield-PSI: 44,600 Condition: ANLD

Lot No. D830 Mill: TELEDYNE WAH Our Order Line No. 3
Description: ZR4 .265 X 4.2 X 93
Chemical Properties:
Al: 45 PPM B: <0.250 C: 110 PPM Co: <10 PPM
Cr: 0.110 Cu: <10 PPM Fe: 0.210 H: <5 PPM
Hf: 70 PPM Mg: <10 PPM Mn: <25 PPM Mo: <10 PPM
N: <24 PPM Nb: <0.250 Ni: <35 PPM Si: 73 PPM
Sn: 1.500 Ti: <25 PPM U: <50 PPM W: <50 PPM
Zr: BAL
Physical Properties:
Not Available

We certify that the Material Test Report is correct to the best of our knowledge and that the material supplied meets your required P.O. specifications.

THANK YOU, Quality Control Dept.

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by

6/1/00

From Page No. _____

05/15/2000 10:41 2563584515

ALSP

PAGE 05

RECEIVED JAN 28 1985

TO Metal Samples Company
ADDRESS Rt. 1 Box 152
Munford, AL 36268TELEDYNE Page 1 of 1
WAH CHANG ALBANY
P.O. BOX 400
ALBANY, OREGON 97321
(503) 826-4211 FAX (510) 596-0073

ATTENTION OF: Don Johnson

IN REGARD TO YOUR
PURCHASE ORDER NO. 3032
ITEM NO. 1
DESCRIPTION Zr4 Plate
DIMENSIONS .265"x4.2"x92.75"
SPECIFICATIONS Per Purchase OrderISSUED DATE: January 18, 1985
REVISED DATE:
DATE SHIPPED Ref. P.L.#1
QUANTITY SHIPPED 1 pc.
WEIGHT SHIPPED 24.9 lbs.
SALES ORDER NO. 1553
HEAT NO. 214450Q Zr4
MACS NO. 35944

THE TEST REPORT FOLLOWS

Material Condition:

INGOT ANALYSIS

COMPOSITION IN PERCENT

Element	Spec.	Top	Middle	Bottom
Sn	1.20-1.70	1.50	1.52	1.44
Fe	0.18-0.24	0.21	0.21	0.19
Cr	0.07-0.13	0.11	0.11	0.10
Fe+Cr	0.28-0.37	0.32	0.32	0.29
Zr				

BALANCE

MAXIMUM IMPURITIES IN PPM

Al	75	45	43	43
B	0.5	<0.25	<0.25	<0.25
C	270	110	120	110
Cd	0.5	<0.25	<0.25	<0.25
Co	20	<10	<10	<10
Cu	50	<10	<10	<10
H	25	<5	<5	6
Hf	100	70	63	76
Mg	20	<10	<10	<10
Mn	50	<25	<25	<25
Mo	50	<10	<10	<10
N	80	24	24	24
NI	70	<35	<35	<35
Si	120	73	72	69
Ti	50	<25	<25	<25
U	3.5	<1.0	<1.0	<1.0
W	100	<50	<50	<50

DB30

C1.05.19.03

C1.03.25.02.06.01-01

MATERIAL
CERTIFIED BY Tony Nelson
kh Tony Nelson
Quality Assurance Dept

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

S.B. [Signature]

6/4/00

TITLE _____

Project No. _____
Book No. _____

From Page No. _____

ZIRCONIUM EXPOSURE TEST - CREVICE SAMPLES IN
18MBG OHM WATER AT 350°C & 300°C

Zirconium Exposure Samples - Pretest Weights

Mettler Model AE240, S/N C-31119

Bldg 57 Sartorius S/N 16704379

Last Cal: 2/7/00, Next: 8/7/00

ZR1 ZR2 ZR2A ZR2B ZRA ZRB ZRC ZRD

(g) (g) (g) (g) (g) (g) (g) (g)

29.10267 29.51858 29.47941 29.56545 29.18177 29.21341 29.06434 29.54845

29.10265 29.51861 29.47946 29.56549 29.18178 29.21339 29.06431 29.54839

29.10265 29.51859 29.47945 29.56546 29.18180 29.21339 29.06428 29.54839

29.10265 29.51859 29.47944 29.56546 29.18179 29.21339 29.06433 29.54844

29.10263 29.51856 29.47943 29.56547 29.18171 29.21333 29.06435 29.54841

29.10264 29.51858 29.47945 29.56544 29.18177 29.21341 29.06430 29.54837

29.10265 29.51860 29.47946 29.56544 29.18177 29.21341 29.06434 29.54838

29.10266 29.51857 29.47944 29.56546 29.18182 29.21338 29.06436 29.54837

29.10267 29.51858 29.47947 29.56544 29.18181 29.21338 29.06428 29.54836

29.10264 29.51857 29.47944 29.56546 29.18186 29.21337 29.06428 29.54836

Mean 29.10265 29.51858 29.47945 29.56546 29.18179 29.21338 29.06432 29.5484
St.Dev 1.286E-05 1.494E-05 1.715E-05 1.475E-05 4.002E-05 3.020E-05 2.828E-05 3.020E-05

Post Test Bldg 57 Sartorius

29.16144 29.61138 29.58755 29.67800 29.18338 29.21403 29.06534 29.54645
29.16157 29.61124 29.58758 29.67809 29.18339 29.21414 29.06547 29.54664
29.16155 29.61105 29.58766 29.67811 29.18350 29.21413 29.06557 29.54661
29.16158 29.61089 29.58761 29.67807 29.18346 29.21418 29.06556 29.54670
29.16154 29.61063 29.58771 29.67812 29.18339 29.21414 29.06547 29.54672
29.16155 29.61052 29.58769 29.67813 29.18351 29.21426 29.06554 29.54672
29.16156 29.61029 29.58763 29.67814 29.18347 29.21421 29.06564 29.54672
29.16152 29.61013 29.58761 29.67814 29.18350 29.21420 29.06556 29.54674
29.16153 29.60996 29.58765 29.67811 29.18350 29.21414 29.06563 29.54669
29.16147 29.60978 29.58760 29.67815 29.18345 29.21424 29.06561 29.54675Mean 29.16153 29.61059 29.58763 29.67811 29.18346 29.21417 29.065539 29.54667
St.Dev 4.43E-05 0.000549 4.93E-05 4.45E-05 5.1E-05 6.62E-05 9.09758E-05 9E-05Change 0.05888 0.092004 0.108184 0.112648 0.001662 0.00079 0.001219 -0.00172
1 Week 350 C 2 Weeks 300 C 1 Week 2 WeeksMean 29.06760 29.54784
St.Dev 6.69324E-05 6.23E-052 Week Change 0.00206 0.00126
4 Week Change 0.00328 -0.00046350°C SERIES COMPLETED 6/16/00 - NEXT SERIES TERMINATED 6/19/00
TEMPERATURE IS TOO HIGH -
300°C SERIES COMPLETED 6/30/00

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

[Signature]

8/8/00

From Page No. _____

ZIRCONIUM EXPOSURE TESTS - CORROSION SPECIMENS
IN 18M Ω OHM WATER AT 300°C

Zirconium Exposure Samples - Second Series Pretest

	Z1	Z2	Z3	Z4	Z5	Z6
	(g)	(g)	(g)	(g)	(g)	(g)
	9.20141	9.18713	9.19986	9.20562	9.10513	9.08750
	9.20145	9.18593	9.19725	9.20302	9.10521	9.08759
	9.20144	9.18590	9.19625	9.20203	9.10525	9.08762
	9.20147	9.18592	9.19586	9.20086	9.10526	9.08767
	9.20147	9.18591	9.19592	9.20004	9.10527	9.08778
	9.20150	9.18587	9.18580	9.19992	9.10527	9.08767
	9.20147	9.18588	9.19580	9.19988	9.10525	9.08762
	9.20143	9.18589	9.19580	9.19988	9.10526	9.08768
	9.20148	9.18584	9.19582	9.19990	9.10525	9.08768
	9.20148	9.18588	9.19585	9.19991	9.10530	9.08772
Mean	9.20146	9.18602	9.19542	9.20111	9.10525	9.08765
St.Dev	0.00003	0.00039	0.00361	0.00193	0.00005	0.00008
Post Test						
	9.20206	9.18697	9.18651	9.20118	9.10718	9.08933
	9.20196	9.18697	9.18655	9.20123	9.10725	9.08931
	9.20197	9.18699	9.18653	9.20118	9.10714	9.08932
	9.20201	9.18703	9.18652	9.20115	9.10713	9.08928
	9.20197	9.18699	9.18651	9.20114	9.10722	9.08925
Mean	9.20199	9.18699	9.18652	9.20118	9.10718	9.08930
St.dev	4.15935E-05	2.45E-05	1.67E-05	3.51E-05	5.13E-05	3.27E-05
Change	0.00053	0.00097	-0.00890	0.00007	0.00194	0.00164

SERIES COMPLETED 8/7/00

To Page No. _____

Witnessed & Understood by me,



Date

8/8/00

Invented by

Recorded by

Date

TITLE _____

From Page No. _____

CENTER FOR NUCLEAR WASTE REGULATORY
ANALYSES

SCIENTIFIC NOTEBOOK REVIEW CHECKLIST RECORD

Scientific Notebook No.: 328

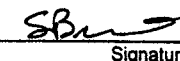
Accomplished

1. Initial entries per QAP-001 ☒ YES
2. Dating of entries ☒ YES
3. Corrections (crossed out, one line through w/initials/date) ☒ NO
4. White out not used ☒ YES
5. Page number visible on original notebook ☒ YES
6. In process entries per QAP-001 ☒ YES
7. Figure numbers present ☒ N/A
8. Text visible ☒ YES
9. Electronic Scientific Notebook changes initialed and dated ☒ N/A
10. Permanent ink or type only ☒ YES
11. Signing of entries (not required on each page) ☒ YES
12. Statement at the end of electronic Scientific Notebook print outs—"No original text removed" ☒ N/A
13. Electronic media in the scientific notebook properly labeled ☒ N/A

Discrepancies have been identified. Yes ☒ No ☐Checker: 

Date: 8/16/00

The discrepancies identified in this Scientific Notebook Review Checklist have been addressed by:


Signature8/16/2000
Date

CNWRA Form QAP-01 (8/2000)

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Recorded by

Date

8/16/2000

From Page No. _____

Copy of pages 71-77 sent to QA

SBS 9/5/2000

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

To Page No. _____

From Page No. _____

wt gains observed during 300°C autoclave exposures on
Zr-4

Zirconium Exposure Samples - Pretest Weights

Mettler Model AE240, S/N C-31119

Last Cal: 2/7/00, Next: 8/7/00

Bldg 57 Sartorius S/N 16704379

ZR1	ZR2	ZR2A	ZR2B	ZRA	ZRB	ZRC	ZRD
(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
29.10267	29.51858	29.47941	29.56545	29.18177	29.21341	29.06434	29.54845
29.10265	29.51861	29.47946	29.56549	29.18178	29.21339	29.06431	29.54839
29.10265	29.51859	29.47945	29.56548	29.18180	29.21339	29.06428	29.54839
29.10265	29.51859	29.47944	29.56548	29.18179	29.21339	29.06433	29.54844
29.10263	29.51856	29.47943	29.56547	29.18171	29.21333	29.06435	29.54841
29.10264	29.51858	29.47945	29.56545	29.18182	29.21332	29.06431	29.54841
29.10265	29.51860	29.47946	29.56544	29.18177	29.21341	29.06430	29.54837
29.10266	29.51857	29.47944	29.56546	29.18182	29.21338	29.06434	29.54838
29.10267	29.51858	29.47947	29.56544	29.18181	29.21338	29.06436	29.54837
29.10264	29.51857	29.47944	29.56548	29.18188	29.21337	29.06428	29.54836

Mean	29.10265	29.51858	29.47945	29.56546	29.18179	29.21338	29.06432	29.5484
St.Dev	1.286E-05	1.494E-05	1.715E-05	1.475E-05	4.002E-05	3.020E-05	2.828E-05	3.020E-05

Post Test Bldg 57 Sartorius

29.16144	29.61138	29.58755	29.67800	29.18338	29.21403	29.06534	29.54645
29.16157	29.61124	29.58758	29.67809	29.18339	29.21414	29.06547	29.54664
29.16155	29.61105	29.58766	29.67811	29.18350	29.21413	29.06557	29.54661
29.16158	29.61089	29.58761	29.67807	29.18346	29.21418	29.06556	29.54670
29.16154	29.61063	29.58771	29.67812	29.18338	29.21414	29.06547	29.54672
29.16155	29.61052	29.58769	29.67813	29.18351	29.21426	29.06554	29.54672
29.16156	29.61029	29.58763	29.67814	29.18347	29.21421	29.06564	29.54672
29.16152	29.61013	29.58761	29.67814	29.18350	29.21420	29.06556	29.54674
29.16153	29.60996	29.58765	29.67811	29.18350	29.21414	29.06563	29.54669
29.16147	29.60978	29.58760	29.67815	29.18345	29.21424	29.06561	29.54675

Mean	29.16153	29.61059	29.58763	29.67811	29.18346	29.21417	29.065539	29.54667
St.Dev	4.43E-05	0.000549	4.93E-05	4.45E-05	5.1E-05	6.62E-05	9.09758E-05	9E-05

Change	0.05888	0.092004	0.108184	0.112848	0.001662	0.00079	0.001219	-0.00172
--------	---------	----------	----------	----------	----------	---------	----------	----------

1 Week	←	2 Weeks	→	1 Week	←	2 Weeks	→
350 C				300 C			

29.06750	29.54784
29.06758	29.54792
29.06766	29.54798
29.06762	29.54793
29.06768	29.54800

Mean	29.06760	29.54793
St.Dev	6.69324E-05	6.23E-05

29.06844	
29.06845	
29.06849	
29.06846	
29.06854	

Mean	29.068476
St.Dev	4.03738E-05

2 Week Change	0.00206	0.00126
4 Week Change	0.00328	-0.00046
8 Week Change	-0.00007	
12 Week Change	0.004158	

2 WKS

4 WKS

12 WKS

0.00387

SBS 4/4/0

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

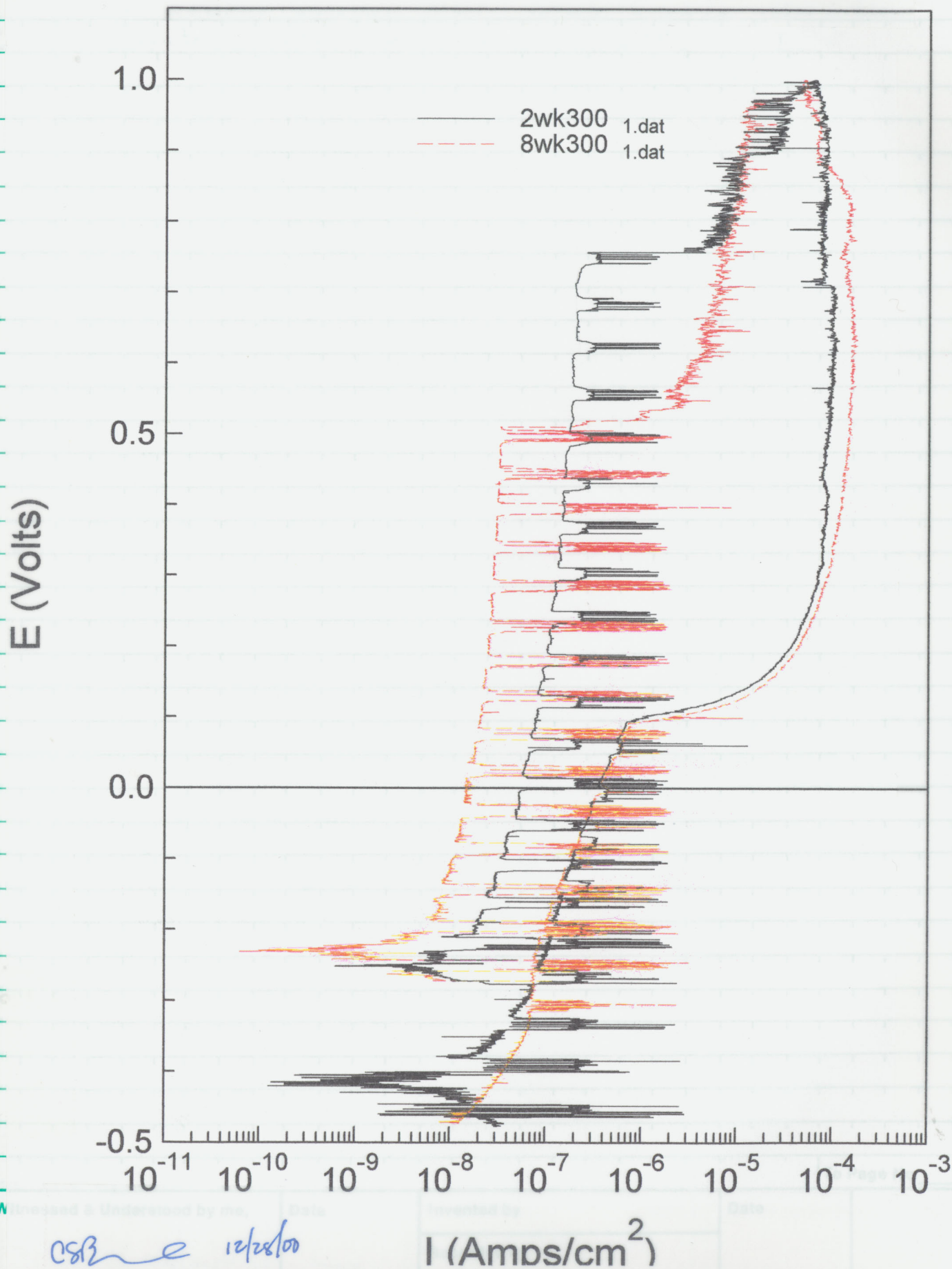
Recorded by _____

12/28/2000

To Page No. _____

From Page No. _____

cpp of 300°C autoclave specimens in 0.1M NaCl at 98°C



TITLE _____

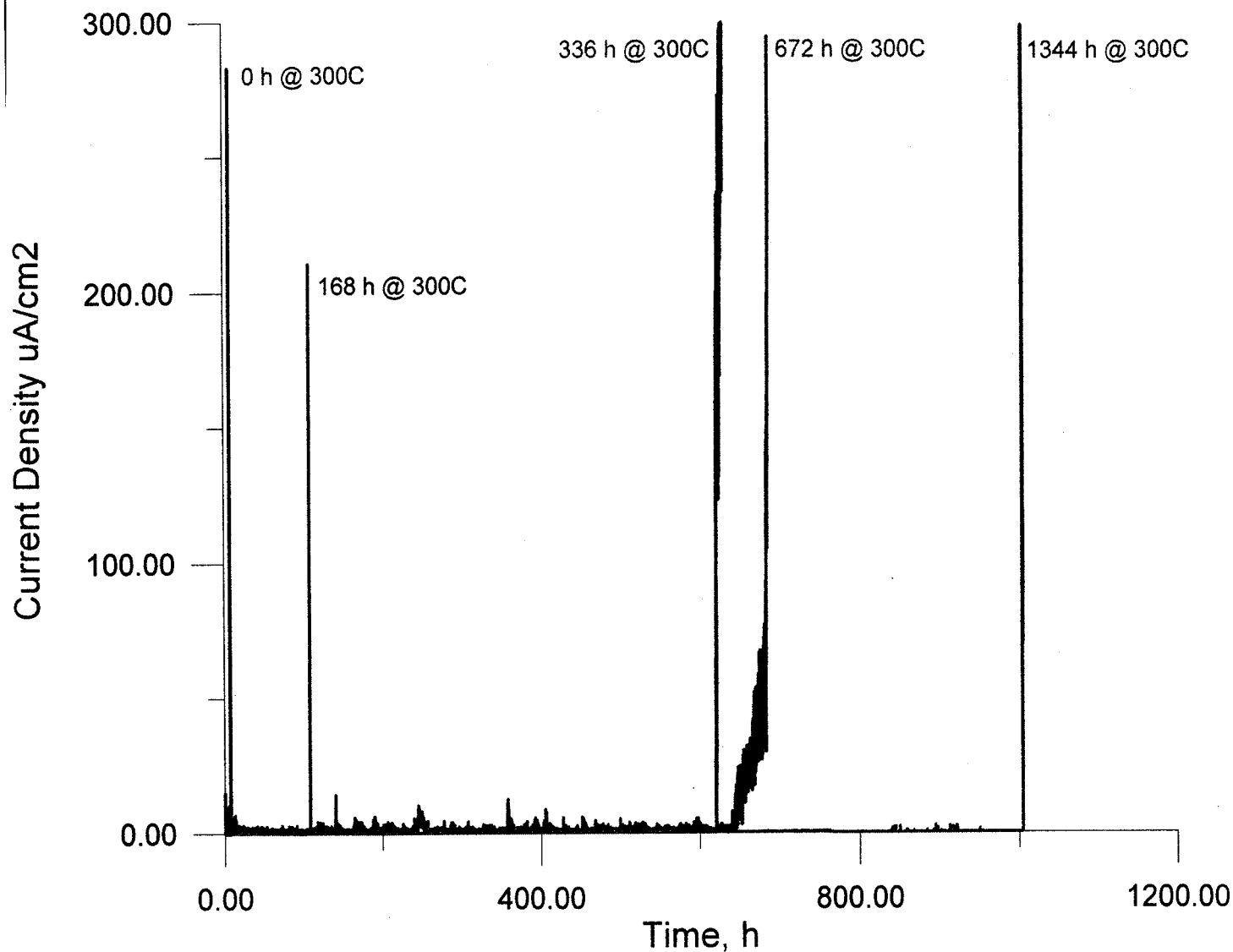
From Page No. _____

Long-term potentiostatic holds in 0.01 M NaCl 95°C
of Zr4 specimens exposed in autoclave at 300°C for
different times

SFB

12/28/00

3/22/01

Zr4, 0.01 M NaCl, 95C, E = 125 mVsce

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

3/22/01



From Page No. _____

Open circuit tests - Long term 2r 4

→ Aerated, 95°C, 0.01 M NaCl w/ additions of H_2O_2 + $FeCl_3$
(on-going) SB → 3/22/01

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

TITLE _____



From Page No. _____

3/22/01 - copy sent to QA through pg 82. SB → 3/22/01

To Page No. _____

Witnessed & Understood by me,

Date

Invented by

Date

Recorded by

Project No. _____

Book No. _____ TITLE _____

From Page No. _____

Impedance Testing

3 specimens of Zr-4; one polished to 600 grit and ultrasonically cleaned in methanol, one exposed to 300°C for 2 weeks, one exposed to 300°C for 8 weeks

Test Solution

0.1 M NaCl → 5.844g to 1000ml DI Filter lot #

pH = 6.222

Test Condition

95°C

De-aerated with Nitrogen gas Ultra High Purity

File Names

polished specimen = ZR4IMP1.Z

2 wk exposure = ZR4IMP2WKb.Z

8 wk exposure = ZR4IMP8WK.Z

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by SL

3-2601

Project No. _____

Book No. _____

TITLE _____

From Page No. _____

~~95°C~~ 80°C 7/26/01
Zr-4, 95°C, N₂, 0.1 M NaCl, 0.01 M NaF
as polished

int wt = 9.128

wt_f = 8.459

pH_i = 6.85

pH_f = 7.58

Zr-4, 2 wk oxidized @ 300°C (autoclave)
N₂, 0.1 M NaCl, 0.01 M NaF

wt_i = 9.110

wt_f = 9.096

pH_i = 6.85

pH_f = 7.58

Zr-4, 2 wk oxidized @ 300°C, N₂, 0.1 M NaCl, 0.0005 M NaF

wt_i = 9.090

wt_f = 9.081

pH_i = 6.24

pH_f = 6.38

Zr-4, as polished, N₂, 0.1 M NaCl, 0.0005 M NaF

wt_i = 9.189g

wt_f = 9.184g

pH_i = 6.27

pH_f = 6.19

To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by SL

3/29/01

From Page No. _____

Potentiodynamic test to be run on three samples -
 Virgin ZR4 polished to 600 grit / ZR4 polished & heat treated @ 300°C for
 2 wks and ZR4 polished and heat treated @ 300°C for 8 wks

Test conditions -200mV to +200mV vs OCP Scan Rate .162mV/s
 Deaerated w/ Nitrogen @ 25°C

Test Solution mixed to 2Lt

.1 M Na_2SO_4 #010224 28.4080 g / 2lt DI

.01 M $\text{K}_3\text{Fe}(\text{CN})_6$ #996057 65.8520 g / 2lt DI

.01 M $\text{K}_4\text{Fe}(\text{CN})_6$ #901620 88.4820 g / 2lt DI

Dwg specimen polished to 600 grit & ultrasonically cleaned in methanol

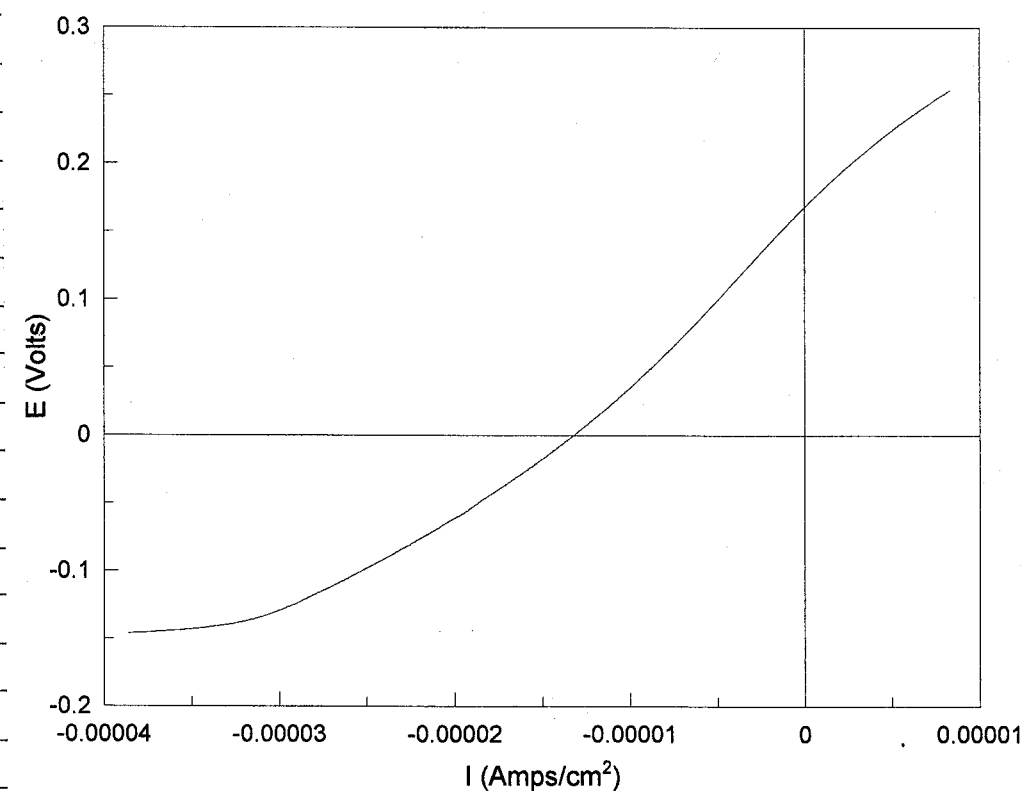
Filename ZR4906A.DAT

Init wt = 29.0743

Final wt = 29.0695

Init pH = 9.206

Final pH = 9.208



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Date _____

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Date _____

Recorded by _____

9/6/01

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Zr4 specimen rinsed in methanol for 1 minute to remove any
 oils

Test Solution from that of page 86

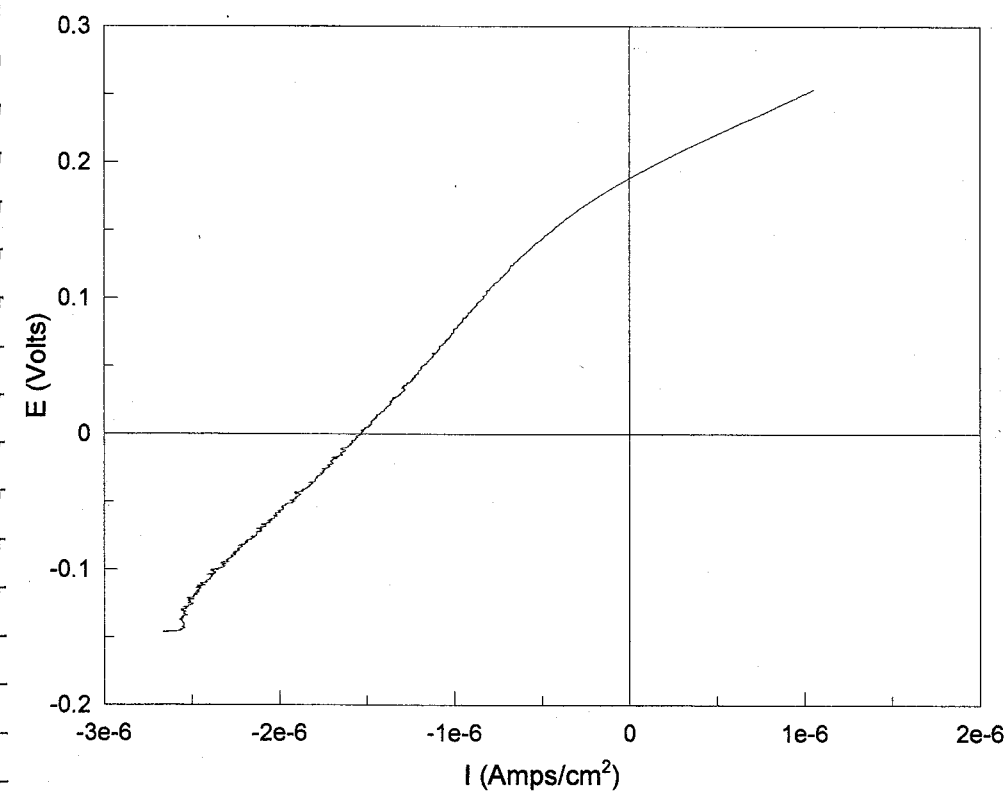
Filename ZR4907B.DAT

Init wt = 29.2153

Final wt = 29.2138

Init pH = 9.208

Final pH = 9.210



To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

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9-7-01

From Page No. 86 Sub specimen rinsed in methanol for 1 minute to remove any oils

Test Solution from that of page 86

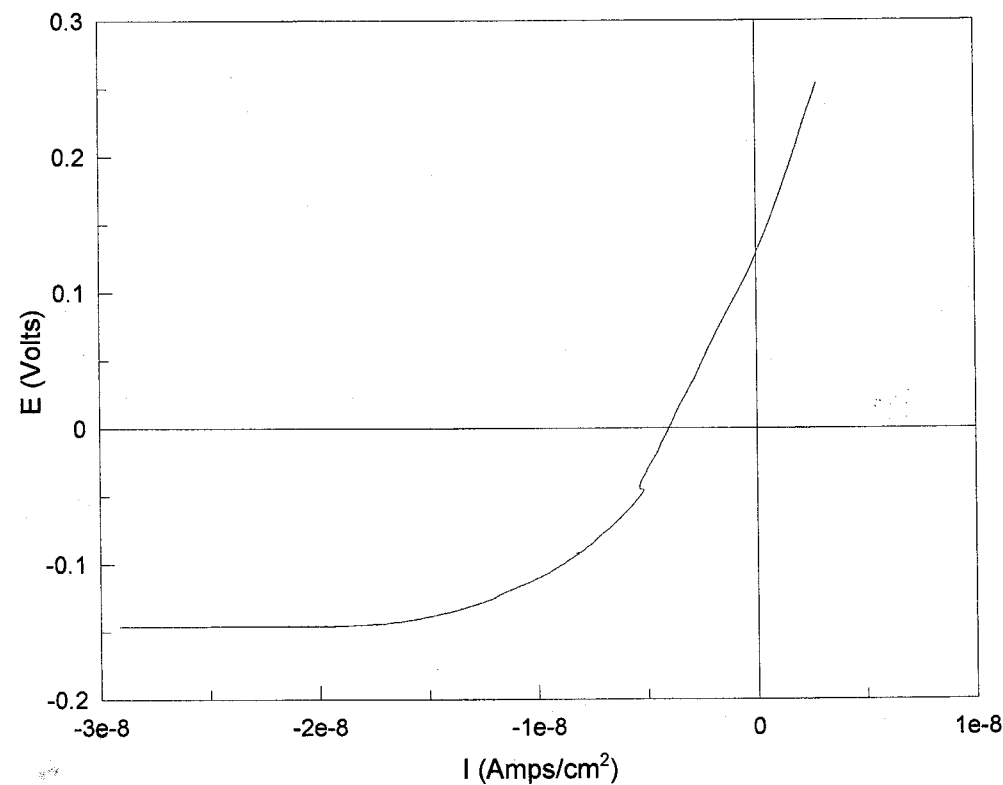
Filename ZR4907C.DAT

Init date 9.2011

Final date 9.2007

Init pH = 9.209

Final pH = 9.210



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Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____

9-7-01

TITLE _____

From Page No. _____

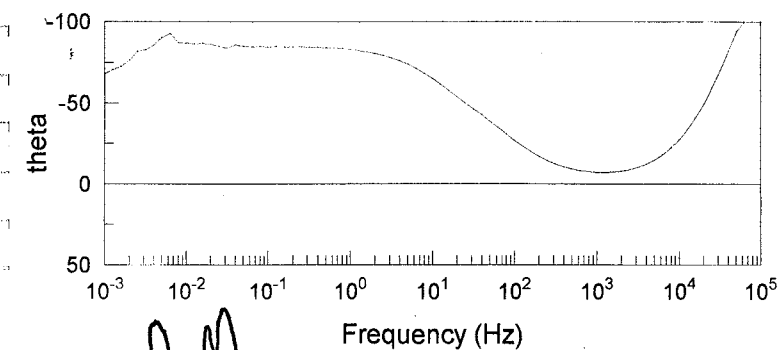
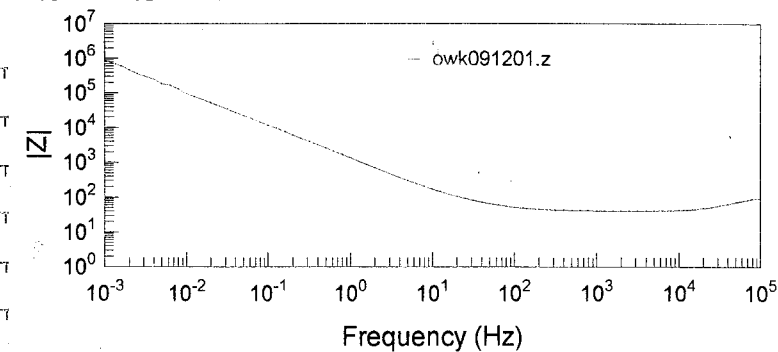
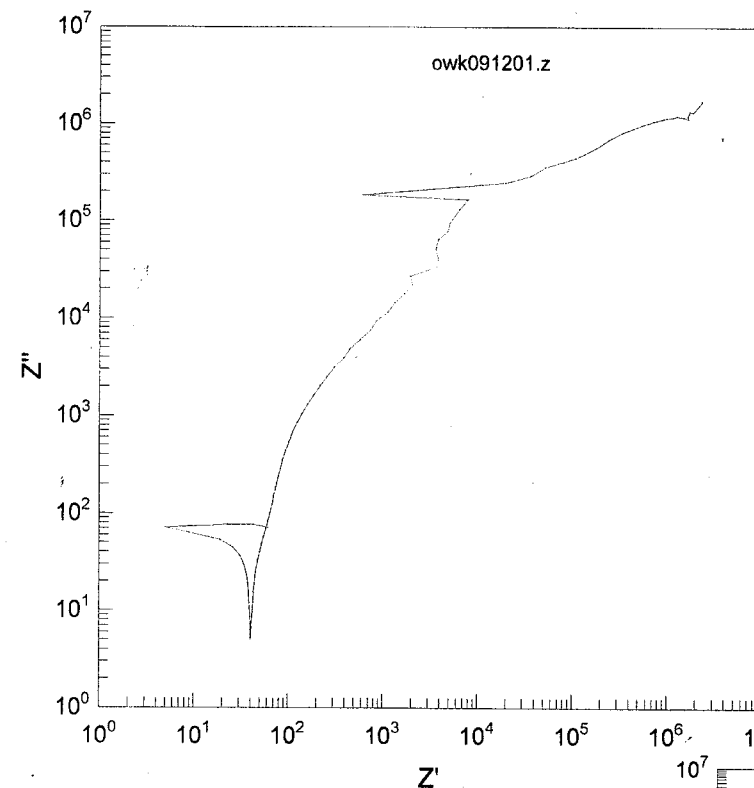
EIS Testing of Dwk exposure specimen rinsed in methanol

Test Solution

0.1 M NaCl → 1.1688g/2L # 015568

Filename = Dwk091201.z

Test in progress 9/17/01 SRB



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9-12-01

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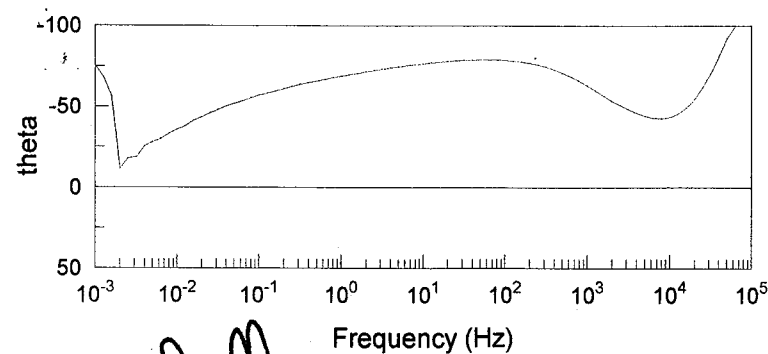
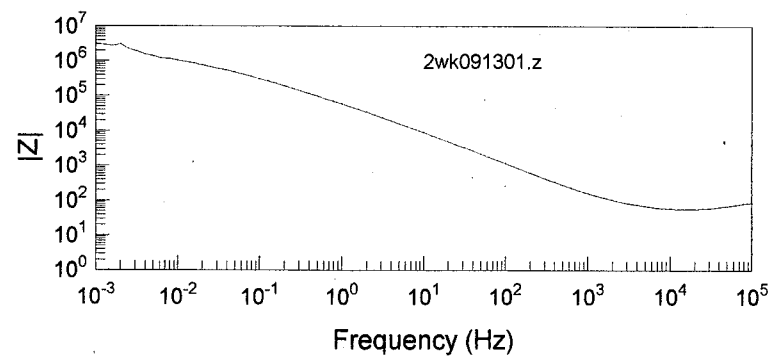
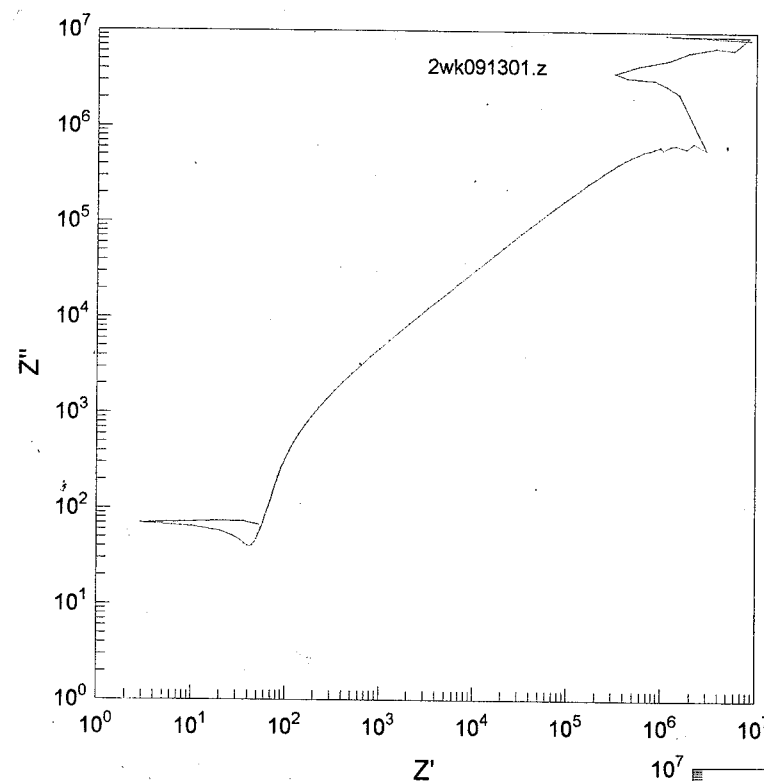
EIS Testing of 2wk 300c exposure specimen

Test Solution

.01 M NaCl \rightarrow 1.1688 g/2L # 015568

Filename = 2wk091301.z

Test in program 9/12/01 SB



Witnessed & Understood by me,

Date

S. Don

9-13-01

TITLE _____

From Page No. _____

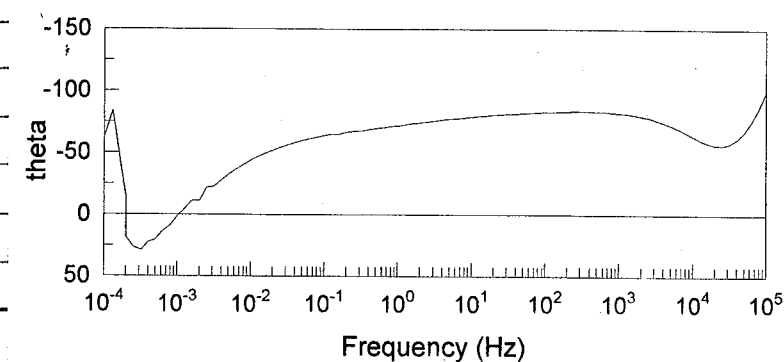
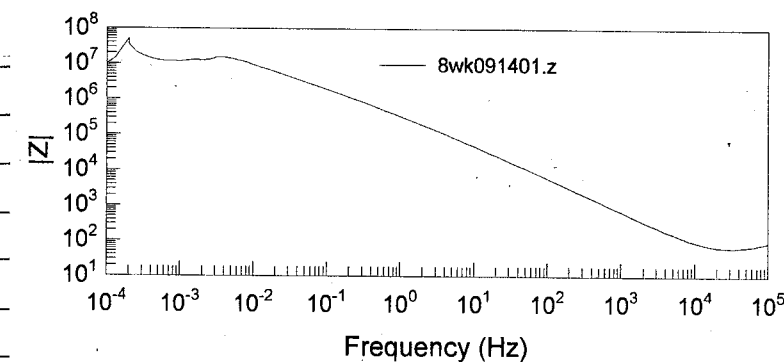
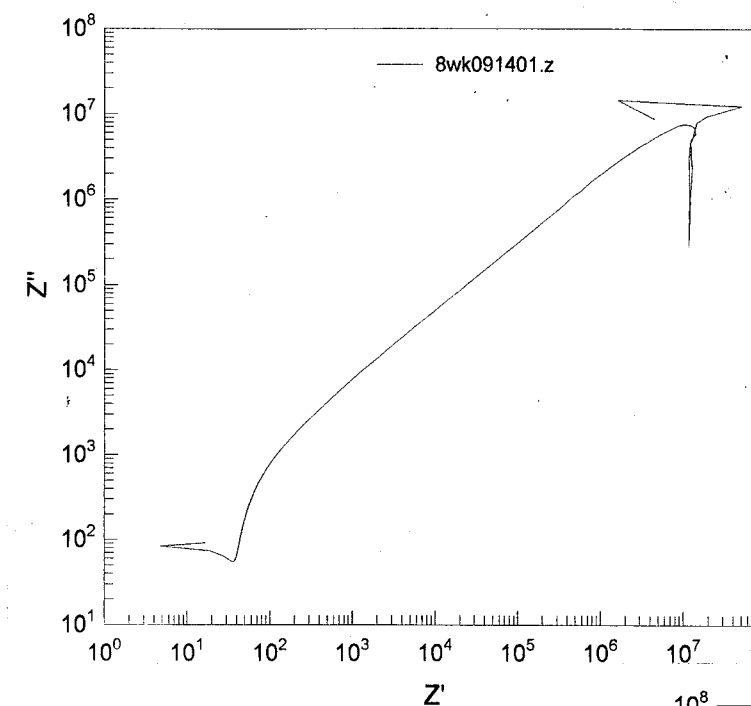
EIS Testing of 8wk 300c exposure specimen

Test Solution

.01 M NaCl \rightarrow 1.1688 g/2L # 015568

Filename = 8wk091401.z

Test in program 9/17/01 SB



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Recorded by

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Galvanic Corrosion Scan

Specimen: ¹ = ZRC 12 wk 300°C Exposure - Specimen Dimensions Pg #2
² = ZR4 wire .040" Diameter - 600 Cont Finish - 6.4cm In Cell
 Start wt: 29.07509g Sartorius Genius SN# 12509099 cal 6/22/01 #1
 End wt: 29.07714g

Solution: 1m Cl
 58.44g NaCl
 + DI water To 1000mls

Temperature: 23°C
 Reference: Fisher 13-620-52 SN# 7079122
 Bubbles with Zero Air

Spike Solution: 0.4m FeCl₃
 6.491g FeCl₃ Lot# 991549
 + DI water To 100mls

1st Spike of solution with 1.25mls on 10/8/01 @ 12:14pm @ 445.8K Into Testing

2nd Spike of solution with 1.25mls on 10/9/01 @ 7:19am @ 514.4K Into Testing

3rd Spike of solution with 10mls on 10/9/01 @ 2:00pm @ 538.4K Into Testing

Restarted 2nd Galvanic Scan 10/10/01 = Same solution with same setup of cell

4th Spike of solution with 12.5mls on 10/11/01 @ 9:26am @ 89.4K Into Testing

Graphs on Following Pages
 →

2:45pm Test Ended 10/15/01

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Date

Invented by

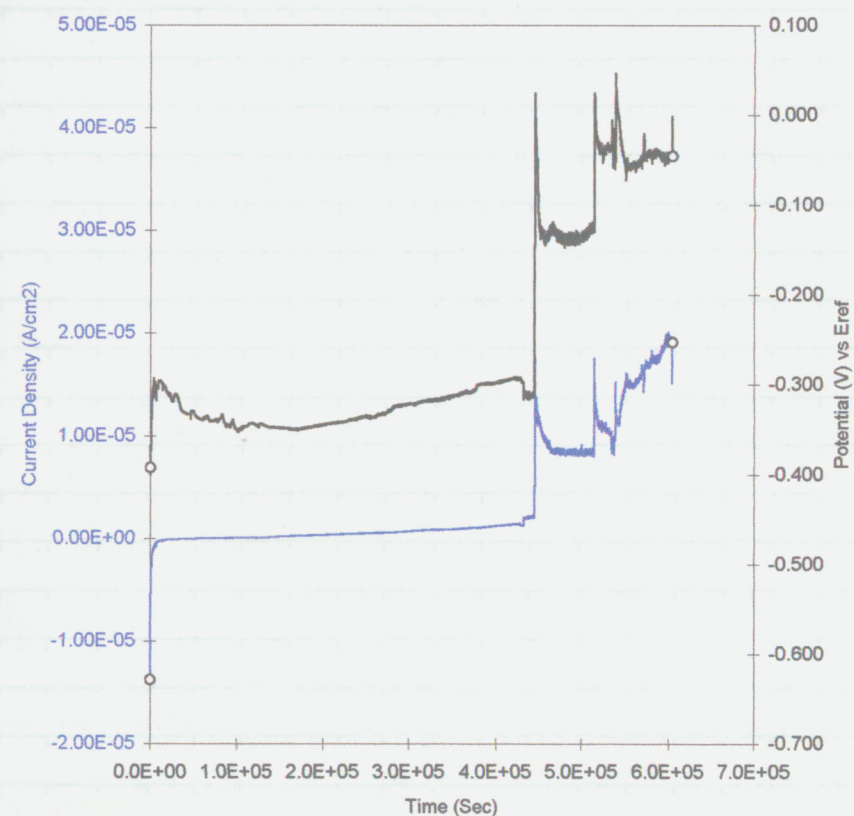
Date

Recorded by

10/3/01

From Page No. _____

Galvanic Corrosion Scan
'Zr-oxide_polished galv1.dta' 10/3/2001-
8:30:1



Pstat: PC4/750
Run Time: 604800 S
Sample Time: 60 S
Cur Limit: 100 mA
Eoc: 0.006261 V
Area: 1 cm2
Electrode: 7.87 gm/cm3, 27.92 g/equiv
Delay: OFF
IR Comp.: OFF

NOTES

ANALYSIS

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Date _____

Invented by _____

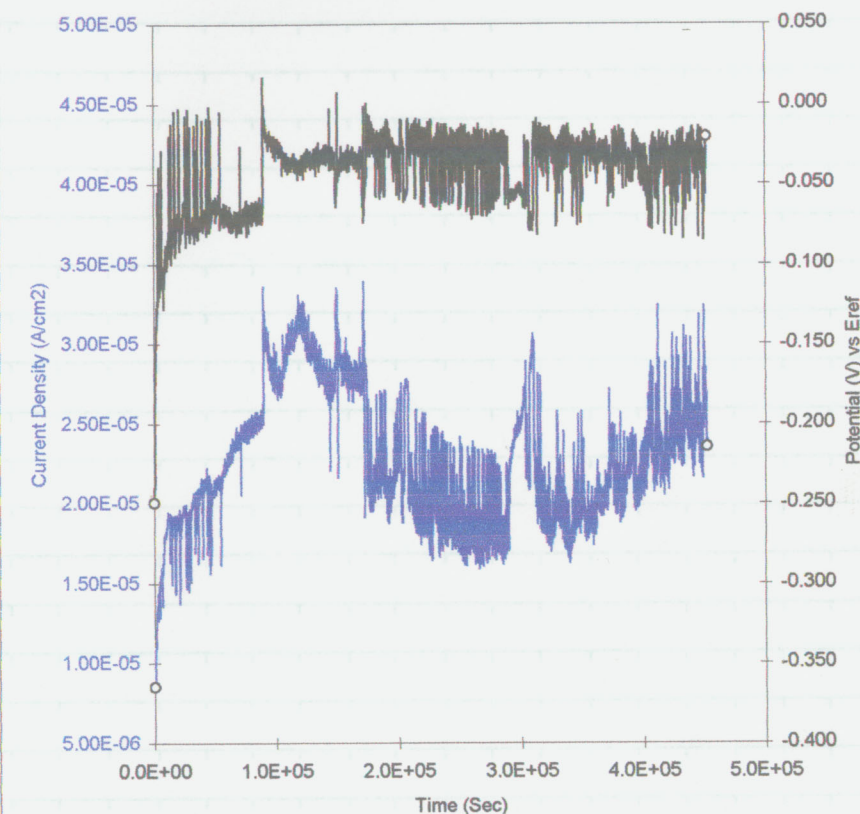
Date _____

Recorded by _____

10/15/01

From Page No. _____

Galvanic Corrosion Scan
'Zr-oxide_polished galv2.dta' 10/10/2001-
8:44:55



Pstat: PC4/750
Run Time: 604800 S
Sample Time: 60 S
Cur Limit: 100 mA
Eoc: -0.353681 V
Area: 1 cm2
Electrode: 7.87 gm/cm3, 27.92 g/equiv
Delay: OFF
IR Comp.: OFF

NOTES

ANALYSIS

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Date _____

Invented by _____

Date _____

Recorded by _____

10/15/01

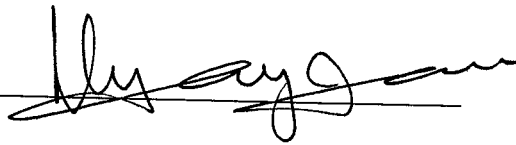
From Page No. _____

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8/12/02

SB

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



To Page No. _____

Witnessed & Understood by me, _____

Date _____

Invented by _____

Date _____

Recorded by _____