308

Scientific Notebook # 260

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CL 57

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Container Life and Source Term

Sean Brossia 256-2809

CNWRA
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Initial Scientific notebook entry for A516 grade 60 carbon steel pit growth kinetics study.

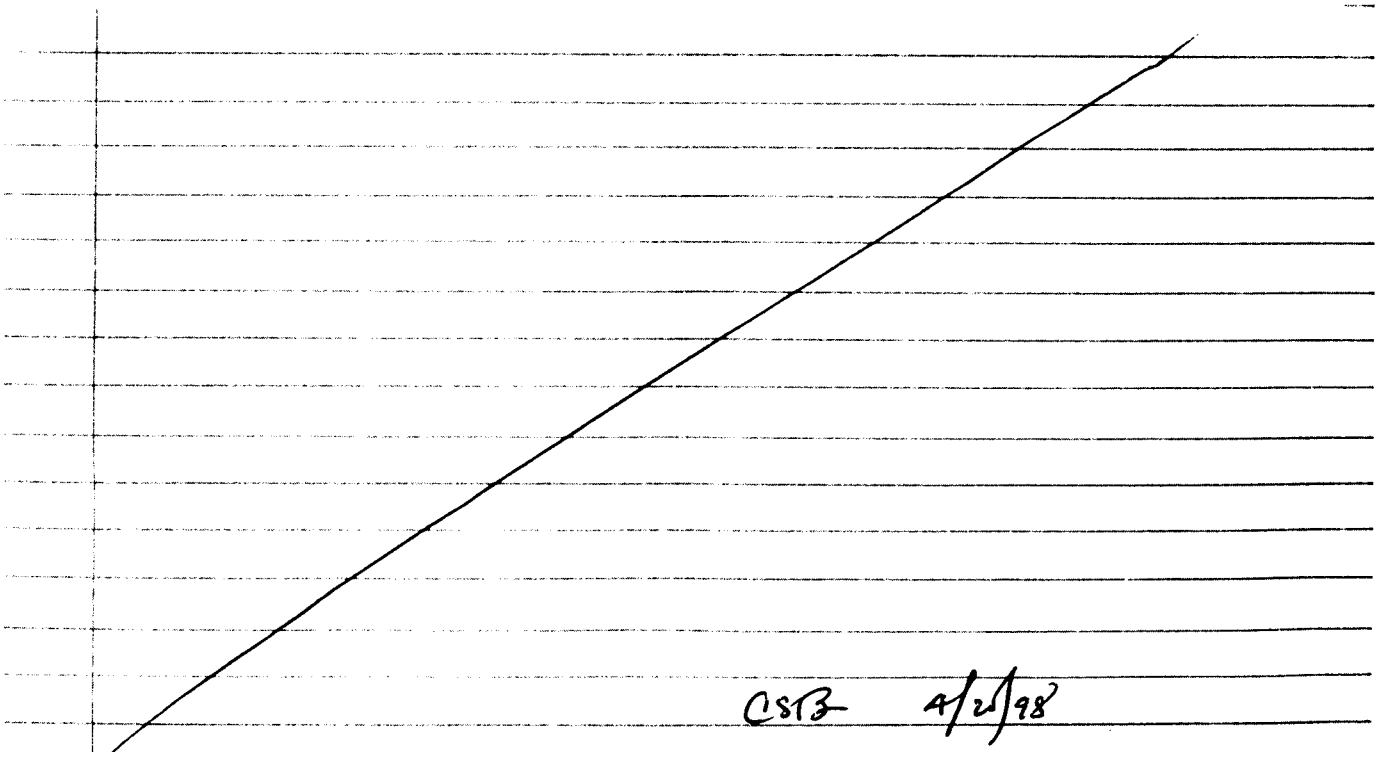
Title: Pit growth in A516 grade 60

Tests Performed by: Sean Brossia; other personnel will be identified as they begin work on specific tests. CSB

Objectives: Determine growth rate of pits in A516 grade 60 as a function of environmental conditions expected in repository. Evaluate if long-term pit propagation likely to continue and lead to perforation. Ascertain if lateral pit growth and/or development of new pits will dominate the corrosion process such that pit penetration arrest occurs. Detailed test objectives identified in initial entries or individual test entries.

Equipment: Specific equipment lists provided in initial entries or in individual test entries. Equipment will be identified and calibrated prior to testing.

Materials: A516 grade 60 carbon steel (Heat #: D84944; composition: 0.03Cr, 0.01Ni, 0.01Mo, 0.02Cu, 1.00Mn, 0.23Si, 0.009S, 0.18C, 0.014P, 0.021Al, 0.001V, 0.001Nb, bal Fe). Other materials and heats to be added and identified prior to testing.



CSB 4/21/98

Screening Tests On Pitting Corrosion of A516 grade 60

Overall Program Title: Pit growth in A516 grade 60

Tests Performed by: Sean Brossia CSBrossia 4/30/98

Objectives: Determine solution composition (pH, [Cl^-]) and temperature that will lead to the development of identifiable individual pits on A516. These compositions will then be used in the long-term (5-year) performance evaluation of this material for use as an overpack in nuclear waste containers -- next phase of this program.

Equipment: ESC/Scribner Associates Multipotentiostat Model 440 (SN9209139); Strawberry Tree data acquisition system consisting of two 8-channel, 16-bit analog input boards and terminal blocks and one 8-channel, 12-bit analog output board and terminal block and a personal computer (Austin 386SX, SN19425) running Strawberry Tree WorkBench PC software (ver 2.1.0); E.G.&G. (Greene) 5-port electrochemical cells and associated glassware and hardware to enable sample exposure, deaeration, a reference electrode and a counter electrode; name heating mantles and controls to perform elevated temperature experiments and thermometer to verify the solution temperature; counter electrodes: Pt foil, Pt mesh, graphite rod; reference electrodes: SCEs (SN5333208, SN5333204, SN3106345)

SN 341151
SN 328185 — Glas-Col PL 112 Control — SN120501A
Glas Col TM 108 Mantle

Materials: A516 grade 60 carbon steel (Heat #: D84944; composition: 0.03Cr, 0.01Ni, 0.01Mo, 0.02Cu, 1.00Mn, 0.23Si, 0.009S, 0.18C, 0.014P, 0.021Al, 0.001V, 0.001Nb, bal Fe);

Test Conditions: The following testing conditions will be investigated:

Set	pH	[Cl^-] (ppm)	E	Temp (°C)	Time (h)
1	9.5 ([HCO_3^-] + [CO_3^{2-}] = 0.012 M)	10 - 1000	E > Erp (E = -600 mVsce)	95	24 - 48
2	9.5 ([HCO_3^-] + [CO_3^{2-}] = 0.012 M)	10 - 1000	E > Erp (E = -525 mVsce)	25	24 - 48
3	6.0	10 - 1000	E > Erp (E = -600 mVsce)	95	24 - 48
4	6.0	10 - 1000	E > Erp (E = -525 mVsce)	25	24 - 48

All solutions will be deaerated by purging with N_2 for at least 30 min prior to testing. All tests will be potentiostatic in nature, using the potentials shown in the above table.

Evaluation: After polarization, the samples will be removed from the test and evaluated for the presence of distinct, individual pits. The depths and diameters of the pits that are present will be measured using a variety of techniques including optical microscopy, micrometer measurements, electron microscopy and cross-sectional analysis.

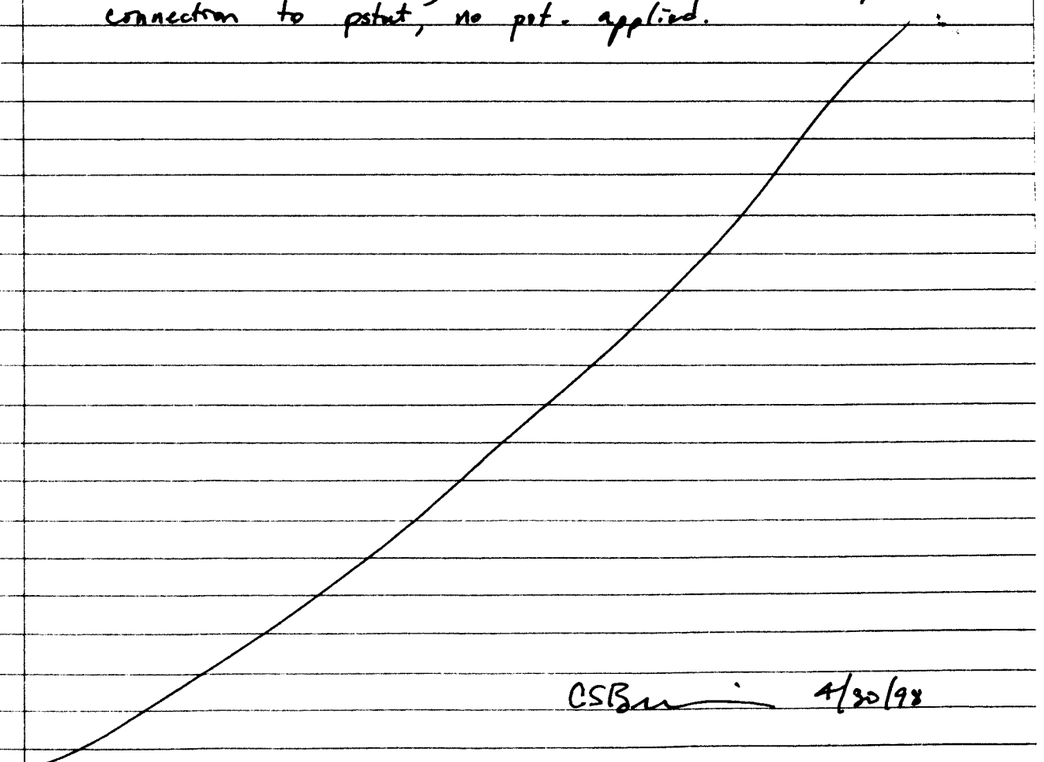
4/30/98

Preliminary testing of exp. set-up
- using 10Ω & 100Ω resistors as "cells" to test and set-up Strawberry Tree DAQ System and Scribner Model 440 Pstat

- using 3 cells

Cell 1 - # 7 pstat slot, # 4 pstat board
Cell 2 - # 3 pstat slot, # 2 pstat board
Cell 3 - # 5 pstat slot, # 3 pstat board

→ cable connections to cell on pstat slot #1 seem to have a problem. Cable doesn't have an open (resistance meas. using Ω-meter was low) but upon connection to pstat, no pot. applied.



CSBrossia 4/30/98

5/1/98 Continuation of DAQ/DSTAT Setup for Screening Tests

Tested 10Ω & 100Ω resistors as loads applying 100 mV via front panel controls.

→ ran overnight from 4/30/98 CSB

→ file names: workbook file Restst3.WBB
data restst1.dat

changed resistors to 10k, 100K & 1MΩ to evaluate min current resolution
Restst2.wbb restst2.dat

→ 1MΩ resistor resulted in "negative" currents
→ sign reversal observed, plus "measured" current too great in magnitude

→ 100K resistors okay - somewhat unstable current readings
→ seems as if min I resolution ~ 0.1 - 1 μA

Pstat	Analog Out signal	Actual E applied (verified w/ Fluke 8050A multimeter SN 25272)	E read w/ DAQ
7	0.091 V	0.100 ✓	
CSB 5			0.0974 V
CSB 53	0.1013183	0.099 V	0.0966 ✓
CSB 35	0.1038 V	0.101 V	0.0983 V

confused about pstat vs cell hook-up. Verified that the above corrections are valid via re-checking and turning pstats off.

CSB 5/1/98

Development of calibration curve for pstats

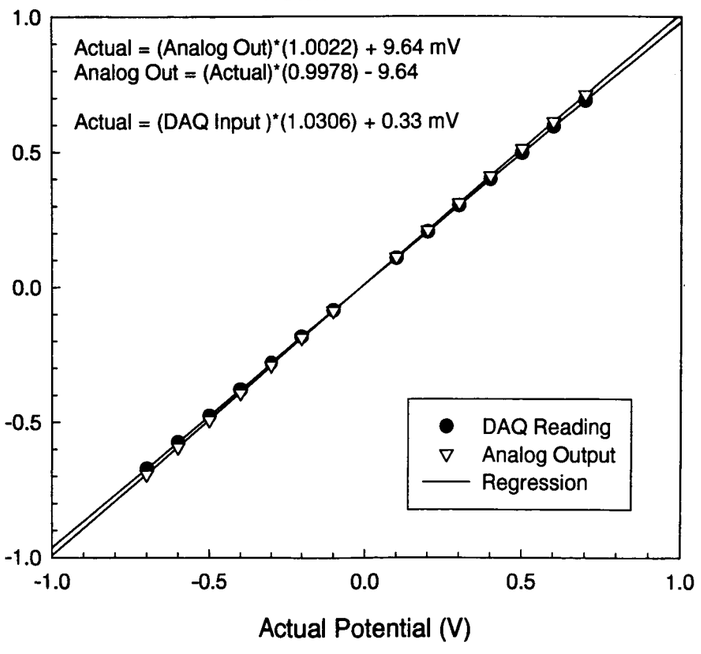
pstat	Analog Out	Actual	DAQ	5/1/98
5	0.100	0.0963	0.0937	
5	0.200	0.196	0.1913	
	0.300	0.297	0.2878	
	0.400	0.397	0.3850	
	0.500	0.497	0.4822	
	0.550	0.546	0.5301	
	0.600	0.597	0.5796	
	0.700	0.697	0.6769	
	-0.100	-0.104	-0.1015	
	-0.200	-0.204	-0.1994	
	-0.300	-0.304	-0.2955	
	-0.400	-0.405	-0.3932	
	-0.500	-0.505	-0.4906	
	-0.550	-0.554	-0.5378	
	-0.600	-0.605	-0.5877	
	-0.700	-0.705	-0.6850	
3	0.100	0.0990	0.0966	
	0.200	0.199	0.1940	
	0.300	0.299	0.2903	
	0.400	0.399	0.3873	
	0.500	0.498	0.4842	
	0.600	0.599	0.5814	
	0.700	0.699	0.6785	
	-0.100	-0.101	-0.0982	
	-0.200	-0.201	-0.1959	
	-0.300	-0.301	-0.2919	
	-0.400	-0.401	-0.3894	
	-0.500	-0.502	-0.4866	
	-0.600	-0.601	-0.5834	
	-0.700	-0.701	-0.6805	
7	+0.100	0.100	0.1069	
	0.200	0.210	0.2045	
	0.300	0.310	0.3010	
	0.400	0.410	0.3982	
	0.500	0.511	0.4953	
	0.600	0.611	0.5926	
	0.700	0.711	0.6900	

SB 5/1/98

cont on p 12

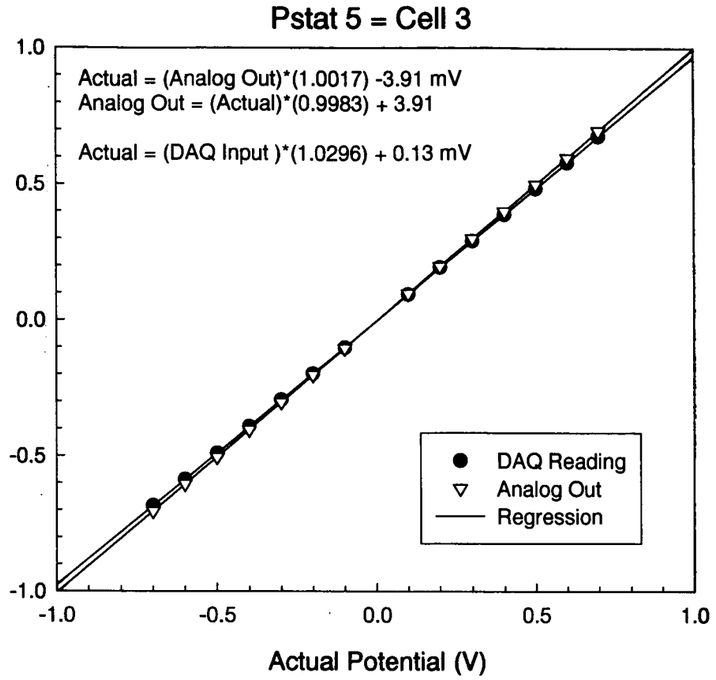
cont from p" 5/1/98

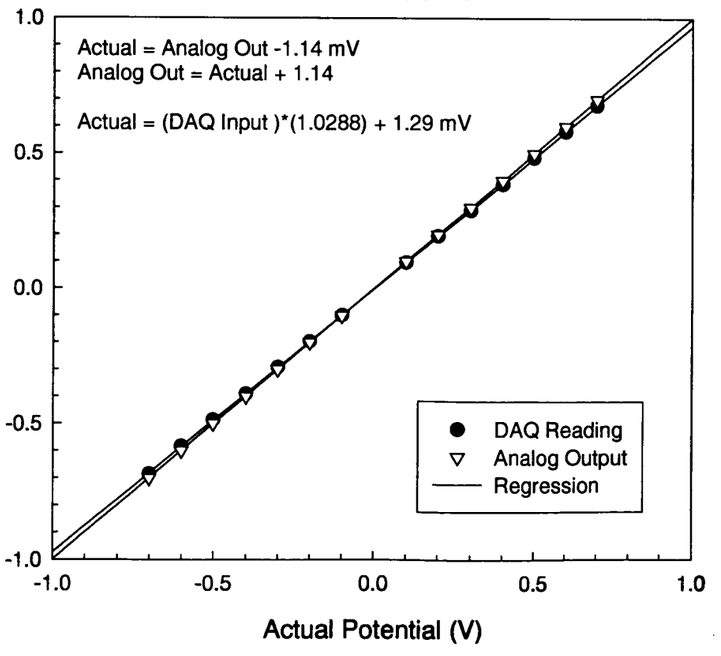
pstat	Analog Out	Actual	DAQ
7	-0.100	-0.090	-0.0881
	-0.200	-0.190	-0.1859
	-0.300	-0.291	-0.2820
	-0.400	-0.391	-0.3798
	-0.500	-0.492	-0.4772
	-0.600	-0.592	-0.5741
	-0.700	-0.692	-0.6713



CSR 5/1/98

5/1/98





CSR 5/1/98

5/4/98 Continued setup cells. Ran N_2 lines & worked on heating mantles. Also put together 2 salt bridges for the heated cell RE's. Used strong & glass wool.

CSB 5/4/98

5/5/98

screening tests
AS16 material - 8 cm^2 area exposed

Test - S9T1.DAT - set 1 = pH 9.5 temp: 95°C
E = -600 mVsce test 1 = 1000 ppm Cl^- RE: 7079122
↳ ~~Blank~~ CSB cell 2 ⇒ output = -0.5986 CE: Pt foil

Test - S2T1.DAT - set 2 = pH 9.5 RE: 7079123 CSB 5/5/98
test 1 = 1000 ppm CE: Pt ~~foil~~ mesh
Cell 1 temp = 21°C
E = -525 mVsce
↳ output = -0.5335

Test S1T2.DAT set 1 = pH 9.5 RE: 5144349
test 2 = 100 ppm Cl^- CE: graphite rod
Cell 3 temp 95°
E = -600 mVsce
↳ output = -0.5951

Solutions for above
0.5 mM Na_2CO_3 = 0.10599 g/2L Fisher Lot # 960685
11.5 mM $NaHCO_3$ = 1.93223 g/2L Fisher Lot # 897789

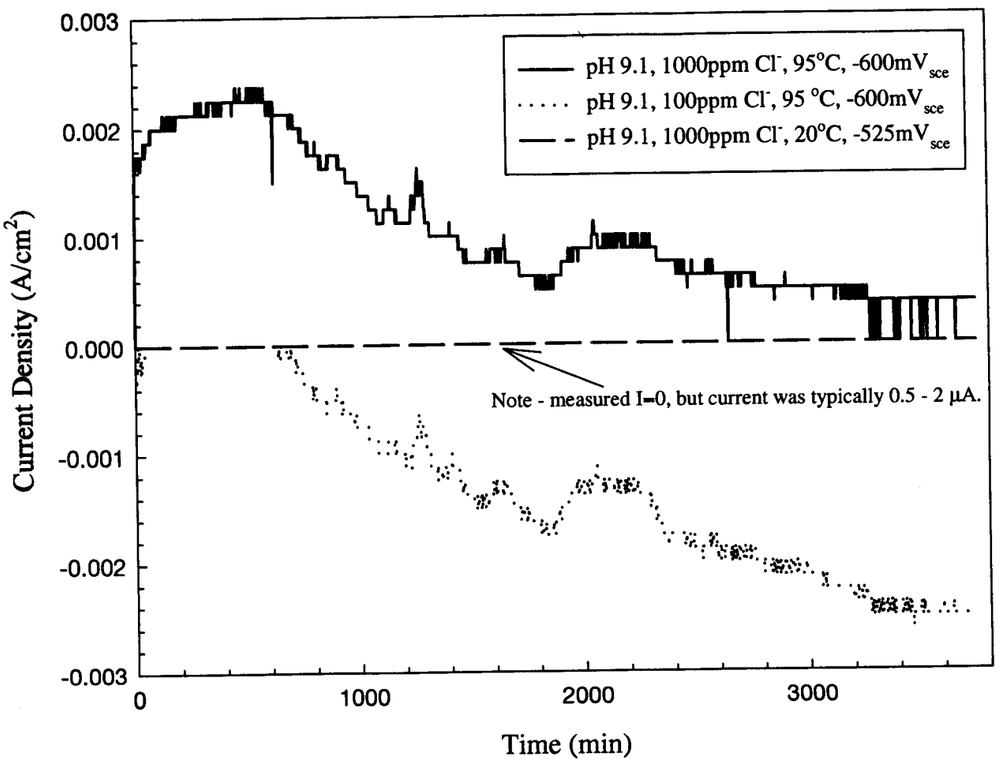
Cl^- added as NaCl Fisher Lot # 972274
100 ppm Cl^- = 0.0823 g/0.5L
1000 ppm Cl^- = 0.823 g/0.5L

DAQ potentials have been corrected

Purged w/ N_2 for 30 min
Samples polished to 600 grit

SB 5/5/98

5/8/98



samples saved ; the corrosion products will be analysed using Raman Spectroscopy

5/8/98

5/8/98

Since samples in previous 3 tests did not pit, going to examine alt. pH & potentials

→ All samples polished to 600 grit & ultrasonically cleaned w/ acetone

Cell 1 : S5T1.DAT

pH = 11.047 (12 mM Na_2CO_3) – Fisher #960685

Cl = 50 ppm → 0.1648 g/2L → 2.54376 g/2L

area = 8 cm² T = 20 °C Fisher #972274

-508.54 ← E = -500 mVsce – ~~Analog out programed~~ CSB 5/8/98 & Analog in programed to compensate for error

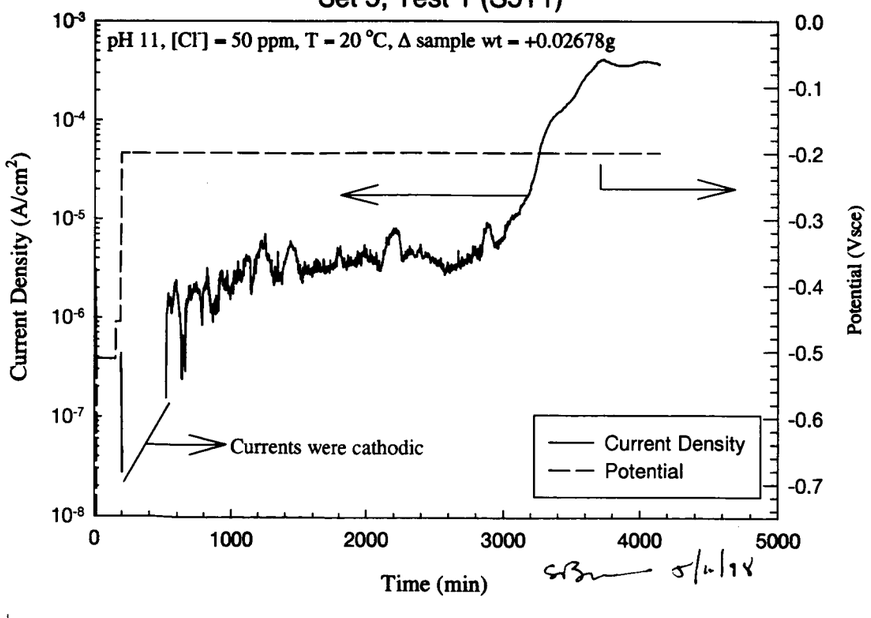
sample initial wt = 11.41334 g

sample final wt = 11.44012 g 5/11/98 SB

initial pH = 11.047

final pH = 11.138 5/11/98 SB

observations – 205 pm, sample "gun metal gray", & cathodic kicked up pot to -450 mV – current went anodic for about 10 s then cathodic again; 240 pm – incr. pot. to -350 mVsce SB 5/8/98 – still cathodic, increased to -300 mVsce still dropping to cathodic currents – increase to -250 mVsce still dropping to cathodic → increased E_{set} to -200 mVsce



Con't from p 18 5/8/98

Cell 2 : S5T2.DAT

HCO_3/CO_3 = 12 mM Na_2CO_3 area = 8 cm²

Cl = 50 ppm Cl

T = 95 °C

E = -600 mVsce → -598.86 mV = Analog Out

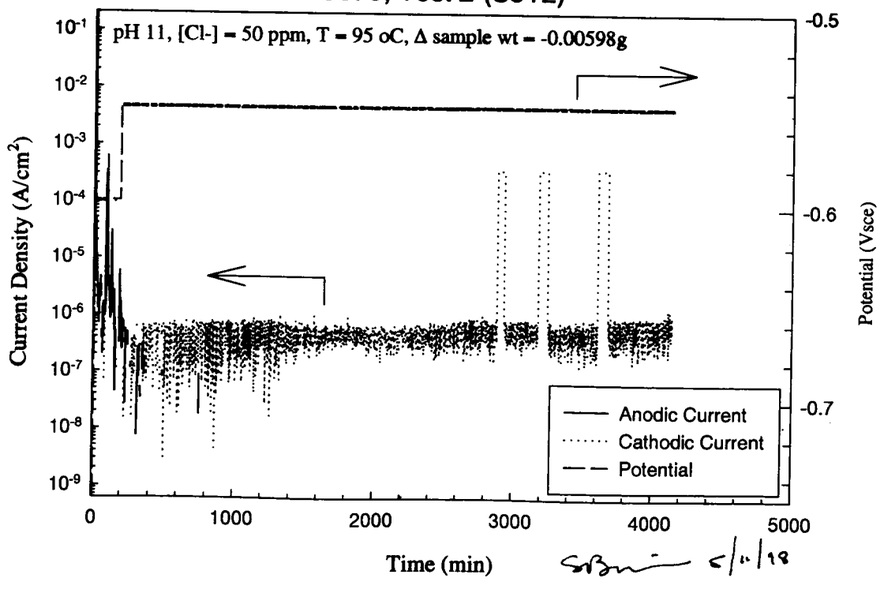
sample init wt = 11.41370 g CSB 5/8/98

sample final wt = 11.40772 g 5/11/98 SB

init pH = 11.047

final pH = 10.549 5/11/98 SB

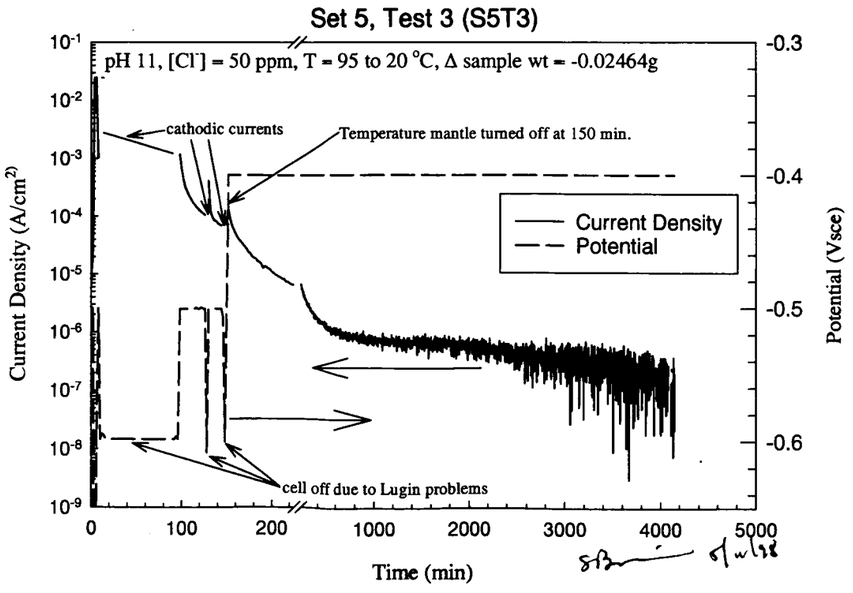
observations – at 12:00 showed evidence of rust spots; at 205 pm, rust spots now black but still distinct individuals, 240 pm – currents starting to fluctuate btwn anodic & cathodic → increased potential to -550 mVsce



cont from p 19 5/8/98

Cell 3: S5T3.DAT

HCO_3^-/CO_3^{2-} = 12 mM CO_3^{2-} area = 8 cm^2
Cl^- = 50ppm
T = 95°C
E = -500 mV$_{sce}$ ⇒ -495.24 mV = Analog Out
sample init wt = 11.44960
sample final wt = 11.42496 5/11/98 SBS
init pH = 11.047
final pH = 10.785 5/2/98 SBS
Observations:
Experienced Lugin problem - no control over potential turned off while repairing - could not repair - used regular Lugin until temp too high. Turned off temp mantle, re-set pot to ~ -400 mV$_{sce}$ @ 2105 pm 5/8/98 and continued expt. using reg Lugin probe



SBS 5/11/98

> New solution compositions for screening tests 5/18/98

Due to difficulty in getting samples of A516 to pit in solns w/ pH ~9.1, a new test matrix has been designed as follows:

pH	[Cl⁻] ppm	Temp	E_{rp}*	E_{set}	*$E_{rp}^{95C,pH11} = -622.9 - 40.2 \log[Cl^-]$
11	10	95	-480	-380	$E_{rp}^{95C,pH9.5} = -576 - 11.6 \log[Cl^-]$
	10	95	-480	-280	from Cragnolino et al,
	10	20	?	-280	Corrosion/98
	100	95	-520	-420	
	100	95	SBS 5/18/98 -520	-320	$E_{set}^1 = E_{rp} + 100$ mV
	100	20	?	-320	$E_{set}^2 = E_{rp} + 200$ mV
	1000	95	-560	-460	$E_{set}^{25C} = E_{set}^2$
	1000	95	-560	-360	
	1000	20	?	-360	
9.5	10	95	-534	-434	
	10	95	-534	-334	
	10	20	?	-334	
	100	95	-546	-446	
	100	95	-546	-346	
	100	20	?	-346	
	1000	95	SBS -558	SBS -458	5/18/98
	1000	95	SBS -558	SBS -358	
	1000	20	?	-358	
12.5	100	95	?	-420	
	100	95	?	-320	
	100	20	?	-320	

SBS 5/18/98

Cont on p 22

5/18/98

Stock solution

cont from p21

pH 11 → 12 mM Na_2CO_3 ⇒ 2.54376 g/2L Fisher Lot # 960685

CSB 5/18/98

10 ppm Cl ⇒ 0.03295B g/2L Fisher Lt # 972274

initial pH = 11.013

All samples polished to 600 grit, ultrasonically cleaned in Acetone

SB 5/18/98

Cont to p.23

from p 22

CELL 1: A516PS01.DAT 5.1.8/98

Temp = 20°C

E_{set} = -280 mV sce ⇒

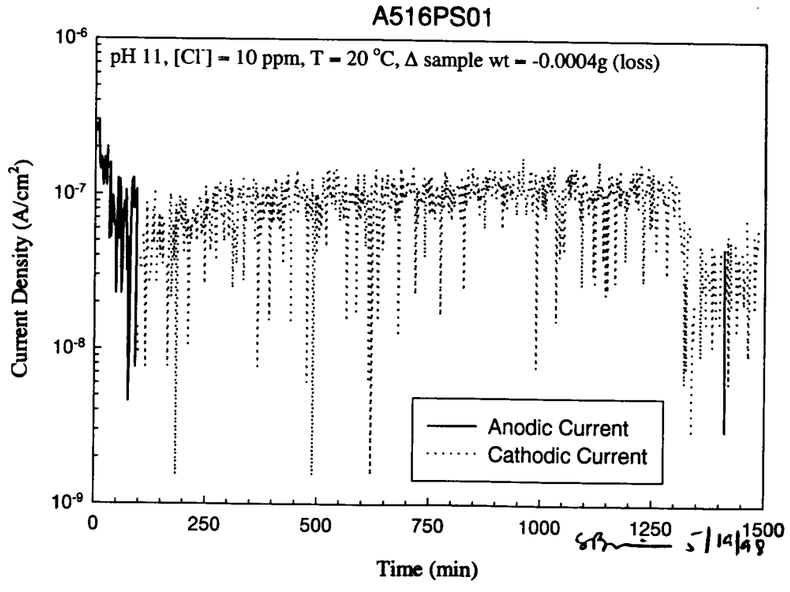
init. sample wt = 11.43081

final sample wt = 11.43045 5/18/98 CSB

final soln pH = 11.246 5/14/98

observations no observable pitting 5/15/98

Steve Clay Div 6 was added to project



SB 5/14/98

to p 24

from p 23

5/18/98 CELL 2: A516PS02.DAT

Temp = 95 °C

E_{sct} = −380 mV$_{sce}$

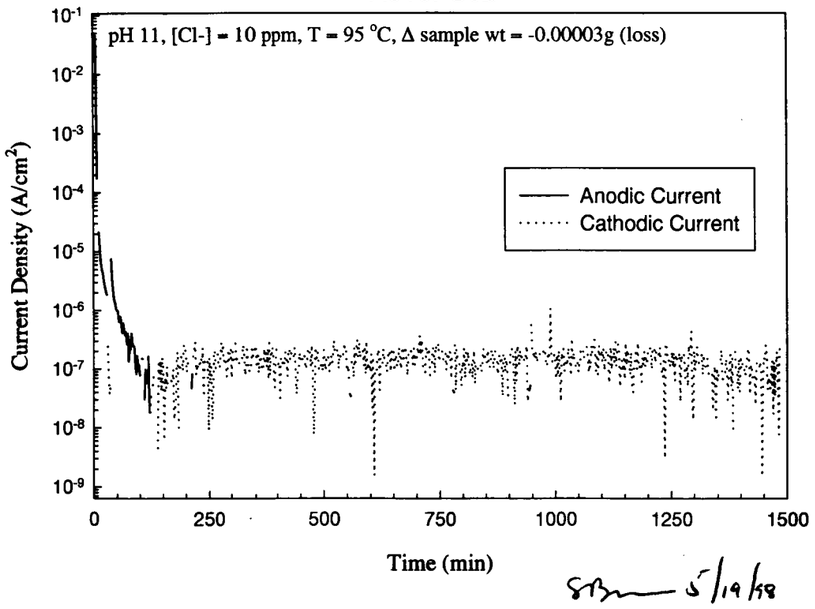
init sample wt = 11.42596

final sample wt = 11.42593 5/19/98 SB

final soln pH = 10.993 5/19/98 SB

observations - Lugin / salt bridge problem
extensive bottom pitting 5/19/98 SB
a few small pits on sides

A516PS02



5/19/98

to p25

5/18/98

from p25

CELL 3: A516PS03.DAT 5/18/98

Temp = 95°C

E_{sct} = −250 mV$_{sce}$

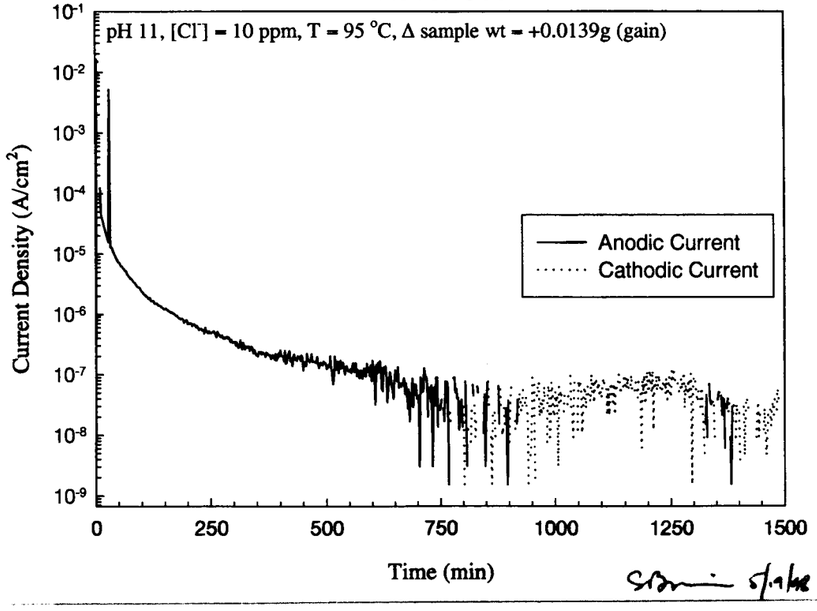
init wt = 11.38749

final sample wt = 11.40141g 5/19/98 SB

final soln. pH = 11.153 5/19/98 SB

observations - extensive pitting on sides especially at scratch areas 5/19/98 SB
bottom fully rusted with a few dark pits

A516PS03



5/19/98

5/18/98

From 25

5/19/98

Stock Solution

PH = 11 → 12 mm Na_2CO_3 => 2.54376 g/2L Fisher Lot # 960685

100 ppm Cl => 0.32958 g/2L Fisher Lot # 972274

Initial PH = 11.251

All samples polished to 600 grit + ultrasonically cleaned in Acetone

Start

Change File Names for new spec

Check pulses for 2 hour

System change Copy + to

to new file names

Change Cell potentials

Note - ALT - Left Mouse Click

turns on ICON

Right Click File Run

Turn on Potentiostat

Shut down

Double Click each Pulse for each Cell

change to 2 minutes low duration

After both drives A+C are

accessed Right Click Select

Stop

ALT Left Click File Iron

+ System Iron

5/19/98

Cont to 27

from 26

A516PS04.DAT

Temp = 20°C

Eset = -320 mV

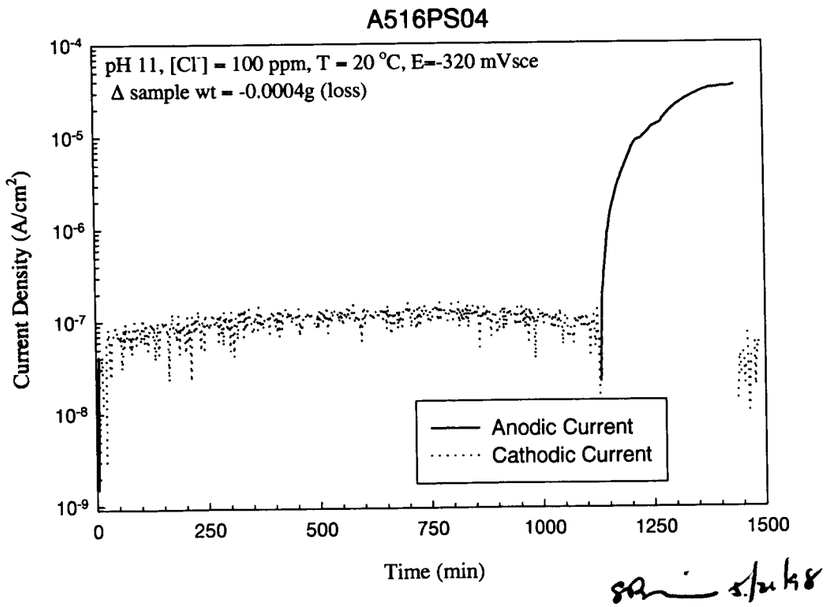
Init Sample wt = 11.42389 g

Final Sample wt = 11.42385 g 5-20-98

Final Solution PH = Dumped before ph taken 5-20-98

Observations Nitrogen low at end of test

Water line buildup on specimen 5-20-98



5/21/98

5/19/98

Cont to 28

from 27

Cell 2 A516PS05.DAT

Temp = 95°C

Eset = -420mv

Init Sample wt = 11.40679 g

Final Sample wt = 11.40632 g

Final Solution PH = 10.968

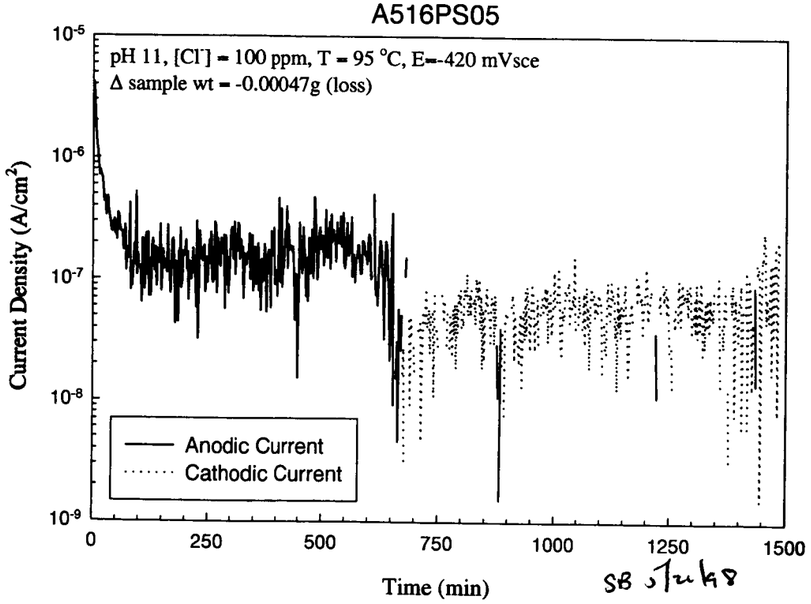
Observations Water line film on specimen, rusting above waterline

No noticeable pitting

5-20-98

5-20-98

5-20-98



SB 5/14/98

5/19/98

Cont to 29

from 28

Cell 3 A516PS06.DAT

Temp = 95°C

Eset = -320mv

Init Sample wt = 11.40362

Final Sample wt = 11.39731

Final Solution PH = 10.795

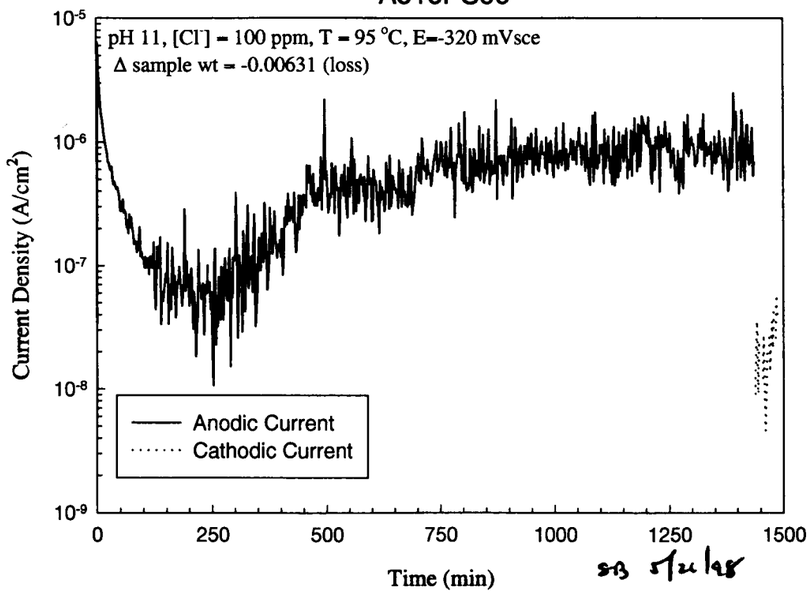
Observations Water line film + rusting above waterline

No noticeable pitting

5-20-98

5-20-98

5-20-98



SB 5/14/98

5/19/98

Cont to 30

from 29

5-20-98

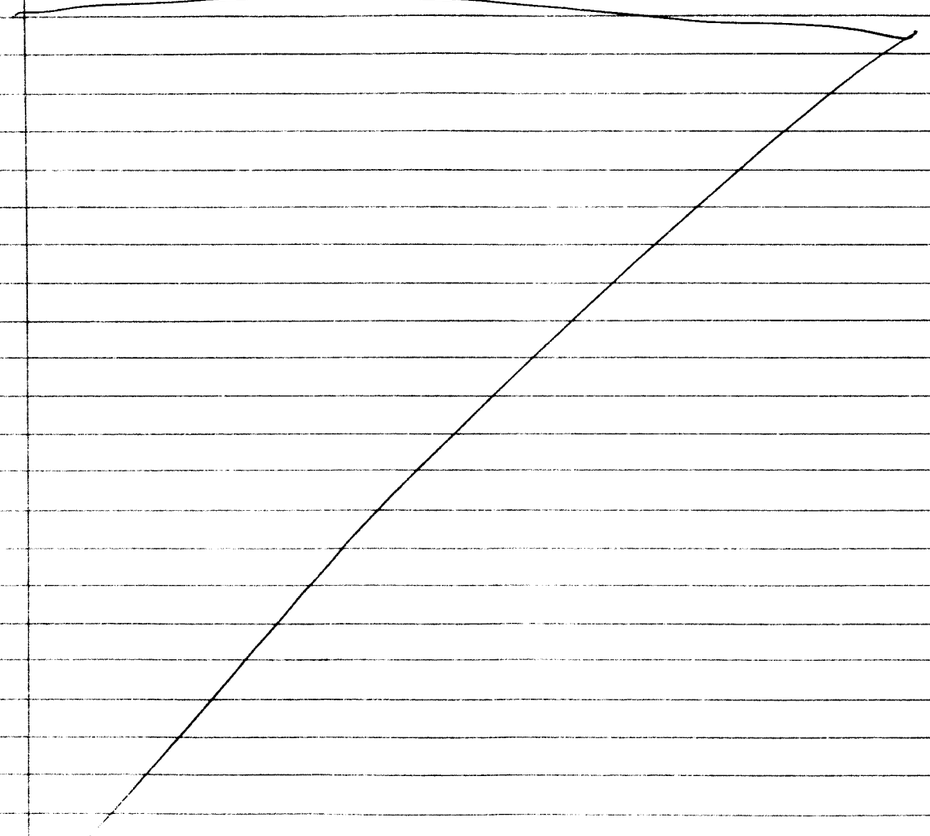
Stock Solution

PH = 11 → 12 mm Na_2CO_3 => 2.54376 g/2L Fisher Lot 960685

1000ppm Cl => 3.2958 g/2L Fisher Lot 972274

Initial PH = 11.205 5-21-98

All Samples ultrasonically cleaned in Acetone after polishing to 600 grit



5-20-98

Cont to 31

from 30

Cell 1 A516PS07.DAT

Temp = 20°C

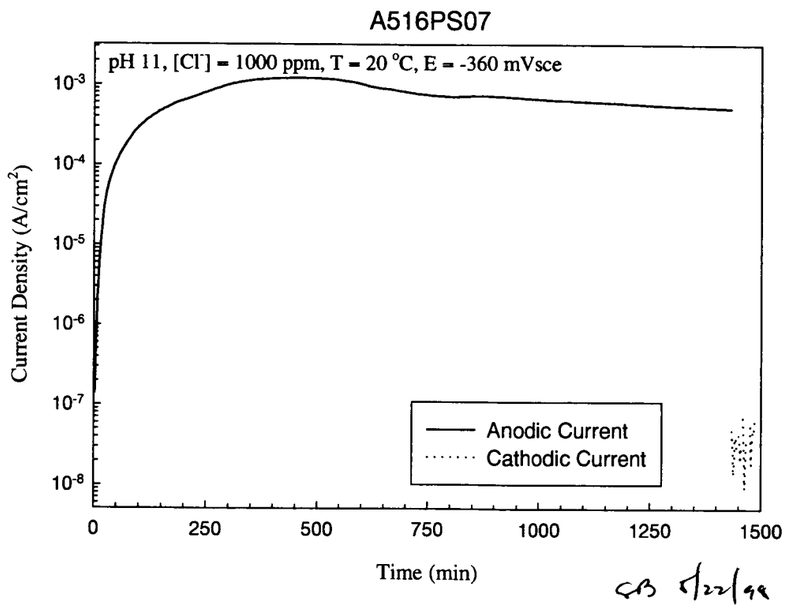
Esct = -360mV

Init Sample wt = 11.39714

Final Sample wt = 11.25644 5-22-98

Final Solution PH = 11.717 5-22-98

Observations Heavy product buildup, some pitting 5-22-98



CB 5/22/98

5-20-98

Cont to 32

from 31

Cell 2 A516PS08.DAT

Temp = 95°C

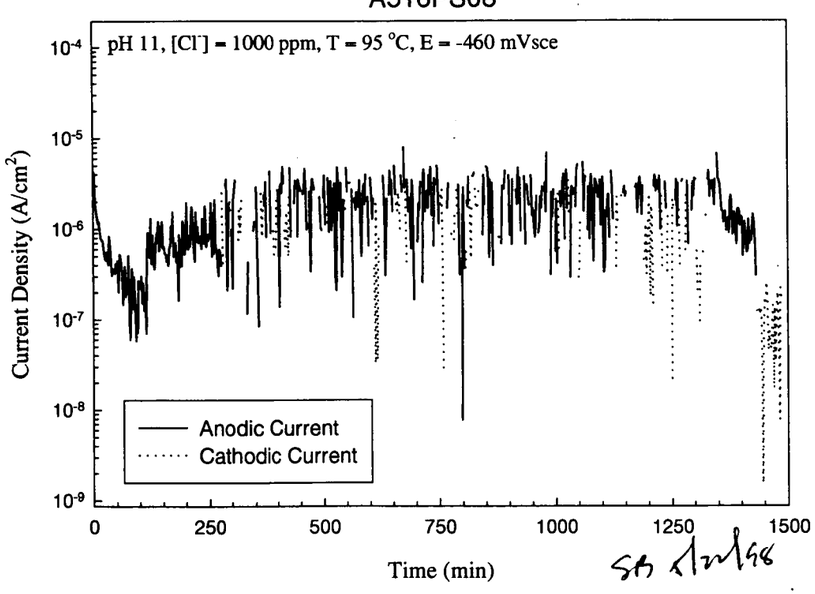
Eset = -460mv

Init Sample wt = 11.46693

Final Sample wt = 11.46901 5-22-98

Final Solution pH = 11.159 5-22-98

Observations No pitting, slight rusting above solution line 5-22-



5-22-98

5-20-98 Cont to 33

from 32

Cell 3 A516PS09.DAT

Temp = 95°C

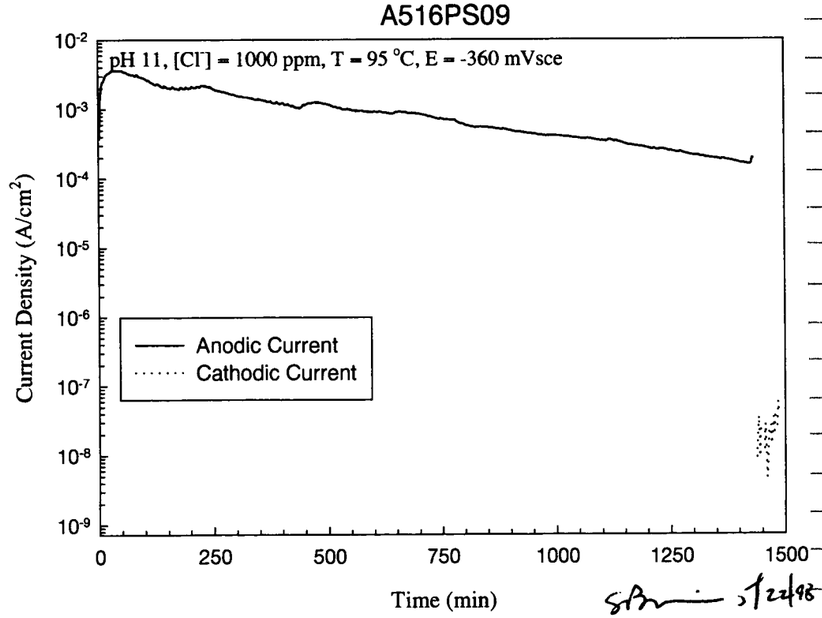
Eset = -360mv

Init Sample wt = 11.44650

Final Sample wt = 11.29136 5-22-98

Final Solution pH = ~~11.317~~ 5-22-98 10.822 5-22-98

Observations Heavy product buildup, rust + pitting, slight rust above solution line 5-22-98



5-22-98

5-20-98

5/25/98

Attempt to make pH 9.5 solution

		100 mL of soln	measured pH
#1	12 mM Na_2CO_3 – 105.986 g/mol	0.1272 g	10.918
	1 mM $NaHCO_3$ – 84.0049 g/mol	0.0084 g	
#2	12 mM Na_2CO_3	0.1272 g	11.018
	0.1 mM $NaHCO_3$	0.00084 g	
		actual 0.00140	
#3	11 mM Na_2CO_3	0.1166 g	11.040
#4	10 mM Na_2CO_3	0.1060	10.880
	1 mM $NaHCO_3$	0.0084	
#5	10 mM Na_2CO_3	0.1060	10.765
	2 mM $NaHCO_3$	0.0168	

After discussion w/ N. Sridhar and G. Cragnolino, key issue seems to be CO_3^{2-}/Cl^- ratio to generate conditions that lead to pitting. Ratios that have worked to some extent thus far:

CO_3^{2-}	Cl^-	ratio
12 mM	1.41 mM (50 ppm)	8.51
12 mM	0.282 mM (10 ppm)	42.6 – best case

ratios that led to general corrosion

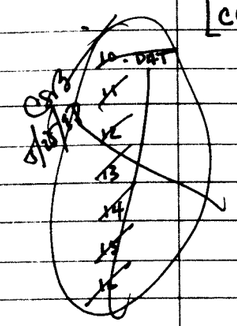
CO_3^{2-}	Cl^-	ratio
11.5 mM	2.82 mM (100 ppm)	4.1
11.5 mM	28.17 mM (1000 ppm)	0.41
12 mM	2.82 mM	4.3
12 mM	28.17 mM	0.43

SRB 5/26/98

cont on p. 35

from p 34

From tests so far given that CO_3^{2-}/Cl^- ratio is critical parameter, following experiments are proposed 5/25/98



$[CO_3^{2-}]$ as Na_2CO_3	ratio	$[Cl^-]$ mM	ppm	g NaCl/2L
12 mM	6:1	2	71	0.233768
	8:1	1.5	53	0.175326
	10:1	1.2	42.6 OSR 5/25/98	0.14026
	20:1	0.6	21	0.07013
	30:1	0.4	14	0.046754
	50:1	0.24	8.5	0.028052
	75:1	0.16	5.7	0.018701
	100:1	0.12	4.3	0.014026
	500:1	0.024	0.85	0.002805
	1000:1	0.012	0.425	0.0014026

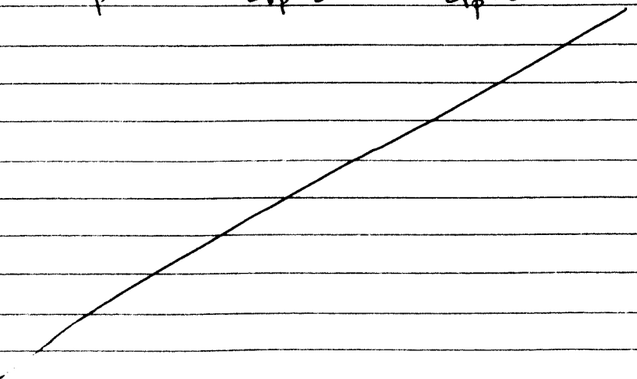
make from stock solution:

0.48 M solution (1 L = 0.02805 g NaCl)

0.024 mM final $[Cl^-]$ in 2L → 100 mL stock Cl^- soln

0.012 mM final $[Cl^-]$ in 2L → 50 mL stock Cl^- soln

	potentials			
pH	E_{rp}	$E_{set}^{95°C}$	$E_{cut}^{95°C}$	$E_{cut}^{25°C}$
6:1	−514	E_{rp} + 100	E_{rp} + 200	E_{rp} + 200
8:1	−509			
10:1	−505			
20:1	−493			
30:1	−486			
50:1	−477			
75:1	−470			
100:1	−465			
500:1	−437			
1000:1	−425			



SRB 5/26/98

Col Pg 36

from 35

5-26-98

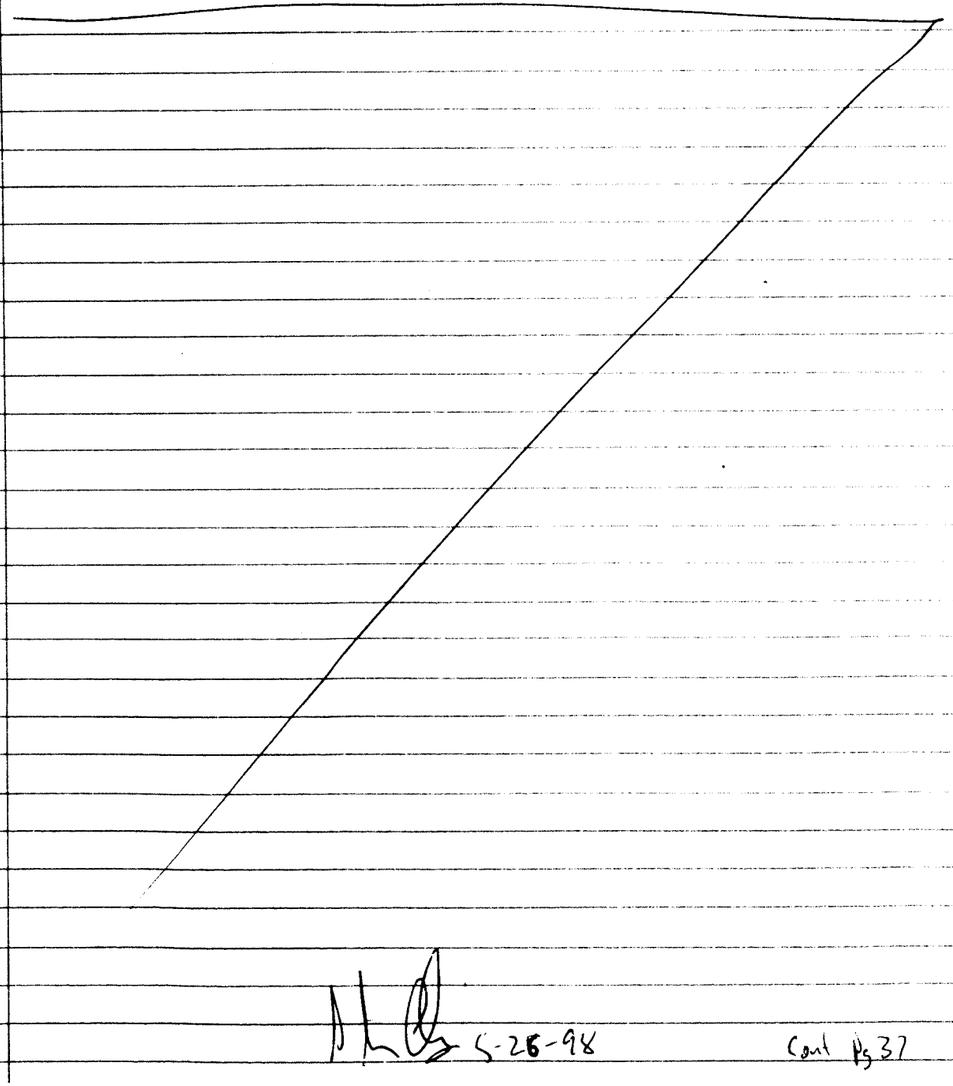
Stock Solution-

PH = 11 → 12mm Na_2CO_3 => 2.54376 g/2L Fisher Lot 960685

→ 71ppm Cl => .233768 g/2L Fisher Lot 972274

Initial PH = 11.075 5-25-98

All samples polished to 600 grit & cleaned w/ freon in Ultrasonic



5-26-98

Cont Pg 37

from 36

Cell: A516PS10.DAT

Temp = 20°C

Eset = -314mV

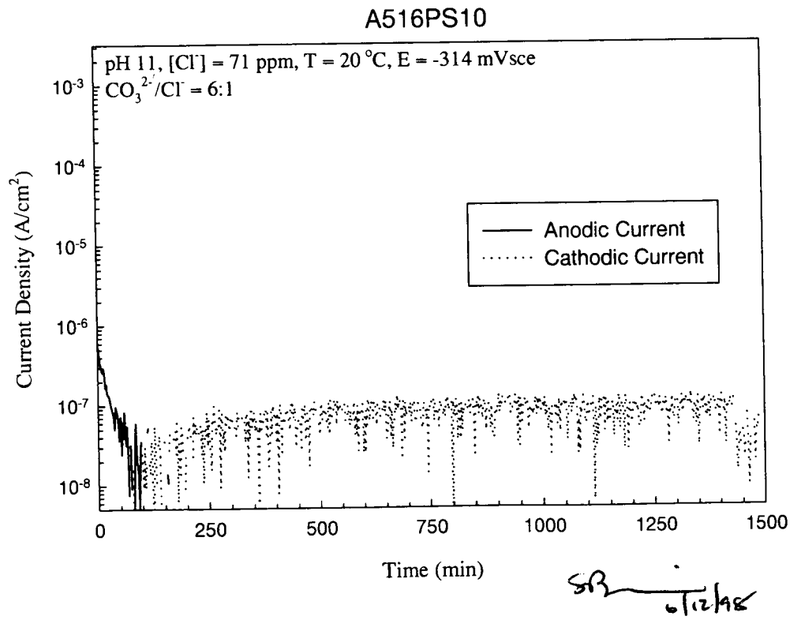
Init Sample wt = 11.44881g

Final Sample wt = 11.44732g M.B 5/27/98

Final Solution PH = 11.030 M.B 5/27/98

Observations

No corrosion/pitting evident on entire sample



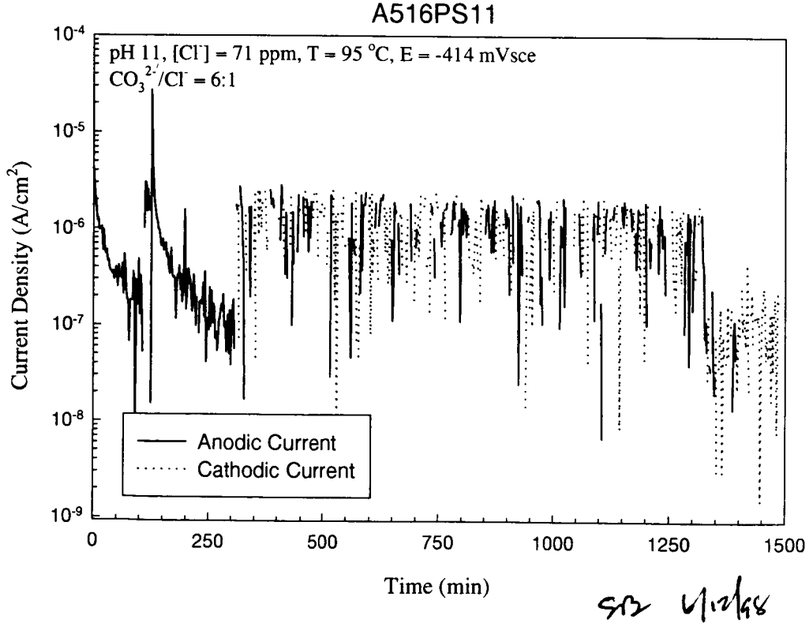
SB 6/12/98

5-26-98

Cont Pg 38

from 37 Cell 2 A516PS11.DAT
Temp: 95°C
Eset = -414 mv
Init Sample wt = 11.40642 g
Final Sample wt = 11.40461 g M.Byfo 5/27/98

Final Solution PH = 11.213 M.Byfo 5/27/98
Observations vapor phase & water line attack

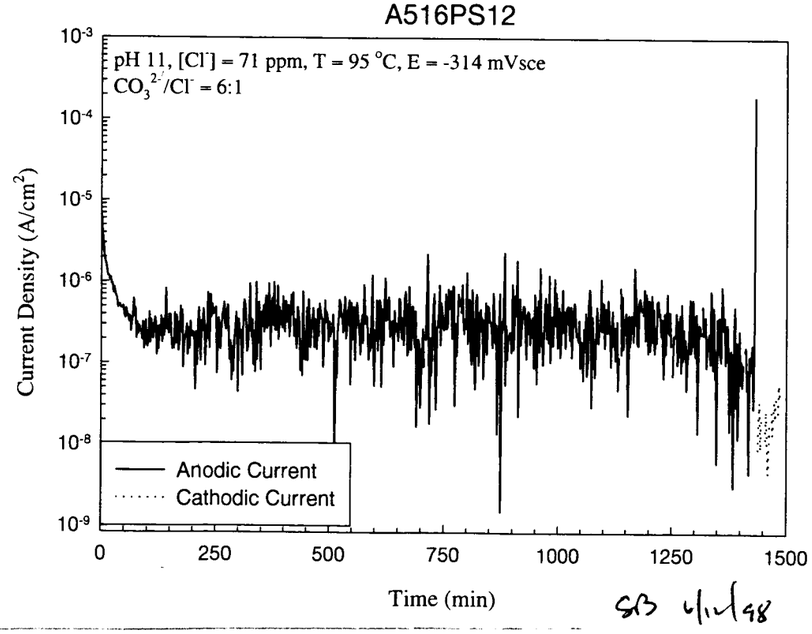


SRB 6/1/98

5-26-98 Cont pg 39

from 38 Cell 3 A516PS12.DAT
Temp = 95°C
Eset = -314 mv
Init Sample wt = 11.38665 g
Final Sample wt = 11.38572 g M.Byfo 5/27/98

Final PH of Solution = 11.271 M.Byfo 5/27/98
Observations vapor phase and water line attack



SRB 6/1/98

5-26-98 Cont pg 40

from 39

5-28-98

Start Solution

pH-11 → 12 mm Na_2CO_3 => 2.54376 g/2Lt Fisher Lot 960685

1 → 53 ppm Cl => .175326 g/2Lt Fisher Lot 972274

Initial PH = 11.058 5-28-98

All Samples polished to 600 grit & ultrasonicated, cleaned in Acetone

5-28-98 Cont 41

from 40

Cell 1 A516PS13.DAT

Temp = 20°C

Eset = -309 mv

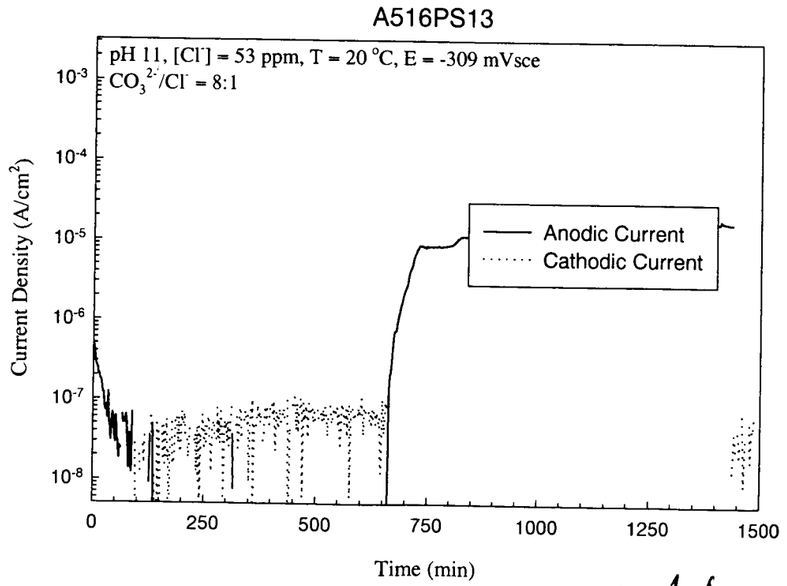
Init Sample wt = 11.45128 g 5-29-98

Final Sample wt = 11.45098 g 5-29-98

Final Solution PH = 11.058 5-29-98

Observations

No pitting, slight buildup at vapor line 5-29-98



GB 6/12/98

5-28-98 Cont 42

from 41

Cell 2 A516PS14.DAT

Temp = 95°C

Eset = -409 mv

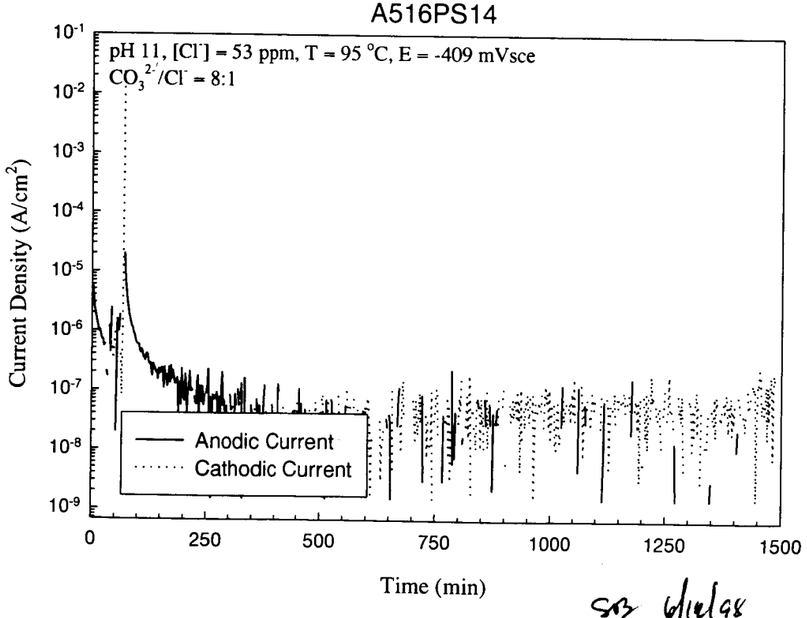
Initial Sample wt = 11.40596g

Final Sample wt = 11.40569g 5-25-98

Final Solution PH = 10.732 5-29-98 5-29-98

Observations

No Pitting, slight vapor changes 5-29-98



SB 6/1/98

5-28-98

Cont 43

from 42

Cell 3 A516PS15.DAT

Temp = 95°C

Eset = -309 mv

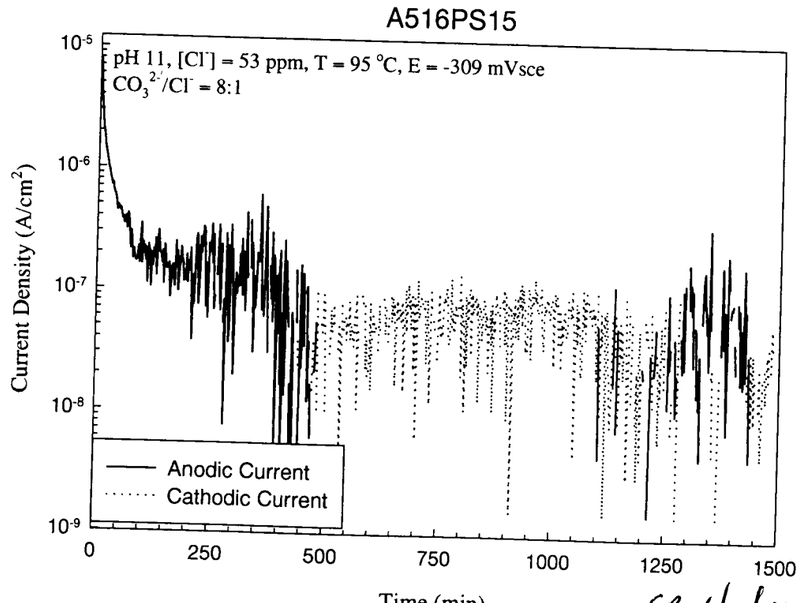
Initial Sample wt = 11.46107g

Final Sample wt = 11.46065g 5-29-98

Final Solution PH = 11.102 5-29-98

Observations

No Pitting, slight vapor attack 5-29-98



SB 6/1/98

5-28-98

Cont 44

from 43

6-1-98

Stock Solution

PH=11 → 12 mm Na_2CO_3 => 2.54376g/2Lt Fisher Lot# 960685

→ 42ppm Cl => .14026g/2Lt Fisher Lot# 972274

Initial PH= 11.071 6-1-98

All Samples polished to 600grit + ultrasonically cleaned in Acetone

6-1-98

Cont 45

from 44

Cell 1 A516PS16.DAT

Temp = 20°c

Eset = -305mv

Init Sample wt = 11.37783g 6-1-98

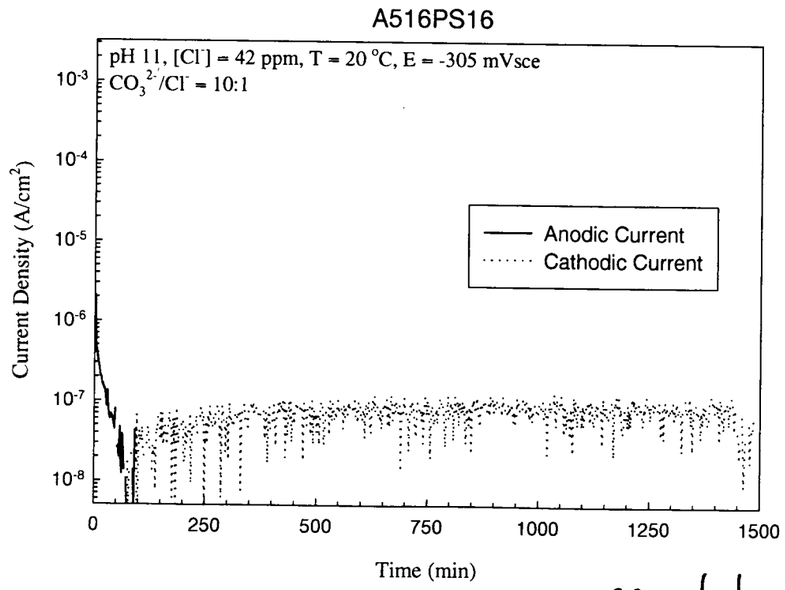
Final Sample wt = 11.37754g 6-2-98

Final Solution pH = 10.842 6-2-98

Observations

No Pitting or changes noticed below vapor line

some small marks in vapor area 6-2-98



SB 6/12/98

6-1-98

Cont 46

from 45 Cell 2 A516PS17.DAT

Temp = 95°C

Eset = -405 mv

Init Sample wt = 11.30289g 6-1-98

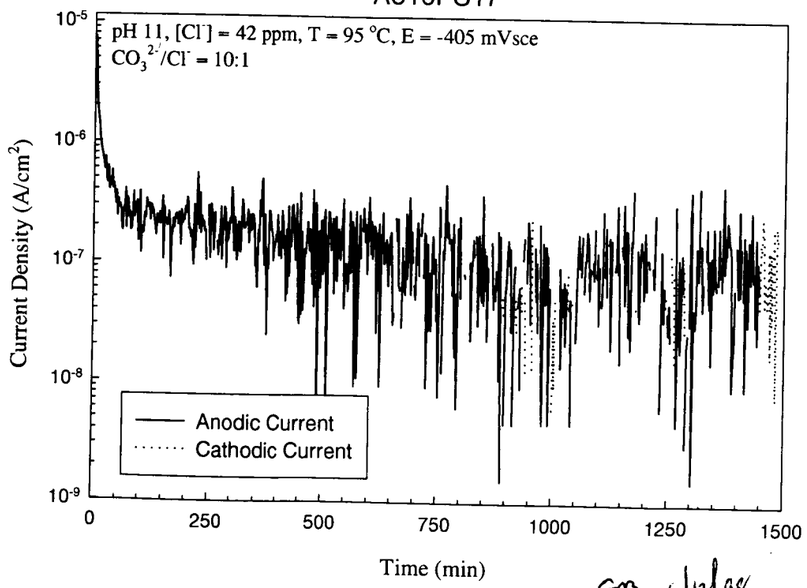
Final Sample wt = 11.29953g 6-2-98

Final Solution pH = 10.770 6-2-98

Observations

No pitting noted below vapor line, discoloration in vapor area 6-2-98

A516PS17



SAS 6/12/98

6-1-98 Cont 47

from 46 Cell 3 A516PS18.DAT

Temp = 95°C

Eset = -305 mv

Init Sample wt = 11.41842g 6-1-98

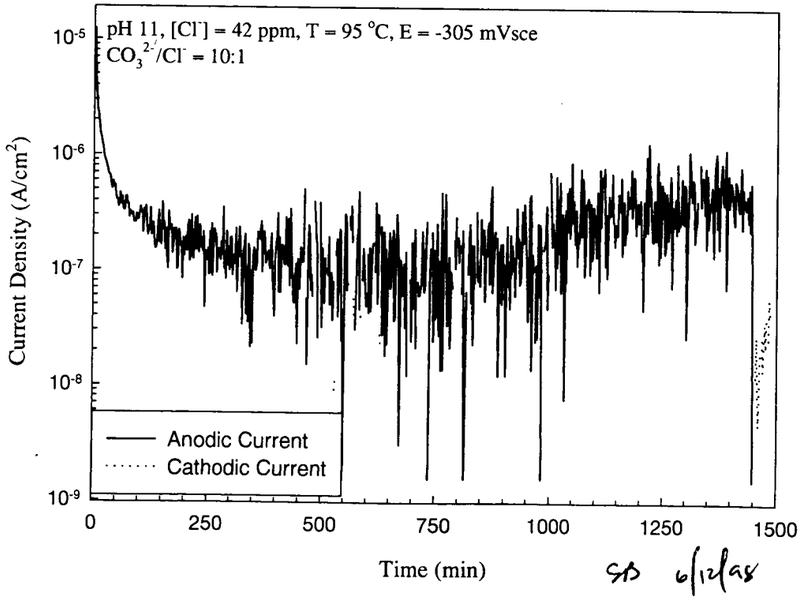
Final Sample wt = 11.41528g 6-2-98

Final Solution pH = 10.861

Observations

No pitting noted below vapor line, discoloration and slight rusting in vapor area 6-2-98

A516PS18



SAS 6/12/98

6-1-98 Cont 48

from 47

6-2-98

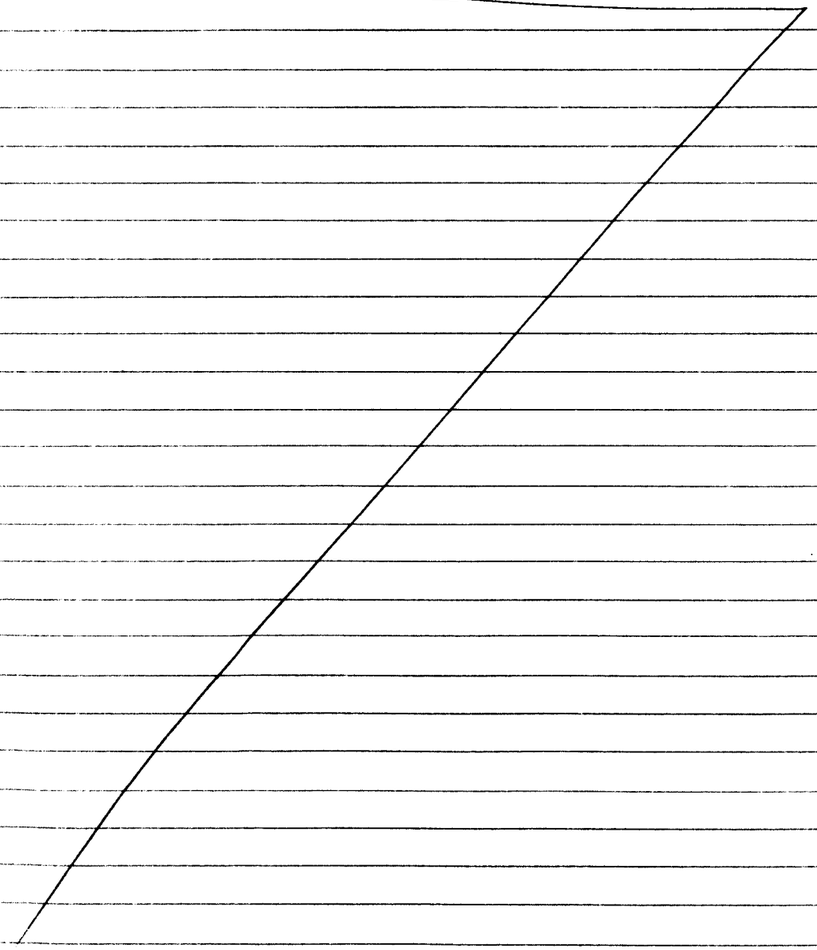
Stock Solution

PH=11 → 12mm Na_2CO_3 => 2.54376 g/2Lt Fisher Lot# 960685

→ 21 ppm Cl => .07013 g/2Lt Fisher Lot# 972274

Initial PH= 11.047 6-2-98

All Samples polished to 600 Grit + ultrasonically cleaned in Acetone



6-2-98

Cont 49

from 48

Cell1 A516PS19.DAT

Temp = 20°c

Eset = -293mv

Init Sample wt = 11.38535g 6-2-98

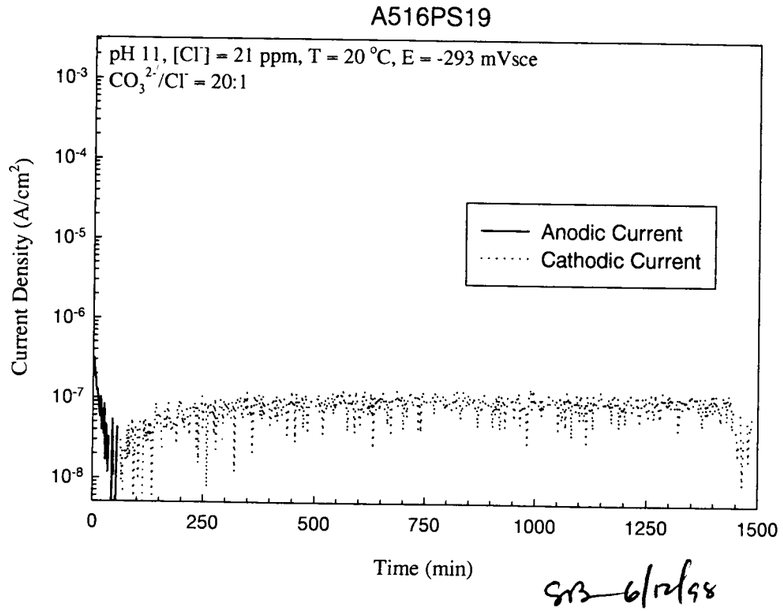
Final Sample wt = 11.38623g 6-3-98

reduced = 11.38593g

Final Solution PH = 11.029 6-3-98

Observations

N_2 purging observed 6-3-98



SRB 6/17/98

6-2-98

Cont 50

from 49

Cell 2 A516PS20.DAT

Temp = 95°C

Eset = -393mv

Init Sample wt = 11.43569g 6-2-98

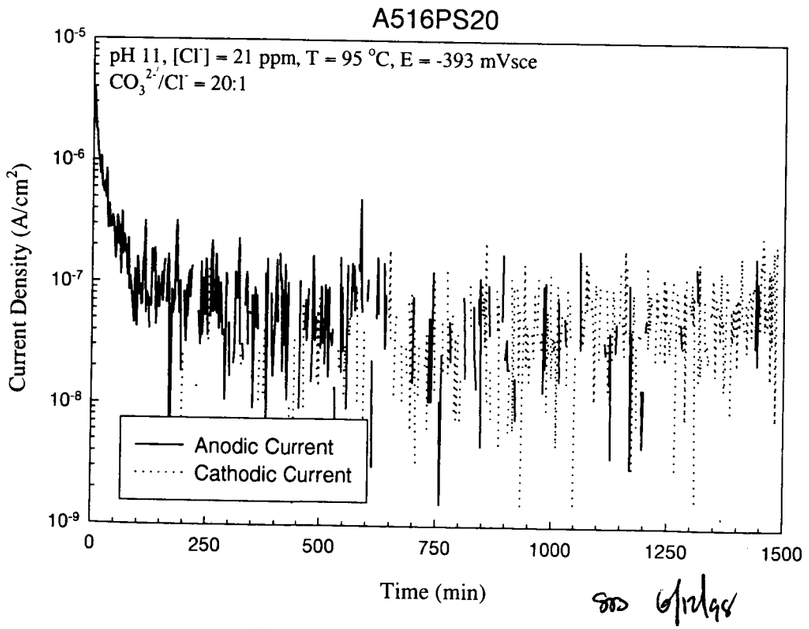
Final Sample wt = 11.43494g 6-3-98

Final Solution PH = 10.741 6-3-98

Observations

¼" loss of solution Some rusting in vapor area

more in solution 6-3-98



SD 6/12/98

6-2-98

Cont 51

from 50

Cell 3 A516PS21.DAT

Temp = 95°C

Eset = -293mv

Init Sample wt = 11.39660 6-2-98

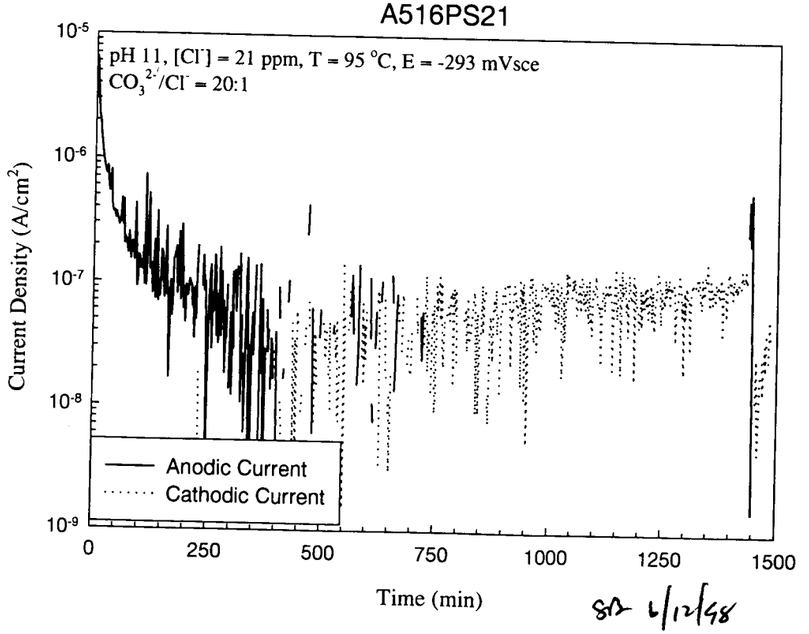
Final Sample wt = 11.39482

Final Solution PH = 11.117

Observations

¼" loss of solution 6-3-98

Some rusting in vapor area more in solution



SD 6/12/98

6-2-98

Cont 52

from 51

6-3-98

Stock Solution

PH = 11 → 12mm Na_2CO_3 => 2.54376g/2Lt Fisher Lot # 960685

→ 14ppm Cl => .046754g/2Lt Fisher Lot # 972274

PH Initial = 10.969

All samples polished to 600 grit + ultrasonically cleaned in Acetone

[signature] 6-3-98

Cont 53

from 52

Cell 1 A516PS22.DAT

Temp = 20°C

Eset = -286mv

Init wt = 11.34903g

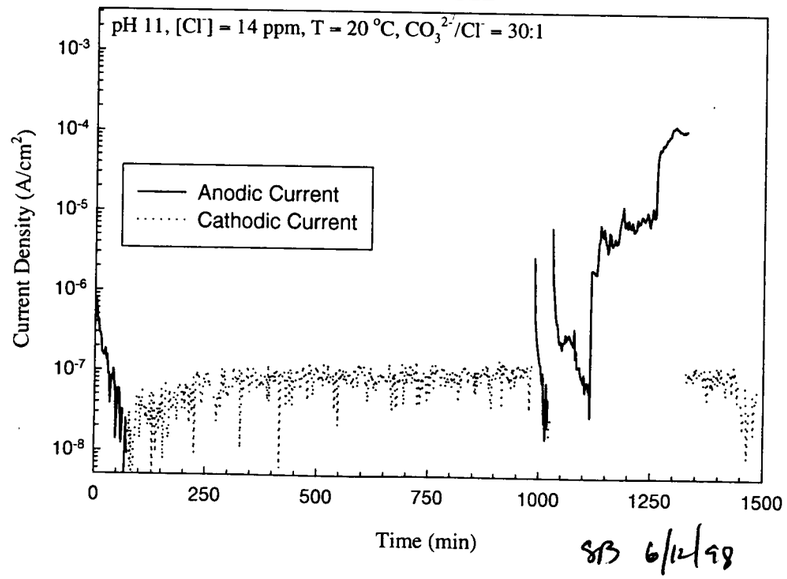
Final wt = ~~11.053~~ 11.34569g [signature] 6-4-98

Final Solution PH = 11.053 [signature] 6-4-98

Observations - no attack observed, ~~[illegible]~~ SSB 6/4/98 incr. potential to ~~[illegible]~~ SB 6/4/98 V sce @ 8:10 Am 6/4/98

SSB 6/4/98 no pitting - increased +62 E to +490 mV - 1 small pit observable 10:15 Am - incr. E to Eint +200 (790 mV) Find at 2:00pm pitting + participate on specimen [signature] 6-4-98

A516PS22



SB 6/12/98

[signature] 6-3-98

Cont 54

from 53

Cell 2 A516PS23.DAT

Temp = 95°C

Eset = -386mv

Init wt = 11.35095g

Final wt = 11.35102g 6-4-98

Final Solution PH = 11.009 6-4-98

Observations - no pitting obs. Erp @95C w/ 14 ppm Cl⁻ is approx -438 mVscc (-196 mVshe)

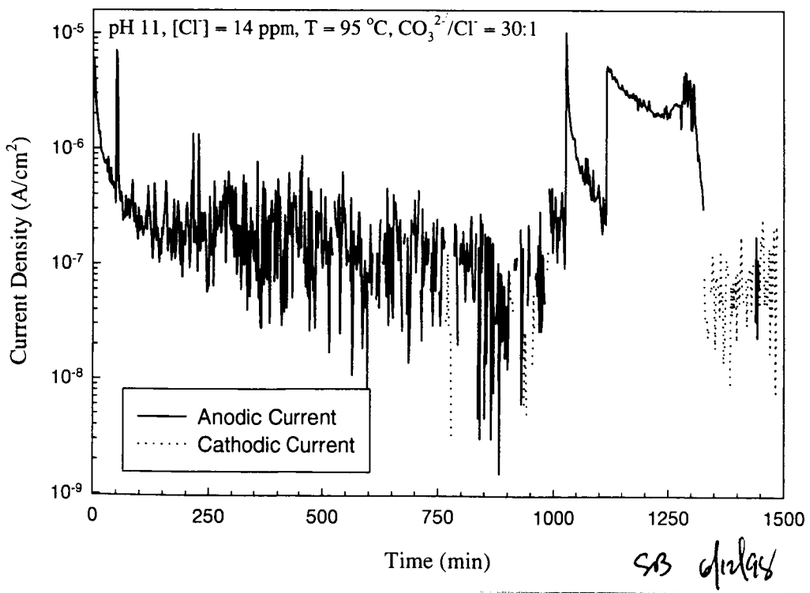
Ept = 590 mVscc; potential increased to Erp + 250 = -188 @ 8:10 am 6/4/98 cgb

9am no pitting - increased E to Ept - 200 = +390

10:15 no pits observable, E incr. to Ept + 100 = 690 mV

End 2:00pm corrosion above water in vapor area no pitting observed in solution 6-4-98

A516PS23



SB 6/12/98

6-3-98 Cont 55

from 54

Cell 3 A516PS24.DAT

Temp = 95°C

Eset = -286mv

Init wt = 11.36644g

Final wt = 11.36351g 6-4-98

Final Solution PH = 11.165 6-4-98

Observations - no pitting as Erp @95°C w/ 14ppm Cl⁻ is approx -438 mVscc (-196 mVshe); Ept = 590 mVscc

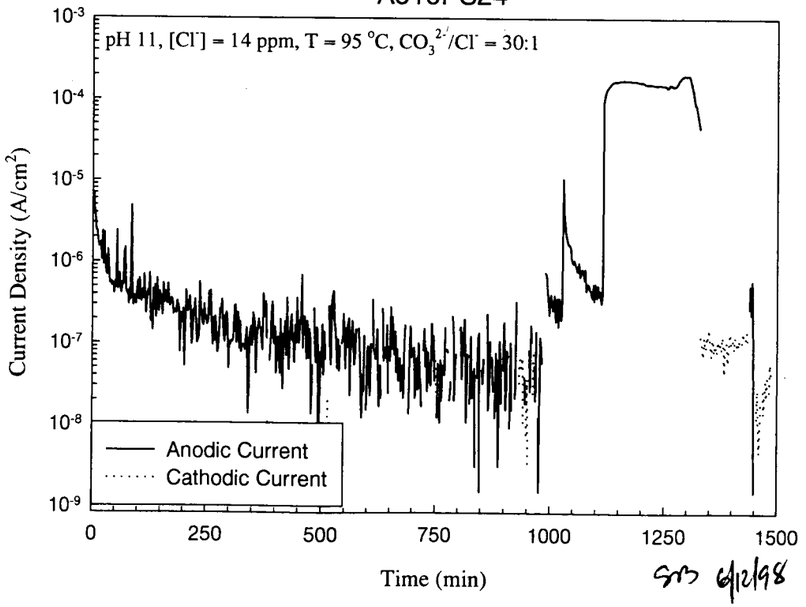
CSB 6/4/98 E increased to Erp + 500 = +62 @ 8:10 am 6/4/98 cgb

9am no pitting, increased E to Ept - 100 = +490

10:15 no pits observable - E incr. to Ept + 200 = 790 mV

End 2:00pm corrosion in vapor area no pitting observed in solution 6-4-98

A516PS24



SB 6/12/98

6-3-98 Cont 56

From 55

6-4-98

Stock Solution

→ 60 mm Na_2CO_3 => 12.71880 g/2lt

→ 100ppm Cl => .32958 g/2lt

Initial PH = 11.277

All samples polished to 600 Grit + ultrasonically cleaned in Acetone

6-4-98

Cont 57

from 56 Cell 1 A516PS25.DAT

Temp = 20°C

Eset = -200mV

Init wt = 11.37092g

Final wt = 11.36997 g 6-5-98

Final Solution PH: 11.286 6-5-98

Observations 8:05 am no noticeable pitting 6/5/98

Ener to 243 mV/sec @ 8:15

9 am - cathode hold for 10 min @ -750 mV

then reinstated to previous potential (+243)

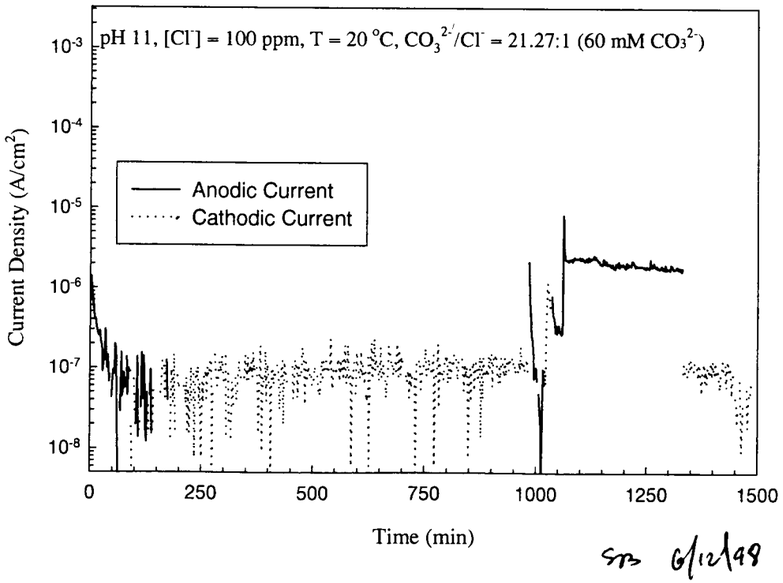
9:30 incr. potential to +750

End 2:00pm 6-5-98 79912 seconds

Some small pitting on sample base to side — to free

slight discoloration overall 6-5-98

A516PS25



6/12/98

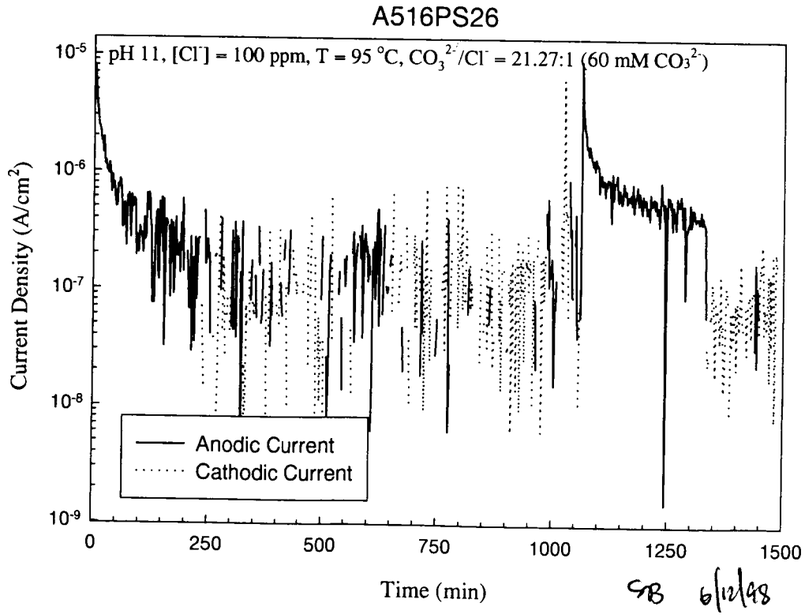
6-4-98

Cont 58

from 57

Cell 2 A516PS26.DAT
Temp = 95°C
Eset = -300mV
Init wt = 11.36067g
Final wt: 11.36031g 6-5-98

Final PH of Solution = 10.981 6-5-98
Observations 8^05 no noticable pitting 6/5/98
Epit (mV SHE) = -159 - 242 log [Cl⁻] ± +343 mVsce
Encri to Epit -200 = 143 mVsce @ 8^15
9hm - cathodic hold @ -750 mV for 10 min then
reinstated to previous potential (+143)
9^30 incr. potential to +650
No pitting observed, slight change in vapor area. 6-5-98



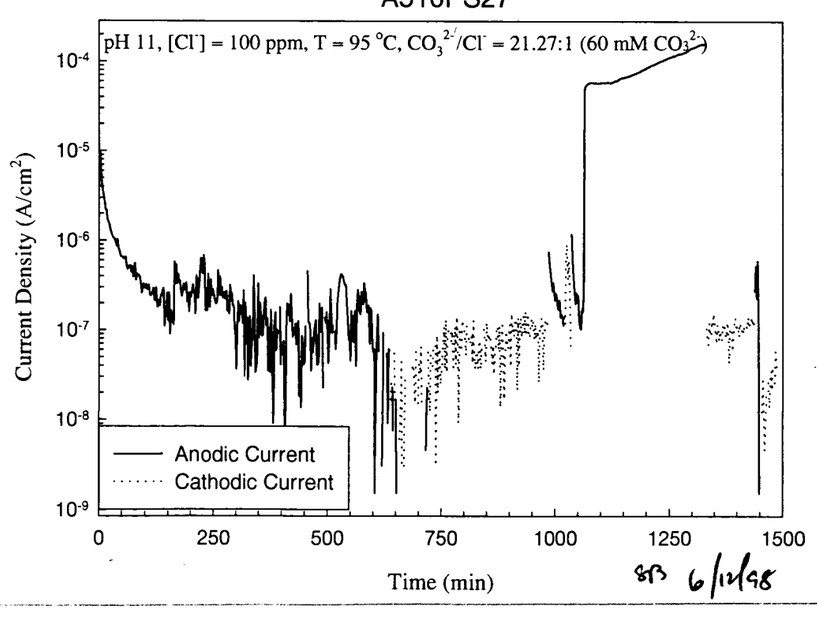
SB 6/12/98

6-4-98

Cont 59

Cell 3 A516PS27.DAT
Temp = 95°C
Eset = -200mV
Init wt = 11.30834g
Final wt = 11.30820g 6-5-98

Final Solution PH = 11.315 6-5-98
Observations 8^05 hm no noticeable pitting 6/5/98
G incr. to Epit -100 = 243 mV sce @ 8^15
9hm - cathodic hold @ -750mV for 10 min then
reinstated to previous potential (+243)
9^30 incr. potential to +750
no pitting. 6-5-98



SB 6/12/98

6-4-98

Cont 60

from 59

6-8-98

Stock Solution

→ 12mm Na_2CO_3 ⇒ 2.54376 g/2lt Fisher Lot# 960685

→ 1000 ppm Cl ⇒ 3.2958 g/2lt Fisher Lot#972274

Initial PH = 11.036 6-8-98

Crevice sample area ~ 1.7cm² for total sample incl. hole CSR 6/12/98

All samples polished to 600 grit + ultrasonically cleaned in Acetone

Crevices cleaned in Methanol (ultrasonic)

Switched to crevice samples to try to induce attack @ specific locations on sample CSR 6/8/98

Crevice formers loaded to 40 in oz torque using torque wrench #2. CSR 6/8/98

6-8-98

Cont 61

from 60

cell 1 A516PS28.DAT - crevice

Temp = 20°C

Eset = -.450 v

Init wt = 29.29312 g

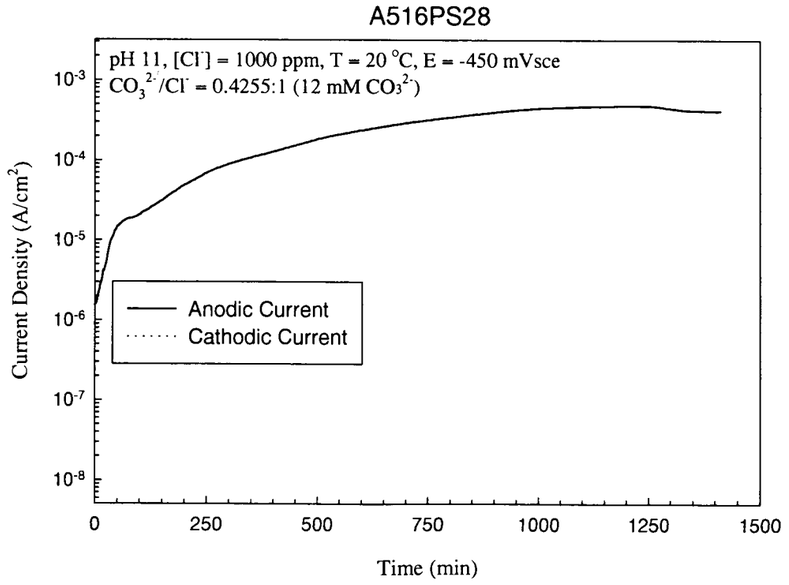
Final wt = 29.17318 g 6-9-98

Final solution PH = 11.934 6-9-98

Observations Acquisition began @ 1685 s

Particle build occured much later in test

Massive pitting observed. 6-9-98



SRB 6/12/98

6-8-98

Cont 62

from 61

Cell 2 A516PS29.DAT - CREVICE

Temp = 65°C

Eset = -.300 v

Init wt = 29.29714g

Final wt = 28.67316g 6-9-98

Final Solution PH = 11.507 6-9-98

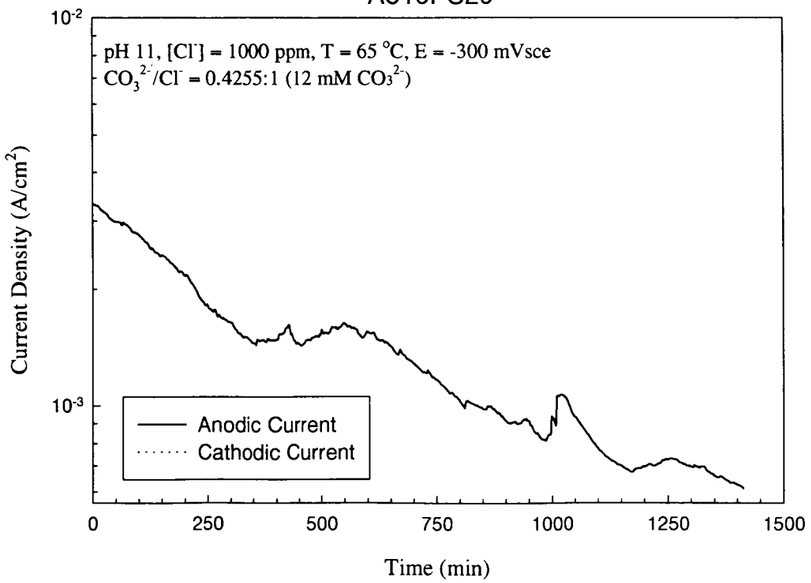
Observations

Immediately after applying potential, wetted buildup accumulating

Acquisition began @ 1685s 6-9-98

Massive pitting

A516PS29



SB 6/12/98

6-8-98

Cont 63

from 62

Cell 3 A516PS30.DAT - CREVICE

Temp = 95°C

Eset = -.050 v

Init wt = 29.34919g

Final wt = 28.08.745g 6-9-98

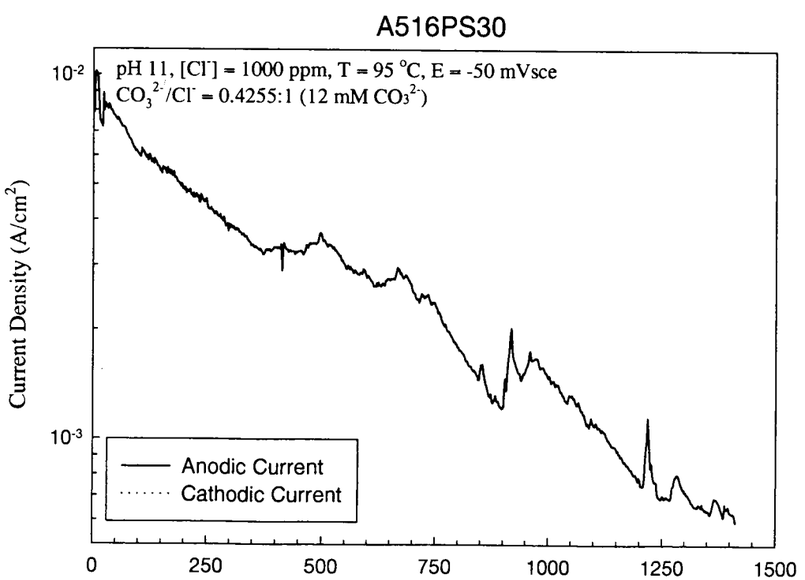
Final Solution PH = 10.850 6-9-98

Observations

Immediately after applying potential, wetted buildup accumulating and rusting taking place. Acquisition began @ 1685s

Massive pitting 6-9-98

A516PS30



SB 6/12/98

6-8-98

from 63

6-9-98

Stock Solution

→ 12 mm Na_2CO_3 => 2.54376g/2Lt Fisher Lot# 960685

→ 100 ppm Cl => .32958g/2Lt Fisher Lot# 972274

Initial PH = 11.087

All samples polished to 600 Grit + ultrasonically cleaned in Acetone

Crevice material ultrasonically cleaned in Methanol

6-9-98

Cont 64

from 64

Cell 1 A516 PS31, DAT — CREVICE

Temp = 20 °C

Eset = -400 mV

Init wt = 29.32179g

Final wt = 29.32128g 6-10-98

Final Solution PH = 11.053 6-10-98

Observations Start 6-9-98 / 15:50

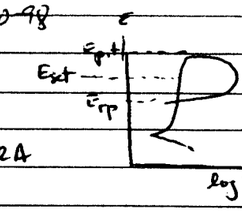
Change Eset to -530mV @ 600 s

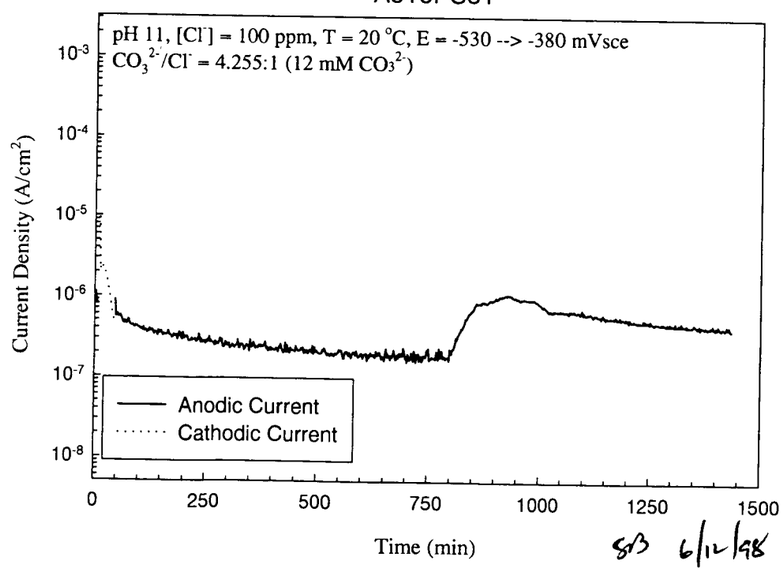
Eset ↑ to -380 mV @ 2700s ~1/2 passive range

Crevice pitting observed 6-10-98

Note "1/2 passive range" = [Epit - Erp]/2

Epit & Erp are from Cragnolino et al (Corrosion/98) and other work in ASIL @ CNWRA in Lab Notebook #157





SB 6/12/98

6-9-98

Cont 65

from 65

Cell2 A516PS32.DAT - CREVICE

Temp = 65°C

Eset = -250mv

Int wt = 29.37832g

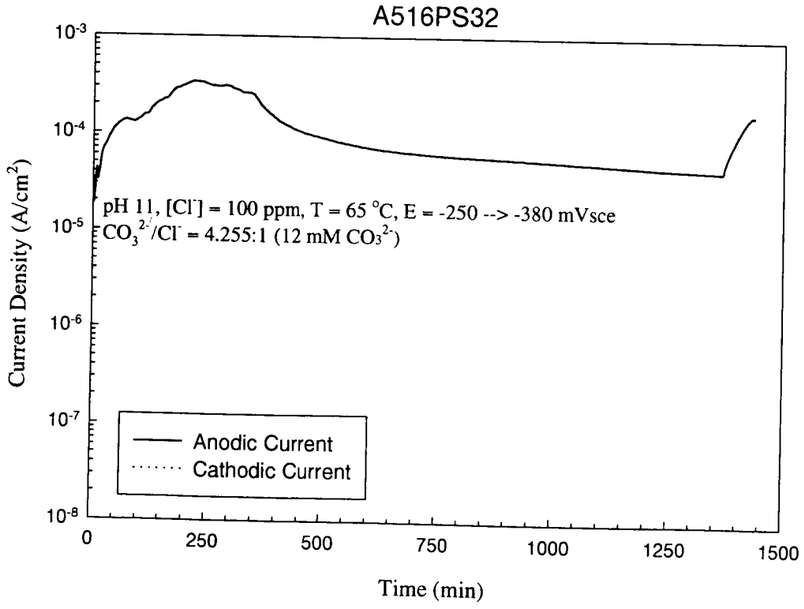
Final wt = 29.32809g 6-10-98

Final Solution PH = 10.983 6-10-98

Observations

Charge Test -380mv — 1/2 passive range

Pitting noted on both crevice surfaces but not where crevice made contact. 6-10-98



SB 6/12/98

6-9-98

C-t 6/8

from 66

Cell3 A516PS33.DAT - CREVICE

Temp = 95°C

Eset = +100mv

Int wt = 29.32629g

Final wt = 29.19822g 6-10-98

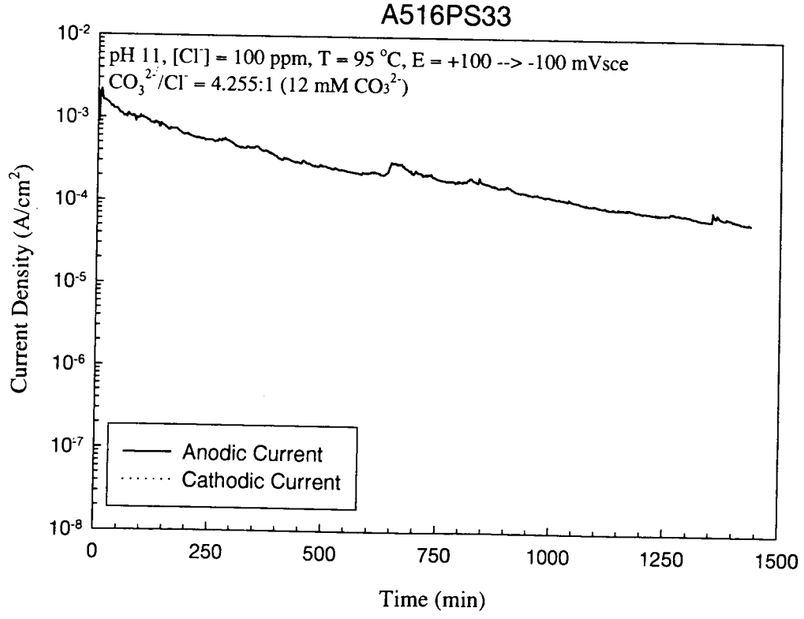
Final Solution PH = 10.668 6-10-98

Observations

Immediate signs of attacking directly after applied potential

Charge Est -100mv 600sec into test - 1/2 passive range

Massive pitting on all surfaces, not where crevice made contact. 6-10-98



SB 6/12/98

6-9-98

from 67

6-11-98

Start Solution

→ 12mm Na_2CO_3 => 2.543376g/2L

→ 10ppm Cl => .032958 g/2L

Initial PH = 11.070 6-11-98

All samples polished to 600grit + ultrasonically cleaned in Acetone

Crevices ultrasonically cleaned in Methanol

Cont 69

6-11-98

from 68

Cell 1 A516PS34.DAT – crevice

Temp = 20°C

Eset = -300mV w/r passive range

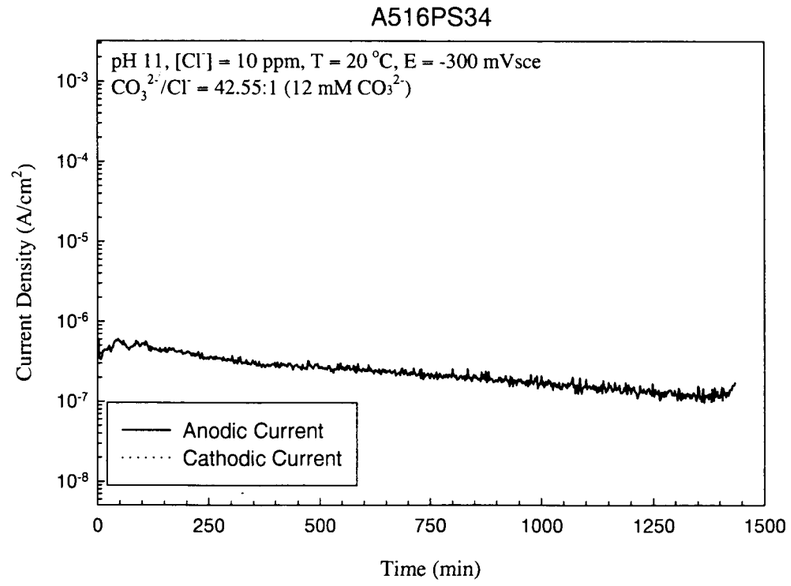
Init wt = 29.23887g 6-12-98

Final wt = 29.23634g 6-12-98

Final Solution PH = 11.048 6-12-98

Observations Begin Test 6-11-98 10:20am

Crevice pitting observed 6-12-98



GS 6/12/98

6-11-98

Cont 70

from 69 Cell 2 A516PS35.DAT

Temp = 65°C

Eset = -250 mv ~ 1/2 passive range

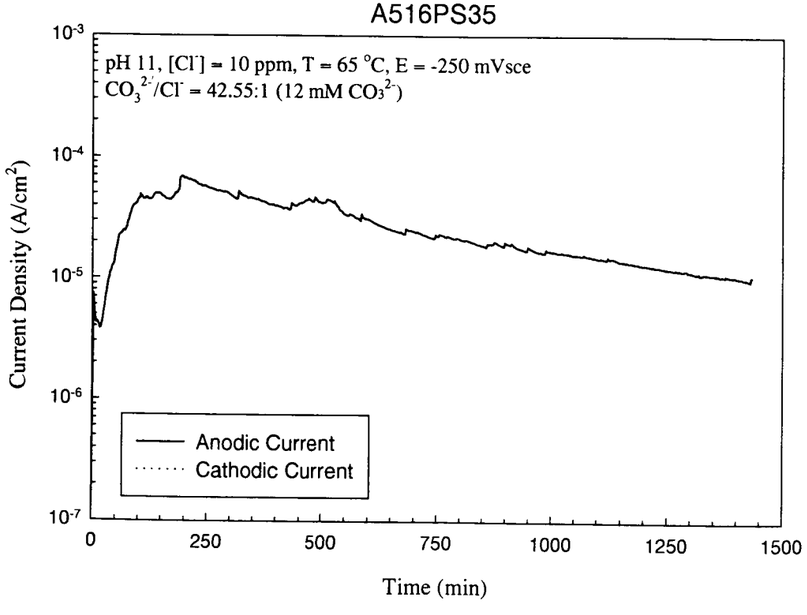
Init wt = 29.350216g

Final wt = 29.239.30g 6-12-98

Final Solution PH = 10.774 6-12-98

Observations

Crevice pitting observed with some pitting attack outside crevice footprint. 6-12-98



SDS 6/12/98

6-11-98 Cell 71

from 70 Cell 3 A516PS36.DAT

Temp = 95°C

Eset = +100 mv ~ 1/2 passive range

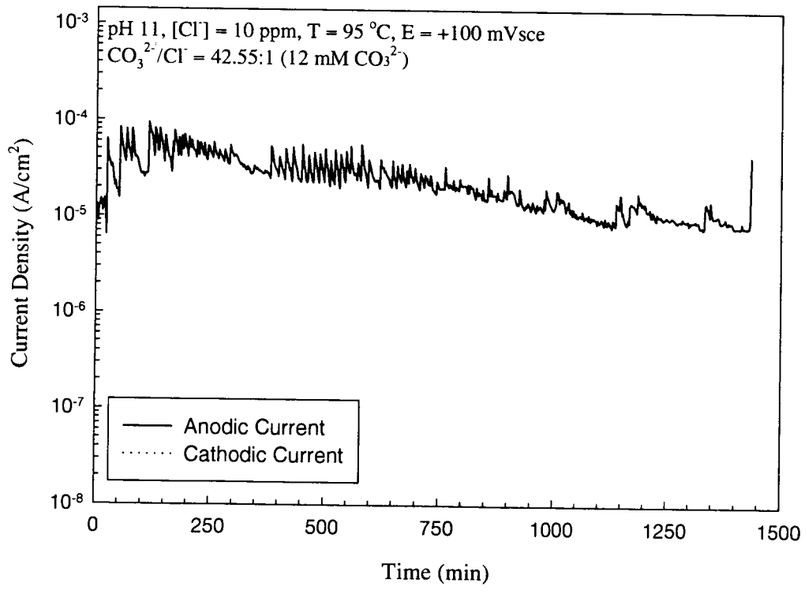
Init wt = 29.294144g

Final wt = 29.28630g 6-12-98

Final Solution PH = 10.796 6-12-98

Observations

Crevice pitting observed. 6-12-98



SDS 6/12/98

6-11-98

fon 71

6-15-98

Stock Solution

→ 120 mm Na_2CO_3 => 25.4376 g/2Lt Fisher lot# 960685

→ 100 ppm Cl => .32958 g/2Lt Fisher lot# 972274

Initial PH = 11.386

All specimens polished to 600 grit and ultrasonically cleaned in Acetone

Crevices ultrasonically cleaned in methanol

6-15-98

Cont 73

from 72

Cell1 A516PS37.DAT - crevice

Temp = 20°C

Eset = -380mv

Init wt = 29.27947g

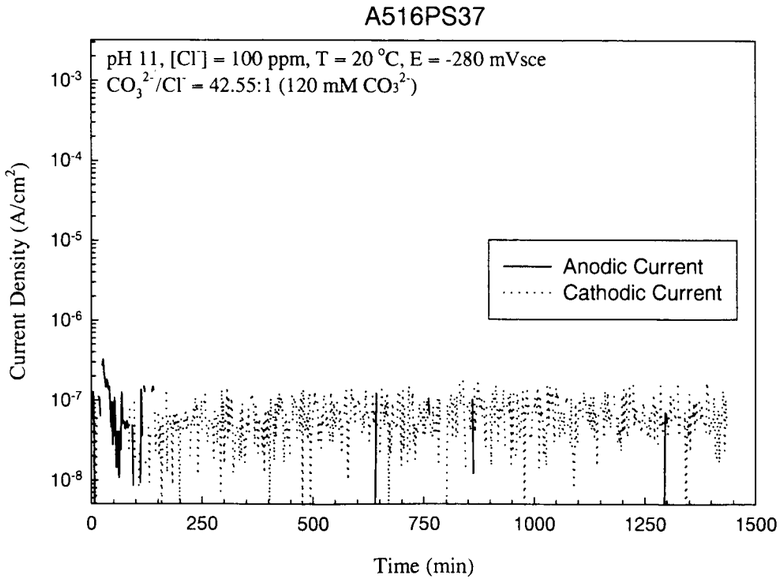
Final wt = 29.27554g 6-16-98

Final Solution PH = 11.352 6-16-98

Observations Start 10:33

Error to -280mV @ 1420s

No Pitting 6-16-98



6-15-98

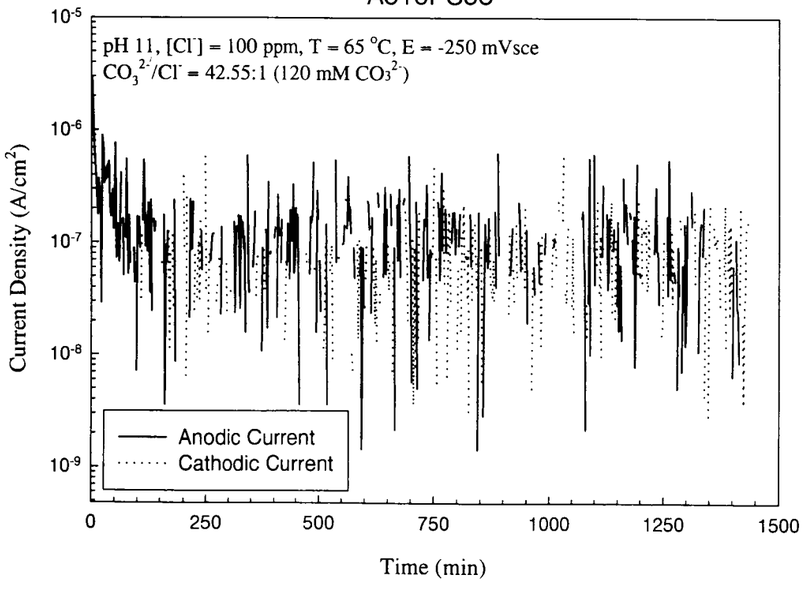
Cont 74

from 73

Cell 2 A516PS38.DAT - creviced
Temp = 65°C
Eset = -350mv
Init wt = 29.23673g
Final wt = 29.23348g 6-16-98

Final Solution PH = 11.114 6-16-98
Observations
Emer. to -350 @ 1420s
No Pitting 6-16-98

A516PS38



6-15-98

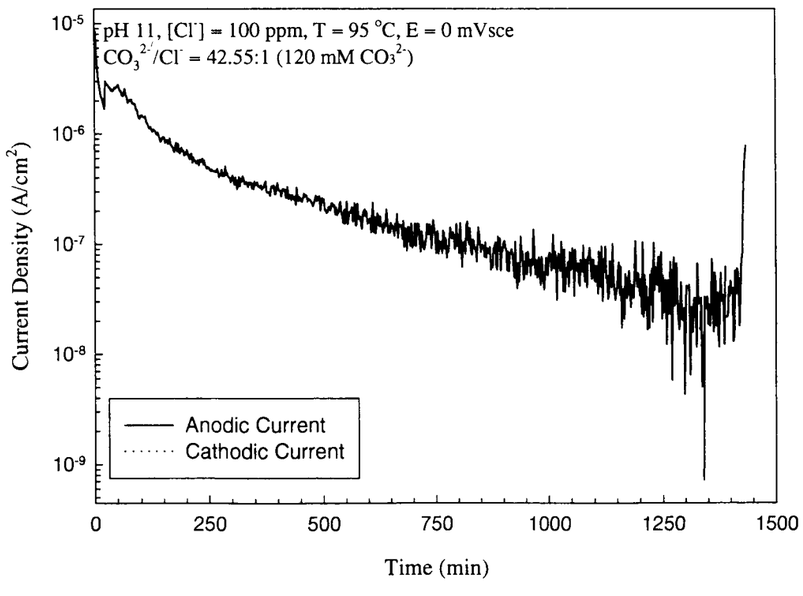
Cat 75

from 74

Cell 3 A516PS39.DAT - creviced
Temp = 95°C
Eset = -100mv
Init wt = 29.33130g
Final wt = 29.32791g 6-16-98

Final Solution PH = 11.107 6-16-98
Observations
Emer. to 0 @ 1424s
Some minor crevicepitting 6-16-98

A516PS39



6-15-98

from 75

6-16-98

Start Solution

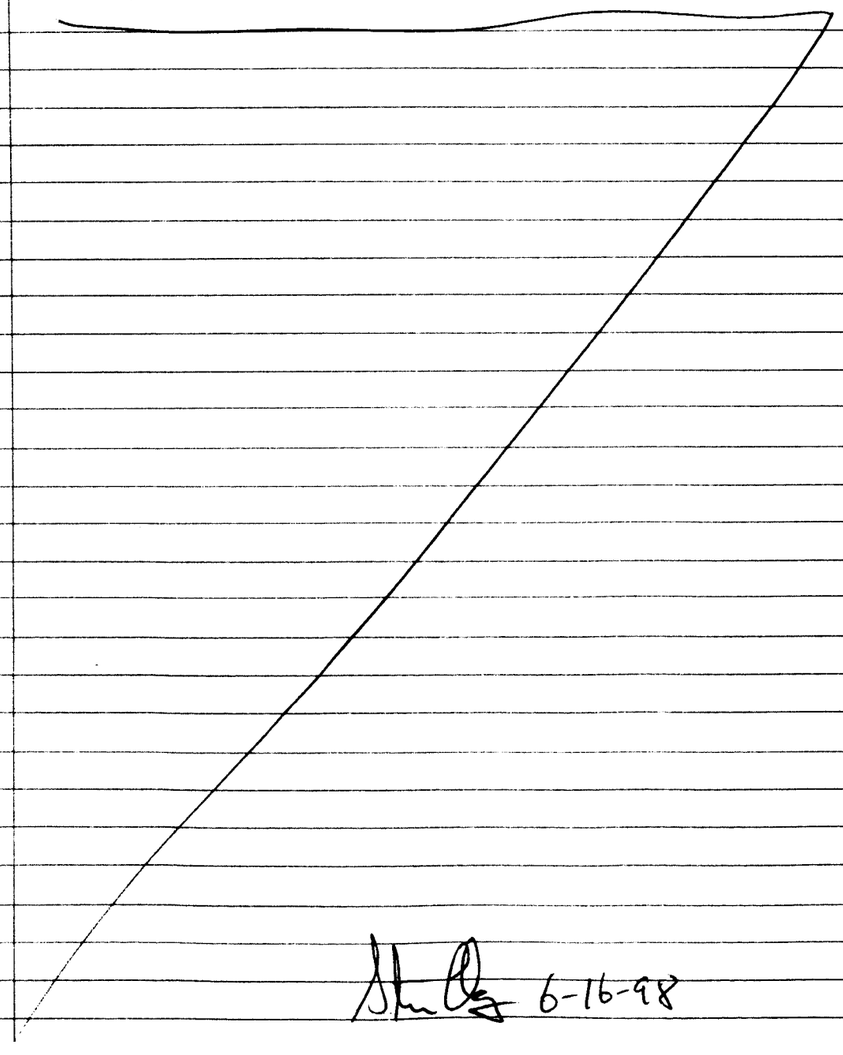
→ 120mm Na_2CO_3 => 25.4376g/2Lt Fisher Lot# 960685

→ 1000ppm Cl => 3.2958g/2Lt Fisher Lot# 972274

Initial PH = 11.366

All specimens polished to 600 grit and ultrasonically cleaned in Acetone

All crevices ultrasonically cleaned in Methanol



6-16-98

Cont 77

from 76

Cell1 A516PS40.DAT – crevice

Temp = 20°C

Eset = -450 mv

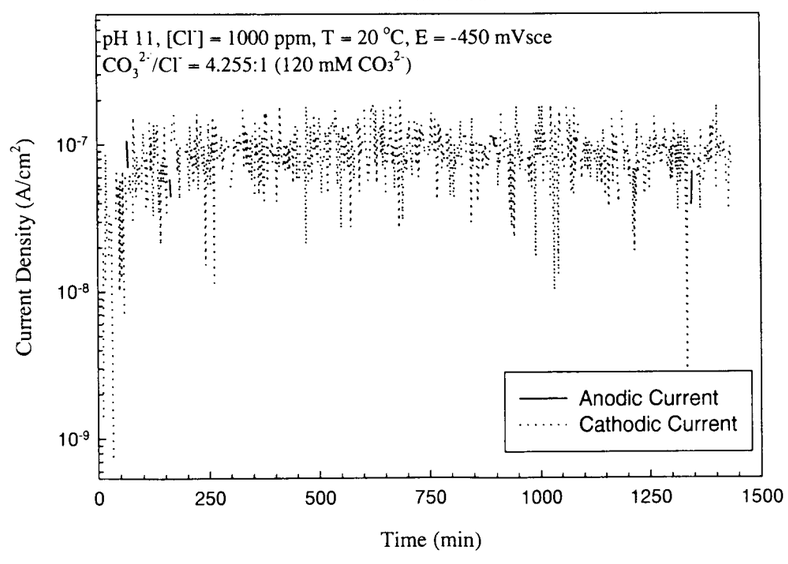
Init wt = ~~29.30116g~~ 29.29899g 6-16-98

Final wt = 29.29662g 6-17-98

Final Solution PH = 11.344 6-17-98

Observations Start 1:43pm

No Pitting 6-17-98



6-16-98

Cont 78

from 77 Cell 2 A516PS41.DAT - crevice

Temp = 65°C

Eset = -300mV

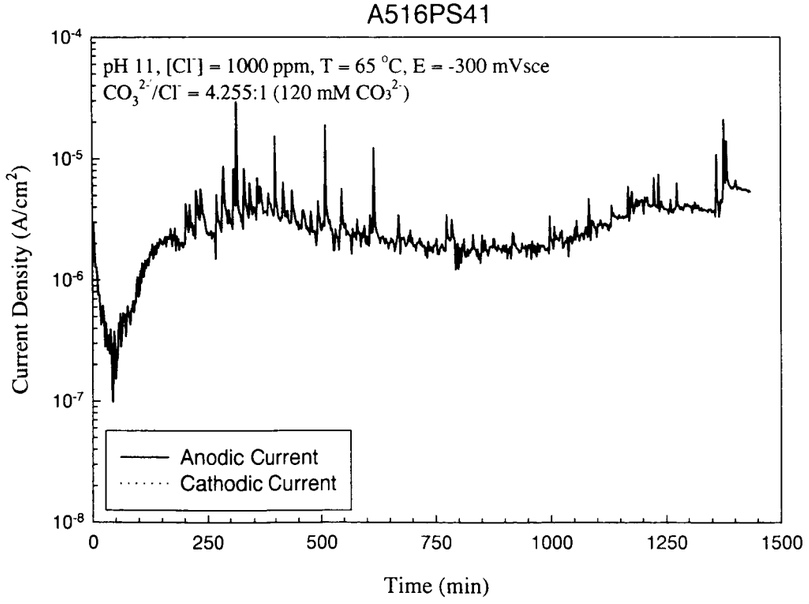
In.wt = 29.24151g

Final wt = 29.23757g 6-17-98

Final Solution pH = 11.129 6-17-98

Observations

MODERATE CREVICE PITTING 6-17-98



6-15-98

Cat 79

from 78 Cell 3 A516PS42.DAT - crevice

Temp = 95°C

Eset = -50mV

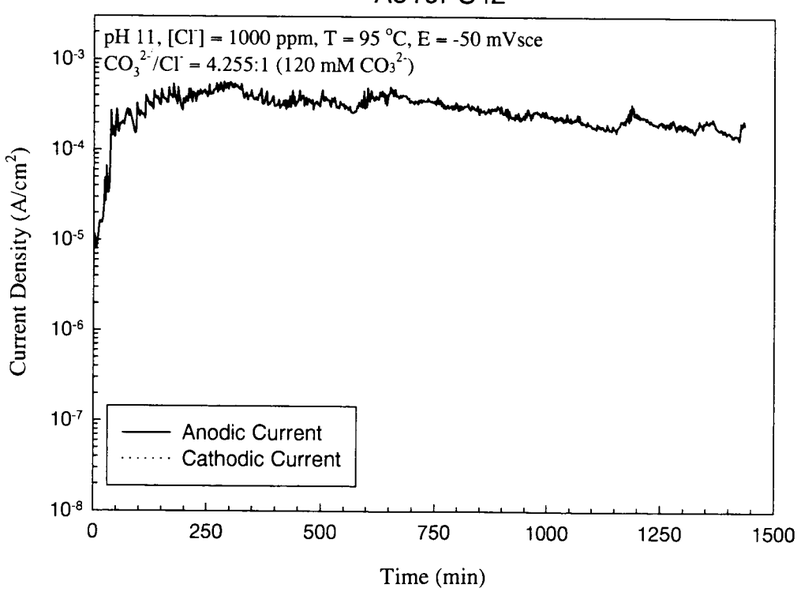
In.wt = 29.32231g

Final wt = 29.31065g 6-17-98

Final Solution pH = 11.253 6-17-98

Observations

Heavy crevice pitting, corrosion inside crevice bolt hole. 6-17-98



6-16-98

from 79

6-17-98

Stat Solution

12mm Na_2CO_3 => 2.54376g/2Lt

100ppm Cl => .32958g/2Lt

Initial PH= 11.042

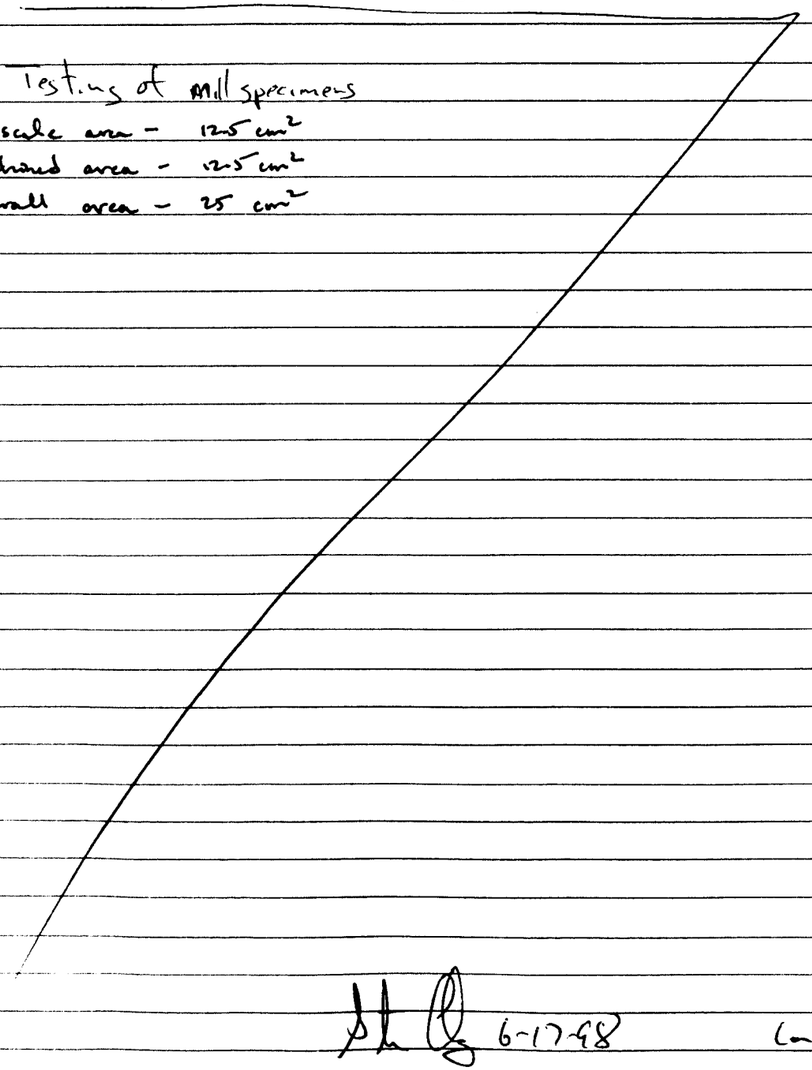
All samples polished on edges to 600 grit + ultrasonically degreased in Acetone

Testing of mill specimens

millscale area - 12.5 cm^2

machined area - 12.5 cm^2

overall area - 25 cm^2



6-17-98

Cont 81

from 80

Cell 1 A516 PS 43.DAT - mill scale

Temp = 20°C

Eset = -380mV

Init wt = ~~69.088~~ ml 69.08234g

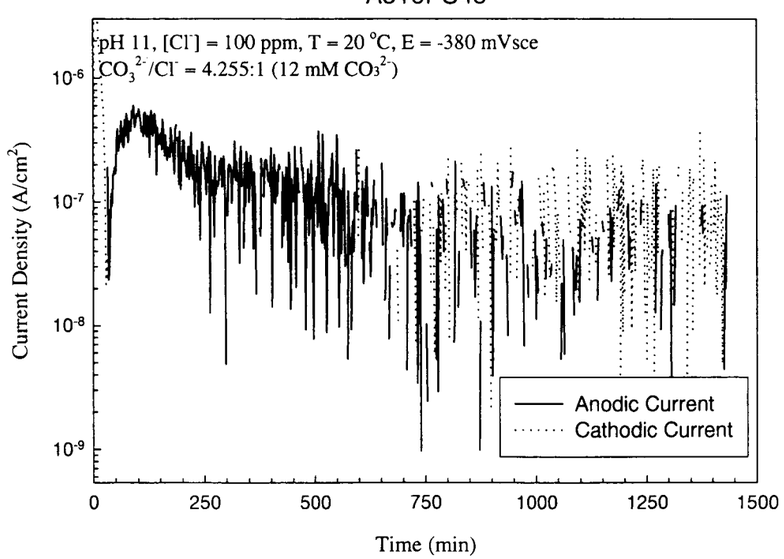
Final wt = 69.07892 g 6-18-98

Final Solution PH= 11.085 6-18-98

Observations Start 3:20pm

No pitting on polished edges or mill surface

some corrosion above vapor line 6-18-98



6-17-98

Cont 82

From 81 Cell 2 A516PS44.DAT – mill scale

Temp = 65°C

Eset = -350 mv

Init wt = 69.81044 g

Final wt = 69.75538 g 6-18-98

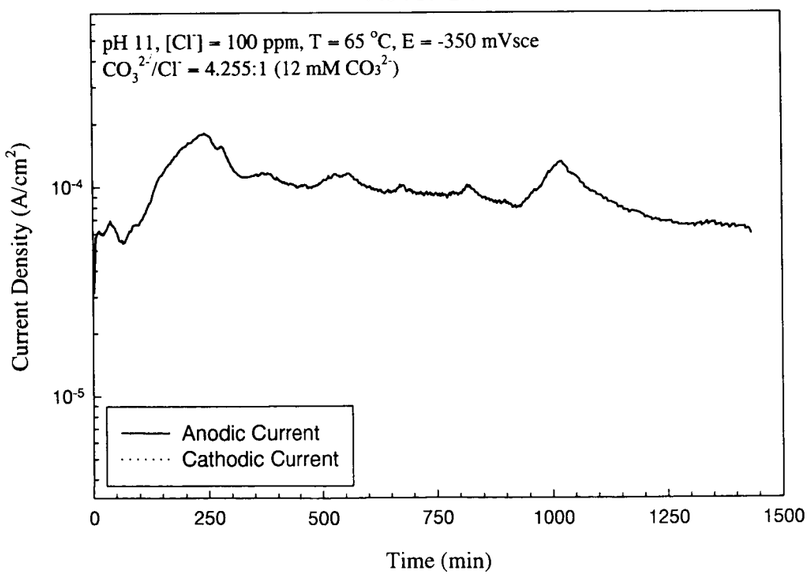
Final Solution PH = 10.962 6-18-98

Observations

No pitting on polished edges or mill surface

some corrosion on top machined surface 6-18-98

A516PS44



6-17-98

Cont 83

From 82 Cell 3 A516PS45.DAT – mM scale

Temp = 95°C ATP

Eset = -100 mv

Init wt = 69.88012 g

Final wt = 69.73813 g 6-18-98

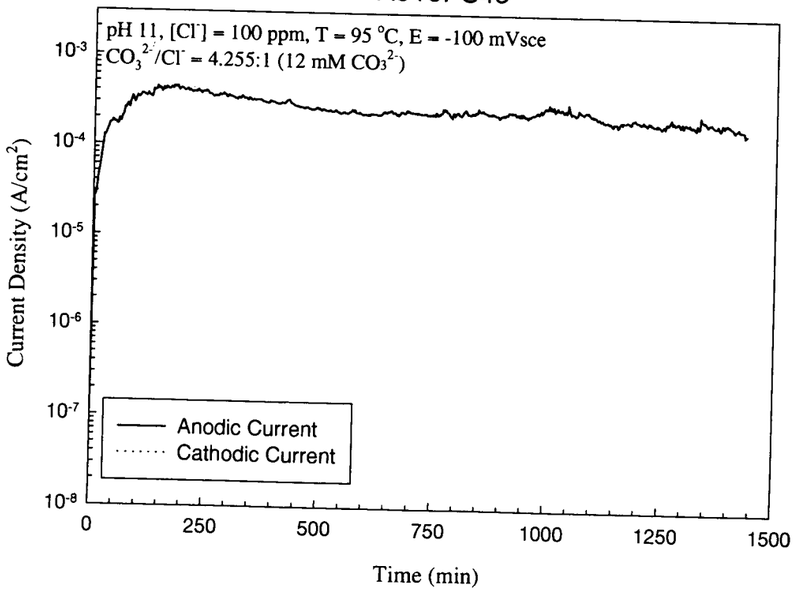
Final Solution PH = ~~11.085~~ 6-18-98 11.478 6-18-98

Observations

Pitting on polished edges but not mill surface

Some corrision on machined top surface 6-18-98

A516PS45



6-17-98

6-18-98
Stock Solution -
12 mM Na_2CO_3 => 2.54376 g/2Lt
10 ppm Cl => .03296 g/2Lt

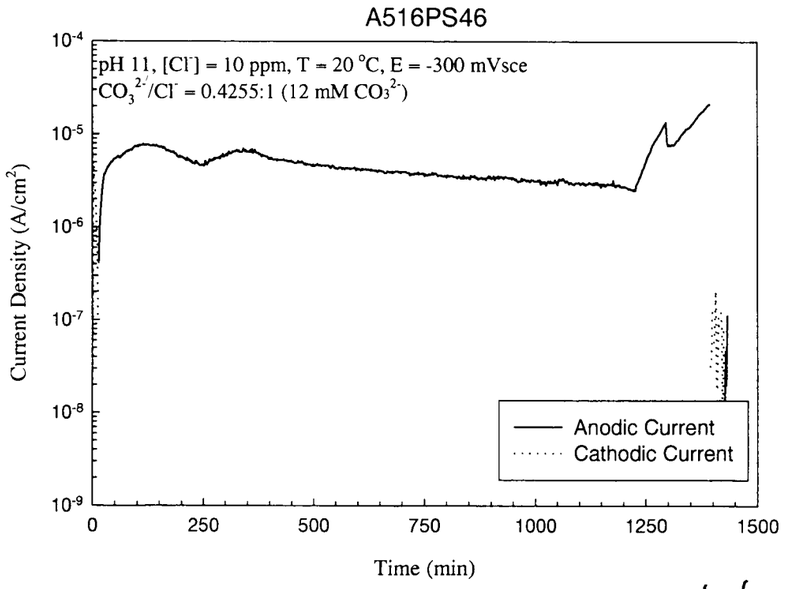
Initial PH = 11.068

All samples edge polished to 600 grit + ultrasonically cleaned in Acetone

CSB 6/24/98

Cell1 A516PS46.DAT — mill scale 6/18/98
Temp = 20°C
Eset = -300 mV
Initwt = 70.00812 g
Finalwt = 70.00176 g CSB 6/18/98

Final Solution PH = 10.950 CSB 6/18/98
Observations
some water-line attack, and corrosion on top of specimen



CSB 6/22/98

6/18/98 Cell 2 A516PS47.DAT - mill scale

Temp = 65°C

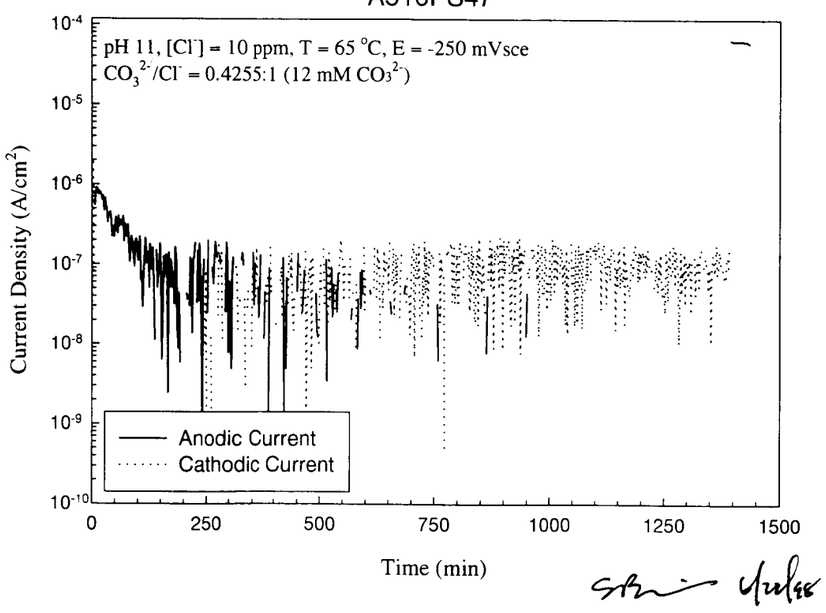
Eset = -250mv

Init wt = 69.90712g

Final wt = ~~69.4044g~~ 69.9044g CSB 6/19/98 - suspect wt loss may be due to cath. reduction of mill scale

Final Solution PH = ~~11.071~~ CSB 6/19/98 10.659 CSB 6/19/98

Observations no noticeable corrosion



SB 6/22/98

Cell 3 A516PS48.DAT - mill scale 6/18/98

Temp = 95°C

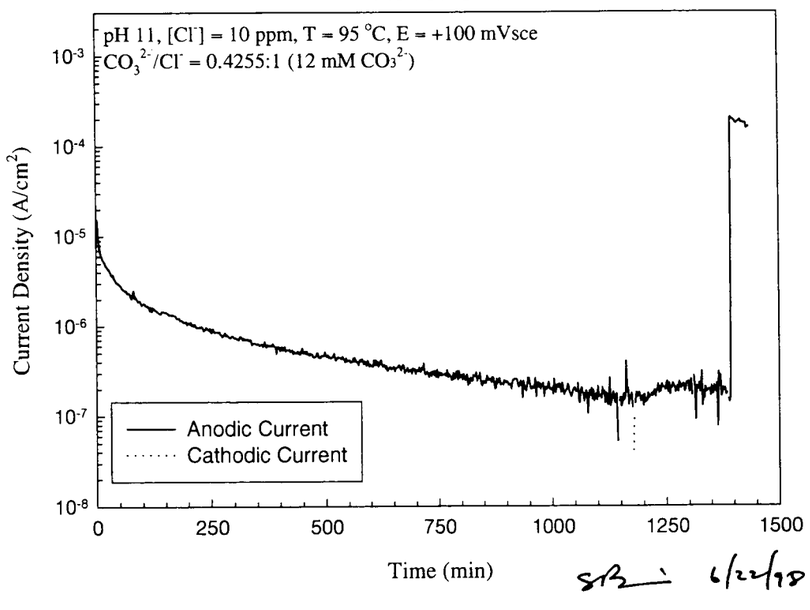
Eset = +100mv

Init wt = 69.51632g

Final wt 69.4044g CSB 6/19/98

Final Solution PH = 10.840 6/19/98

Observations small pits observable on polished areas, but no noticable attack on mill scale



SB 6/22/98

6/22/98

Performance Verification — Actual R = 10.048114

Applying Potential to 10Ω resistor (SN 171002) and measuring current response

PSTAT 7 (Cell 1)

Eapplied (Strawberry Tree DAQ)	Eread	I
-1	-0.9979	-9.4247×10^{-2} A
-0.75	-0.7484	-7.0688×10^{-2} A
-0.50	-0.4994	-4.7157×10^{-2} A
-0.25	-0.2509	-2.3719×10^{-2} A
0	0.0003	6.0177×10^{-6} A
+0.25	0.2519	2.3762×10^{-2} A
+0.50	0.4999	4.7153×10^{-2} A
+0.75	0.7489	7.0644×10^{-2} A
+1	0.9980	9.4145×10^{-2} A

PSTAT 3 (Cell 2)

-1	-0.9918	-9.5267×10^{-2} A
-0.75	-0.7441	-7.1356×10^{-2} A
-0.5	-0.4943	-4.7443×10^{-2} A
-0.25	-0.2476	-2.3852×10^{-2} A
0	0.0020	4.0667×10^{-6} A
+0.25	0.2514	2.3900×10^{-2} A
+0.50	0.4976	4.7433×10^{-2} A
+0.75	0.7472	7.1290×10^{-2} A
+1	0.9945	9.4939×10^{-2} A

CSR 6/22/98

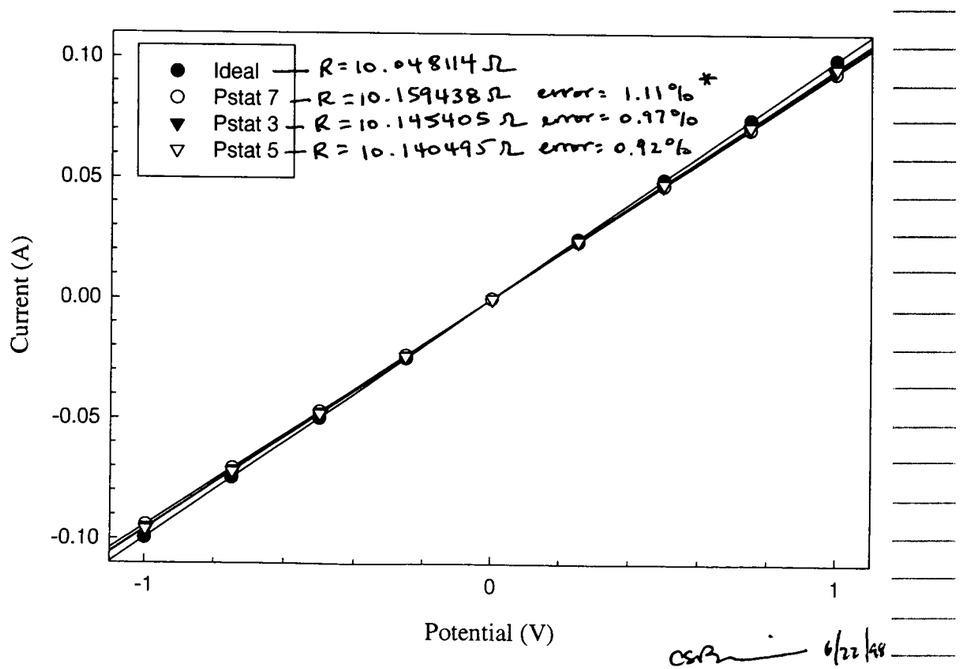
Cont on p89

from p88

6/22/98

PSTAT 5 (Cell 3)

Eapplied	Eread	I
-1	-0.9955	-9.6402×10^{-2} A
-0.75	-0.7489	-7.1999×10^{-2} A
-0.50	-0.4998	-4.8050×10^{-2} A
-0.25	-0.2513	-2.4165×10^{-2} A
0	0.0024	2.315×10^{-5} A
+0.25	+0.2517	2.4204×10^{-2} A
+0.50	+0.4998	4.8026×10^{-2} A
+0.75	+0.7448	7.1952×10^{-2} A
+1	1.0006	9.6111×10^{-2} A



CSR 6/22/98

* Note, outside of tolerance of resistor; according to TOP-22, Potentiostat/data acquisition system is not functioning properly. However, based on limitations of 12-bit control system being used and the small error (< 1.1%) noted between measured & known resistance, it is felt that the system is operating properly.

CSR 6/22/98

from 89

8-10-98

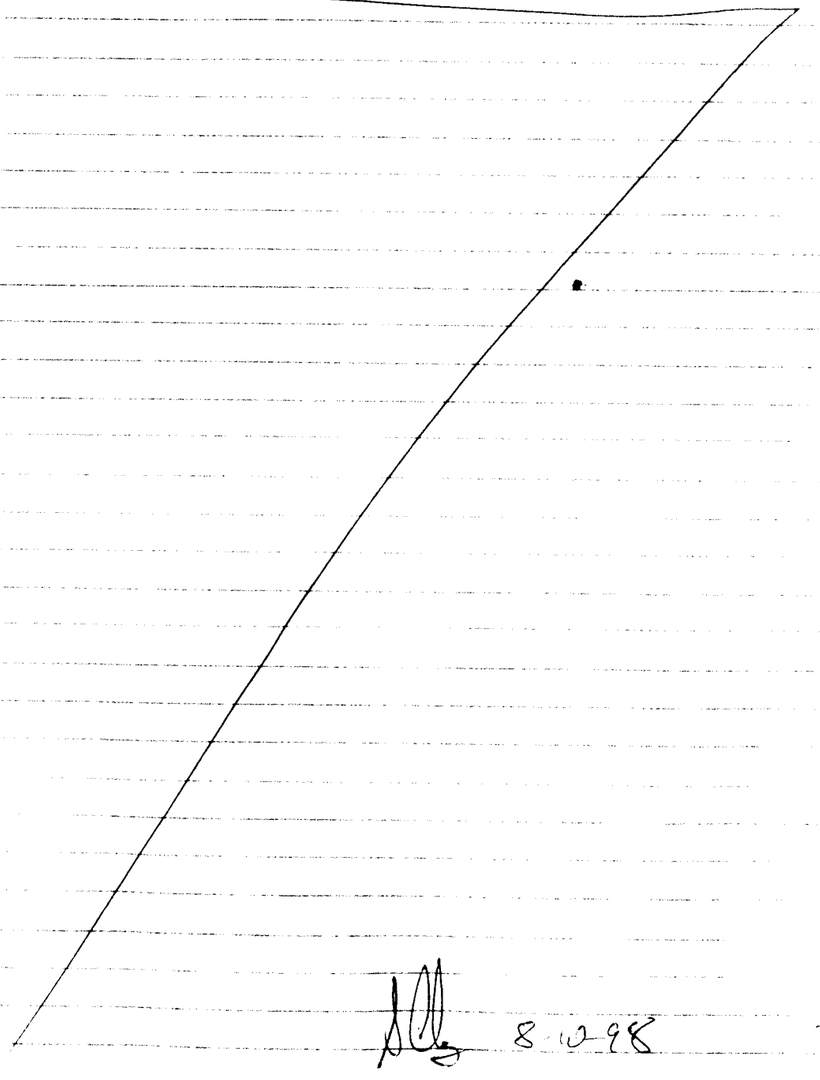
Stock Solution

12 mm Na_2CO_3 => 2.543736g/2Lt

100 ppm Cl => .329558g/2Lt

Initial PH = 11.007 [signature]

All Samples Degreased in Acetone



[signature] 8-10-98

To 91

from 90

Cell 1 A516PS49.DAT Mill scale samples

Temp = 25°C

Eset = -300

Init wt = 70.102065g [signature] 8-17-98

Final wt = 69.822202g [signature] 8-17-98

Final Solution PH = 11.465 [signature] 8-17-98

Observations started @ 12:25 pm 8/10/98

Several corrosion product build up on machined edges [signature] 8-17-98

To 92

CSB 8/10/98

from 91

Cell 2 A516 PS 50. DAT Mullscale Samples

Temp: 65°C

Eset: -200

In. wt: 70.10938g

Final wt: 69.20446g

Final Solution PH 11.234 8-17-98

Observations started @ 12:25 pm 8/13/98

Heavy corrosion product + pitting on machined surfaces

corrosion on non machined surfaces 8-17-98

8/10/98 CSB

from 92

Cell 3 A516 PS 51. DAT Mullscale samples

Temp = 95°C

Eset = +100

In. wt = 70.06316g

Final wt = 69.06042g 8-17-98

Final Solution PH = 10.664 8-17-98 12:25 pm 8/10/98

Observations started @ ~~to~~ CSB 8/10/98

Heavy corrosion product and pitting on machined surfaces

corrosion on non machined surfaces. Corrosion 8-17-98

above vapor line.

OSD 8/10/98

to 94

from 93

8-17-98 Crevice Specimens

Stock Solution

120 mm $NaCO_3$ → 25.4376g/2Lt

12 mm Cl → 1.4026g/2Lt

Initial PH = 11.322

All Samples polished to 600 grit and ultrasonically cleaned in Acetone

8-17-98 To 95

from 94

Cell 1 A516PS52.DAT

Temp = 25°C

Eset = -250mv

Int wt = 28.68414g 8-18-98

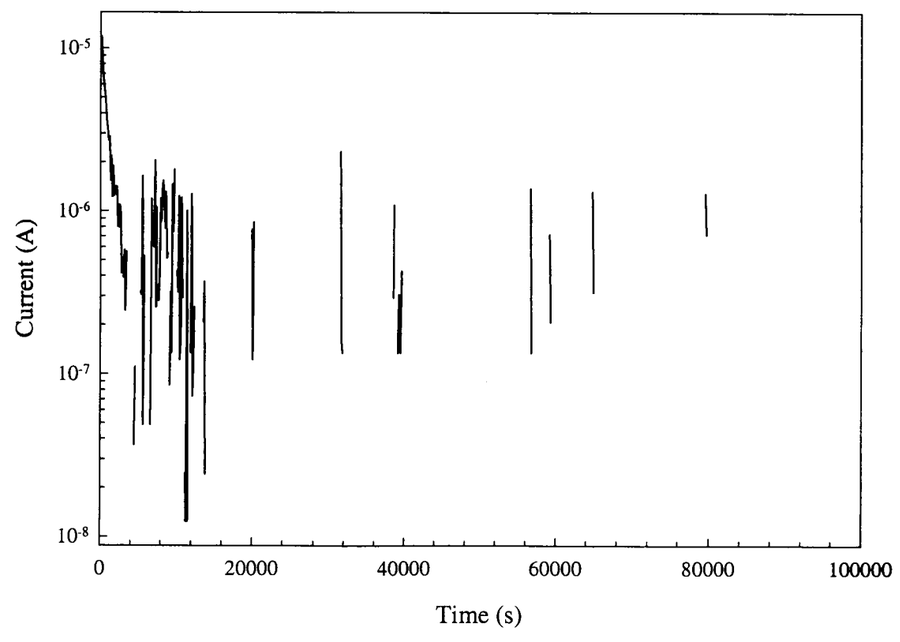
Final wt = 28.68434g 8-18-98

Final Solution PH = 11.301 8-18-98

Observations

No corrosion or pitting

A516PS52



8-17-98 To 96

From 95

Cell 2 A516 PS53.DAT

Temp = 65°C

Eset = -200 mV

Init wt = 28.67494g

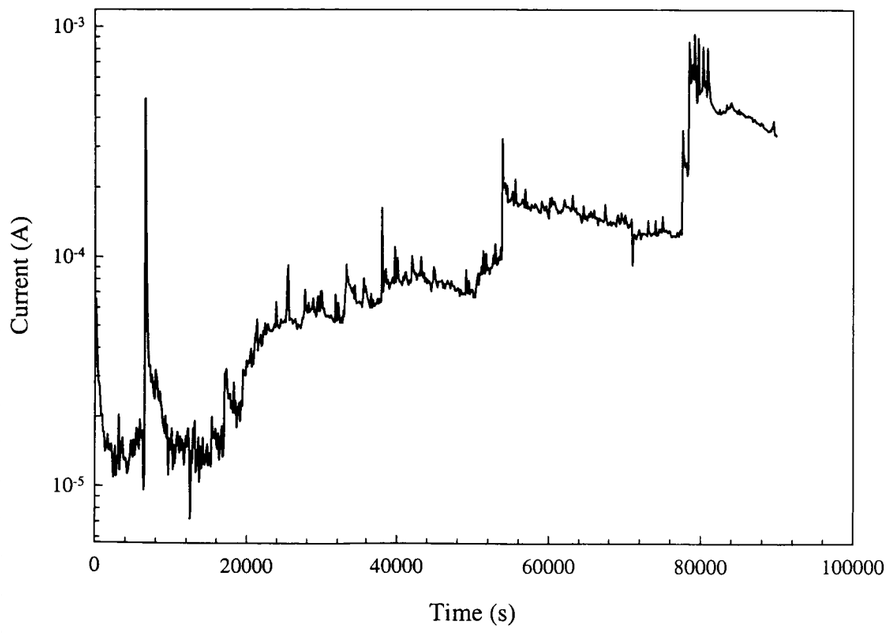
Final wt = 28.67368g 8-18-98

Final Solution pH = 11.145 8-18-98

Observations

Crevice pitting. Some corrosion in inner hole. 8-18-98

A516PS53



To 97

8-17-98

From 96

Cell 3 A516 PS54.DAT

Temp = 95°C

E Set = 0mV

Init wt = 28.97623g

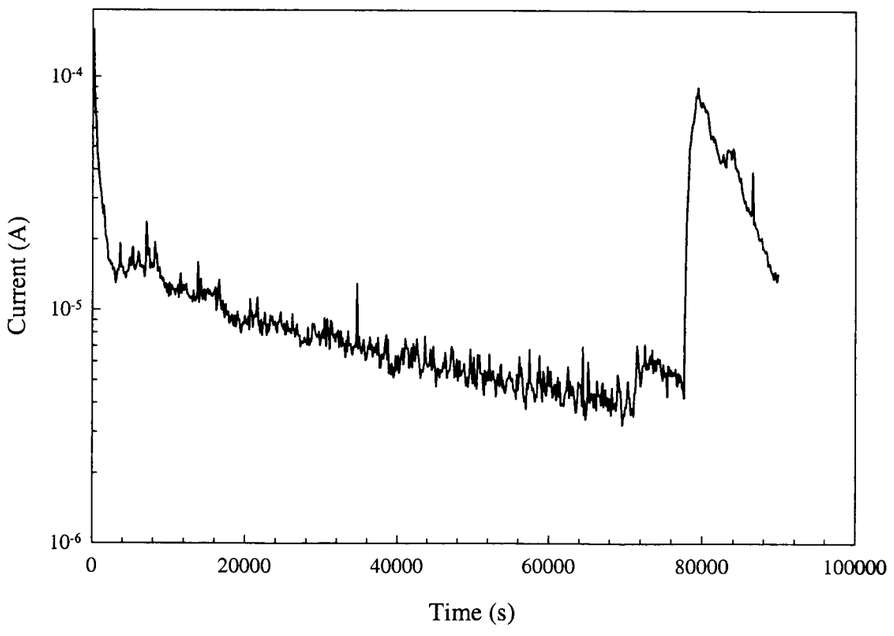
Final wt = 28.97715g 8-18-98

Final Solution pH = 10.857 8-18-98

Observations

Crevice pitting 8-18-98

A516PS54



8-17-98 To 98

from 97

8-18-98

Stock Solution

120 mm Na_2CO_3 → 25.4376 g/2 lt

4.8 mm Cl → 0.5610 g/2 lt

Initial pH = 11.324

All samples polished to 600 grit + cleaned ultrasonically in Acetone
crevices cleaned in Methanol.

8-18-98

To 99

from 98

cell: A516PS55.DAT

Temp = 25°C

Eset = -200mv

Init wt = 28.81722g

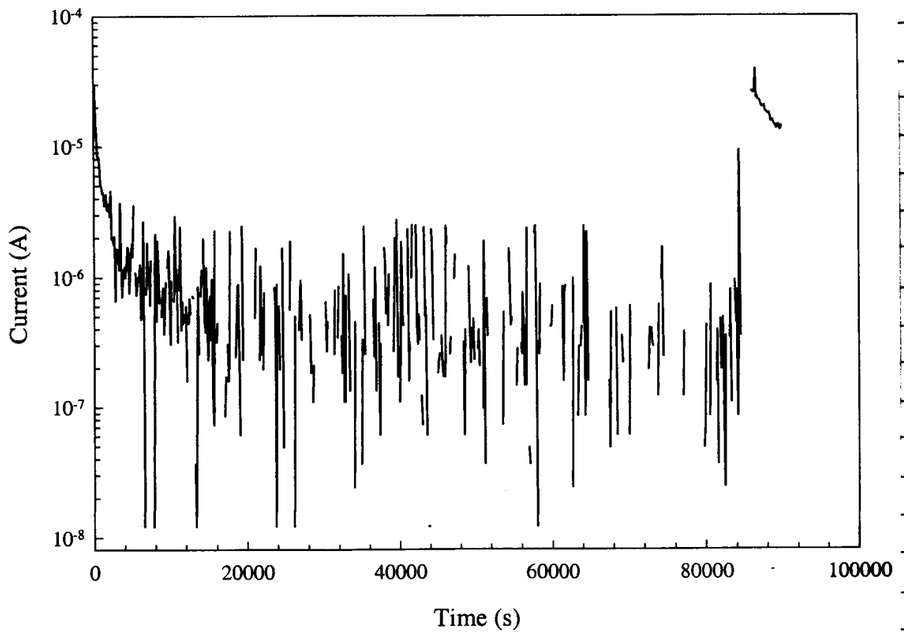
Final wt = 28.81727g 8-19-98

Final pH = 11.288 8-19-98

Observations

No pitting, very light markings at crevice feet 8-19-98

A516PS55



8-18-98

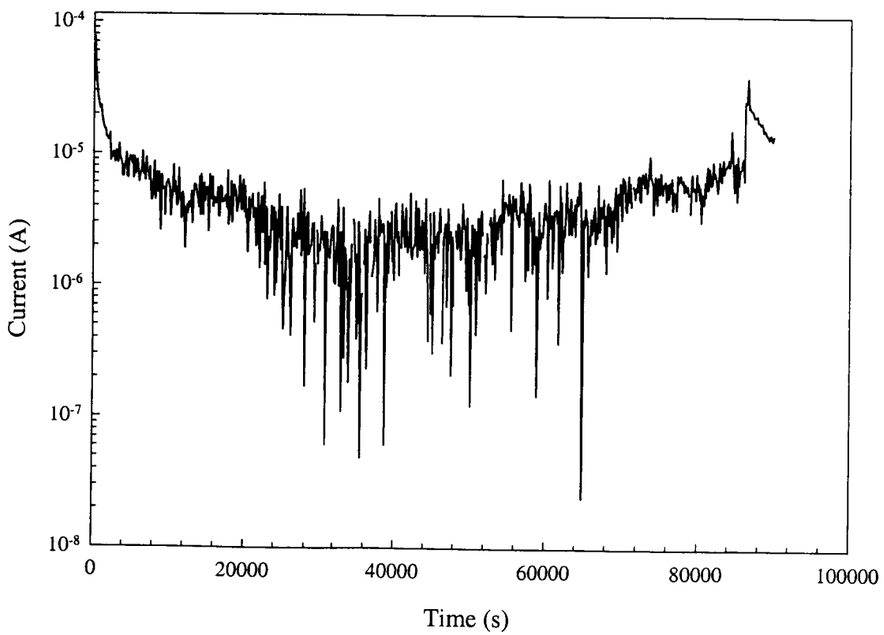
To 100

from 99

Cell 2 A516PS56.DAT
Temp 65°C
Eset
Init wt = 28.68480
Fin wt = 28.68535 g 8-19-98

Final PH 11.144 8-19-98
Observations
very slight pitting on one side of specimen 8-19-98

A516PS56



8-18-98

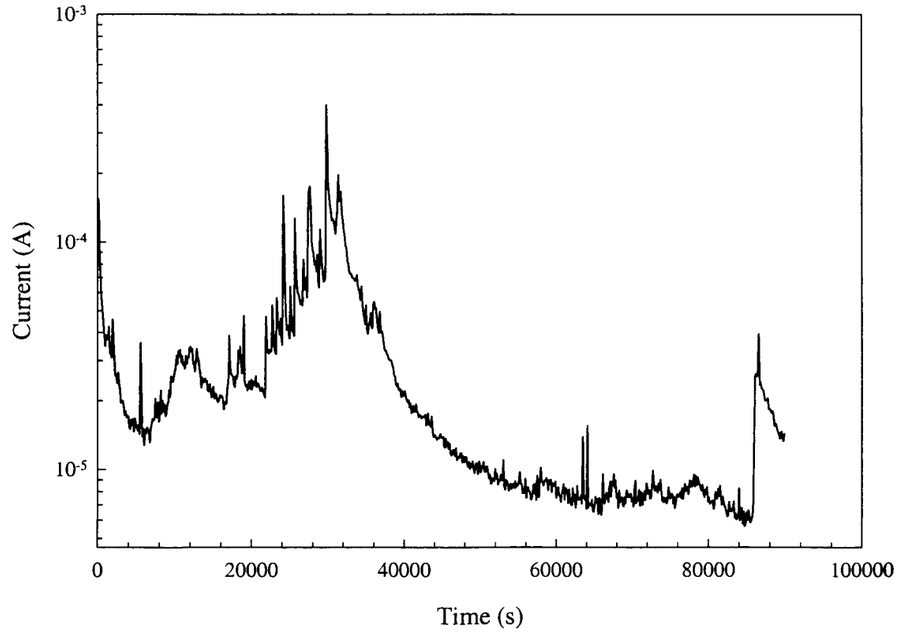
To 101

from 100

Cell 3 A516PS57.DAT
Temp 95°C
Eset
Init wt 28.58329g
Fin wt 28.58506 g 8-19-98

Final pH 10.985 8-19-98
Observations
Very slight pitting at crevice feet on one side of specimen, Uniform pitting on the other 8-17-98

A516PS57



8-18-98

To 102

From 101

8-19-98 St 1 10:00 AM

Stock Solution

120 mm $NaHCO_3$ → 25.4376g/2Lt Fisher Lot # 960685

2.4 mm Cl → 0.2805g/2Lt " 972274

Initial PH = 11.307

All Specimens polished to 600 grit & ultrasonically cleaned in Acetone, previous ultrasonically cleaned in Methanol

8-20-98

To 103

From 102

Cell 1 A516PS58.DAT

T 25°C

Eset = -175mv

Init wt = 28.86627g

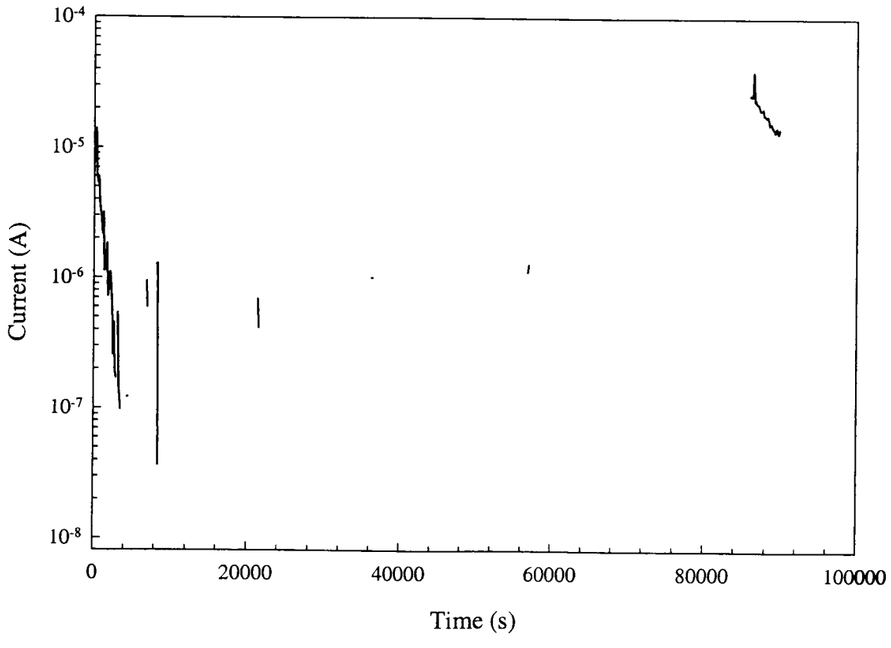
Final wt = 28.80679g 8-21-98

Final Solution PH = 11.283 8-21-98

Observations

No pitting on specimen 8-21-98

A516PS58



8-20-98

To 104

from 103

Cell 2 A516PS59.DAT

T = 65°C

Eset = -125 mv

Init wt = 28.73656 g

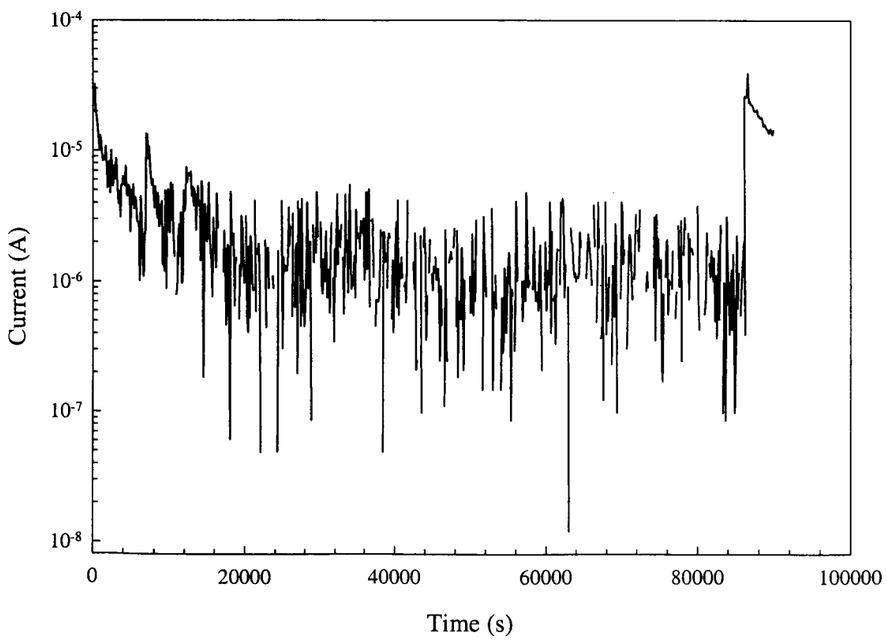
Final wt = 28.73727 g 8-21-98

Final Solution pH = 11.110 8-21-98

Observations

Very minor crevice pting in some sporadic areas 8-21-98

A516PS59



8-20-98 To 105

from 104

Cell 3 A516PS60.DAT

T = 95°C

Eset = 150 mv

Init wt = 28.94290 g

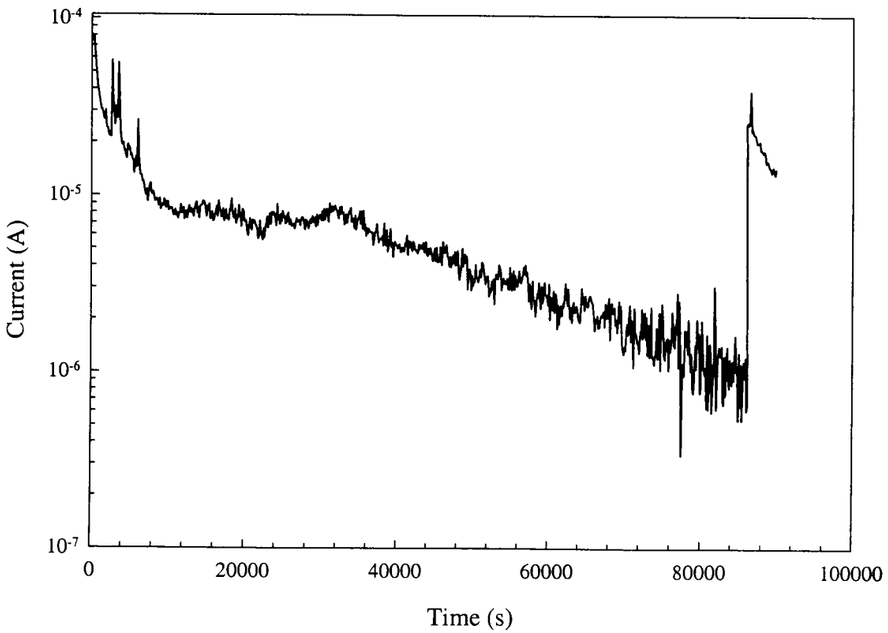
Final wt = 28.94391 g 8-21-98

Final Solution pH = 11.048 8-21-98

Observations

Small crevice pitting noted 8-21-98

A516PS60



8-20-98 To 106

from 105

3-24-98

Stock Solution

		Fisher Lot #
Na_2CO_3	= 120mm → 25.4376g/2Lt	960685
Cl	= 16mm → 0.1870g/2Lt	972274

Initial pH = 11.304

All specimens polished to 600 grit & ultrasonicall, cleaned in Acetone, crevices ultrasonically cleaned in Methanol

8-24-98 to 107

from 106

Cell1 A516PS61.DAT

T = 25°C

Eset = -150mv

Init wt = 28.85143g

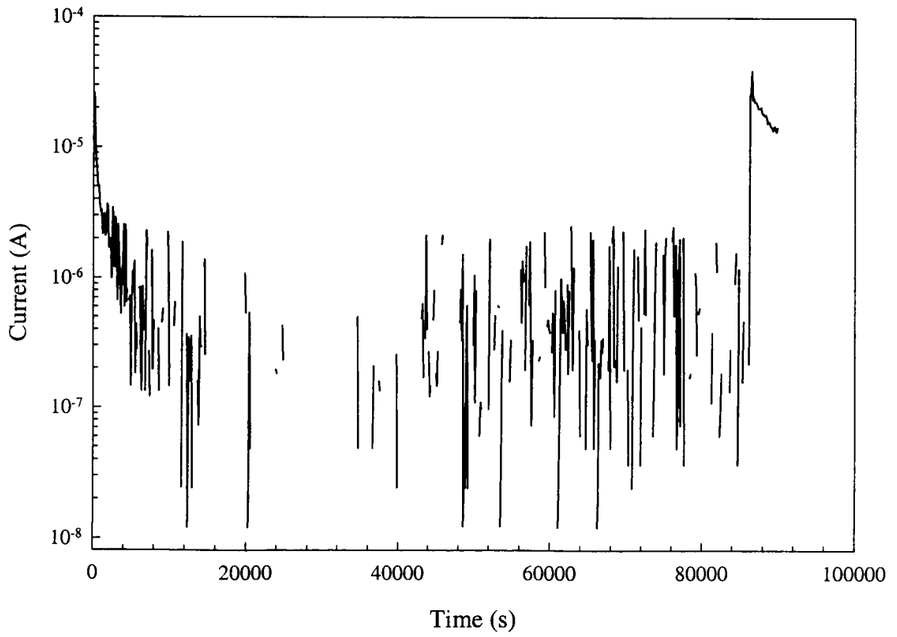
Final wt = 28.85080g 8-25-98

Final Solution pH = 11.281 8-25-98

06 saturations

Very light crevice pitting 8-25-98

A516PS61



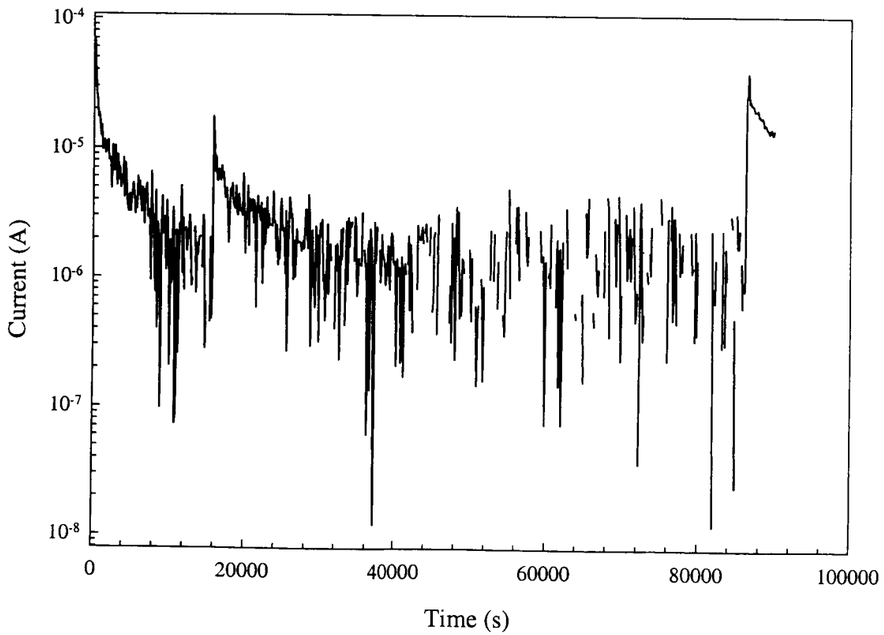
8-24-98 to 108

from 107

Cell 2 A516 PS 62.DAT
T = 65°C
Eset = -100mv
Init wt = 28.95778g
Final wt = 28.95762g 8-25-98

Final Solution pH = 11.120 8-25-98
Observations
Very light crevice pitting 8-25-98

A516PS62



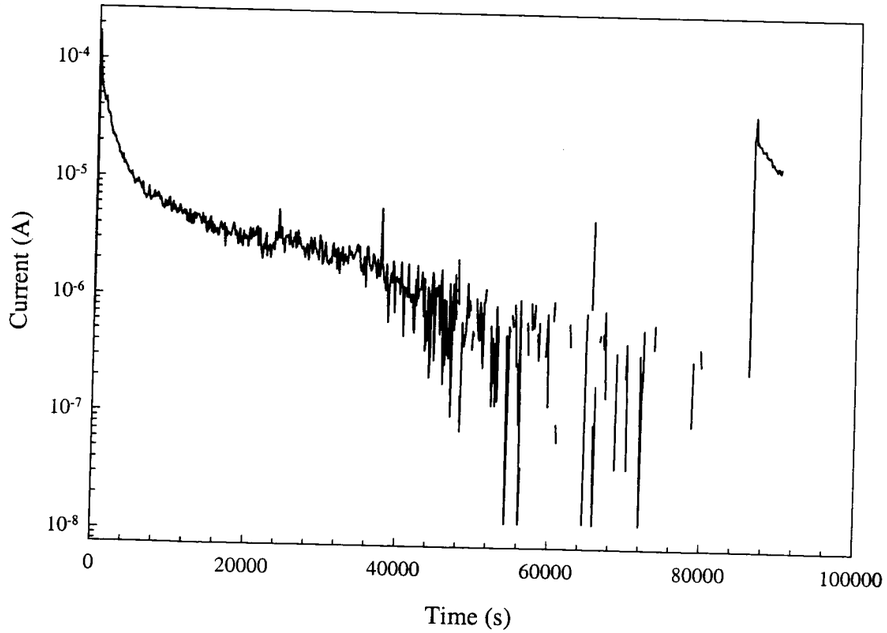
8-27-98 to 109

from 108

Cell 3 A516 PS 63.DAT
T = 95°C
Eset = 200mv
Init wt = 28.76252g
Final wt = 28.76363g 8-25-98

Final Solution pH = 11.084 8-25-98
Observations
Very light crevice pitting 8-25-98

A516PS63



8-27-98 to 110

from 109

8-25-98 Stat 11:33 AM

Stock Solution

$NaCO_3$ → 120 mm → 25.4376g/2Lt Fisher Lot 960685

Cl → 1.2 mm 0.1403g/2Lt 972274

Initial PH = 11.319

All

8-25-98 to 111

from 110

Cell 1 A516PS64.DAT

T = 25°C

Eset = −150mv

Init wt = 28.97570g

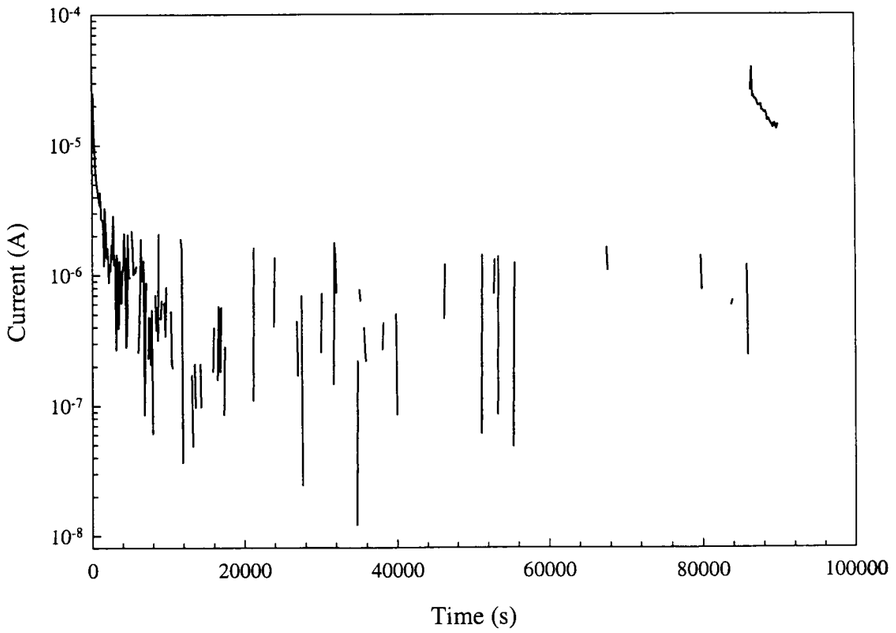
Final wt = 28.96836g 8-26-98

Final Solution PH = 11.297 8-26-98

Observations

No pitting 8-26-98

A516PS64



8-25-98 to 112

from 111

Cell 2 A516PS65.DAT

T = 65°C

Esat = -100 mV

Init wt = 28.90922g

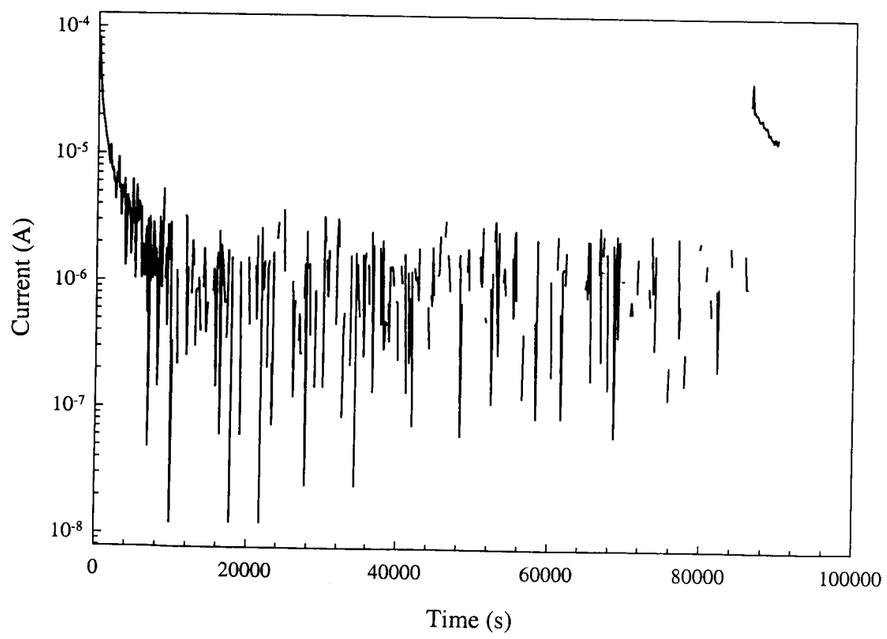
Final wt = 28.90955g 8-26-98

Final Station Pit = 11.132 8-26-98

Observations

Two small pitted spots, very small 8-26-98

A516PS65



8-25-98 to 113

from 112

Cell 3 A516PS66.DAT

T = 95°C

Esat = 225mV

Init wt = 28.77015g

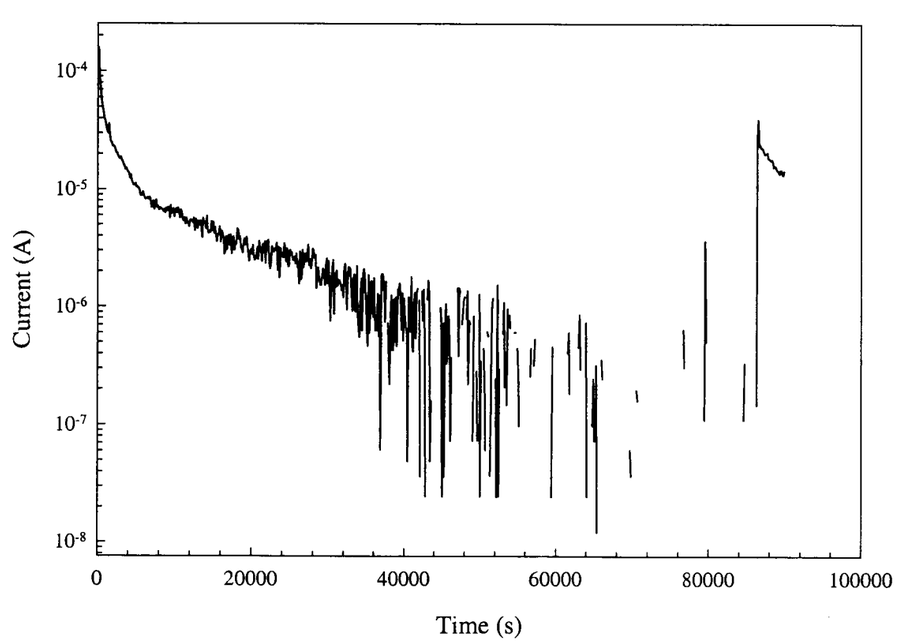
Final wt = 28.77013g 8-26-98

Final Station Pit = 10.944 8-26-98

Observations

Several pits on both sides but very small 8-26-98

A516PS66



8-25-98 to 114

from 113

8-26-98

Stock Solution

		Fisher Lot
$NaHCO_3$ → 120mM → 20.1624g/2Lt		#897789
Cl → 30mM → 3.5065g/2Lt		972274

Initial PH = 8.143

All Specimens polished to 600 grit & ultrasonically cleaned in Acetone, crevices ultrasonically cleaned in Methanol

8-26-98 to 115

from 114

Cell 1 A516PS67.DAT

T = 25°C

Eset = -300mv

Init wt = 29.01942g

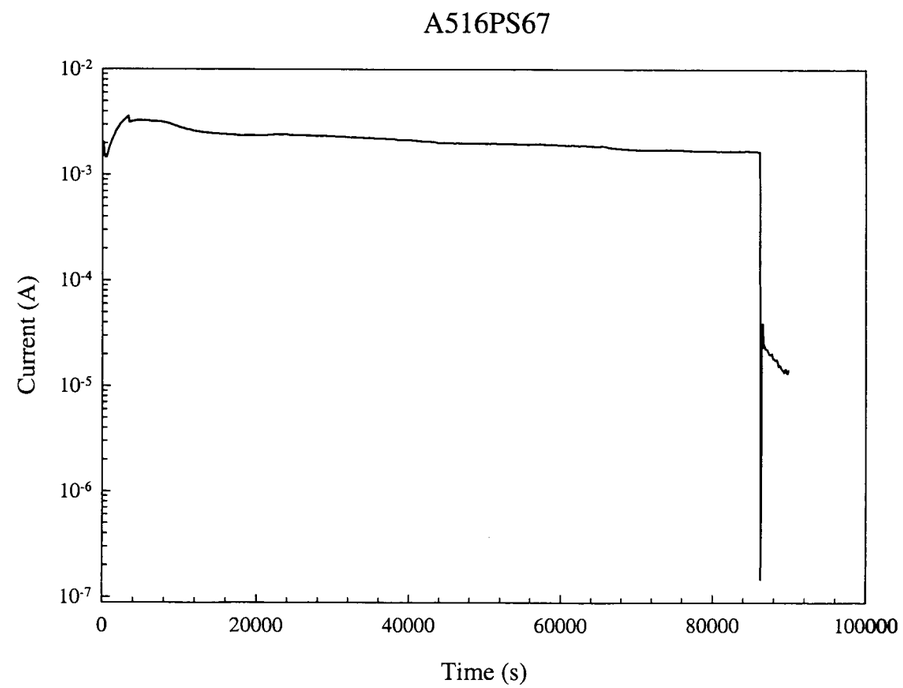
Final wt = 28.97915g 8-27-98

Final PH = 9.359 8-27-98

Observations

General corrosion on specimen.

Crevice foot protected specimen from pitting 8-27-98



8-26-98 to 116

from 115 | Cell 2 | A516PS68.DAT

T = 65 °C

Est = -370 mv

Init wt = 29.06937 g

Final wt = 28.99512 g 8-27-98

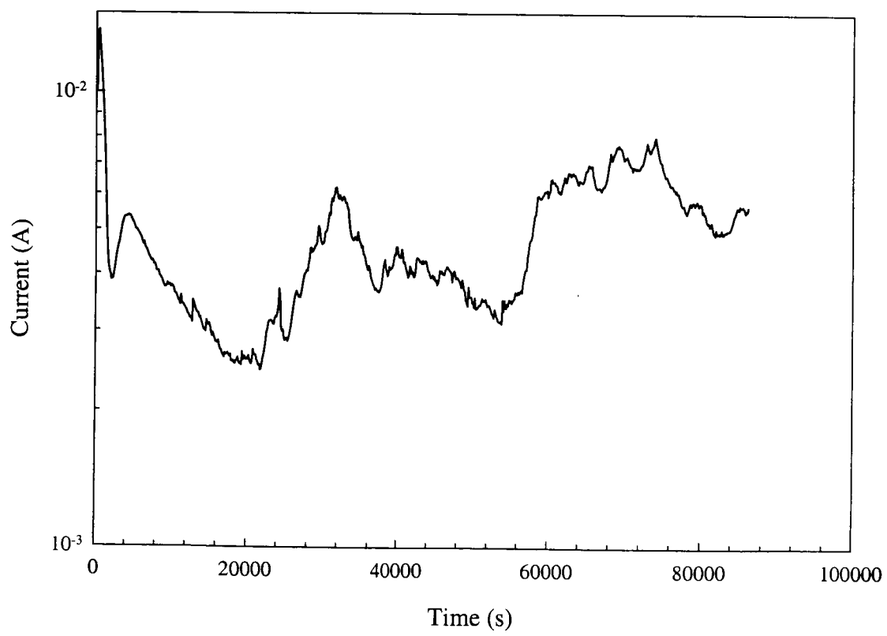
Final PH = 9.859 8-27-98

Observations

General corrosion on specimen with modest deposits

Crevice test protected specimen from pitting 8-27-98

A516PS68



8-26-98

to 117

from 116 | Cell 3 | A516PS69.DAT

T = 95°C

Est = -290 mv

Init wt = ~~29.01942 g~~ 28.82012 g

Final wt = 28.83614 8-27-98

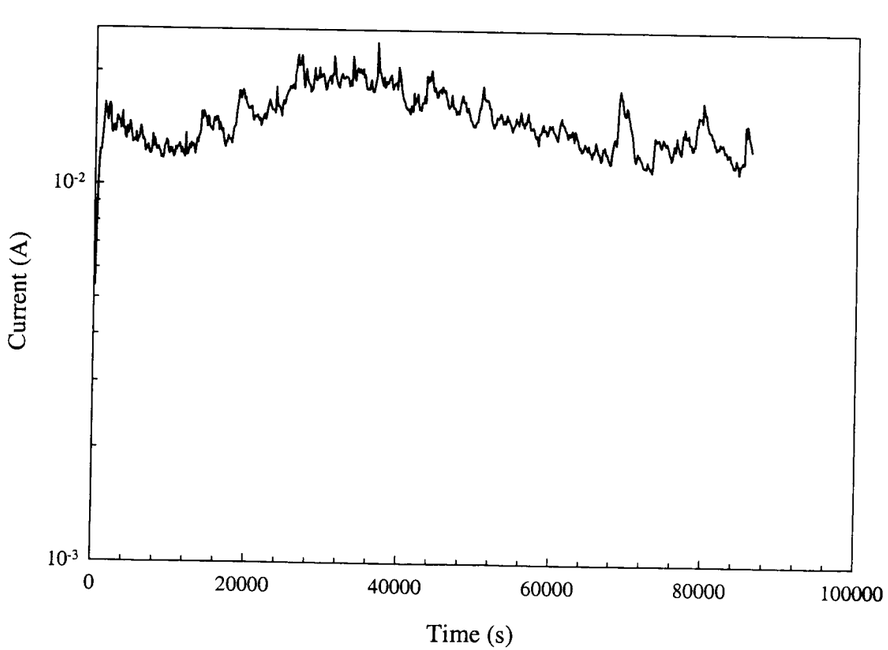
Final PH = 10.170 8-27-98

Observations

General corrosion on specimen with moderate deposits

Crevice test protected from pitting 8-27-98

A516PS69



8-26-98

to 118

from 117

8-27-98

Stock Solution — Fisher Lot

$NaHCO_3$ → 120mM → 20.1624g/2Lt — # 897789

NaCl → 12mM → 1.4026g/2Lt — # 972274

Initial PH = 8.125

All specimens polished to 600grit & ultrasonically cleaned in Acetone, specimens ultrasonically cleaned in methanol

8-27-98

to 119

from 118

Cell 1 A516PS70.DAT

T = 25°C

Fiset = -315mV

Init wt = 28.90293

Final wt = 28.89718g 8-28-98

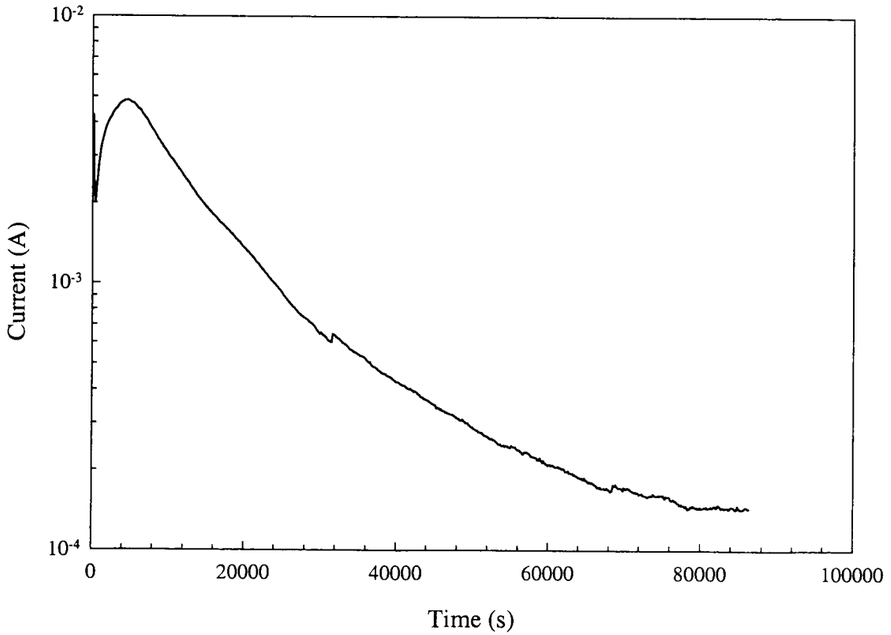
Final Solution ph = 9.169 8-28-98

Observations

General corrosion on specimen

no pitting under feet 8-28-98

A516PS70



8-27-98

to 120

from 119

Cell 2 A516PS71.DAT

T = 65°C

Eset = -340mV

Init wt = 28.79780g

Final wt = 28.77355g 8-28-98

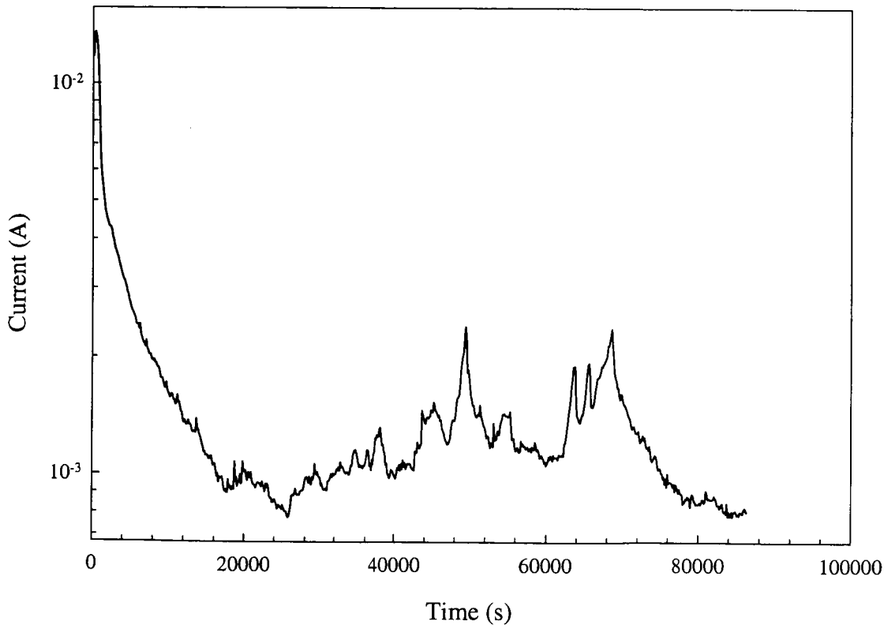
Final Solution pH = 9.821 8-24-98

Observations

General corrosion product on Spec 8-28-98

Minor pitting under crevice feet

A516PS71



8-27-98 to 121

from 120

Cell 3 A516PS72.DAT

T = 95°C

Eset = -260mV

Init wt = 28.87609g

Final wt = 28.84266g 8-24-98

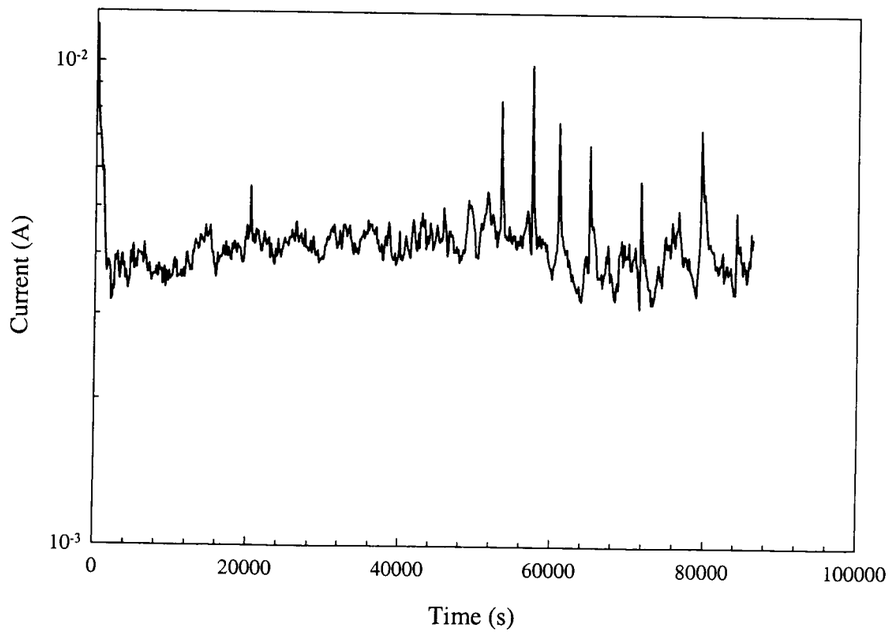
Final Solution pH = 10.077 8-28-98

Observations

General corrosion product on Spec

No pitting under feet 8-28-98

A516PS72



8-27-98 to 122

from 121

8-31-98 Start 9:46 Am

Stock Solution Fisher lot

$NaHCO_3$ → 120mM → 20.1624 g/2Lt # 897789

NaCl → 4.8mM 0.5610 g/2Lt 972274

Init PH = 8.142

All specimens polished to 600 grit & ultrasonically cleaned in Acetone

Crevices ultrasonically cleaned in Methanol

NOTE:

Power Failure occurred, 9-1-98

Data Acquisition Shutdown

8-31-98 to 123

from 122

Cell 1 A516PS73.DAT

T = 25°C

Eset = -305 mV

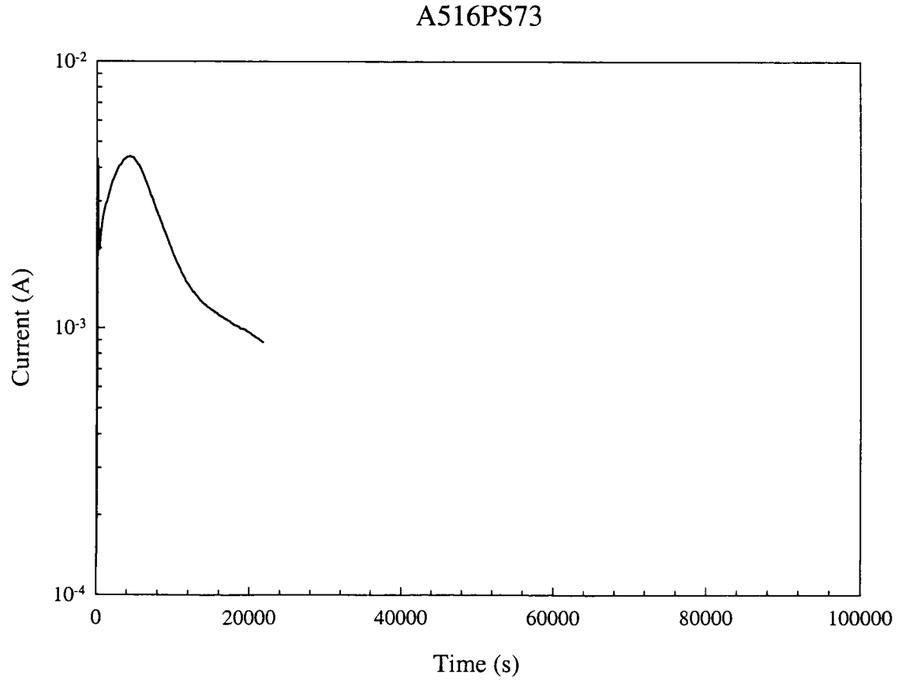
Init wt = 28.79218 g

Final wt = 28.78509 g 9-1-98

Final Solution PH = 9.134 9-1-98

Observations

some general corrosion, some undercutting at feet 9-1-98



8-31-98 to 124

from 123 Cell 2 A516PS74.DAT

T = 65°C

Eset = -310mV

Init wt = 28.73502g

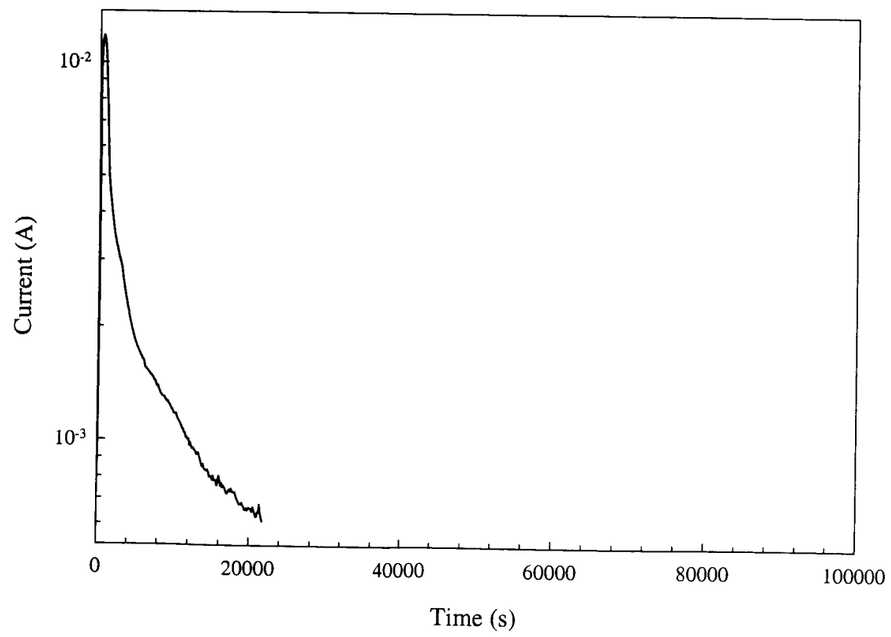
Final wt = 28.73270g 9-1-98

Final Solution pH = 9.755 9-1-98

Observations

Some general corrosion, more under feet 9-1-98

A516PS74



8-31-98 to 125

from 124 Cell 3 A516PS75.DAT

T = 95°C

Eset = -230mV

Init wt = 29.03454g

Final wt = 29.02792g 9-1-98

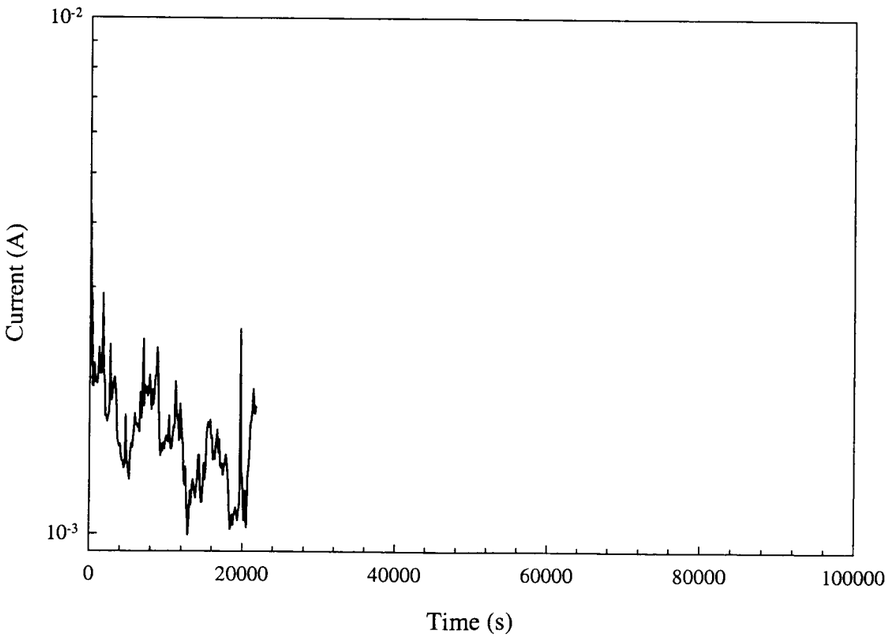
Final Solution pH = 10.526 9-1-98

Observations

some corrosion pitting on bottom of specimen

crevice pitting under feet and on edges of feet 9-1-98

A516PS75



8-31-98 to 126

from 125

9-1-98
Stock Solution
$NaHCO_3$ → 120mM → 20.1624g/2Lt Fisher # 897789
NaCl → 4.8mM → .5610 g/2Lt " 972274

Init pH = 8.149

All Specimens polished to 600 grit + ultrasonically cleaned in de-ione
Crevices ultrasonically cleaned in Methanol

9-1-98

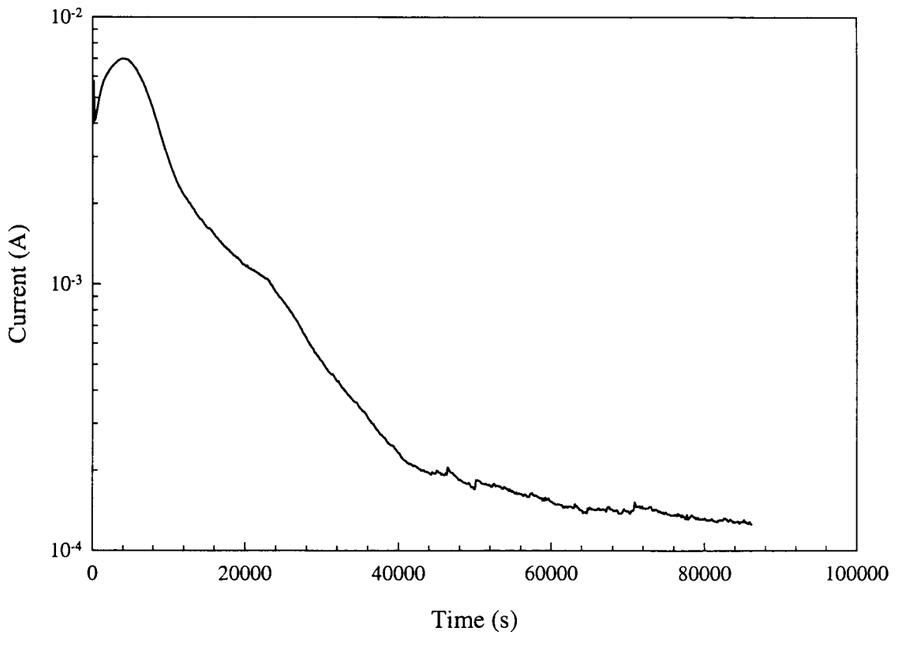
to 127

from 126

Cell 1 A516PS76.DAT
T = 25°C
Eset = -305mV
Init wt = 28.96249g
Final wt = 28.95584g 9-2-98

Final Solution pH = 9.161 9-2-98
Observations
General corrosion, none under feet 9-2-98

A516PS76



9-1-98

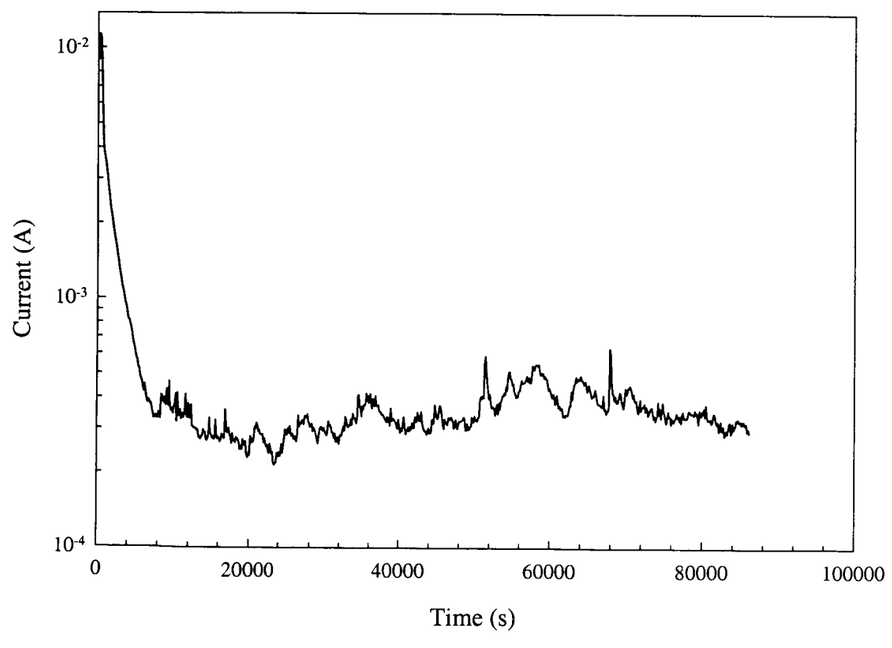
to 128

from 127

Cell 2 A516PS77.DAT
T = 65°C
Eset = -310mv
Init wt = 28.81691g
Final wt = 28.81041g 9-2-98

Final Solution pH = 9.947 9-2-98
Observations
Corrosion above vaperline, some pitting under crevice foot. 9-2-98

A516PS77



9-1-98

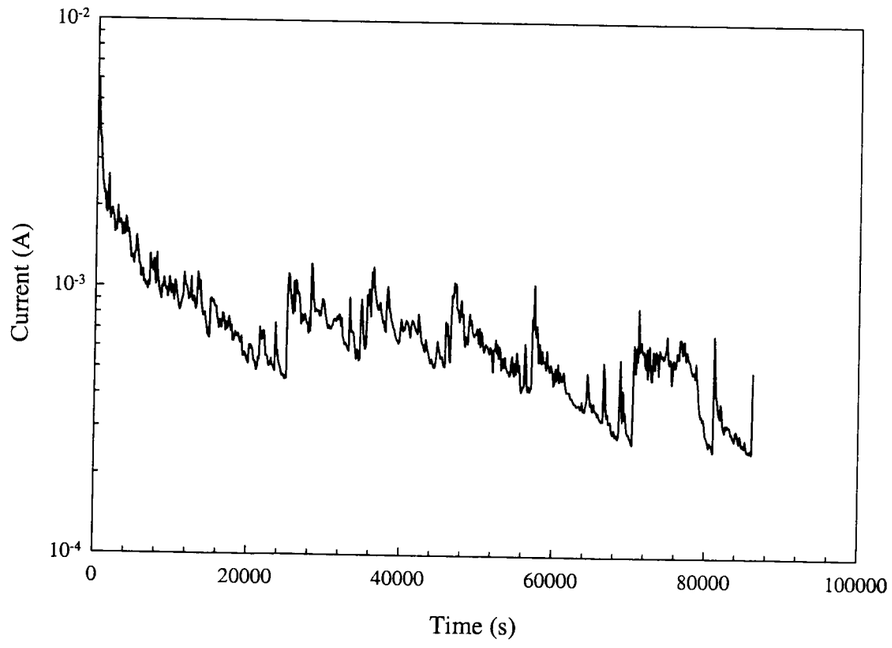
To 129

from 128

Cell 3 A516PS78.DAT
T = 95°C
Eset = -230
Init wt = 28.75483g
Final wt = 28.75318g 9-2-98

Final Solution pH = 11.223 9-2-98
Observations
Crevice pitting, some general corrosion 9-2-98

A516PS78



9-1-98

to 130

from 129

9-2-98

Stock Solution

		F. for Lot #
$NaHCO_3$ → 120 mM	→ 20.1614 g /2Lt	897789
NaCl → 2.4 mM	0.2805 g /2Lt	972274

Initial PH = 8.166

All specimens polished to 600 grit + ultra Sonicall, cleaned in Acetone Crevices ultrasonically cleaned in Methanol

9-2-98

To 131

From 130

Cell 1 A516PS79.MAT

T = 25°C

Eset = -300mv

Init wt 28.70445g

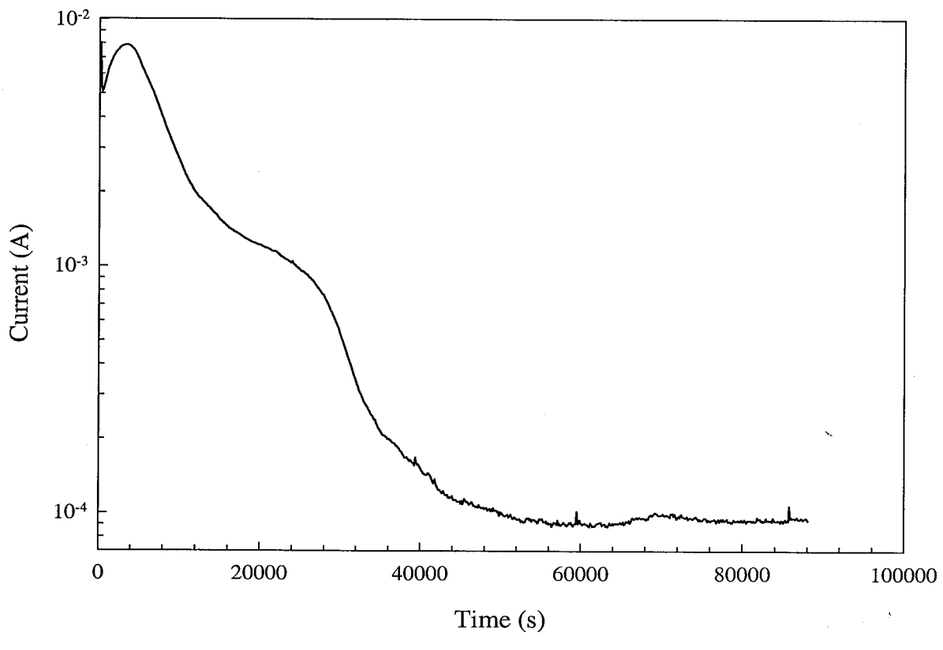
Final wt. 28.69709g 9-3-98

Final PH 9.168 9-3-98

Observations

General corrosion, None under crevice Coat 9-3-98

A516PS79



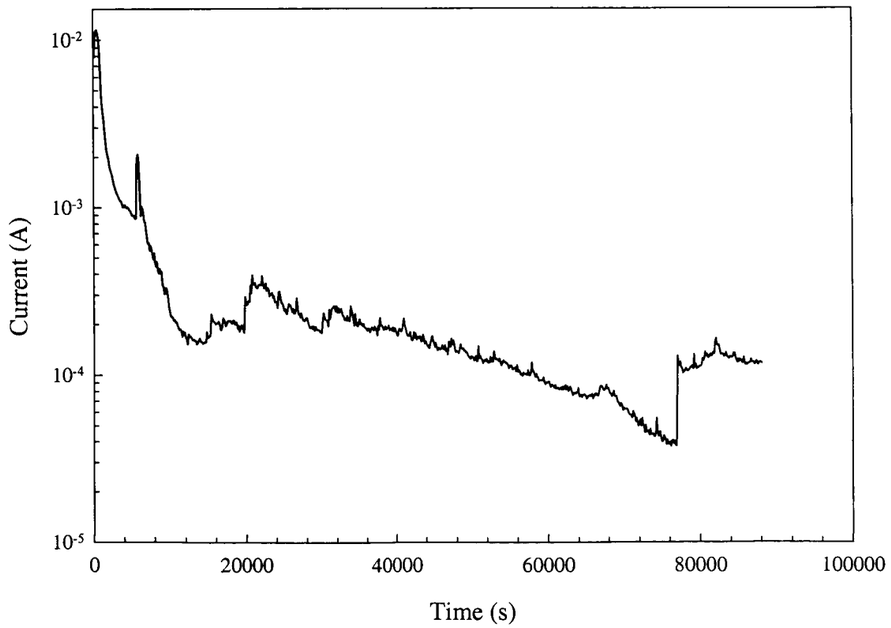
9-2-98

To 132

from 131 Coll 2

A516PS80.DAT
T = 65°C
Eset = -280mv
I-it wt = 28.93610g
F-al wt = 28.93348g 9-3-98
F-al Solution = 9.983 9-3-98
Observation:
Some general corrosion on shaft
minor crevice pitting 9-3-98

A516PS80

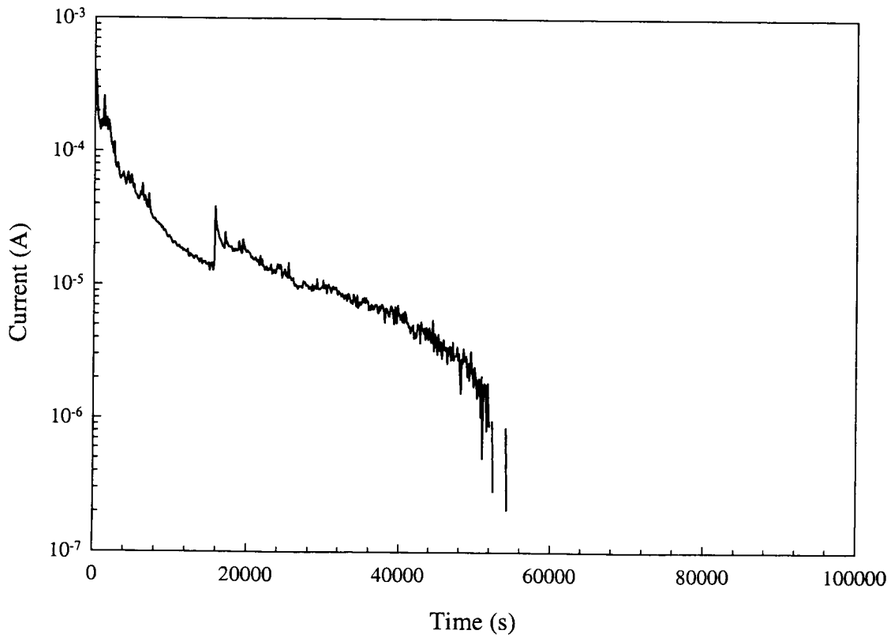


9-2-98 To 133

from 132

A516PS81.DAT
T = 95°C
Eset = -210 mv
I-it wt = 28.60858g
F-al wt = 28.60984g 9-3-98
F-al PH = 11.030 9-3-98
Observations
No general corrosion fairly uniform 9-3-98
crevice pitting

A516PS81



9-2-98 To 134

from 133

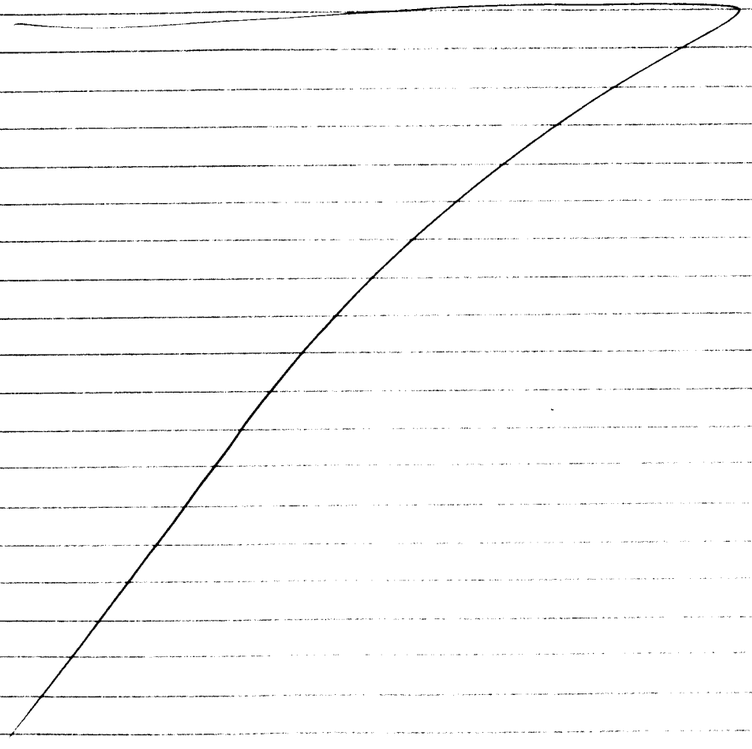
Stock Solution Sol'd 2:37 pm

$NaHCO_3$ → 120mM → 20.164g/2L Fisher # 897398

NaCl → 1.6mM → 0.1870g/2L Fisher # 972274

Initial PH: 8.164

All specimens polished to 600 grit + ultrasonically cleaned in Acetone crevices cleaned in methanol ultrasonically



9-3-98

to 135

from 134

Cell 1 A516PS82.DAT

T = 85°C

Eset = -290mV

In. wt = 28.96751g

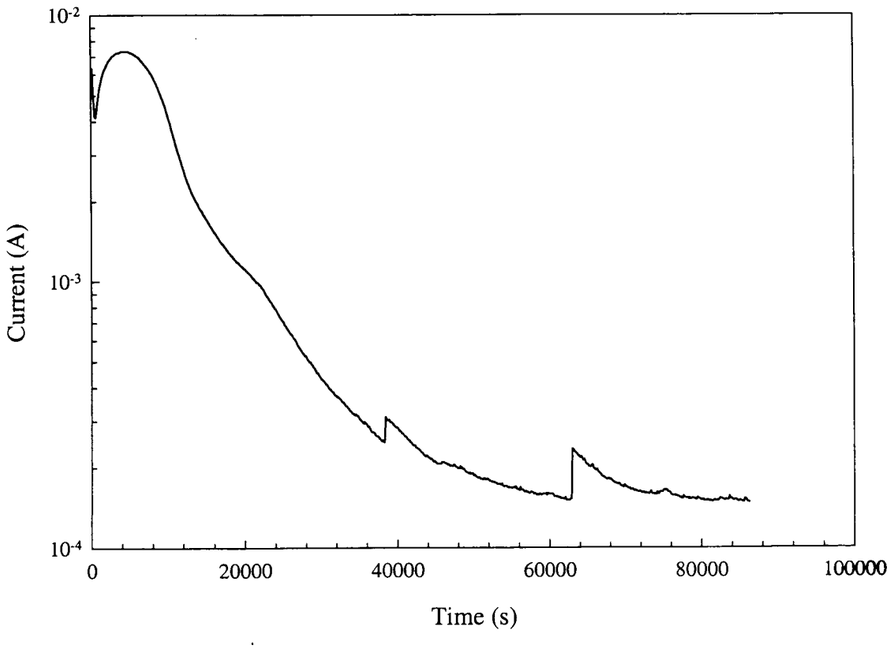
Final wt = 28.95940g 9-4-98

Final Solution PH = 9.145 9-4-98

Observations

General corrosion, more under foot 9-4-98

A516PS82



9-3-98

To 136

from 135 Cell2 A516PS83.DAT

T = 65°C

ESet = -270 mv

In. wt = 28.88775g

Final wt = 28.88693g 9-4-98

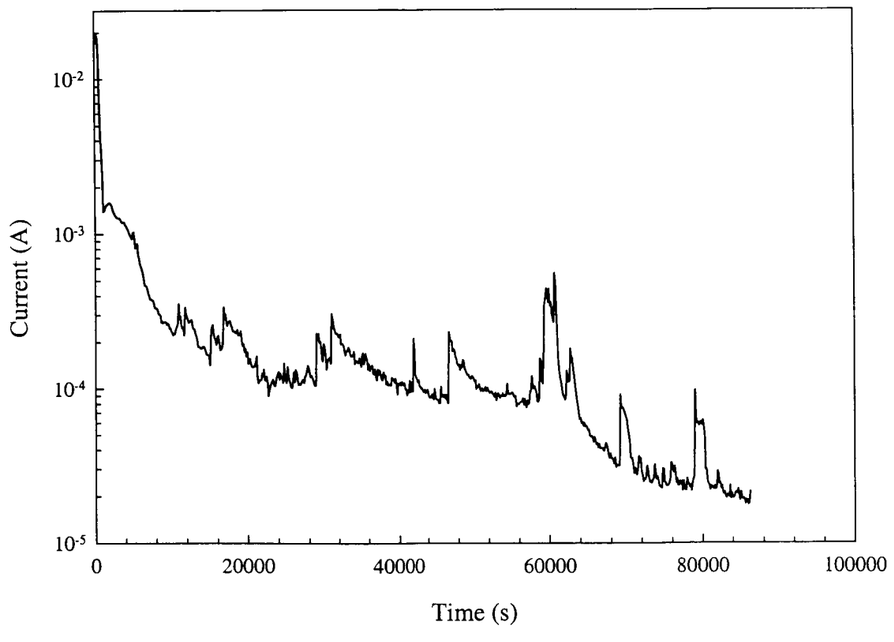
Final Solution PH = 9.858 9-4-98 9-4-98

Observations

Some corrosion above waterline

minor pitting under crevice foot

A516PS83



9-3-98

from 136 Cell3 A516PS84.DAT

T = 95°C

ESet = -200 mv

In. wt = 29.07900g

Final wt = 29.07920g 9-4-98

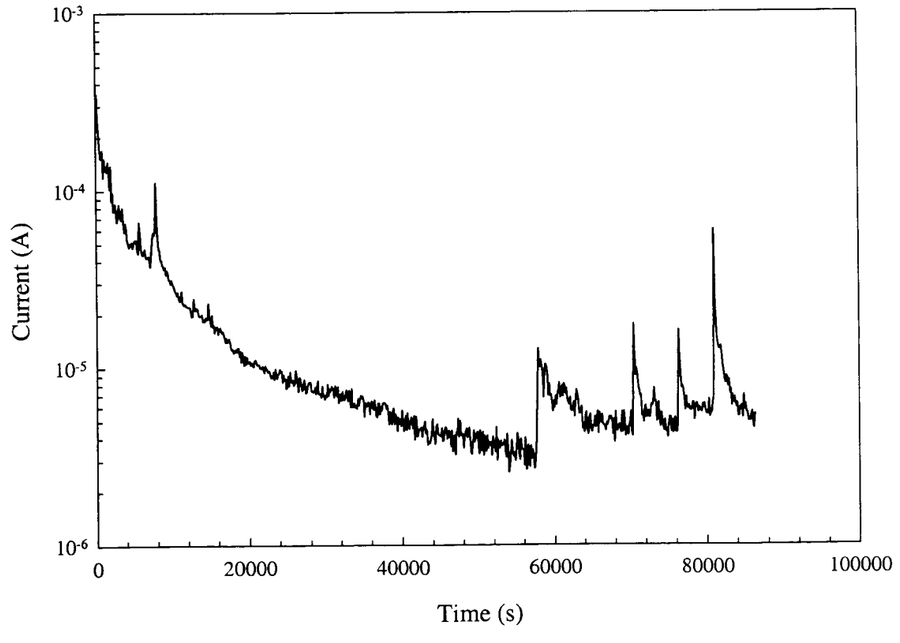
Final Solution PH = 10.685 9-4-98

Observations

crevice pitting under foot 9-4-98

no other corrosion noted

A516PS84



9-3-98

from 137 9-8-98 Start 9:53

Stock Solution Fisher #

$NaHCO_3$ → 120 mM → 20.1624 g / 2 Lt 897789

NaCl → 1.2 mM → 0.1403 g / 2 Lt 972274

Initial pH = 8.180

All specimens polished to 600 grit & ultrasonically cleaned in Acetone
All crevices ultrasonically cleaned in methanol

9-8-98 To 138

from 138 Cell 1 A516PS85.DAT

T = 25°C

Eset = -290 mV

Init wt = 28.92722 g

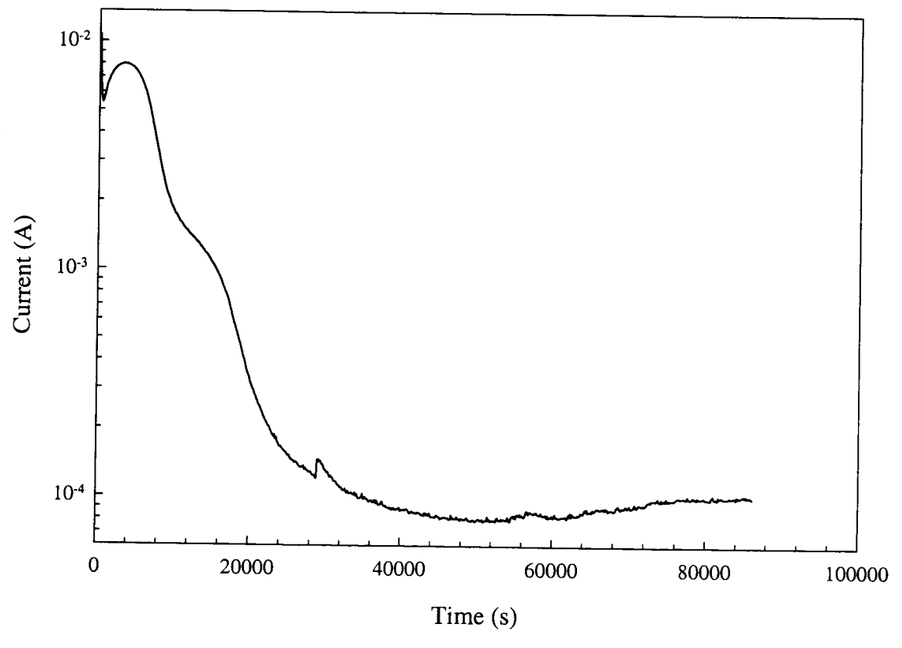
Final wt = 28.91933 g 9-9-98

Final Solution pH = 8.958 9-9-98

Observations

General Corrosion, no crevice pitting 9-9-98

A516PS85



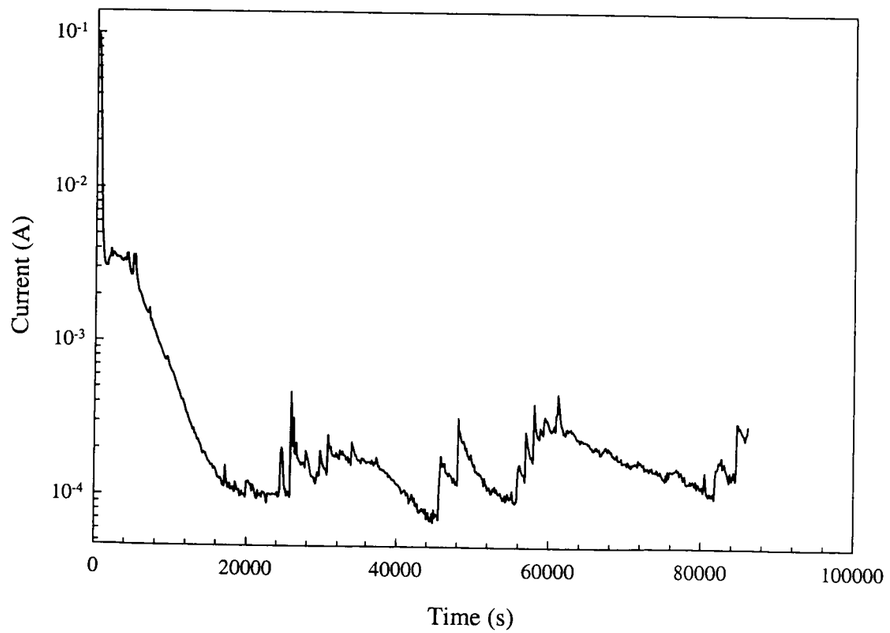
9-8-98 To 140

from 139

Cell 2 A516PS86.DAT
T = 65°C
Eset = -260 mv
Init wt = 28.79420g
Final wt = 28.79300g 9-9-98

Final Solution pH = 9.465 9-9-98
Observations
General Corrosion, no crevice pitting 9-9-98

A516PS86



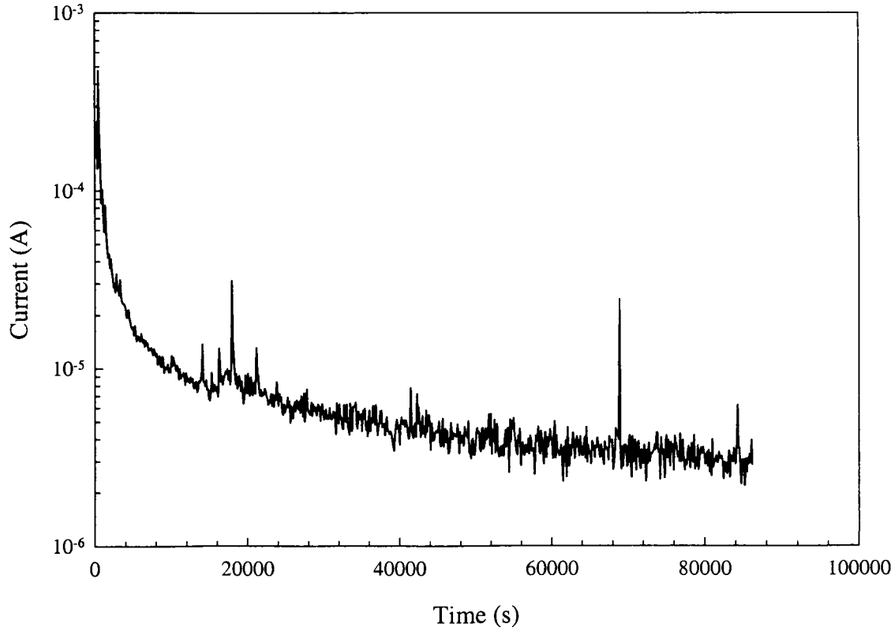
9-8-98 To 141

From 140

Cell 3 A516PS87.DAT
T = 95°C
Eset = -190 mv
Init wt = 28.73913g
Final wt = 28.73965g 9-9-98

Final Solution pH = 10.961 9-9-98
Observations
No general Corrosion, minor crevice pitting 9-9-98

A516PS87



9-8-98 To 142

From 141 9-9-98

Stock Solution

$NaHCO_3$ → 6mM → 1.008g/2Lt Fisher # 897789

NaCl → 1.2mM → 0.1403g/2Lt " 972274

Init PH = 7.57 @ 1min 8.36 after 5min

All specimens polished to 600grit & ultrasonically cleaned in Acetone

All crevices ultrasonically cleaned in Methanol

9-9-98

To 143

From 142 Cell 1 A516PS88.DAT

T = 25°C

Eset = −290mV

Init wt = 28.92371g

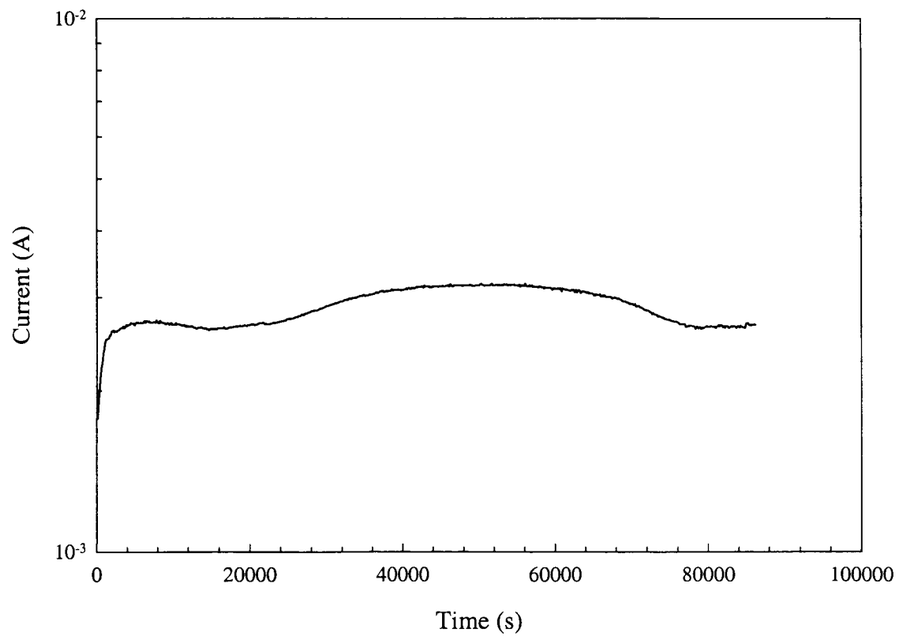
Final wt = 28.84964g 9-10-98

Final Solution PH = 9.428 9-10-98

Observations

General corrosion, crevice feet showed no pitting 9-10-98

A516PS88



9-9-98

To 144

From 143 | Cell 2 | A516 PS89.DAT

T = 65°C

Esat = -260mv

Init wt = 28.78665g

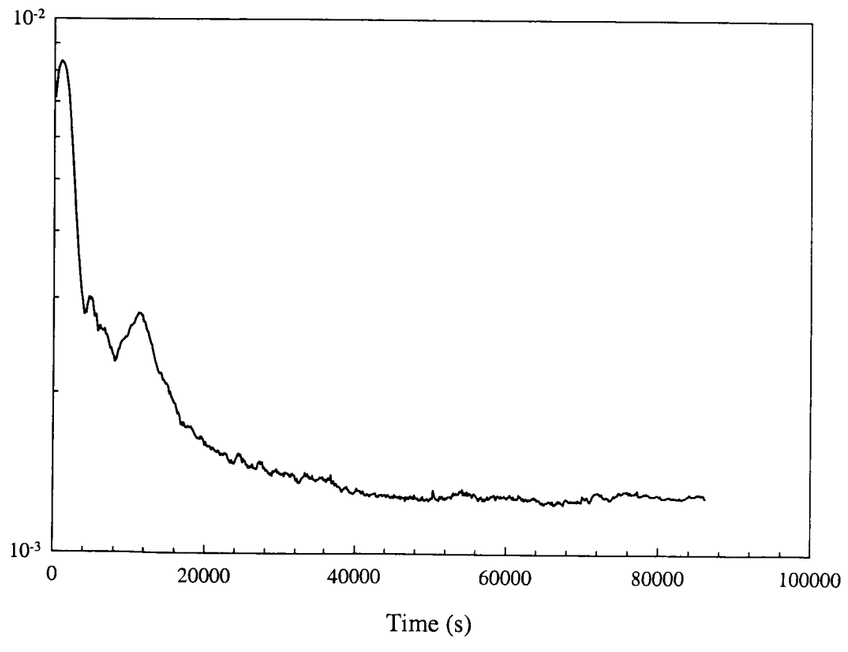
Final wt = 28.75611g 9-10-98

Final Solution PH = 9.625 9-10-98

Observations

General corrosion, No crevice pitting 9-10-98

A516PS89



9-9-98 To 145

From 144 | Cell 3 | A516 PS90.DAT

T = 95°C

Esat = -190mv

Init wt = 29.002 14g

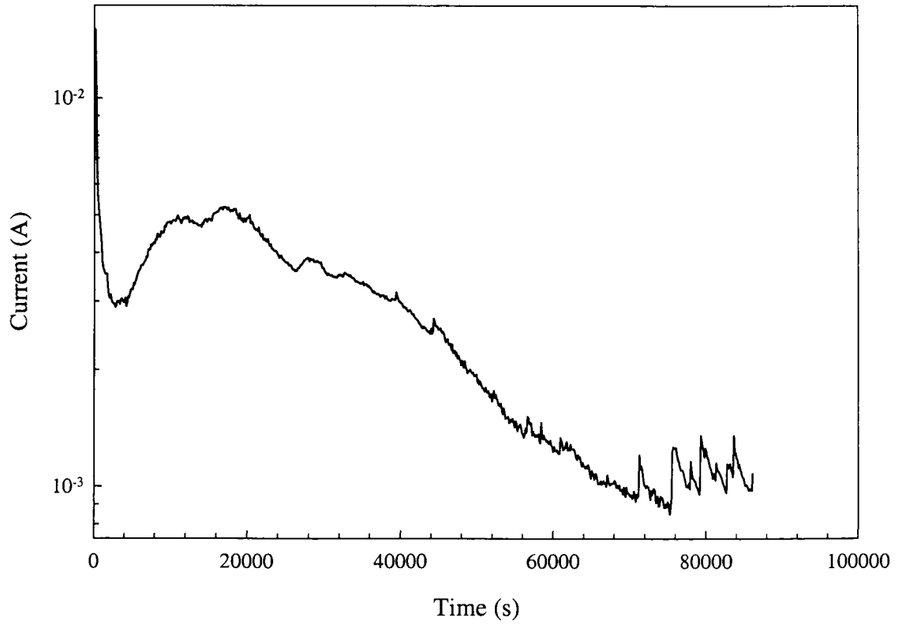
Final wt = 28.9515g 9-10-98

Final Solution PH = 10.342 9-10-98

Observations

Some general corrosion and strital pitting around crevice foot but not under foot 9-10-98

A516PS90



9-9-98 To 146

From 145

9-10-98 Start 1:20pm

Stock Solution Fisher #

$NaHCO_3$ → 6 mM → 1.008 g/2Lt 897789

NaCl → .48 mM → .0561g/2Lt 972274

Init PH = 7.499 @ 1min 8.49 after 5

All specimens polished to 600 grit & ultrasonically cleaned in Acetone

All crevices ultrasonically cleaned in Methanol

9-10-98

To 147

From 146

Cell 1 A516PS91.DAT

T = 25°C

Eset = -270

Init wt = 28.80670g

Final wt = 28.68025g 9-11-98

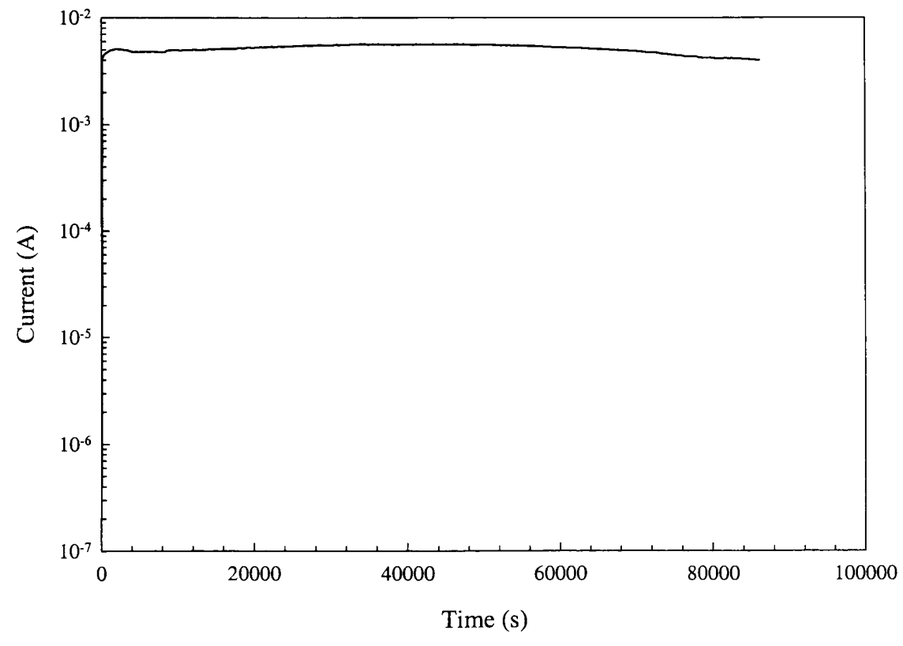
Final Solution PH = 10.082 9-11-98

Observations

Precipitate on specimen, general corrosion 9-11-98

No crevice pitting

A516PS91



9-10-98

To 148

From 147

Cell 2 A516PS92.DAT

T = 65°C

Eset = -250mV

Init wt = 29.04891g

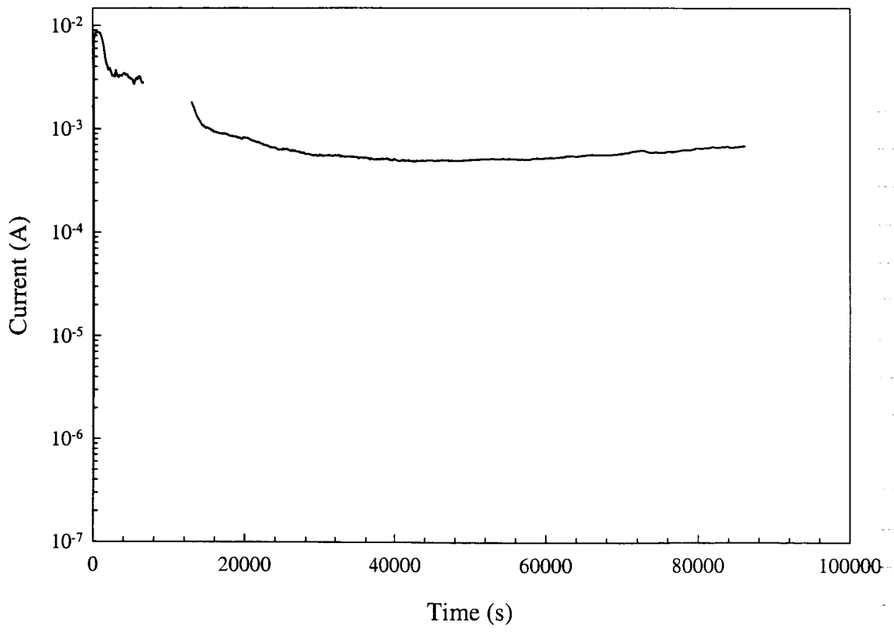
Final wt = 29.03300g 9-11-98

Final Solution PH = 9.756 9-11-98

Observations

General Corrosion, no crevice pitting 9-11-98

A516PS92



9-10-98

To 149

From 148

Cell 3 A516PS93.DAT

T = 95°C

Eset = -170 mV

Init wt = 28.94460g

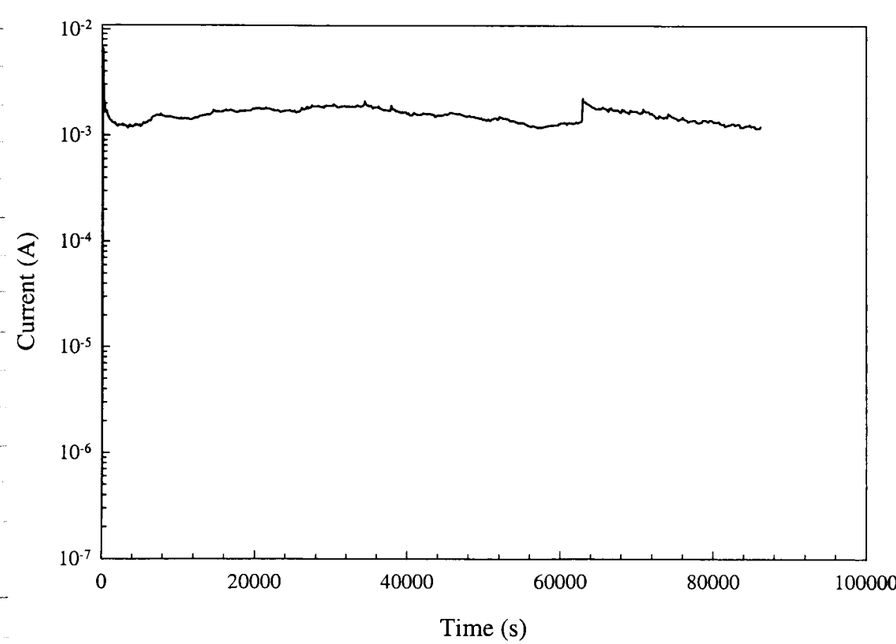
Final wt = 28.91372g 9-11-98

Final Solution PH = 9.743 9-11-98

Observations

Some minor general corrosion crevice pitting 9-11-98

A516PS93



9-10-98

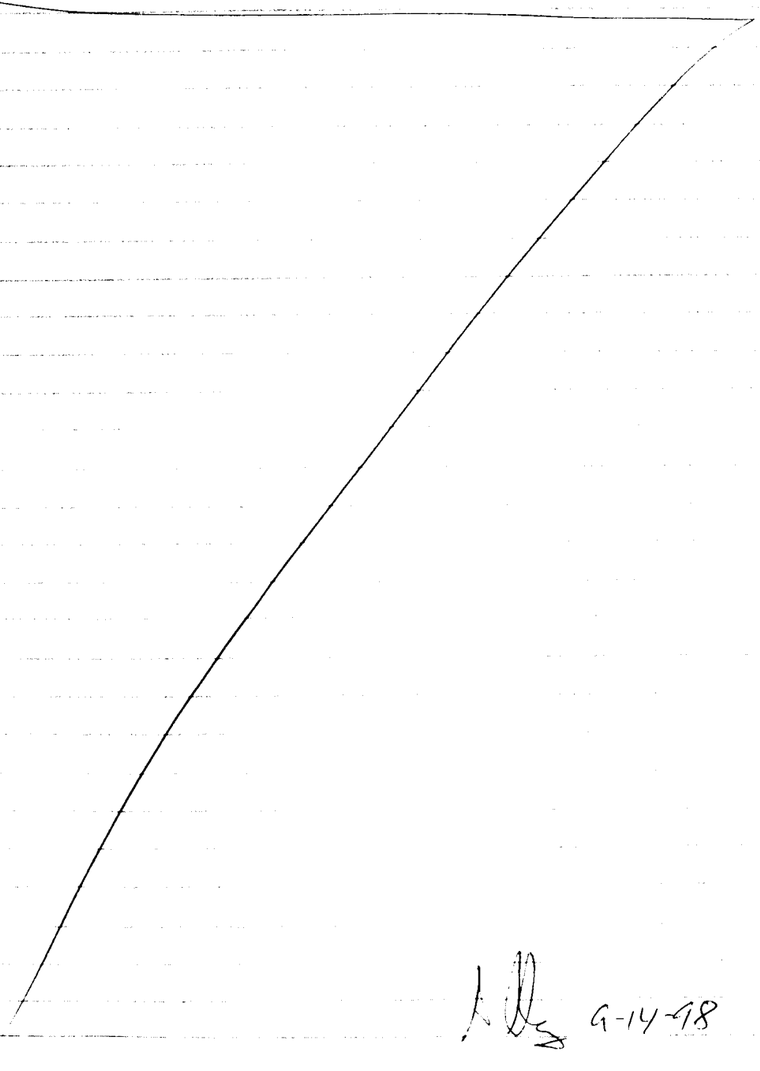
To 150

From 149

Stock Solution
9-14-98
$NaHCO_3$ → 6mM → 1.008g/2Lt Fisher # 897789
NaCl → .12mM → .01403g/2Lt " # 972274

Final pH = 8.428

All Specimens polished to 600grit + ultrasonically cleaned in Acetone
Crevices cleaned ultrasonically in Methanol



9-14-98

To 151

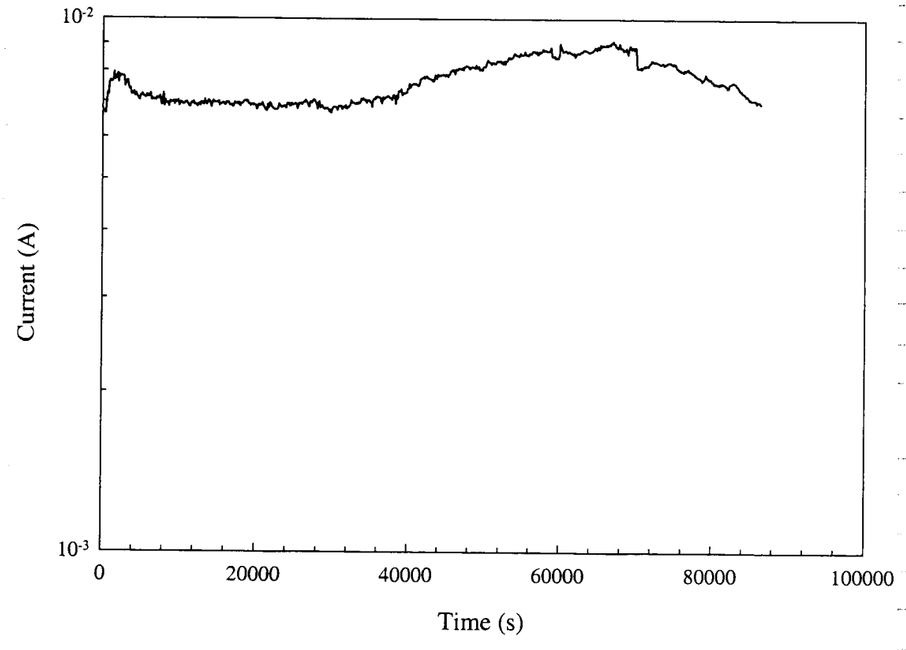
From 150

Cell 1 A516PS94.DAT
T = 25°C
Eset = -250mV
Init wt = 28.90233g 9-15-98
Final wt = 28.71316g

Final Solution pH = 10.247 9-15-98

Observations
General Corrosion, no crevice pitting 9-15-98

A516PS94



9-14-98

To 152

From 151 Cell 2 A516PS95.DAT

T = 65°C

Eset = -220mv

Initwt: 29.01220g

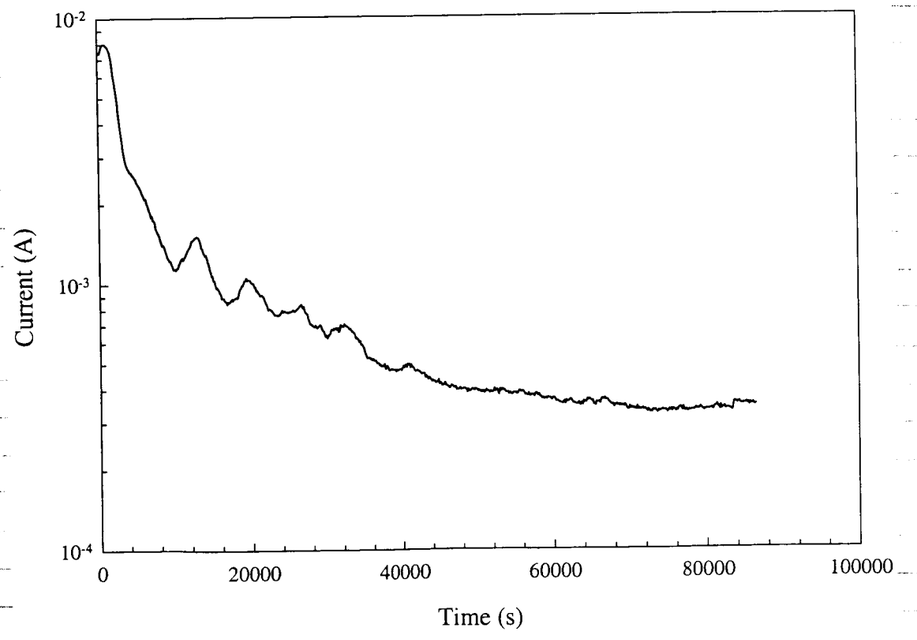
Final wt: 29.00144g 9-15-98

Final Solution PH-9.598 9-15-98

Observations

General Corrosion, No crevice pitting 9-15-98

A516PS95



9-14-98

To 153

From 152 Cell 3 A516PS96.DAT

T = 95°C

Eset: -150mv

Initwt: 29.00522g

Final wt: 28.99275g 9-15-98

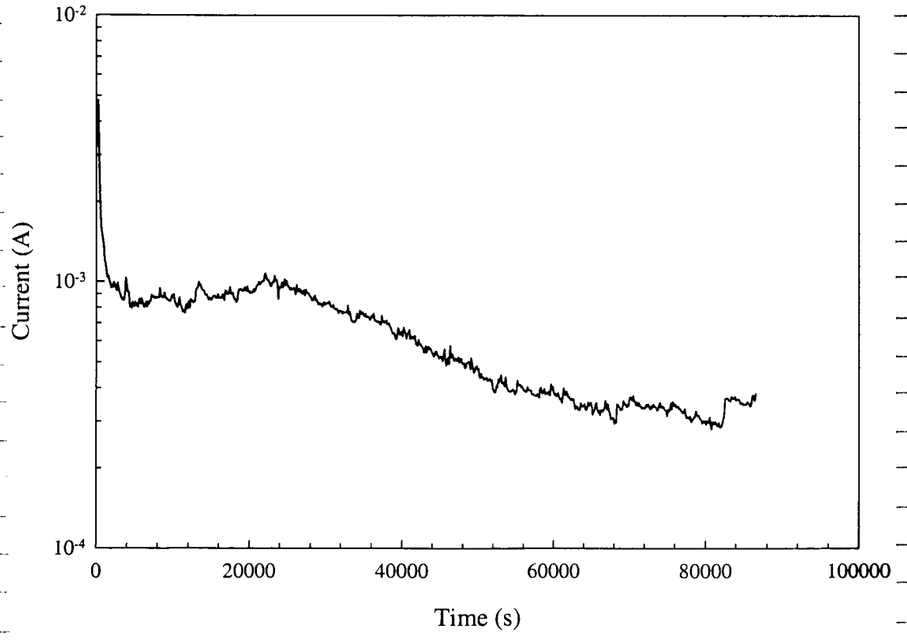
Final Solution PH: 10.504 9-15-98

Observations

One spot of pitting on sample bottom

crevice pitting around crevice edges 9-15-98

A516PS96



9-14-98

To 154

From 153

Stock Solution

Na_2CO_3 → 3mM → .63594g/2Lt Fisher # 960685
$NaHCO_3$ → 3mM → .50406 g/2Lt " 879789
NaCl → 1.2mM → .1403 g/2Lt " 972274

Solution pH = 10.159

All specimens polished to 600grit ultrasonically cleaned in Acetone
Crevices ultrasonically cleaned in Methanol

9-15-98

To 155

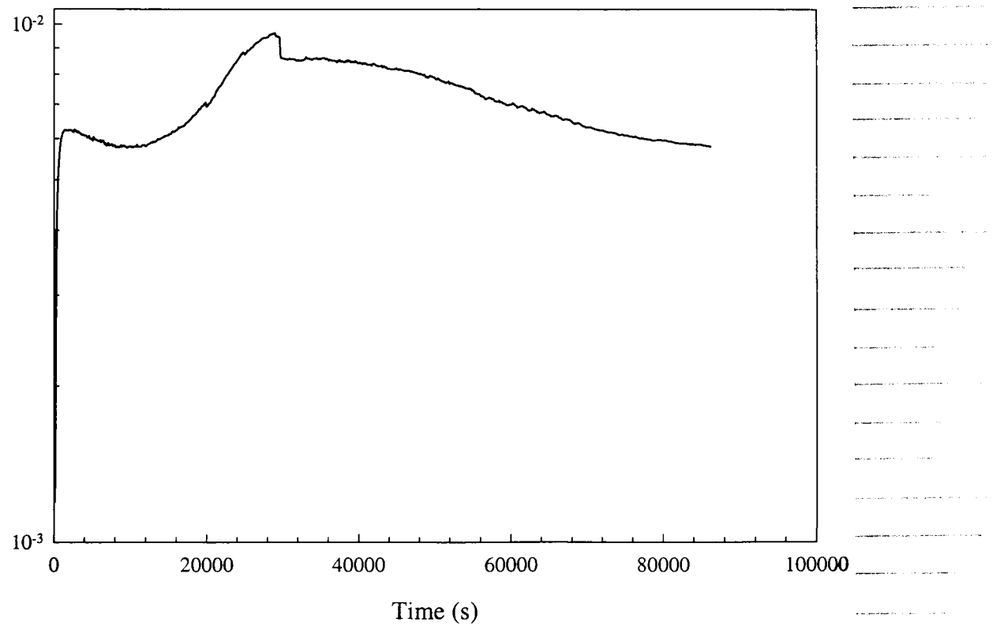
From 154

Cell 1 A516PS97.DAT
T = 25°C
Eset = -290 mV
Initial wt = 28.81978g
Final wt = 28.63596g 9-16-98

Final Solution pH: ~~10.~~ 11.012 9-16-98

Observations
Large amount of precipitate + general corrosion 9-16-98
No crevice pitting

A516PS97



9-15-98

To 156

From 155

Cell2 A516PS98.DAT

T=65°C

Eset=-260mv

Init wt: 28.85600g

Final wt: 28.66316g 9-16-98

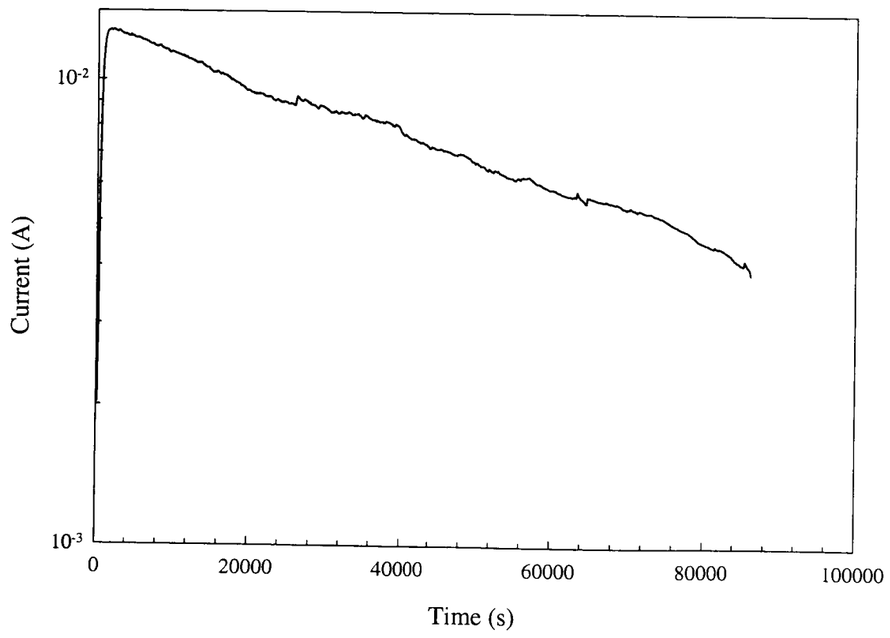
Final Solution PH: 10.762 9-16-98

Observations

Large amount of precipitate and general corrosion 9-16-98

No crevice pitting

A516PS98



9-15-98

To 157

From 156

Cell3 A516PS99.DAT

T=95°C

Eset=-190mv

Init wt: 28.83070g

Final wt: 28.72598g 9-16-98

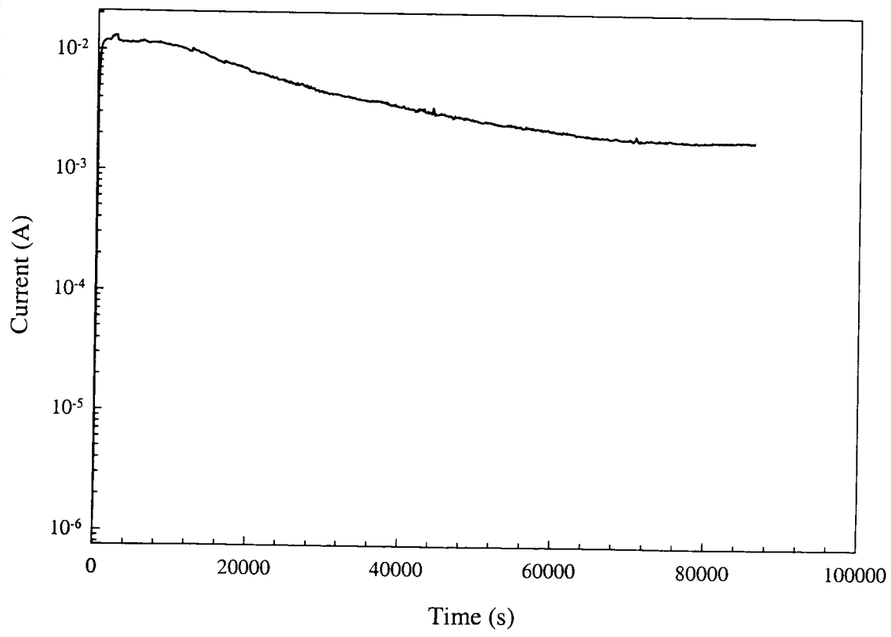
Final Solution PH: 10.743 9-16-98

Observations

Some general corrosion with precipitate

crevice pitting evident 9-16-98

A516PS99



9-15-98

To 158

From 157

9-16-98

Stock Solution

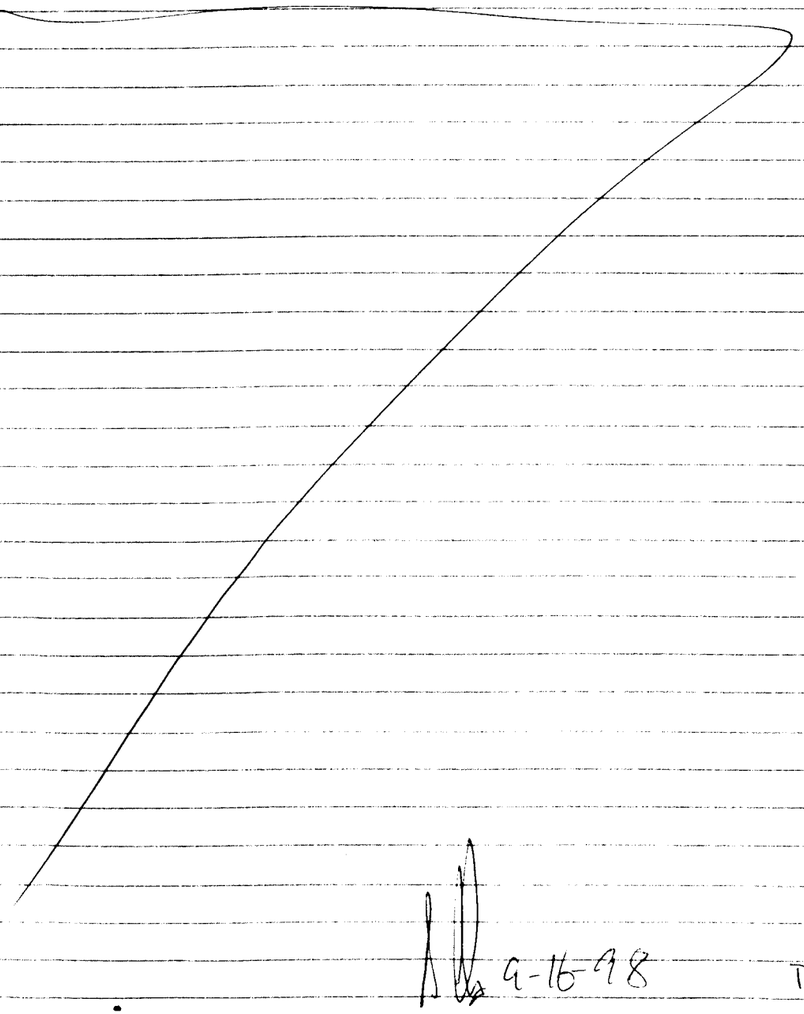
Na_2CO_3 → 3mM → .63594g/2Lt Fisher # 960685

$NaHCO_3$ → 3mM .50406g/2Lt " 879789

NaCl → .48mM → .0561g/2Lt " 972274

PH = 10.170

All specimens polished to 600grit + ultrasonically cleaned in Acetone
crevices ultrasonically cleaned in Methanol



9-16-98

To 159

From 158

Cell 1 516PS100.DAT

T = 25°C

Eset = -270mv

Initwt = 28.67141

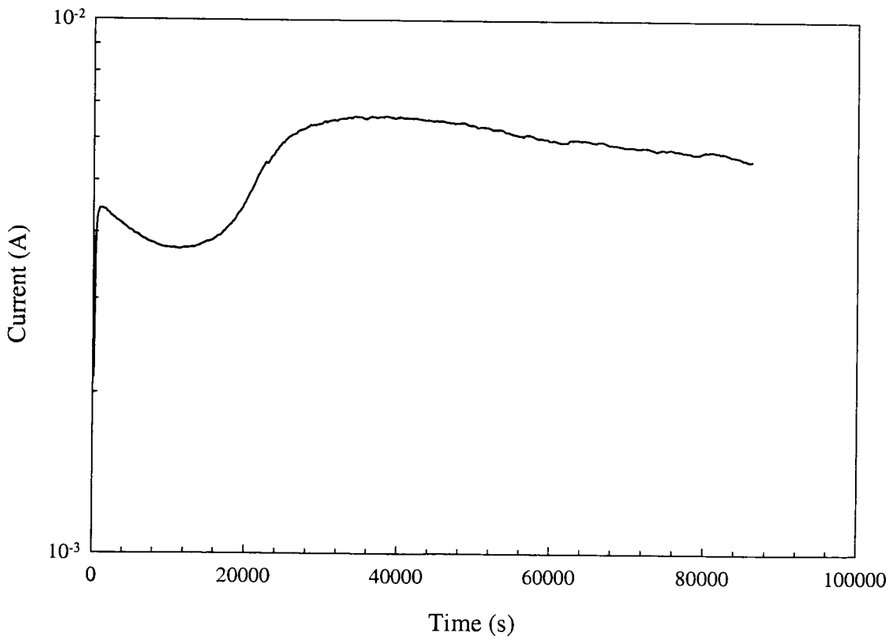
Final wt = 28.52590 9-17-98

Final Solution PH = 10.882 9-17-98

Observations

large amount of precipitate & general corrosion
no crevice pitting 9-17-98 gc

516PS100



9-16-98

To 160

Cell2 516PS101.DAT
T=65°C
Eset=-240mv
Init wt=29.17760g
Final wt=29.08168g 9-17-98
Final PH=10.44 9-17-98
Observations
large amount of precipitate tgenoral corrosion 9-17-98
No service pitting

516PS101

9-16-98

Cont on Page 7 of Book 291

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

2/11/2005