



U.S. Department of Energy  
Office of Civilian Radioactive Waste Management

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# Fabrication Effects on the Corrosion Behavior of Alloy 22

Presented to:  
**Nickel Institute Workshop No. 6**

Presented by:  
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**Las Vegas, Nevada**

# Objectives

- Show the Influence of Fabrication Processes of the Container on the Corrosion Behavior of Alloy 22
- Studied Variables
  - Heat-to-Heat Variability (In the Range of B 575)
  - Effect of Black Annealing Scale on General and Localized Corrosion Behavior
  - Effect of Surface Stress Mitigation
  - Behavior of Specimens Prepared from a Full-Diameter Mock-up Container
- Conclusions



# Part 1

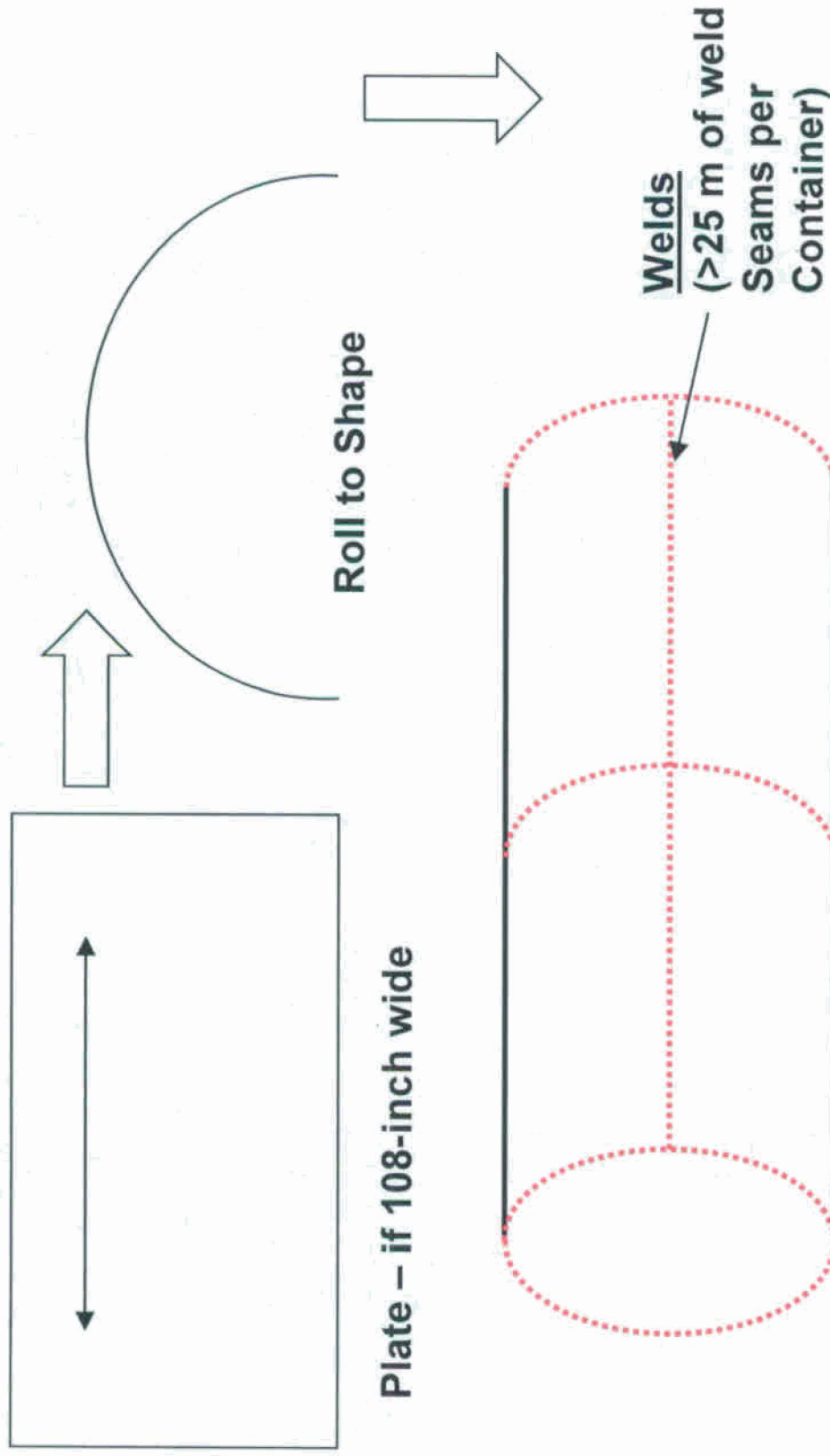
## Heat-to-Heat Variability Studies

### Immersion Tests





# Fabrication by GTA Welding



**Weld longitudinally and 2 cylinders side by side + bottom lid**

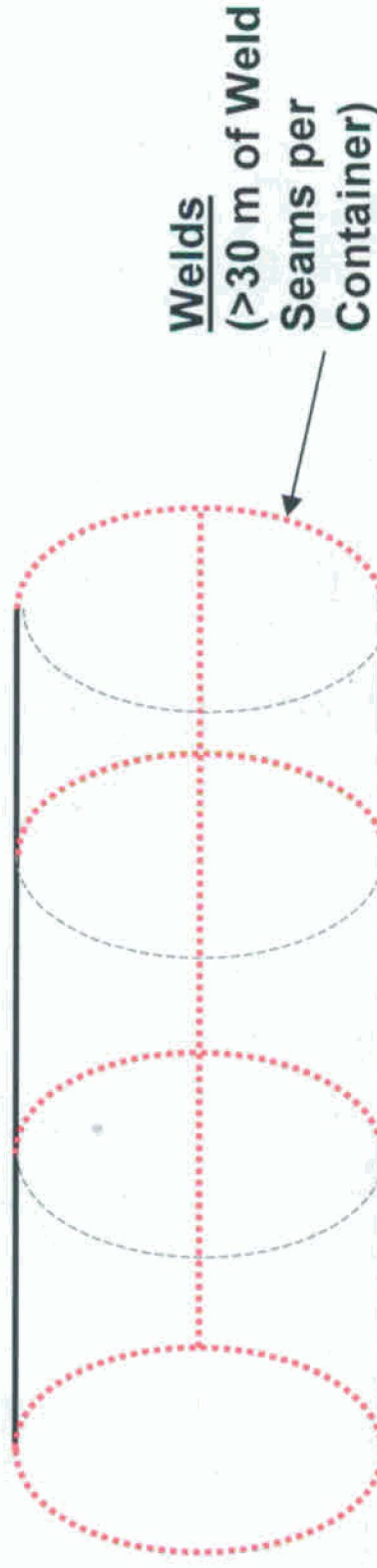




# Fabrication by GTA Welding

All Welds but the Final Closure Weld will be Solution Annealed for Homogenization and To Eliminate Residual Stresses

The final Weld will be treated in-situ to mitigate stresses

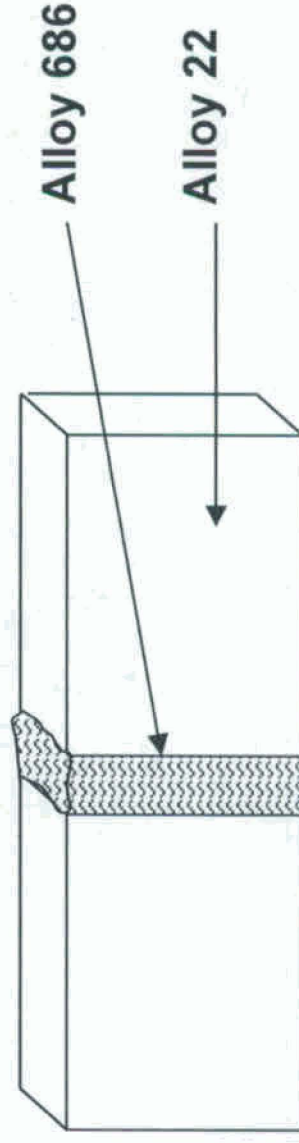


Weld longitudinally and 2 or 3 cylinders side by side + bottom lid



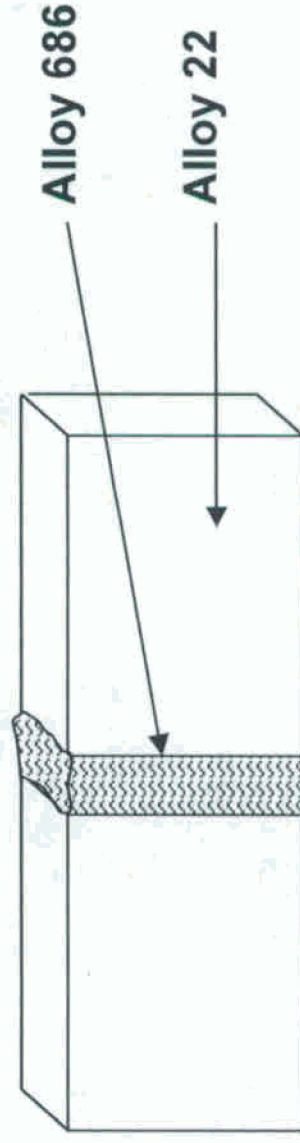
## Preparation of the Plates

- The fabrication of the plates and weld wire was performed at Allegheny Technologies Co. (Brackenridge, PA and Albany, OR)
- Seven (7) heats of plate material (forged-wrought) were welded with seven heats of wire
- Plate was Alloy 22 (N06022) – Heats A through G
- Wire was Alloy 686 (N06686) – Heats 1 through 7



## Preparation of the Plates

- 49 Different Welded Plates, A1 through G7
- Also Plates were studied in the Solution Annealed condition, 2075°F for 30 min. in air, rapid cooling
- At least 196 coupons were tested, one for each condition
- Tested external (First Layer) and internal (Second Layer) coupons





# Composition of Plate and Wire

- Plate of **Alloy 22**, ASTM B 575 = 20-22.5 Cr, 12.5-14.5 Mo, 2.5-3.5 W, 2-6 Fe
  - In 7 Heats, Cr was varied from 20.3 to 22.5
  - In 7 Heats, Mo was varied from 12.7 to 14.2
  - In 7 Heats, W was varied from 2.7 to 3.4
  - In 7 Heats, Fe was varied from 2.5 to 5.8
- Wire of **Alloy 686**
- ASTM B 575 = 19-23 Cr, 15-17 Mo, 3-4.4 W, 5 Fe\*
  - In 7 Heats, Cr was varied from 19.3 to 22.9
  - In 7 Heats, Mo was varied from 15.1 to 16.8
  - In 7 Heats, W was varied from 3.2 to 4.3
  - In 7 Heats, Fe was varied from 0 to 4.03



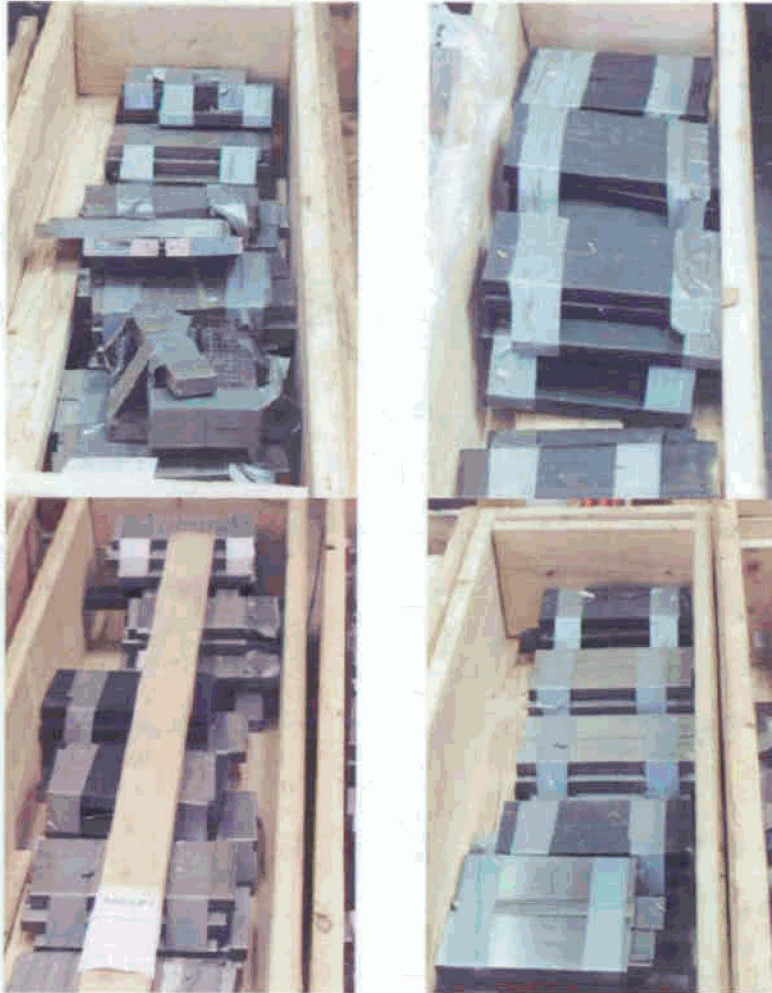
## Immersion Corrosion Tests

- 42 g/L Ferric Sulfate + 50% Sulfuric Acid Solution
- Highly Oxidizing and Acidic – ASTM G 28 A
- Boiling for 24 h
- Corrosion Rate Calculated by Mass (Weight) Loss

$$CR(mm / year) = 8.76 \times 10^4 \frac{\Delta W(g)}{A(cm^2) \cdot t(h) \cdot d(g \cdot cm^{-3})}$$



# Testing Procedure

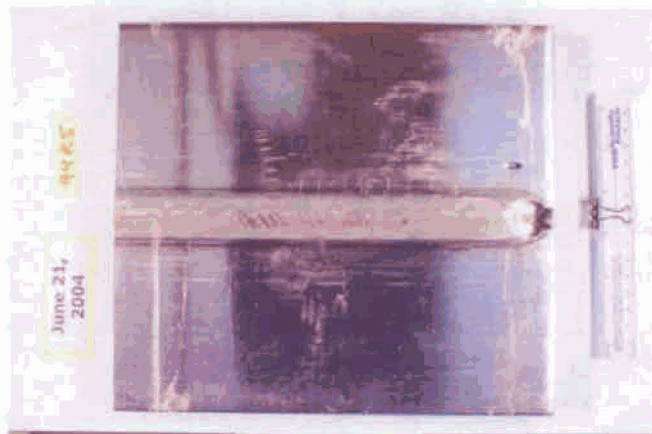
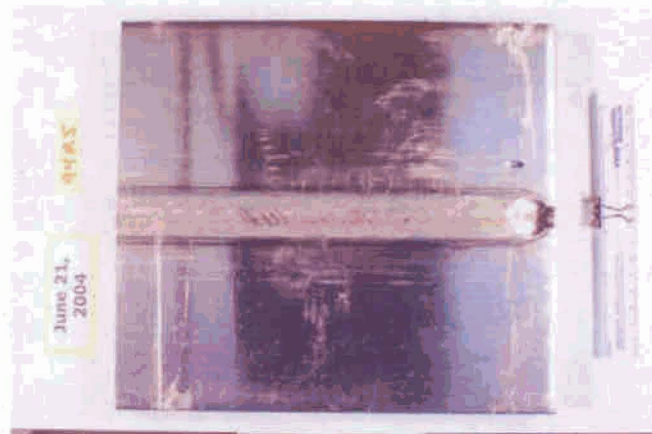


- Plates were sliced about 1-inch thick
- Then ends were cut keeping the weld seam and base metal at both ends of the weld
- Then sliced through the thickness to approximately  $\frac{1}{4}$ -inch thick
- Degreased, rinsed, dried, dimensioned, weighed
- Immersion Test

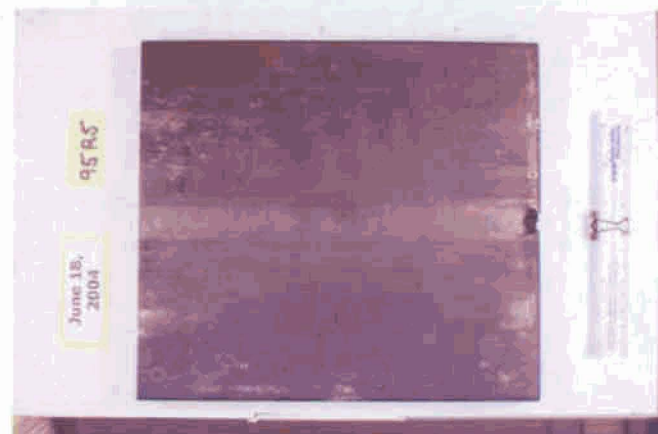
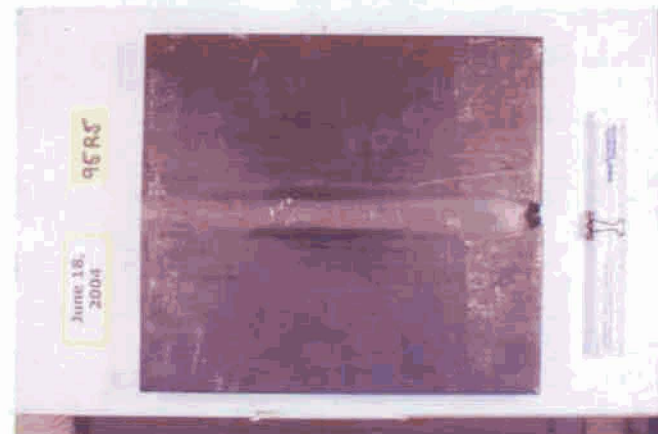




# As-Received D1 Welded Plates



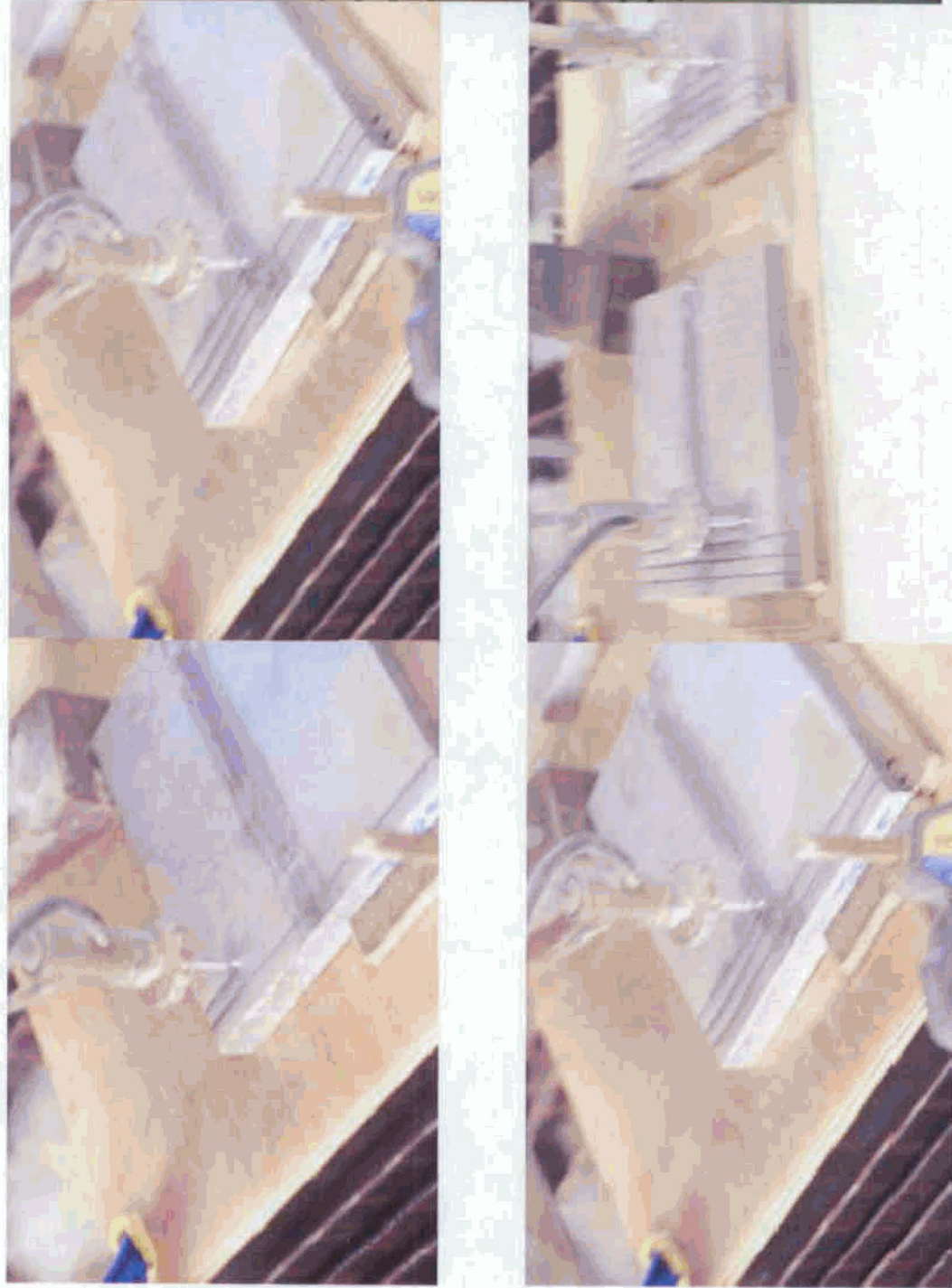
**Plates 94R5  
As-Welded (ASW)**



**Plates 95R5  
Solution Heat Treated (SHT)**

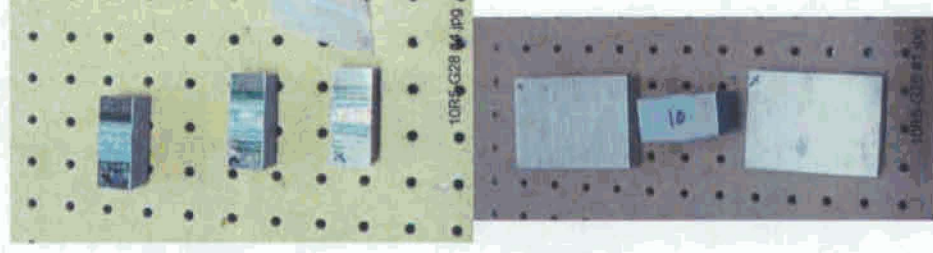
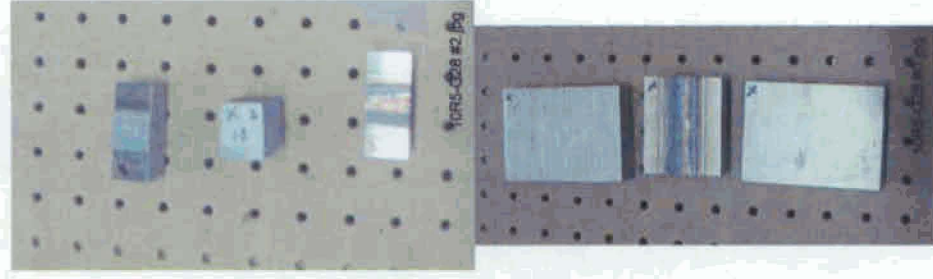
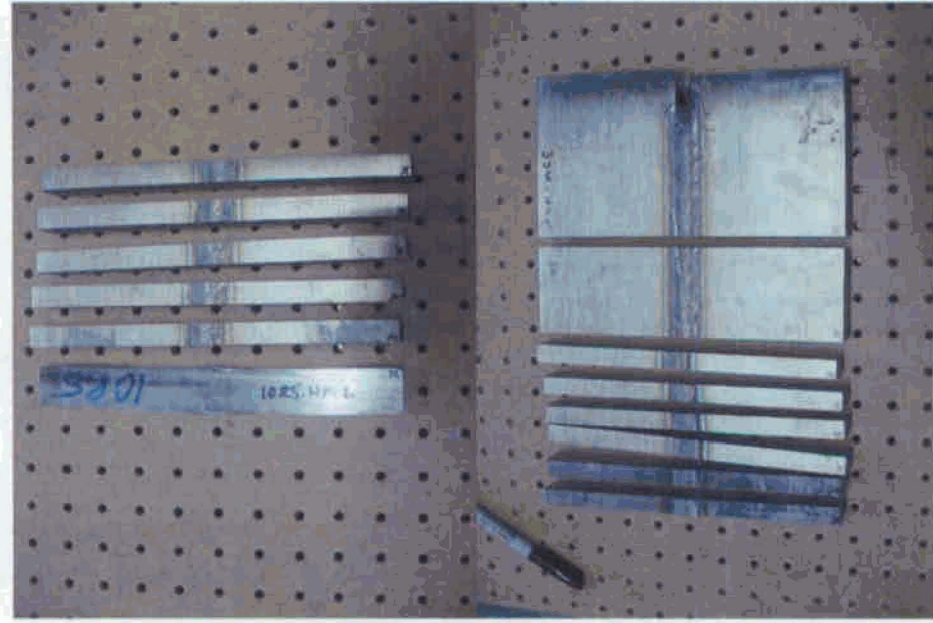


# Water-Jet Slicing



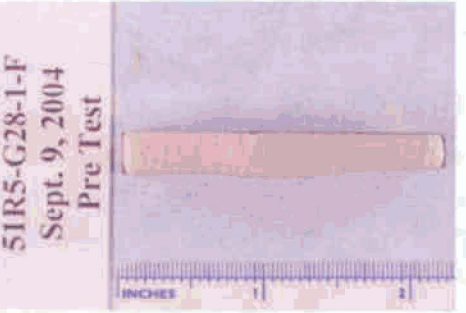
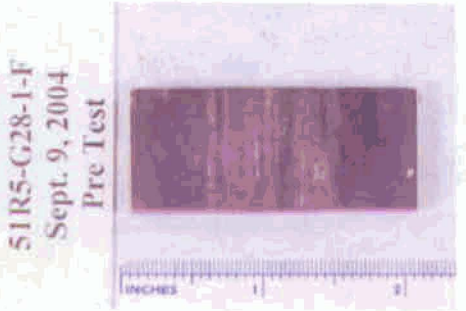
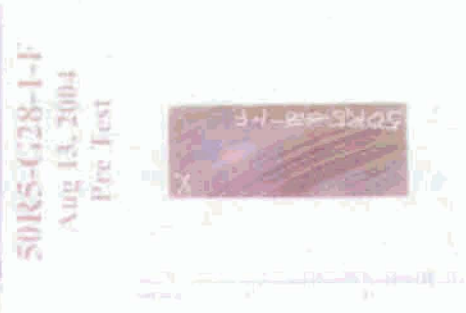
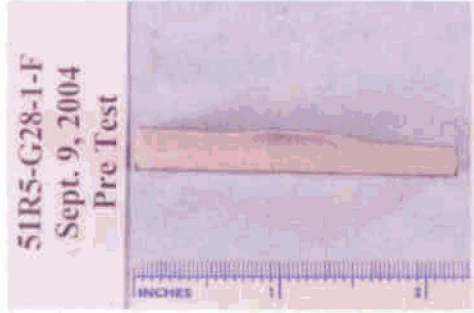
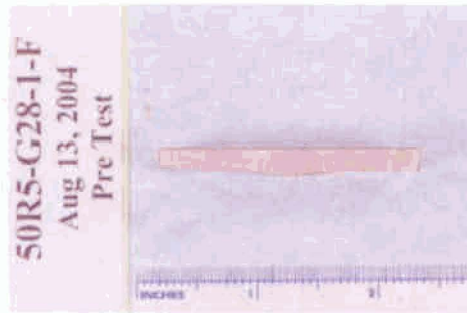


# Coupon from Plate 10R5

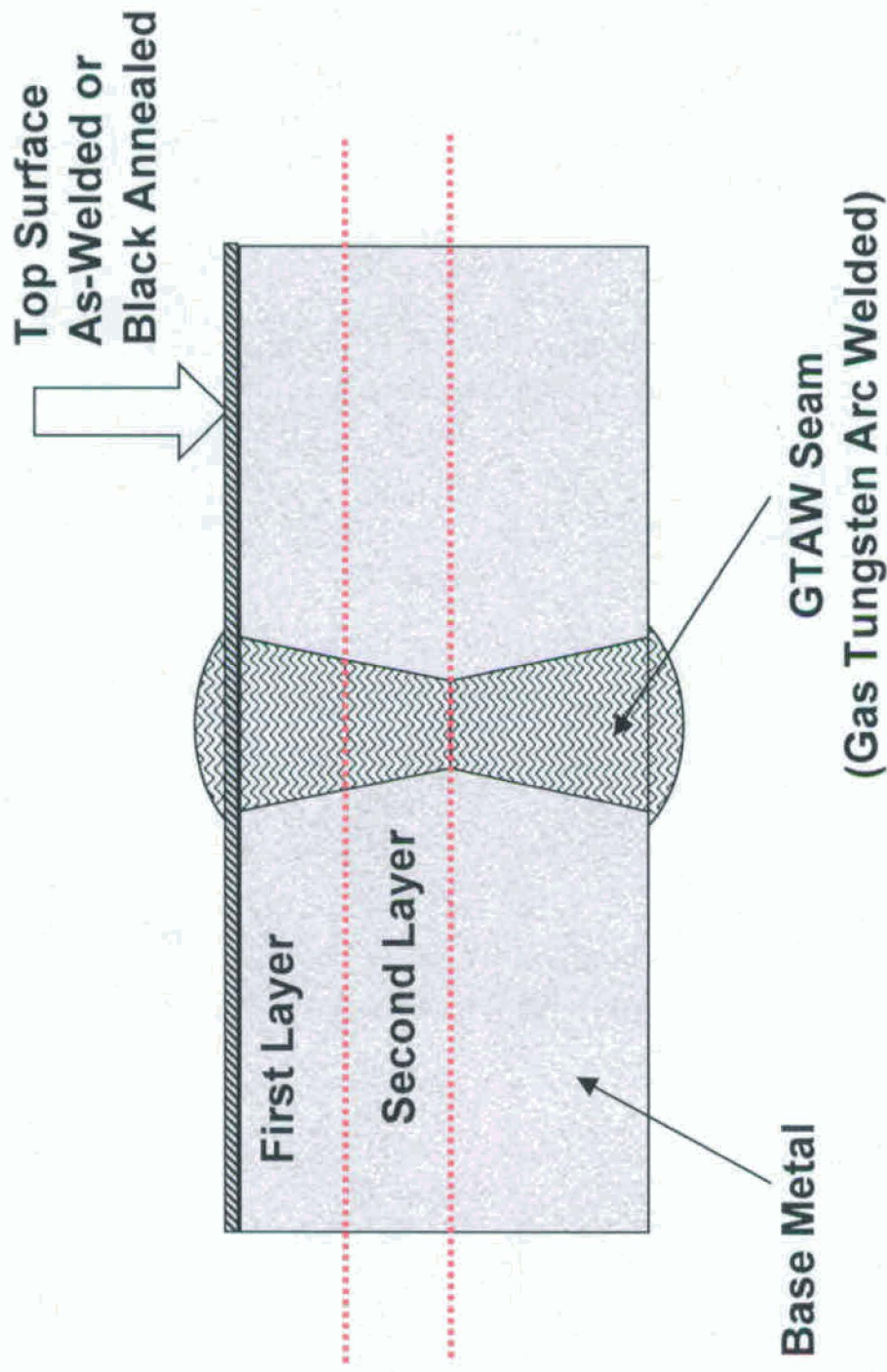




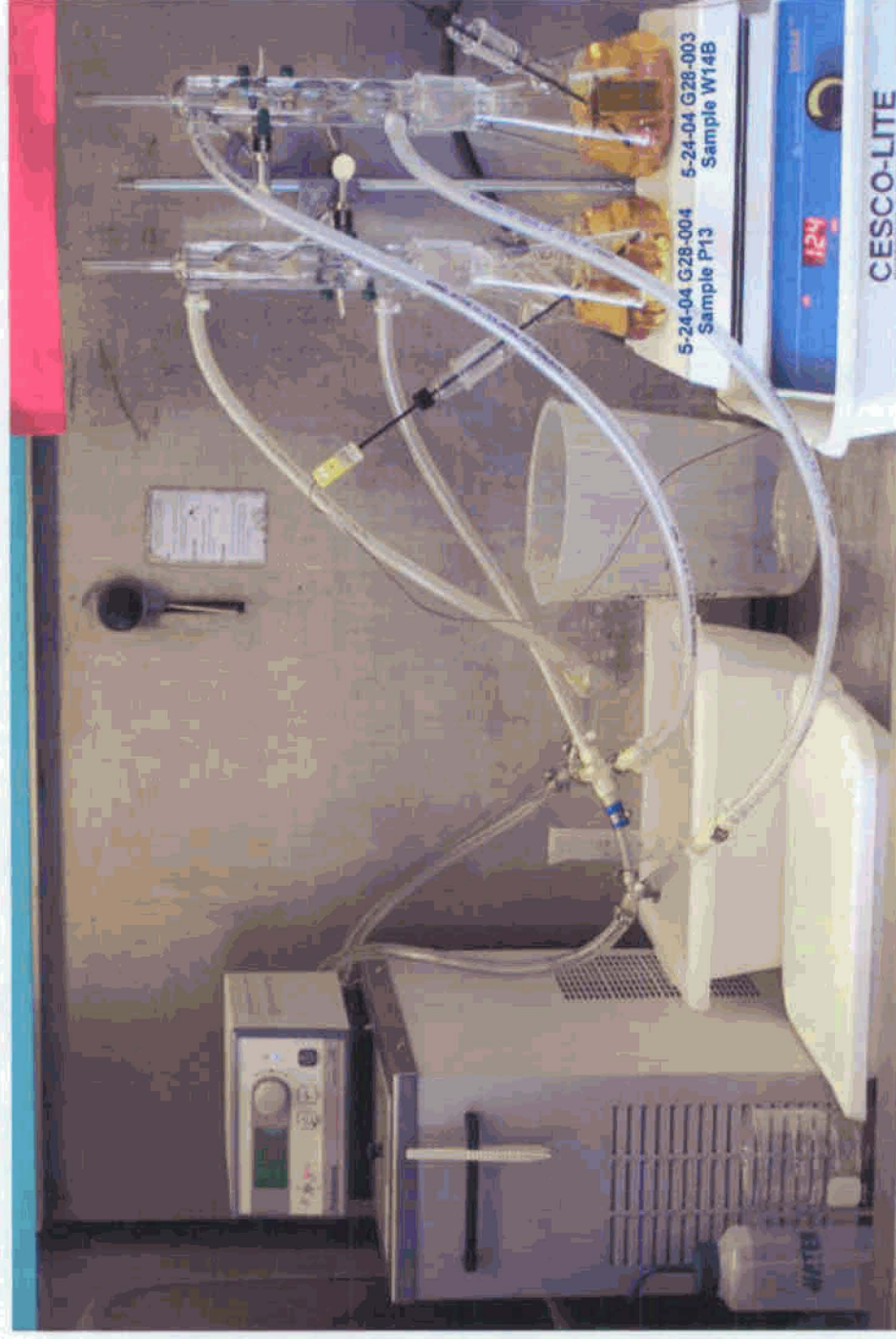
# Preparation of the Coupons



# First and Second Layer Coupons



# Immersion Corrosion Testing





# Corrosion Rate Results

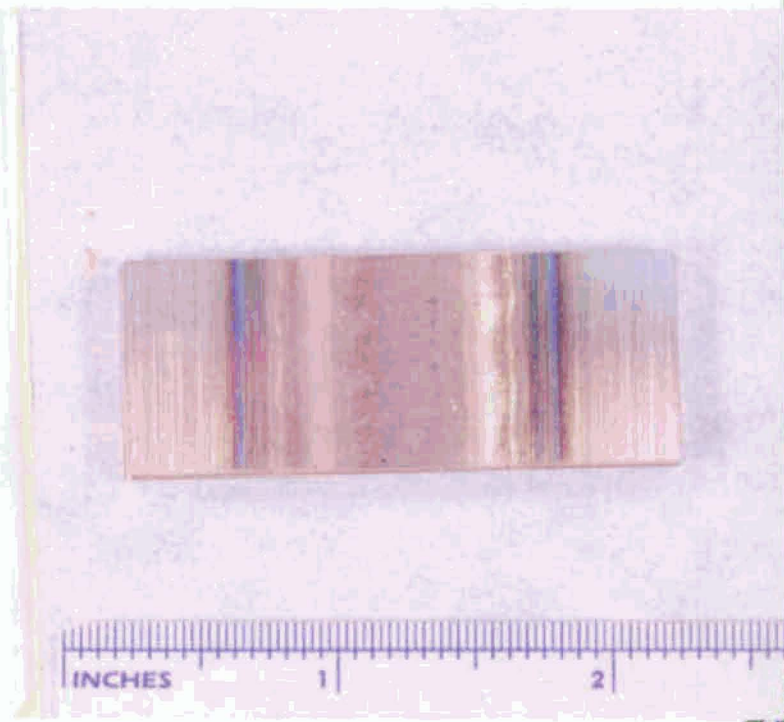


# ASW Coupons - Top Layer Before and After Corrosion Test

28R5-G28-1-F

Aug 17, 2004

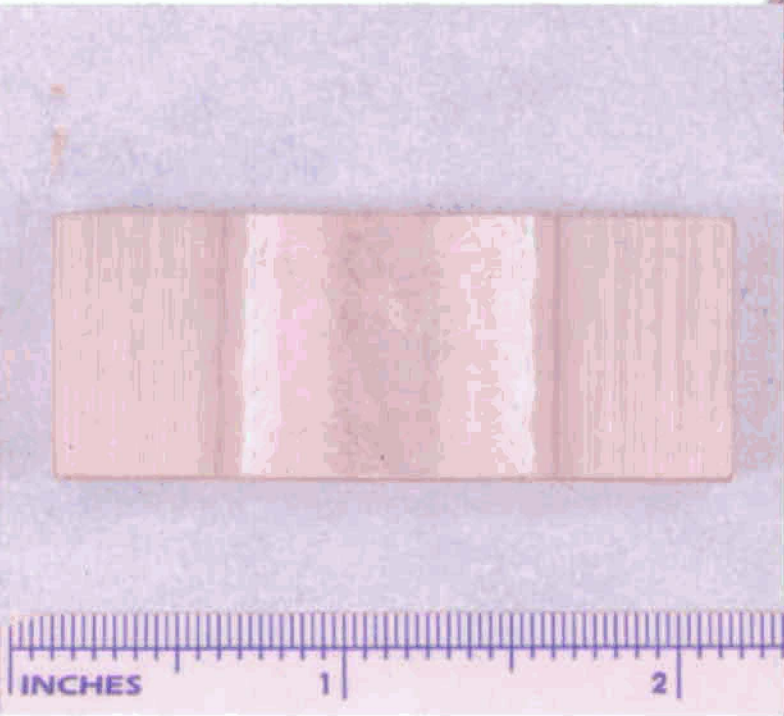
Pre Test



28R5-G28-1-F

Sept. 2, 2004

Post Test

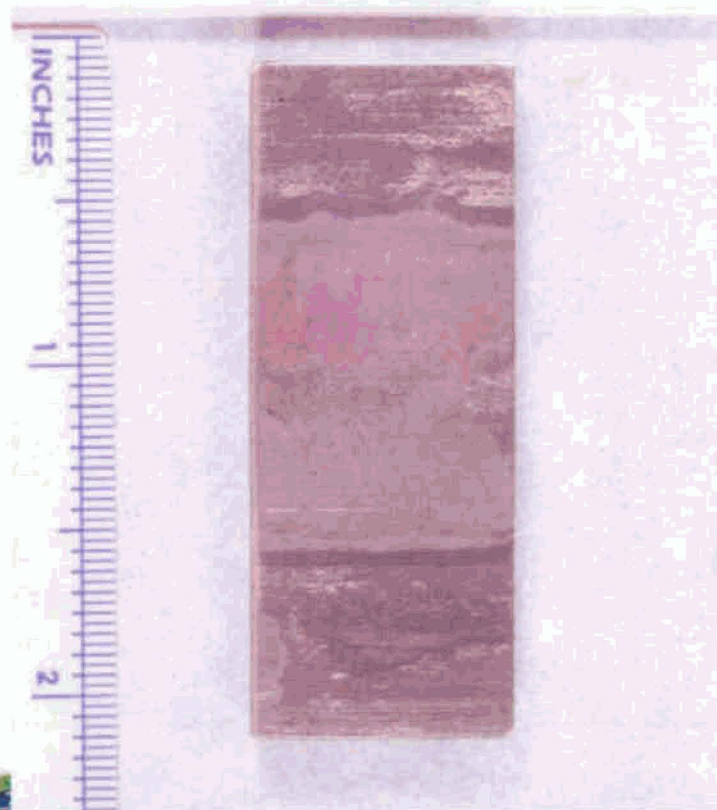


# ASW + SHT Coupons – Top Layer Before and After Corrosion Test

73R5-G28-1-F

Sept. 9, 2004

Pre Test



73R5-G28-1-F

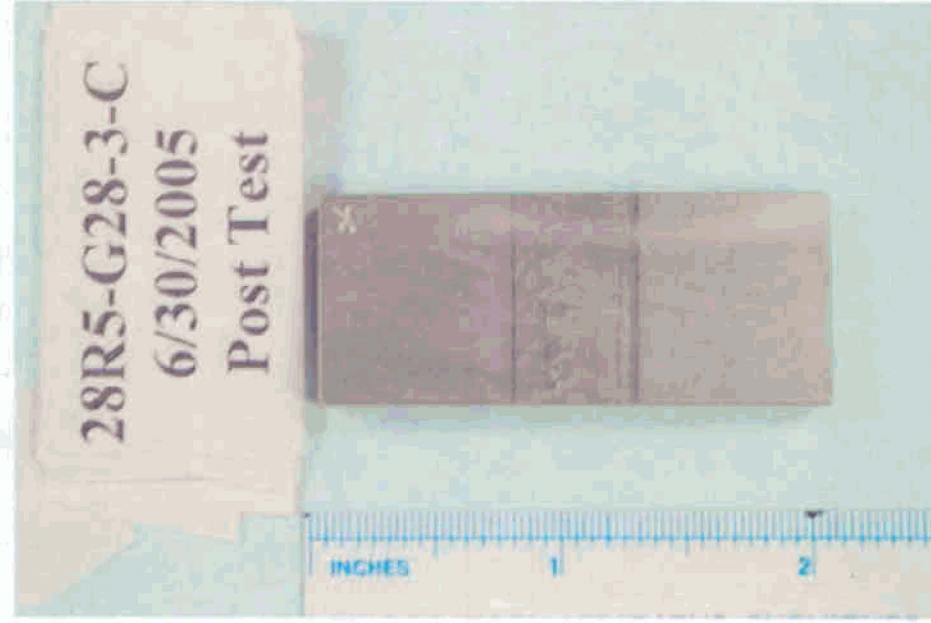
Sept. 16, 2004

Post Test

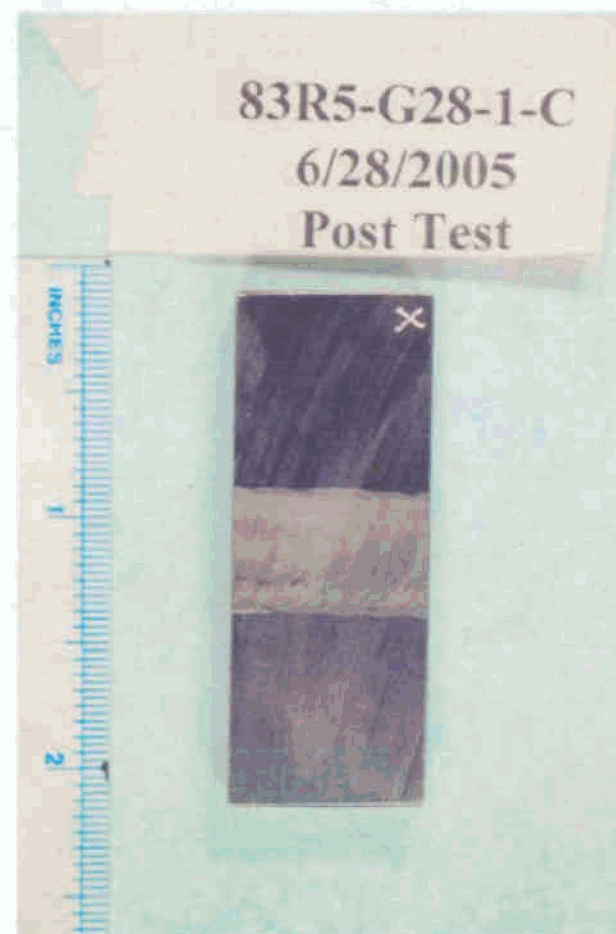




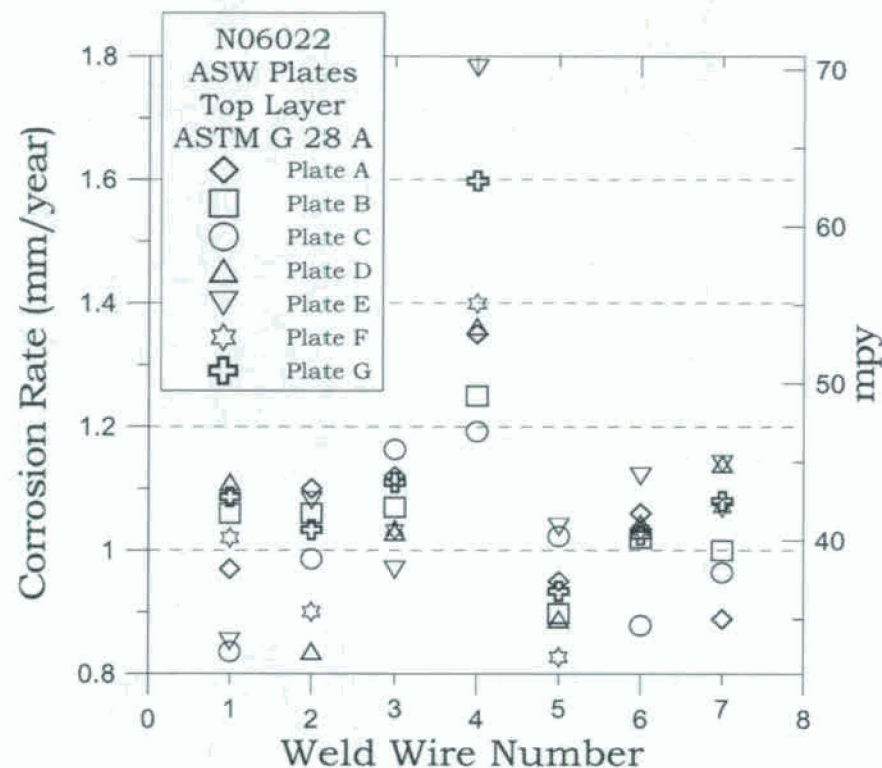
# ASW Coupons - Second Layer Before and After Corrosion Test



# ASW + SHT Coupons – Second Layer Before and After Corrosion Test



# Corrosion Rate of As-Welded Plates First Layer

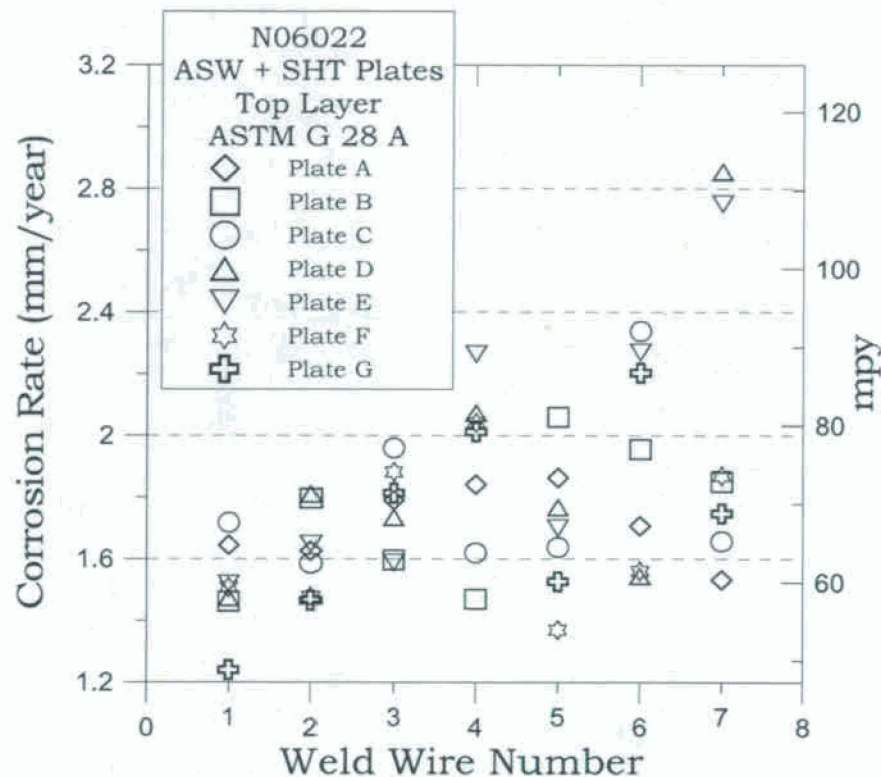


- Corrosion Rate for most of the coupons is between 0.8 and 1.2 mm/year (or 35 and 45 mpy)
- The standard value for Welded Alloy 22 plates with matching filler metal is 40 mpy
- Plates with corrosion rate above 80 mpy are rejected
- A few plates (E4, G4, F4) had higher corrosion rates





# Corrosion Rate of ASW + SHT Plates First Layer

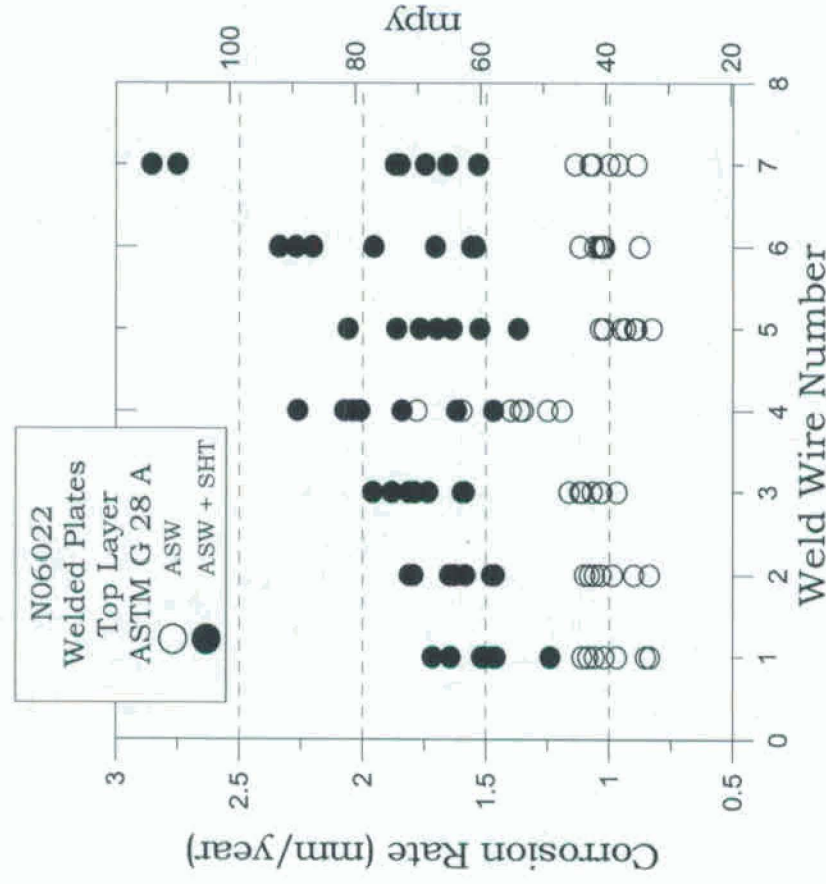


- Corrosion Rate for most of the coupons was higher than for the ASW plates
- Oxide scale loss may have contributed to the higher corrosion rates
- Corrosion Rate increased for the higher heats. Weld 4 heat gives higher than the trend corrosion rates

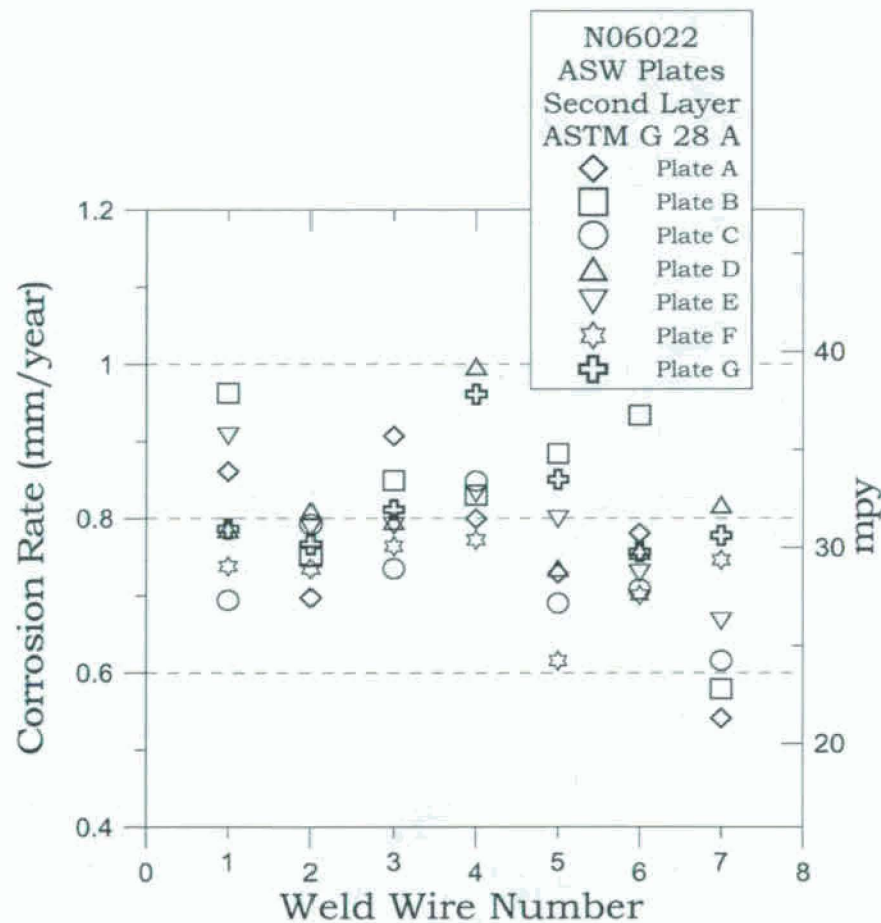


# Comparison of Corrosion Rate of ASW and ASW + SHT Plates - First Layer

- Corrosion Rate for ASW + SHT coupons was higher than for the ASW plates
- Well documented in the literature on the influence of surface finish of the coupons on the corrosion rate in ASTM G 28 A



# Corrosion Rate of As-Welded Plates Second Layer



- Corrosion Rate for most of the coupons is between 0.6 and 1.0 mm/year (or 25 and 40 mpy)
- The standard value for Welded Alloy 22 plates with matching filler metal is 40 mpy
- No much influence of the type of base heat or weld wire

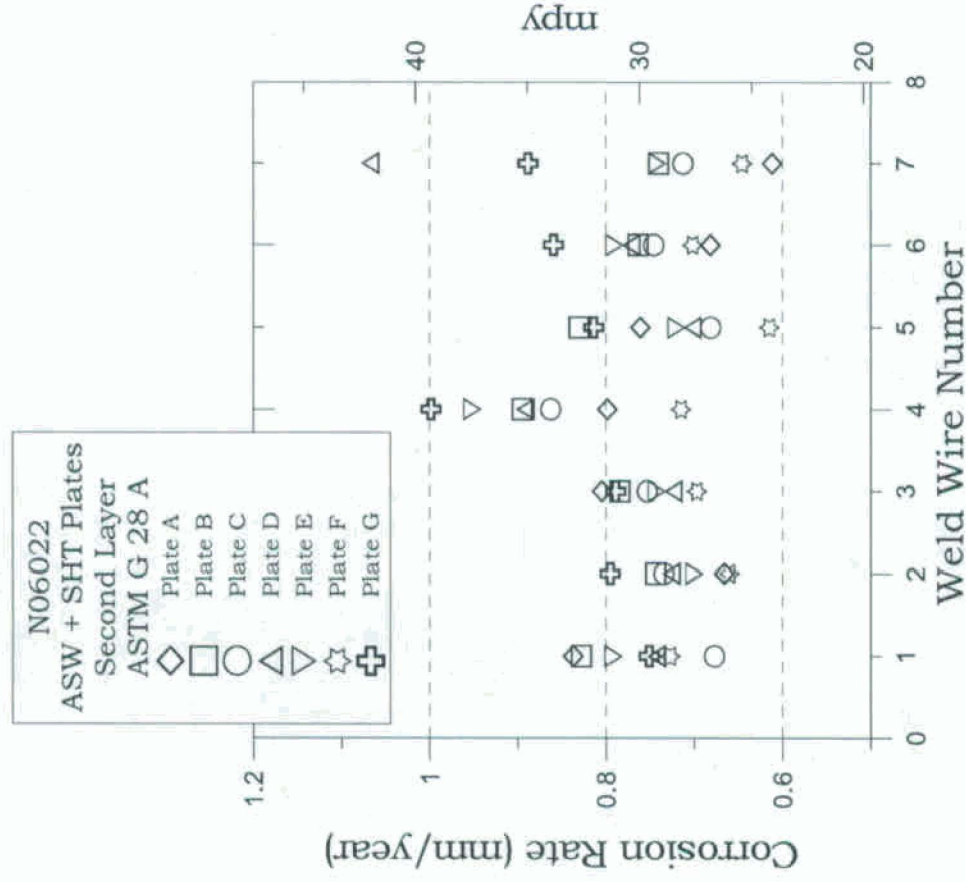




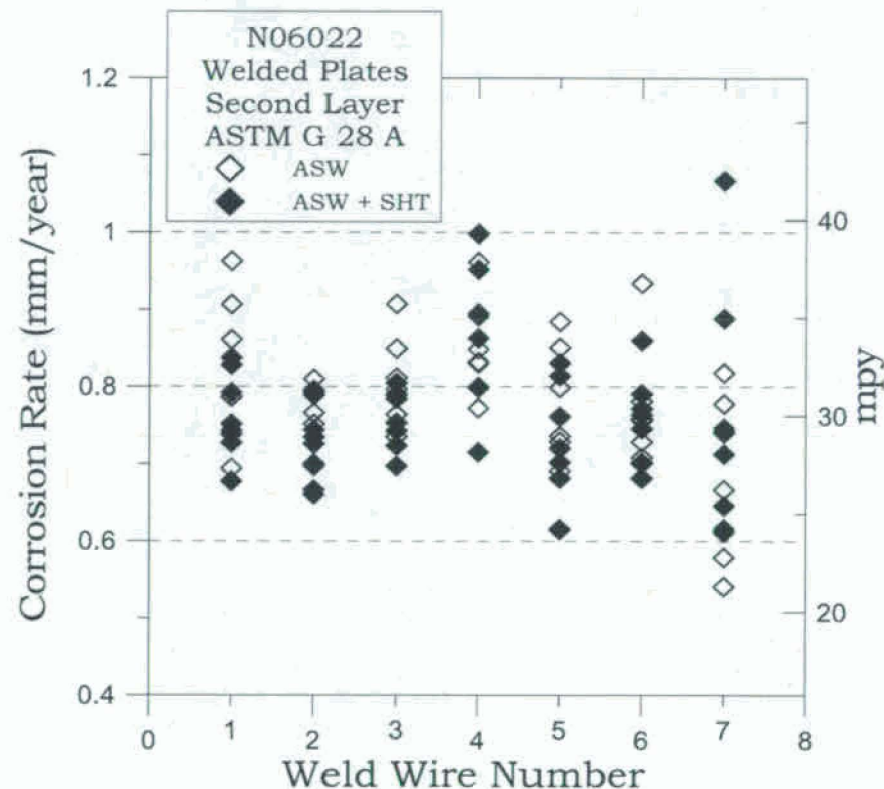
# Corrosion Rate of ASW + SHT Plates

## Second Layer

- Corrosion Rates mostly between 0.6 and 0.8 mm/year
- Weld wire 4 and plate G gave higher corrosion rates



# Comparison of Corrosion Rate of ASW and ASW + SHT Plates - Second Layer

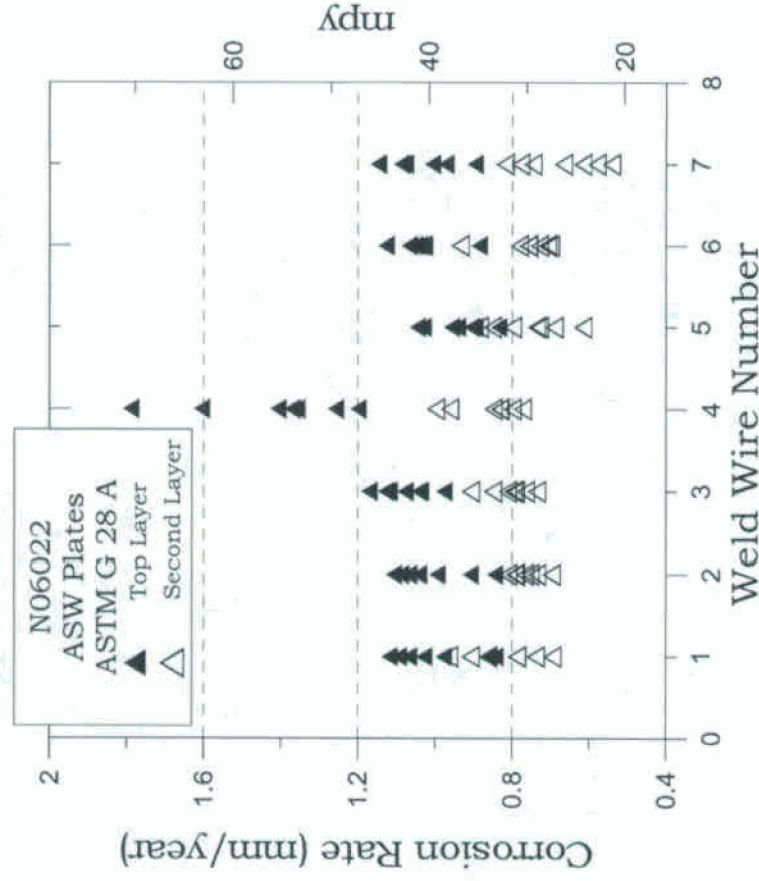


- Corrosion Rate for ASW coupons was higher than for the ASW + SHT coupons
- It is likely that the SHT reduced the amount of intergranular corrosion in the heat affected zone – SEM studies may be needed to quantify this



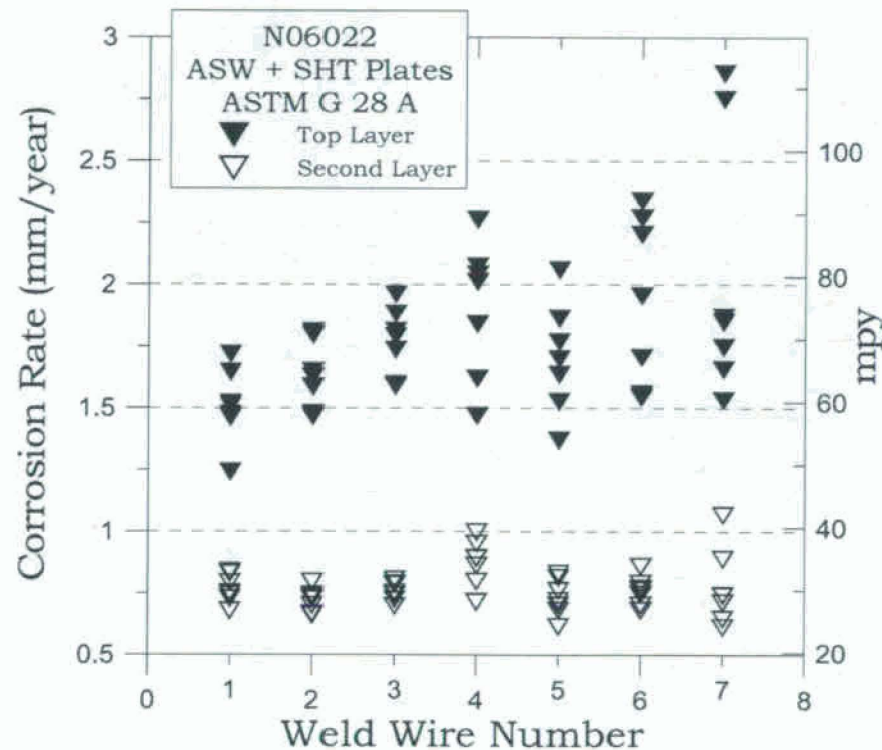
# Corrosion Rate of As-Welded Plates First and Second Layer Comparison

- Corrosion Rate of top layer of ASW coupons higher than of the second layer
- For ASW plates effect less than a factor of 2
- Influence of surface finish well documented in the literature





# Corrosion Rate of ASW + SHT Plates First and Second Layer Comparison



- Corrosion Rate of top layer of ASW + SHT coupons higher than of the second layer
- For ASW plates effect by a factor larger than 2
- Influence of surface finish well documented in the literature



# Conclusion Heat-to-Heat Variability

- Corrosion rates of as-welded (ASW) Alloy 22 plates with Alloy 686 wires are in the order of known values (approximately 40 mpy)
- The top layer had higher corrosion rates than the second layer, especially for the SHT coupons
- For the second layer, the SHT condition gave slightly lower corrosion rates than for the ASW condition
- In the range of accepted chemistry per ASTM standards, the corrosion rate from one heat will be undistinguishable from another heat (in the margin of error)



# **Part 2**

## **Effect of Black Annealing Scale on the General and Localized Corrosion Electrochemical Tests**





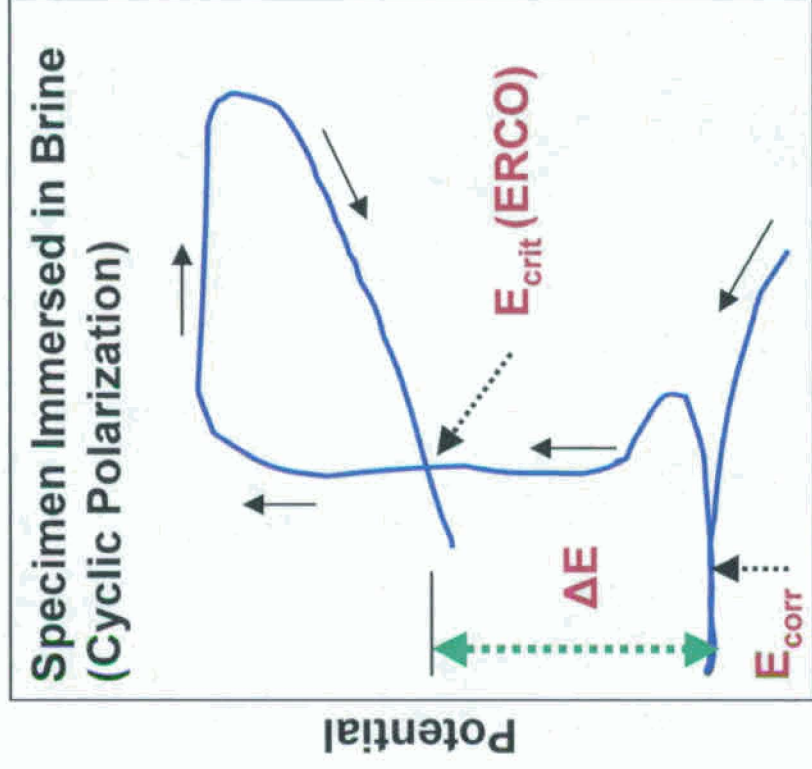
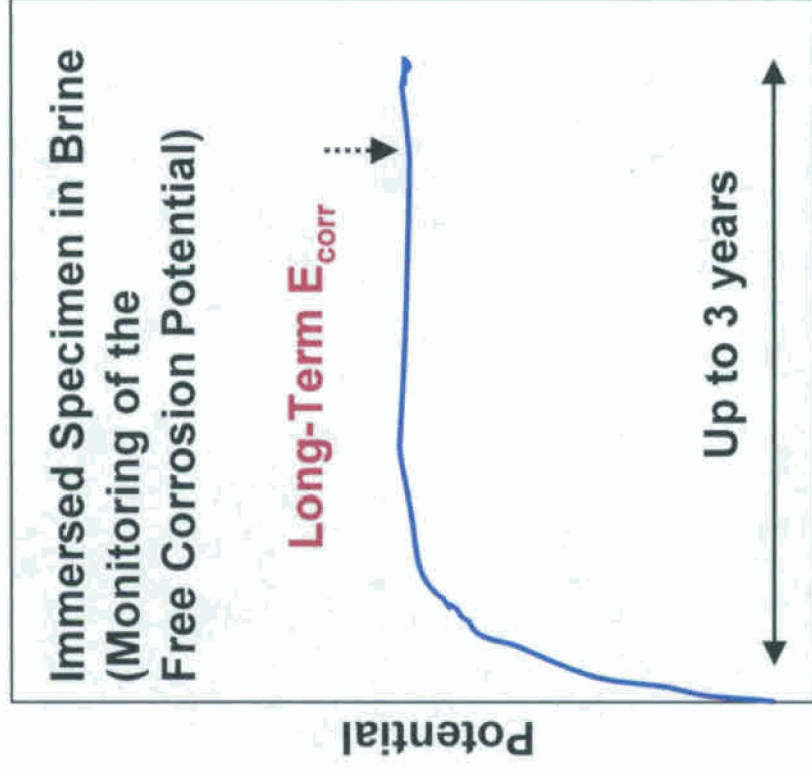
## Model for Localized Corrosion

- Pitting Corrosion does not form easily in Alloy 22. Crevice Corrosion May be Possible
- Crevice Corrosion occurs only above a Critical Potential ( $E_{crit}$ ) such as the Repassivation Potential
- If the steady state Corrosion Potential ( $E_{corr}$ ) in the environment of choice never reaches  $E_{crit}$ , crevice corrosion will not occur
- If  $\Delta E = E_{crit} - E_{corr} > 0$

**Crevice Corrosion will not occur**



# $E_{\text{corr}}$ and $E_{\text{crit}}$ Are measured Independently



# **Current Results**

## **Monitoring of $E_{\text{corr}}$ and $R_p$ vs. Time**

### **Determine Influence of Immersion Time on the Repassivation Potential**





## Monitoring $E_{\text{corr}}$ and Polarization Resistance

- Immerse Artificially Creviced Specimens in Brines and Monitor  $E_{\text{corr}}$  as a Function of Time
- Also Monitor the Polarization Resistance ( $R_p$ ) (or Corrosion Rate) using ASTM G 59 and G 102

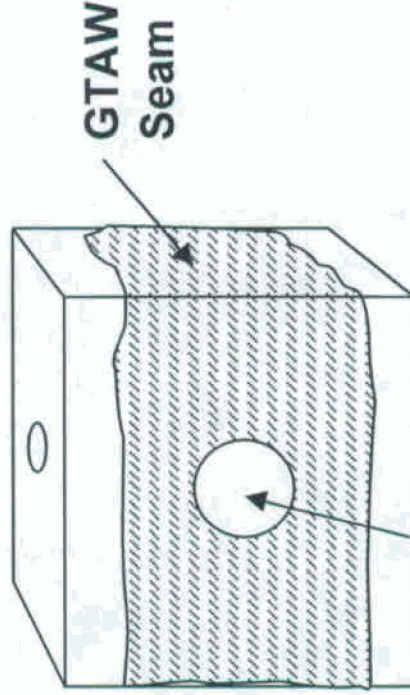
$$i_{\text{corr}} = \frac{1}{R_p} \times \frac{b_a \cdot b_c}{2.303(b_a + b_c)}$$

$$CR(\mu\text{m} / \text{yr}) = k \frac{i_{\text{corr}}}{\rho} EW$$

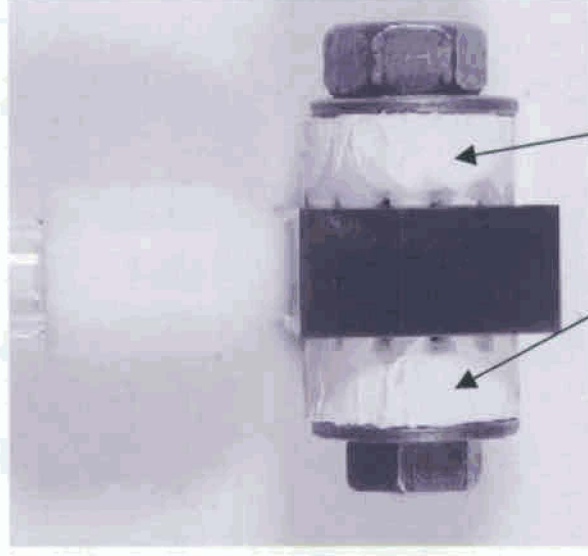


# Prism Creviced Assembly (PCA)

## WELDED PCA SPECIMEN



Hole for  
Crevice Formers



Two Crevice Formers  
With 12 Teeth Each



Crevice Former  
ASTM G 48 or G 78



# Long Term $E_{\text{corr}}$ Creviced Specimens

- Four (4) As-Welded (ASW) Polished PCA and
- Four (4) ASW + Black Annealed PCA Specimens or ASW + SHT (1120°C for 20 min + Water Quenched)





# Monitoring Long-Term Corrosion Potential ( $E_{\text{corr}}$ )

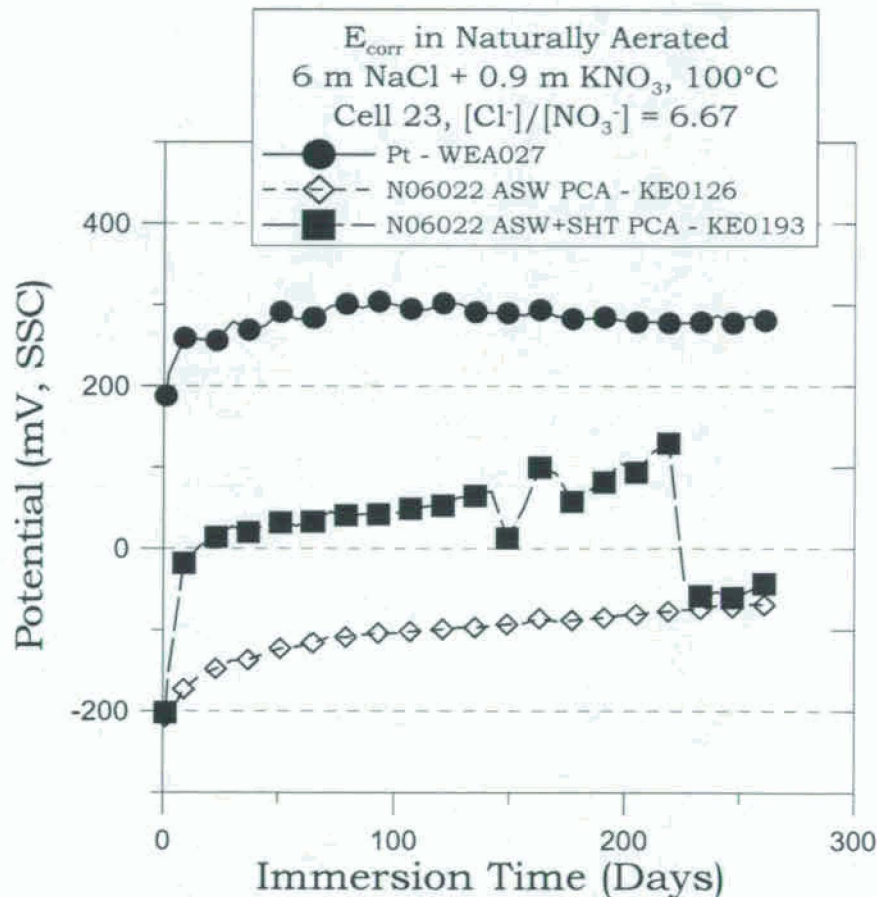


# Long-Term Monitoring of $E_{\text{corr}}$

- $E_{\text{corr}}$  depends on
  - Environment (electrolyte composition, pH, Temperature) and
  - Surface condition of the alloy (which also depends on the metallurgical conditions of the metal)
- The value of  $E_{\text{corr}}$  provides a measure about the expectancy (or not) of localized corrosion
- Test Matrix
  - $T = 100^{\circ}\text{C}$
  - Six Cells, Three  $[\text{Cl}^-]$  and Two  $[\text{NO}_3^-]/[\text{Cl}^-] = 0.15$  and  $0.05$
  - $1\text{ m NaCl} + 0.15\text{ m KNO}_3$  and  $1\text{ m NaCl} + 0.05\text{ m KNO}_3$
  - $3.5\text{ m NaCl} + 0.525\text{ m KNO}_3$  and  $3.5\text{ m NaCl} + 0.175\text{ m KNO}_3$
  - $6\text{ m NaCl} + 0.9\text{ m KNO}_3$  and  $6\text{ m NaCl} + 0.3\text{ m KNO}_3$



# $E_{\text{corr}}$ of Creviced Alloy 22



- 6 m NaCl + 0.9 m KNO<sub>3</sub>
- [Cl<sup>-</sup>]/[NO<sub>3</sub><sup>-</sup>] = 6.67
- [NO<sub>3</sub><sup>-</sup>]/[Cl<sup>-</sup>] = 0.15
- T = 100°C
- Individual Specimens with black annealed film have an  $E_{\text{corr}}$  slightly more positive than the freshly polished specimens
- Variations in the  $E_{\text{corr}}$  of the creviced specimens may indicate onset of crevice corrosion



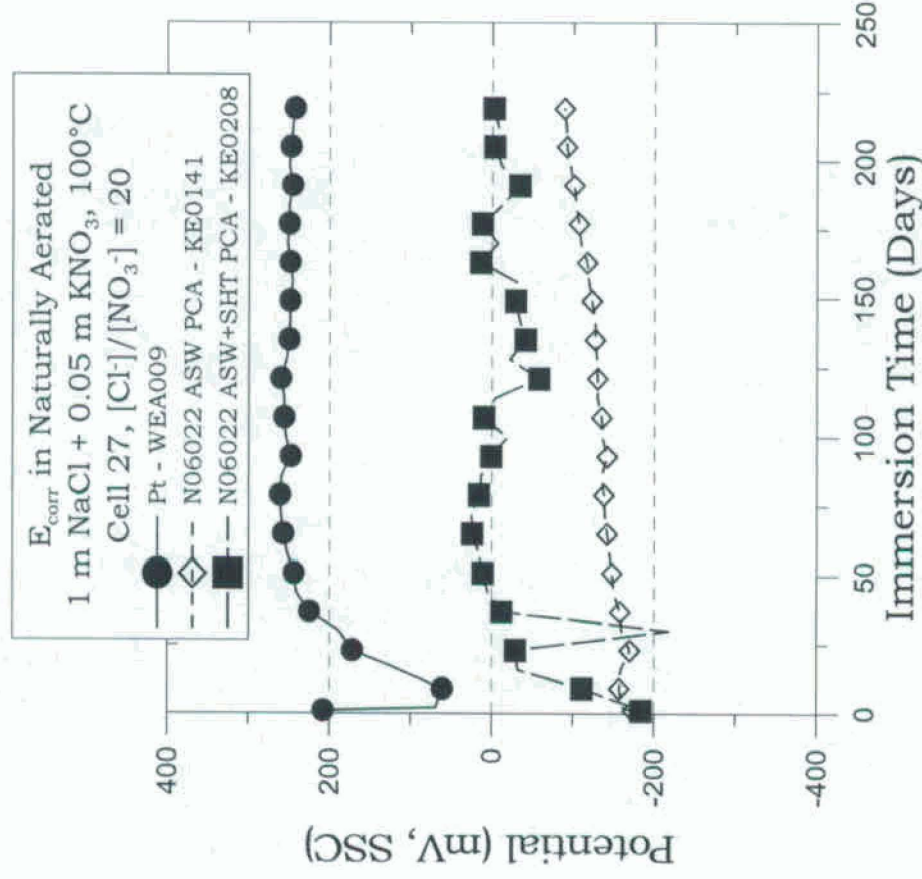


# $E_{\text{corr}}$ of Creviced Alloy 22

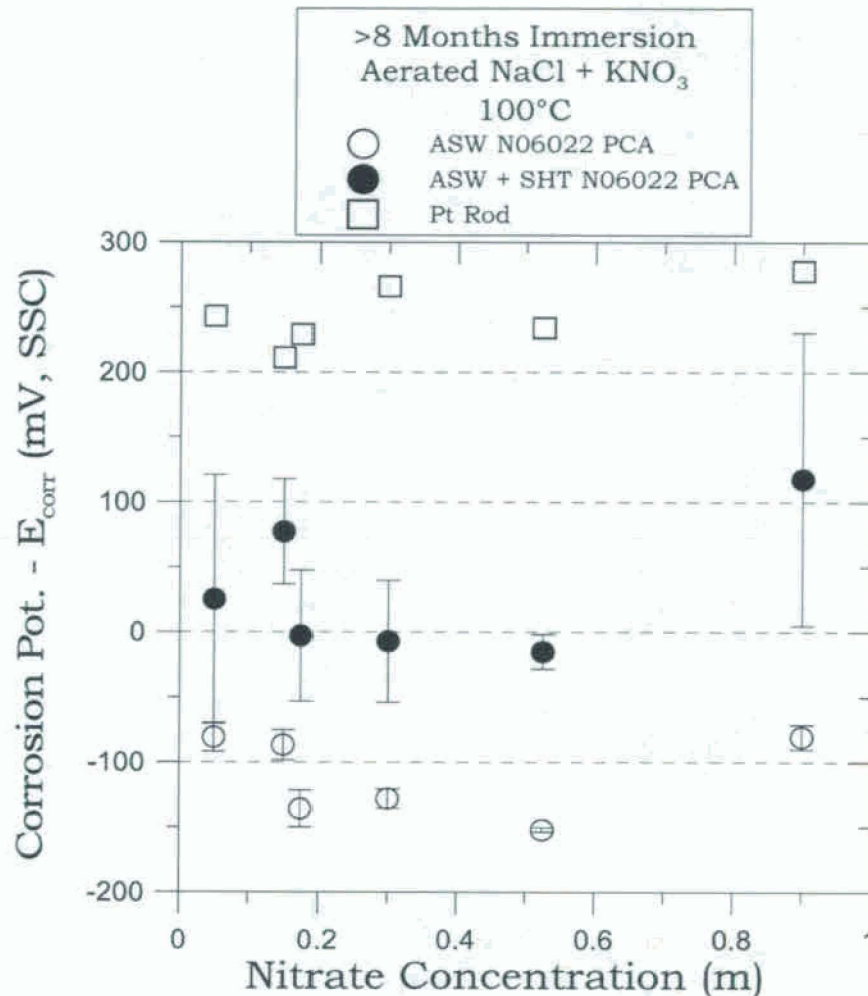
- 1 m NaCl + 0.05 m KNO<sub>3</sub>
- $[\text{Cl}^-]/[\text{NO}_3^-] = 20$
- $[\text{NO}_3^-]/[\text{Cl}^-] = 0.05$
- $T = 100^\circ\text{C}$

• Individual Specimens with black annealed film have an  $E_{\text{corr}}$  slightly more positive than the freshly polished specimens

• Variations in the  $E_{\text{corr}}$  of the creviced specimens may indicate onset of crevice corrosion



# $E_{\text{corr}}$ of Creviced Alloy 22

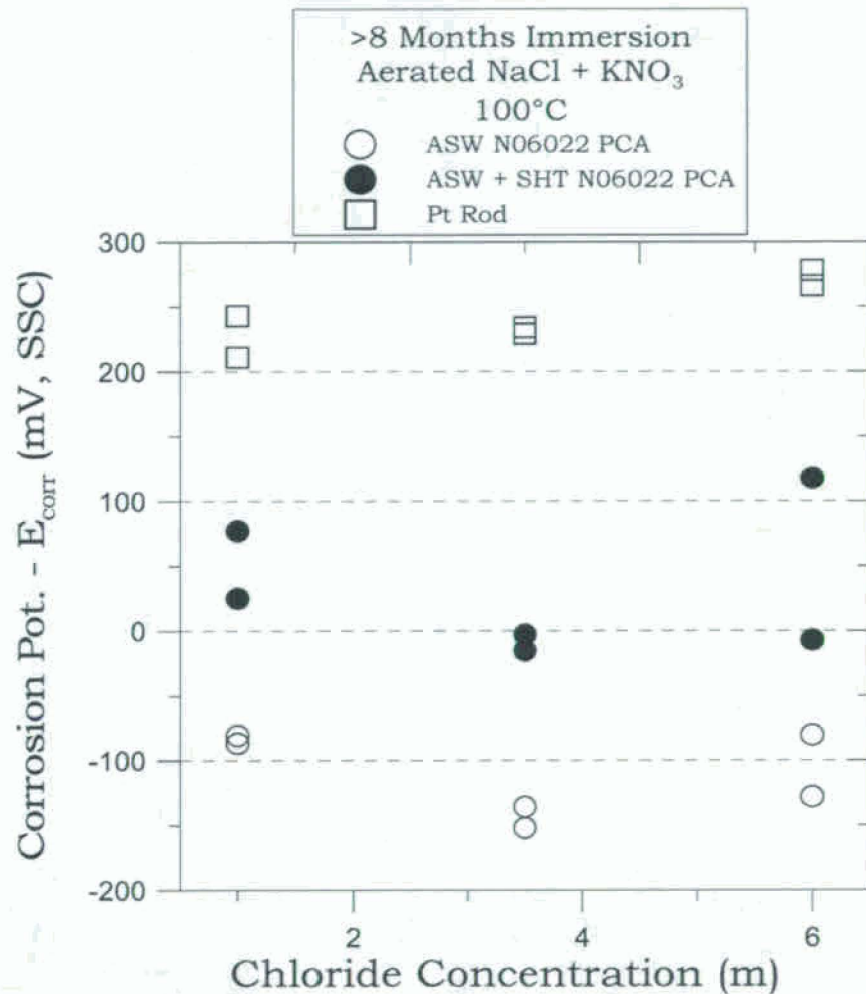


- Average of Four Values of  $E_{\text{corr}}$  at the end of the test as a function of Nitrate Concentration for the Six Cells
- $x \text{ m NaCl} + y \text{ m KNO}_3$
- $T = 100^\circ\text{C}$
- Specimens with black annealed film have less reproducible  $E_{\text{corr}}$  than the freshly polished specimens
- For the tested conditions ( $0.05 < \text{NO}_3/\text{Cl} < 0.15$ ),  $E_{\text{corr}}$  did not depend strongly on total [NO<sub>3</sub>]





# $E_{\text{corr}}$ of Creviced Alloy 22

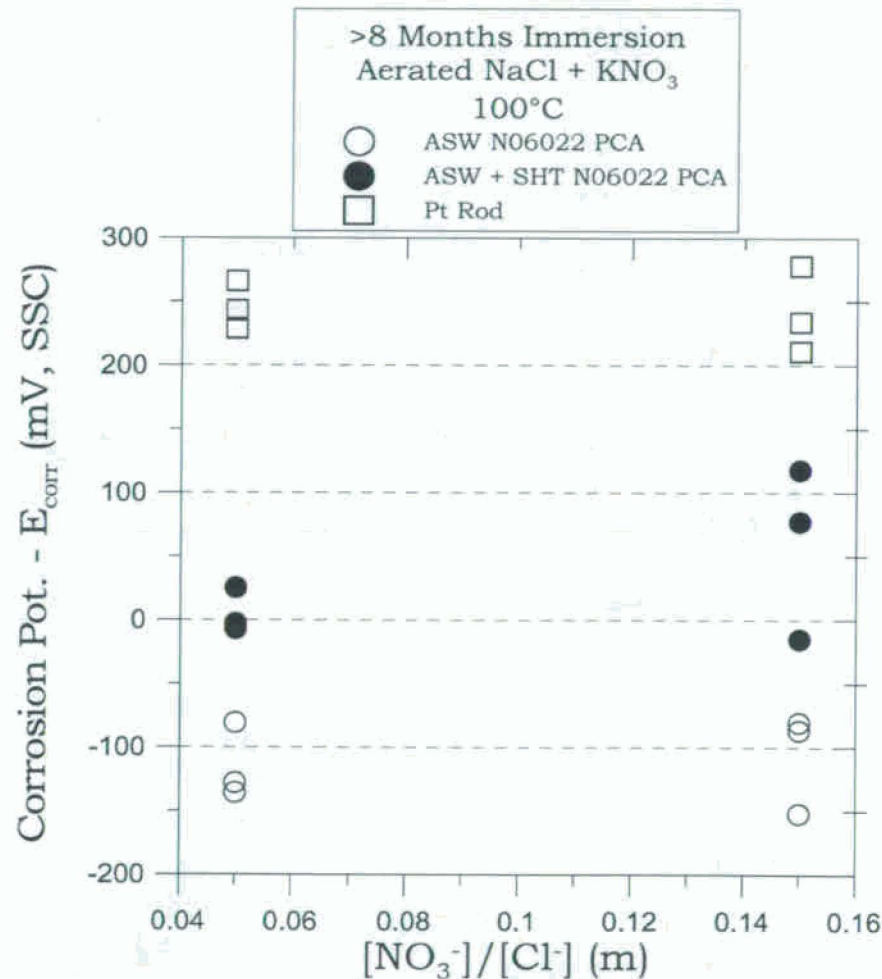


- Average of Four Values of  $E_{\text{corr}}$  as a function of Chloride Concentration for the Six Cells
- x m NaCl + y m KNO<sub>3</sub>
- T = 100°C
- Specimens with black annealed film have less reproducible  $E_{\text{corr}}$  than the freshly polished specimens
- For the tested conditions  $E_{\text{corr}}$  did not depend strongly on total [Cl]





# $E_{\text{corr}}$ of Creviced Alloy 22



- Average of Four Values of  $E_{\text{corr}}$  as a function of Chloride over Nitrate Ratio for the Six Cells
- $x \text{ m NaCl} + y \text{ m KNO}_3$
- $T = 100^\circ\text{C}$
- Specimens with black annealed film have less reproducible  $E_{\text{corr}}$  than the freshly polished specimens
- For the tested conditions  $E_{\text{corr}}$  did not depend strongly on nitrate over chloride ratio

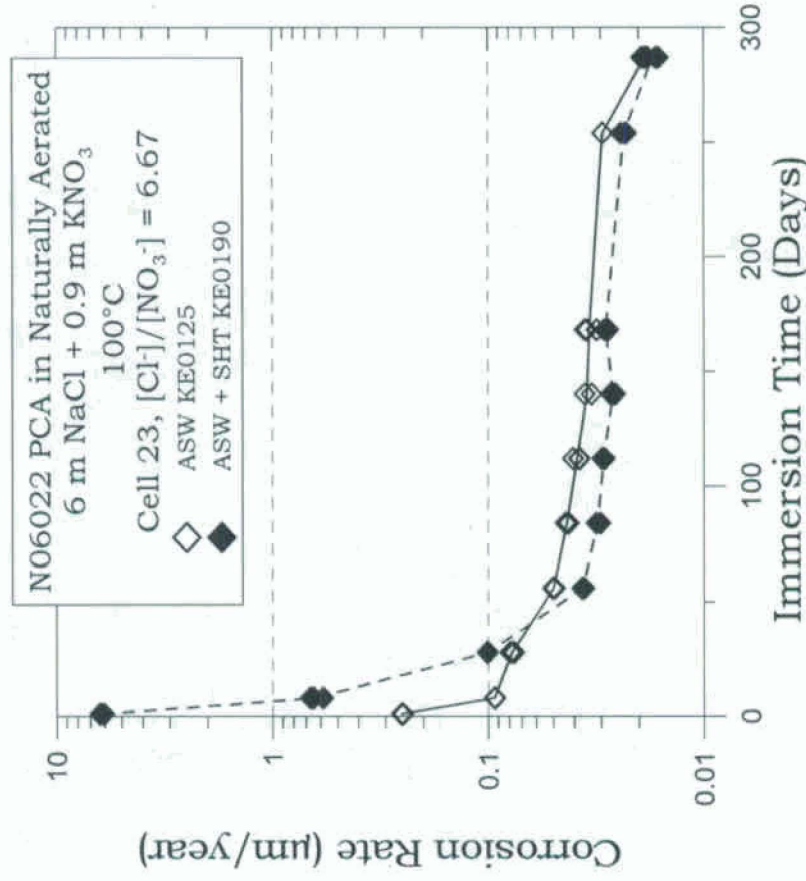


# Monitoring Polarization Resistance and Corrosion Rate



# Corrosion Rate of Creviced Alloy 22

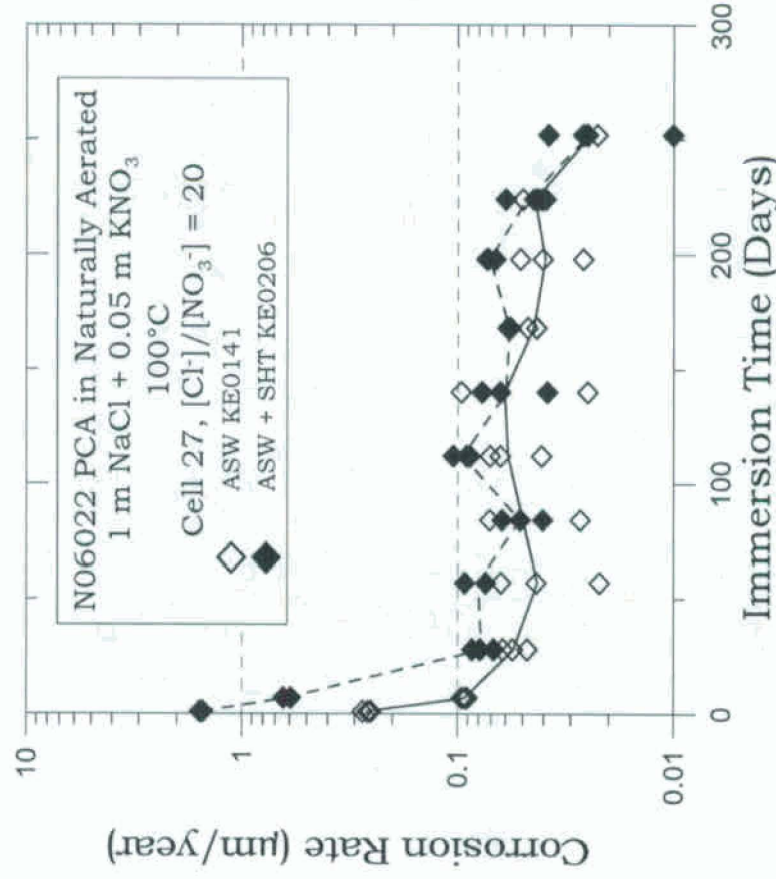
- Individual Values of Corrosion Rate (CR) as a function of Immersion Time
- 6 m NaCl + 0.9 m KNO<sub>3</sub>
- T = 100°C
- Specimens with black annealed film Initially had higher CR than the freshly polished specimens
- CR for both specimens decreased as immersion time increased
- After 8 mo. Immersion it was less than 30 nm/year



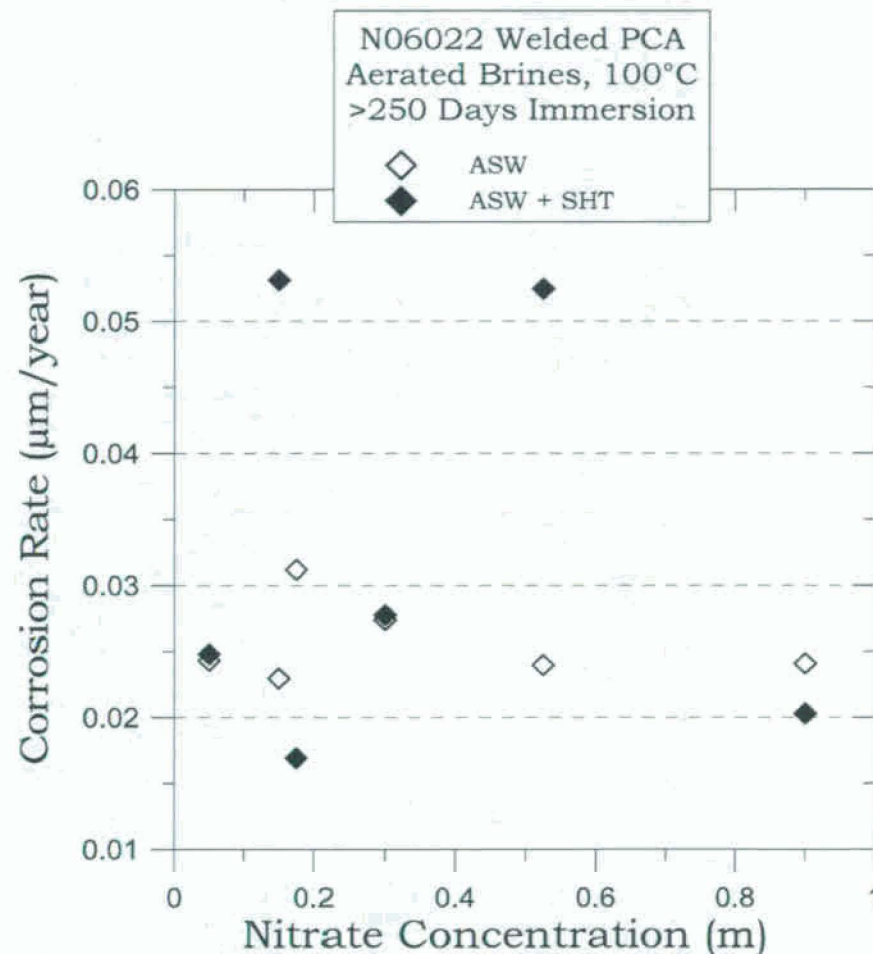


# Corrosion Rate of Creviced Alloy 22

- Individual Values of Corrosion Rate (CR) as a function of Immersion Time
- **1 m NaCl + 0.05 m KNO<sub>3</sub>**
- **T = 100°C**
- **Specimens with black annealed film Initially had higher CR than the freshly polished specimens**
- **CR for approximately stable at 50 nm/year**



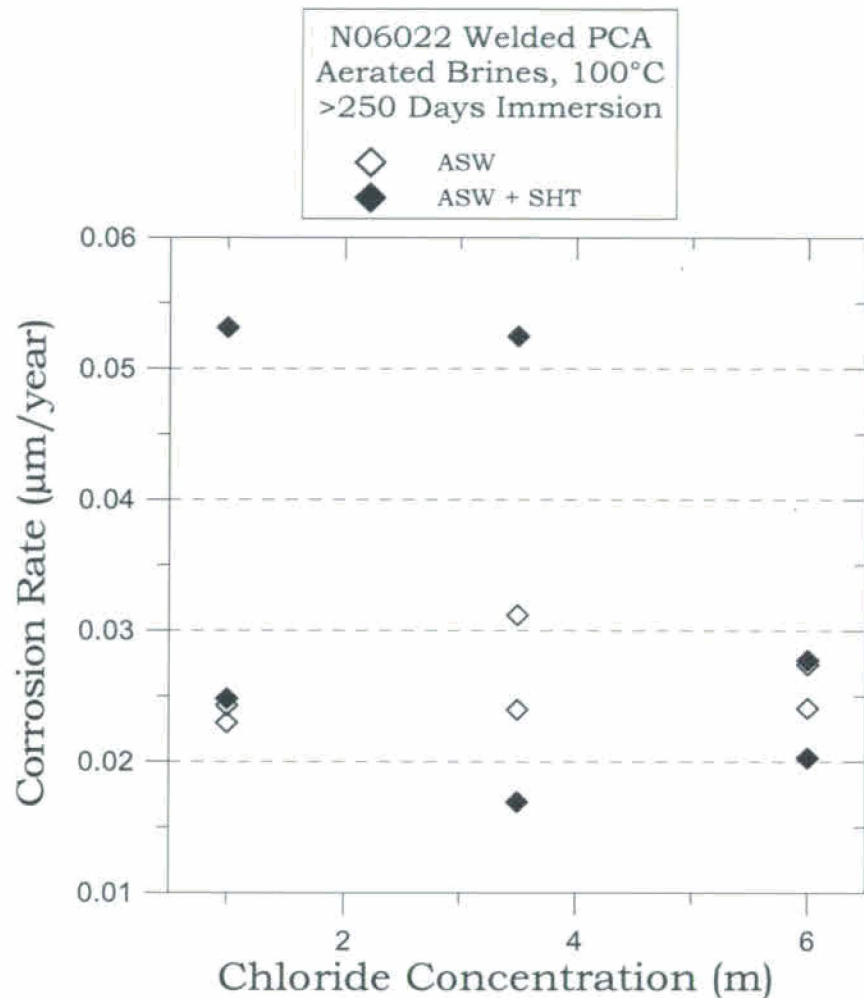
# Corrosion Rate of Creviced Alloy 22



- Average Values of Corrosion Rate (CR) as a function of Nitrate Concentration for Times >250 Days
- $x \text{ m NaCl} + y \text{ m KNO}_3$
- $T = 100^\circ\text{C}$
- No apparent influence of Nitrate concentration
- Average CR for Black Annealed (ASW + SHT) Specimens more erratic



# Corrosion Rate of Creviced Alloy 22



- **Average Values of Corrosion Rate (CR) as a function of Chloride Concentration for Times >250 Days**
- **x m NaCl + y m KNO<sub>3</sub>**
- **T = 100°C**
- **No apparent influence of Chloride Concentration**
- **Average CR for Black Annealed (ASW + SHT) Specimens more erratic**



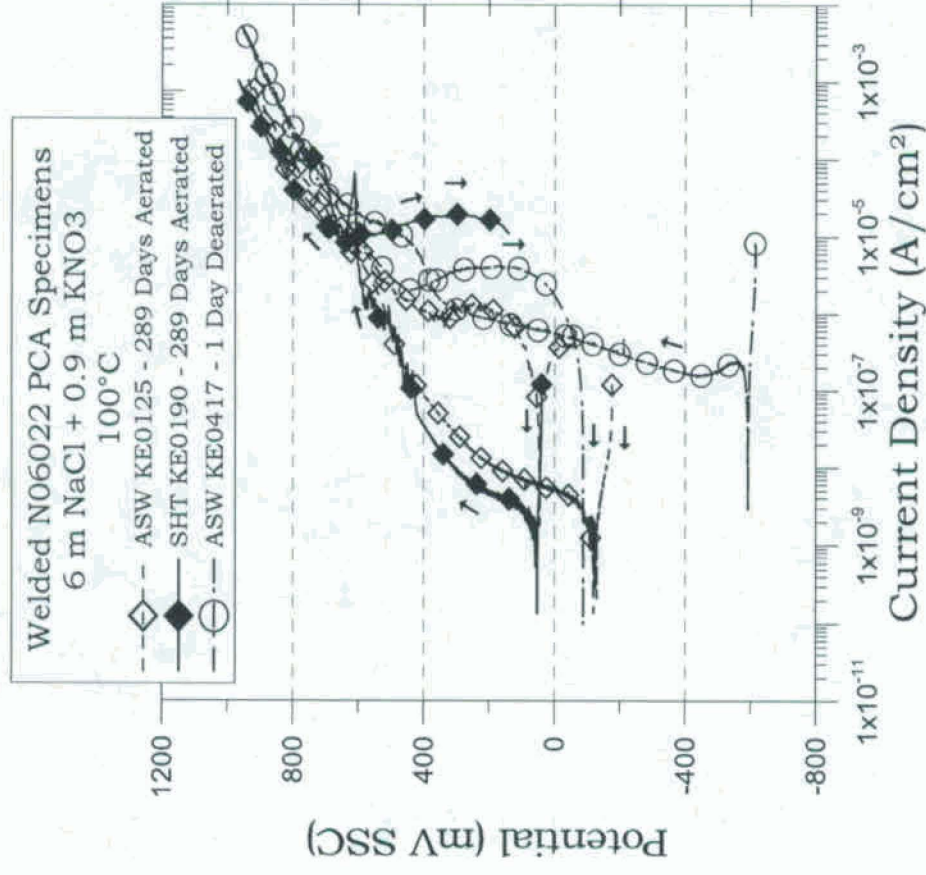


# Effect of Immersion Time on Repassivation Potential

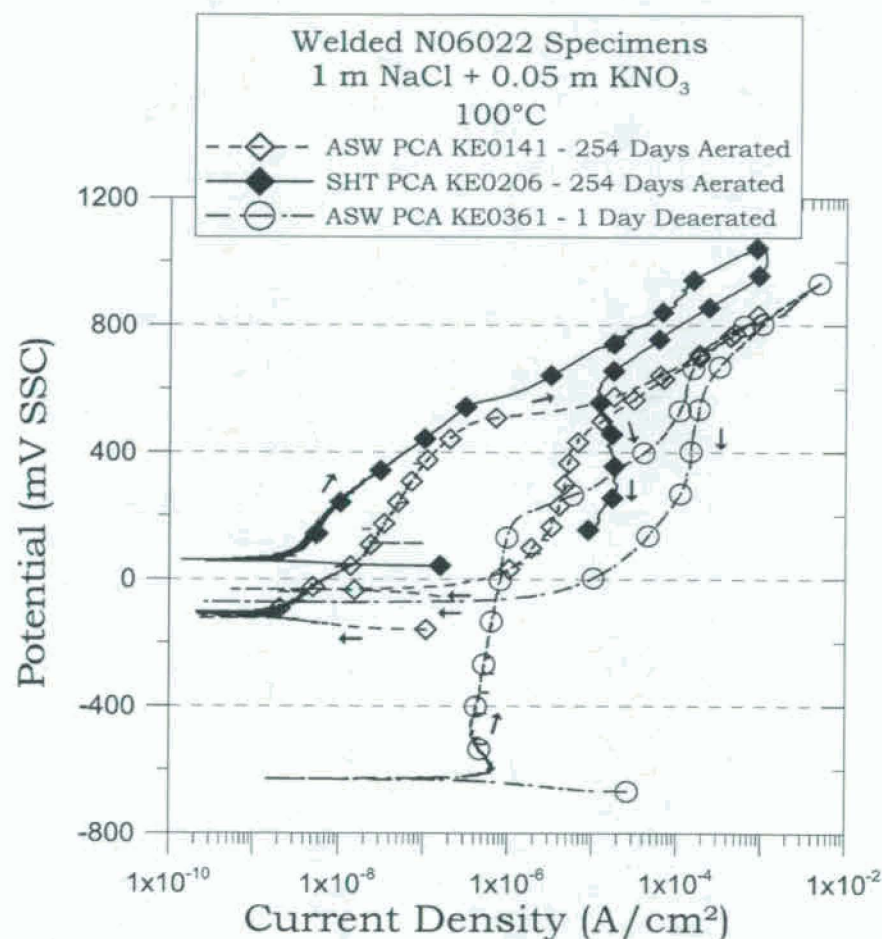


# Effect of Immersion Time on Cyclic Polarization

- Cyclic Potentiodynamic Polarization
  - 1 Day Deaerated (Polished)
  - >8 Months Aerated (Prepolished)
  - >8 Months Aerated (Black Annealed)
- 6 m NaCl + 0.9 m KNO<sub>3</sub>
- T = 100°C
- After 8 Mo. Passive Current Two orders of Magnitude Lower
- After 8 Mo. Repassivation Potential Higher



# Effect of Immersion Time on Cyclic Polarization



- Cyclic Potentiodynamic Polarization
  - 1 Day Deaerated (Polished)
  - >8 Months Aerated (Prepolished)
  - >8 Months Aerated (Black Annealed)
- 1 m NaCl + 0.05 m KNO<sub>3</sub>
- T = 100°C
- After 8 Mo. Passive Current Two orders of Magnitude Lower
- After 8 Mo. Repassivation Potential Higher





# Effect of Immersion Time on Breakdown Potential

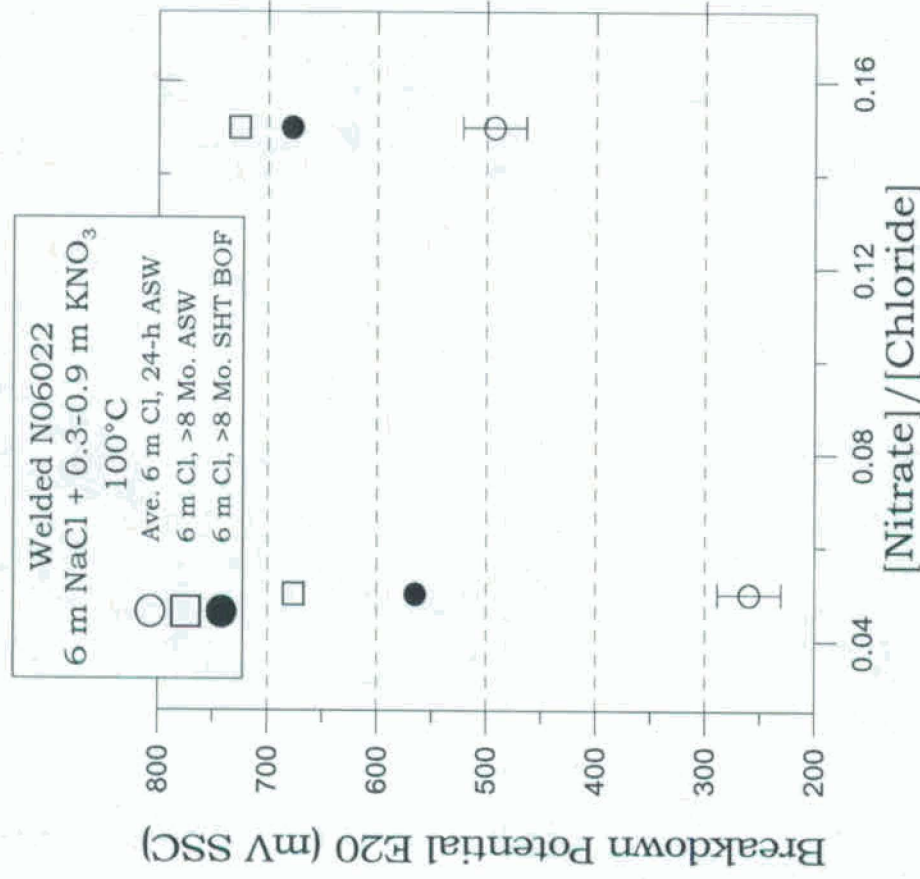
## Cyclic Potentiodynamic Polarization

- 1 Day Deaerated (Polished)
- >8 Months Aerated (Prepolished)
- >8 Months Aerated (Black Annealed)

6 m NaCl + x m KNO<sub>3</sub>

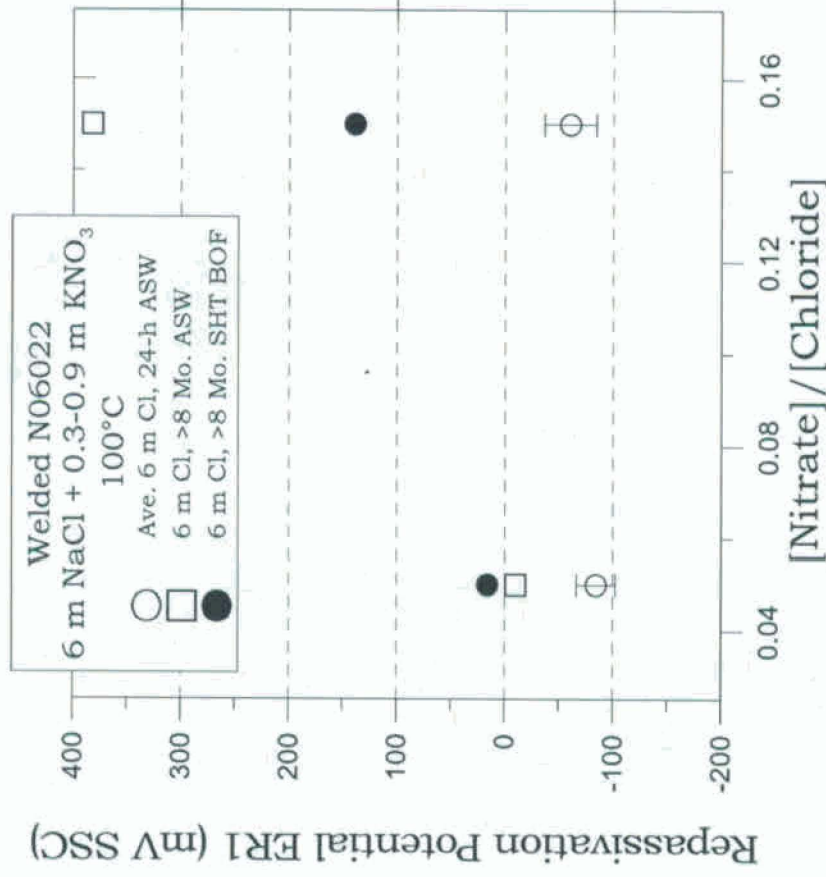
T = 100°C

After 8 Mo. Breakdown Potential Higher



# Effect of Immersion Time on Repassivation Potential

- Cyclic Potentiodynamic Polarization
  - 1 Day Deaerated (Polished)
  - >8 Months Aerated (Prepolished)
  - >8 Months Aerated (Black Annealed)
- 6 m NaCl + x m KNO<sub>3</sub>
- T = 100°C
- After 8 Mo. Repassivation Potential Higher



# Inspection of Specimens After Shut Down (20Nov04)





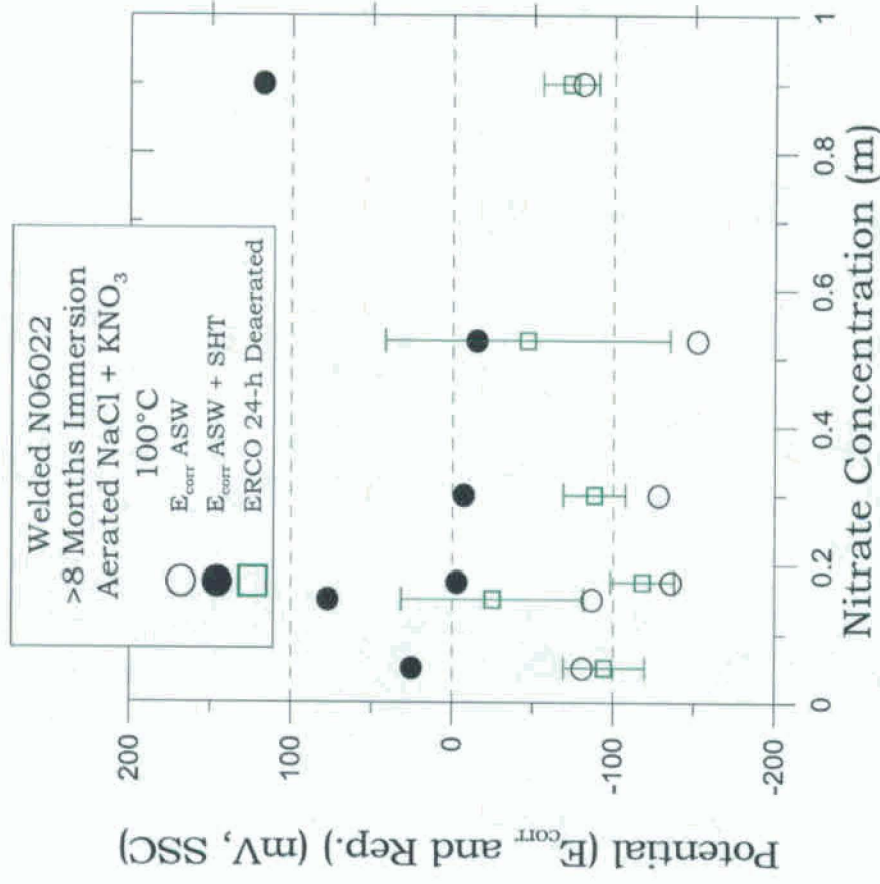
## Six Cells – 48 Specimens

- Each Cell had
  - 4 ASW Polished Creviced Specimens
  - 4 Black Annealed Crevice Specimens
- For each Cell one specimen of each kind was used to Measure Cyclic Polarization Curve
- The Three Other Specimens of Each Kind in Each Cell Were only Exposed to the Open Circuit Potential (Natural Polarization by Air)



# Correlation of Long Term $E_{\text{corr}}$ and $E_{\text{crit}}$

- Cell 27 = 1 m Cl + 0.05 NO3  
ASW = No CC  
ASW + SHT = 3/3 Small CC
- Cell 26 = 1 m Cl + 0.15 NO3  
ASW = No CC  
ASW + SHT = 1/3 Small CC
- Cell 25 = 3.5 m Cl + 0.175 NO3  
ASW = No CC  
ASW + SHT = 2/3 Small CC
- Cell 24 = 3.5 m Cl + 0.525 NO3  
ASW = No CC  
ASW + SHT = No CC
- Cell 22 = 6 m Cl + 0.3 NO3  
ASW = No CC  
ASW + SHT = CC
- Cell 23 = 6 m Cl + 0.9 NO3  
ASW = 2/3 Small CC  
ASW + SHT = CC





# Cell 23 After >8 Months Immersion

6 m NaCl + 0.9 m KNO<sub>3</sub>, NO<sub>3</sub>/Cl = 0.15



ASW KE0127 X 10  
1 Spot Crevice Corrosion



ASW + SHT KE0191 X 10  
1 Spot Crevice Corrosion





# Cell 22 After >8 Months Immersion

6 m NaCl + 0.3 m KNO<sub>3</sub>, NO<sub>3</sub>/Cl = 0.05



ASW KE0122 X 10  
No Crevice Corrosion



ASW + SHT KE0188 X 10  
Small Crevice Corrosion



# > 8 Months Immersion



ASW KE0131  
X8 Cell 24



ASW + SHT KE0196  
X8 Cell 24



ASW KE0136  
X8 Cell 25



ASW + SHT KE0200  
X20 Cell 25





## > 8 Months Immersion



ASW KE0140  
X8 Cell 26



ASW + SHT KE0205  
X8 Cell 26



ASW KE0144  
X8 Cell 27



ASW + SHT KE0209  
X8 Cell 27





# Conclusions Black Annealing Scale

- In the tested conditions, the  $E_{\text{corr}}$  of Alloy 22 specimens increased as time increased
- Increase of  $E_{\text{corr}}$  more gradual for polished ASW PCA specimens
- Long Term  $E_{\text{corr}}$  was higher for Black Annealed specimens
- Little effect of solution composition on  $E_{\text{corr}}$
- Corrosion Rate (CR) of creviced specimens decreased as time increased, both for Polished and Black Annealed
- After 100 days of immersion, the corrosion rate was nominally 30 nm/year, even though salt concentration was ~30% and  $T = 100^{\circ}\text{C}$
- After Long-Term Immersion in Aerated Brines, the Breakdown Potential and Repassivation Potential Increased
- After >6 Months Immersion, ASW + SHT Specimens were more prone to crevice corrosion with natural polarization



# **Part 3**

## **Effect of Surface Stress Mitigation on the General and Localized Corrosion Immersion and Electrochemical Tests**



# Conditions of Tested Plates

- **As-Welded (ASW) – Gas Tungsten Arc Welding (GTAW)**
  - Non-treated
- **Low Plasticity Burnishing (LPB)**
  - Produces compressive stresses by rolling a metallic ball over the surface
- **Laser Shock Peening (LSP)**
  - Produces compressive stresses by means of a laser beam, which produces a high-pressure plasma





# Laser Shock Peening (LSP)

Hill et al.,  
Adv. Mater. and Processes  
August 2003

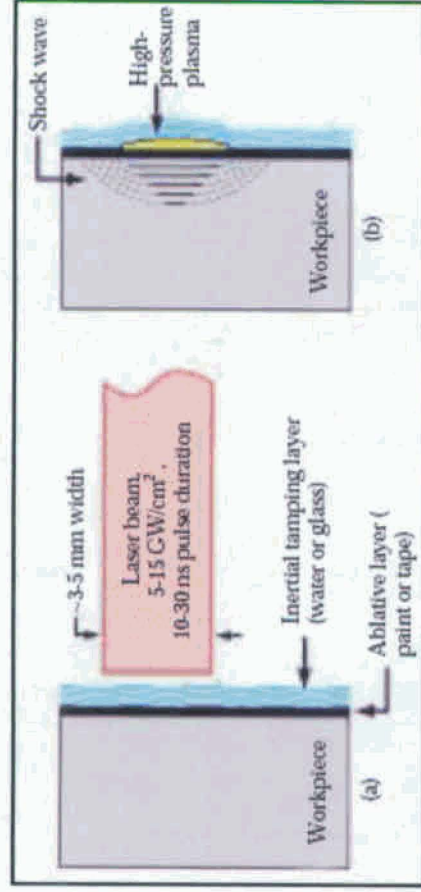


Fig. 1 — Description of laser peening process. (a) Workpiece is covered with a protective ablative layer and an inertial tamping layer. (b) Laser pulse forms high-pressure plasma on the surface of the part, causing a shock wave to travel through the

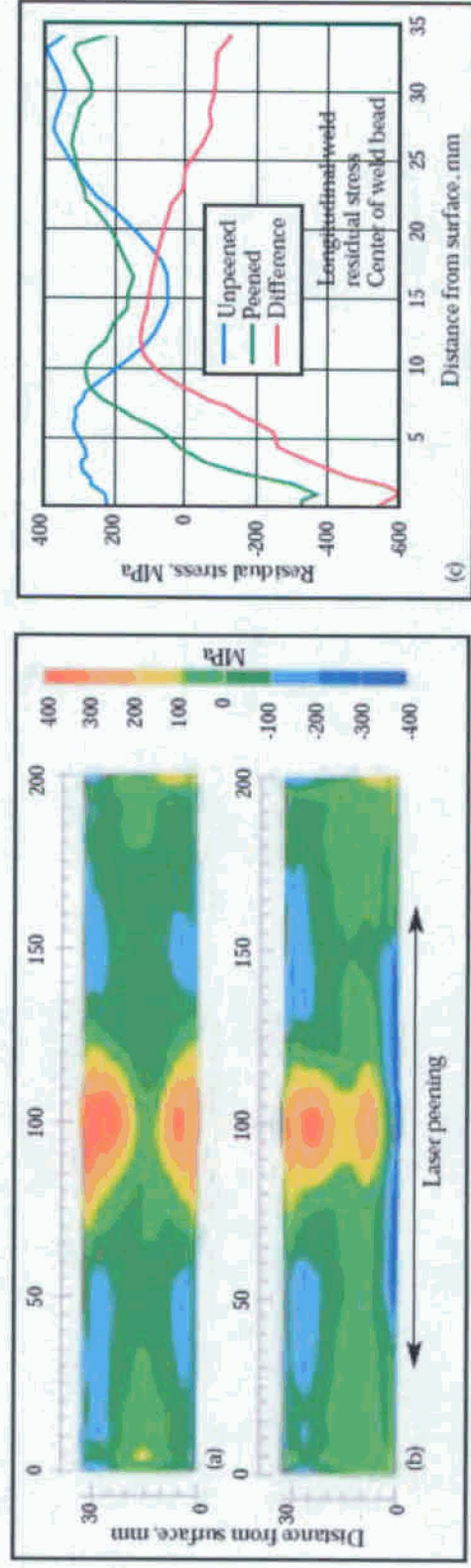


Fig. 2 — Longitudinal residual stress in a 33-mm thick Alloy 22 welded plate: (a) map of residual stress in unpeened weld; (b) map of residual stress in laser-peened weld; (c) line plot of residual stress versus depth at the center of the weld bead.



# Low Plasticity Burnishing (LPB)

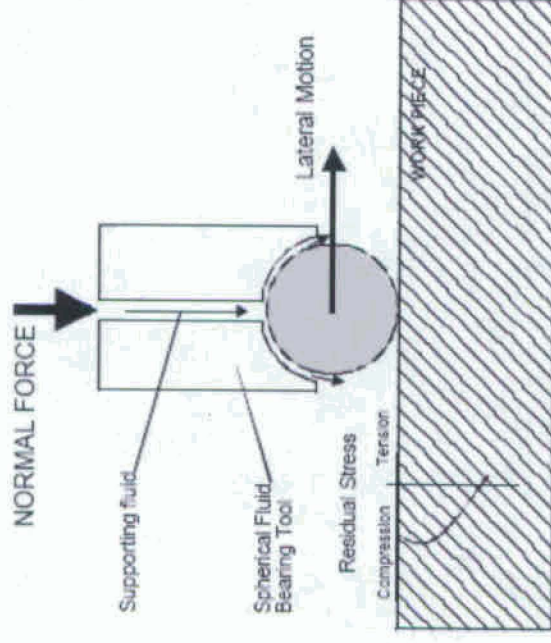
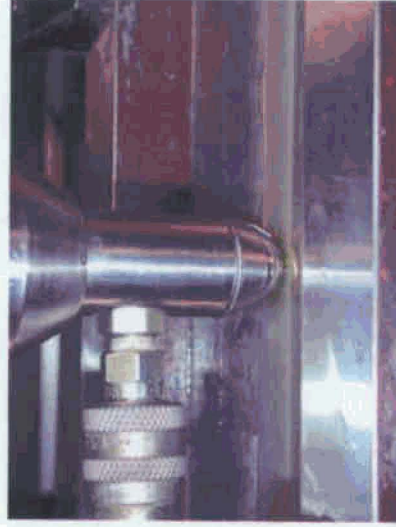
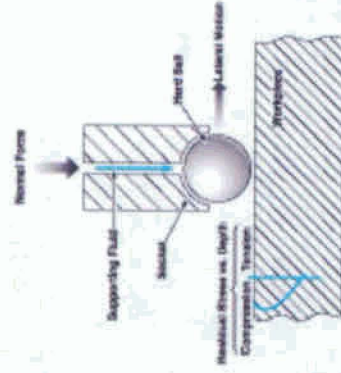


Figure 2 - Low Plasticity Burnishing (LPB) schematic.

Prev y et al., Lambda Research  
High Cycle Fatigue Conf.  
March 2001



# Current Results Immersion Corrosion Testing





## Immersion Corrosion Tests

- 42 g/L Ferric Sulfate + 50% Sulfuric Acid Solution
- Highly Oxidizing and Acidic – ASTM G 28 A
- Boiling for 24 h
- Corrosion Rate Calculated by Mass (Weight) Loss

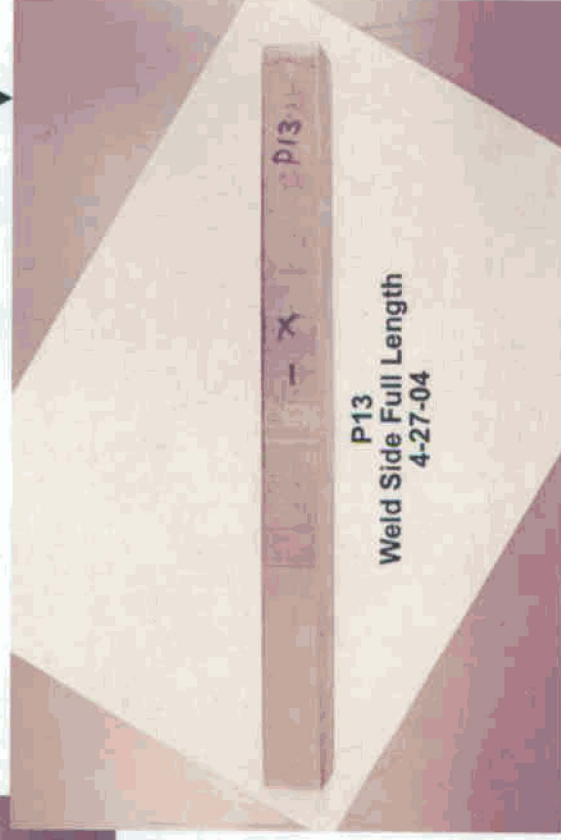
$$CR(mm / year) = 8.76 \times 10^4 \frac{\Delta W(g)}{A(cm^2) \cdot t(h) \cdot d(g \cdot cm^{-3})}$$



# As-Received Material



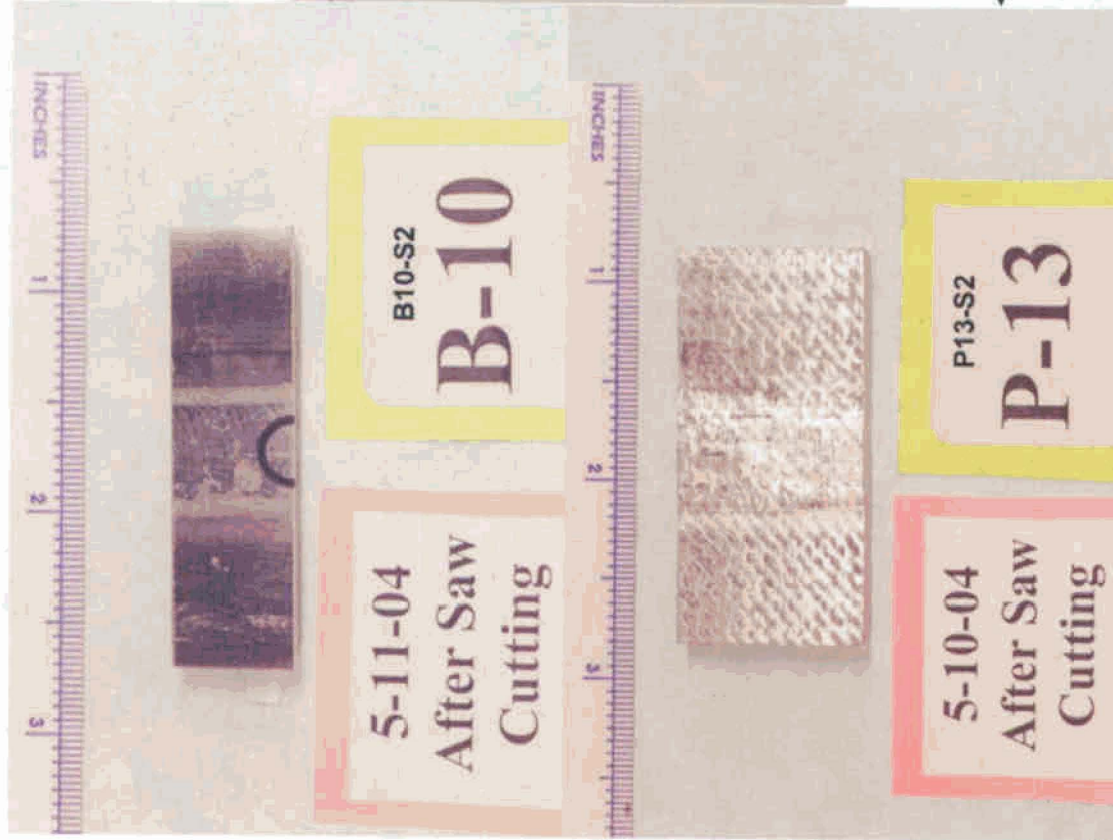
Laser Shock Peening  
LSP



Low Plasticity Burnishing  
LPB



# Coupon Preparation



Pre-Test LPB

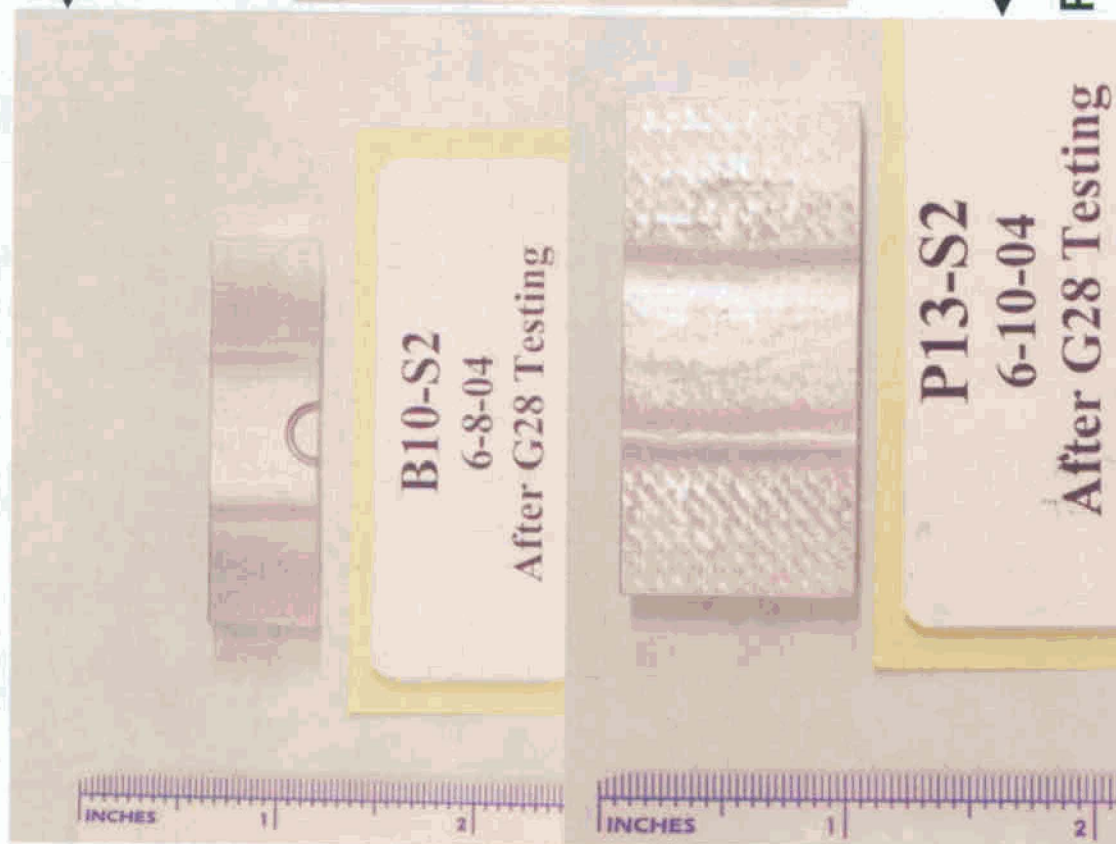
Pre-Test ASW

Pre-Test LSP





# Coupon Preparation



Post-Test LPB  
Post-Test ASW



Post-Test LSP



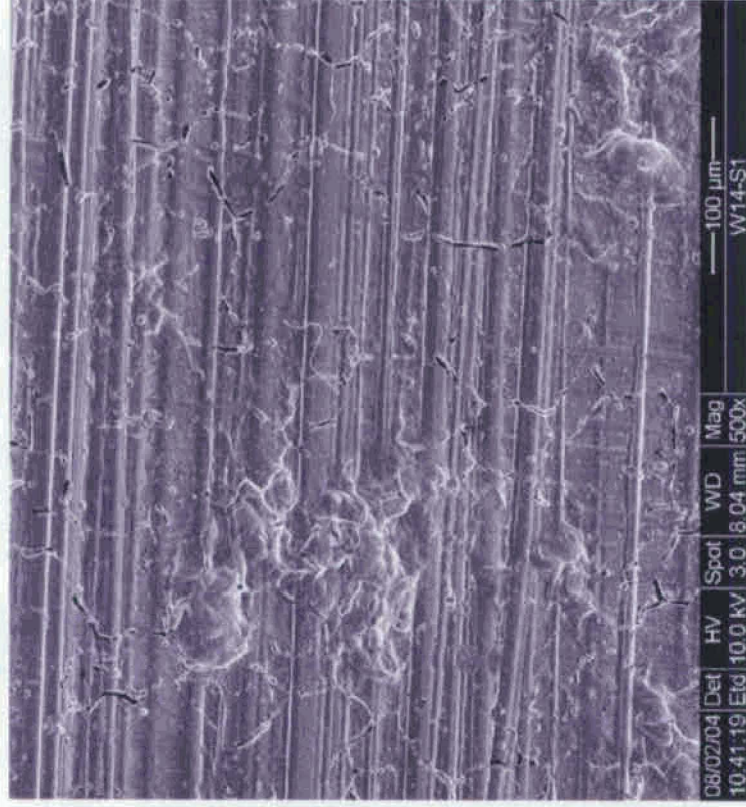
# Corrosion Rates of the Three Materials

|                                                   | ASW               | LPB               | LSP               |
|---------------------------------------------------|-------------------|-------------------|-------------------|
| Corrosion Rate (mm/year) $\pm$ Standard Deviation | 1.303 $\pm$ 0.040 | 1.295 $\pm$ 0.106 | 1.257 $\pm$ 0.055 |
| Observations                                      | IGA in HAZ        | IGA in HAZ        | IGA in HAZ        |





# SEM Observations - ASW



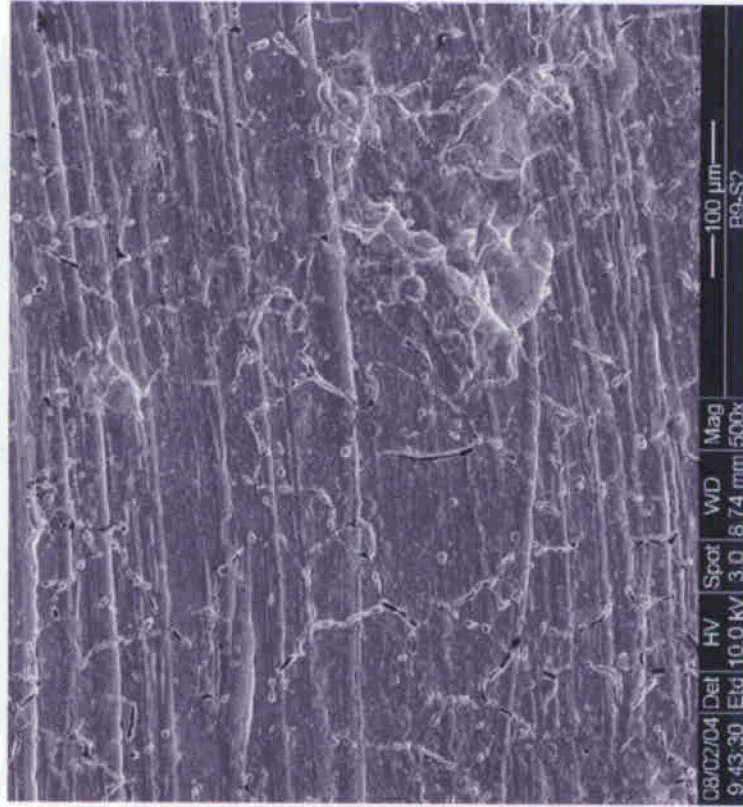
X500, Base

X500, HAZ



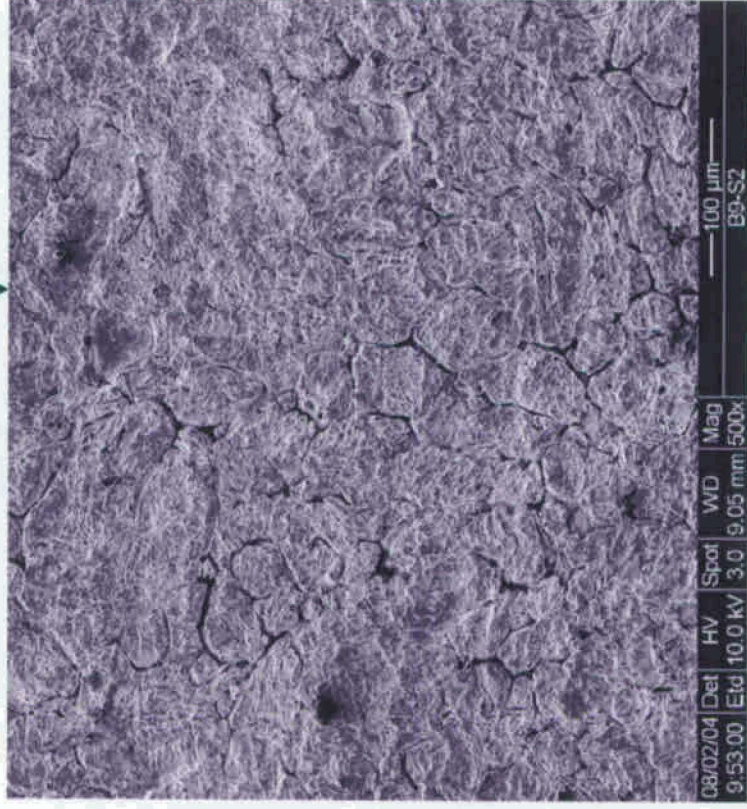


# SEM Observations - LPB

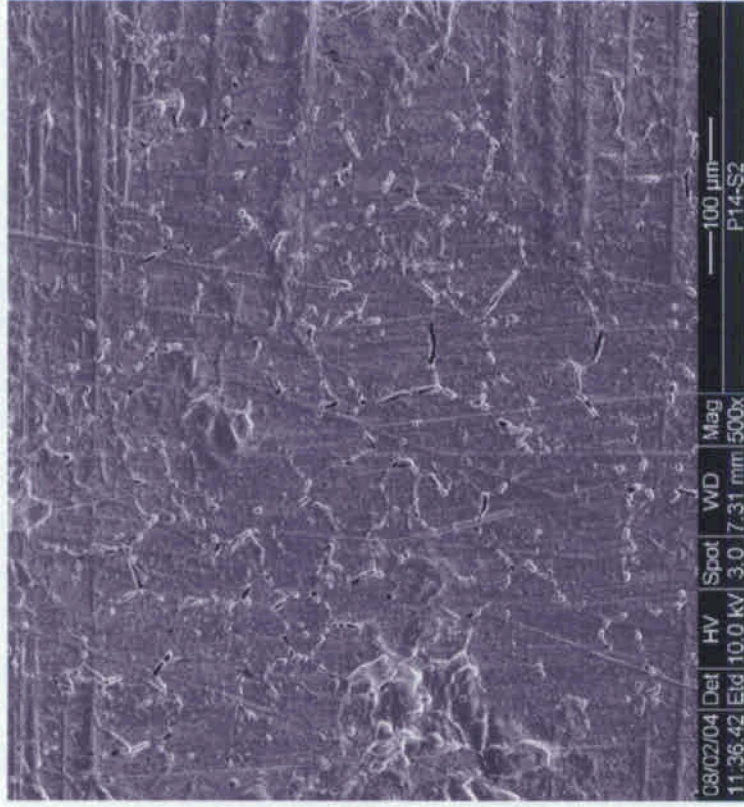


X500, Base

X500, HAZ



# SEM Observations - LSP



X500, Base

X500, HAZ





# Current Results

## Electrochemical Testing

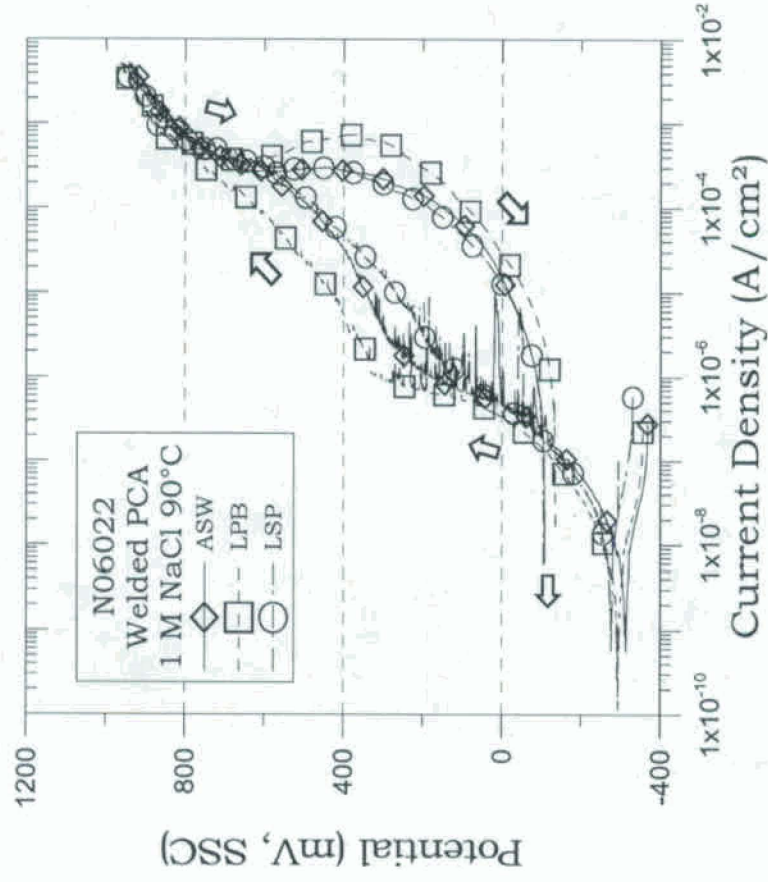
### Cyclic Potentiodynamic Polarization





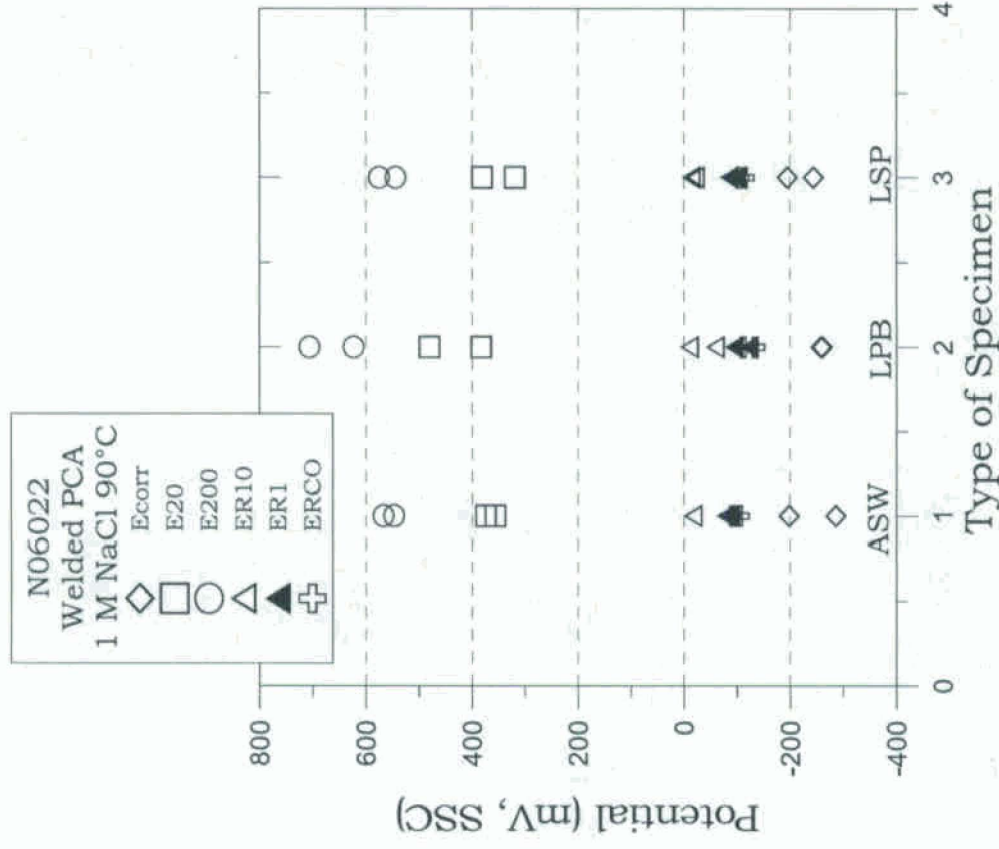
# Cyclic Polarization of Alloy 22

- Three Materials: ASW, LPB, LSP
- 1 M NaCl, 90°C
- All three materials behave the same



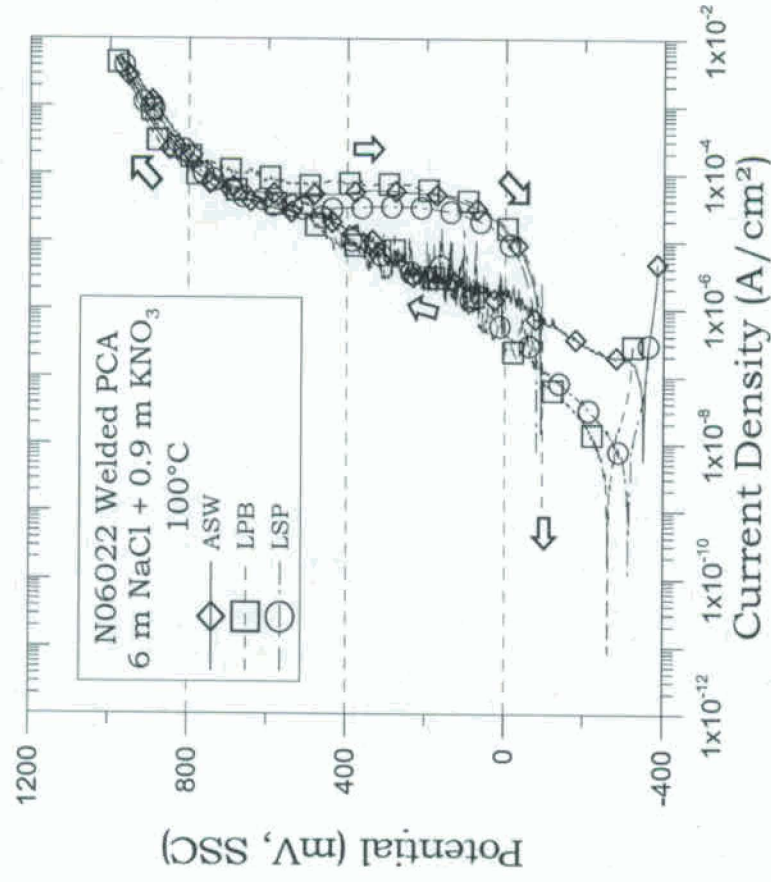
# Characteristic Potentials

- 1 M NaCl, 90°C
- Corrosion, Breakdown and Repassivation Potentials are the same for ASW, LPB and LSP Materials
- Repassivation Potentials higher than Corrosion Potentials



# Cyclic Polarization of Alloy 22

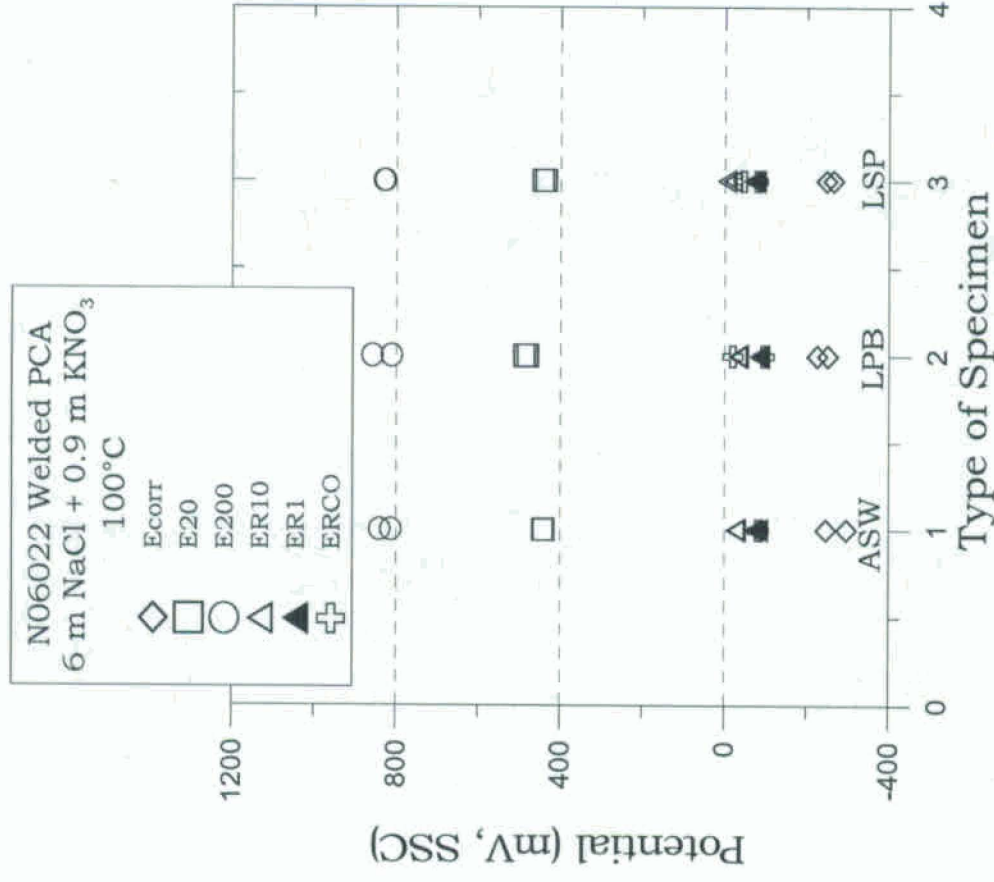
- Three Materials: ASW, LPB, LSP
- 6 m NaCl + 0.9 m KNO<sub>3</sub>, 100°C
- All three materials behave the same





# Characteristic Potentials

- 6 m NaCl + 0.9 m KNO<sub>3</sub>, 100°C
- Corrosion, Breakdown and Repassivation Potentials are the same for ASW, LPB and LSP Materials
- Repassivation Potentials higher than Corrosion Potentials



# SEM of Polarized Specimens



LSP  
Specimen P5  
X500

LPB  
Specimen B3  
X500



Crevice  
Corrosion  
(Inter-dendritic  
Attack)  
In  
1 M NaCl  
90°C

ASW  
Specimen W6  
X250





# Conclusion on Stress Mitigation

- Surface Stress Mitigation such as Low Plasticity Burnishing (LPB) and Laser Shock Peening (LSP) do not Affect the Corrosion Resistance of Alloy 22
- The corrosion by weight-loss in an aggressive oxidizing and acidic solution was the same for the three tested materials (ASW, LPB and LSP)
- Electrochemical Tests showed that the anodic behavior of the three materials was the same
- The repassivation potential was the same
- When corrosion occurred the mode of attack in the three materials was the same





# **Part 4**

## **Testing of Specimens Fabricated from a Mock Up Container Electrochemical Tests**



# Fabrication of the Mockup



# Full Diameter and $\frac{1}{4}$ Length



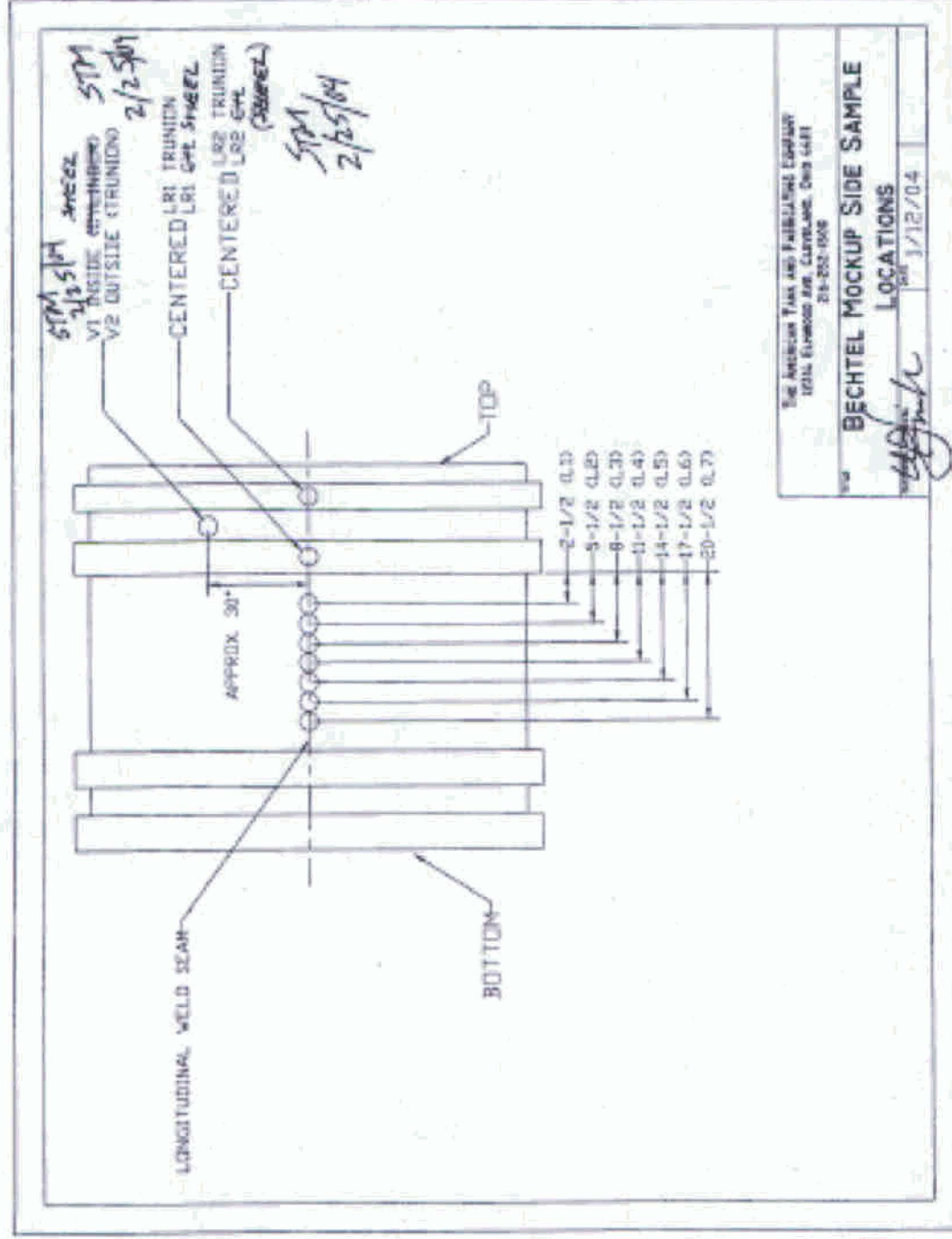


## Specimen Preparation

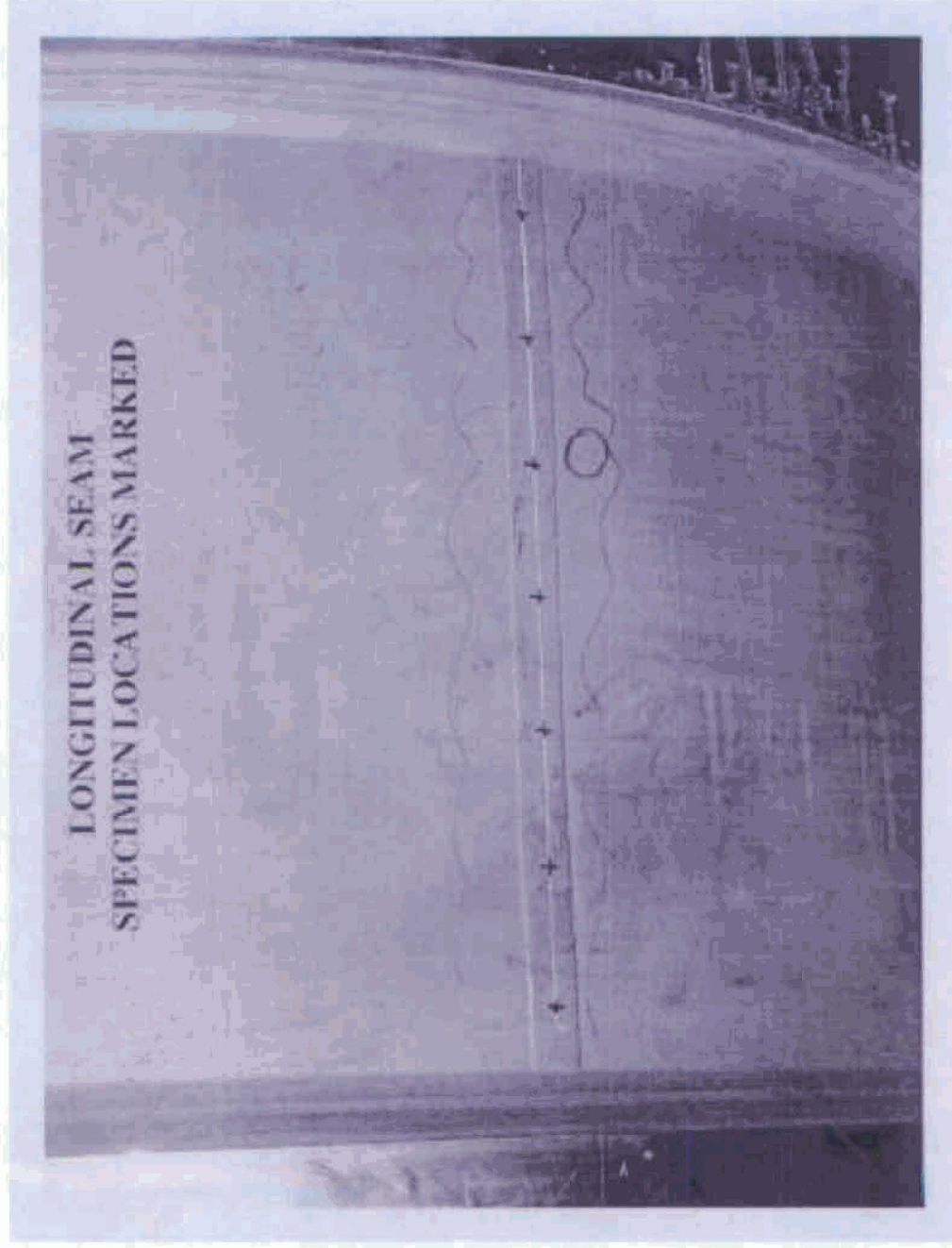
- The container was fabricated at American Tank in Cleveland, Ohio
- The container was solution annealed and water quenched
- Hockey-pucks like samples were prepared from several parts of the mockup container
- The specimens for electrochemical testing were machined from the longitudinal weld of the mockup container



# Location of Specimens in the Container

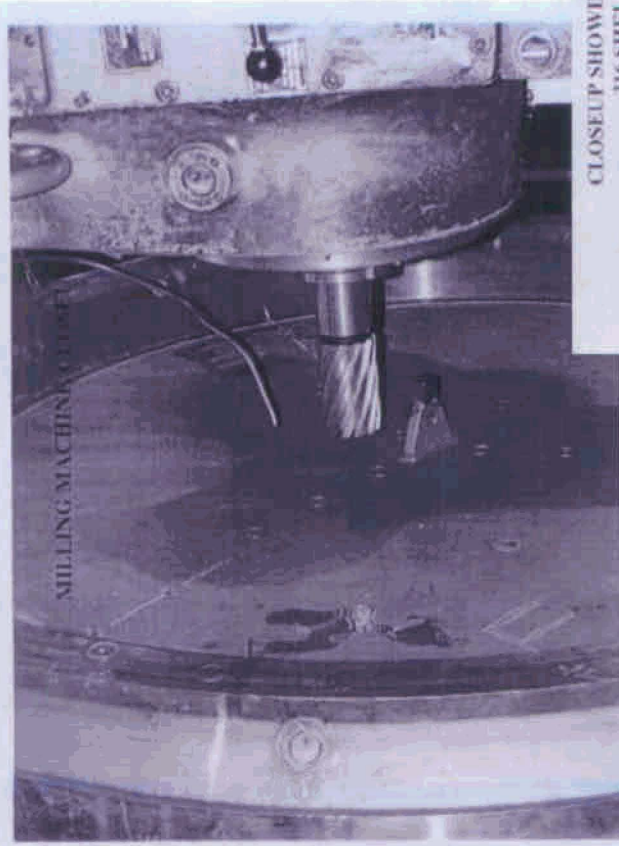


# Location of Specimens in the Container





# Removal of Samples Procedure



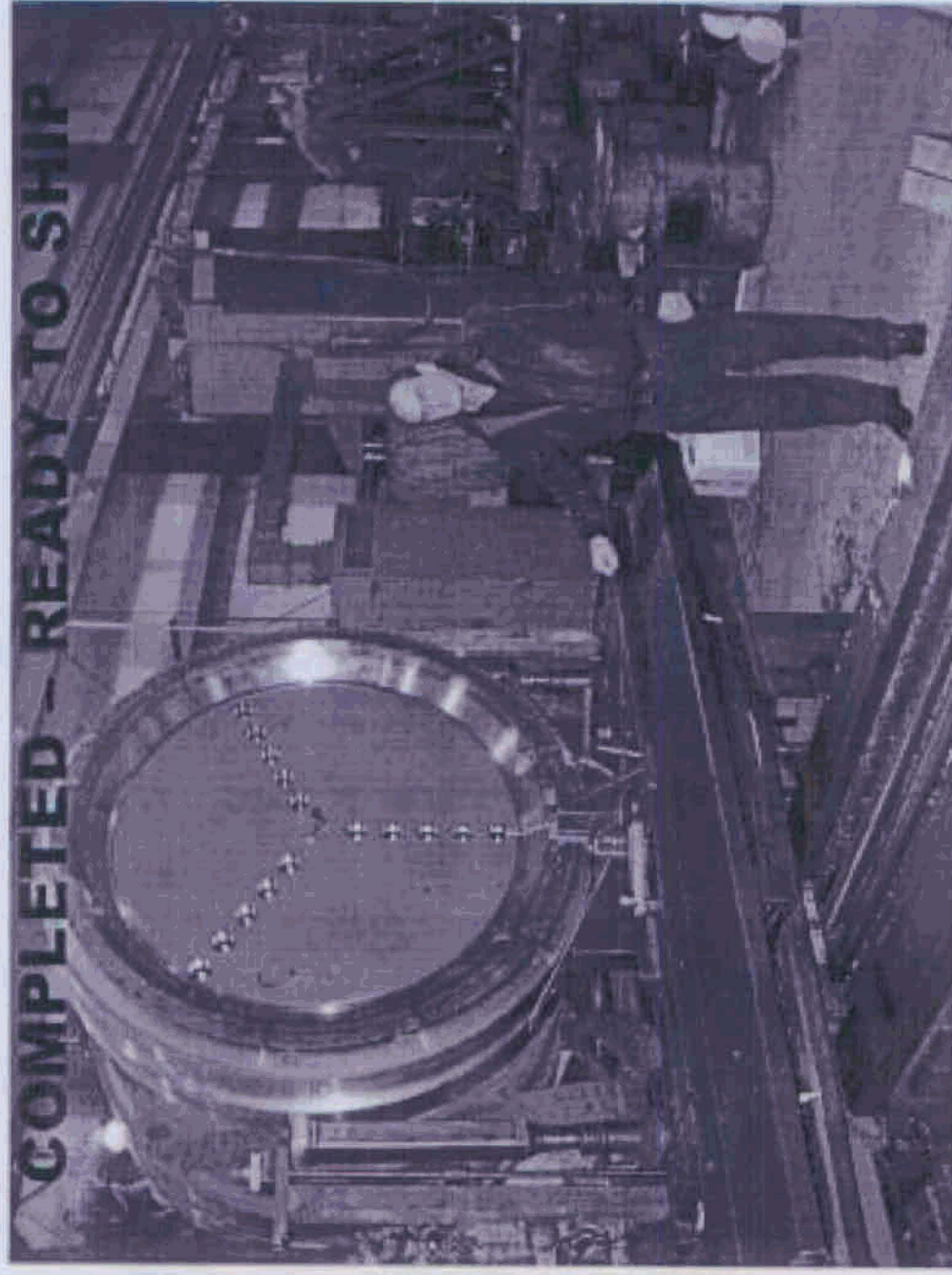
VIEW OF A SPECIMEN REMOVED FROM PROTOTYPE



CLOSEUP SHOWING INNER  
316 SHELL



# Relative Size of the Container



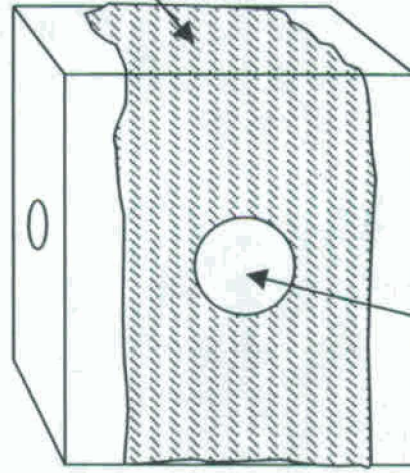
# Final Specimen Characteristics





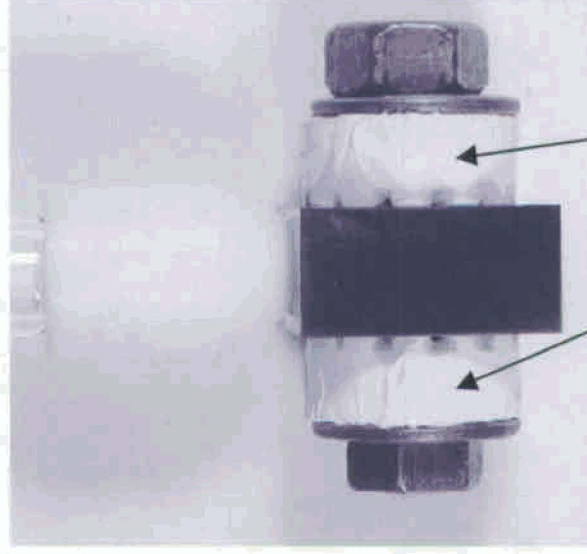
# Prism Creviced Assembly (PCA)

## WELDED PCA SPECIMEN



GTAW  
Seam

Hole for  
Crevice Formers



Two Crevice Formers  
With 12 Teeth Each  
ASTM G 48



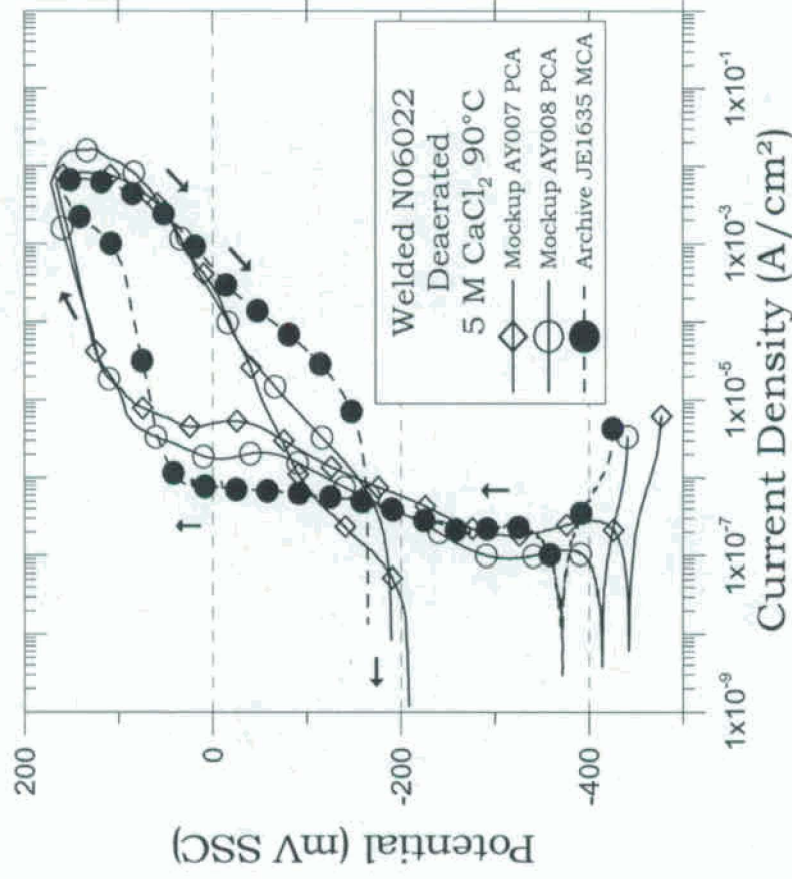
# Electrochemical Tests

- Cyclic Potentiodynamic Polarization (CPP)
  - 0.167 mV/s
- Electrolytes at 80°C, 90°C, 100°C and 130°C
  - 6 m NaCl + 0.3 m KNO<sub>3</sub> (Nitrate/Chloride = 0.05)
  - 6 m NaCl + 0.9 m KNO<sub>3</sub> (Nitrate/Chloride = 0.15)
  - 5 M CaCl<sub>2</sub>
  - 1 M NaCl
  - 12 m CaCl<sub>2</sub> + 6 m KNO<sub>3</sub> (Nitrate/Chloride = 0.5)



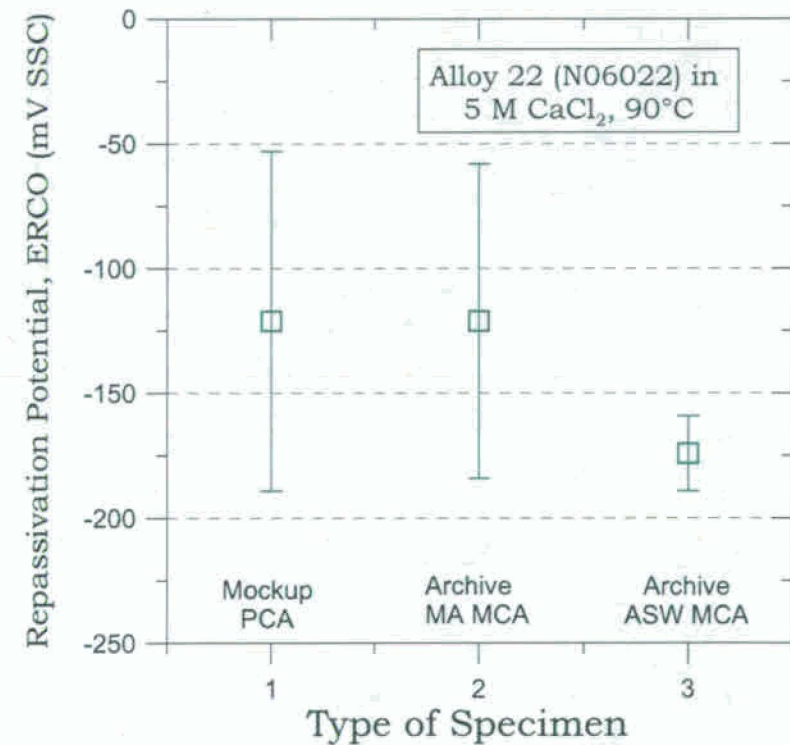
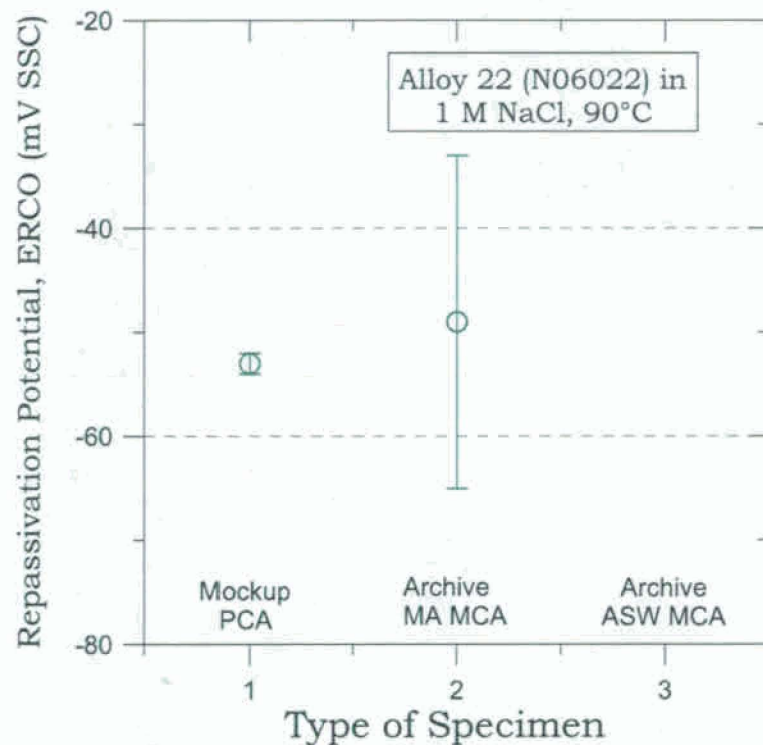
# Cyclic Potentiodynamic Polarization

- No differences in the anodic behavior between two specimens prepared from the mockup container and a specimen from archive data
- Repassivation potentials are also the same

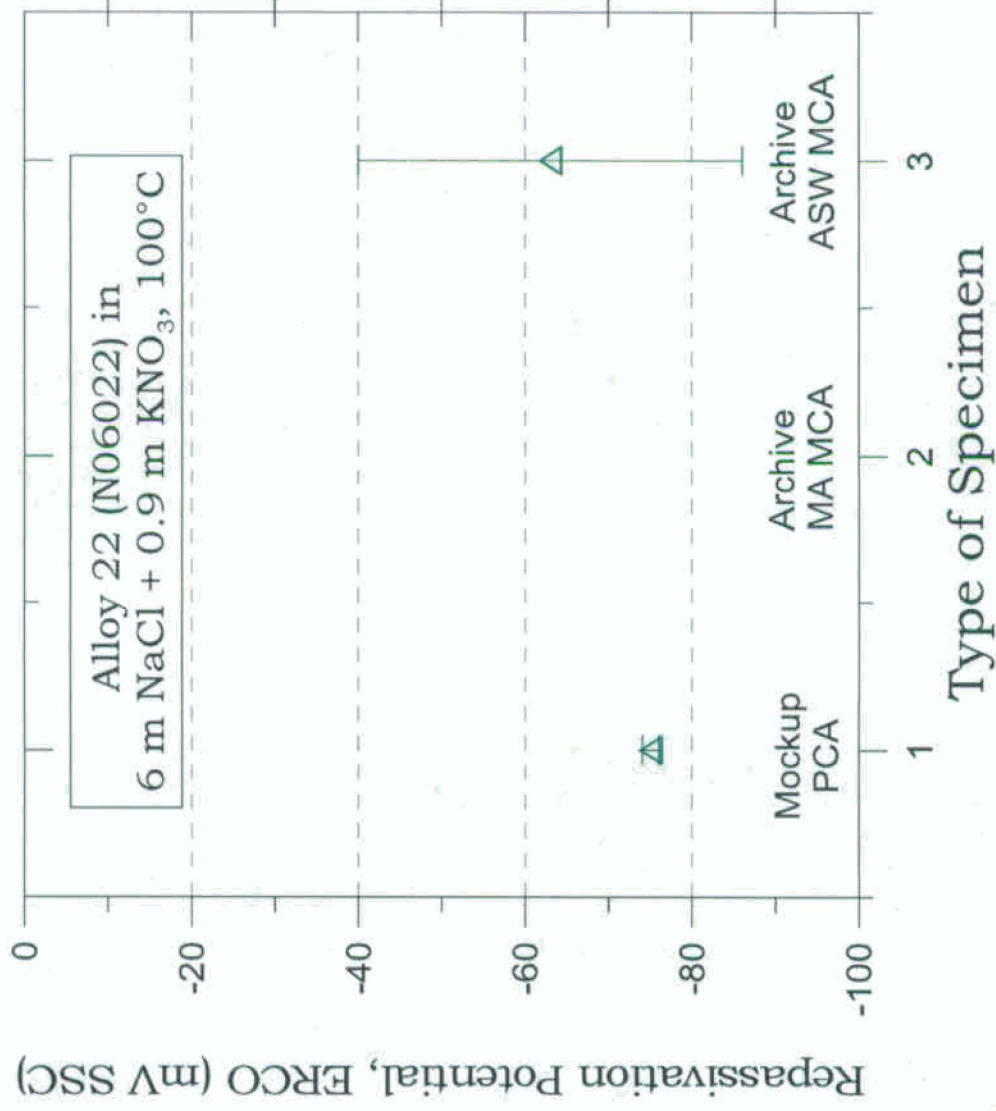




# Crevice Repassivation Potentials Mockup and Archive Data



# Crevice Repassivation Potentials



# Macrographs



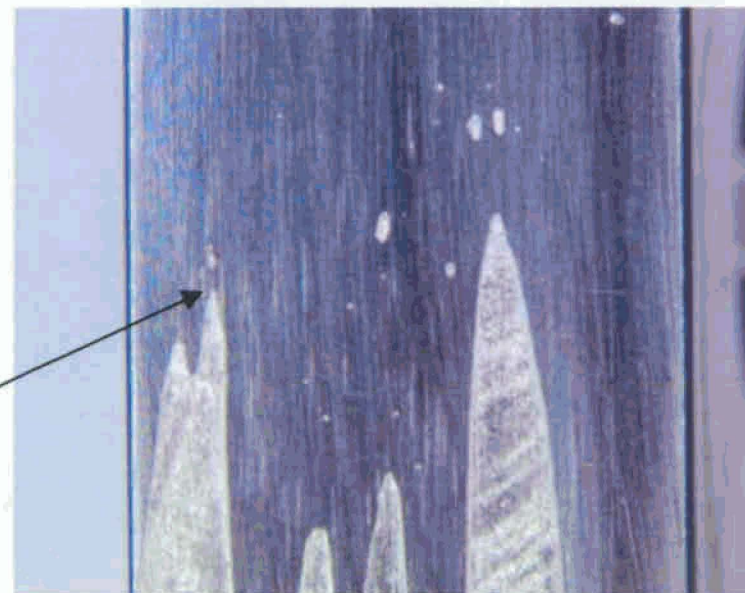


# After The Cyclic Potentiodynamic Polarization in 5 M $\text{CaCl}_2$ at 90°C



No Crevice Corrosion  
Localized Attack outside the  
Crevice Formers and Starting at  
Crevice Formers

Magnification X8



Side (Non-Creviced) View



# After The CPP in 12 m $\text{CaCl}_2$ + 6 m $\text{Ca}(\text{NO}_3)_2$ at 130°C



No Crevice Corrosion  
Localized Attack outside the  
Crevice Formers

Magnification X8

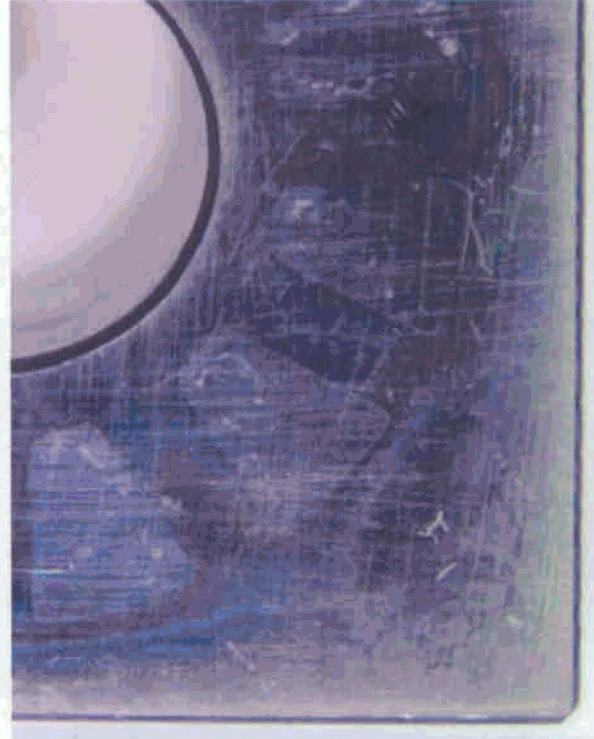


Side (Non-Creviced) View





# After The Cyclic Potentiodynamic Polarization in 6 m NaCl + 0.9 m KNO<sub>3</sub>



80°C

No Crevice Corrosion

Magnification X8



100°C

Minor Dull Crevice Corrosion





# Conclusions on Mock Up Container

- Under the tested conditions, the repassivation potentials (ER1 and ERCO) obtained using specimens prepared from a prototype container (Mockup) had similar values as the repassivation potentials currently available for specimens fabricated using laboratory welded flat plates
- Values of repassivation potential used in the models to predict the lifetime of the container via localized corrosion accurately represent the behavior of a fabricated container.



# Overall Summary

- Alloy 22 is Highly Resistant to General and Localized Corrosion
- In the Tested Conditions No Difference in Behavior was Found Between Welded and Non-Welded Specimens
- Fabrication Processes Do not Affect Corrosion Performance
  - Heat to Heat Variability – No Effect
  - Black Annealing Scale Generally Does Not Have an Effect on General and Localized Corrosion Behavior
  - Surface Stress Mitigation – No Effect
  - Specimens Prepared from a Full-Diameter Mock up Container Behave the Same as Other Laboratory Specimens





# Acknowledgments

- This work was performed under the auspices of the U. S. Department of Energy by the University of California Lawrence Livermore National Laboratory under contract N° W-7405-Eng-48. This work is supported by the Yucca Mountain Project, LLNL which is part of the Office of Civilian Radioactive Waste Management OCRWM-DOE
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