7-7-2004

# Environment-Induced Degradation and Crack-Growth Studies of Candidate Target Materials: Annual Progress Report (May 2003 – May 2004)

Ajit K. Roy
*University of Nevada, Las Vegas*, aroy@unlv.nevada.edu

## Repository Citation

Roy, A. K. (2004). Environment-Induced Degradation and Crack-Growth Studies of Candidate Target Materials: Annual Progress Report (May 2003 – May 2004). 1-9.
**Available at:** https://digitalscholarship.unlv.edu/hrc_trp_sciences_materials/51

# Annual Progress Report
# (May 2003 – May 2004)

## Environment-Induced Degradation and Crack-Growth Studies of Candidate Target Materials

## TRP Task-4

**Principal Investigator**
**Ajit K. Roy, Ph.D.**

**Investigators**
**Mohammad K. Hossain**
**Ramprashad Prabhakaran**
**Sudheer Sama**
**Phani P. Gudipati**
**Venkataramakrishnan Selvaraj**

**University of Nevada, Las Vegas**

**July 07, 2004**

# Environment-Induced Degradation and Crack-Growth Studies of Candidate Target Materials

## Introduction

As indicated in the original proposal, the primary objective of this task was to evaluate the effect of hydrogen on environment-assisted cracking of candidate target structural materials for applications in spallation-neutron-target (SNT) systems such as accelerator production of tritium (APT) and accelerator transmutation of waste (ATW). The materials selected for evaluation and characterization were martensitic stainless steels including Alloy EP 823, HT-9, and Type 422 stainless steel. The susceptibility to stress corrosion cracking (SCC) of these materials were evaluated in neutral and acidic aqueous environments using smooth and notched tensile specimens under constant-load (CL) and slow-strain-rate (SSR) conditions. Further, the localized corrosion (pitting and crevice) behavior of these alloys was evaluated by electrochemical polarization technique. The extent and morphology of cracking and localized corrosion of the tested specimens were determined by optical microscopy and scanning electron microscopy (SEM).

The experimental program proposed in this task was refocused to evaluate the effect of molten lead-bismuth-eutectic (LBE) on the corrosion behavior of similar target structural materials in the presence of oxygen. Since the Materials Performance Laboratory (MPL) at UNLV could not accommodate this type of testing, the LBE loop at the Los Alamos National Laboratory (LANL) is currently being used to contain the stressed test specimens to evaluate the SCC and localized corrosion (pitting and crevice) behavior of all three candidate alloys in the molten LBE environment. Since the magnitude of the applied load/stress during these tests could not monitored or controlled (as in conventional SCC experiments) in the LBE environment, the test specimens were self-loaded. Two types of specimen configurations, namely C-ring and U-bend, were used to perform these experiments. The results of SCC testing being conducted at LANL are not yet available. The stress of principal interest in both types of specimen is the circumferential stress. SCC tests using these types of self-loaded specimens have also been conducted at MPL in aqueous environments having neutral and acidic pH values at ambient and elevated temperatures.

## Personnel

The current project participants are listed below.

Principal Investigator (PI): Dr. Ajit K. Roy
Department of Mechanical Engineering, UNLV
Phone: (702) 895-1463 email: aroy@unlv.nevada.edu

Investigators (UNLV): Mr. Mohammad K. Hossain, Ph. D. Student, Mechanical Engineering
Mr. Ramprashad Prabhakaran, M.S. Student, Mechanical Engineering
Mr. Sudheer Sama, M.S. Student, Mechanical Engineering
Mr. Venkataramakrishnan Selvaraj, M.S. Student, Mechanical Engineering
Mr. Phani P Gudipati, M.S. Student, Mechanical Engineering
Hossain: Phone: (702) 895-5014 email: mkamalh@egr.unlv.edu
Prabhakaran: Phone: (702) 895-5014 email: ram79@egr.unlv.edu
Sama: Phone: (702) 895-5014 email: sudheers@egr.unlv.edu

Selvaraj: Phone: (702) 895-2804 email: svenkat@egr.unlv.edu
Gudipati: Phone: (702) 895-2804 email: gudiphan@egr.unlv.edu

Collaborator (DOE): Dr. Ning Li, LANL, New Mexico
Phone: (505) 665-6677 email: ningli@lanl.gov
Dr. Stuart A. Maloy, LANL, New Mexico
Phone: (505) 667-9784 email: Maloy@lanl.gov

## Accomplishments

- Ambient temperature mechanical properties of Alloys EP-823, HT-9 and Type 422 Stainless Steel (SS) have been evaluated by a calibrated MTS equipment using smooth and notched tensile specimens.

- SCC testing using calibrated proof rings and smooth and notched uniaxial tensile specimens of all three martensitic alloys has been completed in both neutral and acidic aqueous solutions under CL conditions at ambient temperature and 90°C. The results of CL SCC testing indicate that neither Alloy EP-823 nor Type 422 SS showed failure in the neutral environment at applied stresses approaching 90 and 95 percent of the material's YS value at ambient temperature and $90^oC$. However, CL SCC testing of Alloy HT-9 in the 90°C neutral solution showed failure at applied stress corresponding to the 95 percent of its YS value. Data also indicate that failures were observed in all three materials when tested in the $90^oC$ acidic environment at applied stresses corresponding to the 95 percent of the materials' YS values. Further, Alloy HT-9 and Type 422 SS exhibited failure in a similar environment at an applied stress equivalent to 90 and 85 percent of their YS values but did not show any failure at applied stress equivalent to 80 percent of their YS value suggesting their threshold stress ($\sigma_{th}$) value in the vicinity of 80 percent of their measured ambient temperature YS values when tested in the $90^0C$ acidic environment. The applied stress versus TTF for all three alloys is shown in Figure 1 showing their $\sigma_{th}$ values. The presence of a notch in the tensile specimen reduced their $\sigma_{th}$ values to much lower levels due to smaller cross-sectional area at the root of the notch. The $\sigma_{th}$ values for the notched specimens were in the vicinity of 45, 30 and 25 percent of their YS values for Alloys EP-823, HT-9, and Type 422 SS, respectively.

- SCC testing using the SSR technique has also been completed in both aqueous environments at ambient temperature, 60 and $90^oC$ involving smooth and notched tensile specimens of all three martensitic alloys. A typical stress versus strain curve is shown in Figure 2, showing the effect of temperature. The results of SSR testing of Alloy EP-823 are illustrated in Figure 3, elucidating the effects of temperature, solution pH and specimen geometry (smooth versus notched gage section) on percent elongation (%El), percent reduction in area (%RA), time-to-failure (TTF) and failure stress ($\sigma_f$). These data indicate that, for the smooth specimen, the ductility parameters, TTF and $\sigma_f$ were significantly reduced in the $90^oC$ acidic solution, showing a synergistic influence of pH and temperature on the cracking susceptibility of this alloy. The presence of notch in the test specimen further reduced the TTF and the extent of ductility in terms of %El and %RA. However, the magnitude of $\sigma_f$ in this material was increased to some extent in the presence of a notch primarily due to a smaller area at the root of the notch. Similar effects of temperature, pH, and specimen geometry on cracking susceptibility were observed with Alloy HT-9 and Type 422 SS.

- The susceptibility of all three alloys to pitting and crevice corrosion in both neutral and acidic aqueous solutions has been determined by performing cyclic potentiodynamic polarization (CPP) experiments using EG&G Model 273A Potentiostats. A three-electrode polarization technique was used. At the onset, the corrosion potential ($E_{corr}$) was measured with respect to a silver/silver chloride (Ag/AgCl) electrode, followed by forward and reverse potential scans at the ASTM-specified rate of 0.17 mV/sec. The magnitudes of the critical pitting potential ($E_{pit}$) and the protection potential ($E_{prot}$), if any, were determined from the CPP diagram. The results indicate that $E_{corr}$ and $E_{pit}$ became more active (negative) with increasing temperature and reduced pH of the test solution, as expected, as shown in Figures 4 for Alloy EP-823 and Type 422 SS, respectively. A similar behavior was observed with Alloy HT-9.

- The results of SCC testing using the C-ring specimens of Alloy HT-9, loaded at 95 and 98 percent of its YS value, showed cracks at their apex when tested in the acidic solution at 50 and 100°C. However, somewhat reduced cracking tendency was observed in Alloy EP-823 under identical testing conditions. No identifiable cracks were observed with the U-bend specimens of either alloy in similar environments. Some degradation marks, indicative of cracking, were noticed along the specimen surface.

- Extensive efforts have been made to analyze the fracture modes in all broken specimens. Based on these fractographic evaluations, it appears that the primary failure mode at the gage section of specimens tested in the neutral environment at both test temperatures was ductile showing dimpled microstructures, as shown in Figure 5A. On the contrary, the specimens tested in the acidic environment showed intergranular and/or transgranular brittle failures at both test temperatures (Figures 5B, 5C and 5D). However, the extent of cracking was more severe at the elevated temperature. The metallographic evaluation of the secondary cracks along the gage section of the broken specimen by optical microscopy revealed branching of cracks, as illustrated in Figure 6.

## Status of Funds

Expenditures incurred during the third year were within the target amount allocated.

## Highlights

- Seven full-length papers were presented and published in the symposium proceedings. One article has been accepted for publication in a technical journal.
- Eleven abstracts were accepted and presented in numerous conferences.
- Research findings were presented at two international research laboratories.
- Ramprashad Prabhakaran received three scholarships to attend and present papers at international conferences: US DOE scholarship (IYNC 2004, Toronto), ASME scholarship (ICONE12 2004, Arlington) and ORAU scholarship (SNS-JINS-NICEST 2003, Oak Ridge).
- Mohammad Kamal Hossain received outstanding paper award in the ANS Student Conference 2003, Berkeley, CA.

## Publications

- "Environment-Assisted Cracking of Structural Materials under Different Loading Conditions," NACE Corrosion Journal. (In Preparation)
- "Role of Applied Potentials on Cracking of Martensitic Stainless Steels," Electrochemical Society Journal. (In Preparation)
- "Environment-Induced Embrittlement of Martensitic Stainless Steel for Transmutation Applications," Corrosion/2005 Conference, April 3-7, 2005, Houston, Texas. (In Preparation)
- "Stress Corrosion Cracking Evaluation of Target Structural Materials by Different Techniques," Environment-Induced Cracking of Metals Conference, September 19-23, 2004, Alberta, Canada. (In Preparation).
- "Stress Corrosion Cracking of Type 422 Stainless Steel for Transmutation Applications," IYNC 2004, Paper No. 119, May 9-13, 2004, Toronto, Canada.
- "The Effect of Environmental and Mechanical Variables on Stress Corrosion Cracking of a Martensitic Stainless Steel for Transmutation Applications," ASME ICONE12, Paper No. 49399, April 25-29, 2004, Arlington, VA.
- "Environmental Effects on a Candidate Structural Material for Transmutation Application," ANS Student Conference 2004, April 1-3, 2004, Madison, WI.
- "Cracking of Target Structural Material in Different Environments," ANS Student Conference 2004, April 1-3, 2004, Madison, WI.
- "Stress Corrosion Cracking of Structural Target Material Alloy EP-823," ANS Student Conference 2004, April 1-3, 2004, Madison, WI.
- "Stress Corrosion Cracking and Localized Corrosion in Alloy HT-9", ANS Student Conference 2004, April 1-3, 2004, Madison, WI.
- "Cracking of Target Structural Materials for Transmutation Applications," NACE Corrosion 2004, Paper No. 4559, March 28-April 1, 2004, New Orleans, LA.
- "Characterization of Environment-Induced Degradation in Type 422 Stainless Steel," TMS 2004, March 14-18, 2004, Charlotte, NC.
- "Effect of Environmental Variables on Cracking of Martensitic Stainless Steels Under Different Loading Conditions," ANS Global 2003, Paper No. 87869, November 16-20, 2003, New Orleans, LA.
- "Environmental Effects on Materials For Nuclear Applications," MS&T 2003, November 9-12, 2003, Chicago, IL.
- "Cracking of Martensitic Stainless Steels Under Applied Electrochemical Potential," 204th Meeting of ECS, Abstract No. 1255, October 12-17, 2003, Orlando, FL.
- "Environment-Induced Degradation of Spallation Target Materials," ANS AccApp'03, Paper No. 79416, June 1-5, 2003, San Diego, CA.
- "Effects of Environmental Variables and Stress Concentration on Cracking of Spallation Target Materials," 203rd Meeting of ECS, Abstract No. 1283, April 27-May 2, 2003, Paris, France.
- "Stress Corrosion Cracking of Type 422 Stainless Steel," ANS Student Conference, April 2-5, 2003, Berkeley, CA.
- "Effects of Environmental Variables and Stress Concentration on Target Materials," ANS Student Conference, April 2-5, Berkeley, CA.
- "Stress Corrosion Cracking of Target Materials," ANS Student Conference, April 2-5,

Berkeley, CA.

- "Stress Corrosion Cracking of Martensitic Stainless Steel for Transmutation Applications," 10th IHLRWM Conference, March 30-April 3, 2003 Las Vegas, NV.
- "Stress Corrosion Cracking of Type 422 Stainless Steel for Applications in Spallation-Neutron-Target Systems," SNS-JINS-NICEST, March 12, 2003, Oak Ridge, TN.
- "Stress Corrosion Cracking of Transmutation Structural Materials in Aqueous Media," NACE *Materials Performance Journal* (In Press)

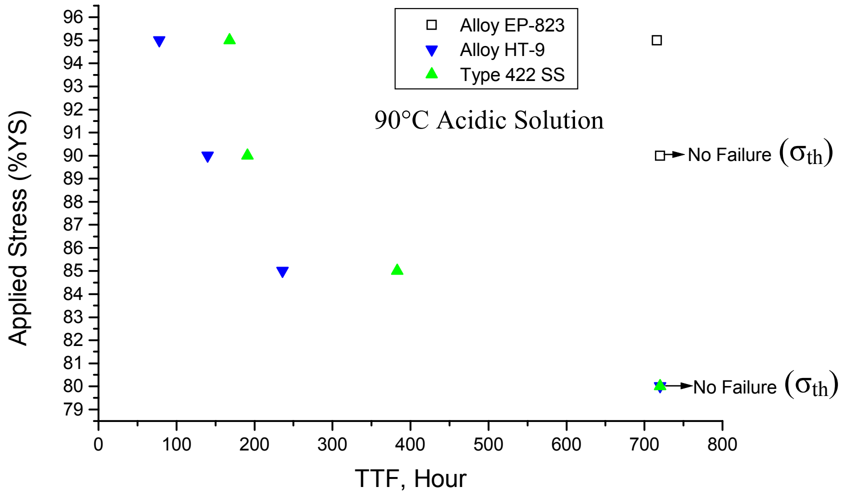


Figure 1: Applied Stress versus Time-To-Failure

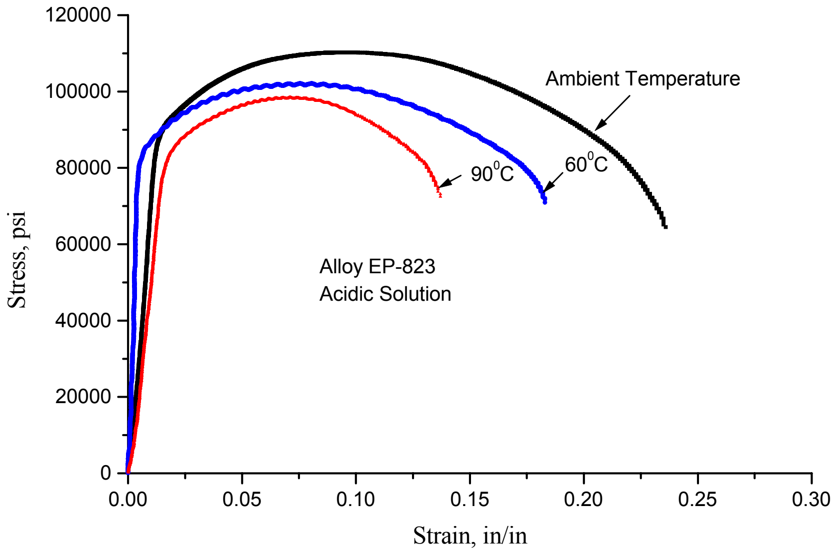


Figure 2: Typical Stress versus Strain Curve

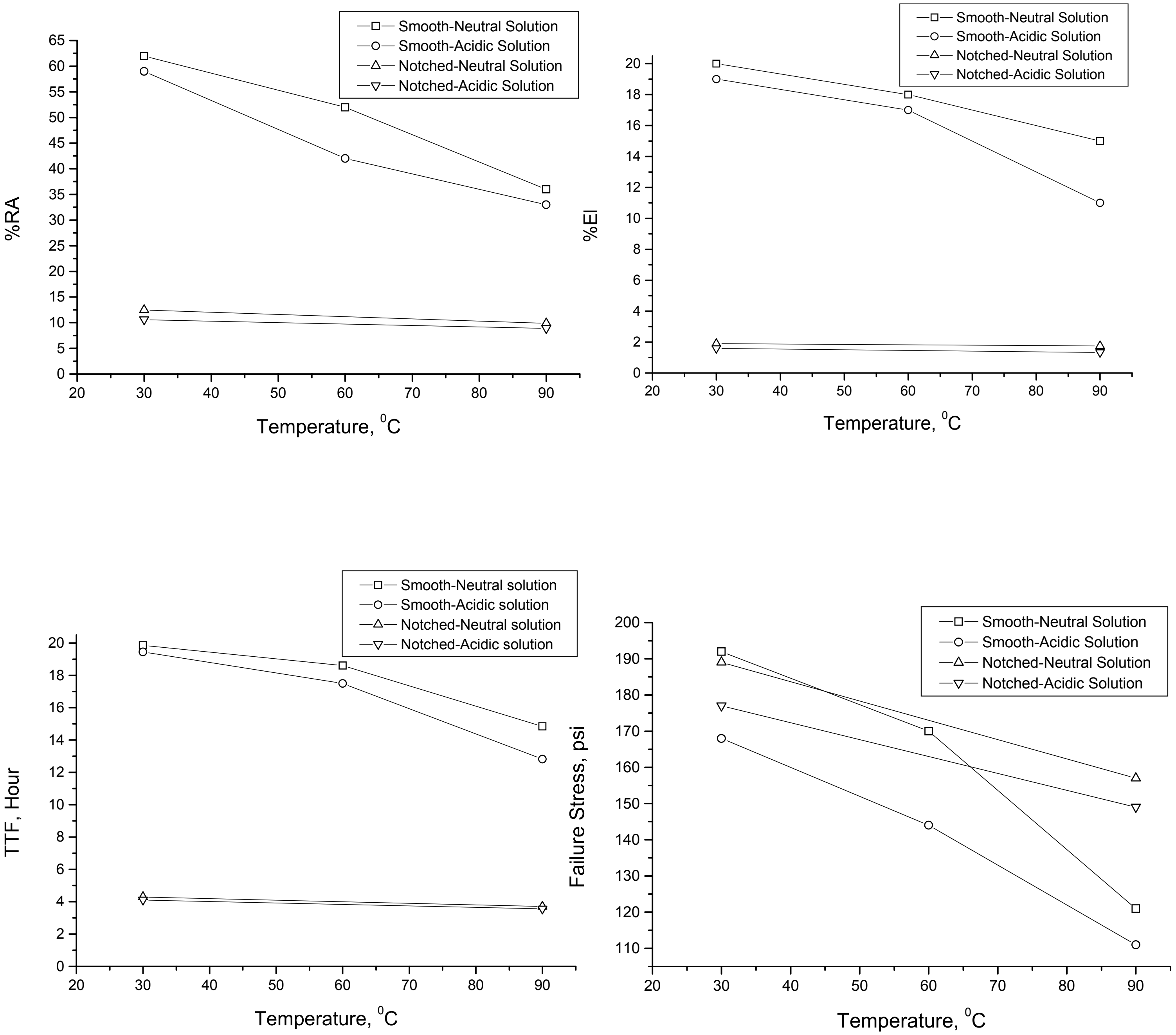


Figure 3: Effects of pH, Temperature and Notch on %RA, %EL, $\sigma_f$ and TTF for Alloy EP-823

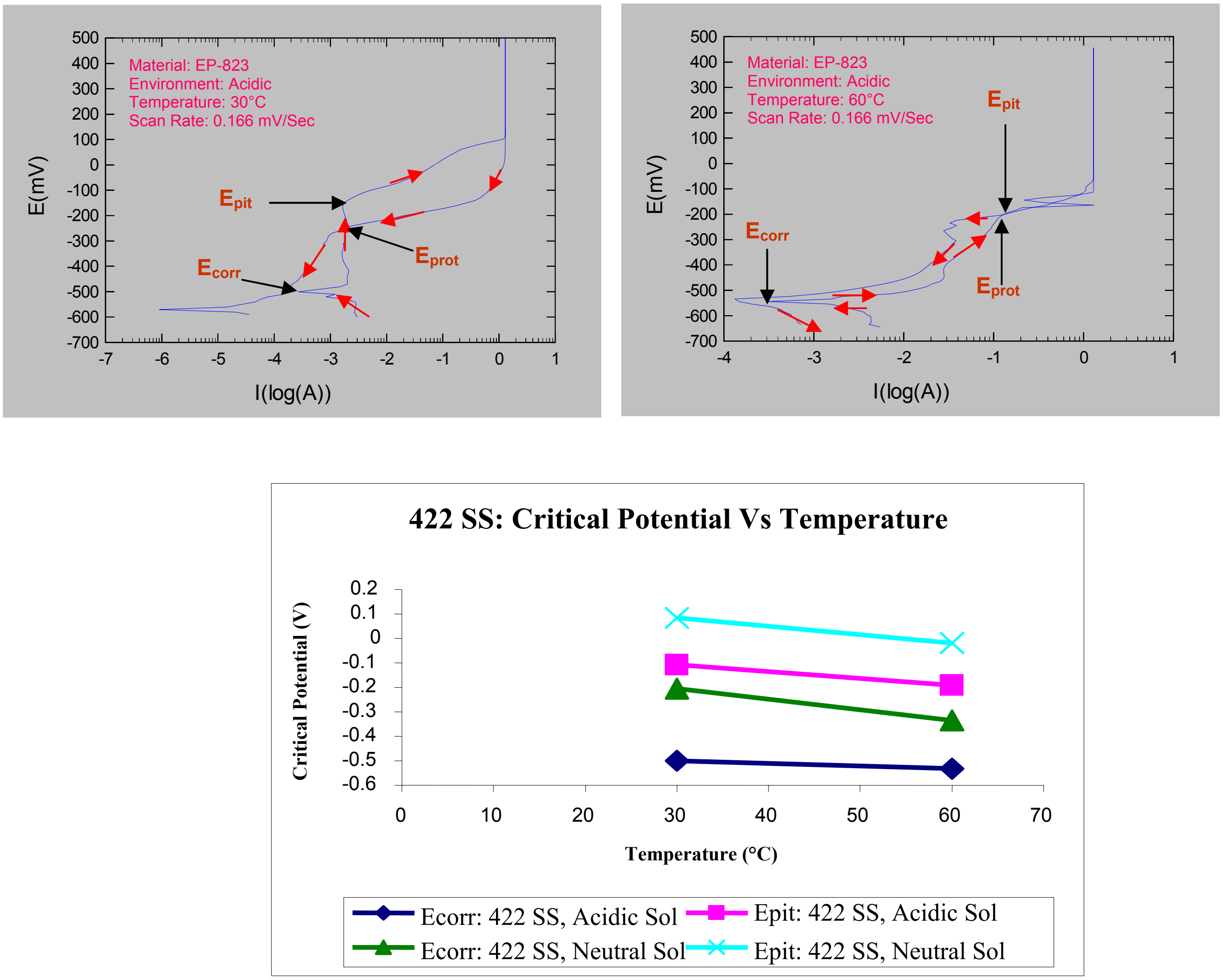
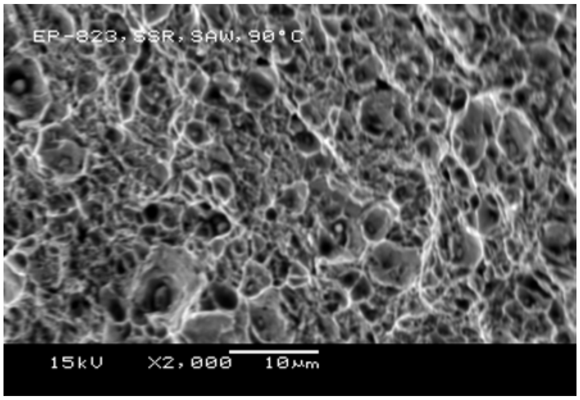
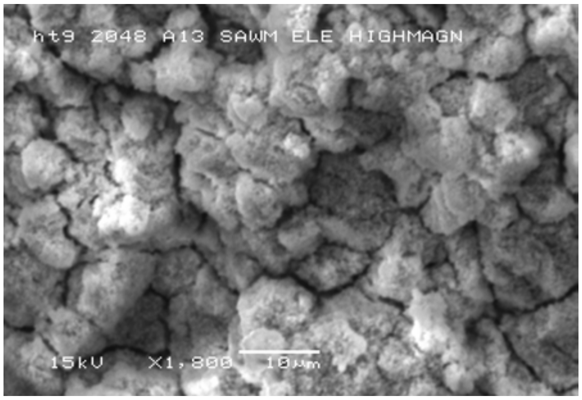


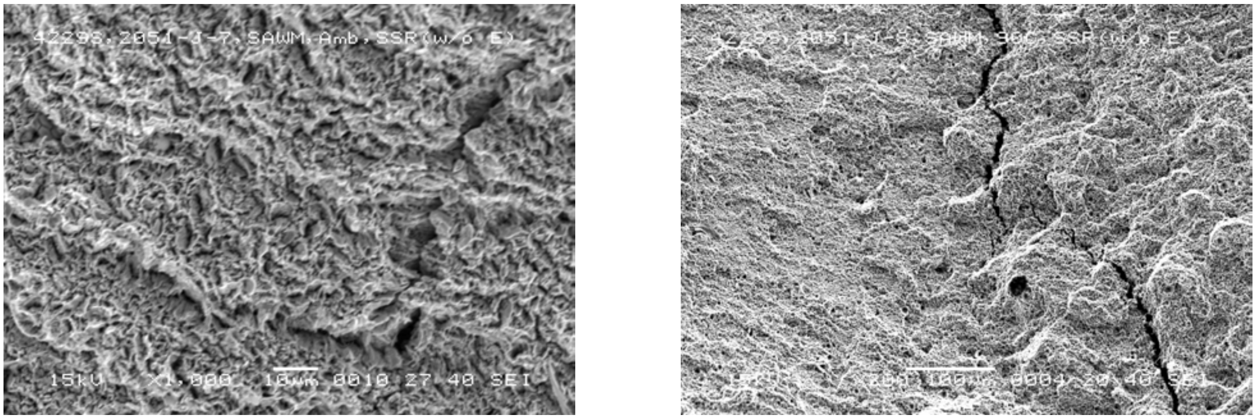
Figure 4: CPP Data



5A. Alloy EP-823 tested in 90°C Neutral Environment
(Ductile Failure)



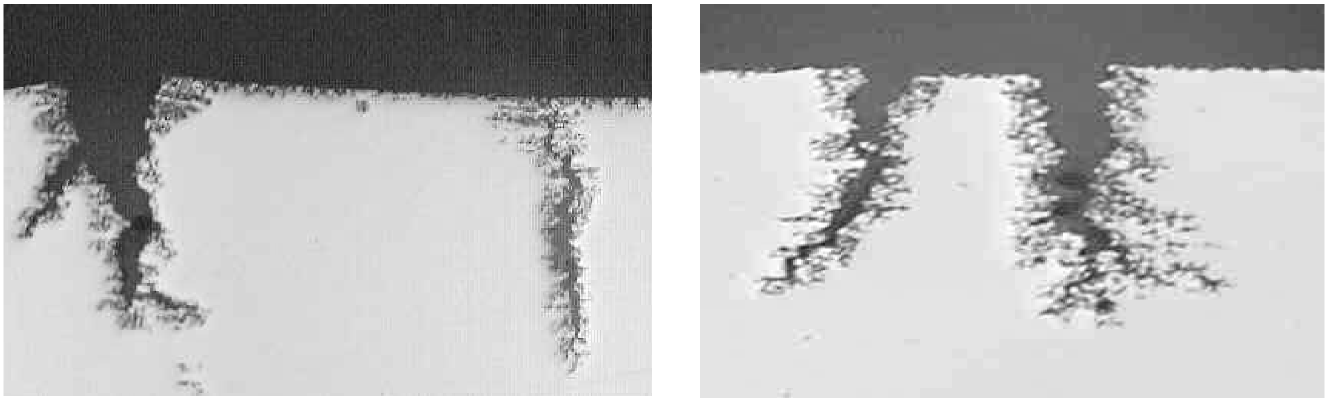
5B. Alloy HT-9 tested in 90°C Acidic Environment
(Intergranular Failure)



5C. Type 422 SS tested in 30°C Acidic Environment
(Intergranular/Transgranular Failure)

5D. Type 422 SS tested in 90°C Acidic Environment
(Transgranular Failure)

Figure 5: SEM Micrographs of Cracked Specimens



5A. Alloy EP-823 (40X, as polished)

5B. Type 422 SS (40X, as polished)

Figure 6: Optical Micrographs in 90°C Acidic Environment Showing Branched Secondary Cracks