Stress corrosion cracking and hydrogen embrittlement of martensitic Alloy Ep -823

Mohammad Kamal Hossain

University of Nevada, Las Vegas

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UMI®
STRESS CORROSION CRACKING AND HYDROGEN EMBRITTLEMENT OF
MARTENSITIC ALLOY EP-823

by

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of the requirements for the

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The Graduate College
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Mohammad Kamal Hossain

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Doctor of Philosophy in Mechanical Engineering

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ABSTRACT

Stress Corrosion Cracking and Hydrogen Embrittlement of Martensitic Alloy EP-823

by

Mohammad Kamal Hossain

Dr. Ajit K. Roy, Examination Committee Chair
Associate Professor of Mechanical Engineering
University of Nevada, Las Vegas

This investigation is focused on the evaluation of stress corrosion cracking (SCC), localized corrosion, and hydrogen embrittlement (HE) susceptibility of martensitic Alloy EP-823 in neutral and acidic solutions at ambient and elevated temperatures. While no failures were observed in smooth specimens in the neutral solution, failures were noticed in the 90°C acidic solution at constant-load (CL) leading to a threshold stress (σth) of 102 ksi. The presence of a notch reduced the σth value to 91 ksi in a similar environment. The ductility (%El and %RA), time-to-failure (TTF), and true failure stress (σf) were gradually reduced in both environments with increasing temperature during SSR testing. The presence of a notch, however, enhanced the σf value. All polarized specimens exhibited localized attack. The application of a controlled cathodic potential further reduced the ductility, TTF and σf showing the characteristics of more brittle failures. Metallographic and fractographic evaluations revealed secondary cracks along the gage section, and combination of ductile and brittle failures at the primary fracture face, respectively.
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CHAPTER 1

INTRODUCTION

A major requirement for sustaining human progress is an adequate source of energy and, at present, nuclear energy is playing a vital role on that issue. Currently, nuclear technology is being used in many applications including medicine, industry, consumer products, scientific research, agriculture, energy, environmental protection, law enforcement and public safety. In essence, with increased demand of energy, nuclear power has become a potential source of energy worldwide. Currently, over 20% of the electricity generated in the United States (U.S.) is provided by approximately 100 nuclear power reactors operating in this country. It is estimated that the amount of used nuclear fuel in the U.S. will reach 140,000 tons by the end of the operational period of current reactors.\(^1\) Even though nuclear technology offers several benefits, the disposal of radioactive waste arising from nuclear-power production or from the dismantling of nuclear weapons poses a severe challenge to many nations employing this technology.\(^2,3\)

Significant efforts are in progress, nationally and internationally, to dispose of the spent nuclear fuel (SNF) and high-level radioactive waste (HLW) generated from the nuclear plants, research reactors and government facilities. Countries like France, United Kingdom, and Japan are reprocessing the SNF to extract uranium and plutonium for further use. The U. S. Department of Energy (DOE) has recently proposed the Yucca Mountain site, near Las Vegas, Nevada to be the nation’s first geologic repository to
contain SNF/HLW without reprocessing. Initially 70,000 metric tons of nuclear wastes were designed to be disposed of in this repository. However, the amount of nuclear waste is expected to gradually increase in the U.S. due to the enhanced consumption of nuclear fuel for power generation, thus, necessitating the development of additional repositories.

Worldwide, more than 250,000 tons of SNF from currently operating reactors will require disposal. These numbers account for only HLW generated by the present-day power reactors. Rather conservative projections of nuclear power growth worldwide in the coming decades indicate that, by the year 2050, almost 1 million tons of discharged fuel requiring disposal could exist. Such projections would indicate the need to build and commission a repository of the scale of the Yucca Mountain disposal site somewhere in the world roughly every three to four years.

SNF contains several unstable nuclei in sufficient quantities to render them quite radioactive for a prolonged period of time. Risks to future generations arising from long-term disposal of SNF are attributable to ~1% of its content. This 1% is primarily made up of plutonium, neptunium, americium, and curium (called transuranic elements) and long-lived isotopes of iodine and technetium created as products from the fission process in power reactors. The disposal for SNF carries an obligation to isolate these materials from the environment for a period of 10,000 years, as proposed by the DOE. This unusually long duration challenges man’s abilities to engineer long-lasting containers and barriers, and therefore, force some reliance on the prediction of geologic behavior to ensure the desired isolation from the natural environment. In order to circumvent the drawback associated with the long-term disposal of future nuclear waste,
DOE is considering alternative methods to reduce the radioactivity of SNF/HLW prior to their disposal in the proposed repository.

One such method is transmutation of minor actinides and fission products from SNF/HLW to reduce their half-lives. Transmutation is currently being used in Europe, particularly in Russia, Sweden, Switzerland and France. During this past decade, these countries have made significant progress in partitioning and transmuting the long-lived actinides from SNF. Due to these advances, the U.S. national laboratories have begun to explore the transmutation concept as an alternative waste management strategy. The transmutation of SNF, shown in Figure 1.1, would provide the benefit of disposing of substantially less radioactive waste inside the proposed geologic repository for a shorter duration.
Transmutation is a process that transmutes the long-life radioactive species to short-life radioactive species and fission products from the SNF with eventual reduction of its radioactivity.\(^{(10)}\) This process involves separating actinides and fission products and then bombarding the long-lived radioactive isotopes with neutrons. The neutrons are produced either by projecting protons generated from an accelerator onto a target material such as lead-bismuth-eutectic (LBE) containing approximately 55 weight percent (wt %) of Bismuth or a nuclear reactor. The molten LBE, which can also act as a coolant, will be contained inside a vessel made of corrosion resistance structural material such as Alloy...
EP-823. The transmutation of fission products and actinides from SNF/HLW by use of neutrons is illustrated in Figure 1.2.

The target structural material such as martensitic Alloy EP-823 will be subjected to high heat and stresses during the transmutation process while in contact with the molten LBE. Further, during the spallation process using neutrons, an abundant amount of hydrogen and helium can be produced, which may cause environment-assisted degradations such as liquid-metal-embrittlement (LME), stress corrosion cracking (SCC),
hydrogen embrittlement (HE), and localized corrosion such as pitting and crevice production.\(^{11-13}\) The temperature encountered during the transmutation process will range between 420 and 550°C.\(^{14}\)

In view of the likelihood of different types of degradations to be experienced by the prospective target structural material, an extensive research program was undertaken at UNLV in collaboration with the Los Alamos National Laboratory (LANL) to address the relevant modes of degradations. Since the molten LBE can cause liquid metal corrosion and embrittlement in target structural material \(^{15}\), it was decided to conduct SCC testing at LANL using self-loaded C-ring and U-bend specimens using its LBE loop. LME can manifest itself in different ways including dissolution, compound formation, and liquid penetration at grain boundaries depending on factors such as exposure time, temperature, thermal gradient, flow velocity, compositions of solid and liquid species.\(^{16}\) While several studies on LME of steel in the presence of molten lead or LBE have been reported in the open literature,\(^{17-25}\) SCC testing could not be accommodated at LANL due to some administrative and scheduling difficulties.

Simultaneously, significant efforts were made at UNLV to evaluate numerous environment-assisted degradation of Alloy EP-823 in aqueous environments of different pH values at ambient and elevated temperatures using numerous state-of-the-art experimental techniques. These tests include SCC, HE, and localized corrosion evaluation. SCC is an environment-assisted cracking of metallic materials resulting from the combined effect of a corrosive environment and a tensile stress. The stress may result from applied forces or locked-in residual stresses. Only specific combinations of alloy and chemical environment can lead to SCC. Usually, SCC begins with the rupturing of
the protective oxide film on the metal surface by either mechanical means or by the action of chemical species, such as chloride ion (Cl⁻). The cracks resulting from SCC may be either ductile or brittle in nature or a combination of both. Cracking may be intergranular, transgranular, or a mixed mode depending on the alloy, its microstructure, and the environment.

As stated earlier, hydrogen and helium produced during nuclear reactions can segregate to vacancy clusters and internal voids, thus eventually leading to HE in the target structural material. The HE process may depend on the origin of the hydrogen and the transport processes involved in moving the hydrogen from its source to the locations where it reacts with the metal to cause embrittlement. Body-centered cubic (BCC) metals are the most susceptible to HE. The primary characteristics of HE are its strain-rate sensitivity, its temperature dependence and its susceptibility to delayed fracture.

Localized corrosion is a degradation mode in which an intense attack takes place at localized sites on the surface of the material while the rest corrodes at a lower rate either because of an inherent property of the component material, such as the formation of a protective oxide film or because of some environmental effect. Indeed, the primary surface may be essentially under satisfactory corrosion control. In such circumstances, if corrosion protection breaks down locally, then corrosion may be initiated at these local sites.

This investigation is focused on the evaluation of SCC, HE, and localized corrosion behavior of martensitic Alloy EP-823 in neutral and acidic aqueous environments at ambient and elevated temperatures. State-of-the-art testing techniques including constant-
load (CL), slow-strain-rate (SSR) and electrochemical polarization have been employed to evaluate these corrosion phenomena. Also, the susceptibility of Alloy EP-823 to HE has been evaluated by applying cathodic potential while the specimen was subjected to tensile loading by the SSR testing technique. Optical microscopy and scanning electron microscopy (SEM) have also been used to analyze the metallurgical microstructures and fractography, respectively, of the tested specimens.

In essence, this dissertation presents the results of SCC, HE, and localized corrosion studies of Alloy EP-823 in aqueous environments that may constitute significant baseline data on the performance of this alloy as a function of different environmental and mechanical variables including temperature, pH, and hydrogen ion (H⁺) concentration, controlled electrochemical potential and the loading mode. The fact that the structural material would be in contact with a molten metal during the transmutation process, clearly warrants that the generated data in the aqueous solutions be compared to those intended to have been developed at LANL. However, due to the lack of corrosion data from LANL, it is practically impossible to make a viable comparison of the generated data to those in the presence of a molten metal. It is well known that mechanistically a susceptible material may undergo embrittlement in a molten metal primarily due to the reduction in cohesive strength among its surface atoms due to their reaction with the molten metal. On the contrary, degradations experienced by the same material in aqueous environments is the results of ionic exchange involving both anodic and cathodic reactions.

In light of the preceding discussion, it would have been worthwhile to compare the surface characteristics of the tested materials exposed to both types of environments.
Therefore, it is proposed that a future effort be focused to develop an experimental facility at UNLV to perform SCC testing involving self-loaded specimens such as C-ring and U-bend in the presence of molten LBE. It is possible that the most effective structural material could then be identified and selected based on the analyses of the comprehensive corrosion data reported in this dissertation and future data yet to be generated at the proposed LBE testing facility at UNLV.
The test matrix is summarized in Table 1.1 for this investigation.

Table 1.1 Test Matrix

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<th>Condition</th>
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<td>Acidic Solution (Avg. pH: 2.35), Ambient to Elevated Temperature, and -1000 mV (Ag/AgCl) Controlled Applied Potential ($E_{cont}$)</td>
<td>True Failure Stress ($\sigma_t$), Time-to-Failure (TTF), Percent Elongation (%El), and Percent Reduction in Area (%RA)</td>
</tr>
<tr>
<td>Optical Microscopy</td>
<td>Polished and/or Etched Gage Section of the Tested Specimen in SSR Condition with/without $E_{cont}$</td>
<td>Secondary Cracks</td>
</tr>
<tr>
<td>Scanning Electron Microscopy (SEM)</td>
<td>Primary Fracture Face of the Tested Specimen in SSR Condition with/without $E_{cont}$</td>
<td>Ductile versus Brittle Failure</td>
</tr>
</tbody>
</table>
CHAPTER 2

TEST MATERIALS, SPECIMENS, AND ENVIRONMENTS

2.1. Test Materials

Martensitic stainless steels were the first stainless steels grades that were commercially developed having a relatively high carbon (C) content (0.1-1.2%) compared to that in other types of stainless steels. These are plain chromium (Cr) steels containing between 12 and 18 percent Cr. These steels are magnetic and hardenable by quenching and tempering like plain carbon steels, and find their applications in cutlery, aerospace and general engineering. The martensitic stainless steels are currently finding extensive applications in nuclear reactors as substitutes for austenitic stainless steels.

Martensitic steels were introduced into the fusion materials programs about 25 years ago after research in fast reactor programs demonstrated their superior swelling resistance and excellent thermal properties compared to those in austenitic stainless steels. The materials initially considered were conventional chromium-molybdenum (Cr-Mo) steels containing 9 to 12% Cr by weight. The development of reduced-activation steels began less than 20 years ago, which are presently being considered to be the primary structural materials for demonstration fusion plants and the fast fusion power reactors.

The material tested in this investigation is a Russian-grade martensitic stainless steel (SS), known as Alloy EP-823. This alloy has been extensively used as a structural
material to contain the molten lead-bismuth-eutectic (LBE) in Europe and Russia, which may also act as a target onto which proton beams generated from an accelerator can be impinged to produce neutrons, as indicated earlier. Since significant amount of heat can be generated during the transmutation process, the molten LBE can also act as a coolant.

Martensitic stainless steels are known for their high tensile strength, moderate creep and fatigue properties, in combination with moderate corrosion and heat resistance. A comparison of the stress-strain diagrams for different types of SS are presented in Figure 2.1, showing relatively higher strength in martensitic SS, compared to those in other types of SS. The effect of temperature on the yield strength (YS) and creep strength (CS) for different types of SS is shown in Figure 2.2. These data indicate that the martensitic stainless steels can retain their high YS and CS even at elevated temperatures.\(^\text{(35)}\)

![Stress-Strain Diagrams for Different Types of SS at 25°C](http://www.outokumpu.com/pages/Page_5832.aspx)

Figure 2.1 Stress-Strain Diagrams for Different Types of SS at 25°C \(^\text{(35)}\)

(Source: Mechanical Properties of Stainless Steel, Outokumpu Stainless Website: http://www.outokumpu.com/pages/Page_5832.aspx)
Martensitic stainless steels containing Cr and Mo can possess a body-centered-cubic (BCC) or body-centered-tetragonal (BCT) crystal structure in the hardened (martensitic) condition. The medium carbon content in the martensitic stainless steels enables the development of the desired metallurgical microstructures and properties resulting from hardening and tempering. The Cr and C contents are balanced to ensure a fully-tempered martensitic microstructure following hardening and subsequent tempering. The presence of Mo can enhance the mechanical properties and the localized corrosion resistance. When higher Cr levels are used to improve the corrosion resistance, the presence of Ni
can also help to maintain the desired microstructure and prevent the formation of excessive free-ferrite.\(^{36}\)

Alloy EP-823, containing a relatively high silicon (Si) level, was developed in Russia to provide adequate corrosion resistance in the oxygen-containing LBE coolant. This alloy possesses significant resistance to swelling during high neutron exposure at temperatures up to 420\(^\circ\)C, low rate of irradiation creep and rather low activation compared to that of the austenitic stainless steels.\(^{37,38}\) This alloy has also been reported to retain its high strength and ductility at elevated temperatures in irradiated conditions.\(^{39}\)

Experimental heats of Alloy EP-823 were melted at the Timken Research Laboratory, Canton, Ohio, by vacuum induction melting practice followed by processes that included forging and hot rolling. The hot rolled products were subsequently cold rolled to produce round bars of different sizes. Since fine-grained and fully-tempered martensitic microstructures were desired in these materials, the cold-rolled bars were initially austenitized at 1010\(^\circ\)C (1850\(^\circ\)F) followed by an oil quench. Hard but brittle martensitic microstructures were developed in these bars due to austenitizing and quenching. Therefore, subsequently, tempering treatments were done at 621\(^\circ\)C (1150\(^\circ\)F) for two different tempering time periods of 1.25 and 1.75 hours to produce fully-tempered martensitic microstructure without formation of any retained austenite, thus producing the desired ductility. The purpose of using different tempering times was to study their effect on the resultant metallurgical properties including microstructures and the tensile properties. The chemical compositions of two different heats of Alloy EP-823 tested in this investigation are shown in Table 2.1.
Table 2.1 Chemical Compositions of Materials Tested (wt %)

<table>
<thead>
<tr>
<th>Material/Heat Number</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>V</th>
<th>W</th>
<th>Cb</th>
<th>B</th>
<th>Ce</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>EP-823/2054</td>
<td>0.16</td>
<td>0.55</td>
<td>0.014</td>
<td>0.004</td>
<td>1.09</td>
<td>11.70</td>
<td>0.66</td>
<td>0.74</td>
<td>0.002</td>
<td>0.30</td>
<td>0.60</td>
<td>0.24</td>
<td>0.009</td>
<td>0.04</td>
<td>Bal</td>
</tr>
<tr>
<td>EP-823/2055</td>
<td>0.16</td>
<td>0.57</td>
<td>0.014</td>
<td>0.004</td>
<td>1.14</td>
<td>11.71</td>
<td>0.67</td>
<td>0.74</td>
<td>0.002</td>
<td>0.31</td>
<td>0.58</td>
<td>0.23</td>
<td>0.009</td>
<td>0.04</td>
<td>Bal</td>
</tr>
</tbody>
</table>

Bal: Balance

2.2. Test Specimens

Smooth cylindrical specimens (4-inch overall length, 1-inch gage length and 0.25-inch gage diameter) were machined from the quenched and tempered round bars of Alloy EP-823 in such a way that the gage section was parallel to the longitudinal rolling direction. The gage length to the diameter (l/d) ratio of these specimens was maintained at 4 according to the ASTM Designation E 8-04. The configuration and the dimensions of the smooth specimens used in tensile testing are shown in Figure 2.3.
Surface imperfections such as flaw, notch or dent may exist in the machined specimens that can influence the resultant tensile data. In view of this rationale, a V-shaped notch of 0.156-inch diameter, with an angle of 60° and a maximum root radius of 0.05 inch around the diameter was added to the center of the gage section of the cylindrical specimen to study the effect of stress concentration on the tensile properties. The configuration and the dimensions of the notched cylindrical specimens used in this investigation are shown in Figure 2.4.
The stress concentration factor ($K_I$) resulting from this notch was approximately 1.45.

The magnitude of $K_I$ was estimated by calculating the values of $D/d$ and $r/d$ of the notched specimen, as shown below. The plots $^{41,42}$ of $K_I$ versus $r/d$ (Figure 2.5), and the following calculations were used to estimate the $K_I$ value.

Figure 2.4 Notched Cylindrical Specimen
\[ \frac{D}{d} = \frac{0.250 \text{ in}}{0.156 \text{ in}} \quad \text{(Equation 3.1)} \]

\[ \frac{D}{d} = 1.60 \]

\[ \frac{r}{d} = \frac{0.05 \text{ in}}{0.156 \text{ in}} \quad \text{(Equation 3.2)} \]

\[ \frac{r}{d} = 0.32 \]

Where,

\[ D = \text{Gage Diameter of the Specimen} \]

\[ d = \text{Notch Diameter of the Specimen} \]

\[ r = \text{Radius of Curvature at the Root of the Notch} \]

Cylindrical specimens having length and diameter of 0.50 and 0.37 in, respectively, with a central blind hole parallel to the cylindrical axis was used for electrochemical
polarization experiments to evaluate the localized corrosion behavior according to the ASTM Designation G 5.\(^{(43,44)}\) The configuration and dimensions of this type of specimen are shown in Figure 2.6.

![Figure 2.6 Electrochemical Polarization Specimen](image)

2.3. Test Environments

The quenched and tempered martensitic stainless steels are usually resistant to atmospheric and aqueous corrosion. However, their corrosion resistance may be adversely affected by exposure to aggressive environments such as seawater.\(^{(45)}\) As
indicated earlier, Alloy EP-823 will be subjected to a molten LBE environment during the transmutation process. Thus, this alloy may become susceptible to liquid-metal corrosion and embrittlement. Very little data exist in the open literature as to the corrosion behavior of this alloy in molten metals. Therefore, experimental work was planned to be performed at LANL involving self-loaded specimens of Alloy EP-823 using its LBE loop. However, due to some administrative and scheduling difficulties, this testing could not be performed.

Since corrosion studies in the presence of a molten LBE environment could not be accommodated at UNLV, numerous types of corrosion experiments were performed at the Materials Performance Laboratory (MPL) of UNLV using different state-of-the-art testing techniques in the presence of aqueous environments of different pH values at ambient and elevated temperatures (60 and 90°C) to establish baseline corrosion data.

The effects of environmental and mechanical variables on the susceptibility of Alloy EP-823 to SCC, HE and localized corrosion have been evaluated in this investigation. Both neutral (average pH: 6.23) and acidic (average pH: 2.35) aqueous solutions were used to evaluate the SCC, HE, and localized corrosion (pitting and crevice production) behavior of this material. The purpose of using neutral versus acidic solution was to evaluate the effect of pH on the corrosion susceptibility of this alloy. It is well known that increased hydrogen ion (H+) concentration can enhance the degradation of a susceptible material exposed to an aqueous solution. The H+ concentration is significantly higher in the acidic solution, thus resulting in a lower pH and subsequently causing enhanced corrosive attack. The compositions of the testing environments are given in Table 2.2.
Table 2.2 Chemical Compositions of Tested Solutions (gm/liter)

<table>
<thead>
<tr>
<th>Environment (pH)</th>
<th>CaCl₂</th>
<th>K₂SO₄</th>
<th>MgSO₄</th>
<th>NaCl</th>
<th>NaNO₃</th>
<th>Na₂SO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neutral Solution (Avg. pH: 6.23)</td>
<td>2.77</td>
<td>7.58</td>
<td>4.95</td>
<td>39.97</td>
<td>31.53</td>
<td>56.74</td>
</tr>
<tr>
<td>Acidic Solution (Avg. pH: 2.35)</td>
<td>Same as above except for an addition of HCl to adjust the pH to the desired range</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
CHAPTER 3

EXPERIMENTAL PROCEDURES

As mentioned earlier, the purpose of this investigation is to evaluate the SCC, HE, and localized corrosion behavior of martensitic Alloy EP-823 as a structural material for transmutation applications. SCC is a cracking phenomena resulting from the combined effect of tensile stress and a corrosive environment. The required tensile stresses may be in the form of directly applied stresses or residual stresses. On the other hand, hydrogen in an atomic form can enter susceptible materials during steel making, welding, corrosion reactions, and cathodic protection. Presence of hydrogen in metallic materials can cause embrittlement leading to the reduction in ductility, and brittle fracture.

Localized corrosion is an intense attack at localized sites on the surface of a component while the rest of the surface corrodes at a much lower rate, either because of an inherent property of the component material such as the formation of a protective oxide film or because of some environmental effect. Thus, it is appropriate to evaluate the SCC, HE, and localized corrosion susceptibility of Alloy EP-823 in hostile environments using different state-of-the-art experimental techniques.

Alloy EP-823 will be in contact with molten LBE during the transmutation process. Molten LBE has a tendency to corrode most engineering materials in the form of liquid-metal corrosion and embrittlement.\(^{13,15}\) Significant amount of stress, heat, hydrogen and helium can be generated during transmutation and hence, the structural material may be
subjected to various environment-induced degradations including SCC, HE, and localized corrosion in susceptible environments.\(^{(13)}\)

Very limited amount of research work has so far been performed in the U.S. to characterize corrosion behavior of Alloy EP-823 subjected to a molten LBE environment. The planned SCC testing involving self-loaded test specimens of this alloy could not be performed at LANL due to some administrative and scheduling difficulties. Accordingly, an ambitious research program was undertaken at UNLV using its state-of-the-art MPL to evaluate both the metallurgical characteristics and the corrosion behavior of Alloy EP-823 in aqueous environments. Constant-load (CL) and slow-strain-rate (SSR) testing techniques were used to determine the susceptibility of this alloy to SCC in the presence of neutral and acidic solutions at room temperature (RT), 60 and 90°C. The effect of stress concentration on the cracking tendency of this material was also investigated. Further, the effect of hydrogen on the cracking susceptibility was evaluated by applying cathodic (negative) electrochemical potential to the tensile specimens by SSR technique.

Prior to the SCC testing, the hardness, the metallurgical microstructural evaluation, and the room-temperature tensile properties of the test materials were determined by using a rockwell hardness testing machine, optical microscopy and a mechanical testing system (MTS), respectively. The susceptibility of this alloy to localized corrosion was determined in similar environments by the cyclic potentiodynamic polarization (CPP) testing technique. The fractographic and metallographic evaluations of all tested specimens were performed by scanning electron microscopy (SEM) and optical microscopy, respectively.
3.1 Hardness Measurement

Upon completion of thermal treatments, three pieces of material were sectioned from each heat treated round bar followed by polishing to achieve smooth surface finish. Two lines were drawn along the diameter at an angle of 90° to each other on each section of the heat treated material. Three indentations were made by using a rockwell hardness testing machine in C scale along these two lines at 3 locations namely the center and one on each side away from the center. An average hardness value in rockwell C scale (Rc) based on three measurements at each location was used to characterize the hardness value under different thermal treatment conditions.

3.2 Mechanical Properties

The tensile properties such as the ultimate tensile strength (UTS), the yield strength (YS) and the ductility parameters such as the percentage elongation (%El) and the percentage reduction in area (%RA) of Alloy EP-823 were evaluated at ambient temperature using an axial/torsional servohydraulic MTS machine. The smooth and notched cylindrical specimens were pulled at a strain rate of $10^3$/sec according to the ASTM Designation E 8-04. A minimum of two specimens were tested at each experimental condition and an average value was determined. The experimental data such as the load, time, and extensometer reading were recorded in the data file at the rate of 100/sec. The engineering stress versus engineering strain curves were developed using these data. The magnitude of the YS was determined by the point of intersection of a line drawn parallel to the linear portion of this curve at a strain offset value of 0.2% of strain. UTS, %El and %RA were also determined from these plots and the dimensions of the cylindrical specimen before and after testing.
The MTS unit model 319.25, shown in Figure 3.1, had an axial load transducer of 55 kip (250 kN) and a torsional load transducer of 20,000 lbf-in (2200 N-m) capacity. It had a hydraulically-controlled actuator with 5.5” stroke and approximately 55° angular rotation. It consisted of a large heavy-duty load frame with an adjustable crosshead attached to the wedge grip at the top, and a movable actuator with another wedge grip at the bottom to enable loading and unloading of the test specimen. The axial motion was controlled by force, displacement, or an external signal from the strain gage. The torsional motion was controlled by torque, angular position, or an external signal from the strain gage. The specimen was mounted between two wedge grips and was pulled by the movable actuator. The load cell, contained in the crosshead, measured the applied force on the tensile specimen. The movement of the crosshead relative to the locked crosshead generated the strain within the specimen and consequently, the corresponding load. The MTS machine had a capability of evaluating the tensile properties of materials of interest at temperatures ranging from ambient to 600°C.
3.3 Stress Corrosion Cracking

The SCC evaluation of a material involves the use of a specimen type and testing technique that can take both metallurgical and environmental factors into consideration. The susceptibility of Alloy EP-823 to SCC was determined in this investigation by using both CL and SSR testing techniques. A minimum of two tests was performed under each experimental condition. The experimental details for both types of testing are given below.
3.3.1. Constant-Load Testing

One of the most common and basic methods utilized in the SCC tests is the use of a constant applied tensile load that can act as a driving force for SCC to occur. Both smooth and notched cylindrical specimens were used to load them at various levels of applied stress/load according to the ASTM Designation G-49.\textsuperscript{48,49}

A calibrated proof ring was used for the CL testing. Proof rings fabricated by Cortest Inc. from precision-machined alloy steel were designed to precisely determine the SCC susceptibility of the test material to meet the requirements of the National Association of Corrosion Engineers (NACE) Standards.\textsuperscript{50} The SCC test setup at CL is shown in Figure 3.2.

![Figure 3.2 Constant-Load Test Setup](image)

The basic CL testing unit consisted of a calibrated proof ring, proof ring base, specimen grips, environmental chamber, microswitch, dial indicator, thermocouple, heating coil, temperature controller, elapsed timer controller and accessory support. Each
individually calibrated proof ring was provided with a calibration curve showing the load versus deflection of this ring, which was used to determine the applied load during the CL testing. A typical calibration curve for the proof ring is shown in Figure 3.3.

Micrometers with dial indicator were used to measure the ring deflection. The operation of the proof ring was based on the ability to transfer the load of a deflected proof ring to a tensile specimen to obtain a sustained loading. The entire test method was performed in accordance with the NACE Test Standard TM-01-77. Load was applied on the proof ring by using a standard wrench on the tension-adjusting screw and lock nut. A thrust bearing was employed to distribute the load and prevent seizure.

![Figure 3.3 A Typical Calibration Curve for the Proof Ring](Source: Cortest Incorporation)

The specimen grips on the proof rings were made of stainless steel to be fully-resistant to the testing environment. A standard clear, durable acrylic/pyrex glass environment chamber was used for the ambient-temperature testing to permit the visual
observation of the specimen. A Hastelloy C-276 vessel was used for the high-temperature testing. The environmental chamber was firmly secured by using O-ring seals to prevent any leakage. For high-temperature testing, a heating cartridge was connected to the bottom cover of the environmental chamber and a thermocouple was added in the top cover to monitor the testing temperature by the use of a temperature controller. The test specimens were electrically isolated by means of nylon bushings and all tube fittings were wrapped with Teflon tapes to prevent any leakage. Elapsed time monitors were used to record and provide digital display of the time-to-failure (TTF).

The magnitude of the applied load was based on the ambient-temperature tensile yield strength (YS) of the test material. The applied load was calculated by using an equation shown below. Cylindrical specimens with a notch at the center of the gage section were also used to study the effect of stress concentration on the cracking tendency.

\[ P = S \times A \]  \hspace{1cm} (3.1)

Where,

\[ P = \text{Applied Load} \]
\[ A = \text{Initial Cross-Sectional Area at the Gage Section (} d = 0.25 \text{ and } 0.156 \text{ in for smooth and notched specimens, respectively)} \]
\[ S = \text{Ambient-Temperature} \text{YS of Alloy EP-823} \]

The amount of deflection needed to apply the desired load on the smooth and notched specimens was obtained from the calibration curve of each proof ring. The specimens were loaded at stress values equivalent to the different percentages of the material’s YS values for the smooth and notched specimens. The corresponding TTF was recorded by a
timer attached to the test specimen. During the CL testing, once cracking was initiated in the specimen, the cross-sectional area was reduced and hence, the applied stress was increased. Thus, the specimen often failed soon after the initiation of cracking in susceptible environments. The determination of the cracking tendency using this method was based on the TTF for the maximum test duration of 30 days. The cracking tendency in the CL testing was expressed in terms of a threshold stress ($\sigma_{th}$) for a particular testing condition, below which failure did not occur during the maximum test period of 30 days.

3.3.2. Slow-Strain-Rate Testing

The slow-strain-rate (SSR) testing, also known as the constant extension rate testing (CERT), is a dynamic SCC evaluation technique (Figure 3.4). During SSR testing, the specimen was continuously strained in tension until fracture, according to the ASTM Designation G129 \(^{(51)}\), in contrast to more conventional SCC testing conducted under a sustained loading condition. The application of a dynamic plastic strain reduced the initiation time for the onset of cracking and caused an accelerated rupture of the surface films in the test material. This type of testing can cause failure in a material that probably might not occur under a CL condition or might have taken a prohibitively longer duration to initiate cracks in producing failures in it.\(^{(52)}\)
The primary advantage of the SSR testing technique is that it allows the evaluation of the effect of metallurgical variables such as alloy composition, heat-treatment, microstructure, and/or environmental parameters, in a relatively short duration. The SSR unit employed in this study has a load capacity of 7500 pounds with linear extension rates ranging from $10^5$ to $10^8$ in/sec. This unit contained a heavy-duty load-frame to minimize the system compliance but at the same time maintained the precise axial alignment of the load train. An all-gear drive system provided the consistent extension rate.

The SSR test setup used in this investigation consisted of a top-loaded actuator, testing chamber, linear variable differential transducer (LVDT), load cell, specimen grips, stepper motor, motion controller, thermocouple, heating coil, temperature controller and accessory support, as shown in Figure 3.5. The top-loaded actuator was intended to pull the specimen at a specified strain rate, so that the spilled solution, if any, would not
damage the actuator. A heating coil was connected to the bottom cover of the environmental chamber for elevated-temperature testing. A thermocouple was connected through the top cover of this chamber to monitor the testing temperature. The load cell was intended to measure load through an interface with the front panel. The LVDT was used to record the displacement of the gage section during testing.

Prior to the SCC testing by the SSR technique, the load-frame-compliance factor (LFCF), which is defined as the deflection in the frame per unit load, was determined by using a ferritic Type 430 stainless steel specimen during calibration. The generated LFCF data are shown in Figure 3.6. The LFCF values were fed to the load frame acquisition system, prior to the SCC testing to obtain the accurate results from the SSR unit.

Figure 3.5 SSR Test Setup with Accessory
A strain rate of $3.3 \times 10^{-6}$ sec$^{-1}$ was used during the SSR testing. This strain rate was selected based upon prior research work performed at the Lawrence Livermore National Laboratory (LLNL)$^{(53-54)}$. SCC occurrence is an effect of two significant factors such as the applied/residual stress and a susceptible environment. If the stress is applied at a very fast rate to the test specimen, while it is exposed to the aqueous environment, the resultant failure may not be different from the conventional mechanical deformation produced without an environment. On the other hand, if the strain rate is too slow, the resultant failure may simply be attributed to the corrosive damage due to environmental interaction with the material, thus, causing breakdown of the protective surface film. In view of this rationale, the SSR testing at LLNL was initially conducted at strain rates ranging between $10^5$ and $10^7$ sec$^{-1}$. Based upon this experimental work, it was determined that a strain rate of around $10^{-6}$ sec$^{-1}$ would provide the most effective
contributions of both the mechanical and environmental variables in characterizing the environment-induced cracking susceptibility during the SSR testing.\(^{(55)}\)

The susceptibility of Alloy EP-823 to SCC was evaluated by using both smooth and notched tensile specimens in neutral and acidic environments at ambient temperature, 60 and 90°C. The load versus displacement curves and engineering stress versus engineering strain curves for this alloy were generated during these tests. The dimensions (length and gage diameter) of the test specimens before and after testing were measured. During the SSR testing, the test specimens were subjected to a continuously changing stress. Hence, the cracking tendency of the test material was characterized by the TTF and ductility parameters such as the percent elongation (\(^{\%}\)El) and percent reduction in area (\(^{\%}\)RA). Further, the failure load (\(P_f\)) and true failure stress (\(\sigma_f\)) was also utilized to characterize the cracking tendency. The value of \(P_f\) was determined from load versus displacement curves. The magnitudes of \(^{\%}\)El, \(^{\%}\)RA, and \(\sigma_f\) were calculated by using the following equations.

\[
^{\%}\text{El} = \left(\frac{L_f - L_o}{L_o}\right) \times 100 \quad ; \quad L_f > L_o
\]  \hspace{1cm} (Equation 3.1)

\[
^{\%}\text{RA} = \left(\frac{A_o - A_f}{A_o}\right) \times 100 \quad ; \quad A_o > A_f
\]  \hspace{1cm} (Equation 3.2)

\[
\sigma_f = \frac{P_f}{A_f}
\]  \hspace{1cm} (Equation 3.3)

\[
A_o = \frac{\pi \times D_o^2}{4}
\]  \hspace{1cm} (Equation 3.4)

\[
A_f = \frac{\pi \times D_f^2}{4}
\]  \hspace{1cm} (Equation 3.5)

Where,
A_o = Initial Cross Sectional Area

A_f = Cross Sectional Area at Failure

P_f = Failure Load

\( \sigma_f \) = True Failure Stress

L_o = Initial Length

L_f = Final Length

D_o = Initial Diameter

D_f = Final Diameter

3.4 Localized Corrosion

Localized corrosion is a type of corrosion where there is intense attack at localized sites on the surface of a component while the rest of the surface undergoes damage at a slower rate. \(^{(28)}\) Localized corrosion is a serious concern due to its ability to cause more damage individually compared to other destructive processes such as stress, fatigue, erosion and other forms of chemical attack. The presence of halide ions such as chloride, and concentration cells developed on the surface of the alloys tend to induce degradation due to localized corrosion. The resistance to localized corrosion varies greatly among materials, and is dependent upon environmental factors. Hence, it was important to study the susceptibility of Alloy EP-823 to localized corrosion as a function of different environmental variables.

3.4.1. Cyclic Potentiodynamic Polarization Testing

Electrochemistry plays a significant role in understanding the corrosion mechanism of metallic materials. The polarization techniques can provide significant information on the
corrosion mechanisms, corrosion rate and susceptibility of materials to localized corrosion in different environments. The equilibrium conditions existing during an electrochemical process can be disturbed by applying either an external potential or current. This phenomenon is known as polarization. The magnitude of polarization is generally described by an overvoltage ($\eta$), which is a measure of polarized potential with respect to the corrosion potential ($E_{corr}$) of an electrode.\(^{(56)}\) The magnitude of $\eta$ can be either positive or negative depending on the applied electrochemical potential during the polarization of a metallic specimen of interest. The $\eta$ is given by the following equation.

$$\eta = E_{app} - E_{corr}$$

Where,

$$E_{app} = \text{Applied electrochemical potential}$$

$$E_{corr} = \text{Corrosion/open-circuit potential}$$

The magnitude of $\eta$ is positive for an anodic polarization while a negative $\eta$ indicates a cathodic polarization.

Cyclic potentiodynamic polarization (CPP) is a testing technique by which both anodic and cathodic polarizations can be performed in a cyclic manner. It is often used to evaluate the susceptibility of a material to pitting corrosion, which is usually characterized by a change in slope during the forward potential scan at a potential known as critical pitting potential ($E_{pit}$). At this potential, the material undergoes localized breakdown of its protective surface film causing initiation of pits. Materials that are capable of repassivation by formation of a protective film during the reverse potential scan are characterized by the development of a repassivation/protection potential ($E_{prot}$).
The larger the difference between $E_{\text{pit}}$ and $E_{\text{prot}}$, the greater is the resistance of the material to localized attack, in particular, to pitting corrosion.

The susceptibility of Alloy EP-823 to localized corrosion was determined by CPP experiments in neutral and acidic environments at 30, 60 and 90°C using a potentiostat, as shown in Figure 3.7.

Figure 3.7 Cyclic Potentiodynamic Polarization Test Setup

A potentiostat is an electronic device that controls the potential between the working and reference electrodes at a preset value. It allows the necessary current to flow between the working and counter electrodes to maintain the desired potential, as long as the required cell potential and current do not exceed the compliance limits of the potentiostat.\(^{(57)}\)
The potentiostats used in this investigation were calibrated prior to the performance of the CPP testing according to the ASTM Designation G 5. These potentiostats were calibrated to generate a characteristic polarization curve for a ferritic Type 430 SS specimen in an 1 Normal (1N) sulfuric acid (H₂SO₄) solution at 30°C using a potential scan rate of 0.166 mV/sec. A typical calibration curve showing potential versus current density for ferritic Type 430 stainless steel is illustrated in Figure 3.8.

The potentiostat used in the CPP tests involving the test material was considered to be functioning accurately if the generated calibration curve showed a similar shape and an acceptable \( E_{\text{corr}} \) value, as shown in the standard ASTM G 5 polarization diagram. A typical potentiodynamic polarization curve generated in this investigation is illustrated in Figure 3.8.
Figure 3.9 showing a similar pattern. In order to obtain reasonable polarization data, calibration tests were performed after every 20 CPP experiments.

![Generated ASTM G 5 Potentiodynamic Polarization Curve](image)

The three-electrode polarization system used in this investigation consisted of the test specimen as an anode, two graphite rods as cathodes and silver/silver chloride (Ag/AgCl) as the reference electrode. This reference electrode was contained in a Luggin probe placed within 2-3 mm from the center of the specimen that also acted as a salt bridge, as shown in Figure 3.10.\(^{(43)}\)

An initial delay time of 50 minutes was given before performing the forward and reverse scans to attain a stable \(E_{\text{corr}}\) value. The \(E_{\text{corr}}\) value of the test material in the desired test environment was determined with respect to the Ag/AgCl reference.
electrode, followed by forward and reverse potential scans at the ASTM specified rate of 0.166 mV/sec. The magnitudes of the $E_{\text{pit}}$ and $E_{\text{prot}}$, if any, were obtained from the CPP diagram. Duplicate tests were performed under each experimental condition.

![Diagram of Luggin Probe Arrangement](image)

Figure 3.10 Luggin Probe Arrangement

3.5. SCC Testing at Controlled Cathodic Potential

During the transmutation process, a significant amount of hydrogen may be generated, which can cause hydrogen-induced embrittlement (HE) of the target structural material, such as Alloy EP-823. HE is a delayed brittle failure resulting from the interaction of atomic hydrogen (H) diffused into the metal lattice. Hydrogen can be produced from an external environment or during the melting of a material of interest. However, the role of hydrogen studied in this investigation is primarily due to the atomic hydrogen produced during cathodic potentiostatic polarization. Electrochemically speaking, a metal is considered to be in equilibrium in an aqueous environment when the
rate of oxidation reaction (metal dissolution) and the rate of reduction reaction (hydrogen reduction) become equal. However, this equilibrium condition may be disturbed by polarizing the test specimen due to the application of either an external potential or a current. Subsequently, the resultant current and potential, respectively are recorded on a polarization diagram.

The potentiostatic polarization performed in this investigation involved the application of a negative potential to the gage section of the specimen, thereby producing atomic hydrogen ($\text{H}^+ + e = \text{H}$). The hydrogen generated by this process is nascent in nature, thus enabling its diffusion into the metal lattice in the form of an atomic hydrogen. With time, the concentration of atomic hydrogen may be increased inside the metal lattice, which may then interact at the weakest region of the metal lattice in the vicinity of the triaxial stress region, thus, enhancing internal stresses with subsequent formation of cracks. This phenomenon by which a metallic material undergoes failure in a hydrogen-containing environment due to the combined effect of applied/internal stress and the presence of aggressive chemical species, such as $\text{H}$, is known as HE. The susceptibility of a material to HE becomes more pronounced in the acidic environment due to the presence of increased concentration of hydrogen ion ($\text{H}^+$), compared to that in a neutral solution, generated during an electrochemical reaction.

The potentiostatic polarization under a cathodic applied potential is a common method of evaluating HE in susceptible materials. In this investigation, a constant potential, cathodic to the measured $E_{\text{corr}}$ value, was applied to the test specimen, which was spot-welded (Figure 3.11) with a conductive metallic wire for electron-flow. This wire was coated with lacquer to prevent contact with the test solution during polarization.
The spot-welded specimen was continuously strained in tension in the test environment using a similar strain rate of $3.3 \times 10^{-6}$ sec$^{-1}$, while simultaneously applying the controlled cathodic potential ($E_{\text{con}}$) to the specimen. The experimental setup used during cathodic charging is shown in Figure 3.12.$^{(53, 64-66)}$

![Figure 3.11 Spot-Welded Tensile Specimen](image-url)

Figure 3.11 Spot-Welded Tensile Specimen
3.6. Surface Analyses

The metallurgical properties of an engineering material depend on its chemical composition, thermal treatment and the resultant microstructures. The microstructure plays an important role in differentiating the properties of one alloy from that of the other. Thus, the evaluation of the metallurgical microstructure of a material of interest constitutes a significant step in characterizing its performance when exposed to a hostile environment under tensile loading. In view of this rationale, it is always customary to evaluate the metallurgical microstructure by optical microscopy at meaningful magnifications. Simultaneously, the characterization of failure, in particular, the extent and morphology of primary and secondary failure by SEM and optical microscopy,
respectively, is useful to develop a fundamental understanding of the failure mechanism of a material tested under certain environmental conditions.

Accordingly, significant emphasis was placed in this investigation to characterize the metallurgical microstructure and fractography of Alloy EP-823 using optical microscopy and SEM, respectively. The detailed analytical procedures are discussed in the following sub-sections.

3.6.1. Optical Microscopy

The metallographic technique using an optical microscope enables the characterization of phases present, their distributions within grains and their sizes that depend on the chemical composition and thermal treatments performed on a material of interest. The principle of an optical microscope is based on the impingement of a light source perpendicular to the surface of the test specimen. The light rays pass through the system of condensing lenses and the shutters up to the half-penetrating mirror. This brings the light rays through the objective to the surface of the specimen. Light rays reflected off the surface of the sample then return to the objective, where they are gathered and focused to form the primary image. This image is then projected to the magnifying system of the eyepiece. The contrast observed under the microscope results from either an inherent difference in intensity or wavelength of the light absorption characteristics of the phases present. It may also be induced by preferential staining or attack of the surface by etching with a chemical reagent.

The metallurgical microstructural evaluations of test specimens were performed by using standard metallographic techniques. These techniques include sectioning of the heat treated specimen at a desired location, mounting in an epoxy resin, grinding up to certain
depth, and finally polishing the mounted specimens in polishing wheel manufactured by Buehler Corporation. Some of the specimens were also etched in relevant chemical reagent (Fry's Reagent) to expose the thermal treated specimen surface to detailed microstructural analysis and identification of secondary cracks in tested specimens, if any, of Alloy EP-823. The polished and/or etched specimens were then evaluated by placing them on a top mounted platform attached to a Leica optical microscopy for the determination of metallurgical characteristics at different magnifications. Care was taken to ensure that the specimen was not over-etched. The specimen was then immediately washed with deionized water and subsequently dried with acetone and alcohol prior to metallographic evaluation by a Leica optical microscope. This microscope is capable of resolution of up to 1000X.

3.6.2. Scanning Electron Microscopy

In a scanning electron microscope (SEM), electrons from a metal filament are collected and focused just like light waves into a narrow beam. The beam scans across the subject, synchronized with a spot on a computer screen. Electrons scattered from the subject are detected creating a current, the strength of which makes the spot on the computer brighter or darker. This creates a photograph-like image with an exceptional depth of field. Magnifications of several thousand times are possible. Normally, SEM provides black and white micrographs. A JEOL-5600 scanning electron microscope, capable of resolution of up to 50 nm at magnifications of up to 100,000 times, was used in this study. The manual stage of this SEM can accommodate four 1 cm diameter samples or one sample with up to 3.2 cm diameter.
The extent and morphology of the primary failure in the SCC specimens were determined by SEM. The test specimens were sectioned into ½ to 3/4 of an inch in length to accommodate them in the vacuum chamber of the SEM. Usually, failure can occur by one or more of several mechanisms including surface damage, such as corrosion or wear, elastic or plastic deformation and fracture. Failures can be classified as ductile or brittle. Dimpled microstructure is a characteristic of ductile failure. Brittle failure can be of two types, intergranular and transgranular. An intergranular brittle failure is characterized by crack propagation along the grain boundaries while a transgranular failure is characterized by crack propagation across the grains.
CHAPTER 4

RESULTS

4.1 Effect of Heat Treatment on Hardness

The hardness values of Alloy EP-823 using a Rockwell hardness testing machine under different thermal-treatment conditions are shown in Table 4.1. These results indicate that the hardness value in the Rockwell C scale (Rc) was significantly reduced in the quenched and tempered (Q & T) condition, compared to that in the quenched only condition. The effect of tempering on hardness may be attributed to the homogenization of the metallurgical microstructure and relaxation of internal stresses resulting from the tempering operation, showing more pronounced effect at longer tempering time. A graphical representation of the effect of tempering time on the hardness value is shown in Figure 4.1. The resultant hardness based on three measurements is shown in Table 4.1, as presented in Appendix E.

<table>
<thead>
<tr>
<th>Heat Treatment Condition</th>
<th>Hardness, Rc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitized and Quenched</td>
<td>41</td>
</tr>
<tr>
<td>Quenched and Tempered for 1.25 hr</td>
<td>28</td>
</tr>
<tr>
<td>Quenched and Tempered for 1.75 hr</td>
<td>26</td>
</tr>
</tbody>
</table>

Table 4.1 Hardness of Alloy EP-823 under Different Heat-Treatment Conditions
4.2 Effect of Heat-Treatment on Microstructure

The results of metallographic evaluation of Alloy EP-823 by optical microscopy under different thermal treatment conditions are shown in Figure 4.2. An examination of these optical micrographs reveals that fine-grained and fully-tempered microstructure, characteristic of a martensitic stainless steel, resulted following quenching and tempering of this alloy. Some delta-ferrites were also observed in this material primarily due to the presence of significant amount of Cr, which is known to promote ferrite formation. It is interesting to note that the difference in tempering time did not influence the resultant metallurgical microstructure, in particular, the grain size of this alloy.
Figure 4.2 Optical Micrographs of Alloy EP-823 (a) Austenitized and Quenched, (b) Quenched and Tempered for 1.25 hours (c) Quenched and Tempered for 1.75 hours, Etched in Fry's Reagent, 10X
4.3. Ambient-Temperature Tensile Properties

The ambient-temperature tensile properties of Q & T Alloy EP-823, tempered for 1.25 and 1.75 hours, are shown in Figure 4.3, providing a comparison of the stress-strain (σ-ε) diagram as a function of the tempering time using smooth specimens. The magnitudes of YS, UTS, %El, and %RA, based on these plots and the dimensions of the tested specimens, are given in Table 4.2. The extent of uncertainty in the measured values is also shown for each parameter. It also be noted that the level of uncertainty, where applicable, have been included in relevant tables. An evaluation of the overall ambient-temperature tensile data indicates that, as expected, both YS and UTS were reduced at longer tempering time giving rise to somewhat improved ductility in terms of %El and %RA. The results shown in Table 4.2 are based on at least duplicate tests performed under each experimental condition, as shown in Appendices A and E, respectively.

Figure 4.3 Comparison of Stress-Strain Diagrams at Different Tempering Times
Table 4.2 Ambient-Temperature Tensile Properties using Smooth Specimens

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>YS (ksi)</th>
<th>UTS (ksi)</th>
<th>% El</th>
<th>% RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2055V</td>
<td>111±0.89</td>
<td>135±1.00</td>
<td>21.07±0.10</td>
<td>60.00±0.60</td>
</tr>
<tr>
<td>Alloy EP-823/2055W</td>
<td>108±0.86</td>
<td>121±0.97</td>
<td>26.02±0.10</td>
<td>62.62±0.57</td>
</tr>
</tbody>
</table>

V: Tempered for 1.25 Hour  
W: Tempered for 1.75 Hour

The results of room-temperature (RT) tensile testing using notched specimens of Alloy EP-823, quenched and tempered for 1.25 and 1.75 hours are given in Table 4.3, showing much higher failure strength, compared to those of the smooth specimens. A comparison of the σ-ε diagrams obtained by using smooth and notched specimens under similar thermal-treatment condition is illustrated in Figure 4.4. It is obvious from this comparison that, while the strength (YS/UTS) was enhanced, the ductility in terms of %El and %RA was significantly reduced in the notched specimen due to the plastic constraint resulting from the stress concentration effect. At least duplicate tests were performed using MTS under each heat treated condition, as presented in Appendices A and E, respectively.

Table 4.3 Ambient-Temperature Tensile Properties Using Notched Specimen

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>YS (ksi)</th>
<th>UTS (ksi)</th>
<th>% El</th>
<th>% RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2055V</td>
<td>211±2.71</td>
<td>224±2.87</td>
<td>3.00±0.10</td>
<td>15.09±1.61</td>
</tr>
<tr>
<td>Alloy EP-823/2055W</td>
<td>190±2.44</td>
<td>203±2.60</td>
<td>3.86±0.10</td>
<td>16.77±1.58</td>
</tr>
</tbody>
</table>

V: Tempered for 1.25 Hour  
W: Tempered for 1.75 Hour
4.4. Constant-Load SCC Testing

The results of SCC testing at constant-load (CL) involving smooth cylindrical specimens of Alloy EP-823 are shown in Tables 4.4. These results indicate that Alloy EP-823 did not exhibit any failure in the neutral environment at applied stresses (σ_{app}) corresponding to 90 and 95 percent of the material’s YS value at ambient temperature and 90°C. No failure was also observed with this alloy in the 90°C acidic solution at a σ_{app} value of 0.90YS. However, a failure was observed in this material when tested in the 90°C acidic environment at a σ_{app} value corresponding to the 95 percent of the material’s YS value.

The basic difference between the two tested environments lies in their pH values in that the acidic environment had much lower average pH value than that of the neutral solution (2.23 versus 6.35). Obviously, the cracking observed in the 90°C acidic solution...
may be attributed to the combined effect of the acidic pH and the elevated testing temperature. The resultant SCC data obtained with smooth specimens may suggest that the $\sigma_{th}$ value for a fully-tempered Alloy EP-823 may lie between 90 and 95 percent of its measured ambient temperature YS value when tested in the 90°C acidic environment, as shown in Figure 4.5. The magnitude of the $\sigma_{th}$ value under this experimental condition was approximately $102 \pm 0.10$ ksi (704 MPa). This result is based on at least duplicate tests performed under each experimental condition, as shown in Appendix B.

Table 4.4 SCC Test Results at CL using Smooth Specimens

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>Applied Stress (%)</th>
<th>TTF (Hour)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>%YS</td>
<td>Stress (ksi)</td>
</tr>
<tr>
<td>Alloy EP-823/2055V</td>
<td>Neutral Solution (Avg. pH ≈ 6.23)</td>
<td></td>
<td>90</td>
<td>99.90±0.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>95</td>
<td>105.45±0.84</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td>99.90±0.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>95</td>
<td>105.45±0.84</td>
</tr>
<tr>
<td></td>
<td>Acidic Solution (Avg. pH ≈ 2.35)</td>
<td></td>
<td>90</td>
<td>99.90±0.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>95</td>
<td>105.45±0.84</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

F: Failure  
NF: No Failure  
V: Tempered for 1.25 Hour
The results of SCC testing at CL using notched specimens are shown in Table 4.5. These data indicate that no failure was observed with Alloy EP-823 in the neutral solution at ambient temperature and 90°C at applied stresses of 0.30, 0.35, 0.40, 0.45 or 0.50 of its YS value. The notched specimens could not be loaded at stresses above the 0.50YS value since the specimens broke during the loading stage. Thus, a maximum applied stress corresponding to 50% of this material's YS value was used for CL SCC evaluation using the notched specimens. The data shown in Table 4.5 indicate that the notched specimens suffered from failure in the 90°C acidic solution at a $\sigma_{\text{app}}$ value of 0.50YS. The presence of a notch reduced the $\sigma_{\text{th}}$ value of Alloy EP-823 to 95±1.68 ksi (655 MPa) in the 90°C acidic solution, as shown in Figure 4.6. This result is based on at least duplicate tests performed under each experimental condition, as shown in Appendix B.
<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment (Avg. pH)</th>
<th>Temperature (°C)</th>
<th>Applied Stress % YS (ksi)</th>
<th>TTF (Hour)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Neutral Solution</td>
<td>Ambient 30</td>
<td>63.30±0.81</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 30</td>
<td>63.30±0.81</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 35</td>
<td>73.85±0.95</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 35</td>
<td>73.85±0.95</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 40</td>
<td>84.40±1.08</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 40</td>
<td>84.40±1.08</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 50</td>
<td>105.50±1.35</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 50</td>
<td>105.50±1.35</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td>Acidic Solution</td>
<td>Ambient 25</td>
<td>52.75±0.68</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 25</td>
<td>52.75±0.68</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 30</td>
<td>63.30±0.81</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 30</td>
<td>63.30±0.81</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 40</td>
<td>84.40±1.08</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 40</td>
<td>84.40±1.08</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 45</td>
<td>94.95±1.22</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 45</td>
<td>94.95±1.22</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ambient 50</td>
<td>105.50±1.35</td>
<td>NF</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 50</td>
<td>105.50±1.35</td>
<td>F (415 ± 0.05)</td>
</tr>
</tbody>
</table>

F: Failure
NF: No Failure
V: Tempered for 1.25 Hour
4.5. Slow-Strain-Rate Testing

The SSR testing was also performed to evaluate the susceptibility of Alloy EP-823 to SCC using smooth and notched cylindrical specimens in neutral and acidic solutions at ambient and elevated temperatures. During SSR testing, the specimens were subjected to a continuously changing stress at an applied strain rate of $3.3 \times 10^6$ sec$^{-1}$ until fracture. The cracking tendency was characterized by the TTF and ductility parameters such as %E1 and %RA. Further, the $\sigma_f$ was also used to characterize the SCC susceptibility.

4.5.1 Results using Smooth Specimens

The stress-strain ($\sigma$-$\varepsilon$) diagrams of Alloy EP-823, quenched and tempered (Q & T) for 1.25 and 1.75 hours, obtained by using smooth specimens in neutral and acidic solutions at room temperature (RT) and elevated temperatures, are shown in Figures 4.7 through 4.10. The $\sigma$-$\varepsilon$ diagram for Alloy EP-823 in air is also superimposed in these figures for comparison purpose. An examination of these figures indicates that the
magnitude of strain was gradually reduced with increasing temperature, irrespective of the testing environment.

Figure 4.7 Stress-Strain Diagrams in Neutral Solution

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Figure 4.8 Stress-Strain Diagrams in Neutral Solution

Figure 4.9 Stress-Strain Diagrams in Acidic Solution
Comparisons of $\sigma$-e diagrams obtained in neutral and acidic environments using smooth specimens tempered for 1.25 and 1.75 hours, respectively, are shown in Figures 4.11 and 4.12 as a function of the testing temperature. An examination of these diagrams reveals that the magnitude of strain was reduced with increasing temperature in both testing environments, showing more pronounced effect in the acidic solution.

Figure 4.10 Stress-Strain Diagrams in Acidic Solution

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The resultant SSR testing data obtained from Figures 4.11 and 4.12 and the specimen dimensions before and after testing are shown in Tables 4.6 and 4.7, respectively, for specimens quenched and tempered for 1.25 hour and 1.75 hours. These data indicate that
the magnitude of ductility parameters, TTF, and $\sigma_f$ were gradually reduced with increasing temperature, showing more pronounced effect in the acidic environment due to the synergistic effects of lower pH and higher testing temperature on the cracking susceptibility of the tested material.

Table 4.6 SSR Testing Results using Smooth Specimens Tempered for 1.25 Hours

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>$\sigma_f$ (ksi)</th>
<th>%EI</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2055V</td>
<td>Air</td>
<td>Ambient</td>
<td>197±2.53</td>
<td>21.20±0.10</td>
<td>59.50±0.60</td>
<td>20.86</td>
</tr>
<tr>
<td></td>
<td>Neutral Solution</td>
<td>Ambient</td>
<td>192±2.43</td>
<td>19.65±0.10</td>
<td>57.50±0.62</td>
<td>19.85</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90</td>
<td>117±1.22</td>
<td>13.70±0.10</td>
<td>35.50±0.82</td>
<td>14.84</td>
</tr>
<tr>
<td></td>
<td>Acidic Solution</td>
<td>Ambient</td>
<td>168±2.09</td>
<td>18.90±0.10</td>
<td>56.00±0.64</td>
<td>19.45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90</td>
<td>106±1.03</td>
<td>11.85±0.10</td>
<td>25.00±0.92</td>
<td>12.81</td>
</tr>
</tbody>
</table>

V: Tempered for 1.25 Hours
Table 4.7 SSR Testing Results using Smooth Specimens Tempered for 1.75 Hours

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>$\sigma_t$ (ksi)</th>
<th>%E1</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2055W</td>
<td>Air</td>
<td>Ambient</td>
<td>174±2.28</td>
<td>24.35±0.10</td>
<td>60.67±0.59</td>
<td>23.15</td>
</tr>
<tr>
<td>Neutral Solution (Avg. pH ≈ 6.23)</td>
<td>Ambient</td>
<td>171±2.22</td>
<td>23.00±0.10</td>
<td>60.00±0.60</td>
<td>21.97</td>
<td></td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>158±1.89</td>
<td>21.20±0.10</td>
<td>52.50±0.67</td>
<td>20.19</td>
<td></td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>108±1.15</td>
<td>16.13±0.10</td>
<td>39.00±0.79</td>
<td>15.00</td>
<td></td>
</tr>
<tr>
<td>Acidic Solution (Avg. pH ≈ 2.35)</td>
<td>Ambient</td>
<td>150±1.87</td>
<td>22.05±0.10</td>
<td>56.50±0.63</td>
<td>20.46</td>
<td></td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>119±1.25</td>
<td>17.50±0.10</td>
<td>36.50±0.82</td>
<td>16.85</td>
<td></td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>99±0.97</td>
<td>12.80±0.10</td>
<td>27.00±0.90</td>
<td>13.09</td>
<td></td>
</tr>
</tbody>
</table>

W: Tempered for 1.75 Hours

The SSR test data, shown in Tables 4.6 and 4.7, are graphically reproduced in Figures 4.13 through 4.16, showing the effect of pH, temperature, and tempering time on %E1, %RA, TTF, and $\sigma_t$. Examination of these figures indicates that the first three parameters were reduced in both testing environments at elevated temperatures, showing more pronounced effect in the acidic solution. However, the magnitude of $\sigma_t$ was relatively lower for specimens tempered for longer duration due to their lower strengths resulting from the release of internal stresses, on the other hand, the TTF and the ductility parameters were enhanced at the longer tempering time. These results are based on at least duplicate tests performed under each experimental condition, as shown in Appendices C and E.
Figure 4.13 Effects of pH, Temperature, and Tempering Time on %EI

Figure 4.14 Effects of pH, Temperature, and Tempering Time on %RA
4.5.2 Results using Notched Specimens

The stress-strain diagrams obtained by using notched specimens of Alloy EP-823 tempered for 1.75 hours in neutral and acidic solutions at 30, 60, and 90°C are shown in
Figures 4.17 and 4.18, respectively. These figures also show a gradual reduction in strain at higher temperatures, irrespective of the testing environment.

Figure 4.17 Stress-Strain Diagrams using Notched Specimens in Neutral Solution

Figure 4.18 Stress-Strain Diagrams using Notched Specimens in Acidic Solution
Comparisons of σ-ε diagrams using notched specimens tested in neutral and acidic solutions at different test temperatures are superimposed in Figure 4.19. An analysis of these diagrams clearly reveal that the strain was reduced with increasing temperature in both environments, showing more pronounced effect in the acidic solution.

![Figure 4.19 Comparison of Stress-Strain Diagrams using Notched Specimens in Neutral and Acidic Solutions](image)

The results shown in Figures 4.17 and 4.18 are reproduced in Table 4.8 showing all relevant parameters. The effects of pH and temperature on these parameters are illustrated in Figures 4.20 through 4.23. An examination of these figures indicates that the %RA and σf were appreciably reduced in both environments at elevated temperatures. However, very little reduction was observed with %El and TTF in either environment due to the presence of the notch. At least duplicate tests were performed under each experimental condition, as presented in Appendices C and E.

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Table 4.8 Results of SSR Testing using Notched Specimens Tempered for 1.75 Hours

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>σf (ksi)</th>
<th>%EI</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2054T</td>
<td>Air</td>
<td>Ambient</td>
<td>224±3.22</td>
<td>1.80±0.10</td>
<td>16.67±1.59</td>
<td>4.60</td>
</tr>
<tr>
<td></td>
<td>Neutral Solution</td>
<td>Ambient</td>
<td>216±3.03</td>
<td>1.75±0.10</td>
<td>12.48±1.64</td>
<td>4.26</td>
</tr>
<tr>
<td></td>
<td>Neutral Solution</td>
<td>60</td>
<td>201±2.79</td>
<td>1.50±0.10</td>
<td>10.75±1.67</td>
<td>4.02</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td></td>
<td>174±2.41</td>
<td>1.39±0.10</td>
<td>9.87±1.68</td>
<td>3.69</td>
</tr>
<tr>
<td>Acidic Solution (pH ≈ 2.35)</td>
<td>Ambient</td>
<td>198±2.75</td>
<td>1.64±0.10</td>
<td>10.60±1.67</td>
<td>4.11</td>
<td></td>
</tr>
<tr>
<td>Acidic Solution (pH ≈ 2.35)</td>
<td>60</td>
<td></td>
<td>172±2.38</td>
<td>1.31±0.10</td>
<td>9.56±1.68</td>
<td>3.56</td>
</tr>
<tr>
<td>Acidic Solution (pH ≈ 2.35)</td>
<td>90</td>
<td></td>
<td>164±2.26</td>
<td>1.20±0.10</td>
<td>8.89±1.69</td>
<td>3.45</td>
</tr>
</tbody>
</table>

T: Tempered for 1.75 Hours

Figure 4.20 Effects of pH and Temperature on %RA

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Figure 4.21 Effects of pH and Temperature on Failure Stress

Figure 4.22 Effects of pH and Temperature on % El
4.5.3. Results using Smooth versus Notched Specimens

Comparisons of σ-ε diagrams using smooth and notched cylindrical specimens in neutral and acidic environments at room temperature (RT), 60 and 90°C are shown in Figures 4.24 and 4.25, respectively as a function of the specimen geometry, pH, and temperature. It is obvious from these plots that the presence of a notch in the cylindrical specimen resulted in an enhancement of σf irrespective of the testing environment. With respect to the strain value obtained with smooth and notched cylindrical specimens, a general observation was its reduction due to the presence of the notch, as shown in Table 4.9. It should, however, be noted that higher reduction in the σf value was seen in the acidic solution at comparable testing temperatures.
Figure 4.24 Comparison of Stress-Strain Diagrams using Smooth versus Notched Specimens

Figure 4.25 Comparison of Stress-Strain Diagrams using Smooth versus Notched Specimens
Table 4.9 Comparison of SSR Testing Results using Smooth versus Notched Specimens

<table>
<thead>
<tr>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>$\sigma_f$ (ksi)</th>
<th>$%E_1$</th>
<th>$%R_A$</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Smooth</td>
<td>Notch</td>
<td>Smooth</td>
<td>Notch</td>
</tr>
<tr>
<td>Air</td>
<td>Ambient</td>
<td>174</td>
<td>224</td>
<td>24.35</td>
<td>1.80</td>
</tr>
<tr>
<td>Neutral Solution (Avg. pH = 6.23)</td>
<td>Ambient</td>
<td>171</td>
<td>216</td>
<td>23.00</td>
<td>1.77</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>158</td>
<td>201</td>
<td>21.20</td>
<td>1.50</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>108</td>
<td>174</td>
<td>16.13</td>
<td>1.39</td>
</tr>
<tr>
<td>Acidic Solution (Avg. pH = 2.35)</td>
<td>Ambient</td>
<td>150</td>
<td>198</td>
<td>22.05</td>
<td>1.64</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>119</td>
<td>172</td>
<td>14.00</td>
<td>1.31</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>99</td>
<td>164</td>
<td>12.80</td>
<td>1.20</td>
</tr>
</tbody>
</table>

The SSR testing data, shown in Table 4.9 using smooth and notched cylindrical specimens, are graphically illustrated in Figures 4.26 through 4.29, showing the effects of pH, temperature and specimen geometry on $%E_1$, $%R_A$, TTF, and $\sigma_f$. Examination of these figures reveals that all these parameters were reduced in both environments at elevated temperatures, showing more pronounced effect in the acidic solution. The presence of a notch significantly reduced the $%E_1$, $%R_A$, and TTF due to the smaller cross-sectional area at the root of the notch. However, it is interesting to note that the presence of a notch in the cylindrical specimens resulted in higher $\sigma_f$ values compared to those obtained using smooth specimens. This phenomenon may be attributed to the development of plastic constraint due to the triaxial state of stress in the vicinity of the notch and its smaller cross-sectional area at the root of the notch.
Figure 4.26 Effects of pH, Temperature, and Specimen Geometry on %EI

Figure 4.27 Effects of pH, Temperature, and Specimen Geometry on %RA
4.6 Cyclic Potentiodynamic Polarization Testing

The susceptibility of Alloy EP-823 to localized corrosion was evaluated by performing cyclic potentiodynamic polarization (CPP) experiments in neutral and acidic
environments at 30, 60 and 90°C by using a potentiostat. The effects of testing
temperature and pH on critical potentials were evaluated by this technique.

4.6.1 CPP Results in Neutral Solution

The results of CPP experiments performed in the neutral solution are illustrated in
Figures 4.30 through 4.32, showing corrosion potential ($E_{corr}$), critical pitting potential
($E_{pit}$), and protection potential ($E_{prot}$), if any, as a function of the testing temperature.
These data indicate that the $E_{corr}$ value became more active (negative) in the neutral
solution due to change in temperature from 30 to 60°C. However, the $E_{pit}$ was gradually
reduced to more active values with increasing temperature from 30 to 60 to 90°C, as
shown in these figures. The average values of $E_{corr}$, $E_{pit}$, and $E_{prot}$, if any, in the neutral
solution are shown in Table 4.10 at different testing temperatures. These results shown in
Table 4.10 are based on at least duplicate tests performed under each experimental
condition, as shown in Appendices D and E.
Figure 4.30 CPP Diagram in 30°C Neutral Solution

Figure 4.31 CPP Diagram in 60°C Neutral Solution
Table 4.10 CPP Test Results in Neutral Solution

<table>
<thead>
<tr>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>Critical Potentials (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>E_{corr}</td>
<td>E_{pit}</td>
</tr>
<tr>
<td>Neutral Solution</td>
<td>30</td>
<td>-625±1.88</td>
</tr>
<tr>
<td>(Avg. pH: 6.23)</td>
<td>60</td>
<td>-850±2.55</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>-780±2.34</td>
</tr>
</tbody>
</table>

4.6.2 CPP Results in Acidic Solution

The results of CPP testing conducted in the acidic solution are shown in Figures 4.33 through 4.35, identifying \(E_{corr}, E_{pit}, \) and \(E_{prot}\) as a function of the testing temperature. An examination of these diagrams indicates that, once again, the \(E_{corr}\) value became more active in the acidic solution at 60°C. However, the \(E_{pit}\) value gradually became more active with increasing temperature. The magnitudes of all critical potentials are given in
Table 4.11. The results shown in this table are based on at least two tests performed under each experimental condition, as presented in Appendices D and E.

Figure 4.33 CPP Diagram in 30°C Acidic Solution

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Figure 4.34 CPP Diagram in 60°C Acidic Solution

Figure 4.35 CPP Diagram in 90°C Acidic Solution

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Table 4.11 CPP Test Results in Acidic Solution

<table>
<thead>
<tr>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>Critical Potentials (mV)</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidic Solution</td>
<td>30</td>
<td>-500±1.5</td>
<td>-144±0.43</td>
<td>-185±0.56</td>
<td></td>
</tr>
<tr>
<td>(Avg. pH: 2.35)</td>
<td>60</td>
<td>-547±1.64</td>
<td>-298±0.89</td>
<td>None</td>
<td></td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>-415±1.25</td>
<td>-342±1.03</td>
<td>None</td>
<td></td>
</tr>
</tbody>
</table>

4.6.3 Comparison of CPP Data in Neutral and Acidic Solutions

The overall CPP results obtained in the neutral and acidic solutions are given in Table 4.12 as a function of the testing temperature. These data indicate that the \(E_{\text{corr}}\) value became more active in either environment as the temperature was increased from 30 to 60°C. However, the \(E_{\text{corr}}\) value became somewhat more noble at 90°C compared to that at 60°C, irrespective of the testing environment. A similar effect of temperature on the critical pitting potential has been reported by other investigators.\(^{67}\) It is interesting to note that the \(E_{\text{corr}}\) value was more active in the neutral solution compared to that in the acidic solution. This phenomenon cannot be explained at this time based on basic understanding of electrochemical principles. The extent of localized attack was, however, more pronounced in the 90°C acidic solution. The effects of temperature and pH on \(E_{\text{corr}}\) and \(E_{\text{pit}}\) are shown in Figures 4.36 and 4.37, respectively.
Table 4.12 Results of the CPP Testing in Neutral and Acidic Solutions

<table>
<thead>
<tr>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>Critical Potentials (mV)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$E_{\text{corr}}$</td>
<td>$E_{\text{pit}}$</td>
</tr>
<tr>
<td>Neutral Solution (Avg. pH: 6.23)</td>
<td>30</td>
<td>-620</td>
<td>-125</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>-850</td>
<td>-235</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>-780</td>
<td>-315</td>
</tr>
<tr>
<td>Acidic Solution (Avg. pH: 2.35)</td>
<td>30</td>
<td>-500</td>
<td>-144</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>-547</td>
<td>-298</td>
</tr>
<tr>
<td></td>
<td>90</td>
<td>-415</td>
<td>-342</td>
</tr>
</tbody>
</table>

Figure 4.36 Effects of pH and Temperature on Corrosion Potential

Alloy EP-823, Q & T
Heat No. 2055W

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The results of visual examination of the polarized specimens (Figures 4.38 and 4.39) indicate that Alloy EP-823 suffered from both pitting and crevice corrosion in either testing environment, showing enhanced localized corrosion susceptibility in the 90°C acidic solution. It is interesting to note that the test specimens showed more surface dissolution in specimens tested in the acidic solution compared to those in the neutral solution. Further, the depth of penetration was more pronounced in the acidic solution, in particular, at 90°C, as illustrated in Figure 4.39. However, with respect to the distribution of pits on the specimen surface, more shallow pits were observed in specimens tested in the neutral solution, as shown in Figure 4.38. A similar observation on the distribution of pits in neutral environment has been reported by other investigators.\(^{68}\)
Figure 4.38 Appearances of Polarized Specimens in Neutral Solution

(a) 30°C

(b) 60°C

(c) 90°C
Figure 4.39 Appearances of Polarized Specimens in Acidic Solution
4.7 SSR Testing Under $E_{\text{cont}}$

As indicated earlier, hydrogen can be generated during the transmutation process, thus, promoting hydrogen-induced damage in target structural material such as Alloy EP-823. In view of this rationale, SSR tests were performed in the acidic solution at 30, 60, and 90°C under a cathodic electrochemical potential ($E_{\text{cont}}$) of -1000 mV with respect to the Ag/AgCl reference electrode. The magnitude of this $E_{\text{cont}}$ was based on the measured $E_{\text{corr}}$ value in a similar environment, as shown in Table 4.12.

4.7.1 Results of $E_{\text{cont}}$ Testing

The results of SCC testing using smooth cylindrical specimens in the acidic solution under SSR condition at an $E_{\text{cont}}$ of -1000 mV (Ag/AgCl) are shown in Figure 4.40. An evaluation of these data indicates that the cracking susceptibility in terms of failure strain was enhanced in the acidic solution with increasing temperature under $E_{\text{cont}}$. The data shown in Figure 4.40 are reproduced in Table 4.13, showing the magnitude of $\sigma_f$, TTF, %EI, and %RA as a function of temperature. These data indicate that all these parameters gradually became reduced in the acidic solution with increasing temperature. The results shown in Table 4.13 are based on at least duplicate tests performed under each experimental condition, as shown in Appendices C and E. Typical current versus time plots generated during the potentiostatic polarization experiment are shown in Figures 4.41 through 4.43. These data indicate that some stable current values were reached after polarizing for some time.
Table 4.13 SSR Test Results using Smooth Specimens under $E_{cont}$

<table>
<thead>
<tr>
<th>Material/Heat No.</th>
<th>Environment</th>
<th>$E_{cont}$ (mV)</th>
<th>Temperature ($^\circ$C)</th>
<th>$\sigma_f$ (ksi)</th>
<th>%El</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy EP-823/2055W</td>
<td>Acidic Solution</td>
<td>-1000</td>
<td>Ambien</td>
<td>145±1.77</td>
<td>20.55±0.10</td>
<td>54.50±0.65</td>
<td>19.05</td>
</tr>
<tr>
<td></td>
<td>(Avg. pH = 2.35)</td>
<td></td>
<td>60</td>
<td>111±1.14</td>
<td>17.00±0.10</td>
<td>33.00±0.85</td>
<td>14.52</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>90</td>
<td>94±0.91</td>
<td>11.75±0.10</td>
<td>24.50±0.92</td>
<td>10.56</td>
</tr>
</tbody>
</table>

W: Tempering Time 1.75 hr

Figure 4.40 Comparison of Stress-Strain Diagrams under $E_{cont}$
Figure 4.41 Current-Time Diagram in 30°C Acidic Solution

Figure 4.42 Current-Time Diagram in 60°C Acidic Solution
4.7.2 Comparison of SCC Data with and without $E_{\text{cont}}$

The results of SCC testing in the acidic solution using SSR technique with and without $E_{\text{cont}}$ are shown in Figure 4.44. This figure illustrates comparative analyses of the stress-strain diagrams of Alloy EP-823 generated both in air and acidic solution. Obviously, failure strain in air was significantly higher compared to those in the acidic solution at different temperatures with and without $E_{\text{cont}}$. As to the effect of $E_{\text{cont}}$ on the failure strain in the presence of the acidic solution, a significant reduction in strain was observed due to the application of an $E_{\text{cont}}$ value of -1000 mV (Ag/AgCl), the effect being more pronounced at higher testing temperatures.

The overall data generated with and without applied potential are shown in Table 4.14 once again, showing the detrimental effect of $E_{\text{cont}}$ on the resultant parameters including $\sigma_f$, TTF, %El, and %RA. The effects of $E_{\text{cont}}$ on these parameters are graphically shown.

Figure 4.43 Current-Time Diagram in 90°C Acidic Solution
in Figures 4.45 through 4.48, indicating a gradual reduction in each parameter with increasing temperature.

![Graph showing stress-strain diagrams with and without E_cont](image)

Figure 4.44 Comparison of Stress-Strain Diagrams with and without $E_{\text{cont}}$

Table 4.14 Comparison of SSR Test Results with and without $E_{\text{cont}}$

<table>
<thead>
<tr>
<th>Environment</th>
<th>Temperature (°C)</th>
<th>$E_{\text{cont}}$ (mV)</th>
<th>$\sigma_t$ (ksi)</th>
<th>%El</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>Ambient</td>
<td>0</td>
<td>174.00</td>
<td>24.35</td>
<td>60.67</td>
<td>23.15</td>
</tr>
<tr>
<td>Acidic Solution (Avg. pH ≈ 2.35)</td>
<td>Ambient</td>
<td>0</td>
<td>150.00</td>
<td>22.05</td>
<td>56.50</td>
<td>20.46</td>
</tr>
<tr>
<td></td>
<td>-1000</td>
<td>-150.00</td>
<td>20.55</td>
<td>54.50</td>
<td>19.05</td>
<td></td>
</tr>
<tr>
<td>60</td>
<td>0</td>
<td>119.00</td>
<td>17.50</td>
<td>36.50</td>
<td>16.85</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-1000</td>
<td>111.00</td>
<td>17.00</td>
<td>33.00</td>
<td>14.52</td>
<td></td>
</tr>
<tr>
<td>90</td>
<td>0</td>
<td>99.00</td>
<td>12.80</td>
<td>27.00</td>
<td>13.09</td>
<td></td>
</tr>
<tr>
<td></td>
<td>-1000</td>
<td>94.00</td>
<td>11.75</td>
<td>24.5</td>
<td>10.56</td>
<td></td>
</tr>
</tbody>
</table>

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Figure 4.45 Effects of Temperature and \( E_{\text{cont}} \) on Failure Stress in Acidic Solution

Figure 4.46 Effects of Temperature and \( E_{\text{cont}} \) on TTF in Acidic Solution
4.8 Optical Microscopy

Metallographic evaluations were performed using optical microscopy to examine and characterize the presence of the secondary cracks along the gage section of the Alloy EP-
823 specimens tested under SSR condition. The surface characteristics of the polarized specimens were also analyzed.

4.8.1 Results of Optical Microscopy in Neutral and Acidic Solutions

The cylindrical specimens tested in the neutral and acidic solutions using the SSR technique at different testing temperatures were examined for the presence of secondary cracks in both polished and etched conditions. The results shown in Figure 4.49 indicate that no secondary cracks were observed in the 30°C. However, secondary cracking was noticed at 60 and 90°C. The extent of secondary cracking was more pronounced at 90°C. The optical micrographs, shown in Figure 4.50, indicate that cracking along the gage section was more severe in the acidic solution, showing much larger and branched cracks.
Polished, 10X
Etched in Fry's Reagent, 10X
(a) 30°C

Polished, 10X
Etched in Fry's Reagent, 10X
(b) 60°C

Polished-10X
Etched (Fry's Reagent-10X)
(c) 90°C

Figure 4.49 Optical Micrographs of Tested Specimens in Neutral Solution
Polished-10X  Etched in Fry’s Reagent, 10X
(a) 30°C

Polished, 10X  Etched in Fry’s Reagent, 10X
(b) 60°C

Polished, 10X  Etched in Fry’s Reagent, 10X
(c) 90°C

Figure 4.50 Optical Micrographs of Tested Specimens in Acidic Solution
4.8.2 Results of Optical Microscopy of $E_{cont}$ Specimens

Specimens tested in the acidic solution under $E_{cont}$ were also examined for secondary cracks using optical microscopy. The results, shown in Figure 4.51, illustrate larger secondary cracks in Alloy EP-823, compared to those seen in specimens with an $E_{cont}$. Once again, the extent of secondary cracks was more pronounced at higher testing temperatures.

Figure 4.51 Optical Micrographs of Tested Specimens in Acidic Solution with $E_{cont}$

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4.8.3 Results of Optical Microscopy of CPP Testing in Neutral and Acidic Solutions

The results of optical microscopic evaluations involving the specimens polarized in neutral and acidic solutions at 90°C are shown in Figures 4.52 and 4.53, respectively. As indicated earlier, more scattered pits were observed in the neutral solution, but pit sizes were larger and deeper in the acidic solution.

Figure 4.52 Optical Micrograph of CPP Specimen in 90°C Neutral Solution, 10X
4.9 Results of Fractographic Examination

Fractography was performed on the smooth and notched cylindrical specimens of Alloy EP-823 using SEM to study the extent and morphology of failure at their primary fracture faces as a function of the testing environment. It appears that the failure mode in the neutral solution at all three testing temperatures was predominantly ductile, showing dimpled microstructure (Figure 4.54). However, brittle failures were observed in the acidic solution, as illustrated in Figure 4.55. A combination of intergranular and transgranular cracks was observed in the SEM micrographs in this environment.
Figure 4.54 SEM Micrographs of Specimens Tested in Neutral Solution

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Figure 4.55 SEM Micrographs of Specimens Tested in Acidic Solution

(a) 30°C, 750X

(b) 60°C, 750X

(c) 90°C, 750X
Fractographic evaluations using notched specimens in the neutral solution are shown in Figure 4.56, which illustrate dimpled microstructures indicating ductile failures. Slight cracks were noticed in the acidic solution (Figure 4.57). Finally, the application of an $E_{\text{cont}}$ to the test specimens during the SSR testing in the acidic solution resulted in increasing cracking susceptibility due to the hydrogen damage, as illustrated by the brittle failures in Figure 4.58.

Figure 4.56 SEM Micrographs of Notched Specimens Tested in Neutral Solution

(a) 30°C, 950X

(b) 90°C, 950X

Figure 4.56 SEM Micrographs of Notched Specimens Tested in Neutral Solution

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Figure 4.57 SEM Micrographs of Tested Notched Specimens in Acidic Solution

(a) 30°C, 950X

(b) 90°C, 950X

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Figure 4.58 SEM Micrographs of Specimens Tested in Acidic Solution under an $E_{\text{cont}}$
CHAPTER 5

DISCUSSION

This dissertation is focused on elucidating the susceptibility of Alloy EP-823, a leading candidate target structure material, to stress-corrosion-cracking (SCC), hydrogen-embrittlement (HE) and localized corrosion (pitting and crevice) in aqueous environments. Numerous state-of-the-art experimental techniques have been utilized to evaluate these degradation modes. The propensity to SCC has been determined using smooth and notched specimens in neutral and acidic solutions under constant-load (CL) and slow-strain-rate (SSR) conditions at different temperature. The localized corrosion behavior has been studied by using electrochemical cyclic potentiodynamic polarization (CPP) method. The effect of hydrogen on the cracking (HE) susceptibility has been evaluated by using SSR testing technique under a cathodic applied potential (E_{cont}). Microstructural evaluation and characterization of secondary cracks along the gage section of the tested specimens and the surface characterization of the tested CPP specimens have been conducted by optical microscopy. The extent and morphology of the primary failure of the tested tensile specimens were determined by SEM. Discussions on the overall data generated in this investigation are presented below.

5.1 Thermal Treatment Effect on Metallurgical Characteristics

As mentioned earlier, Alloy EP-823 was quenched and tempered (Q & T) to achieve a fine-grained and fully-tempered martensitic microstructure, providing the desired
In order to study the effect of tempering time on the hardness, metallurgical microstructure, and resultant tensile properties, Alloy EP-823 was tempered for 1.25 and 1.75 hours following the austenitizing and quenching operations. It is well known that austenitizing and quenching can produce hard but brittle martensite which may cause problems during the fabrication processes. In view of the drawback associated with the hardened material, subsequently tempering was performed to relieve the internal stresses generated inside the metal matrix due to rapid cooling during the quenching operation. This relaxation of stresses through homogenization of metallurgical microstructure for some specified time period can enhance the ductility of a metallic material involving martensitic Alloy EP-823.

As shown in the previous section, the hardness of the Q & T material was significantly reduced compared to that of austenitized and quenched Alloy EP-823, showing the beneficial effect of tempering. The beneficial effect of longer tempering time on the hardness has also been demonstrated. However, no significant effect of tempering time on the resultant metallurgical microstructure was observed. As to the effect of tempering time on the metallurgical properties such as the stress-strain relationship, the magnitude of the yield strength (YS), ultimate tensile strength (UTS), and failure stress ($\sigma_f$) was reduced but the ductility in terms of strain was enhanced due to the tempering operation for a longer duration as shown in Figure 4.3. In essence, a longer tempering time promotes an improved ductility.

A comparison of the stress-strain diagrams involving smooth and notched cylindrical specimens clearly demonstrated that the magnitude of $\sigma_f$ was significantly higher in the presence of a notch primarily due to the stress concentration effect. Obviously, a reduced
ductility was also noticed along with this enhanced failure strength due to the presence of a notch. A plastic constraint is developed in the vicinity of a notch in a tensile specimen due to the development of a triaxial state of stress. In addition to the axial stress, radial and transverse stresses can also be developed due to Poisson’s effect, leading to lateral contractions in the necked region of a specimen made of a ductile material such as Alloy EP-823. Since the material immediately adjacent to the necked region can experience a much lower stress level, the notched region would resist such constraint by exerting tensile stresses to retard the deepening of the notch. Consequently, a triaxial stress field would plastically constrain the material from deforming in the necked region. Thus, the axial stress would be increased in the presence of a notch to induce plastic flow, as seen with Alloy EP-823. A similar behavior has also been observed with Alloys HT-9 and 422.\(^{(71-72)}\)

5.2 Constant-Load Testing

The results of SCC testing at CL using smooth specimens of Alloy EP-823 indicate that no failures were observed in neutral solution at any tested temperature. However, failures were observed in the 90°C acidic solutions when tested at an applied stress \(\sigma_a\) corresponding to 95% of the material’s ambient-temperature YS value. Since no failures were observed in the acidic solution at a \(\sigma_a\) value of 0.90YS, it can be construed that the magnitude of the threshold stress \(\sigma_{th}\) for cracking for this material may lie in between 90 and 95% of its YS value (approximately 102±1.00 ksi). It is interesting to note that, compared to the other martensitic stainless steels,\(^{(71-72)}\) Alloy EP-823 exhibited superior resistance to SCC in terms of \(\sigma_{th}\) when tested in the 90°C acidic solution. For example, for Alloys HT-9 and 422, the magnitude of \(\sigma_{th}\) was in the vicinity of 80% of their
ambient-temperature YS values. Such improved cracking resistance of Alloy EP-823 may be attributed to the presence of a higher silicon (Si) content (1.12 versus 0.4 wt %). The beneficial effect of an optimum Si content on the corrosion resistance of martensitic alloys has also been reported elsewhere.\(^\text{(73)}\) The presence of a notch, however, reduced the \(\sigma_{th}\) value in the 90°C acidic solution to 0.45YS (approximately 95±1.68 ksi). Despite this reduction in \(\sigma_{th}\) value due to the notch, this value was much higher compared to those for Alloys HT-9 and 422.\(^\text{(71-72)}\) The effect of stress concentration on the cracking susceptibility has been demonstrated by other investigators.\(^\text{(74-75)}\)

It is well known that hydrogen ion (\(\text{H}^+\)) in increased concentration may exist in an acidic solution due to the increased rate of cathodic reaction, thus, leading to enhanced cracking susceptibility in metallic materials and alllloys.\(^\text{(46)}\) Therefore, the enhanced cracking susceptibility of Alloy EP-823 in the 90°C acidic solution may be attributed to the synergistic effect of an acidic pH and higher testing temperature. Temperature dependence of cracking in this alloy may be the result of temperature-induced changes in the protective properties of passive film on the metal surface thereby, resulting in rapid breakdown of this film with increasing temperature.\(^\text{(76)}\) These changes can be rationalized either by an increasing porosity or by an intrinsic modification of the chemical composition and/or physical structure of passive film at higher testing temperature.\(^\text{(77-78)}\) The detrimental effect of temperature and pH on environment-induced cracking of metals and alloys has also been reported by other investigators.\(^\text{(79-80)}\)

5.3 Slow-Strain-Rate Testing

The results of SSR testing, presented in the previous section, indicate that the magnitude of strain was reduced due to a change in temperature from room temperature
(RT) to 60 to 90°C in both tested environments. This detrimental effect of higher testing temperature was more pronounced in the acidic solution. The reduction in strain in the acidic solution may be due to the increased hydrogen ion (H+) concentration that may accelerate the breakdown of surface film. The extent of ductility (%EL and %RA), TTF, and σf, determined from the stress-strain curves and the specimen dimensions, were gradually reduced with increasing temperature in the acidic solution, once again, showing more pronounced effect in this environment. The combined effect of acidic pH and higher testing temperature on cracking susceptibility has also been cited by other researchers.

The results of SSR testing using smooth specimens, tempered for 1.25 and 1.75 hours, revealed enhanced TTF and ductility at longer tempering time in both environments. However, the magnitude of σf was appreciably reduced due to tempering for a longer duration. Such reduction in σf may be attributed to the relief of the materials' internal stresses during tempering for a longer duration.

The presence of a notch further reduced the magnitude of %EL, %RA and TTF, irrespective of the testing environment, as shown earlier in a previous section. However, the magnitude of σf was enhanced due to the plastic constraint and reduced cross-sectional area at the root of the notch. A similar observation has been made by other investigators.

5.4 CPP Testing

Based on the results of the localized corrosion study presented earlier, it is clear that Alloy EP-823 may undergo localized attack, including pitting and crevice corrosion, irrespective of the testing environment and temperature. A similar behavior was also

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noticed by some recent investigators.\(^{47, 82}\) The initiation of pitting in susceptible metals and alloys is usually the result of the breakdown of the passive films on their surface in the presence of aggressive anions, such as chloride ions (Cl\(^-\)) and the subsequent establishment of an electrochemical cell in which the damaged site acts as an anode and the surrounding passive surface acts as a cathode.\(^{67}\) It is well known that the initiation of crevice corrosion in metallic materials immersed in chloride-containing aqueous solutions may involve the dissolution of metals to metallic ions and maintenance of a high degree of acidity within the crevice region by the hydrolysis of the dissolved metal ions.\(^{83}\)

The results of the CPP experiments performed in both the neural and acidic solutions, indicate that the magnitude of the corrosion potential (E\(_{\text{corr}}\)) became more active (negative) due to a change in temperature from 30 to 60°C. However, the E\(_{\text{corr}}\) value became somewhat noble (positive) at 90°C. A similar behavior was also noticed by other researchers,\(^{67}\) who tested different materials, however, no viable explanation can be provided as to this phenomena based on basic understanding of electrochemical principles. Nevertheless, a consistent pattern on the effect of temperature on the critical pitting potential (E\(_{\text{pit}}\)) was noticed in that its value gradually became more active with increasing temperature, irrespective of the testing environment.\(^{47, 82, 84}\) It is well known that the magnitude of these critical potentials may become more active (negative) with increasing temperature.\(^{46, 76-78}\) A similar phenomenon was also observed in this investigation, showing a consistent pattern on the effect of temperature on the E\(_{\text{pit}}\) value.

Protection potential (E\(_{\text{prot}}\)), which is a characteristic of repassivation of the material surface, was not consistently observed in all CPP diagrams, as shown earlier in the
previous section. The difference in the magnitude of $E_{\text{pit}}$ and $E_{\text{prot}}$ of a material of interest can influence its susceptibility to localized attack. The larger this difference the better is the resistance of the material to pitting corrosion. The tested polarization specimens exhibited enhanced dissolution in the acidic solution, showing a lower distribution of pits on the surface. However, the localized penetrations (pits) were deeper in specimens polarized in the acidic solution. The extent of localized attack was, however, more severe in this environment at elevated temperature.\(^{(46, 76-78)} \) The detrimental effect of lower pH and higher temperature on the breakdown of surface film can, thus, be attributed to this phenomenon.\(^{(79-80)} \)

5.5 SSR Testing under $E_{\text{cont}}$

It is well known that an electrochemical reaction is a combination of an anodic and a cathodic reaction. The anodic (oxidation) reaction produces a metal ion ($M^+$) and an electron, which is used in the reduction of hydrogen ion ($H^+$) to produce atomic hydrogen ($H$). During potentiostatic polarization of a metal in an acidic solution under cathodic control, more $H^+$ ions can be generated causing increased $H^+$ concentration that can diffuse into the metal lattice at some susceptible temperature regime.

The results of SCC testing under cathodic applied potential of $-1000$ mV (Ag/AgCl) using the SSR technique indicate that the magnitude of $\sigma_t$, TTF, $\%$EI, and $\%$RA was reduced, compared to those obtained without an $E_{\text{cont}}$ due to the generation of more hydrogen ions ($H^+$) in the acidic solution due to the cathodic charging. However, as discussed in a previous section, the extent of reduction in all four parameters was more pronounced at $90^\circ$C indicating a detrimental effect of higher testing temperature on the cracking susceptibility of Alloy EP-823, with or without an $E_{\text{cont}}$. A similar effect of
cathodic applied potential on the cracking susceptibility of metallic materials has been reported by other investigators.\(^{(53, 59-66, 85-87)}\)

5.6 Explanation of Change in Slope

5.6.1 Change in Slope in MTS Testing

The metallurgical properties of a material of interest are dependent on many factors including thermal treatment imparted to achieve these properties. Alloy EP-823 was tempered for two different times to study their effect on the resultant properties. It is interesting to note that a slight change in slope was observed in the stress-strain diagram of Alloy EP-823 as a function of the tempering time. Even though this change is insignificant, it is possible that this variation in the slope, thus, the modulus too, can be attributed to different tempering times.

5.6.2 Change in Slope in SSR Testing

During the SSR testing, the stress-strain diagram may exhibit slight changes in slope as a function of different testing environments, as observed in the current investigation. It is interesting to note that the effect of environment on the slope (such as elastic modulus) has not been investigated extensively by other researchers. However, one should realize that the stress-strain diagram in mechanical testing is entirely different from under combined influence of applied stress and deleterious environment such as salt solution containing acidic species. In essence, the presence of an aggressive environment can influence this parameter primarily due to the interaction of this environment with the metallurgical structure of the candidate material, thereby causing some inherent changes in this parameter. In view of this rationale, it is not surprising that slight changes in the slope may occur during SCC testing using the SSR concept. Further, in the presence of a
notch, the structural stability of the test specimen may become impaired resulting in stepwise data accumulation, as seen in this investigation.

5.7 Metallographic Evaluation

Metallographic evaluations performed on the quenched and tempered specimens of Alloy EP-823 by optical microscopy showed conventional fine-grained and fully-tempered martensitic microstructures, as expected.

The tensile specimens tested in the neutral solution under SSR conditions did not show any secondary cracks at RT. Very few secondary cracks were observed along the gage section when tested in the 60°C neutral solution. However, numerous secondary cracks were observed in this solution at 90°C. No secondary cracks were observed in the tested specimens in the acidic solution at RT. However, branched secondary cracks were noticed in specimens tested in the acidic solution at 60 and 90°C, showing more severe cracks at 90°C. A similar observation has been made by other investigator. (53)

The polarized specimens showed both pitting and crevice corrosion in the neutral solution, irrespective of the testing temperature. However, the distribution and severity of the pits were enhanced at higher temperature. In general, the specimens tested in the acidic solution showed surface dissolution, irrespective of the testing temperature. However, increased dissolution was observed at higher temperatures, showing deepest pits at 90°C.

The specimens tested in the acidic solution under controlled potential showed larger secondary cracks at RT and 60°C compared to those seen in absence of E_{cont}. Branched secondary cracks were also observed in the 90°C acidic solution under the influence of the controlled potential.
5.8 Fractographic Evaluation

Fractographic evaluations by SEM revealed ductile failures in the smooth specimens tested in the neutral solution at RT using SSR testing technique, irrespective of the testing temperature. Intergranular brittle failures were observed in the acidic solution at RT. A combination of intergranular and transgranular brittle failures were observed in the 60 and 90°C acidic solutions, showing more pronounced effect at 90°C. Similar types of cracking morphology have been reported elsewhere.\(^{53,88-90}\)

The notched specimens tested in the neutral solution using SSR condition showed ductile failure showing dimpled microstructure, irrespective of the testing temperature. However, combined intergranular and cleavage type of failures were observed in the acidic solution at RT and 90°C in the presence of a notch.

SEM study performed on specimens subjected to controlled electrochemical potential in the RT acidic solution revealed intergranular brittle failures. However, both intergranular and transgranular brittle failures were observed in this solution at 60 and 90°C.

5.9 Implication of the Results

An analysis of the overall data presented in this dissertation indicates that martensitic Alloy EP-823 having higher silicon (Si) content (1.12% by weight) may be the most suitable target structural material compared to the other two candidate alloys, namely Alloys HT-9 and 422 (0.20 and 0.51% by weight of Si, respectively).\(^{71-72}\) It should, however, be noted that Si is not the only element that can enhance the protectiveness of the surface film in this alloy. There are other alloying elements such as Cr, Ni, Mo, and niobium/columbium (Nb/Cb), which can also significantly influence the
metallurgical/corrosion behavior of a structural material. For example, superior corrosion resistance is attributed to the formation of chromium-oxide \((\text{Cr}_2\text{O}_3)\) films. Simultaneously, Nb/Cb has stronger affinity for carbon (C), thereby producing carbide, a phenomena which can prevent preferential segregation of chromium-carbide at the grain boundary, thus, retaining its superior corrosion resistance. Presence of Mo can also inhibit the localized corrosion susceptibility by stabilizing the oxide film.

In order to precisely evaluate the effect of Si on corrosion resistance, additional work is needed. It is worth-mentioning at this time that a new research project has recently been initiated at UNLV to study the effect of Si content on both metallurgical and corrosion characteristics of martensitic stainless steels in the presence of both molten metal and aqueous environments. The effect of radiation on these phenomena has also been proposed to be investigated.

Superior metallurgical and corrosion properties, cited and evaluated under this investigation, are critical in selecting the appropriate structural materials for numerous engineering applications. For example, high strength and ductility are considered during fabrication of an engineering component such as a structural vessel to contain molten metal. Simultaneously, appreciable corrosion resistance is desired to ensure the structural integrity of a component while it is in contact with an aggressive environment such as molten LBE. Thus, it should suffice to state the applicability of all these properties in selecting suitable materials for specific applications.

Even though the transmutation process may occur in the temperature regime 420 to 550°C, testing could not be performed in this temperature range under the current
investigation. Thus, it is suggested that additional corrosion testing be performed at higher testing temperature in the presence of molten LBE.
CHAPTER 6

SUMMARY AND CONCLUSIONS

Martensitic Alloy EP-823 has been proposed to be a candidate structural material to contain the molten lead-bismuth-eutectic (LBE) target during the transmutation process. This investigation is focused on the evaluation of the susceptibility of Alloy EP-823 to environment-induced degradations such as stress corrosion cracking (SCC), localized corrosion (pitting/crevice production), and hydrogen embrittlement (HE) in neutral and acidic solutions at different temperatures. The SCC susceptibility was evaluated by the SSR and CL testing techniques using smooth and notched cylindrical specimens. The localized corrosion behavior was determined by cyclic potentiodynamic polarization (CPP) method. The effect of hydrogen on the cracking behavior was evaluated by applying cathodic potential ($E_{\text{cont}}$) to the test specimens while in tension. Metallographic and fractographic evaluations of the tested specimens were performed by optical microscopy and SEM, respectively. The significant conclusions derived from this study are given below.

- The hardness ($R_c$) value was reduced significantly due to tempering, showing more pronounced effect at the longer tempering time. Ductility was also enhanced due to the longer tempering time. However, the strength was reduced due to the relaxation of internal stresses.
- Fine-grained and fully-tempered martensitic microstructure resulting from quenching and tempering was observed by optical microscopy. However, no effect of tempering time on grain size was noticed.

- No failures were observed in the neutral solution at CL using smooth specimens, irrespective of the applied stress and temperature. No failure was observed in the notched specimens tested in a similar neutral environment at applied stress up to 50 percent of the material’s YS value.

- Alloy EP-823 did not exhibit cracking in the 90°C acidic solution at an applied stress of 0.90YS. However, failures were observed in this environment at 0.95YS resulting in a $\sigma_{th}$ value of approximately 102±1.00 ksi. The presence of a notch reduced the $\sigma_{th}$ value to 95±1.68 ksi when tested in a similar environment.

- Since no failures were observed in the acidic solution at ambient temperature, the enhanced cracking tendency at 90°C may be the result of the combined effect of elevated temperature and pH.

- The magnitude of the ductility parameters (%El and %RA), TTF, and $\sigma_f$ was gradually reduced with increasing temperature in both environments. However, the extent of reduction of these parameters was more pronounced in the acidic solution, implying a synergistic effect of acidic solution pH and temperature on the cracking susceptibility.

- The presence of a notch enhanced the magnitude of $\sigma_f$ due to the plastic constraint and reduced cross-sectional area at the root of the notch, irrespective of the testing environments.
• All polarized specimens exhibited localized corrosion susceptibility in neutral and acidic solutions at ambient and elevated temperatures. A scattered distribution of shallow pits was observed in the neutral solution. However, general dissolution of the polarized specimens was noticed in the acidic solution. The pits developed in the acidic solution were deeper and wider.

• The magnitude of critical pitting potential ($E_{pit}$) consistently became more active (negative) in both environments with increasing temperature. The corrosion potential ($E_{corr}$) also became more active due to a change in temperature from 30 to 60°C.

• An application of a cathodic electrochemical potential (-1000 mV, Ag/AgCl) to the test specimens during straining resulted in a reduced ductility in terms of strain, $\%E_1$, and $\%RA$. The magnitude of failure stress was also further reduced due to cathodic charging, as anticipated.

• The observed reduction in ductility and strength in Alloy EP-823 by potentiostatic cathodic polarization may be attributed to the generation of atomic hydrogen and its diffusion into the metal lattice during the straining process.

• Secondary failures were observed along the gage section of the tested specimens by optical microscopy that revealed branched cracking, the extent of cracking being more pronounced in the acidic solution.

• Characterization of the polarized specimens by optical microscopy showed wider and deeper pits in the acidic solution.

• Fractographic evaluations of the primary fracture face by scanning electron microscopy (SEM) revealed a combination of ductile and brittle failures. The
ductile failures were dimpled microstructure. The brittle failures included intergranular and transgranular cracks.

- A general inference, based on the overall results, is that Alloy EP-823 may be the most viable target structural material, compared to other two candidate materials (Alloys HT-9 and 422), possessing the desired metallurgical properties and suitable corrosion resistance for transmutation applications.
CHAPTER 7

FUTURE WORK

The following additional work for further evaluations is suggested:

- Due to the lack of data in the presence of molten lead-bismuth-eutectic (LBE), it is warranted that corrosion studies be performed in this environment should an LBE testing facility becomes available either at UNLV or at LANL.
- Develop an understanding of the molten metal corrosion mechanism as opposed to electrochemical corrosion elucidated in the current investigation.
- Perform potentiostatic anodic polarization of Alloy EP-823 to investigate the effect of surface film rupture on cracking.
APPENDIX A

MTS DATA

A1. Stress-Strain Diagram using Smooth Specimens Tempered for 1.25 Hours

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A2. Stress-Strain Diagram using Smooth Specimens Tempered for 1.75 Hours
A3. Stress-Strain Diagram using Notched Specimens Tempered for 1.25 Hours

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A4. Stress-Strain Diagram using Notched Specimens Tempered for 1.75 Hours

![Diagram of Stress-Strain for Sample 1]

![Diagram of Stress-Strain for Sample 2]

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APPENDIX B

CONSTANT-LOAD (CL) DATA

B1. SMOOTH SPECIMENS

<table>
<thead>
<tr>
<th>Environment/Temperature (°C)/Applied Stress (%YS)</th>
<th>Failure (Hour)/No Failure</th>
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</thead>
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<tr>
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<td>Neutral/Ambient/0.90YS</td>
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<td><strong>Neutral/Ambient/0.90YS (Mean)</strong></td>
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<tr>
<td>Acidic/Ambient/0.90YS</td>
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<tr>
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<td><strong>Neutral/90/0.95YS (Mean)</strong></td>
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<td>Acidic/90/0.95YS</td>
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### B2. NOTCHED SPECIMENS

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<td>90</td>
<td>30</td>
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</tr>
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APPENDIX C

SLOW-STRAIN-RATE (SSR) DATA

C1. Stress-Strain Diagram in Air using Smooth Specimens Tempered for 1.25 Hours

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C2. Stress-Strain Diagram in Air using Smooth Specimens Tempered for 1.75 Hours

Sample 1-Air

Sample 2-Air

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C3. Stress-Strain Diagram in Air using Notched Specimens Tempered for 1.75 Hours

[Graphs showing stress-strain relationships for samples 1, 2, and 3 in air for Alloy EP-823]
C4. Stress-Strain Diagram in Neutral Solution using Smooth Specimens Tempered for 1.25 Hours

Sample 1-30°C

Sample 2-30°C
Sample 1-90°C

Sample 2-90°C
C5. Stress-Strain Diagram in Neutral Solution using Smooth Specimens Tempered for 1.75 Hours

Sample 1-30°C

Sample 2-30°C
Sample 1-60°C

Sample 2-60°C
Alloy EP-823

Sample 1-90°C

Sample 2-90°C
C6. Stress-Strain Diagram in Neutral Solution using Notched Specimens Tempered for 1.75 Hours

Sample 1-30°C

Sample 2-30°C
C7. Stress-Strain Diagram in Acidic Solution using Smooth Specimens Tempered for 1.25 Hours

Sample 1-30°C

Sample 2-30°C

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C8. Stress-Strain Diagram in Acidic Solution using Smooth Specimens Tempered for 1.75 Hours

Sample 1-30°C

Sample 2-30°C
Alloy EP-823

Sample 1-90°C

Sample 2-90°C

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C9. Stress-Strain Diagram in Acidic Solution using Notched Specimens Tempered for
1.75 Hours

Sample 1-30°C

Sample 2-30°C
Sample 1-60°C

Sample 2-60°C

Sample 3-60°C
C10. Stress-Strain Diagram in Acidic Solution under $E_{\text{cont}}$
Alloy EP-823

Sample 1-90°C

Sample 2-90°C
APPENDIX D

CYCLIC POTENTIODYNAMIC POLARIZATION (CPP) DATA

D1. CPP Data in Neutral Solution

CPP Diagram in 30°C Neutral Solution (Sample 1)

CPP Diagram in 30°C Neutral Solution (Sample 2)

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CPP Diagram in 60°C Neutral Solution (Sample 1)

CPP Diagram in 60°C Neutral Solution (Sample 2)
CPP Diagram in 90°C Neutral Solution (Sample 1)

CPP Diagram in 90°C Neutral Solution (Sample 2)
D2. CPP Data in Acidic Solution

CPP Diagram in 30°C Acidic Solution (Sample 1)

CPP Diagram in 30°C Acidic Solution (Sample 2)
CPP Diagram in 30°C Acidic Solution (Sample 3)

CPP Diagram in 60°C Acidic Solution (Sample 1)
CPP Diagram in 60°C Acidic Solution (Sample 2)

CPP Diagram in 60°C Acidic Solution (Sample 3)
Cyclic Polarization Scan

Alloy EP-823

CPP Diagram in 60°C Acidic Solution (Sample 4)

CPP Diagram in 90°C Acidic Solution (Sample 1)
CPP Diagram in 90°C Acidic Solution (Sample 2)
APPENDIX E

CALCULATION OF MEAN VALUE OF ALL EXPERIMENTAL RESULTS

Hardness of Alloy EP-823 after Austenitizing and Quenching Operations

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<tr>
<th>Specimen</th>
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<th>Average Hardness (Rc)</th>
<th>Overall Average Harness (Rc)</th>
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Hardness of Alloy EP-823 quenching and Tempered for 1.25 Hours

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Hardness of Alloy EP-823 quenching and Tempered for 1.75 Hours

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### Results of MTS Testing using Smooth Specimens

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<th>UTS (ksi)</th>
<th>%El</th>
<th>%RA</th>
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### Results of MTS Testing using Notched Specimens

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## Results of SSR SCC Testing using Smooth Specimens Tempered for 1.25 Hours

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## Results of SSR SCC Testing using Smooth Specimens Tempered for 1.75 Hours

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Results of SSR SCC Testing using Notched Specimens Tempered for 1.75 Hours

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<th>Environment</th>
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<td>90</td>
<td>163.00</td>
<td>1.27</td>
<td>8.82</td>
<td>3.44</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>165.00</td>
<td>1.13</td>
<td>8.96</td>
<td>3.46</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>90</td>
<td>164.00</td>
<td>1.20</td>
<td>8.89</td>
<td>3.45</td>
<td></td>
</tr>
</tbody>
</table>
## Results of SSR SCC Testing using Smooth Specimens under $E_{\text{cont}}$

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Environment (pH ≈ 2-3)</th>
<th>Temperature ($^\circ$C)</th>
<th>$\sigma_f$ (ksi)</th>
<th>%EL</th>
<th>%RA</th>
<th>TTF (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ambient</td>
<td>143.00</td>
<td>20.60</td>
<td>55.00</td>
<td>20.71</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Ambient</td>
<td>147.00</td>
<td>20.50</td>
<td>54.00</td>
<td>17.30</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>Acidic Solution</td>
<td>145.00</td>
<td>20.55</td>
<td>54.50</td>
<td>19.05</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>60</td>
<td>112.50</td>
<td>16.40</td>
<td>32.00</td>
<td>14.06</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>60</td>
<td>109.50</td>
<td>17.60</td>
<td>34.00</td>
<td>14.98</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>60</td>
<td>111.00</td>
<td>17.00</td>
<td>33.00</td>
<td>14.52</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>90</td>
<td>95.00</td>
<td>11.90</td>
<td>25.00</td>
<td>10.52</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>90</td>
<td>93.00</td>
<td>11.60</td>
<td>24.00</td>
<td>10.60</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td>90</td>
<td>94.00</td>
<td>11.75</td>
<td>24.50</td>
<td>10.56</td>
<td></td>
</tr>
</tbody>
</table>

## Results of the CPP Testing in Neutral and Acidic Solutions

<table>
<thead>
<tr>
<th>Environment (Avg. pH)</th>
<th>Specimen</th>
<th>Temperature ($^\circ$C)</th>
<th>Critical Potentials (mV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>$E_{\text{corr}}$</td>
</tr>
<tr>
<td>Neutral Solution</td>
<td>1</td>
<td>30</td>
<td>-825</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>30</td>
<td>-425</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>60</td>
<td>-620</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>60</td>
<td>-840</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>60</td>
<td>-850</td>
</tr>
<tr>
<td>Acidic Solution</td>
<td>1</td>
<td>90</td>
<td>-760</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>90</td>
<td>-800</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>90</td>
<td>-780</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>90</td>
<td>-500</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>30</td>
<td>-500</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>30</td>
<td>-500</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>60</td>
<td>-500</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>60</td>
<td>-530</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>90</td>
<td>-564</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>90</td>
<td>-547</td>
</tr>
<tr>
<td>Mean</td>
<td>1</td>
<td>90</td>
<td>-460</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>90</td>
<td>-370</td>
</tr>
</tbody>
</table>

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APPENDIX F

UNCERTAINTY ANALYSES OF EXPERIMENTAL RESULTS

A more precise method of estimating uncertainty in experimental results has been presented by Kline and McClintock. The method is based on a careful specification of the uncertainties in the various primary experimental measurements.\(^{(91)}\) For example, the maximum load-cell reading of the slow-strain-rate (SSR) unit is 7500 lbs ± 0.3% lbs.

When the plus or minus notation is used to designate the uncertainty, the person making this designation is stating the degree of accuracy with which he or she believes the measurement has been made. It is notable that this specification is in itself uncertain because the experiment is naturally uncertain about the accuracy of these measurements.\(^{(91)}\)

If a very careful calibration of an instrument has been performed recently, with standards of very high precision, then the experimentalist will be justified in assigning a much lower uncertainty to measurements than if they were performed with a gage or instrument of unknown calibration history.\(^{(91)}\)

Most of the instruments in the Materials Performance Laboratory (MPL) were calibrated on a regular basis by Bechtel Nevada using standards with very high precision. Thus, it is expected that the resultant data presented in this dissertation would have very insignificant uncertainty. The uncertainties in the results of this investigation are
calculated by using the Kline and McClintock Method. The equation used for this method is given below.

\[
W_R = \left[ \left( \frac{\partial R}{\partial x_1} w_1 \right)^2 + \left( \frac{\partial R}{\partial x_2} w_2 \right)^2 + \cdots + \left( \frac{\partial R}{\partial x_n} w_n \right)^2 \right]^{\frac{1}{2}} \quad \cdots \cdots \cdots \quad (F^{91})
\]

Where, \( W_R \) = the uncertainty in the results

\( R \) = the given function of the independent variables \( x_1, x_2, \ldots, x_n \)

\( R = R(x_1, x_2, \ldots, x_n) \)

\( w_1, w_2, \ldots, w_n \) = the uncertainty in the independent variables

F.1 Uncertainty Calculation in MTS Results

The results generated from the MTS testing are stress (\( \sigma \)), percentage elongation (%El), and percentage reduction in area (%RA). The stress is based on the load (\( P \)) and the initial cross-sectional area (\( A_i \)) of the tested specimen. The %El is based on the change in length (\( \Delta l \)) during the testing and the %RA is based on the initial and final cross-sectional areas (\( A_i \) and \( A_f \)). The magnitude of \( P \) was obtained from the load-cell of the MTS unit. However, the values for \( \Delta l, A_i, \) and \( A_f \) were calculated based measurements by a caliper. The uncertainties in load-cell and caliper were ± 0.03% lbs and ± 0.001 in, respectively, obtained from the calibration. The uncertainty in the initial notched diameter was ± 0.001, which was provided by the manufacturer and the uncertainty in the final notched diameter was ± 0.001 obtained by using the caliper.

F.1.1 Calculation of Uncertainty in Stress (\( \sigma \))

\( \sigma = \sigma (P, A_i) \)
\[ A_i = A_i(D_i) \]

Uncertainty in load-cell = ± 0.03% lb

Uncertainty in caliper = ± 0.001 in

Sample calculation:

For yield stress (YS) = 111 ksi

Corresponding average yield load (YL) for the specimen tempered for 1.25 hr = 5449 lbs

Uncertainty in load (\(\mu P\)) = 5449*0.0003

= ± 1.64

Uncertainty in cross-sectional area (\(\mu A_i\)) for the smooth specimen:

Initial Diameter (\(D_i\)) = 0.25 in

Uncertainty in diameter (\(\mu D\)) = ± 0.001 in

\[
\text{Area (} A_i \text{)} = \frac{\pi D_i^2}{4}
\]

= 0.049

\[
\frac{dA_i}{dD_i} = \frac{\pi D_i}{2}
\]

= 0.393

Uncertainty in area, \(\mu A_i\) = \[ \left( \frac{dA_i}{dD_i} \cdot \mu D_i \right)^2 \]

= 0.393*0.001

= ± 0.000393

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Uncertainty in stress, \( u = \left[ \left( \frac{\partial \sigma}{\partial P} \right)^2 + \left( \frac{\partial \sigma}{\partial A_i} \right)^2 \right]^{\frac{1}{2}} \) \( \ldots \) (F.1.1)

\[
\sigma = \frac{P}{A_i}
\]

\[
\frac{\partial \sigma}{\partial P} = \frac{1}{A_i}
\]

= 20.41

\[
\frac{\partial \sigma}{\partial A_i} = -\frac{P}{A_i^2}
\]

= -2269471.05

Now providing all the numerical values in equation F.1.1 obtained from the calculation, it is found that,

\[ u = \left[ (20.41 \times 1.64)^2 + (-2269471.05 \times 0.000393) \right]^{\frac{1}{2}} \]

= 892.53 psi

= ± 0.89 ksi

Similarly, uncertainties in all stresses (YS and UTS) obtained from the MTS were calculated using Mathcad, as shown in the tables of the experimental results.

F.1.2 Calculation of Uncertainty in Percentage Elongation (\( \omega \% El \))

Sample calculation:

Change in length (\( \Delta l \)) = 0.2107 in

Gage length (l) = 1 in
\[
\%\text{El} = \frac{\Delta l}{l} \cdot 100
\]

Uncertainty in \(\Delta l\) \((u_{\Delta l})\) = \pm 0.001

Uncertainty in \(\%\text{El}\) \((u_{\%\text{El}})\),

\[
u_{\%\text{El}} = \left(\frac{d\%\text{El}}{d\Delta l} u_{\Delta l}\right)^{1/2}
\] .......................... (F.1.2)

\[
\frac{d\%\text{El}}{d\Delta l} = \frac{100}{l} = 100
\]

Providing all the calculated values in equation F.1.2, it is found that,

\[
u_{\%\text{El}} = \left[(100 \times 0.001)^2\right]^{1/2}
\]

\(u_{\%\text{El}} = \pm 0.1\)

Similarly, all uncertainty calculations of \(\%\text{El}\) were performed using Mathcad, as presented in the resultant tables.

F.1.3 Calculation of Uncertainty in Percentage Reduction in Area \((u_{\%\text{RA}})\)

Sample calculation:

For \(\%\text{RA} = 60.00\%\)

Uncertainty in initial cross-sectional area \((u_{A_i})\) for the smooth specimen:

Initial Diameter \((D_i) = 0.25\) in

Uncertainty in initial diameter,

\((u_{D_i}) = \pm 0.001\) in

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Area \( A_i \) = \( \frac{\pi D_i^2}{4} \)

\[
= 0.049
\]

\[
\frac{dA_i}{dD_i} = \frac{\pi D_i}{2}
\]

\[
= 0.393
\]

Uncertainty in initial cross-sectional area,

\[
\sigma A_i = \left[ \left( \frac{dA_i}{dD_i} \sigma D_i \right)^2 \right]^{1/2}
\]

\[
= 0.393 \times 0.001
\]

\[
= \pm 0.000393
\]

Uncertainty in final cross-sectional area \( \sigma A_f \) for the smooth specimen:

Final Diameter \( D_i = 0.158 \) in

Uncertainty in final diameter \( \sigma D_i \),

\[
= \pm 0.001 \text{ in}
\]

Area \( A_f \) = \( \frac{\pi D_f^2}{4} \)

\[
= 0.02
\]

\[
\frac{dA_f}{dD_f} = \frac{\pi D_f}{2}
\]

\[
= 0.248
\]
Uncertainty in final cross-sectional area,

\[ u_{A_f} = \left[ \left( \frac{dA_f}{dD_f} u_{D_f} \right)^2 \right]^{\frac{1}{2}} \]

\[ = 0.248 \times 0.001 \]

\[ = 0.000248 \]

Uncertainty in \( \%RA \),

\[ u_{\%RA} = \left[ \left( \frac{\partial \%RA}{\partial A_i} u_{A_i} \right)^2 + \left( \frac{\partial \%RA}{\partial A_f} u_{A_f} \right)^2 \right]^{\frac{1}{2}} \] \hspace{1cm} (F.1.3)

\[ \%RA = \left( \frac{A_i - A_f}{A_i} \right) \times 100 \]

\[ = \left( 1 - \frac{A_f}{A_i} \right) \times 100 \]

\[ \frac{\partial \%RA}{\partial A_i} = \frac{100A_f}{A_i^2} \]

\[ = 832.99 \]

\[ \frac{\partial \%RA}{\partial A_f} = - \frac{100}{A_i} \]

\[ = -2040.82 \]

Now assigning all the calculated values in equation F.1.3, it is found that,

\[ u_{\%RA} = \left[ (832.99 \times 0.000393)^2 + (-2040.82 \times 0.000248)^2 \right]^{\frac{1}{2}} \]

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Similarly, uncertainty in %RA was calculated using Mathcad, as presented in the resultant tables of the results.

F.1 Uncertainty Calculation in Constant-load (CL) Results

Uncertainty Calculation in Applied Stress ($\sigma_a$):
The uncertainty in applied stresses was calculated using equation F.1.1 and is shown in the resultant tables of the results.

Uncertainty Calculation in Threshold Stress ($\sigma_{th}$):
The uncertainty in the $\sigma_{th}$ was introduced from the $\sigma_a$ and the dial indicator. The uncertainty in the dial is \( \pm 0.0005 \) in which corresponds to \( \pm 0.57 \) ksi. Therefore, the uncertainty in the $\sigma_{th}$ for the smooth specimen,

\[
= \sqrt{0.82^2 + 0.57^2} = 0.998649 = \pm 1 \text{ ksi}
\]

The uncertainty in the $\sigma_{th}$ for the notched specimen,

\[
= \sqrt{1.22^2 + 1.16^2} = 1.68 \text{ ksi}
\]

The calculated uncertainties in the $\sigma_{th}$ have been shown in the result.

Uncertainty Calculation in Time-To-Failure (TTF):

\[
= 0.60
\]
The uncertainty in the automatic timer of the CL test setup is ± 0.50 which is shown in the resultant tables of the CL results.

F.3 Uncertainty Calculation in Slow-Strain-Rate (SSR) Testing

F.3.1 Uncertainty Calculation in True Failure Stress ($\sigma_f$)

The uncertainty in the $\sigma_f$ is based on the failure load ($P_f$) and the final cross-sectional area ($A_f$) of the tested specimen. The uncertainty in the $\sigma_f$ was calculated based on the $P_f$ and $A_f$ using the equation F.1.1. The uncertainty results are shown in the relevant tables.

F.3.2 Uncertainty Calculation in Percentage Elongation ($u_{\%El}$)

The uncertainty in the $\%El$ was calculated using the equation F.1.2 and shown in the results section.

F.3.3 Uncertainty Calculation in Percentage Reduction in Area ($u_{\%RA}$)

The uncertainty in the $\%RA$ was calculated based on the equation F.1.3 and presented in the resultant tables of the results.

F.3.4 Uncertainty Calculation in Time-To-Failure (TTF)

The TTF is obtained from the Field Point software of the slow-strain-rate (SSR) unit, which is accurate up to $\frac{1}{100}$th of a second in finding the TTF. Therefore, the uncertainty of the TTF in the SSR testing is negligible.

F.4 Uncertainty Calculation in Cyclic Potentiodynamic Polarization (CPP) Testing

The accuracy of the potentiostat provided by the manufacturer is ± 0.003 mV within a range of 1 mV.
Sample calculation: For corrosion potential ($E_{\text{corr}}$) = -625 mV

The uncertainty in $E_{\text{corr}}$ = 625 * 0.003

\[ = 1.875 \]

\[ = \pm 1.88 \text{ mV} \]

Similarly, uncertainties in all critical potentials were calculated and presented in the resultant tables.
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