Characterization of alloy C-276 as a structural material for high-temperature heat exchangers

Narendra V Kothapalli

University of Nevada, Las Vegas

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CHARACTERIZATION OF ALLOY C-276 AS A STRUCTURAL MATERIAL FOR
HIGH-TEMPERATURE HEAT EXCHANGERS

by

Narendra V. Kothapalli

Bachelor of Science in Engineering Technology
Birla Institute of Technology & Sciences, Pilani, India
December 2001

A thesis submitted in partial fulfillment
of the requirements for the

Master of Science Degree in Mechanical Engineering
Department of Mechanical Engineering
Howard R. Hughes College of Engineering

Graduate College
University of Nevada, Las Vegas
August 2005

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The Thesis prepared by

Narendra V. Kothapalli

Entitled

Characterization of Alloy C-276 as a Structural Material for High-Temperature Heat Exchangers

is approved in partial fulfillment of the requirements for the degree of

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Examination Committee Chair

Dean of the Graduate College

Examination Committee Member

Examination Committee Member

Graduate College Faculty Representative
ABSTRACT

Characterization of Alloy C-276 as a Structural Material for High-Temperature Heat Exchangers

by

Narendra V. Kothapalli

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

This investigation was focused on characterizing the high-temperature tensile behavior and corrosion susceptibility of nickel-based Alloy C-276, a leading structural material for heat-exchangers in nuclear hydrogen generation. The tensile properties, evaluated at different temperatures, indicate that both the yield strength and ultimate tensile strength were gradually reduced with increasing temperature. However, the ductility parameters were not significantly influenced by the testing temperature. While no failures were observed in stress corrosion cracking (SCC) tests in an acidic solution under a constant-load, the true failure stress ($\sigma_f$) was significantly reduced under a slow-strain-rate condition using both smooth and notched specimens. The critical potentials, determined in a similar environment by an electrochemical method, became more active at the elevated temperature. Cracking was enhanced at anodic and cathodic applied potentials, showing reduced failure strain and $\sigma_f$. The fractographic evaluations of the primary fracture surface revealed dimpled microstructures, indicating ductile failures.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>vi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>vii</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>ix</td>
</tr>
<tr>
<td>CHAPTER 1 INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>CHAPTER 2 MATERIAL, TEST SPECIMENS AND ENVIRONMENTS</td>
<td>7</td>
</tr>
<tr>
<td>2.1 Test Material</td>
<td>7</td>
</tr>
<tr>
<td>2.2 Test Specimens</td>
<td>9</td>
</tr>
<tr>
<td>2.3 Test Environments</td>
<td>13</td>
</tr>
<tr>
<td>CHAPTER 3 EXPERIMENTAL PROCEDURES</td>
<td>14</td>
</tr>
<tr>
<td>3.1 Tensile Testing</td>
<td>14</td>
</tr>
<tr>
<td>3.2 Constant-Load Testing</td>
<td>17</td>
</tr>
<tr>
<td>3.3 Slow-Strain-Rate Testing</td>
<td>19</td>
</tr>
<tr>
<td>3.4 Cyclic Potentiodynamic Polarization Testing</td>
<td>24</td>
</tr>
<tr>
<td>3.5 SCC Testing at Controlled Potentials</td>
<td>26</td>
</tr>
<tr>
<td>3.6 Metallography by Optical Microscope</td>
<td>28</td>
</tr>
<tr>
<td>3.7 Fractographic Evaluation by SEM</td>
<td>30</td>
</tr>
<tr>
<td>CHAPTER 4 RESULTS</td>
<td>32</td>
</tr>
<tr>
<td>4.1 Tensile Properties at Ambient Temperature</td>
<td>32</td>
</tr>
<tr>
<td>4.2 Tensile Properties at Elevated Temperatures</td>
<td>32</td>
</tr>
<tr>
<td>4.3 SCC Testing at Constant-Load</td>
<td>34</td>
</tr>
<tr>
<td>4.4 SCC Testing under SSR Condition</td>
<td>36</td>
</tr>
<tr>
<td>4.5 Localized Corrosion using CPP</td>
<td>39</td>
</tr>
<tr>
<td>4.6 SSR Testing under $E_{\text{cont}}$</td>
<td>42</td>
</tr>
<tr>
<td>4.7 Metallographic Evaluations</td>
<td>47</td>
</tr>
<tr>
<td>4.8 Fractographic Evaluations</td>
<td>48</td>
</tr>
<tr>
<td>CHAPTER 5 DISCUSSION</td>
<td>52</td>
</tr>
<tr>
<td>CHAPTER 6 SUMMARY AND CONCLUSIONS</td>
<td>55</td>
</tr>
</tbody>
</table>

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LIST OF TABLES

Table 2-1  Physical Properties of Hastelloy C-276 ............................................. 8
Table 2-2  Chemical Composition of Hastelloy C-276 (wt %) .................................. 9
Table 2-3  Ambient-Temperature Mechanical Properties of Hastelloy C-276 ............ 9
Table 2-4  Chemical Composition of Test Solutions (gram/liter) ............................... 13
Table 3-1  Minimum Time to Reach the Desired Temperature ................................. 17
Table 4-1  Comparison of Room Temperature Tensile Properties ............................ 32
Table 4-2  Tensile Properties of Alloy C-276 at Different Temperatures .................... 34
Table 4-3  Constant-Load SCC Test Results ......................................................... 36
Table 4-4  SSR Test Results ................................................................................... 38
Table 4-5  Critical Potentials vs. Temperature ......................................................... 41
Table 4-5  Results of SSR Testing with and without Cathodic $E_{cont}$ ......................... 43
Table 4-6  Results of SSR Testing with and without Anodic $E_{cont}$ ......................... 47
LIST OF FIGURES

Figure 1-1  The S-I Water Splitting Cycle for Hydrogen Generation ........................................ 3
Figure 2-1  Stress Concentration Factors for Grooved Shafts ........................................................ 10
Figure 2-2  Schematic view of the Tensile Specimens ................................................................... 11
Figure 2-3  Schematic view of Polarization Specimen .................................................................... 12
Figure 3-1  Tensile Testing Setup .................................................................................................. 16
Figure 3-2  Constant-load Test Setup ............................................................................................ 18
Figure 3-3  A Typical Calibration Curve of a Proof Ring ............................................................... 19
Figure 3-4  CERT Machine for SSR Testing .................................................................................. 20
Figure 3-5  SSR Test Setup .......................................................................................................... 21
Figure 3-6  Load Frame Compliance Test ....................................................................................... 22
Figure 3-7  Electrochemical Testing Setup .................................................................................... 24
Figure 3-8  Luggin Probe Arrangement .......................................................................................... 25
Figure 3-9  Standard Potentiodynamic Polarization Plot (ASTM G 5) ......................................... 26
Figure 3-10 Spot-Welded Cylindrical Specimen ........................................................................... 27
Figure 3-11 HE Test Setup ............................................................................................................ 28
Figure 3-12 Leica Optical Microscope .......................................................................................... 30
Figure 3-13 Scanning Electron Microscope .................................................................................. 31
Figure 4-1  Stress-Strain Diagram vs. Temperature ........................................................................ 33
Figure 4-2  Comparison of s-e diagrams in Air and S-I solution (Smooth Specimen) .................. 37
Figure 4-3  Comparison of s-e diagrams in Air and S-I solution (Notch Specimen) ................. 38

vii

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Figure 4-4  CPP Diagram in Room Temperature S-I Solution ....................................... 40
Figure 4-5  CPP Diagram in 90°C S-I Solution ................................................................ 40
Figure 4-6  $E_{\text{cont}}$ vs. Temperature ................................................................. 41
Figure 4-7  $E_{\text{pit}}$ vs. Temperature ........................................................................ 42
Figure 4-8  s-e Diagram with and without Cathodic $E_{\text{cont}}$.............................. 43
Figure 4-9 (a) The effect of Cathodic $E_{\text{cont}}$ on $\%$EI ................................................. 44
Figure 4-9 (b) The effect of Cathodic $E_{\text{cont}}$ on RA .............................................. 44
Figure 4-9 (c) The effect of Cathodic $E_{\text{cont}}$ on TTF ............................................. 45
Figure 4-9 (d) The effect of Cathodic $E_{\text{cont}}$ on $\sigma_f$ .............................................. 45
Figure 4-10 s-e Diagram with and without Anodic $E_{\text{cont}}$ ............................. 46
Figure 4-11 Optical Micrograph of Sectioned Bar Material .................................. 47
Figure 4-12 SEM Micrographs of Smooth Tensile Specimen ................................. 49
Figure 4-13 SEM Micrographs of Smooth Specimen used in SSR Testing .......... 50
Figure 4-14 SEM Micrographs of Notch Specimen used in SSR Testing .............. 51
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CHAPTER 1

INTRODUCTION

Fossil fuels including oil and gas have been known to be major energy sources for quite sometime. These types of fuels are extensively being used in residential, commercial, transportation and industrial applications. Due to the growing demand for oil and gas, their productions are expected to hit the maximum levels soon after 2010 with their supplies dwindling afterwards, thus causing significant shortage of fuels. Since 55% of the fuel needs in the United States are met by import and the oil and gas prices are increasing at alarming rates, there are needs for the development of non-fossil fuels. In view of this rationale, the US Department of Energy (USDOE) has initiated a major effort to develop alternate sources of energy. One such energy is hydrogen that may be commercially generated thus, eliminating the dependence on foreign oil and gas.

Hydrogen has the potential to revolutionize the way Americans produce, store, and utilize energy. Hydrogen would be attractive as an energy carrier if it can be demonstrated that it could be produced cleanly and cost-effectively on a large scale. Hydrogen, along with electricity, is expected to dominate the world energy system in the long term. As the US and the rest of the world transitions to a hydrogen economy, hydrogen will be used increasingly by the transportation, residential, industrial, and commercial sectors of the energy market. A promising alternative source of hydrogen is to use process heat from a high-temperature nuclear reactor to drive a set of chemical
reactions that can produce hydrogen. Thermochemical production of hydrogen involves the dissociation of water into hydrogen and oxygen through chemical reactions at high temperatures. Ideally, this dissociation can be accomplished directly (thermolysis). However, this process requires temperatures in excess of 2500°C. Since these temperatures are impractical, thermochemical water-splitting cycles can be used to generate hydrogen at lower temperatures.

A thermochemical water-splitting cycle consists of a series of chemical reactions, some at rather high temperatures. Chemicals are chosen to create a closed loop, where water can be fed to the process, oxygen and hydrogen gas are collected, and all other reactants are regenerated and recycled. Recent studies conducted through the Nuclear Energy Research Initiative (NERI) have identified more than 100 thermochemical water splitting cycles. A few of the most promising cycles have been selected for further research and development due to the simplicity of the cycle, the efficiency of the process, and the ability to separate a pure hydrogen product. The two leading systems for production of nuclear hydrogen are the sulfur-iodine (S-I) thermochemical cycle and high-temperature-electrolysis (HTE). A second thermochemical process that may operate efficiently at lower temperatures is the calcium-bromine (Ca-Br) cycle, which is also under consideration by the USDOE.

1.1 S-I Cycle

The sulfur-iodine (S-I) process is a thermochemical water splitting cycle developed at General Atomics, and first described in the mid 1970's. This process consists of three chemical reactions, which sum to the dissociation of water. As shown in the Figure 1-1, the S-I cycle uses iodine (I₂) and sulfur dioxide (SO₂) as chemical catalysts to split water.
First, water reacts with I\textsubscript{2} and SO\textsubscript{2} at 120°C to form hydrogen iodide (HI) and sulfuric acid (H\textsubscript{2}SO\textsubscript{4}), as shown below. Subsequently, HI and H\textsubscript{2}SO\textsubscript{4} are separated from each other. I\textsubscript{2} and SO\textsubscript{2} are then recovered as byproducts from the breakdown of HI and H\textsubscript{2}SO\textsubscript{4} and recycled. Hydrogen and oxygen gases are then collected. The reactions associated with the S-I cycle are given below.

\[
\begin{align*}
\text{I}_2 + \text{SO}_2 + 2\text{H}_2\text{O} &\rightarrow 2\text{HI} + \text{H}_2\text{SO}_4 \quad \text{(temperatures ~ 120°C)} & \text{(Reaction 1.1)} \\
\text{H}_2\text{SO}_4 &\rightarrow \text{H}_2\text{O} + \text{SO}_2 + \frac{1}{2}\text{O}_2 \quad \text{(temperatures > 800°C)} & \text{(Reaction 1.2)} \\
2\text{HI} &\rightarrow \text{H}_2 + \text{I}_2 \quad \text{(temperatures ~ 450°C)} & \text{(Reaction 1.3)}
\end{align*}
\]

As shown above, the reaction that requires the greatest heat input is the thermal decomposition of H\textsubscript{2}SO\textsubscript{4}, at temperatures in the vicinity of 800°C. High temperatures are necessary to produce large quantities of hydrogen in a cost-effective manner, because the
efficiency of the process (hydrogen produced per unit of heat input) decreases rapidly at reduced temperatures.

1.2 Ca-Br Cycle

The calcium-bromine (Ca-Br) cycle for hydrogen production is a variant of UT-3 thermochemical cycle investigated by the Japan Atomic Energy Research Institute (JAERI). The Ca-Br cycle is designed to operate at temperatures lower than that of the S-I system. The UT-3 cycle is based on two pairs of chemical reactions. The first two reactions ensure the formation of hydrobromic acid releasing oxygen, and the other two are used to ensure the reduction of water by bromide.

\[
\text{CaO} + \text{Br}_2 \rightarrow \text{CaBr}_2 + \frac{1}{2}\text{O}_2 \quad \text{(temperatures ~ 550°C)} \tag{Reaction 1.4}
\]

\[
\text{CaBr}_2 + \text{H}_2\text{O} \rightarrow \text{CaO} + 2\text{HBr} \quad \text{(temperatures ~ 730°C)} \tag{Reaction 1.5}
\]

\[
\text{Fe}_3\text{O}_4 + 8\text{HBr} \rightarrow 3\text{FeBr}_2 + 4\text{H}_2\text{O} + \text{Br}_2 \quad \text{(temperatures~220°C)} \tag{Reaction 1.6}
\]

\[
3\text{FeBr}_2 + 4\text{H}_2\text{O} \rightarrow \text{Fe}_3\text{O}_4 + 6\text{HBr} + \text{H}_2 \quad \text{(temperatures~ 650°C)} \tag{Reaction 1.7}
\]

The thermodynamics of these reactions have been found favorable. However, the hydrogen production efficiency of the process is limited to about 40% due to the melting point of CaBr\(_2\) at 760°C.

1.3 High-Temperature-Electrolysis

High-temperature electrolysis (HTE) uses the technology of solid-oxide-fuel-cells (SOFCs) to split steam into hydrogen and oxygen. These cells operate at temperatures of 750 to 900°C, and have the electrical potential reversed from that of a typical fuel-cell mode. Nuclear hydrogen production using HTE utilizes the energy input from nuclear-reactor process heat to reduce the electrical power required to split water into hydrogen and oxygen by electrolysis, thereby improving the overall efficiency of the process.
The HTE system is conceptually simpler in comparison to those of the thermochemical cycles currently being considered for nuclear hydrogen generation. In the HTE system, water is heated to superheated steam in a series of steam generators and superheaters, electrolyzed into hydrogen and oxygen in a water-containing process stream in the cell, and then separated into its constituents by condensation and/or the use of organic membranes. The only other major components are the heat exchangers used to recover the waste heat from the hydrogen and oxygen product streams prior to their discharge.

1.4 Objective of this Investigation

In light of the preceding discussions, it is obvious that the proposed heat-exchanger system may experience temperatures ranging from ambient to 900°C, presence of damaging chemical species, and a pH of less than one. Under such hostile operating conditions, the structural materials may become susceptible to environment-induced degradations such as general corrosion, localized corrosion, stress corrosion cracking (SCC), and/or hydrogen embrittlement (HE). In addition, they can undergo plastic deformation at elevated temperatures. Thus, the paramount materials needs for heat-exchanger applications are centered around the availability of suitable structural materials possessing superior corrosion resistance and enhanced high temperature tensile properties.

Hastelloy C-276 is a wrought nickel (Ni)-based superalloy possessing high strength and excellent corrosion resistance in a wide variety of hostile environments. The high strength and corrosion resistance of this alloy is attributed to the continuous matrix of face centered cubic (FCC) solid solution of chromium (Cr), cobalt (Co), molybdenum
(Mo), tungsten (W), iron (Fe) etc. in large amounts. [1] This material has been extensively used as equipment in chemical industries, manufacturing of high-temperature autoclaves, test chambers etc. that warrants exceptional corrosion resistance. In view of these superior properties, Hastelloy C-276 has been identified to be a candidate structural material for high-temperature heat exchangers (HTHXs).

It is obvious that the structural materials to be used in a HTHX must possess excellent resistance to high-temperature deformation and suitable corrosion resistance in an aggressive environment relevant to the S-I cycle. Therefore, this thesis is focused on the evaluation of both the metallurgical behavior of Alloy C-276 and its corrosion characteristics in a simulated aqueous test environment. The results of SCC tests using state of the art experimental techniques have been presented in this thesis. The susceptibility to localized corrosion using electrochemical polarization techniques has also been determined. Tensile testing of Alloy C-276 has been performed at ambient and elevated temperatures. The effect of applied potential (anodic/cathodic) on the cracking susceptibility of Alloy C-276 has also been studied in a similar environment. The metallurgical microstructures have been determined by optical microscopy. The extent and morphology of failure in the tested specimens have been evaluated by scanning electron microscopy (SEM). The comprehensive test results including the fundamental understanding on the metallurgical and corrosion behavior of Alloy C-276 has been presented in this thesis.
2.1 Test Material

The identification and selection of structural materials for HTHX will depend on their abilities to meet the design and service requirements, and be fabricated and assembled according to the industry standard. The capabilities of these materials to meet the desired requirements will be determined by their mechanical, physical and corrosion properties, as well as their forming, shaping and bonding capabilities, as determined by applicable methods.

The material tested in this investigation is a Ni-based alloy commonly known as Hastelloy C-276 (UNS N10276) manufactured by the Haynes International Inc. The Ni and Ni-based alloys have been extensively used in modern-day industrial applications due to their exceptional capabilities to withstand severe operating conditions including highly corrosive environments, elevated temperatures, high stresses, and a combination of all these factors. The enhanced ductility and toughness in Ni-containing austenitic superalloys such as Alloy C-276 may be attributed to their stable FCC phase structures maintained even up to their melting temperatures. The presence of substantial Cr content in these alloys can render them highly resistant to corrosion in the normal atmosphere, sea water and acidic environments. They are highly resistant to corrosive degradations in sulfuric acid, hydrochloric acid and chlorinated organics. The presence of Mo can also
promote significant resistance to localized corrosion in many hostile environments. Further, the intermetallic phases formed with other alloying elements enable these alloys to withstand high temperature deformation for applications in refineries and deep drilling of oil and gas wells.

Alloy C-276 is readily weldable and can outperform many commercially available stainless steels. This material can be used in the as-welded conditions, eliminating the need for postweld thermal treatments. The presence of significantly low carbon and silicon contents in Alloy C-276 is beneficial in preventing grain boundary precipitation/segregation during heating cycles associated with the welding operation. The typical physical properties of Alloy C-276 are given in Table 2-1.

<table>
<thead>
<tr>
<th>Physical Property</th>
<th>Temperature, °C</th>
<th>Metric Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>22</td>
<td>8.89 g/cm³</td>
</tr>
<tr>
<td>Melting Range</td>
<td>1323-1371</td>
<td>--</td>
</tr>
<tr>
<td>Electrical Resistivity</td>
<td>24</td>
<td>1.30 microhm-m</td>
</tr>
<tr>
<td>Mean Coefficient of Thermal Expansion</td>
<td>24-93</td>
<td>11.2 x 10⁻⁶ m/m.K</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td>38</td>
<td>10.2 W/m.K</td>
</tr>
<tr>
<td>Specific Heat (Calculated)</td>
<td>Room</td>
<td>427 J/Kg.K</td>
</tr>
</tbody>
</table>

The flat and round bars of Hastelloy C-276 were received from the Haynes International Inc. in a heat treated condition. They were solution annealed at 2050° F (1121° C) followed by rapid cooling, thus, providing a fully austenitic microstructure. The chemical composition and the tensile properties of the as-received bars of Alloy C-276 are given in Tables 2-2 and 2-3, respectively.
Table 2-2 Chemical Composition of Hastelloy C-276 (wt %) [4]

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>C</th>
<th>Co</th>
<th>Cr</th>
<th>Fe</th>
<th>Mn</th>
<th>Mo</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>V</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>2760-1-3939</td>
<td>0.005</td>
<td>1.42</td>
<td>15.87</td>
<td>5.43</td>
<td>0.52</td>
<td>15.62</td>
<td>Bal</td>
<td>0.007</td>
<td>0.003</td>
<td>0.02</td>
<td>0.15</td>
<td>3.56</td>
</tr>
</tbody>
</table>

Table 2-3 Ambient-Temperature Mechanical Properties of Hastelloy C-276 [4]

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Yield Strength, Ksi (Mpa)</th>
<th>Ultimate Strength, Ksi (Mpa)</th>
<th>%El</th>
</tr>
</thead>
<tbody>
<tr>
<td>2760-1-3939</td>
<td>50 (345)</td>
<td>114 (786)</td>
<td>64</td>
</tr>
</tbody>
</table>

2.2. Test Specimens

The specimens tested in this investigation include cylindrical specimens (with/without notch) of different dimensions for tensile and SCC testing, and small cylindrical specimens for electrochemical polarization studies. Smooth cylindrical specimens having 4-inch overall length, 1-inch gage length and 0.25-inch gage diameter were used for tensile properties, and SCC evaluations in a simulated S-I environment.

For SCC testing under controlled electrochemical potentials (anodic/cathodic), the dimension of the smooth cylindrical specimen was modified to reduce the gage length (l) and diameter (d), while maintaining the l/d ratio at 4, as prescribed by the ASTM Designation E 8. [5] The notched specimen had a circular notch of 0.156-inch diameter at
the center of the gage section with a root radius of 0.0468-inch. The notched specimens were used to study the effect of stress concentration on the tensile properties and the SCC susceptibility of Alloy C-276. The stress concentration factor ($K_t$) corresponding to this notch configuration was approximately 1.45. The magnitude of $K_t$ \[^6\] was determined from the following calculations, and the plot shown in Figure 2-1.

$$\frac{D}{d} = \frac{0.250 \text{ in}}{0.156 \text{ in}} \quad \text{(Equation 3.1)}$$

$$\frac{D}{d} = 1.60$$

$$\frac{r}{d} = \frac{0.0468 \text{ in}}{0.156 \text{ in}} \quad \text{(Equation 3.2)}$$

Where,

$D$ = gage diameter,

$d$ = notch diameter

$r$ = radius of curvature at the root of the notch

![Figure 2-1 Stress Concentration Factors for Grooved Shafts \[^7\]](image-url)
Specimens for localized corrosion studies were also manufactured from the same heat of material. These specimens had smaller length and diameter of 0.4881-inch and 0.37-inch, respectively with a central blind hole for holding it with a stainless steel rod. The geometric configurations of all types of tested specimen in this investigation are shown in Figures 2-2 and 2-3.

(a) Smooth Specimen
(b) Notched Specimen

Figure 2-2 Schematic view of the Tensile Specimens

Figure 2-3 Schematic view of Polarization Specimen
2.3. Test Environment

Environment can have a profound influence on the performance of the heat exchanger materials. A material may undergo destruction or deterioration due to its reaction with the environment depending on the type of environment. As indicated earlier, there are three viable options to produce hydrogen using nuclear power. However, this investigation is concentrated on the S-I cycle only. Since hydrogen is proposed to be generated by thermochemical reactions involving sulfuric acid (H₂SO₄) leading to the production of oxygen and sulfur dioxides, different types of corrosion-related degradations can be experienced by a candidate heat exchanger material such as Alloy C-276.

Three different types of degradations that can be encountered by Alloy C-276 in a prototypic environment are general corrosion, localized corrosion (pitting and crevice), SCC and/or hydrogen embrittlement (HE). The susceptibility to these degradation modes may be influenced by factors such as the type of material, a potent environment and a critical stress level, tensile in nature. In view of the applicability of the S-I cycle, a simulated environment was used in all testing, which consisted of sulfuric acid, sodium iodide and deionized water. The pH of this solution was approximately 1.0. The composition of this test solution is given in Table 2-4.

<table>
<thead>
<tr>
<th>Solution (pH)</th>
<th>NaI</th>
<th>H₂SO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidic (1.0)</td>
<td>10.0</td>
<td>Added to adjust the desired pH</td>
</tr>
</tbody>
</table>

Table 2-4 Chemical Composition of Test Solutions (gram/liter)
CHAPTER 3

EXPERIMENTAL PROCEDURES

The tensile properties of Hastelloy C-276 were evaluated at ambient and elevated temperatures by using an axial/torsional material testing system (MTS). The susceptibility of this alloy to SCC in acidic environment was evaluated by using constant-load testing and slow-strain-rate (SSR) testing techniques at ambient temperature and 90°C. The electrochemical cyclic potentiodynamic polarization (CPP) method was used to evaluate the localized corrosion (pitting/crevice) behavior using a Gamry Potentiostat. Further, fractographic and metallographic evaluations were performed by SEM and optical microscopy, respectively. The detailed experimental techniques are given in the following subsections.

3.1. Tensile Testing

The tensile properties including the yield strength (YS), ultimate tensile strength (UTS), and the ductility in terms of percentage elongation (%El) were evaluated at ambient temperature, 450 and 600°C using the MTS unit model 319.25. The smooth cylindrical specimens were strained in tension at a strain rate of $10^{-3}$/sec according to the ASTM Designation E 8. 5 A minimum of two specimens were tested under each condition and the average value was recorded. 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 diagrams were developed from
these data. The magnitude of the yield strength (YS) at each temperature 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 and %El were also determined from this plot,
and the dimensions of the cylindrical specimen before and after testing.

The MTS unit, 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 can be controlled by
force, displacement, or an external signal from the strain gage. The torsional motion can
be 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 was equipped with an 8-channel signal-conditioning box for
monitoring the strain gages, extensometers, and temperature sensors. Signals from this
box were processed directly by the MTS control software programs (Test Star 790.00 v
4.0E and Test Ware SX v 4.0D) that automatically controlled all signals during testing. A
laser extensometer having a scan rate of 100 scans/sec was added to this MTS unit to
measure the elongation of the gage section of test specimen during plastic deformation
under tensile loading.
The MTS unit was modified to accommodate high-temperature testing in the presence of nitrogen using a ceramic-lined custom-made chamber. The testing temperature inside this chamber was monitored by two K-type thermocouples. A pair of custom-built water-cooled specimen grips made of maraging steel (M250) was attached to the MTS machine to prevent these grips from being heated during testing at elevated temperatures. Temperature profiles were developed to determine the times needed to achieve the desired test temperatures as a part of the furnace calibration process. The resultant times and the environment chamber set point temperatures for different target temperatures in the presence of nitrogen gas at 20 bar on Argon scale are shown in Table 3-1. It should,
however, be noted that testing was performed at 450 and 600°C in the presence of nitrogen.

Table 3-1 Minimum Time to Reach the Desired Temperature

<table>
<thead>
<tr>
<th>Material</th>
<th>Target temperature (°C)</th>
<th>Environmental chamber set point temperature (°C)</th>
<th>Minimum time to reach the target temperature (minutes)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy C-276</td>
<td>100</td>
<td>143</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>363</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>467</td>
<td>55</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>563</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>600</td>
<td>658</td>
<td>50</td>
</tr>
</tbody>
</table>

3.2. Constant-load Testing

A calibrated proof ring was used for constant-load (CL) testing. The proof rings were specifically-designed to meet the National Association of Corrosion Engineers (NACE) standard TM-01-77. Each individually-calibrated proof ring was made by Cortest Inc. and was accompanied by a calibration curve showing the load versus deflection of the ring. The test specimens were loaded under a stress state of uniaxial tension. The ring deflection was measured with a 8-9" diameter micrometer, with the supplied dial indicator providing a check. The proof rings were fabricated from precision-machined alloy steel. Tension on the proof ring was quickly and easily adjusted using a standard wrench on the tension-adjusting screw and lock nut. A thrust bearing distributed the load and prevented seizure. Specimen grips in these proof rings were made of stainless steel, fully-resistant to the testing environments. The environmental test chamber was secured with O-ring seals that prevented any leakage during testing. The environmental chambers
made of highly corrosion-resistant Hastelloy C-276 were used for testing at elevated temperatures. The experimental setup is shown in Figure 3-2.

![Figure 3-2 Constant-load Test Setup](image)

The amount of deflection needed to apply the desired load in the CL testing was determined by use of the calibration curve of each proof ring, as shown in Figure 3-3. The magnitude of the applied stress was based on the ambient temperature tensile YS of the test material. The specimens were loaded at stress values equivalent to different percentages of the test material's YS value, and the corresponding time-to-failure (TTF) was recorded. The determination of the SCC tendency using this technique was based on the TTF for the maximum test duration of 30 days. An automatic timer attached to the test specimen recorded the TTF. The cracking susceptibility was expressed in terms of a threshold stress ($\sigma_{th}$) below which cracking did not occur during the maximum test duration of 30 days.$^9$
3.3. Slow-Strain-Rate Testing

Slow-strain-rate (SSR) testing was performed using a specially-designed system known as a constant-extension-rate-testing (CERT) machine, as shown in Figure 3-4. The CERT machines enabled testing to simulate a broad range of load, temperature, strain-rate and environmental conditions using both mechanical and electrochemical corrosion testing techniques. These machines, designed and manufactured by Cortest Inc., offered accuracy and flexibility in testing the effect of strain rate, providing up to 7500 lbs of load capacity with linear extension rates ranging from $10^{-5}$ to $10^{-8}$ in/sec. To ensure the maximum accuracy in test results, this apparatus was comprised of a heavy duty load-frame that minimized the system compliance while maintaining precise axial alignment.
of the load train. An all-gear drive system provided consistent extension rate. Added features included a quick-hand wheel to apply a pre-load prior to the operation.

![CERT Machine for SSR Testing](image)

**Figure 3-4 CERT Machine for SSR Testing**

The SSR test setup used in this investigation consisted of a top-loaded actuator, testing chamber, linear variable differential transducer (LVDT) and load cell 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 performance of SCC testing by the SSR technique, the load-frame-compliance factor (LFCF), which indicates the deflection in the frame per unit load, was determined by using ferritic Type 430 stainless steel specimens. The generated LFCF data are shown in Figure 3-6. These LFCF values were given to the load frame acquisition system prior to the SCC testing.

The SCC phenomenon is a consequence of two major factors including the applied or residual stress and a potent environment. If the stress is applied to the specimen at a very fast rate while it is exposed to the aqueous environment, the resultant failure may not be different from the typical mechanical deformation produced without an environment. Conversely, if the strain rate is too slow, the resultant failure may simply be attributed to the corrosion damage due to the breakdown of the protective surface film by interaction of the test material with the susceptible environment.
A strain rate of $3.3 \times 10^6$ sec$^{-1}$ was used to evaluate the combined effect of the applied load and the environment, during the SCC testing. The selection of this optimum strain rate was based on the results of SSR testing obtained at the Lawrence Livermore National Laboratory (LLNL). The specimen was continuously strained in tension until fracture, according to the ASTM Designation G 129, in contrast to more conventional SCC test conducted under a sustained loading condition. The application of a slow dynamic straining during the SSR testing to the specimen caused failure that probably might not occur under a constant load or might have taken a prohibitively longer duration to initiate cracks in producing failures in the tested cylindrical specimens.

Load versus displacement and stress versus strain curves were plotted during these tests. Dimensions (length and diameter) of the test specimens were measured before and after testing. The cracking tendency of the test specimen was expressed by the time-to-failure (TTF), and a number of ductility parameters such as $\%E_l$ and $\%R_A$. Further,
the true failure stress ($\sigma_f$) determined from the stress-strain diagram and the final dimensions were taken into consideration to characterize the cracking tendency. The magnitudes of $\% E_l$, $\% RA$ and $\sigma_f$ were calculated using the following equations:

$$\% E_l = \left( \frac{L_f - L_o}{L_o} \right) \times 100$$  \hspace{1cm} \text{(Equation 3.1)}

$$\% RA = \left( \frac{A_o - A_f}{A_o} \right) \times 100$$  \hspace{1cm} \text{(Equation 3.2)}

$$\sigma_f = \frac{P_f}{A_f}$$  \hspace{1cm} \text{(Equation 3.3)}

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

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

Where,

$A_o$ = Initial cross sectional area

$A_f$ = Final 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. Cyclic Potentiodynamic Polarization Testing

The susceptibility of Hastelloy C-276 to localized (pitting and crevice) corrosion was determined by performing CPP experiments in the acidic solution using a Model PCI4 GAMRY potentiostat. The CPP testing was based on a three-electrode-polarization concept, in which the working electrode (specimen) acted as an anode and two graphite electrodes (counter electrodes) acted as cathodes as shown in Figure 3-7. The reference electrode was made of Ag/AgCl solution contained inside a Luggin probe having the test solution that acted as a salt-bridge. The tip of the Luggin probe was placed within a distance of 2 to 3 mm from the test specimen as shown in Figure 3-8.

Figure 3-7 Electrochemical Testing Setup
At the onset of the CPP testing, the corrosion or the open circuit potential ($E_{corr}$) of the specimen was determined with respect to the Ag/AgCl reference electrode, followed by forward and reverse potential scans at the ASTM specified $^{[15]}$ rate of 0.17 mV/sec. An initial delay time of 50 minutes was given for attaining a stable $E_{corr}$ value. The magnitudes of the critical pitting potential ($E_{pit}$) and protection potential ($E_{prot}$), if any, were obtained from the CPP diagram. Prior to the CPP testing, the potentiostat was calibrated according to the ASTM Designation G 5. $^{[16]}$ Calibration of the potentiostat was performed to generate a characteristic potentiodynamic polarization curve (Figure 3-9) using small cylindrical test specimens of ferritic Type 430 stainless steel in 1 Normal (1N) sulfuric acid ($H_2SO_4$) solution at 30°C.
3.5. SCC Testing at Controlled Potentials

It is a well known that structural materials used in many engineering applications may become susceptible to enhanced cracking in a potent environment under tensile loading due to the application of an external potential. In order to verify the effect of externally applied potential on the cracking susceptibility of Alloy C-276, SCC testing was performed in the S-I environment under both anodic and cathodic potentiostatic controls. The mechanisms of cracking, however, are different for SCC testing using anodic and cathodic controlled potentials ($E_{\text{cont}}$).

SCC of a material under cathodic $E_{\text{cont}}$ is commonly known as hydrogen embrittlement (HE), resulting from the generation of atomic hydrogen due to cathodic polarization of the test specimen. The atomic hydrogen (H) can eventually be diffused into the metal lattice, thus causing failure due to the combined effect of applied
stress/stress gradient in the gage section and the presence of increased hydrogen ion (H⁺) concentration. On the contrary, cracking of metallic material under anodic $E_{\text{cont}}$ usually occurs due to the breakdown of protective films on its surface due to the application of a potential above the $E_{\text{pit}}$. The susceptibility to SCC under $E_{\text{cont}}$ can, therefore, be evaluated by applying potential either active (negative) to the $E_{\text{corr}}$ value or noble (positive) to the $E_{\text{pit}}$ value for cathodic and anodic $E_{\text{cont}}$ testing, respectively.

In order to study the effect of $E_{\text{cont}}$, the cylindrical specimens of Alloy C-276 were spot welded with a conductive metallic wire at the top shoulder of the specimen above its gage section. The purpose of this conductive wire was to apply the external potential so that electron flow can occur to induce polarization of the test specimen. The metallic conductive wire was coated with a lacquer to prevent its contact with the test solution during straining of the specimen under the SSR condition. The schematic view of the spot welded cylindrical specimen is illustrated in Figure 3-10, which was polarized cathodically/anodically using three-electrode polarization technique discussed earlier in this thesis.

![Figure 3-10 Spot-Welded Cylindrical Specimen](image)

Figure 3-10 Spot-Welded Cylindrical Specimen
The experimental setup used in $E_{\text{cont}}$ testing is shown in Figure 3-11. The $E_{\text{corr}}$ and $E_{\text{pit}}$ values were obtained by the CPP technique discussed earlier in this thesis. The magnitude of $E_{\text{cont}}$ for cathodic potentiostatic straining was based on the measured $E_{\text{corr}}$ value in a similar environment. On the other hand, $E_{\text{cont}}$ testing under anodic potentiostatic condition was based on the measured $E_{\text{pit}}$ value in the same environment. The cracking susceptibility using these techniques was expressed in terms of TTF, $\sigma_f$, and ductility parameters including $% El$ and $% RA$.

Figure 3-11 HE Test Setup

3.6 Metallography by Optical Microscope

The metallographic technique using an optical microscope enables the characterization of phases present, their distributions within grains and their sizes which depend on the typical chemical composition and thermal treatments performed on a
material of interest. Characterization of metallurgical microstructures of the test specimens before and after testing by optical microscopy is of paramount importance. The principle of an optical microscope is based on the impingement of a light source perpendicular to 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 test specimens were sectioned and mounted using the standard metallographic technique, followed by polishing and etching to reveal the microstructures including the grain boundaries. The etchant composition is: 5% of 70% concentrated HNO₃ and 95% of Methanol. The polished and etched specimens were rinsed in deionized water, and dried with acetone and alcohol prior to their evaluation by a Leica optical microscope, shown in Figure 3-12. This microscope is capable of resolution of up to 1000X.
3.7 Fractographic Evaluation by SEM

The extent and morphology of failure in the tested specimens were determined using a Scanning Electron Microscope (SEM). Analysis of failure in metals and alloys involves identification of the type of failure. The test specimens were sectioned into 1/2 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.

In a 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 and create 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, shown in Figure 3-13, 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.

Figure 3-13 Scanning Electron Microscope
CHAPTER 4

RESULTS

4.1 Tensile Properties at Ambient Temperature

The results of tensile testing of Alloy C-276 at ambient temperature are shown in Table 4-1. An effort was made to compare these data to those obtained from the vendor. A comparison of the tensile properties determined in this investigation and those obtained from the vendor showed very similar values for the average YS, UTS and %El, as shown in Table 4-1.

Table 4-1 Comparison of Room Temperature Tensile Properties (Vendor vs. Laboratory)

<table>
<thead>
<tr>
<th>Data Source</th>
<th>Y.S, ksi (Mpa)</th>
<th>UTS, ksi (Mpa)</th>
<th>%El</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vendor</td>
<td>50 (345)</td>
<td>114 (786)</td>
<td>64</td>
</tr>
<tr>
<td>Materials Performance</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laboratory (MPL) (Average)</td>
<td>53 (365)</td>
<td>117 (807)</td>
<td>66</td>
</tr>
</tbody>
</table>

4.2 Tensile Properties at Elevated Temperatures

The engineering stress versus engineering strain (s-e) diagrams for Alloy C-276 obtained in tensile testing at ambient temperature, 450 and 600°C are shown in Figure 4-
1. The magnitudes of YS, UTS, and %El determined from these s-e diagrams, and the dimensions of test specimens (before/after) are shown in Table 4-2. As expected, the magnitude of YS and UTS was gradually reduced with increasing temperature due to enhanced plasticity at elevated temperatures thus, producing less resistance to deformation at the applied tensile load. It is, however, interesting to note that the magnitude of YS and UTS was still quite high even at temperatures of 450 and 600°C. These results may suggest that Alloy C-276 will have the capability to maintain fairly high strength at elevated temperatures, thus providing reasonably high resistance to plastic deformation at temperatures up to 600°C, which is very close to the operating temperatures for the sulfuric acid decomposition process in the S-I cycle during nuclear hydrogen generation.

![Stress-Strain Diagram vs. Temperature](image)
Table 4-2 Tensile Properties of Alloy C-276 at Different Temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>YS, ksi (Mpa) Avg.</th>
<th>UTS, ksi (Mpa) Avg.</th>
<th>%EI Avg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>53 (365)</td>
<td>117 (807)</td>
<td>66</td>
</tr>
<tr>
<td>450</td>
<td>37 (255)</td>
<td>95 (655)</td>
<td>80</td>
</tr>
<tr>
<td>600</td>
<td>37 (255)</td>
<td>90 (621)</td>
<td>80</td>
</tr>
</tbody>
</table>

The magnitudes of %EI obtained from these tensile tests at different temperatures are also shown in Table 4-2. An evaluation of these data indicates that the ductility in terms of %EI was significantly high even at temperatures up to 600°C. It is well known that, for a fcc nickel-based material such as Alloy C-276, there are numerous slip planes oriented in random directions, thus allowing simultaneous plastic deformation along these slip planes. This phenomenon might have contributed to the enhanced ductility in terms of %EI even at the elevated temperatures. Thus, the overall data in terms of all measured parameters may suggest that Alloy C-276 may be a suitable structural material for heat-exchanger applications due to its resistance to plastic deformation while simultaneously maintaining high ductility in terms of %EI even at significantly high testing temperatures.

4.3 SCC Testing at Constant-Load

As indicated earlier in this thesis, the structural materials for heat-exchanger applications may become susceptible to SCC while exposed to the S-I environment at the operating temperatures related to the nuclear hydrogen generation. Since the current experimental facility at the Materials Performance Laboratory (MPL) is capable of...
performing SCC testing at temperatures up to the boiling point of water, constant-load (CL) testing was performed to evaluate the SCC susceptibility in an environment relevant to the S-I cycle at temperatures only up to 90°C. Both smooth and notched cylindrical specimens were tested in this environment using the calibrated proof rings as indicated earlier. The results of SCC testing of Alloy C-276 using smooth specimens loaded to 95% of its ambient temperature YS value are shown in Table 4-3. These results indicate that this alloy did not exhibit any failure at this applied stress level. Since no failure was observed based on duplicate testing at this applied stress level, no additional SCC tests were performed at any other load levels. Thus, the threshold stress (\( \sigma_{th} \)) for SCC of this alloy in the tested environment may lie in the vicinity of 0.95YS.

Efforts were also made to load the notched cylindrical specimens at various levels with respect to the yielding load (YL) at ambient temperature. However, these specimens could not be loaded at load levels above 0.7YL since they broke while loading them in the proof ring. The results shown in Table 4-3 also indicate that the notched specimen did not exhibit failure at applied loads corresponding to 50 and 70% of the YL of the test material. These results, suggest that the threshold load for SCC in Alloy C-276 in the presence of a notch may lie in the vicinity of 0.7YS.
Table 4-3 Constant-Load SCC Test Results

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Environment, pH and Temperature</th>
<th>%Applied Stress/ Stress (ksi)</th>
<th>Failure/No Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Smooth Tensile</td>
<td>S-I,</td>
<td>95 % YS / 50</td>
<td>No Failure</td>
</tr>
<tr>
<td>Smooth Tensile</td>
<td>pH=1</td>
<td>95 % YS / 50</td>
<td>No Failure</td>
</tr>
<tr>
<td>Notch Tensile</td>
<td>pH=1</td>
<td>50 % YS / 64</td>
<td>No Failure</td>
</tr>
<tr>
<td>Notch Tensile</td>
<td>90 °C</td>
<td>70 % YS / 89</td>
<td>No Failure</td>
</tr>
<tr>
<td>Notch Tensile</td>
<td>90 °C</td>
<td>70 % YS / 89</td>
<td>No Failure</td>
</tr>
</tbody>
</table>

4.4 SCC Testing under SSR Condition

The results of SCC testing of Alloy C-276 using the SSR testing technique involving both smooth and notched cylindrical specimens are shown in Figures 4-2 and 4-3, respectively. The cracking susceptibility in these SSR testing is shown in the form of a s-e diagram for both types of specimens. The s-e diagram in air is also superimposed in these plots for comparison purpose. The ductility parameters (%El and %RA), $\sigma_t$ and TTF determined from these s-e diagrams and the specimen dimensions (before/after) are shown in Table 4-4. An evaluation of these parameters indicate that Alloy C-276 exhibited significant resistance to SCC in terms of %El, %RA and TTF showing very little changes in these parameters even under the combined effect of applied stress and the testing environment.

The SCC results under SSR condition are not surprising in view of the fact that Alloy C-276 is known\textsuperscript{18,19} to withstand aggressive chemical environment even under the
influence of different types of loading. It is, however, interesting to note that the magnitude of $\sigma_f$ was reduced at 90°C while exposed to the S-I environment. This reduction in the $\sigma_f$ value is attributed to the reduction in load-bearing capability of any engineering alloys including Alloy C-276 due to the synergistic effect of applied stress and an aggressive chemical environment.

Figure 4-2 Comparison of s-e diagrams in Air and S-I solution
Figure 4-3 Comparison of s-e diagrams in Air and S-I solution

Table 4-4 SSR Test Results

<table>
<thead>
<tr>
<th>Environment/Specimen Type</th>
<th>Temperature (°C)</th>
<th>%El (Avg.)</th>
<th>%RA (Avg.)</th>
<th>σf, ksi (Mpa) (Avg.)</th>
<th>TTF, hours (Avg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air/ Smooth Tensile</td>
<td>RT</td>
<td>20.6</td>
<td>81.6</td>
<td>274 (1892)</td>
<td>72.1</td>
</tr>
<tr>
<td>S-I/ Smooth Tensile</td>
<td>RT</td>
<td>21.1</td>
<td>80.8</td>
<td>265 (1829)</td>
<td>73.1</td>
</tr>
<tr>
<td>S-I/ Smooth Tensile</td>
<td>90</td>
<td>20.5</td>
<td>79.0</td>
<td>247 (1704)</td>
<td>71.6</td>
</tr>
<tr>
<td>Air/ Notch Tensile</td>
<td>RT</td>
<td>2.9</td>
<td>58.0</td>
<td>174 (1202)</td>
<td>10.6</td>
</tr>
<tr>
<td>S-I/ Notch Tensile</td>
<td>RT</td>
<td>2.7</td>
<td>54.5</td>
<td>162 (1116)</td>
<td>10.5</td>
</tr>
<tr>
<td>S-I/ Notch Tensile</td>
<td>90</td>
<td>2.9</td>
<td>55.9</td>
<td>146 (1008)</td>
<td>11.1</td>
</tr>
</tbody>
</table>

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The results of SCC testing involving notched specimens of Alloy C-276 under the SSR condition, shown in Table 4-4, indicate that the magnitude of TTF was significantly lower compared to that of the smooth specimen. Further, the %E1 was substantially reduced in the presence of a notch indicating enhanced cracking susceptibility due to the stress concentration effect. The ductility in terms of %RA was also substantially lower in the notched specimen compared to that of the smooth specimens. In addition, the magnitude of σf in the notched specimen was substantially lower compared to that of the smooth specimen. It should, however, be noted that the magnitude of σf was reduced substantially at 90°C in the S-I solution, irrespective of the type of specimen used in the SSR testing.

4.5 Localized Corrosion using CPP

The results of localized corrosion study using the cyclic potentiodynamic polarization (CPP) technique in 30 and 90°C S-I environment are shown in Figures 4-4 and 4-5, respectively. An examination of these figures indicates that even though an active-passive behavior was noted at both temperatures, no positive hysteresis loop was experienced during reverse potential scan. Instead, the polarization diagram was reversed during cyclic polarization indicating the formation of protective oxide film due to oxygen generation at high current density, thus reducing the corrosion rate. Such phenomenon in the CPP diagram is very common\textsuperscript{[20]} with metals and alloys possessing significant resistance to localized corrosion even in the presence of many hostile chemical species including H\textsubscript{2}SO\textsubscript{4} and iodine.
Figure 4-4 CPP Diagram in Room Temperature S-I Solution

Figure 4-5 CPP Diagram in 90°C S-I Solution
The magnitude of the corrosion potential ($E_{\text{corr}}$) and the critical pitting potential ($E_{\text{pit}}$), determined from these polarization diagrams, are shown in Table 4-5. An examination of these data indicates that both $E_{\text{corr}}$ and $E_{\text{pit}}$ became more active (negative) at 90°C, showing a pattern which is consistent with the observations made by other investigators.\(^{[21]}\) The variation of $E_{\text{corr}}$ and $E_{\text{pit}}$ with temperature is shown in Figures 4-6 and 4-7, respectively. The visual examination of the polarized specimens exhibited no localized attack in them.

### Table 4-5 Critical Potentials vs. Temperature

<table>
<thead>
<tr>
<th>Temperature</th>
<th>$E_{\text{corr}}$, mV (Ag/AgCl)</th>
<th>$E_{\text{pit}}$, mV (Ag/AgCl)</th>
<th>$E_{\text{corr}}$, mV (Ag/AgCl)</th>
<th>$E_{\text{pit}}$, mV (Ag/AgCl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room Temperature</td>
<td>254.2</td>
<td>578.1</td>
<td>225.5</td>
<td>494.9</td>
</tr>
<tr>
<td>90°C</td>
<td>232.1</td>
<td>606.6</td>
<td>200.0</td>
<td>503.6</td>
</tr>
</tbody>
</table>

![Figure 4-6 $E_{\text{corr}}$ vs. Temperature](image-url)
4.6 SSR Testing under $E_{\text{cont}}$

It is well known\cite{22, 23, 24} that materials susceptible to SCC may be influenced by the external potential/current during straining under different loading conditions. In view of this rationale, the susceptibility of Alloy C-276 to SCC was determined under both anodic and cathodic applied potentials. As indicated earlier, the selection of these applied potentials was based on either $E_{\text{corr}}$ or $E_{\text{pit}}$ determined in a similar acidic environment by the CPP technique. SCC testing under cathodic $E_{\text{cont}}$ was performed by SSR technique at potential active (negative) to the $E_{\text{corr}}$ value. On the other hand, the SSR testing under anodic $E_{\text{cont}}$ was performed at potentials noble (positive) to the $E_{\text{pit}}$ value. The resultant data are discussed in the following two subsections.

4.6.1 SCC Testing under Cathodic $E_{\text{cont}}$

The results of SSR testing involving smooth cylindrical specimens, with and without cathodic $E_{\text{cont}}$, are shown in Figure 4-8 in the form of s-e diagram. $E_{\text{cont}}$ values corresponding to -100 and -300mV with respect to the $E_{\text{corr}}$ value were applied in these tests. An examination of these diagrams clearly indicates that the failure stress was

![Figure 4-7 $E_{\text{pit}}$ vs. Temperature](image)
gradually reduced while the specimens were strained under more active \( E_{\text{cont}} \) values. Simultaneously, the failure strain was also gradually reduced with more active \( E_{\text{cont}} \) values. The ductility parameters (%E1 and %RA), true failure stress (\( \sigma_f \)) and TTF obtained from these s-e diagrams and the specimen dimensions are shown in Table 4-5. The variations of these parameters with the \( E_{\text{cont}} \) values are illustrated in Figure 4-9 (a-d), once again demonstrating that the cracking susceptibility of this alloy may be enhanced in the acidic solution due to the cathodic \( E_{\text{cont}} \).

![Figure 4-8 s-e Diagram with and without Cathodic \( E_{\text{cont}} \)](image)

Table 4-5 Results of SSR Testing with and without Cathodic \( E_{\text{cont}} \)

<table>
<thead>
<tr>
<th>Environment</th>
<th>( E_{\text{cont}} ), mV (Ag/AgCl)</th>
<th>%E1</th>
<th>%RA</th>
<th>( \sigma_f ) (ksi)</th>
<th>TTF (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90°C, S-I</td>
<td>None</td>
<td>7.04</td>
<td>72.12</td>
<td>300</td>
<td>25.96</td>
</tr>
<tr>
<td></td>
<td>Cathodic, +143</td>
<td>6.94</td>
<td>69.67</td>
<td>285</td>
<td>23.92</td>
</tr>
<tr>
<td></td>
<td>Cathodic, -60</td>
<td>6.79</td>
<td>66.36</td>
<td>267</td>
<td>23.79</td>
</tr>
</tbody>
</table>

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(a) %El vs. $E_{\text{cont}}$

(b) %RA vs. $E_{\text{cont}}$
Figure 4-9 Variation of %El, %RA, TTF and $\sigma_f$ vs. $E_{cont}$

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4.6.2 SCC Testing under Anodic $E_{cont}$

The cracking susceptibility of Alloy C-276 in the acidic solution under $E_{cont}$ values corresponding to +200 and +300mV relative to the $E_{pit}$ value is shown in Figure 4-10. An evaluation of the s-e diagrams, shown in this figure, indicates that both the failure stress and strain were gradually reduced with more noble $E_{cont}$ values. Once again, the detrimental effect of anodic $E_{cont}$ on the cracking susceptibility is demonstrated in this figure. The magnitudes of the ductility parameters, TTF and $\sigma_f$ obtained from these anodic $E_{cont}$ testing are given in Table 4-6. It is interesting to note that the cracking tendency of Alloy C-276 was more pronounced under anodic $E_{cont}$ in terms of $\sigma_f$, as shown in this table.
Table 4-6 Results of SSR Testing with and without Anodic $E_{cont}$

<table>
<thead>
<tr>
<th>Environment</th>
<th>$E_{cont}$, mV (Ag/AgCl)</th>
<th>%El</th>
<th>%RA</th>
<th>$\sigma_f$ (ksi)</th>
<th>TTF (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>90°C, S-I</td>
<td>None</td>
<td>7.04</td>
<td>72.12</td>
<td>300</td>
<td>25.96</td>
</tr>
<tr>
<td></td>
<td>Anodic, +200</td>
<td>6.74</td>
<td>68.64</td>
<td>286</td>
<td>23.29</td>
</tr>
<tr>
<td></td>
<td>Anodic, +300</td>
<td>6.42</td>
<td>63.58</td>
<td>233</td>
<td>22.68</td>
</tr>
</tbody>
</table>

4.7 Metallographic Evaluations

The optical micrograph of Alloy C-276 in an etched condition is shown in Figure 4-11. This micrograph reveals conventional microstructure of a typical nickel-based alloy, including the presence of annealing twins, which are the characteristics of solution annealed nickel-based alloy. [25]

Figure 4-11 Optical Micrograph of Sectioned Bar Material at 50X
4.8 Fractographic Evaluations

The characteristics of failure at the primary fracture surface of both the tensile and the SCC specimens were determined by SEM. The SEM micrographs of the fracture surface of the tensile specimens are shown in Figure 4-12 at three different temperatures. These micrographs are characterized by dimples, indicating ductile failure both at ambient and elevated temperatures. An evaluation of the primary fracture surface of both smooth and notch specimens used in SSR testing also revealed the formation of dimpled microstructures, as evidenced by Figures 4-13 and 4-14, respectively. Thus, it is obvious that irrespective of the testing method, the failure of Alloy C-276 will predominantly be ductile in nature. These results may demonstrate the suitability of Alloy C-276 as a structural material possessing superior resistance to deformation and corrosive attack.
Figure 4-12 SEM Micrographs of Smooth Tensile Specimen

(a) Ambient Temperature, 100X

(b) 450°C, 100X

(c) 600°C, 100X

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Figure 4-13 SEM Micrographs of Smooth Specimen used in SSR Testing

(a) Ambient Temperature, 500X

(b) Ambient Temperature, S-I Solution, 500X

(c) 90°C, S-I solution, 500X
Figure 4-14 SEM Micrographs of Notch Specimen used in SSR Testing
CHAPTER 5

DISCUSSION

The identification and selection of structural materials for applications in heat exchangers in nuclear hydrogen generation using the S-I cycles are undoubtedly two major challenges to the engineering and scientific communities. A nickel-based alloy known as Alloy C-276 has been identified and characterized for its high temperature tensile properties, and corrosion resistance in an environment relevant to the S-I cycle proposed for hydrogen generation using nuclear power.

Even though, the maximum operating temperature in the S-I cycle may be in the vicinity of 850-900°C, the tensile properties of Alloy C-276 have been determined in this investigation at ambient temperature, 450 and 600°C. The results of tensile testing using smooth cylindrical specimens indicate that Alloy C-276 is capable of maintaining high tensile strength even at a temperature of 600°C. Very little changes in failure strength were noted in the temperature regime of 450-600°C. In addition, this alloy exhibited excellent ductility in terms of %EI at all tested temperatures. While efforts should be made to determine the tensile strength of Alloy C-276 at temperatures above 600°C using the newly installed Instron testing machine at the materials performance laboratory (UNLV), it may be concluded that Alloy C-276 may be a suitable structural material for heat exchanger applications at least up to an operating temperature of 600°C as noted in this investigation.
The precise operating environment for hydrogen generation using the S-I cycle is yet to be finalized. Since the hostile environments associated with the S-I cycle could not be accommodated at MPL, an aqueous environment containing $\text{H}_2\text{SO}_4$ and NaI was used to evaluate the corrosion behavior of Alloy C-276 using state-of-the-art experimental techniques. The results of SCC testing performed in this environment at CL indicate that no failures were observed with the smooth specimens even at an applied stress corresponding to 95% of this material's YS value. However, a maximum temperature of only 90°C could be accommodated in these tests using the existing facility. Therefore, additional SCC testing is warranted in a similar environment at much higher testing temperatures. In view of this rationale, SCC testing using self-loaded specimens (C-ring and U-bend) is recommended for future researchers involving the newly installed high temperature autoclave system. SCC testing using the notched specimens of Alloy C-276 exhibited enhanced cracking tendency due to the stress concentration effect, as anticipated.

The susceptibility of Alloy C-276 to SCC was also determined in the similar acidic solution at 90°C by using the SSR technique. The results involving smooth specimens indicate that the cracking tendency in terms of ductility parameters and TTF was not significantly influenced by the variation in temperature from ambient to 90°C. However, the magnitude of $\sigma_f$ was substantially reduced at the elevated temperature. The magnitude of $\sigma_f$ in the notched specimen was significantly lower due to the stress concentration effect, and short failure time due to the presence of the notch.

The results of CPP testing indicate that Alloy C-276 exhibited an active/passive behavior, and significant resistance to localized attack by demonstrating a negative...
hysteresis loop during the reverse potential scan. This phenomenon is commonly associated with the formation of protective oxide film on the specimen surface due to the evolution of oxygen by electrolysis of water at high current densities. The results of CPP testing also indicate that both $E_{\text{corr}}$ and $E_{\text{pit}}$ became more active at 90°C. This observation is consistent with the findings of other investigators\textsuperscript{[21]} on the effect of temperature on critical potentials.

The results of SSR testing under $E_{\text{corr}}$ may suggest that the cracking susceptibility of a nickel-based alloy such as Alloy C-276 may be enhanced under anodic applied potential as opposed to cathodic applied potentials. Under anodic $E_{\text{cont}}$, the susceptible material may undergo dissolution at a localized region causing pitting, the extent of which may be enhanced at potentials above the $E_{\text{pit}}$ value. Thus, the application of a more noble $E_{\text{cont}}$ (with respect to $E_{\text{pit}}$) may lead to enhanced localized corrosion, eventually resulting in increased cracking susceptibility. This phenomenon was also noticed in this investigation showing a significant reduction in $\sigma_f$ value at more noble potentials. However, it should be mentioned that a detrimental effect of cathodic $E_{\text{cont}}$ on cracking susceptibility was also noted in the SSR testing.

The optical micrograph revealed a typical microstructure for a fcc nickel-based alloy. The fractographic evaluations of the broken specimens, both in tensile and corrosion testing, by SEM revealed dimpled microstructures, indicating ductile failures.
SUMMARY AND CONCLUSIONS

The tensile properties of Alloy C-276 have been determined at ambient and elevated temperatures. The cracking susceptibility of this alloy has been determined in an acidic solution under constant-load and slow-strain-rate conditions. The effect of applied potential on cracking behavior has been studied under anodic and cathodic controlled potentials. The localized corrosion behavior has been studied using a polarization technique. Metallographic and fractographic evaluations have been performed by optical microscopy and SEM. The significant conclusions derived from this investigation are summarized below.

- The tensile data indicate that Alloy C-276 may be capable of maintaining appreciably high tensile strength even at a temperature of 600°C. The ductility in terms of %El was enhanced at elevated temperatures. The enhanced ductility may be attributed to increased plasticity at elevated temperatures and the presence of numerous randomly oriented slip planes associated with a nickel-based alloy.

- No failure was observed at constant-load when tested in a 90°C acidic environment.
• The true failure stress ($\sigma_f$) was substantially reduced at an elevated temperature for both smooth and notched specimens tested under a SSR condition. However, the magnitude of $\%El$, $\%RA$ and TTF was not significantly influenced by the variation in the testing temperature.

• The susceptibility of Alloy C-276 to SCC was more pronounced with the notched specimens in SSR tests due to the stress concentration effect.

• The application of anodic and cathodic potentials during straining of the specimens resulted in enhanced cracking susceptibility in terms of the ductility parameters, TTF and $\sigma_f$, which were all reduced due to the applied potentials.

• Both $E_{corr}$ and $E_{pit}$, determined by the CPP technique, became more active with increasing temperature.

• Fractographic evaluations by SEM revealed dimpled microstructures at the primary fracture surface, indicating ductile failures. A metallurgical microstructure typical of a fcc nickel-base material was observed in the optical micrograph.
CHAPTER 7

SUGGESTED FUTURE WORK

- Evaluation of tensile properties at temperatures above 600°C, preferably up to 1000°C, using the Instron testing machine.
- Evaluation of stress corrosion cracking susceptibility using self-loaded specimens (C-ring, U-bend and double cantilever beam) at elevated temperatures using an autoclave.
- Evaluation of general corrosion rate as a function of temperature using an autoclave.
- Determination of fracture toughness (K_IC) using compact tension specimen in the Instron testing machine.
APPENDIX A

MTS DATA

A1. Stress-Strain Diagrams using Smooth Specimens Tested at Room Temperature
(Conversion factor 1 ksi = 6.8948 Mpa can be used wherever applicable)

Sample 1

Sample 2

58
A2. Stress-Strain Diagrams using Smooth Specimens Tested at 450°C

Sample 1

Sample 2
A3. Stress-Strain Diagrams using Smooth Specimens Tested at 600°C

Sample 1

Sample 2

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APPENDIX B

SSR DATA

B1. Stress-Strain Diagrams in Air using Smooth Specimens
(Conversion factor 1 ksi = 6.8948 Mpa can be used wherever applicable)

Sample 1

Sample 2
B2. Stress-Strain Diagrams in S-I solution at Room Temperature using Smooth Specimens

Sample 1

Sample 2
B3. Stress-Strain Diagrams in S-I solution at 90°C using Smooth Specimens

Sample 1

Sample 2
B4. Stress-Strain Diagrams in Air using Notch Specimens

Sample 1

Sample 2

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B5. Stress-Strain Diagrams in S-I solution at Room Temperature using Notch Specimens

Sample 1

Sample 2

65
B6. Stress-Strain Diagrams in S-I solution at 90°C using Notch Specimens

Sample 1

Sample 2
APPENDIX C

CPP DATA

C1. CPP Diagrams at 30°C

Sample 1

Sample 2
C2. CPP Diagrams at 90°C

Sample 1

Sample 2
APPENDIX D

Econt DATA

D1. Cathodic $E_{\text{cont}}$ in S-I, 90°C at +143 mV
   (Conversion factor 1 ksi = 6.8948 Mpa can be used wherever applicable)

D2. Cathodic $E_{\text{cont}}$ in S-I, 90°C at -60 mV

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D3. Anodic $E_{\text{cont}}$ in S-I, 90°C at +200 mV

D4. Anodic $E_{\text{cont}}$ in S-I, 90°C at +300 mV
APPENDIX E

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. 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.

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.

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.

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

(1)\textsuperscript{26}

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

\[
R = \text{the given function of the independent variables } x_1, x_2, \ldots, x_n
\]

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

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

E1.1 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 \( \pm 0.03\% \) lbs and \( \pm 0.001 \) in, respectively, obtained from the calibration. The uncertainty in the initial notched diameter was \( \pm 0.001 \), which was provided by the manufacturer and the uncertainty in the final notched diameter was \( \pm 0.001 \) obtained by using the caliper.

E1.1.1 Calculation of Uncertainty in Stress (\( u\sigma \))

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

\[
u A_i = (uD_i)^2
\]

Uncertainty in load-cell = \( \pm 0.03\% \) lb
Uncertainty in caliper = ± 0.001 in

**Sample calculation:**

For yield stress (YS) = 53 ksi

Uncertainty in load (\(u_P\)) = 2597*0.0003

= ± 0.7791

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

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

Uncertainty in diameter (\(u_D\)) = ± 0.001 in

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

= 0.049

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

= 0.393

Uncertainty in area, \(u_{A_i}\) = \[\left(\frac{dA_i}{dD_i} \cdot u_D\right)^2\] \[\frac{1}{2}\]

= 0.393*0.001

= ± 0.000393

Uncertainty in stress, \(u_\sigma\) = \[\left(\frac{\partial \sigma}{\partial P} \cdot u_P\right)^2 + \left(\frac{\partial \sigma}{\partial A_i} \cdot u_{A_i}\right)^2\] \[\frac{1}{2}\]

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

\[
\frac{\partial \sigma}{\partial P} = \frac{1}{A_i}
\]
\[ \text{d} \sigma = \frac{P}{A_i^2} \]

\[ = -1081632.65 \]

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

\[ \sigma = \left( (20.41 \times 0.7791)^2 + (-1081632.65 \times 0.000393)^2 \right)^{\frac{1}{2}} \]

\[ = 425.37 \text{ psi} \]

\[ = \pm 0.42 \text{ ksi} \]

One example of the use of the uncertainty analysis is shown in this section. This can be implemented to all experimental results discussed in this thesis.

E1.2 Calculation of Uncertainty in Percentage Elongation (\( \% \text{El} \))

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 \( \Delta l \) (\( \sigma_{\Delta l} \)) = \pm 0.001

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

\[ \sigma_{\% \text{El}} = \left[ \left( \frac{d\% \text{El}}{d\Delta l} \cdot \sigma_{\Delta l} \right)^2 \right]^{\frac{1}{2}} \]

\[ \frac{d\% \text{El}}{d\Delta l} = \frac{100}{l} \]
Providing all the calculated values in equation 1.2, it is found that,

\[
_u \% El = \left[ \left(100 + 0.001 \right)^2 \right]^{\frac{1}{2}}
\]

\[u \% El = \pm 0.1\]

One example of the use of the uncertainty analysis is shown in this section. This can be implemented to all experimental results discussed in this thesis.

E2 Uncertainty Calculation in Constant-load (CL) Results

E2.1 Uncertainty Calculation in Applied Stress (\(\sigma_a\)):
The uncertainty in applied stresses was calculated using equation 1.1.

E2.2 Uncertainty Calculation in Time-To-Failure (TTF):
The uncertainty in the automatic timer of the CL test setup is \(\pm 0.50\).

E3 Uncertainty Calculation in Slow-Strain-Rate (SSR) Testing

E3.1 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 1.1.

E3.2 Uncertainty Calculation in Percentage Elongation (\(u \% El\))
The uncertainty in the \(\% El\) was calculated using the equation 1.2.

E3.3 Calculation of Uncertainty in Percentage Reduction in Area (\(u \% RA\))
Sample calculation:

For \(\% RA = 81.6\%\)
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

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,

$u_{A_i} = \left[ \left( \frac{dA_i}{dD_i} \cdot u_{D_i} \right)^2 \right]^\frac{1}{2}$

= 0.393*0.001

= $\pm 0.000393$

Uncertainty in final cross-sectional area ($u_{A_f}$) for the smooth specimen:

Final Diameter ($D_f$) = 0.1070 in

Uncertainty in final diameter ($u_{D_f}$),

$u_{D_f} = \pm 0.001$ in

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

= 0.009

$\frac{dA_f}{dD_f} = \frac{\pi D_f}{2}$
Uncertainty in final cross-sectional area,

\[ u_{A_f} = \left[ \frac{dA_f}{DD_f} \right]^{\frac{1}{2}} \]

\[ = 0.014 \times 0.001 \]

\[ = 0.000014 \]

Uncertainty in \( u \% RA \),

\[ u \% RA = \left[ \left( \frac{\partial \% RA}{\partial A_i} \right)^2 + \left( \frac{\partial \% RA}{\partial A_f} \right)^2 \right]^{\frac{1}{2}} \]

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

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

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

\[ = 374.84 \]

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

\[ = -2040.82 \]

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

\[ u \% RA = \left[ (374.84 \times 0.000393)^2 + (-2040.82 \times 0.000014)^2 \right]^{\frac{1}{2}} \]

\[ = 0.15 \]
One example of the use of the uncertainty analysis is shown in this section. This can be implemented to all experimental results discussed in this thesis.

E3.4 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.

E4 Uncertainty Calculation in Cyclic Potentiodynamic Polarization (CPP) Testing

The accuracy of the potentiostat provided by the manufacturer is $\pm 0.003\, \text{mV}$ within a range of 1 mV.

Sample calculation: For corrosion potential ($E_{\text{corr}}$) = 254.2 mV

The uncertainty in $E_{\text{corr}} = 254.2 \times 0.003$

$= 0.762$

$= \pm 0.762\, \text{mV}$

One example of the use of the uncertainty analysis is shown in this section. This can be implemented to all experimental results discussed in this thesis.


3. Haynes International Inc.


7. Robert C. Juvinall and Kurt M. Marshek, “Fundamentals of Machine Component Design,” Published by John Wiley and Sons, Third Edition, Figure No. 4.36(b), pp. 146

   http://www.cortest.com


79

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13. Slow-Strain-Rate Testing, Corrosion Source Website
   http://www.corrosionsource.com/handbook/testing/ssrt.htm

   http://www.gamry.com/Homepage/Index.html


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