Effects of metallurgical and environmental variables on the performance of alloy 718 for heat exchanger applications

Anand Venkatesh

University of Nevada, Las Vegas

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EFFECTS OF METALLURGICAL AND ENVIRONMENTAL VARIABLES ON THE PERFORMANCE OF ALLOY 718 FOR HEAT EXCHANGER APPLICATIONS

by

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The Dissertation prepared by

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ABSTRACT

Effects of Metallurgical and Environmental Variables on the Performance of
Alloy 718 for Heat Exchanger Applications

By
Anand Venkatesh

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

Austenitic Alloy 718 was extensively investigated, evaluating its tensile properties, crack-growth-rate (CGR), impact resistance, and resistance to stress-corrosion-cracking (SCC) and localized corrosion. The tensile data enabled a development of mechanisms on dynamic strain aging (DSA) and yield strength anomaly (YSA). The magnitudes of dislocation density, activation energy and work-hardening index, related to the DSA phenomenon, were estimated using different analytical approaches. The occurrence of YSA was verified based on transmission electron microscopic evaluations. The steady state CGR followed the Paris equation, showing a linear relationship. Alloy 718 exhibited a respectable impact resistance irrespective of the testing temperature. The SCC susceptibility of this alloy was enhanced in the 90°C acidic solution, showing reduced ductility and true failure stress. The corrosion potential, measured by polarization technique, became more active with increasing temperature. The failure morphologies of all tested specimens were characterized by scanning electron microscopy.
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CHAPTER 1

INTRODUCTION

The price of conventional fossil fuels, such as oil and gas, has been escalating at an alarming rate in recent years. Current statistics indicate that fossil fuels provide 66% of the world’s electricity and about 90% of the global energy demands.\textsuperscript{[1,2]} Extensive use of these types of fuels for residential, transportation and industrial applications has created a significant disparity between their supply and demand. Moreover, these fuels have now been known to contaminate natural environment, causing global warming due to greenhouse effect resulting from carbon dioxide (CO\textsubscript{2}) emission.\textsuperscript{[3]}

In order to avoid continued dependence on fossil fuels and prevent/minimize detrimental effect of pollutants due to extensive fossil fuel utilization, the United States Department of Energy (USDOE) had been considering the development of alternate sources of energy for quite sometime. Hydrogen is one such energy, which is currently being explored by USDOE that can circumvent numerous problems associated with the usage of conventional fossil fuels. While different methods of hydrogen generation currently exist, a major challenge rests in the development of hydrogen using a cost-effective technique.

Conventionally, hydrogen can be generated by electrolysis. However, substantial energy in the form of electricity has to be used for efficient dissociation of water into hydrogen (H\textsubscript{2}) and oxygen (O\textsubscript{2}). Thus, the use of electricity defeats the purpose of H\textsubscript{2}
generation in a cost-effective manner, resulting in reduced efficiency of the electrolytic process.\textsuperscript{[4, 5]} In order to achieve a maximum possible efficiency in \( \text{H}_2 \) generation, the USDOE has more recently proposed the utilization of heat from the nations’ existing nuclear power plants. The nuclear hydrogen initiative (NHI) is centered around the formation of \( \text{H}_2 \) using thermochemical processes, which include sulfur-iodine (S-I)\textsuperscript{[6-8]} and calcium-bromine (Ca-Br)\textsuperscript{[9]} cycles. Both cycles involve generation of \( \text{H}_2 \) using chemical reactions at elevated temperatures. However, the USDOE has been more inclined towards the utilization of the S-I process due to its relatively higher efficiency (52\%) in \( \text{H}_2 \) generation.\textsuperscript{[7, 10]}

The S-I cycle, invented in the mid 1970’s by the General Atomics Corporation (GA) of California\textsuperscript{[11]}, consists of the formation and decomposition of sulfuric acid (\( \text{H}_2\text{SO}_4 \)) and hydrogen iodide (HI) at different temperatures. The temperatures needed for such chemical reactions are currently being envisioned to be provided by gas-cooled nuclear reactors through use of an intermediate heat exchanger located between the reactor and the hydrogen generation plant, as illustrated in Figure 1.1.\textsuperscript{[11]} The reactions associated with the S-I process will occur within closed loops, where water (\( \text{H}_2\text{O} \)) can be fed to the process, \( \text{O}_2 \) and \( \text{H}_2 \) be collected, and other reactants be recycled.

As illustrated in Figure 1.2\textsuperscript{[11]}, \( \text{H}_2\text{SO}_4 \) and HI will be formed at 120°C involving chemical reactions among sulfur dioxide (\( \text{SO}_2 \)), iodine (\( \text{I}_2 \)) and \( \text{H}_2\text{O} \). Subsequently, \( \text{H}_2\text{SO}_4 \) and HI will be separated and transferred to two different reaction chambers, where \( \text{H}_2\text{SO}_4 \) and HI will be subjected to decomposition at different temperatures. The decomposition of \( \text{H}_2\text{SO}_4 \) into \( \text{SO}_2 \), \( \text{O}_2 \) and \( \text{H}_2\text{O} \) will occur at a maximum temperature of 950°C. On the other hand, the decomposition of HI will occur at a much lower
temperature of 400°C. The generated O₂ and H₂ resulting from these chemical reactions will subsequently be transferred to separate storage containers. SO₂ and I₂ will be acting as catalysts, which will be recycled to react with H₂O producing H₂SO₄ and HI. The three chemical reactions associated with the S-I process are given as Reactions 1.1, 1.2 and 1.3, respectively. These reactions will be repeated for continuous generation of H₂ and O₂.

Figure 1.1 Concept of NHI

Figure 1.2 S-I Cycle
\[
\begin{align*}
I_2 + SO_2 + 2H_2O & \Rightarrow 2HI + H_2SO_4 \text{ (Maximum Temperature ~ 120°C)} \quad \text{(Reaction 1.1)} \\
H_2SO_4 & \Rightarrow H_2O + SO_2 + \frac{1}{2} O_2 \text{ (Maximum Temperature ~ 950°C)} \quad \text{(Reaction 1.2)} \\
2HI & \Rightarrow H_2 + I_2 \text{ (Maximum Temperature ~ 400°C)} \quad \text{(Reaction 1.3)}
\end{align*}
\]

In light of the preceding discussion, it is obvious that the two major challenges to \( H_2 \) generation using the S-I process are the identification and selection of suitable structural materials possessing superior tensile properties at elevated temperatures, and excellent corrosion resistance in hostile chemical environments. Substantial research activities have been ongoing for the past three years at the Materials Performance Laboratory (MPL) of UNLV to characterize the tensile deformation, crack-growth and corrosion behavior of several candidate structural materials at ambient and elevated temperatures. More recently, some of these materials are also being considered for high temperature applications in the Next Generation Nuclear Plant (NGNP) program. Thus, the characterization of both the metallurgical and the corrosion behavior of these alloys are vital for successful implementation of both NHI and NGNP programs.

Many candidate structural materials had been recommended by the members of the Materials Advisory Committee at the onset of the NHI program based on their prior experience, and literature review performed by the MPL researchers.\cite{12-15} One such material is austenitic iron-nickel-chromium-molybdenum (Fe-Ni-Cr-Mo) alloy, commercially known as Alloy 718 (UNS N07718). This alloy is known to possess excellent high-temperature tensile strength, superior fatigue resistance, and substantial corrosion and oxidation resistance in many hostile environments.\cite{16} Alloy 718 currently accounts for 45% of wrought Ni-base superalloy production.\cite{17,18} Compared to other Ni-base superalloys, hardened by aluminum or titanium, Alloy 718 has superior weldability
and formability. This alloy finds extensive industrial applications due to its excellent tensile, fatigue, creep and rupture strength along with its capability to be fabricated at moderate cost. Such applications include liquid-fueled rockets, rings, casings and various formed sheet metal parts for aircraft and land-based gas turbine engines, and cryogenic storage tanks. It is also used for fasteners and instrumentation parts.\(^{[19]}\)

The unusually high tensile strength of Alloy 718 may be attributed to the presence of high Ni content that can lead to the development of a continuous matrix of face-centered-cubic (fcc) solid solution of Cr, Mo, cobalt (Co), tungsten (W), and iron (Fe) in large concentrations.\(^{[20]}\) The superior corrosion resistance of this alloy in both oxidizing and reducing environments, and mixtures of acids with chloride ions has been attributed to the formation of niobium (Nb)-rich Cr-oxide layers (Cr\(_2\)O\(_3\)).\(^{[17]}\) The presence of high Cr and Mo content in this alloy can prevent localized attack and stress-corrosion-cracking (SCC) susceptibility in many hostile environments.\(^{[16,17,19]}\) Cr can also provide an ability to withstand attack by oxidizing media and sulfur-containing compounds. The presence of Mo is known to enhance the resistance of metals and alloys to pitting corrosion in many hostile aqueous media containing halides. In view of these superior metallurgical and corrosion properties, Alloy 718 was identified to be a candidate structural material for evaluation of its performance under conditions relevant to the S-I process using numerous state-of-the-art experimental techniques.

Alloy 718 has been tested in this investigation under tensile loading at temperatures up to 1000°C to determine its structural strength and ductility to comply with the maximum operating temperature of 950°C, proposed for \(\text{H}_2\text{SO}_4\) decomposition using the S-I process.\(^{[7,11]}\) A maximum temperature of 1000°C was used to satisfy the requirements
of the American Society of Mechanical Engineers (ASME) Pressure Vessel Committee that requires a structural material be tested at a temperature equivalent to at least 50°C above the maximum design temperature. The magnitudes of tensile properties including the yield strength (YS), the ultimate tensile strength (UTS) and ductility in terms of the percent elongation (%E1) and the percent reduction in area (%RA) have been determined as functions of temperature and strain rate.

Two interesting phenomena related to tensile deformation of Alloy 718 were noted in this investigation. They are dynamic strain aging (DSA) and yield strength anomaly (YSA) that are occasionally experienced by metals and alloys at different temperatures. DSA is known [21-26] to be characterized by reduced plastic strain and formation of serrations in the engineering stress versus strain (s-e) diagrams within susceptible temperature regimes. Such occurrence of reduced ductility, and formation of serrations in the s-e diagrams during tensile loading are the result of diffusion of solute/solvent elements into the metal matrix and the grain boundary regions. The precipitation of these elements near lattice defects such as dislocations, and in the vicinity of grain boundaries can cause reduced dislocation mobility, thus, leading to reduced plastic strain even under tensile loading within specific temperature regimes. The phenomenon of DSA has been extensively investigated by previous UNLV researchers involving several other nickel-base superalloys.[27, 28] Since Alloy 718 has also exhibited a tendency to DSA, extensive efforts have been made in this investigation to characterize defects, and determine dislocation density (ρ) using transmission electron micrographs (TEM) of specimens tested at different temperatures.
It has been well-established \cite{21-32} that both temperature and strain rate can influence the DSA behavior of susceptible materials by virtue of their mutual dependence on the resultant work-hardening index (n) and the activation energy (Q). In view of this rationale, the tensile properties of Alloy 718 have been evaluated under different strain rates at the susceptible temperature regime and beyond. While the phenomenon of DSA is associated with reduced ductility within specific temperature regimes, the concept of YSA is manifested in enhanced tensile strength in terms of YS at relatively higher temperatures.\cite{33-35} Therefore, attempts have also been made to develop a basic understanding of YSA using an analytical approach.

The structural materials to be used in the S-I process may also undergo cracking and their subsequent growth due to the presence of preexisting minute flaws/cracks, and thermal stresses that may fluctuate depending on the operating temperatures.\cite{36} In view of this rationale, the crack growth rate of Alloy 718 has been determined under cyclic loading at ambient and elevated temperatures using fracture-mechanics-based compact-tension (CT) specimens. A novel in-situ crack monitoring device, known as the direct-current-potential-drop (DCPD) technique, has been employed to evaluate the crack propagation rate under cyclic loading ($\frac{da}{dN}$).

The impact toughness of structural materials can play an important role in the selection of a suitable material for high-temperature applications such as the S-I process. Thus, the determination of its impact toughness, in terms of the energies absorbed in fracturing notched specimens at different temperatures, and a ductile-to-brittle-transition-temperature (DBTT) is essential to characterize its resistance to failure under impact loading. This type of testing can simulate very high strain rates in causing plastic
deformation/failure of a structural material at different temperatures.\cite{37} Therefore, the impact resistance of Alloy 718 has been determined by using Charpy v-notch (CVN) specimens in terms of energy absorbed by them during fracture at cryogenic and elevated temperatures.

The thermal stresses developed in the presence of aggressive chemical species (SO$_2$ and I$_2$) may adversely influence the performance of Alloy 718 as a structural material for application in the NHI program. This alloy may suffer from numerous environment-assisted degradations such as stress-corrosion-cracking (SCC), general corrosion and localized corrosion (pitting and crevice). Therefore, efforts have been made in this study to evaluate the susceptibility of Alloy 718 to SCC and localized corrosion in an acidic solution at temperatures up to 100°C using different state-of-the-art experimental techniques. The cracking susceptibility of this alloy has been determined by using both slow-strain-rate (SSR) and fracture-mechanics based double-cantilever-beam (DCB) techniques. The use of pre-cracked and wedge-loaded DCB specimens in the SCC testing also enabled the determination of average crack-growth-rate (CGR) in an acidic environment. Further, cyclic-potentiodynamic-polarization (CPP) technique, based on classical electrochemical principles, was used to determine the critical potentials, at which localized attack may occur in a similar environment.

Metallurgical microstructures of the tested materials were evaluated using conventional optical microscopy. Attempts were also made to analyze phase changes, if any, by using X-ray diffractometry (XRD). The extent and morphology of failure of specimens tested under loaded conditions, with and without environment, were determined by using scanning electron microscopy (SEM). The overall experimental
data, and their analyses and explanations led to the development of a plausible mechanistic understanding of deformation and degradations of Alloy 718 for prospective applications the S-I process.
CHAPTER 2

TEST MATERIAL, SPECIMENS AND ENVIRONMENT

2.1 Test Material

The application of the proposed S-I cycle involving high temperature (up to 950°C) and the presence of aggressive chemical species (SO$_2$, I$_2$) necessitates the identification and selection of suitable structural materials to sustain such hostile operating conditions. Based on a literature search \cite{12-16} and the recommendation of the Materials Advisory Committee, an austenitic Alloy 718 containing iron (Fe), nickel (Ni), chromium (Cr) and molybdenum (Mo) was selected as one of the candidate structural materials for evaluation of its metallurgical and corrosion properties. This alloy has been extensively used in numerous industrial applications \cite{19} due to its excellent tensile and corrosion properties.\cite{16-19} In addition to its superior tensile strength, this alloy also possesses enhanced ductility and toughness, which can be attributed to the formation of a stable face-centered-cubic (FCC) crystal structure that can be retained even at unusually high temperatures.\cite{20}

Alloy 718 is a gamma double-prime ($\gamma''$)-strengthened superalloy resulting from the addition of substantial amount of niobium (Nb) in it.\cite{38} The presence of high Ni content in this alloy enables plastic deformation in multiple slip planes and can, thus, provide enhanced ductility when strained under tensile loading.\cite{21} The superior corrosion resistance of Alloy 718 in many hostile aqueous environments including seawater, and
solutions containing sulfuric acid (H$_2$SO$_4$), hydrochloric acid (HCl), and chlorinated organics can be attributed to the formation of high concentration of Nb-rich Cr-oxide layers (Cr$_2$O$_3$). The presence of Cr and Mo can promote significant resistance to localized corrosion and stress corrosion cracking (SCC) in many corrosive environments. Further, the intermetallic phases formed with other alloying elements enable this alloy to withstand high-temperature deformation. Alloy 718 also possesses excellent weldability and formability, similar to other conventional Ni-base superalloys. The physical properties of Alloy 718 are given in Table 2.1.

<table>
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<tr>
<td>Melting Range</td>
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<tr>
<td>Electrical Resistivity</td>
</tr>
<tr>
<td>Mean Coefficient of Thermal Expansion</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
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<tr>
<td>Specific Heat</td>
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</table>

Round and flat bars of Alloy 718 were procured from multiple vendors in a solution-annealed condition. They were austenitized at 1750°F (954°C) for 1 hour followed by a water-quench. Alloy 718 is conventionally used in a solution-annealed and aged condition to achieve high tensile strength for applications in oil and gas exploration and high temperature applications, such as turbine blades. Even though a high strength of this material may be beneficial for structural applications, it may suffer from poor ductility. The structural materials to be used in nuclear hydrogen generation must possess a
combination of high tensile strength and superior ductility at elevated temperatures. In view of this rationale, Alloy 718 was not subjected to ageing following solution-annealing treatment. The chemical compositions of multiple heats of this alloy \cite{40-44} used in the preparation of different types of test specimens are given in Table 2.2.

Table 2.2 Chemical Compositions of Alloy 718 (wt %)

<table>
<thead>
<tr>
<th>Element</th>
<th>Heat Number/Specimen Type</th>
<th>T673/ Tensile</th>
<th>411434/ Tensile</th>
<th>A356/ Polarization</th>
<th>063417102/ CT</th>
<th>HT0937EK/ DCB</th>
<th>218049766/ CVN</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td></td>
<td>0.021</td>
<td>0.03</td>
<td>0.023</td>
<td>0.049</td>
<td>0.03</td>
<td>0.05</td>
</tr>
<tr>
<td>Mn</td>
<td></td>
<td>0.08</td>
<td>0.06</td>
<td>0.06</td>
<td>0.073</td>
<td>0.09</td>
<td>0.241</td>
</tr>
<tr>
<td>Fe</td>
<td></td>
<td>18.08</td>
<td>18.67</td>
<td>18.14</td>
<td>20.66</td>
<td>17.76</td>
<td>18.50</td>
</tr>
<tr>
<td>S</td>
<td></td>
<td>&lt;0.0003</td>
<td>0.001</td>
<td>&lt;0.0003</td>
<td>0.0004</td>
<td>0.0002</td>
<td>0.004</td>
</tr>
<tr>
<td>Si</td>
<td></td>
<td>0.07</td>
<td>0.09</td>
<td>0.05</td>
<td>0.069</td>
<td>0.06</td>
<td>0.01</td>
</tr>
<tr>
<td>Cu</td>
<td></td>
<td>0.05</td>
<td>0.04</td>
<td>0.02</td>
<td>0.042</td>
<td>0.14</td>
<td>0.0251</td>
</tr>
<tr>
<td>Ni</td>
<td></td>
<td>53.95</td>
<td>52.45</td>
<td>53.89</td>
<td>51.28</td>
<td>53.64</td>
<td>53.4</td>
</tr>
<tr>
<td>Cr</td>
<td></td>
<td>17.93</td>
<td>18.42</td>
<td>18.11</td>
<td>18.03</td>
<td>18.40</td>
<td>18.30</td>
</tr>
<tr>
<td>Al</td>
<td></td>
<td>0.49</td>
<td>0.58</td>
<td>0.33</td>
<td>0.57</td>
<td>0.52</td>
<td>0.55</td>
</tr>
<tr>
<td>Ti</td>
<td></td>
<td>1.02</td>
<td>0.99</td>
<td>0.88</td>
<td>1.03</td>
<td>1.04</td>
<td>1.02</td>
</tr>
<tr>
<td>Co</td>
<td></td>
<td>0.17</td>
<td>0.24</td>
<td>0.19</td>
<td>0.29</td>
<td>0.23</td>
<td>0.21</td>
</tr>
<tr>
<td>Mo</td>
<td></td>
<td>2.91</td>
<td>2.89</td>
<td>2.94</td>
<td>2.89</td>
<td>3.06</td>
<td>3.03</td>
</tr>
<tr>
<td>Nb</td>
<td></td>
<td>5.14</td>
<td>5.37</td>
<td>5.36</td>
<td>5.01</td>
<td>5.00</td>
<td>5.23</td>
</tr>
<tr>
<td>Ta</td>
<td></td>
<td>&lt;0.01</td>
<td>0.01</td>
<td>&lt;0.01</td>
<td>0.002</td>
<td>&lt;0.01</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>P</td>
<td></td>
<td>0.007</td>
<td>0.006</td>
<td>0.010</td>
<td>0.009</td>
<td>0.011</td>
<td>0.005</td>
</tr>
<tr>
<td>B</td>
<td></td>
<td>0.003</td>
<td>0.0038</td>
<td>0.004</td>
<td>0.0012</td>
<td>0.002</td>
<td>0.0041</td>
</tr>
</tbody>
</table>

CT: Compact-Tension
DCB: Double-Cantilever-Beam
CVN: Charpy V-Notch
2.2 Test Specimens

2.2.1 Tensile Specimen

Smooth cylindrical specimens used in tensile and SCC testing were machined from two heats of Alloy 718 (heat numbers T673 and 411434, respectively). These specimens were machined from the heat-treated round bars in such a way that the gage-section was parallel to the longitudinal rolling direction. A ratio of 4 was maintained between the gage-length and the gage-diameter of these specimens to comply with the ASTM Designation E 08 2004. These specimens had an overall length of 101.6-mm (4-inch), a gage-length of 25.4-mm (1-inch) and a gage-diameter of 6.35-mm (0.25-inch). The dimensions and a pictorial view of the cylindrical specimen is illustrated in Figure 2.1.
2.2.2 Polarization Specimen

Cylindrical specimens having a 12.7-mm (0.5-inch) length and a 9.5-mm (0.375-inch) diameter were used to evaluate the localized corrosion (pitting/crevice) behavior of Alloy 718 using an electrochemical polarization technique. These specimens were fabricated from the heat number A356. The dimensions and a pictorial view of the polarization specimen are shown in Figure 2.2.
Compact Tension Specimen

Compact Tension (CT) specimens used in crack-growth-rate (CGR) measurements were machined from a flat bar (heat number 063417L02) of Alloy 718. Pre-cracked CT specimens having a 31.75-mm (1.25-inches) length, a 30.48-mm (1.2-inches) width, and a 6.35-mm (0.25-inch) thickness (Figure 2.3) were used in CGR testing. The machining of these specimens was done to comply with the size requirements prescribed by the ASTM Designations E 399-1990, E 647-2000 and E 1820-2001. The intersection of the crack starter notch tips with the two specimen surfaces was made equidistant from the top and bottom edges of the specimen within 0.005W, where W is the width of the specimen. A root radius of 0.25 mm (0.003-inch) was provided for the straight-through slot terminating in the V-notch of the specimen to facilitate fatigue pre-cracking at low stress intensity levels. A width (W) to thickness (B) ratio (W/B) of 4 was maintained to comply with the size requirements provided by the ASTM Designations E 399-1990, E 647-2000 and E 1820-2001. The detailed dimensions and a pictorial view of the CT specimen are shown in Figure 2.3.
Rectangular double-cantilever-beam (DCB) specimens, having a 101.6-mm (4-inches) length, a 25.4-mm (1-inch) width and a 9.525-mm (0.375-inch) thickness, were machined from the heat number HT0937EK to evaluate the SCC susceptibility of Alloy 718 in the presence of an acidic environment. The DCB specimens were loaded by inserting double-taper wedges of different thickness into a slot, as shown in Figure 2.4. These specimens had V-shaped side grooves on opposite sides, extended from the slot, to the opposite end to prevent branching of cracks, if any. The machining of these specimens was done according to the NACE Standard TM0177-1990. The side grooves were machined as 20% of the wall thickness, thus maintaining a web thickness (B_n) equal to 60% of the overall wall thickness (i.e. 5.715 mm or 0.225-inch in this case). The fabrication of the DCB specimens was done in such a way that the crack plane was perpendicular to the short-transverse direction, thus, ensuring that crack propagation would occur in the longitudinal rolling direction. The machining of the side grooves was done carefully to avoid overheating and cold-working. A total of 0.05 mm (0.002-inches)
of metal was removed during the final two passes of machining. The DCB specimens were capable to determine the average crack growth rate of Alloy 718, when exposed to the testing solution under a loaded condition resulting from the insertion of the wedges. The cracking susceptibility, determined by this technique, was measured in terms of the changes in crack length and the resultant CGR based on two exposure periods. The configuration of the DCB specimen, loading-wedges, and their pictorial views are illustrated in Figures 2.4 and 2.5.
Figure 2.4 Wedge-loaded DCB Specimen

Figure 2.5 Double-Taper Wedge
The pre-cracked DCB specimens were loaded by inserting double-taper wedges made of the same material (Alloy 718) to prevent galvanic interaction. Wedges of different thickness were inserted into the specimen to apply the desired load resulting from the separation of two arms. The thickness of the wedges was varied from 3.10-mm (0.12-inch) to 3.43-mm (0.13-inch) to provide arm-displacements ranging from 0.74-mm (0.02-inch) to 1.07-mm (0.04-inch), as given in Table 2.3.

<table>
<thead>
<tr>
<th>Wedge Thickness (mm (inch))</th>
<th>Arm-Displacement (mm (inch))</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.43 (0.13)</td>
<td>1.07 (0.04)</td>
</tr>
<tr>
<td>3.35 (0.13)</td>
<td>0.99 (0.03)</td>
</tr>
<tr>
<td>3.12 (0.12)</td>
<td>0.76 (0.02)</td>
</tr>
<tr>
<td>3.10 (0.12)</td>
<td>0.74 (0.02)</td>
</tr>
</tbody>
</table>

2.2.5 Charpy V-Notch Specimen

The structural materials to be used in the S-I cycle may also be subjected to impact loading due to a flow-induced vibration. Thus, it is necessary to evaluate the impact resistance of a material of interest at different temperatures in terms of its impact energy and a ductile-brittle-transition-temperature (DBTT). A conventional method to determine the impact energy is to use notched rectangular specimens having a configuration, shown in Figure 2.6. These specimens, known as the Charpy V-notch
(CVN) specimens, were subjected to impact loading by striking them with a pendulum at a central location opposite to the notched region. The CVN specimens of Alloy 718 machined from the heat number 218049766, had a 54.965-mm (2.164-inches) length, a 10-mm (0.394-inch) width, a 10-mm (0.394-inch) thickness, and a notch angle of 45°. These specimens were machined in such a way that the length of these specimens was parallel to the longitudinal rolling direction. The dimensions, shown in Figure 2.6, comply with the size requirements prescribed by the ASTM Designation E 23.\textsuperscript{[51]}

\begin{figure}
\centering
\includegraphics[width=\textwidth]{figure2.6.png}
\caption{Configuration of CVN Specimen}
\end{figure}

(a) Dimensions

(b) Pictorial View

Figure 2.6 Configuration of CVN Specimen
2.3 Testing Environment

An operating environment associated with the proposed S-I cycle can play an important role on the performance of the structural materials to be used in hydrogen generation using nuclear heat. As mentioned in Chapter 1, the S-I process will involve the formation and decomposition of H₂SO₄ and HI at different elevated temperatures. However, such elevated temperatures could not be accommodated in the Materials Performance Laboratory (MPL) of UNLV. Therefore, an aqueous solution containing H₂SO₄ and sodium iodide (NaI) was used for the evaluation of the SCC and the localized corrosion susceptibility (pitting/crevice) of Alloy 718 using the slow-strain-rate (SSR) and electrochemical polarization techniques, respectively. SCC evaluation using the DCB specimens was performed in an acidic solution of similar pH without the addition of NaI. The testing environment was contained inside an autoclave, which accommodated the pre-cracked and wedge-loaded DCB specimens for evaluation of their crack-growth rate (CGR) values as a function of the exposure period. The maximum temperatures used in the SSR and DCB testing were 90 and 100°C, respectively. However, the CPP experiments were performed at temperatures of 30, 60 and 90°C. The compositions of the testing environments are given in Table 2.4.

<table>
<thead>
<tr>
<th>Testing Type</th>
<th>Solution (pH)</th>
<th>NaI (gm)</th>
<th>Deionized Water (ml)</th>
<th>H₂SO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>SSR and Polarization</td>
<td>Acidity (1.0)</td>
<td>60.0</td>
<td>6000</td>
<td>Added to achieve the desired pH</td>
</tr>
<tr>
<td>DCB</td>
<td>Acidity (1.0)</td>
<td>------</td>
<td>6000</td>
<td>Added to achieve the desired pH</td>
</tr>
</tbody>
</table>
CHAPTER 3

EXPERIMENTAL PROCEDURES

The structural materials to be used in heat-exchangers associated with the S-I process must be capable of withstanding high temperatures approaching 1000°C in the presence of aggressive chemical species including H_2SO_4 and HI. In view of this rationale, this investigation has been focused on the evaluation of the metallurgical and environmental degradation behavior of Alloy 718 under conditions relevant to the S-I cycle. The metallurgical study included tensile properties evaluation at temperatures up to 1000°C in an inert atmosphere. Further, the crack-propagation-rate (CGR) of Alloy 718 has been determined at ambient and elevated temperatures using the in-situ DCPD crack monitoring device. Also, Charpy v-notch (CVN) specimens have been used to evaluate the impact toughness of this alloy at temperatures ranging from -40°C to 330°C. As to the corrosion studies, the evaluation of the susceptibility of Alloy 718 to environment-assisted degradations including stress corrosion cracking (SCC), localized corrosion (pitting/Crevice), and crack-growth has been extensively performed using different experimental techniques.

The metallographic and fractographic evaluations of all tested specimens have been performed by using optical microscopy and SEM, respectively. Further, the characterizations of defects due to plastic deformation, and precipitates resulting from phase transformation at elevated temperatures have been performed by TEM and
XRD, respectively. The different experimental techniques employed in this investigation for evaluation of both the metallurgical and corrosion behavior of Alloy 718 have been described 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 percent elongation (%El) and percent reduction in area (%RA) were evaluated using smooth cylindrical specimens. An Instron model 8862 testing equipment (Figure 3.1) was used to determine the tensile properties both at ambient and elevated temperatures. Initially, the tensile specimens were loaded at a strain rate of $10^{-3}$ sec$^{-1}$ according to the ASTM Designation E 8-2004.$^{[45]}$ Triplicate testing was performed at temperatures ranging from ambient to 1000°C, and an average value of each measured tensile parameter was recorded. Later, strain rates of $10^{-2}$ and $10^{-4}$ sec$^{-1}$ were used for testing Alloy 718 under tensile loading at several selected temperatures. The experimental data including the load, extension, engineering stress, and engineering strain were recorded in the data file. The engineering stress-strain (s-e) diagrams were automatically generated using a software program (Bluehill 2) $^{[52]}$, which also enabled acquisition of the resultant data. The magnitudes of YS, UTS, %El and, %RA were determined using this software. A linear-variable-displacement-transducer (LVDT) was used to determine the extension of the tensile specimen. Upon completion of testing, the magnitudes of %El and %RA were calculated using Equations 3.1 through 3.4.

\[
%\text{El} = \left[ \frac{L_f - L_o}{L_o} \right] \quad L_f > L_o \tag{Equation 3.1}
\]
\[
\%RA = \left[ \frac{A_o - A_f}{A_o} \right] A_o > A_f \quad \text{Equation 3.2}
\]

\[
A_o = \frac{\pi \times D_o^2}{4} \quad \text{Equation 3.3}
\]

\[
A_f = \frac{\pi \times D_f^2}{4} \quad \text{Equation 3.4}
\]

where \( L_o = \text{Initial gage length (inch)} \)

\( L_f = \text{Final gage length (inch)} \)

\( A_o = \text{Initial cross sectional area at the gage section (inch}^2\text{)} \)

\( A_f = \text{Final cross sectional area at the gage section (inch}^2\text{)} \)

\( D_o = \text{Initial gage diameter (inch)} \)

\( D_f = \text{Final gage diameter (inch)} \)

The Instron testing machine, shown in Figure 3.1, had an axial load transducer capacity of 22.5 kip (100 kN). It had a single screw electromechanical top actuator that was developed for both static and quasi-dynamic cyclic testing at multiple speed levels. This equipment consisted of a large heavy-duty load frame with an adjustable crosshead attached to the top grip, and a movable actuator with another grip located 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 specimen was mounted between two grips and pulled by the movable actuator. The load cell was used to measure the applied force on the tensile specimen. The movement of the upper
crosshead relative to the lower one measured the resultant strain of the specimen. The key specifications of the Instron equipment are given in Table 3.1.\cite{53}

![Intron Testing Machine](image)

**Figure 3.1 Intron Testing Machine**

**Table 3.1 Specifications of Intron Machine**

<table>
<thead>
<tr>
<th>Load Capacity at RT</th>
<th>Total Actuator Stroke</th>
<th>Maximum Ramp Rate</th>
<th>Actuator Attachment Threads</th>
<th>Load Cell Attachment Threads</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 kN</td>
<td>100 mm</td>
<td>350 mm/min</td>
<td>M30 × 2</td>
<td>M30 × 2</td>
</tr>
</tbody>
</table>
A split furnace (model MDS1735A) was attached to the testing system for loading the smooth tensile specimen at elevated temperatures. This furnace was capable of sustaining a maximum temperature of 1500°C, and consisted of two water-cooled stainless steel jackets that provided a safe ergonomic outer surface for operation. Two layers of micropores and ceramic fibers were placed inside this furnace. Six U-shaped molybdenum disilicide heating elements were used for attaining the desired testing temperature. The desired testing temperature was monitored by using three B-type thermocouples contained inside the test chamber. A separate control panel (model CU666F) was used to perform the overall monitoring of temperature during tensile loading. This control panel could accommodate a maximum heating rate of 10°C per minute. However, a slower heating rate of 4°C per minute was used during tensile testing to prevent any thermal shock of the pull rods and the fixtures inside the furnace. Since the grip material could undergo plastic deformation and phase transformation at elevated temperatures during straining of the specimen, a pair of custom-made grips made of high-strength and temperature-resistant MarM 246 alloy was used to hold the tensile specimen in an aligned position. A positive pressure was maintained inside the heating chamber by continuously purging nitrogen through it, which also ensured the elimination of oxygen from the test chamber, thus, preventing surface contamination/oxidation of the specimen.

3.2 Parameters Related to Tensile Deformation

The plastic deformation of a structural material at different temperatures and under different strain rates is known to be influenced by several metallurgical parameters, namely, dislocation density ($\rho$), activation energy ($Q$), and work hardening index ($n$).
Therefore, extensive efforts have been made in this investigation to determine the magnitudes of $\rho$, $Q$, and $n$ to develop a plausible mechanism of tensile deformation of Alloy 718 at different temperatures and strain rates. The analytical methods used to calculate these parameters are described in the following subsections.

3.2.1 Dislocation Density Calculation

The movement of dislocations, generated during plastic deformation of an alloy, can be influenced by the diffusion of solute or solvent elements as functions of temperature and strain rate. The precipitation of these elements can impede dislocation motion through its matrix as well as in the vicinity of its grain boundaries, thus, showing reduced plastic strains in the s-e diagrams. Therefore, substantial efforts have been made in this study to determine the magnitude of $\rho$ in the tested tensile specimens using their TEM micrographs. A line intersection method \cite{54} has been used to estimate the value of $\rho$ based on the superimposition of a grid consisting of horizontal and vertical test lines onto the TEM micrographs that contained dislocations. Grids of similar size were placed at ten locations of multiple TEM micrographs of the same tested specimen, as illustrated in Figure 3.2.\cite{21, 27, 54-57} Equation 3.5 was used to compute the magnitude of $\rho$. Electron-energy-loss-spectroscopy (EELS) was employed to determine the average thickness of the TEM samples using Equation 3.6.\cite{58} EELS used for such purpose was connected to the Tecnai $G^2$ S-TWIN TEM used in the development of micrographs. A TEM micrograph, showing thickness measurements at three locations of a sample, is displayed in Figure 3.3.
Figure 3.2 Line Intersection Method to Calculate $\rho$

Figure 3.3 Thickness Measurement by EELS
\[
\rho = \frac{1}{t} \left( \frac{\sum n_v}{\sum L_v} + \frac{\sum n_h}{\sum L_h} \right) \tag{3.5}
\]

\[t = \lambda \ln \left( \frac{I_t}{I_o} \right) \tag{3.6}\]

where

- \(\sum n_v\) = Number of intersections of vertical test lines with dislocations
- \(\sum n_h\) = Number of intersections of horizontal test lines with dislocations
- \(\sum L_v\) = Total length of vertical test lines (meters)
- \(\sum L_h\) = Total length of horizontal test lines (meters)
- \(t\) = Average thickness of the TEM sample
- \(\lambda\) = mean free path
- \(I_t\) = total intensity reaching the spectrometer
- \(I_o\) = zero-loss intensity reaching the spectrometer

3.2.2 Activation Energy Calculation

As mentioned in Chapter 1, the occurrence of DSA in Alloy 718, showing reduced failure strain (\(e_f\)) and serrations in the s-e diagrams within certain temperature regimes, could be a function of both temperature and strain rate. Even though the initial tensile testing was performed at temperatures ranging from ambient to 1000°C under an intermediate strain rate of \(10^{-3}\) sec\(^{-1}\), strain rates of \(10^{-2}\) and \(10^{-4}\) sec\(^{-1}\) were later used at temperatures of 100, 200 and 600°C, where serrations of greater heights were observed. For reliability, duplicate testing was performed under these experimental conditions.
It has been well-established\textsuperscript{[21-26]} that the occurrence of DSA is the result of diffusion of interstitial and/or substitutional solute elements into the matrix of a susceptible metallic material. The precipitation of such solute elements near the grain boundaries can lead to impaired dislocation mobility, thus, needing an enhanced driving force to activate the movement of dislocations. Such a driving force, also known as an activation energy (Q), can be estimated using a standard relationship\textsuperscript{[23, 26, 29, 59-60]} between a critical true plastic strain ($\varepsilon^*$) to initiate serrations, and a true strain rate ($\dot{\varepsilon}$), as given in Equation 3.7.\textsuperscript{[59-60]} It should be noted that both $\varepsilon^*$ and $\dot{\varepsilon}$ are influenced by temperature. It has been postulated\textsuperscript{[23, 26]} that $m$ and $\beta$ can be related to $C_v$ and $\rho_m$, respectively that are also dependent on true strain ($\varepsilon$) according to two empirical relationships, expressed by Equations 3.8 and 3.9.

$$
\varepsilon^* = K \varepsilon^0 \exp\left(\frac{Q}{RT}\right)
$$
Equation 3.7

$$
C_v \propto \varepsilon^m
$$
Equation 3.8

$$
\rho_m \propto \varepsilon^\beta
$$
Equation 3.9

where

- $Q =$ Activation energy at the onset of serrations (KJ/mole)
- $R =$ Universal gas constant (8.3144 J/mole Kelvin)
- $T =$ Absolute temperature (Kelvin)
- $m, \beta =$ Exponents related to the variation of vacancy concentration ($C_v$) and mobile dislocation density ($\rho_m$)
- $K =$ Constant
The magnitude of $Q$ was determined from the slope \( \left( \frac{Q}{R(m + \beta)} \right) \) of a straight line obtained by modifying Equation 3.7 into Equation 3.10. Knowing the value of \((m + \beta)\) from Equation 3.11, the magnitude of $Q$ was then computed from the slope of \( \left( \frac{Q}{R(m + \beta)} \right) \) indicated earlier. The mathematical analyses related to such modifications of equations are given below.

Taking natural logarithm of Equation 3.7,

\[
\ln(e^{(m+\beta)}) = \ln K e^{\left(\frac{Q}{RT}\right)}
\]

or \((m + \beta)\ln \varepsilon_c = \ln K + \ln \varepsilon + \frac{Q}{RT}\)

\[
\ln \varepsilon_c = \frac{Q}{R(m + \beta)} \times \frac{1}{T} + \frac{\ln K + \ln \varepsilon_c}{(m + \beta)}
\]

Equation 3.10

\[
\ln \left(\varepsilon^\ast\right) = (m + \beta)\ln \varepsilon_c - \left[\ln K + \frac{Q}{RT}\right]
\]

Equation 3.11

3.2.3 Computation of Strain-Hardening Exponent

Metals and alloys can experience work-hardening due to plastic deformation resulting from tensile loading beyond elasticity. The extent of work-hardening, which also provides a measure of deformability, is known to be related to both the true stress ($\sigma$) and the true strain ($\varepsilon$) by Equation 3.12. This equation is known as the flow equation or
Hollomon equation. The work-hardening index (n) can be determined from the slope of a line derived by plotting $\sigma$ versus $\varepsilon$ on a logarithmic scale. K is simply a constant (intercept), also known as the strain hardening coefficient.

$$\sigma = Ke^n$$  
Equation 3.12

Even though plastic flow in conventional metallic materials may be governed by the Hollomon relationship, a linear behavior was not observed between $\log \sigma$ and $\log \varepsilon$ for Alloy 718. A similar non-linear relationship between these two terms has also been reported in the literature, and in several recent investigations performed at MPL involving different alloys. In view of the resultant non-ideal situation, an alternate relationship proposed by Ludwigson was used to determine the magnitude of n. This relationship is given by Equation 3.13, where a new term ($\Delta$) was added to the classical Hollomon equation. $\Delta$ can be given by Equation 3.14, where $K_1$ and $n'$ are the intercept and the slope of a line, respectively, when $\log \Delta$ is plotted against $\varepsilon$. The value of n was determined by superimposing both the linear and non-linear plots of $\log \sigma$ versus $\log \varepsilon$, and extrapolating the coincided linear portion of both plots such that a minimum value of $\Delta$ (approaching zero) could be achieved. A schematic representation of this approach for determination of n is illustrated in Figure 3.4.

$$\sigma = (Ke^n + \Delta)$$  
Equation 3.13

$$\Delta = \exp\left(K_1 + n'\varepsilon\right)$$  
Equation 3.14
3.3 Crack Propagation Study

Crack-growth-rate (CGR) testing involving pre-cracked CT specimens of Alloy 718 was performed at ambient temperature, 100, 150 and 200°C in an aerated condition. The pre-cracking of the CT specimens was done under cyclic loading according to the ASTM Designation E 399-1999. The pre-cracked specimens were subjected to cyclic loading for evaluation of CGR, satisfying the conditions prescribed by the ASTM Designation E 647-2000. A load ratio (R = $\frac{\sigma_{\min}}{\sigma_{\max}}$) of 0.1 and a frequency of 1 Hz were used for both pre-cracking, and conducting CGR experiments under the influence of alternating stresses. The magnitudes of $\sigma_{\max}$ and $\sigma_{\min}$ were, however, varied to study the effect of stress range on CGR. An in-situ crack-monitoring device, based on direct-current-
potential-drop (DCPD), enabled the determination of crack extension (Δa), which led to the estimation of CGR and stress-intensity-factor (K) using a software program provided by the vendor.

In essence, the DCPD technique is based on the measurement of potential difference between two fixed points on either side of the pre-existing crack on a CT specimen when an external current is applied.\(^{[48, 70]}\) The resultant potential difference or an increase in potential drop due to an increase in electrical resistance can provide an accurate measurement of crack-growth and other related parameters, as described in the ASTM Designation E 647-2000.\(^{[48]}\) The magnitude of the current input was kept constant during DCPD measurements so that Ohm’s law could be obeyed, showing a direct proportionality between the resultant electrical resistance and the potential difference as cracking progressed. The determination of the crack length using this technique was based on the Johnson’s equation (Equation 3.15).

\[
a = \frac{W}{\pi} \cos^{-1} \left[ \cosh \left( \frac{\pi}{W} \times Y_o \right) \right] \\
\cosh \left( \frac{V}{V_r} \times \cosh^{-1} \left[ \cosh \left( \frac{\pi}{W} \times Y_o \right) \right] \right) \cosh \left( \frac{\pi}{W} \times a_r \right) \cos \left( \frac{\pi}{W} \times a_r \right)
\]

Equation 3.15

where \(a = \) Crack length (as defined in Test Method E 647)

\(a_r = \) Reference crack length from some other method
\( W = \text{Specimen width } (0 \leq 2a/W \leq 1) \)

\( V = \text{Measured change in voltage (volts)} \)

\( V = \text{Measured voltage corresponding to } a_r \)

\( Y_0 = \text{Voltage measurement lead spacing from the crack plane} \)

Subsequently, CGR under cyclic loading \( \left( \frac{da}{dN} \right) \) was plotted as a function of stress intensity range \( (\Delta K) \) at different testing temperatures. The steady state CGR is known to be governed by the Paris law, given by Equation 3.16. This equation can be modified in a logarithmic form (Equation 3.17), showing a linear relationship between \( \left( \frac{da}{dN} \right) \) and \( \Delta K \), from which the slope (m) of the resultant line could be estimated.

\[
\frac{da}{dN} = A(\Delta K)^m \quad \text{Equation 3.16}
\]

\[
\log \left( \frac{da}{dN} \right) = \log(A) + m \log(\Delta K) \quad \text{Equation 3.17}
\]

where \( \frac{da}{dN} = \text{CGR under cyclic loading, mm/cycle} \)

\( \Delta K = (K_{\text{max}} - K_{\text{min}}), \text{ MPa } \sqrt{m} \)

\( K_{\text{max}} = \text{Maximum stress intensity factor, MPa } \sqrt{m} \)

\( K_{\text{min}} = \text{Minimum stress intensity factor, MPa } \sqrt{m} \)

\( a = \text{Crack length, mm} \)

\( N = \text{Number of cycles of loading} \)
\[ A = \text{Constant} \]
\[ m = \text{Slope of the linear portion of the plot} \]

3.4 Impact Toughness Evaluation

The load-bearing capacity of a structural material can be impaired by the presence of flaws including cracks and/or notches on its surfaces. The presence of such flaws can create a triaxial state of stress around them. Thus, it is essential that the structural integrity of a metallic component containing such flaws be evaluated either by using fracture mechanics criteria or by determination of its impact resistance using CVN specimens. The impact resistance of a material of interest is commonly determined in terms of the energy absorbed by CVN specimens prior to their fracture due to the application of a sudden load imparted by a pendulum at a very high strain rate using an equipment, shown in Figure 3.5.
In general, medium and low-strength FCC materials and most HCP metals and alloys possess superior impact toughness even in the presence of a notch. On the contrary, high-strength materials are very susceptible to brittle failure under impact loading at all operating temperatures. However, the extent of failure with major engineering materials is more pronounced at lower temperatures, approaching cryogenic conditions. Thus, engineering materials can exhibit notch tough to notch brittle behavior with reducing temperature beyond a critical value, which is known as the ductile-brittle-transition-temperature (DBTT). The lower the magnitude of DBTT, the greater is the impact toughness of a material of interest. The precise determination of DBTT is a very difficult task. Conventionally, the impact energy of a material is determined as a function of temperature ranging from very low to appreciably high temperature. Lower values of
impact energy signify reduced ductility of a material. On the other hand, the lowest possible critical temperature is an indication of greater toughness.

The Charpy impact tester (model SI-1K3), used in this investigation, consisted of a heavy duty steel base on which the specimen holder (vise) and a heavy pendulum was mounted from a vertical shaft. This shaft was supported by precision ball bearings. A single lever located on top of this head assembly performed three functions of applying brake, releasing the break, and placing a latch. Prior to impact loading, the hammer was placed at a fixed position followed by the release of the latch, causing the rupture of the specimens. An indicator dial attached to this equipment recorded the absorbed energy in terms of either joules or feet-pounds. The CVN specimens were tested at temperatures of -40, -20, 125 and 330°C. Cryogenic temperatures (-40 and -20°C) were attained by immersing the CVN specimens into a mixture of dry ice and denatured alcohol contained inside an insulated styrofoam box. For testing at 125 and 330°C, heating of the CVN specimens was conducted using a portable furnace.

3.5 Slow-Strain-Rate Testing for SCC Evaluation

The susceptibility of Alloy 718 to SCC was determined by using smooth cylindrical specimens, subjected to continuous straining at a rate of 3.3 x 10^-6 sec^-1 in air and in the presence of an aqueous solution (pH~1) containing H_2SO_4 and NaI. This type of dynamic testing, also known as the slow-strain-rate (SSR) testing, was performed according to the ASTM Designation G 129. SSR testing is also known as the constant-extension-rate-testing (CERT).
A specially designed equipment, manufactured by Cortest Inc, was used to perform SSR testing at ambient temperature and 90°C. These machines provided accuracy and flexibility in evaluating the effect of a slower strain rate under a maximum loading capacity of 7500 lbs and linear extension rates ranging from $10^{-5}$ and $10^8$ in/sec. The SSR test setup consisted of a top-loaded actuator, a testing chamber, LVDT and a load cell. A top-loaded actuator was used to prevent its damage from the spilled solution, if any. A heating cartridge was connected to the bottom lid of the environmental chamber for elevated-temperature testing. A thermocouple was connected to the top cover of this chamber to monitor the temperature of the test solution. The load cell measured the applied load through an interface with the front panel. The LVDT recorded the displacement of the gage section of the specimen during straining. The experimental setup used in SSR testing is illustrated in Figure 3.6.

Figure 3.6 CERT Machines for SSR Testing
At relatively faster strain rates \((10^{-2} \text{ and } 10^{-3} \text{ sec}^{-1})\), a cylindrical specimen subjected to tensile loading can solely exhibit a mechanical failure even in the presence of a corrosive environment due to an inadequate environmental interaction. Similarly, at very slow strain rates \((10^{-7} - 10^{-8} \text{ sec}^{-1})\), the environmental interaction with the test specimen would be so vigorous that it may undergo dissolution at a very rapid rate. Therefore, a strain rate of \(3.3 \times 10^{-6} \text{ sec}^{-1}\) was selected in this study to optimize a synergistic effect of the applied stress and the testing environment to promote cracking.\(^{[72]}\)

Load versus displacement, and engineering stress-strain curves were automatically generated during SSR testing. Dimensions (length and diameter) of the test specimens were measured before and after testing. The cracking tendency, determined by this technique, was evaluated in terms of the time-to-failure (TTF), the true failure stress \((\sigma_f)\), and the ductility parameters including \(%E_1\) and \(%RA\). These parameters were determined from the stress-strain diagram, and the initial and final dimensions of the tested specimens. The magnitudes of \(%E_1\) and \(%RA\) were determined by using Equations 3.1 through 3.4, given in subsection 3.1. The magnitude of \(\sigma_f\) was computed by using Equation 3.18, where \(P_f\) and \(A_f\) are the failure load and the cross-sectional area at failure, respectively.

\[
\sigma_f = \frac{P_f}{A_f}
\]  

\text{Equation 3.18}
3.6 SCC Testing using DCB Specimens

The double-cantilever-beam (DCB) specimens, described in Chapter 2, were loaded by inserting double-taper wedges of different thicknesses into their slots according to a prescribed method.\cite{50, 73} The DCB specimens were precracked up to an approximate length of 2 mm using the Instron testing equipment. Precracking was performed according to the ASTM Designation E 399-1990 \cite{47} using a load ratio and frequency of 0.1 and 1 Hz, respectively. In order to determine the wedge thicknesses corresponding to low and high levels of stress intensity factor (K), a load (P) versus displacement (δ) curve was generated in the Instron machine using a DCB specimen. Subsequently, the linear portion (elastic region) of this curve was used to select two levels of displacement corresponding to two levels of load. The thickness of the different wedges was then computed using Equation 3.19.

\[ W = (t + \delta) \]  

Equation 3.19

\[ W = \text{Wedge thickness} \]

\[ t = \text{Gap between the two arms of the DCB specimen} \]

\[ \delta = \text{Displacement corresponding to the desired load (from the elastic P-δ curve)} \]

The precracked and wedge-loaded DCB specimens were immersed into a 100°C acidic solution contained inside an autoclave (Figure 3.7) for periods of 30 and 45 days. The specimens, upon completion of testing, were unloaded by removing the wedges.
using a tensile force in the Instron machine. Subsequently, the unloaded specimens were broken apart under tensile loading and the fracture morphologies were evaluated by SEM. The SEM micrographs developed on the broken surfaces enabled the identification of different types of failure corresponding to fatigue precrack, environment-assisted cracking (SCC), and fast fracture resulting from ductile tearing of the tested specimen. A montage was developed encompassing different fracture modes, which enabled the determination of the extent of environmental cracking beyond the precracked region.

Figure 3.7 Autoclave Test System

3.7 Electrochemical Testing for Localized Corrosion Evaluation

The susceptibility of Alloy 718 to localized corrosion (pitting and crevice) in an acidic solution (pH ~1) was evaluated by using the cyclic potentiodynamic polarization (CPP) technique. Testing was performed at 30, 60 and 90°C in accordance with the ASTM Designation G 61.[46] 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. The reference electrode was made of Ag/AgCl solution contained inside a Luggin probe having the test solution that also acted as a salt bridge. The CPP test set-up is shown in Figure 3.8. In order to maintain a continuity of the test solution during electrochemical polarization, the tip of the Luggin probe was placed within a distance of 2-3 mm from the specimen surface, as shown in Figure 3.9.

Figure 3.8  CPP Test Setup
Prior to the performance of the CPP experiments, the potentiostat was calibrated according to the ASTM Designation G 5. A typical calibration curve generated in a 30°C 1N (1 Normal) H$_2$SO$_4$ solution involving Type 430 ferritic stainless steel is illustrated in Figure 3.10. Upon verification of the reliability of this potentiostat, CPP experiments were performed under desired experimental conditions using small cylindrical specimens of Alloy 718, described in the previous chapter. The corrosion or the open circuit potential ($E_{\text{corr}}$) of the specimen was initially determined by immersing it into the test solution for approximately 30 minutes, followed by a potential scan in both forward and reverse direction, respectively at the rate of 0.17 mv/ sec. The localized corrosion susceptibility was determined in terms of the critical pitting potential ($E_{\text{pit}}$) and protection potential ($E_{\text{prot}}$), if any, from the resultant CPP diagram.
3.8 Metallographic Evaluation by Optical Microscopy

The metallurgical microstructure plays an important role in differentiating properties of one alloy versus other. Thus, the evaluation of microstructure constitutes a significant step in characterizing the performance of a material of interest. The metallographic techniques using an optical microscope enable the characterization of phases present, their distributions within grains, and their sizes that depend on the chemical composition of a material, and thermal treatments imparted to it. The operating 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 are reflected off the surface of the sample, which 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 samples of Alloy 718 were sectioned and subsequently mounted using the standard metallographic techniques. This procedure consisted of mounting the sample using a proper ratio of epoxy and hardener. The mounted specimens were ground with rotating discs containing the abrasive papers. The grinding procedure involved several stages using a finer paper each time. The sample was oriented perpendicular to the previous scratches after every step. The polished sample was then washed with deionized water to prevent any contamination and dried with ethanol and acetone. Finally, the polished specimen was etched using a mixture of 90 ml of hydrochloric acid (HCl) and 10 ml of hydrogen peroxide (H₂O₂), followed by microstructural evaluation using a Leica optical microscope, shown in Figure 3.11. This microscope had a maximum magnification of 1000X. A digital camera with a resolution of 1 mega pixel enabled image capture on a computer screen through use of an image acquisition software program.
3.9 Fractographic Evaluation by SEM

The extent and morphology of failure of the tested specimens were determined using scanning electron microscopy (SEM). Failures, in general, can be classified into two types including ductile and brittle.[61] 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, also known as cleavage, is characterized by crack propagation across the grains. For fractographic evaluations, the tested specimens were sectioned into 1/2 to 3/4 of an inch in length to accommodate them in a vacuum chamber of the SEM.

SEM works on a principle of collecting electrons from a metal filament and focusing them into a narrow beam in a manner similar to that of light waves. The beam scans
across the subject (specimen), which is synchronized with a spot on a computer screen. Electrons scattered from the subject are detected and can create a current, the strength of which makes the spot on the computer brighter or darker. This current can create a photograph-like image with an exceptional depth of field. Magnifications of several thousands could be achieved by use of an SEM. A JEOL-5600 SEM (Figure 3.12) having a maximum resolution of 100,000X was used in fractographic evaluations of this study.

![Figure 3.12 Scanning Electron Microscope](image)

3.10 Transmission Electron Microscopy

A Tecnai G² F30 S-TWIN transmission electron microscope (TEM), illustrated in Figure 3.13, was used to characterize dislocations and phases resulting from transformation of Alloy 718. This TEM, having an acceleration voltage of 300kV
allowed a point-to-point resolution of 0.2 nanometer and magnifications up to 1,000,000. This microscope was also equipped with a high-angle-annular-dark-field (HAADF) detector, an X-ray energy-dispersive-spectrometer (EDS), and a Gatan-image-filter (GIF). The TEM sample preparation steps are given next.

Figure 3.13 Tecnai G² F30 S-TWIN TEM

Sample preparation for the TEM study involved the utilization of both conventional and state-of-the-art techniques. Thin foil samples were prepared to ensure electron transparency, the thickness of these samples being in the range 50-100 μm. This was achieved through a series of operations, as described below.\(^{[77-81]}\)
• Initially, multiple circular disc-shaped samples were cut from the gage length of the failed tensile specimens up to a thickness of 500–700μm using a precision cutter.

• These discs were then mechanically ground to about 50-100 μm using a Beuhler grinding equipment (Figure 3.14). This process involved two steps; rough-grinding and fine-polishing. Specimen thickness was monitored during this process.

• These ground discs were punched into 3 mm diameter discs using a disc puncher, shown in Figure 3.15.

• Finally, jet electro-polishing was done to achieve the desired specimen thickness. A twin-jet TenuPol-5 electro polisher (Figure 3.16) was used for this purpose. This process involved removal of material from the sample surface as well as surface finish prior to the TEM evaluation. The thinnest area was obtained around the perforated area. The composition of the electrolyte used in this process was 5% perchloric acid (HClO₄) and 95% ethyl alcohol (C₂H₅OH). An applied potential of 55V, and a cryogenic temperature of -25°C were used during jet polishing. Extreme care was taken to control the flow of electrolyte to prevent the formation of anodic films that could cause etching of the specimen instead of polishing.
3.11 Phase Characterization

X-ray diffractometry (XRD) was used to characterize initial phases and phase changes, if any, in tensile specimens tested at ambient temperature, 700 and 800°C. A PANalytical X’Pert Pro X-Ray diffraction spectrometer (Figure 3.17) with Copper (Cu) Kα radiation was used for this purpose. Measurements were performed within an angular range (2θ) of 6 and 120°, with a step size of 0.017°. The d-spacings were calculated from the intensity (number of counts) vs. 2θ plots according to Equation 3.20, which is known
as Bragg’s law.\textsuperscript{[83]} Phase identification was performed by comparing the resultant d-spacing values to those existing in available standards and open literature.

$$n\lambda = 2d \sin \theta$$ \hspace{1cm} \text{Equation 3.20}

where $\lambda$ = Wave length of x-rays

$d$ = Spacing between planes in the atomic lattices

$\theta$ = Angle between the incident ray and the scattering planes

$n$ = An integer (1, in this investigation)

Figure 3.17 Panalytical X’PERT Pro XRD Spectrometer
CHAPTER 4

RESULTS

A candidate structural material identified for application in the S-I process within the purview of the NHI program must possess adequate tensile strength and excellent resistance to environment-assisted-degradations including SCC and localized corrosion. Additionally, they should be resistant to accelerated rate of cracking in the presence of flaws under operating conditions related to the hydrogen generation process. Based on these rationales, Alloy 718 has been subjected to numerous testing, as described in the previous chapter. An in-depth characterization of the tested specimens has also been performed by using conventional microscopic techniques. The overall results are presented in the following subsections, elucidating a mechanistic understanding of deformation and degradation observed in the tested specimens.

4.1 Microstructural Evaluation

The metallurgical microstructures of two heats of solution-annealed Alloy 718 are illustrated in Figure 4.1. Both micrographs revealed large austenitic grains, with a limited number of annealing twins in one heat, as expected for a Ni-base superalloy. Random precipitates were also observed, which were later analyzed by transmission electron microscopy (TEM).
Figure 4.1 Optical Micrographs, 90 ml HCl+10 ml H₂O₂
4.2 Tensile Properties Evaluation

The results of tensile testing of Alloy 718 are shown in Figure 4.2 in the form of an engineering stress versus engineering strain (s-e) diagram, superimposed as a function of the testing temperature. An evaluation of these s-e diagrams indicates that the magnitude of the yield strength (YS) was gradually reduced with an increase in temperature from ambient to 600°C, followed by its sudden enhancement at 700 and 800°C. Beyond 800°C, a significant drop in YS was observed. Interestingly, the strength in terms of the ultimate tensile strength (UTS) was gradually reduced with increasing temperature up to 1000°C. No distinction could, however, be made between the YS and UTS at temperatures ranging between 800°C and 1000°C.

Figure 4.2 s-e Diagram vs. Temperature
The average values of the different tensile properties including YS, UTS, percent elongation (%El) and percent reduction in area (%RA), derived from the s-e diagrams and the specimen dimensions (before and after testing) are given in Table 4.1. An evaluation of these data indicates that the lowest ductility in terms of %El was observed at 100°C, followed by its larger values up to a temperature of 600°C. The magnitude of %El was reduced again at temperatures of 700 and 800°C, followed by its significant enhancement at temperatures beyond 800°C, possibly due to increased plasticity at higher temperatures. The reduced ductility in terms of %El at 700 and 800°C is consistent with a greater strength in terms of YS at these two temperatures, as discussed earlier.

Table 4.1 Average Tensile Properties versus Temperature

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>YS MPa (ksi)</th>
<th>UTS MPa (ksi)</th>
<th>%El</th>
<th>%RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>407 (59)</td>
<td>855 (124)</td>
<td>66.35</td>
<td>57.98</td>
</tr>
<tr>
<td>100</td>
<td>365 (53)</td>
<td>807 (117)</td>
<td>64.86</td>
<td>52.49</td>
</tr>
<tr>
<td>200</td>
<td>345 (50)</td>
<td>793 (115)</td>
<td>68.92</td>
<td>50.41</td>
</tr>
<tr>
<td>300</td>
<td>324 (47)</td>
<td>786 (114)</td>
<td>68.90</td>
<td>51.37</td>
</tr>
<tr>
<td>400</td>
<td>317 (46)</td>
<td>765 (111)</td>
<td>69.19</td>
<td>54.32</td>
</tr>
<tr>
<td>500</td>
<td>310 (45)</td>
<td>758 (110)</td>
<td>70.66</td>
<td>49.63</td>
</tr>
<tr>
<td>600</td>
<td>317 (46)</td>
<td>738 (107)</td>
<td>72.32</td>
<td>50.66</td>
</tr>
<tr>
<td>700</td>
<td>483 (70)</td>
<td>717 (104)</td>
<td>42.98</td>
<td>36.64</td>
</tr>
<tr>
<td>800</td>
<td>510 (74)</td>
<td>531 (77)</td>
<td>49.77</td>
<td>78.45</td>
</tr>
<tr>
<td>900</td>
<td>165 (24)</td>
<td>172 (25)</td>
<td>101.92</td>
<td>95.03</td>
</tr>
<tr>
<td>1000</td>
<td>76 (11)</td>
<td>83 (12)</td>
<td>109.68</td>
<td>84.39</td>
</tr>
</tbody>
</table>

A careful examination of the s-e diagrams, presented in Figure 4.2, indicates that serrations of different heights were developed within a temperature regime of 100 to 600°C. The reduced ductility in terms of %El or failure strain (εf) at 100°C and the formation of serrations within a specific temperature range due to tensile loading of Alloy
718 can be attributed to a complex metallurgical phenomenon known as dynamic strain aging (DSA).\cite{21-32} A similar phenomenon has also been observed with different structural materials, recently studied at MPL by several investigators.\cite{27,28,84}

DSA of susceptible structural materials is known \cite{21-26} to be associated with the diffusion of either interstitial or substitutional solute elements into their matrices, and their subsequent precipitation in the vicinity of the grain boundaries, thus, impeding dislocation motion from one grain to other. Since the plastic deformation in a metallic material is activated by the movement of dislocations through its lattice, a reduced dislocation mobility will lead to lower plastic strain, as observed in this investigation when testing was performed at 100°C. A comprehensive characterization of the DSA phenomenon is generally performed by analyzing parameters such as the dislocation density ($\rho$), an activation energy ($Q$) for diffusion of solute/solvent elements and initiation of serrations, and a measure of the ease of plastic deformation in terms of work-hardening index ($n$). Analysis of each individual parameter will be presented later in different subsections.

The tensile data, presented in Table 4.1, revealed that Alloy 718 exhibited higher tensile strength and reduced ductility in terms of YS and %EI, respectively when testing was performed at 700 and 800°C. Such anomalous hardening of this alloy at these two temperatures could possibly be attributed to a phenomenon, known as yield strength anomaly (YSA). Different mechanisms \cite{33-35} have been proposed by numerous investigators to account for such an anomaly in the tensile behavior of structural materials. However, an unified mechanism of YSA in structural materials has not yet been established due to compositional and microstructural variations, and a difference in
the associated susceptible temperature regime. TEM and x-ray diffractometry (XRD) have been utilized in this study to develop a basic understanding of YSA in Alloy 718. A detailed analysis of the resultant data has been presented in a different subsection within this chapter.

The graphical representations of different tensile properties including YS, UTS, %El and %RA are illustrated in Figures 4.3 through 4.6 as a function of the testing temperature. The anomalous behavior of YS is illustrated in Figure 4.3, showing its gradual drop up to a temperature of 600°C, followed by a sudden enhancement up to a temperature of 800°C beyond which a drastic drop in YS was noted. Simultaneously, a sudden drop in ductility in terms of %El was noted at 700°C, as shown in Figure 4.5. A reduction in ductility in terms of %RA was also noted at this temperature, as illustrated in Figure 4.6. As to the variation of UTS, it was gradually reduced with an increase in temperature up to 700°C beyond which a drastic drop was observed.

![Figure 4.3 YS vs. Temperature](image-url)
1 ksi = 6.895 Mpa

Figure 4.4 UTS vs. Temperature

Figure 4.5 %El vs. Temperature
4.2.1 Dislocation Density Calculation

The occurrence of reduced $e_t$ and the formation of serrations in the s-e diagram at different temperatures have been cited $^{[59, 60]}$ to be the result of impaired dislocation mobility through the matrix and grain boundaries of a structural material. A reduced dislocation motion can be attributed to the diffusion and subsequent precipitation of solute/solvent elements at temperatures above ambient. Therefore, it was necessary to calculate the dislocation density ($\rho$) of specimens tested at and around the susceptible temperature regime where a tendency to DSA was observed with Alloy 718. The magnitude of $\rho$, determined for specimens tested at ambient temperature, 100, 200 and 300°C, was based on the emplacement of grids onto their TEM micrographs (Figures 4.7 through 4.10), as described in the previous chapter. Computation of $\rho$ at an ambient
temperature was performed to compare the resultant value to those obtained at three elevated temperatures. The average values of $\rho$, calculated by this method, are given in Table 4.2.

Figure 4.7 TEM Micrograph of Alloy 718 Tested at Ambient Temperature
Figure 4.8 TEM Micrograph of Alloy 718 Tested at 100°C

Figure 4.9 TEM Micrograph of Alloy 718 Tested at 200°C
Figure 4.10 TEM Micrograph of Alloy 718 Tested at 300°C

Table 4.2 $\rho$ versus Temperature

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\rho$ (Number/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>9.03E+16</td>
</tr>
<tr>
<td>100</td>
<td>1.29E+17</td>
</tr>
<tr>
<td>200</td>
<td>5.00E+16</td>
</tr>
<tr>
<td>300</td>
<td>6.26E+16</td>
</tr>
</tbody>
</table>
An evaluation of the data, presented in Table 4.2, indicates that a maximum value of \( p \) was observed with the tensile specimen tested at 100°C, which is also consistent with the lowest \( \varepsilon_f \) value seen in the s-e diagram. The variation of \( p \) with temperature is illustrated in Figure 4.11. A similar pattern on the variation of \( p \) with temperature has also been observed by investigators \([27, 28, 85, 86]\) at MPL involving austenitic and martensitic alloys that exhibited the DSA phenomenon.

![Graph showing \( \rho \) vs. Temperature](image)

**Figure 4.11 \( \rho \) vs. Temperature**

### 4.2.2 Activation Energy Calculation

A combination of different temperature and strain rate \( (\varepsilon) \) has been used to determine the magnitude of \( Q \) needed for the diffusion of solute/solvent elements, causing the initiation of serrations in the s-e diagram. A graphical representation, showing the superimposed s-e diagrams generated at different temperatures under an \( \varepsilon \) of \( 10^{-3} \text{ sec}^{-1} \)
has already been illustrated in Figure 4.1. The superimposed s-e diagrams, generated at 100, 200 and 600°C using \( \varepsilon \) values of \( 10^{-2} \) and \( 10^{-4} \) sec\(^{-1} \) are shown in Figures 4.12 and 4.13, respectively.

Figure 4.12 s-e Diagram vs. Temperature

Figure 4.13 s-e Diagram vs. Temperature
A detailed procedure to determine the magnitude of Q has been given in the previous chapter, showing the dependence of critical plastic strain \( (\varepsilon_c) \) on both true strain rate \( (\varepsilon) \) and temperature \( (T) \). The variation of \( \varepsilon_c \) with temperature at three different strain rates is illustrated in Figure 4.14, showing a gradual drop in \( \varepsilon_c \) with increasing temperature at a specific \( \varepsilon \). It should be noted that \( \varepsilon \) can be taken as \( \varepsilon \) due to an insignificant difference between both parameters at the onset of serrations. A similar pattern on the variation of \( \varepsilon_c \) with temperature, as a function of \( \varepsilon \), has been reported by other investigators.\([23, 27, 28]\)

![Figure 4.14 \( \varepsilon_c \) vs. Temperature](image)

**Figure 4.14 \( \varepsilon_c \) vs. Temperature**
The magnitude of Q was determined from the slope \( \frac{Q}{R(m + \beta)} \) of a straight line represented by Equation 3.10, given in the previous chapter. Knowing the value of \((m + \beta)\) from Equation 3.11, also given earlier, the magnitude of Q was then computed. The variation of \( \ln(e^c) \) versus \( \frac{1}{T} \) at different \( \varepsilon \) values are illustrated in Figure 4.15, showing a linear relationship with a slope of \( \frac{Q}{R(m + \beta)} \). The magnitude of \((m + \beta)\) was determined from the plot of \( \ln(e) \) versus \( \ln(e_c) \) as a function of several testing temperatures, as shown in Figure 4.16. Substituting the value of \((m + \beta)\) obtained from these plots, the average value of Q was computed, as given in Table 4.3. This table also shows the measured values of \((m + \beta)\) as functions of T and \( \varepsilon \). The resultant data also indicate that the magnitude of Q was gradually reduced with increasing temperature, suggesting that relatively lower driving forces would be necessary for dislocations to move from one grain to other during plastic deformation. An average Q value of approximately 43 KJ/mole was determined for the onset of serrations for the tested material. This value falls very close to the Q values estimated by other MPL researchers \([21, 27, 28, 86]\) on similar type of Ni-base alloys.
Figure 4.15 \( \ln(\varepsilon_c) \) vs. \( \frac{1}{T} \)

Figure 4.16 \( \ln(\dot{\varepsilon}) \) vs. \( \ln(\varepsilon_c) \)
Table 4.3 Calculated Values of \((m + \beta)\) and \(Q\)

<table>
<thead>
<tr>
<th>Temperature ((^\circ \text{C}))</th>
<th>(\varepsilon \text{ (sec}^{-1}) ()</th>
<th>(Q) ((\text{KJ/mole}))</th>
<th>(\text{Average }Q) ((\text{KJ/mole}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>(10^{-2})</td>
<td>25.59</td>
<td>25.59</td>
</tr>
<tr>
<td></td>
<td>(10^{-3})</td>
<td>25.59</td>
<td>25.59</td>
</tr>
<tr>
<td></td>
<td>(10^{-4})</td>
<td>25.59</td>
<td>64.1</td>
</tr>
<tr>
<td>200</td>
<td>(10^{-2})</td>
<td>12.39</td>
<td>12.39</td>
</tr>
<tr>
<td></td>
<td>(10^{-3})</td>
<td>12.39</td>
<td>36.1</td>
</tr>
<tr>
<td></td>
<td>(10^{-4})</td>
<td>36.1</td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>(10^{-2})</td>
<td>8.85</td>
<td>8.85</td>
</tr>
<tr>
<td></td>
<td>(10^{-3})</td>
<td>8.85</td>
<td>29.7</td>
</tr>
<tr>
<td></td>
<td>(10^{-4})</td>
<td>29.7</td>
<td></td>
</tr>
</tbody>
</table>

4.2.3 Determination of Work-Hardening Index

The average values of \(n\), determined from triplicate testing under tensile loading at temperatures of ambient to 600\(^\circ\)C under a strain rate of \(10^{-3} \text{ sec}^{-1}\), are given in Table 4.4. Obviously, very little or insignificant variation in \(n\) value was observed under these experimental conditions. As indicated earlier, the concept of DSA is also influenced by a variation in strain rate \(\varepsilon\) at a constant temperature. Therefore, testing was also performed at three selected temperatures of 100, 200 and 600\(^\circ\)C using strain rates of \(10^{-2}\) and \(10^{-4} \text{ sec}^{-1}\). The magnitudes of \(n\) corresponding to different strain rates applied during tensile loading at these selected temperatures are given in Table 4.5, showing a random variation in the \(n\) value.
Table 4.4 $n$ vs. Temperature

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>$\varepsilon$ (sec$^{-1}$)</th>
<th>$n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>$10^{-3}$</td>
<td>0.56</td>
</tr>
<tr>
<td>100</td>
<td>0.60</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>0.57</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>0.60</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>0.61</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td>0.63</td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>0.62</td>
<td></td>
</tr>
</tbody>
</table>

Table 4.5 $n$ vs. Temperature and Strain Rate

<table>
<thead>
<tr>
<th>Strain Rate (sec$^{-1}$)</th>
<th>Average $n$ Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature ($^\circ$C)</td>
</tr>
<tr>
<td></td>
<td>100</td>
</tr>
<tr>
<td>$10^{-2}$</td>
<td>0.61</td>
</tr>
<tr>
<td>$10^{-3}$</td>
<td>0.65</td>
</tr>
<tr>
<td>$10^{-4}$</td>
<td>0.65</td>
</tr>
</tbody>
</table>
4.2.4 Characterization of YSA

Alloy 718 exhibited an anomalous increase in YS when testing was performed at 700 and 800°C. Even though such anomalous behavior of YS has often been attributed to a phenomenon known as yield strength anomaly (YSA), a generalized interpretation of such behavior of structural materials is yet to be developed. In general, YSA has been observed in superalloys due to the precipitation of transformation products formed during plastic deformation at some critical temperatures.\[33-35, 87-90\]

Conventionally, Alloy 718 is used in an aged condition, which can promote strengthening of this alloy primarily due to the formation of gamma double prime (γ") phase, having a body-centered-tetragonal (BCT) crystal structure.\[91\] However, in order to achieve a reasonably high tensile strength with enhanced ductility for application in the NHI and NGNP programs, this alloy was tested in a solution-annealed condition. Thus, the formation of γ" was not anticipated in the tested material. However, the anomalous strengthening of Alloy 718 at 700 and 800°C could possibly be the result of γ" phase formation. Other undesirable phases, including delta (δ) phase, formed at these temperatures, may also cause hardening of Alloy 718, as cited in the open literature.\[92-95\]

While both phases can be identified with an Ni3Nb composition, the δ phase is characterized by an orthorhombic crystal structure.\[91\]

TEM was used to develop a basic understanding of hardening of Alloy 718 at 700°C. An evaluation of a TEM micrograph, illustrated in Figure 4.17, indicates the formation of spheroidal γ" precipitates that could have impaired the movement of dislocations by a process, known as Orowan strengthening mechanism.\[96, 97\] The presence of γ" was verified by using scanning transmission electron microscopy (STEM), coupled with
energy dispersive spectrometry (EDS), as shown in Figures 4.18 and 4.19. The EDS spectra exhibited a substantial amount of Ni and Nb in the precipitates, as expected in the $\gamma''$ phase. A time-temperature-transformation (TTT) diagram, cited in the open literature, justified the formation of $\gamma''$ at a temperature of 700°C.

Figure 4.17 TEM Micrograph (Bright Field) Showing $\gamma''$ Precipitates
Figure 4.18 STEM Image of $\gamma''$ Precipitates

Figure 4.19 EDS Spectra for $\gamma''$ Precipitate
As proposed by Orowan, dislocation loops (Figure 4.20) were formed around precipitates of γ" phases. In essence, hardening of Alloy 718 at 700°C may be rationalized by virtue of initial reduced dislocation motion due to the precipitation of γ", followed by forced passage of dislocations by bowing around these precipitates, causing Orowan loops. Such process can produce enhanced stresses, leading to an anomalous strengthening of Alloy 718, as seen in this study. It is interesting to note that dislocation pairs, also known as superlattice dislocations, were formed during such hardening of Alloy 718 at 700°C, as shown in Figure 4.21. A similar observation has also been made by other researchers investigating different types of Ni-base superalloys.
Figure 4.21 TEM Micrograph Showing Superdislocations

STEM micrograph of the specimen tested at 700°C, shown in Figure 4.22, also exhibited the formation of acicular precipitates that were subsequently analyzed by EDS. Once again, the EDS spectra showed the presence of Ni and Nb that could indicate the formation of equilibrium orthorhombic (Ni$_3$Nb) $\delta$ phase resulting from the transformation of $\gamma''$ phase at this temperature. Such observation is consistent with the findings of several other investigators [99] on a similar alloy. As to the phase characterization by XRD, the resultant spectra exhibited common constituents of Alloy 718, without showing any phase changes (Appendix G).
Figure 4.22 STEM Micrograph and EDS Spectra Showing Morphology and Composition of Acicular δ Phase Particles

4.3 Crack-Growth-Rate Evaluation

A highest operating temperature of 950°C has been recommended to achieve a maximum possible efficiency in H₂ generation using the S-I process within the NHI program. However, reduced temperatures may also be used during this process depending on the operating circumstances. Such fluctuation in the operating temperature, causing
thermal stresses, and the presence of minute surface flaws in the structural material may develop cracking that would eventually propagate with time. In order to address this issue, replicate CGR testing was performed under cyclic loading at several selected temperatures (ambient, 100, 150 and 200°C) involving precracked CT specimens of Alloy 718. A maximum testing temperature of 200°C was selected in this study to prevent blunting of the crack tip, if any.

A three stage curve is commonly observed when the rate of crack propagation under cyclic loading \( \frac{da}{dN} \) is plotted against a range in stress intensity factor \((\Delta K)\) on a logarithmic scale. Region I of such a curve, shown in Figure 4.23 \(^{100}\), is bounded by a threshold value \((\Delta K_{th})\) below which no measurable crack growth is commonly observed. In essence, these cracks behave as non-propagating cracks. Region II follows the Paris law, which represents a linear relationship between \( \log\left(\frac{da}{dN}\right) \) and \( \log(\Delta K) \) having a slope of \( m \). Finally, region III is a region of accelerated crack growth, at which \( K_{max} \) approaches a critical stress intensity factor \((K_{IC})\) of a material of interest.
Figure 4.23 $\log\left(\frac{da}{dN}\right)$ vs. $\log(\Delta K)$
The variations of \( \log\left(\frac{da}{dN}\right) \) versus \( \log(\Delta K) \) as a function of temperature are illustrated in Figures 4.24 through 4.27. These plots were all generated under a loading ratio (R) and frequency of 0.1 and 1 Hz, respectively. As expected, these figures revealed three distinct regions. The linear portion of these plots were subsequently reproduced in Figures 4.28 through 4.31, showing the value of m as a function of temperature.

Figure 4.24 CGR Data at Ambient Temperature
Figure 4.25 CGR Data at 100°C

Figure 4.26 CGR Data at 150°C
Figure 4.27 CGR Data at 200°C

Figure 4.28 CGR Data for Linear Region at Ambient Temperature
Figure 4.29 CGR Data for Linear Region at 100°C

Figure 4.30 CGR Data for Linear Region at 150°C
The overall CGR data are given in Table 4.6, showing the average values of all related parameters. The number of cycles (N), generated up to the final stage of the resultant plot, is graphically presented in Figure 4.32 as a function of the testing temperature. An evaluation of these data indicates that the magnitude of N was significantly reduced with an increase in temperature from ambient to 100°C. This result suggests that, for a comparable crack extension, the magnitude of N could be appreciably reduced at a higher temperature, even though the other experimental variables may remain constant. Interestingly, a negligible variation in the N value was observed for crack extension at temperatures between 100 and 150°C. However, a relatively higher reduction in N value was noted at 200°C, possibly due to the blunting of the crack tip resulting from increased plastic deformation at this temperature.
Table 4.6 Results of CGR Testing

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>ai (mm)</th>
<th>ar (mm)</th>
<th>Δa (mm)</th>
<th>$K_{\text{min}}$ (MPa $\sqrt{m}$)</th>
<th>$K_{\text{max}}$ (MPa $\sqrt{m}$)</th>
<th>m</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>5.08</td>
<td>16.16</td>
<td>11.08</td>
<td>2.55</td>
<td>7.82</td>
<td>4.08</td>
<td>78736</td>
</tr>
<tr>
<td>100</td>
<td>5.08</td>
<td>16.12</td>
<td>11.02</td>
<td>2.49</td>
<td>7.71</td>
<td>4.40</td>
<td>65099</td>
</tr>
<tr>
<td>150</td>
<td>5.08</td>
<td>15.14</td>
<td>10.06</td>
<td>2.49</td>
<td>6.58</td>
<td>4.71</td>
<td>64601</td>
</tr>
<tr>
<td>200</td>
<td>5.08</td>
<td>14.82</td>
<td>9.74</td>
<td>2.49</td>
<td>6.32</td>
<td>4.76</td>
<td>61844</td>
</tr>
</tbody>
</table>

Figure 4.32 N vs. Temperature
The data, shown in Table 4.6, indicate that the average value of $m$ within the linear portion of the CGR plot ranged from 4.08 and 4.76. The magnitude of $m$ has been cited to vary from 2.7 to 7.0 depending on a material of interest. Thus, the resultant $m$ values match well within the suggested range. The variations of $\Delta K_{(\text{final})}$ to the overall CGR in terms of $\left( \frac{da}{dN} \right)$ are given in Table 4.7 as a function of temperature. It is interesting to note that, between ambient temperature and 150°C, insignificant variation in CGR resulted even though the magnitude of $\Delta K_{(\text{final})}$ showed relatively higher values at lower temperatures. Finally, at 200°C, $\left( \frac{da}{dN} \right)$ was reduced by an order of magnitude, while $\Delta K_{(\text{final})}$ was further reduced, possibly due to the blunting of crack tip associated with enhanced plastic deformation at this temperature.

Table 4.7 $\Delta K_{(\text{final})}$ vs. $\left( \frac{da}{dN} \right)$ at Different Temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\Delta K_{(\text{final})}$ (MPa $\sqrt{m}$)</th>
<th>$\left( \frac{da}{dN} \right)$ (mm/Cycle)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>70.12</td>
<td>3.24E-2</td>
</tr>
<tr>
<td>100</td>
<td>69.25</td>
<td>1.11E-2</td>
</tr>
<tr>
<td>150</td>
<td>59.35</td>
<td>1.04E-2</td>
</tr>
<tr>
<td>200</td>
<td>56.84</td>
<td>8.34E-3</td>
</tr>
</tbody>
</table>
4.4 Impact Toughness Evaluation

A limited number of testing was performed to evaluate the impact resistance of Alloy 718 at temperatures of -40, -20, 125 and 330°C. A wide range in temperature was selected to evaluate the resistance of CVN specimens to failure due to dynamic loading at cryogenic and elevated temperatures. The results, given in Table 4.8, indicate that between -40 and 125°C, insignificant variation in the average energy absorbed in fracturing the specimens was observed. However, at 330°C, Alloy 718 exhibited a greater resistance to failure, showing higher impact energy that signifies enhanced ductility. The overall Charpy data suggest that Alloy 718 would be capable of withstanding impact loading despite a wide variation in service temperature. Compared to the impact resistance of typical Fe-base martensitic steels [85] recently studied at MPL, the resultant data generated on Alloy 718 was quite respectable from a performance point of view.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Impact Energy (ft. lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-40°C</td>
<td>70</td>
</tr>
<tr>
<td>-20°C</td>
<td>73</td>
</tr>
<tr>
<td>125°C</td>
<td>79</td>
</tr>
<tr>
<td>330°C</td>
<td>90</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Impact Energy (ft. lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-40°C</td>
<td>71</td>
</tr>
<tr>
<td>-20°C</td>
<td>77</td>
</tr>
<tr>
<td>125°C</td>
<td>77</td>
</tr>
<tr>
<td>330°C</td>
<td>93</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Impact Energy (ft. lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-40°C</td>
<td>76</td>
</tr>
<tr>
<td>-20°C</td>
<td>69</td>
</tr>
<tr>
<td>125°C</td>
<td>77</td>
</tr>
<tr>
<td>330°C</td>
<td>92</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Impact Energy (ft. lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>-40°C</td>
<td>72.3</td>
</tr>
<tr>
<td>-20°C</td>
<td>73</td>
</tr>
<tr>
<td>125°C</td>
<td>77.6</td>
</tr>
<tr>
<td>330°C</td>
<td>91.6</td>
</tr>
</tbody>
</table>
4.5 Results of SSR Testing

The results of duplicate SCC testing, performed under a slow-strain-rate (SSR) condition, are illustrated in Figure 4.33 in the form of engineering stress versus strain (s-e) diagram, superimposed as functions of the testing environment and temperature. An examination of these diagrams indicates a gradual reduction in failure strain \( (e_f) \) as the environmental condition was changed from air to RT-solution to 90°C-solution. The magnitudes of different SCC parameters including \( \%E_1 \), \%RA, TTF and true failure stress \( (\sigma_f) \), determined from the s-e diagrams and the specimen dimensions, are given in Table 4.9. These results indicate that the cracking tendency of Alloy 718 in the 90°C acidic solution was enhanced by virtue of the reduced \%E_1 and \( \sigma_f \) values.

![Figure 4.33 Comparison of s-e Diagrams under Different Testing Conditions](image-url)
Table 4.9 SSR Test Results

<table>
<thead>
<tr>
<th>Environment</th>
<th>%EI</th>
<th>%RA</th>
<th>$\sigma_r$, MPa (ksi)</th>
<th>TTF (hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT-Air</td>
<td>63.1</td>
<td>53.5</td>
<td>1593 (231)</td>
<td>56.7</td>
</tr>
<tr>
<td>RT-Solution</td>
<td>62.5</td>
<td>53.6</td>
<td>1594 (231)</td>
<td>55.7</td>
</tr>
<tr>
<td>90°C-Solution</td>
<td>60.7</td>
<td>53.9</td>
<td>1529 (222)</td>
<td>55.0</td>
</tr>
</tbody>
</table>

4.6 Results of DCB Testing

Table 4.10 shows the results of SCC testing involving pre-cracked and wedge-loaded DCB specimens of Alloy 718. While, specimens 1 and 2 were loaded by using thinner wedges, thicker wedges were used in specimens 3 and 4. The resultant data indicate that, the specimens (no. 2 and 4), subjected to longer exposure period (45 days), exhibited measurable crack extension ($\Delta a$) and average CGR. However, the DCB specimens (no. 1 and 3) tested for 30 days did not show any measurable crack extension, irrespective of the wedge thickness. The resultant data also suggest that the specimen 4 loaded with a thicker wedge suffered from greater crack extension and CGR, compared to those encountered by specimen 2 that was loaded with a thinner wedge for a comparable exposure period of 45 days.
Table 4.10 Results of DCB Testing *

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>ai (mm)</th>
<th>af (mm)</th>
<th>Δa (mm)</th>
<th>Test Duration (days)</th>
<th>CGR (mm/sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>30.5739</td>
<td>30.5739</td>
<td>0</td>
<td>30</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>30.5739</td>
<td>31.1079</td>
<td>0.534</td>
<td>45</td>
<td>1.37E-7</td>
</tr>
<tr>
<td>3</td>
<td>30.5739</td>
<td>30.5739</td>
<td>0</td>
<td>30</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>30.5739</td>
<td>31.3339</td>
<td>0.76</td>
<td>45</td>
<td>1.95E-7</td>
</tr>
</tbody>
</table>

* Environment: 100°C Acidic Solution (pH~1.0)

4.7 Results of Electrochemical Testing

The results of localized corrosion study using the electrochemical cyclic potentiodynamic polarization (CPP) technique are illustrated in Figures 4.34 through 4.36. None of these CPP diagrams did exhibit any classical positive hysterisis loop during reverse potential scan. On the contrary, negative hysterisis loop, indicating the formation of passive film, was noted.\[^{103, 104}\] Since no intersection of the reverse polarization curve with the passive region occurred, the magnitude of protection potential (E\text{prot}) could not be determined. The measured corrosion potential (E\text{corr}) and the critical pitting potential (E\text{pit}), obtained from these diagrams are given in Table 4.11 as a function of the testing temperature. The magnitude of E\text{corr} became more active (negative) with increasing temperature, confirming observations made by other investigators.\[^{103, 104}\] However, no consistent pattern on the effect of temperature on E\text{pit} was observed.
$E_{corr} = 284\text{ mV (Ag/AgCl)}$

$E_{pit} = 650\text{ mV (Ag/AgCl)}$

$E_{pit} = 830\text{ mV (Ag/AgCl)}$

$E_{corr} = 258\text{ mV (Ag/AgCl)}$

Figure 4.34 CPP Diagram Generated in 30°C Acidic Solution

Figure 4.35 CPP Diagram Generated in 60°C Acidic Solution
Figure 4.36 CPP Diagram Generated in 90°C Acidic Solution

Table 4.11 Results of CPP Testing

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$E_{\text{corr}}$, mV (Ag/AgCl)</th>
<th>$E_{\text{pit}}$, mV (Ag/AgCl)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>284</td>
<td>650</td>
</tr>
<tr>
<td>60</td>
<td>258</td>
<td>830</td>
</tr>
<tr>
<td>90</td>
<td>239</td>
<td>680</td>
</tr>
</tbody>
</table>
4.8 Fractographic Evaluations

The results of fractographic evaluations of tensile specimens tested at ambient temperature, 100, 400, 600 and 700°C under a strain rate \(\dot{\varepsilon}\) of \(10^{-3}\) sec\(^{-1}\) are illustrated in Figure 4.37. An evaluation of the SEM micrographs, shown in Figure 4.37 (a, b and c) revealed very minute intergranular cracking, and predominantly dimpled microstructures in specimens tested at ambient temperature, 100 and 400°C. However, the specimen tested at 600°C solely exhibited dimpled microstructures (Figure 4.37d), indicating a ductile failure. On the other hand, the specimen that exhibited an anomalous increase in YS at 700°C was characterized by combined intergranular and transgranular brittle failures, as shown in Figure 4.37e. The tensile specimens tested at 100°C under \(\dot{\varepsilon}\) values of \(10^{-2}\) and \(10^{-4}\) sec\(^{-1}\) also revealed predominantly dimpled microstructures (Figures 4.38 and 4.39, respectively) and insignificant cracking similar to that observed with the specimen tested at the same temperature but under an intermediate \(\dot{\varepsilon}\) value of \(10^{-3}\) sec\(^{-1}\).
(a) Ambient Temperature

(b) 100°C
Figure 4.37 SEM Micrographs of Tensile Specimens ($\dot{\varepsilon} \sim 10^{-3} \text{ sec}^{-1}$)

Figure 4.38 SEM Micrograph of Tensile Specimen ($\dot{\varepsilon} \sim 10^{-2} \text{ sec}^{-1}$)
Figure 4.39 SEM Micrograph of Tensile Specimen ($\dot{e} \sim 10^{-4}$ sec$^{-1}$)

The failure morphologies of CT specimens of Alloy 718, tested under cyclic loading (CGR testing) at different temperatures, are illustrated in SEM micrographs presented in Figures 4.40 through 4.43. While all tested specimens exhibited a combination of striations and dimpled microstructures corresponding to cyclic loading and ductile tearing, respectively, the specimens tested at the elevated temperatures showed well-defined striations, spaced uniformly within the matrix of the test material. Further, the dimples became larger in size at higher temperatures due to enhanced plasticity.
(a) Striations (Fatigue Failure)

(b) Dimples (Ductile Failure)

Figure 4.40 SEM Micrograph of CT Specimen (CGR Testing, Room Temperature)
(a) Striations (Fatigue Failure)

(b) Dimples (Ductile Failure)

Figure 4.41 SEM Micrograph of CT Specimen (CGR Testing, 100°C)
(a) Striations (Fatigue Failure)

(b) Dimples (Ductile Failure)

Figure 4.42 SEM Micrograph of CT Specimen (CGR Testing, 150°C)
Figure 4.43 SEM Micrograph of CT Specimen (CGR Testing, 200°C)
The SEM micrographs of the CVN specimens, tested at -40 and 330°C, are illustrated in Figures 4.44 and 4.45, respectively. A cursory examination of these fractographs revealed numerous cracks on the broken surface of the specimen tested at the cryogenic temperature. On the contrary, the specimen tested at 330°C exhibited ductile failures characterized by dimples alone.

Figure 4.44 SEM Micrograph of CVN Specimen Tested at -40°C
The SEM micrographs of the primary fracture surface of the smooth cylindrical specimens of Alloy 718, tested under a SSR condition, are illustrated in Figure 4.46 (a, b and c). An examination of these micrographs clearly revealed predominantly ductile failures characterized by dimples, irrespective of the testing temperature and environment. However, a few tiny cracks were observed in all three micrographs. The morphologies of failure at three distinct regions of the fracture surface of a DCB specimen, exposed for 45 days in the 100°C acidic solution, are shown in Figure 4.47. As expected, these three regions were characterized by striations due to cyclic loading, cleavage failure due to SCC, and dimples resulting from ductile tearing.
(a) Room Temperature-Air

(b) Room Temperature-Acidic Solution
Figure 4.46 SEM Micrographs of SSR Specimens Tested Under Different Experimental Conditions

(c) 90°C - Acidic Solution
Figure 4.47 Three Regions of Failure in DCB Specimen used in SCC Testing
CHAPTER 5

DISCUSSION

Nuclear hydrogen generation using the proposed thermochemical S-I process necessitates the identification and selection of a suitable structural material possessing adequate tensile strength, resistance to plastic deformation under cyclic and impact loading, and excellent resistance to environment-assisted degradations. These requirements are warranted in view of unusually high operating temperatures, and the presence of hostile chemical species involved in the decomposition of \( \text{H}_2\text{SO}_4 \). Austenitic Alloy 718 has been identified to be one of the candidate structural materials for evaluation of its metallurgical and corrosion behavior under conditions relevant to the S-I cycle. The structural stability of this alloy has been determined by performing tensile testing as functions of metallurgical and mechanical variables that led to the development of a basic understanding of its deformation behavior. Simultaneously, an estimation of crack-growth-rate (CGR) has been achieved under a combined effect of cyclic loading and flaws at ambient and elevated temperatures. Environmental degradations of Alloy 718 including stress-corrosion-cracking (SCC) and localized corrosion have also been addressed in this study. Finally, extensive microscopic evaluations have been performed on the tested specimens, which provided a rationalization to the proposed mechanistic understanding of both metallurgical and environmental degradation phenomena observed with Alloy 718.
Two important metallurgical phenomena, namely dynamic strain aging (DSA) and yield strength anomaly (YSA) have been identified based on the resultant tensile data, generated at ambient and elevated temperatures under an applied engineering strain rate ($\varepsilon^\circ$) of $10^{-3}$ sec$^{-1}$. The DSA phenomenon of Alloy 718 has been characterized by reduced plastic strain ($\varepsilon_f$) at 100°C, and formation of serrations of different heights within a temperature range of 100 to 600°C. Simultaneously, an anomalous increase in yield strength (YS) was observed at 700 and 800°C that has been verified to be the result of the occurrence of YSA phenomenon.

DSA of engineering metals and alloys has been known to be associated with the diffusion of solute/solvent elements through their matrices at elevated temperatures, and their subsequent precipitation near dislocations and grain boundaries. Such incidents can lead to a minimization or reduction in dislocation mobility through the grain boundaries, causing reduced $\varepsilon_f$, as seen in the s-\varepsilon diagram. The formation of serrations within the susceptible temperature regime is also a function of the precipitation of diffused elements, thus, needing a greater driving force for continued deformation. Obviously, alternate higher and lower tensile stresses would be generated during such progressive deformation, as evidenced by the resultant serrations. Since plasticity is governed by the movement of dislocations at elevated temperatures, the dislocation density ($\rho$) has been computed at several temperatures within and beyond the susceptible temperature regime. Justifiably, a maximum value of $\rho$ was observed at 100°C, where the magnitude of $\varepsilon_f$ was minimum.

The onset of serrations at different temperatures is also a function of true strain rate ($\varepsilon^\circ$). Accordingly, activation energies (Q) needed for the initiation of serrations were
determined as functions of temperature and $\varepsilon$. An average $Q$ value of 43 KJ/mole has been computed for Alloy 718, which is very close to the $Q$ values estimated on other Ni-base superalloys recently studied by several MPL investigators. As to the effect of temperature and $\varepsilon$ on the work-hardening exponent ($n$), a random variation in $n$ was observed.

Based on the transmission electron microscopic (TEM) evaluations, the occurrence of YSA phenomenon of Alloy 718 at susceptible temperatures could be attributed to the formation of $\gamma''$ and $\delta$ phases, both having an Ni$_3$Nb composition. The formation of spheroidal $\gamma''$ precipitates could have impaired dislocation motion, as proposed by Orowan. Strengthening, according to this mechanism, was thought to be the result of enhanced driving forces needed for the passage of dislocations through precipitates, thus, forming Orowan loops. The generation of superlattice dislocations could also account for the anomalous increase in YS of this alloy. The formation of acicular $\delta$ precipitates due to the transformation of $\gamma''$ phase has been verified based on evaluations by scanning transmission electron microscopy (STEM) coupled with energy dispersive spectroscopy (EDS).

Consistent with the observations made by other researchers, the results of crack-growth-rate (CGR) study involving pre-cracked CT specimens exhibited three distinct regions in the $\log\left(\frac{da}{dN}\right)$ versus $\log(\Delta K)$ plots. The linear portion of these plots followed the Paris equation, showing slopes ranging from 4.08 to 4.76, depending on the testing temperature. The number of cycles ($N$) needed for crack extension was drastically reduced as the temperature was increased from ambient to 100°C. The magnitude of
\( \frac{da}{dN} \) was substantially reduced at 200°C, possibly due to a plasticity-induced blunting of crack tip in the CT specimen. As to the effect of temperature on the impact resistance of Alloy 718, an insignificant variation in impact energy was observed at the cryogenic temperatures and 125°C. A relatively higher impact energy was noted at 330°C, indicating a greater ductility due to enhanced plasticity.

The susceptibility of Alloy 718 to SCC, as determined by the SSR technique, was slightly more pronounced in the 90°C acidic solution by virtue of the resultant greater percent elongation (\(\%\text{El}\)) and true failure stress (\(\sigma_f\)). The SCC study involving pre-cracked and wedge-loaded double-cantilever-beam (DCB) specimens showed measurable crack extension (\(\Delta a\)) and CGR, when testing was performed in an acidic solution for a duration of 45 days. As to the localized corrosion behavior of this alloy, the magnitude of corrosion potential (\(E_{\text{corr}}\)) became more active with increasing temperature. However, an inconsistent pattern on the effect of temperature on the critical pitting potential (\(E_{\text{pit}}\)) was noted.

Fractographic evaluations of the tensile specimens by scanning electron microscopy (SEM) revealed predominantly ductile failures along with a few minute cracks at ambient temperature, 100 and 400°C. The morphology of failure in similar specimens tested at 100°C was not altered due to a change in \(e\). While ductile failures, characterized by dimples, were observed in the specimen tested at 600°C, a combination of intergranular and transgranular brittle failure was seen in the specimen tested at 700°C. All specimens, used in CGR testing, showed striations and dimples in the SEM micrographs, which are the characteristics of fatigue failure and ductile tearing, respectively. The CVN specimen
tested at a cryogenic temperature revealed multiple cracks and dimples on its fracture surface. However, solely ductile failures, characterized by dimpled microstructures, were seen with the specimen tested at 330°C. With respect to the failure morphology of specimens used in the SSR testing, a very few tiny cracks were observed within the dimpled microstructures along their primary fracture surfaces. Finally, the broken surface of a DCB specimen, that showed measurable crack-growth, exhibited characteristics of fatigue failure, SCC and fast fracture in the SEM micrographs.
CHAPTER 6

SUMMARY AND CONCLUSIONS

The metallurgical and corrosion behavior of Alloy 718 have been evaluated as functions of different experimental variables. The metallurgical properties studied include plastic deformation under tensile loading, crack propagation under cyclic loading, and impact resistance. As to the corrosion behavior, the susceptibility of this alloy to stress-corrosion-cracking (SCC) and localized corrosion has been determined in an acidic solution. A mechanistic understanding of plastic deformation has also been developed using transmission electron microscopy. Further, scanning electron microscopy (SEM) has been used to determine the morphology of failure of all tested specimens. The overall data, and conclusions derived from this investigation are summarized below.

- As expected, the metallurgical microstructures consisted of large austenitic grains resulting from solution-annealing.
- Reduced plastic strain ($\varepsilon_f$) and formation of serrations, observed in the superimposed s-c diagrams during tensile testing, were related to a metallurgical phenomenon, known as dynamic strain aging.
- The magnitude of $\varepsilon_f$ was minimum at 100°C, where a maximum dislocation density ($\rho$) was computed. A higher value of $\rho$ at this temperature could be the result of blockage of dislocation motion due to the diffusion and precipitation of
solute/solvent elements in the vicinity of grain boundaries. Such reduction in
dislocation mobility would lead to reduced plastic flow, thus causing lower value
of $\varepsilon_f$ as seen in this study.

- The average activation energy for the onset of serrations was approximately 43
  KJ/mole that was estimated based on testing performed under different
temperatures and strain rates ($\varepsilon$).

- No consistent pattern on the variation of work-hardening index with temperature
  and $\varepsilon$ was observed.

- An anomalous increase in yield strength and reduced ductility at 700 and 800°C
could be attributed to the occurrence of yield strength anomaly (YSA). Such
anomalous behavior could be the result of $\gamma''$ and $\delta$ phase formation at these
temperatures, causing enhanced hardening during the passage of dislocations
through these precipitates, forming Orowan loops. Superlattice dislocations were
also generated during this hardening process.

- The steady-state region of $\log \left(\frac{da}{dN}\right)$ versus $\log(\Delta K)$ plots, obtained during
  crack-growth-rate (CGR) testing, followed the Paris law, showing a linear
  relationship. A drastic reduction in the number of cycles (N) needed for crack
  extension was noted at 100°C. However, a minimum value of N was observed at
  200°C, possibly due to plasticity-induced blunting of the crack tip.

- Between cryogenic temperatures and 125°C, Alloy 718 did not exhibit any
  appreciable variation in impact resistance. At a higher temperature, enhanced
ductility, showing greater impact energy, was observed.
• The susceptibility of Alloy 718 to SCC, using the slow-strain-rate (SSR) technique, was more pronounced in the 90°C acidic solution. SCC study, using double-cantilever-beam (DCB) specimens, showed relatively higher crack-growth following longer exposure time in an acidic solution.

• The magnitude of corrosion potential became more active with increasing temperature, as determined by the cyclic potentiodynamic polarization technique.

• The specimens, tested under tensile loading at ambient, 100 and 400°C, exhibited predominantly dimpled microstructures and a few tiny cracks in the SEM micrographs. However, at 600°C, solely ductile failures characterized by dimples were seen. On the contrary, the fracture morphology of a specimen that exhibited the YSA phenomenon was characterized by combined intergranular and transgranular brittle failures.

• The SEM micrographs of the compact tension specimens used in the CGR testing revealed striations and dimples, indicating cyclic loading and ductile tearing, respectively. The Charpy V-notch specimens, tested at cryogenic temperatures, showed numerous cracks on its broken surface.

• Irrespective of the testing temperature and environment, the specimens used in the SSR testing revealed predominantly ductile failures. The DCB specimens, tested for a longer duration, showed striations, cleavage facets and dimples associated with cyclic loading, SCC and fast fracture, respectively.
CHAPTER 7

SUGGESTED FUTURE WORK

Crack-growth-rate study of Alloy 718 may be performed at temperatures above 200°C to determine a threshold temperature above which crack-growth measurements may become insignificant. Additionally, efforts should be made to determine its fracture toughness under different experimental conditions.
APPENDIX A

TENSILE TESTING DATA

A1 Stress-Strain Diagrams using smooth specimens tested at a strain rate of $10^3$ sec$^{-1}$.

(A conversion factor of 1 ksi = 6.895 MPa can be used wherever applicable)

A1.1 Stress-Strain Diagrams at Room Temperature

Sample 1
A1.2 Stress-Strain Diagrams at 100°C

Sample 1

Sample 2
Sample 3

A1.3 Stress-Strain Diagrams at 200°C

Sample 1
Sample 2

Sample 3
A1.4 Stress-Strain Diagrams at 300°C

Sample 1

Sample 2
A1.5 Stress-Strain Diagrams at 400°C
Sample 2

Sample 3

122
A1.6 Stress-Strain Diagrams at 500°C

Sample 1

Sample 2
Sample 3

A1.7 Stress-Strain Diagrams at 600°C

Sample 1
Sample 2

Sample 3
A1.8 Stress-Strain Diagrams at 700°C

Sample 1

Sample 2
Sample 3

A1.9 Stress-Strain Diagrams at 800°C

Sample 1
A1.10 Stress-Strain Diagrams at 900°C

Sample 1

Sample 2
Sample 3

A1.11 Stress-Strain Diagrams at 1000°C

Sample 1
Sample 2

Sample 3
Table A1 Combined Tensile Properties at a Strain Rate of $10^{-3}$ sec$^{-1}$ (1 ksi = 6.895 MPa)

<table>
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<th>Sample #</th>
<th>YS (ksi)</th>
<th>UTS (ksi)</th>
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<th>%RA</th>
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A2 Stress-Strain Diagrams using Smooth Specimens Tested at a Strain Rate of $10^{-2}$ sec$^{-1}$

(Conversion factor of 1 ksi = 6.895 MPa can be used wherever applicable)

A2.1 Stress-Strain Diagrams at 100°C

Sample 1
Sample 2

A2.2 Stress-Strain Diagrams at 200°C

Sample 1
Sample 2

A2.3 Stress-Strain Diagrams at 600°C

Sample 1
Sample 2
A3 Stress-Strain Diagrams using Smooth Specimens Tested at a Strain Rate of $10^{-4}$ sec$^{-1}$

(Conversion factor of 1 ksi = 6.895 MPa can be used where ever applicable)

A3.1 Stress-Strain Diagrams at 100°C

Sample 1
A3.2 Stress-Strain Diagrams at 200°C

Sample 1

Sample 2
A3.3 Stress-Strain Diagrams at 600°C
Sample 2
A4 Strain Hardening Exponent (n) versus Temperature

A4.1 n vs. Temperature at a Strain Rate of $10^3$ sec$^{-1}$

n vs. Temperature (based on triplicate testing)
A4.2 n vs. Temperature and Strain Rate ($10^{-2}$, $10^{-3}$ and $10^{-4}$ sec$^{-1}$)

Set 1

Set 2
APPENDIX B

RESULTS OF CGR TESTING

Ambient Temperature Set 1:

CGR Data at Ambient Temperature
log \left( \frac{da}{dN} \right) vs. \ log(\Delta K), \ Linear \ Region

Ambient \ Temperature \ Set2:

CGR Data at Ambient Temperature
$m = 4.17$

Alloy 718
Ambient Temperature

$log \left( \frac{da}{dN} \right)$ vs. $log(\Delta K)$, Linear Region

100°C Set 1:

CGR Data at 100°C
\[ \log\left( \frac{da}{dN} \right) \text{ vs. } \log(\Delta K), \text{ Linear Region} \]

100°C Set 2:

CGR Data at 100°C
log\left(\frac{da}{dN}\right) \text{ vs. } \log(\Delta K), \text{ Linear Region}

150°C Set 1:

CGR Data at 150°C
log \left( \frac{da}{dN} \right) vs. \log(\Delta K), Linear Region

150^\circ C Set 2:

CGR Data at 150^\circ C
$\log\left(\frac{da}{dN}\right)$ vs. $\log(\Delta K)$, Linear Region

200°C Set 1:

CGR Data at 200°C
$\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region

200°C Set 2:

CGR Data at 200°C
\[
\log \left( \frac{da}{dN} \right) \text{ vs. } \log(\Delta K), \text{ Linear Region}
\]
APPENDIX C

SSR TEST DATA

Stress-Strain Diagrams using smooth specimens tested at a strain rate of $3.3 \times 10^6 \text{ sec}^{-1}$.

(A conversion factor of 1 ksi = 6.895 MPa can be used wherever applicable)

C1.1 Stress-Strain Diagrams in Room Temperature-Air

![Stress-Strain Diagram](image)

Sample 1
C1.2 Stress-Strain Diagrams in Room Temperature-Acidic Solution

Sample 1

Sample 2
C1.3 Stress-Strain Diagrams in 90°C-Acidic Solution
APPENDIX D

CPP TEST DATA

D1.1 CPP Diagram in 30°C Acidic Solution
D1.2 CPP Diagram in 60°C Acidic Solution

D1.3 CPP Diagram in 90°C Acidic Solution
APPENDIX E

FRACTOGRAPHY

E1.1 Fractography of CVN Specimens

SEM Micrograph of CVN Specimen Tested at -40°C
SEM Micrograph of CVN Specimen Tested at 330°C
E1.2 Fractography of DCB Specimens

SEM Montage of DCB Specimen Showing Three Distinct Regions
Montage of DCB Specimen (High Stress Intensity Factor, 30 Days Exposure)

DCB Specimen (High Stress Intensity Factor, 30 Days Exposure)
Montage of DCB Specimen (High Stress Intensity Factor, 45 Days Exposure)

DCB Specimen (High Stress Intensity Factor, 45 Days Exposure)
Montage of DCB Specimen (Low Stress Intensity Factor, 30 Days Exposure)

DCB Specimen (Low Stress Intensity Factor, 30 Days Exposure)
Montage of DCB Specimen (Low Stress Intensity Factor, 45 Days Exposure)

DCB Specimen (Low Stress Intensity Factor, 45 Days Exposure)
APPENDIX F

TRANSMISSION ELECTRON MICROGRAPHS

F1 TEM Micrographs of Specimens Tested at Room Temperature

120000X 120000X

F2 TEM Micrographs of Specimen Tested at 100°C

63000X 63000X
F3 TEM Micrographs of Specimen Tested at 200°C

F4 TEM Micrographs of Specimen Tested at 300°C
F5 TEM Micrographs of Specimen Tested at 700°C

63000X (γ" Precipitates)  Selected Area Diffraction (SAD) Pattern

97000X (Orowan Loops)
APPENDIX G

X-RAY DIFFRACTION DATA

G1 XRD Spectra of Specimen Tested at Ambient Temperature
G2 XRD Spectra of Specimen Tested at 700°C

G3 XRD Spectra of Specimen Tested at 800°C
APPENDIX H

UNCERTAINTY ANALYSES OF EXPERIMENTAL RESULTS

The parameters like length, time etc. are directly measured in any experiment. The instruments used to determine these measurements may vary in quality every time they are being used. Also, the parameters that are out of control of the researcher might cause undesired variations in these measurements. Efforts were taken to repeat all the experiments in order to determine an average value. However, the undesired variations might have caused variations in the derived results. These variations if caused by the machines used in the experiments can be reduced by calibrating them on a timely basis. However the variations caused due to human errors and environmental factors cannot be avoided. Uncertainty analysis of these derived results determines the variations/ errors in them.

A method for 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. 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. The use of a meticulous calibration instrument / procedure ensures a lower uncertainty in such measured parameters.
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 thesis 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.

$$W_r = \left[ \left( \frac{\partial R}{\partial x_1} w_1 \right)^2 + \left( \frac{\partial R}{\partial x_2} w_2 \right)^2 + \cdots + \left( \frac{\partial R}{\partial x_n} w_n \right)^2 \right]^{\frac{1}{2}} \quad \text{Equation H1}$$

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

## H.1 Uncertainty Calculation in Instron Results

The results generated from the Instron testing equipment are stress ($\sigma$), percentage elongation ($\%E_l$), 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 $\%E_l$ 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 Instron unit. The values for $\Delta l$, $A_i$, and $A_f$ were calculated based on measurements by a caliper. The uncertainties in load-cell and caliper were $\pm 0.03\%$ lbs.
and ± 0.001 inch, 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.

\[
\sigma = \frac{P}{A_i} \quad \text{Equation H2}
\]

H.1.1 Calculation of Uncertainty in Stress (\(U_\sigma\))

\[
U_\sigma = U_{(P, A_i)}
\]

\[
U_{A_i} = (U_{D_i})^2
\]

Uncertainty in load-cell = ± 0.03\% lb
Uncertainty in caliper = ± 0.001 inch.

Sample calculation:

For yield stress (YS) = 57.66 ksi
The measured load (P) = 2808.82 lbf
Uncertainty in load (\(U_P\)) = 2808.82 * 0.0003
= ± 0.84264

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

Initial Diameter (\(D_i\)) = 0.2500 inch.
Uncertainty in diameter (\(U_{D_i}\)) = ± 0.001 inch.

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

= 0.049087 inch\(^2\)

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

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Uncertainty in area, $U_a = \left( \frac{dA_i}{dD_i} \cdot Ud_i \right)^2 \frac{1}{2}$

= 0.392 * 0.001

= ± 0.000392

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

Equation H3

$\sigma = \frac{P}{A_i}$

$\frac{\partial \sigma}{\partial P} = \frac{1}{A_i}$

= 20.371

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

= -1165713.164

Now providing all the numerical values in Equation A3 obtained from the calculation, it is found that,

$U_\sigma = \left[ (20.371 \times 0.84264)^2 + (-1165713.164 \times 0.000392)^2 \right]^{\frac{1}{2}}$

= 457.28 psi = ± 0.46 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 dissertation.
H.1.2 Calculation of Uncertainty in Percentage Elongation (U%EI)

Sample calculation:

Change in length (Δl) = 0.6513 inch.

Gage length (l) = 1 inch.

\[
\% EI = \frac{\Delta l}{l} \times 100
\]

Uncertainty in Δl (U_Δl) = ± 0.001

Uncertainty in %EI (U%EI),

\[
U%EI = \left[ \left( \frac{d\%EI}{d\Delta l} \cdot U_{\Delta l} \right)^2 \right]^{\frac{1}{2}}
\]

Equation H4

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

= 100

Providing all the calculated values in Equation A4, it is found that,

\[
U%EI = \left[ (100 \times 0.001)^2 \right]^{\frac{1}{2}}
\]

U%EI = ± 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 dissertation.

H.1.3 Calculation of Uncertainty in Percentage Reduction in Area (U%RA)

Sample calculation:

%RA = 59.55%

Uncertainty in initial cross-sectional area (U_Ai) for the smooth specimen:

Initial Diameter (D_i) = 0.2500 inch.

Uncertainty in initial diameter,
(U_{D_i}) = \pm 0.001 \text{ in}

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

= 0.049087 \text{ inch}^2

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

= 0.392

Uncertainty in initial cross-sectional area,

U_{A_i} = \left[ \left( \frac{dA_i}{dD_i} \cdot UD_i \right)^2 \right]^{1/2}

= 0.392 \times 0.001

= \pm 0.000392

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

Final Diameter \quad (D_f) = 0.1590 \text{ inch.}

Uncertainty in final diameter (U_{D_f}),

= \pm 0.001 \text{ inch.}

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

= 0.019855 \text{ inch}^2

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

= 0.2497
Uncertainty in final cross-sectional area,

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

\[ = 0.2497 \times 0.001 \]

\[ = 0.0002497 \]

Uncertainty in %RA,

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

\[ = \frac{824.01}{A_i} \times 100 \]

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

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

\[ = 824.01 \]

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

\[ = -2037.19 \]

Now assigning all the calculated values in Equation A5, it is found that,

\[ U_{\%RA} = \left[ (824.01 \times 0.000392)^2 + (-2037.19 \times 0.0002497)^2 \right]^{\frac{1}{3}} \]

\[ = 0.602 \]

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 dissertation.
H.2 Uncertainty Calculation for Parameters Derived from SSR Results

H.2.1 Calculation of Uncertainty in Time to failure ($U_{TTF}$)

Field Point Software of the SSR unit is used to obtain the TTF, which is accurate up to $1/100^{th}$ of a second in finding the TTF. Therefore, the uncertainty of the TTF in the SSR testing is negligible.

H.3 Uncertainty Calculations for Parameters derived from CPP Results

The uncertainty of the potentiostat provided by the manufacturer is ± 0.003 mV.

**Sample calculation:**

For corrosion potential ($E_{corr}$) = 284 mV

The uncertainty in $E_{corr} = 284 \times (±0.003) = ± 0.852$ mV

For pitting potential ($E_{pit}$) = 650 mV

The uncertainty in $E_{pit} = 650 \times (±0.003) = ± 1.95$ 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 dissertation.
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