Tensile deformation, toughness and crack propagation studies of alloy 617

Vikram Marthandam
University of Nevada, Las Vegas
TENSILE DEFORMATION, TOUGHNESS AND CRACK PROPAGATION
STUDIES OF ALLOY 617

by

Vikram Marthandam

Bachelor of Engineering in Mechanical Engineering
University of Madras, India

Master of Science in Mechanical Engineering, 2004
University of Nevada, Las Vegas, Las Vegas, NV 89154

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Cipin K. Roy
Examination Committee Chair

Dean of the Graduate College

Examination Committee Member
Examination Committee Member
Graduate College Faculty Representative
ABSTRACT

Tensile Deformation, Toughness and Crack Propagation Studies of Alloy 617

By

Vikram Marthandam

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

Austenitic nickel-base Alloy 617 has been tested under tensile loading at temperatures relevant to both the Nuclear Hydrogen Initiative and the Next Generation Nuclear Plant programs of the U.S. Department of Energy. Two important metallurgical phenomena, namely dynamic strain aging (DSA) and yield strength anomaly (YSA), have been identified based on the resultant data. DSA was characterized by reduced plastic strain and serrations in the engineering stress versus engineering strain diagrams. YSA was manifested by enhanced yield strength at relatively higher temperatures. A mechanistic understanding of DSA and YSA phenomena has been developed based on the estimation of dislocation density, activation energy, work-hardening index and precipitate morphology, where applicable. Stress-intensity-factor and crack-growth-rates of this alloy have been evaluated with and without the presence of an acidic solution at different temperatures using fracture-mechanics-based principles. Fractographic evaluations of all tested specimens have also been performed to characterize the extent and morphology of failures resulting from different modes of loading.
# TABLE OF CONTENTS

ABSTRACT ................................................................................................................................. iii 

LIST OF TABLES ......................................................................................................................... vi 

LIST OF FIGURES ......................................................................................................................... vii 

ACKNOWLEDGEMENTS ............................................................................................................... x 

CHAPTER 1 INTRODUCTION ......................................................................................................... 1 

CHAPTER 2 TEST MATERIAL AND SPECIMENS AND ENVIRONMENT ................................. 9  
  2.1 Test Material ......................................................................................................................... 9 
  2.2 Test Specimens ................................................................................................................... 11  
     2.2.1 Tensile Specimen ........................................................................................................ 12  
     2.2.2 Compact-Tension Specimens .................................................................................. 13  
     2.2.3 DCB Specimen .......................................................................................................... 16  
  2.3 Test Environment ................................................................................................................. 19 

CHAPTER 3 EXPERIMENTAL PROCEDURES ........................................................................... 21  
  3.1 Tensile Testing .................................................................................................................... 22  
  3.2 Parameters Related to Tensile Deformation ..................................................................... 25  
     3.2.1 Dislocation Density Calculation ............................................................................. 26  
     3.2.2 Activation Energy Calculation ............................................................................... 28  
     3.2.3 Computation of Strain Hardening Exponent ......................................................... 31  
  3.3 Fracture Toughness Measurement ...................................................................................... 33  
  3.4 Crack Propagation Study .................................................................................................... 35  
  3.5 SCC Testing using DCB Specimens .................................................................................. 37  
  3.6 Metallographic Evaluations ............................................................................................... 39  
  3.7 Fractographic Evaluations ................................................................................................. 40  
  3.8 Transmission Electron Microscopic Evaluations ............................................................ 41  
     3.8.1 TEM Sample Preparation ...................................................................................... 42  
  3.9 Phase Characterization ....................................................................................................... 44 

CHAPTER 4 RESULTS .................................................................................................................. 46  
  4.1 Microstructural Evaluations ............................................................................................... 46  
  4.2 Tensile Properties Evaluation ............................................................................................ 48  
     4.2.1 Dislocation Density Calculation ............................................................................. 54  
     4.2.2 Activation Energy Calculation ............................................................................... 58  
     4.2.3 Estimation of Work-Hardening Index .................................................................... 64
# LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 2.1</td>
<td>Physical Properties of Alloy 617</td>
<td>10</td>
</tr>
<tr>
<td>Table 2.2</td>
<td>Chemical Composition of Alloy 617 (wt%)</td>
<td>11</td>
</tr>
<tr>
<td>Table 2.3</td>
<td>Wedge Thickness and Arm-Displacement</td>
<td>19</td>
</tr>
<tr>
<td>Table 2.4</td>
<td>Chemical Composition of the Test Solution</td>
<td>20</td>
</tr>
<tr>
<td>Table 3.1</td>
<td>Specifications of Instron Machine</td>
<td>24</td>
</tr>
<tr>
<td>Table 4.1</td>
<td>Average Tensile Properties versus Temperature</td>
<td>50</td>
</tr>
<tr>
<td>Table 4.2</td>
<td>ρ versus Temperature</td>
<td>57</td>
</tr>
<tr>
<td>Table 4.3</td>
<td>(m + β) and Q Values (Method One)</td>
<td>62</td>
</tr>
<tr>
<td>Table 4.4</td>
<td>Q Value (Method Two)</td>
<td>63</td>
</tr>
<tr>
<td>Table 4.5</td>
<td>n vs. Temperature</td>
<td>65</td>
</tr>
<tr>
<td>Table 4.6</td>
<td>n vs. Temperatures and Strain Rate</td>
<td>65</td>
</tr>
<tr>
<td>Table 4.7</td>
<td>Compositional Variation at Precipitate vs. Matrix</td>
<td>70</td>
</tr>
<tr>
<td>Table 4.8</td>
<td>Comparison of Calculated d-spacing to ICDD Standard</td>
<td>73</td>
</tr>
<tr>
<td>Table 4.9</td>
<td>Results of K_{IC} Measurements</td>
<td>75</td>
</tr>
<tr>
<td>Table 4.10</td>
<td>Results of CGR Testing</td>
<td>82</td>
</tr>
<tr>
<td>Table 4.11</td>
<td>ΔK_{(final)} vs. ( \frac{da}{dN} ) at Different Temperatures</td>
<td>84</td>
</tr>
<tr>
<td>Table 4.12</td>
<td>Results of DCB Testing</td>
<td>85</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Figure 1.1 Schematic View of the S-I Process ................................................................. 3
Figure 1.2 Reactions Involved in the S-I Process .......................................................... 3
Figure 2.1 Tensile Specimen ......................................................................................... 13
(a) Specimen Dimensions ......................................................................................... 13
(b) Pictorial View ....................................................................................................... 13
Figure 2.2 CT Specimen used in $K_{IC}$ Measurements .............................................. 14
(a) Specimen Dimensions ......................................................................................... 14
(b) Pictorial View ....................................................................................................... 14
Figure 2.3 CT Specimen used in CGR Evaluation ....................................................... 15
(a) Specimen Dimensions ......................................................................................... 15
(b) Pictorial View ....................................................................................................... 16
Figure 2.4 Wedge-loaded DCB Specimen ..................................................................... 17
(a) Configuration of DCB Specimen ......................................................................... 17
(b) Pictorial View of DCB Specimen .......................................................................... 18
Figure 2.5 Double Taper Wedge .................................................................................. 18
(a) Configuration of Double Taper Wedge ................................................................ 18
(b) Pictorial View of Double Taper Wedge ............................................................... 18
Figure 3.1 Instron Testing Machine .............................................................................. 24
Figure 3.2 Line Intersection Method to Calculate $\rho$ .................................................. 27
Figure 3.3 Hollomon vs. Ludwigson Relationship ....................................................... 32
Figure 3.4 Three Types of Load-Displacement Curves ................................................. 34
Figure 3.5 Autoclave Test System ............................................................................... 39
Figure 3.6 Leica Optical Microscope .......................................................................... 40
Figure 3.7 Jeol-5600 Scanning Electron Microscope ................................................ 41
Figure 3.8 Tecnai G$^2$ S-TWIN TEM ......................................................................... 42
Figure 3.9 Grinding Accessories .................................................................................. 44
Figure 3.10 Disc Puncher ............................................................................................. 44
Figure 3.11 TenuPol-5 Electropolisher ......................................................................... 44
Figure 3.12 Panalytical X'PERT Pro XRD Spectrometer ........................................... 45
Figure 4.1 Optical Micrographs, Kalling's Reagent ...................................................... 47
(a) Heat # HV0860A ................................................................................................. 47
(b) Heat # HV1160 .................................................................................................... 47
Figure 4.2 s-e Diagrams vs. Temperature ................................................................. 49
Figure 4.3 YS vs. Temperature .................................................................................... 52
Figure 4.4 UTS vs. Temperature ................................................................................ 52
Figure 4.5 %E1 vs. Temperature ................................................................................ 53
Figure 4.6 %RA vs. Temperature ................................................................................ 53
Figure 4.7 TEM Micrograph of Alloy 617 Tested at Ambient Temperature ............... 55
Figure 4.8 TEM Micrograph of Alloy 617 Tested at 100°C ............................................55
Figure 4.9 TEM Micrograph of Alloy 617 Tested at 200°C ............................................56
Figure 4.10 TEM Micrograph of Alloy 617 Tested at 300°C ............................................56
Figure 4.11 $\rho$ vs. Temperature ...................................................................................................57
Figure 4.12 s-e Diagram vs. Temperature ...................................................................59
Figure 4.13 s-e Diagram vs. Temperature ...................................................................59
Figure 4.14 $\epsilon_c$ vs. Temperature .........................................................................................60

Figure 4.15 $\ln(\epsilon_c)$ vs. $\frac{1}{T}$ ................................................................................................61

Figure 4.16 $\ln \epsilon$ vs. $\ln \epsilon_c$ ......................................................................................................61

Figure 4.17 $\ln \left[ \frac{\epsilon_c^{(m+\beta)}}{T} \right]$ vs. $\frac{1}{T}$ ..............................................................................63

Figure 4.18 TEM Micrograph of Specimen Tested at 900°C............................................67
Figure 4.19 TEM Micrograph Showing Subgrains ............................................................68
Figure 4.20 TEM Micrograph Showing Precipitates ............................................................68
Figure 4.21 Spectra for Precipitate and Matrix ......................................................................70
Figure 4.22 Line Scan Elemental Analysis ............................................................................71
(a) STEM Image ..................................................................................................................71
(b) Line Scan Spectra ..........................................................................................................71

Figure 4.23 Intensity vs. $2\theta$ at 800°C ................................................................................72
Figure 4.24 Intensity vs. $2\theta$ at 900°C ................................................................................73
Figure 4.25 P- v Curve at Ambient Temperature ......................................................................74
Figure 4.26 P- v Curve at 100°C ...........................................................................................75

Figure 4.27 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$ ...................................................................................77

Figure 4.28 CGR Data at Ambient Temperature .................................................................78
Figure 4.29 CGR Data at 100°C .............................................................................................78
Figure 4.30 CGR Data at 150°C .............................................................................................79
Figure 4.31 CGR Data at 200°C .............................................................................................79

Figure 4.32 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region .....................................................................80

Figure 4.33 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region .....................................................................80

Figure 4.34 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region .....................................................................81

Figure 4.35 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region .....................................................................81

Figure 4.36 N vs. Temperature ...............................................................................................83
Figure 4.37 SEM Micrographs of Specimens Tested at $10^3$ sec$^{-1}$ .....................................86
(a) Ambient Temperature ..................................................................................................86
(b) 100°C ..................................................................................................................................87
(c) 200°C ..................................................................................................................................87
(d) 300°C .................................................................................................................. 88
(e) 400°C .................................................................................................................. 88
(f) 500°C .................................................................................................................. 89
(g) 600°C .................................................................................................................. 89
(h) 700°C .................................................................................................................. 90
(i) 800°C .................................................................................................................. 90
(j) 900°C .................................................................................................................. 91
(k) 1000°C ............................................................................................................... 91

Figure 4.38 SEM Micrographs of Specimens Tested at 10^{-2} sec^{-1} .................. 86
(a) 100°C ............................................................................................................... 92
(b) 500°C ............................................................................................................... 93
(c) 600°C ............................................................................................................... 93

Figure 4.39 SEM Micrographs of Specimens Tested at 10^{-4} sec^{-1} .............. 94
(a) 100°C ............................................................................................................... 94
(b) 500°C ............................................................................................................... 94
(c) 600°C ............................................................................................................... 95

Figure 4.40 SEM Micrograph of CT Specimen Used in $K_Q$ Evaluation at Ambient Temperature .......................................................... 96
(a) Fatigue Failure (Striations) ........................................................................... 96
(b) Fast Fracture (Dimples) ................................................................................. 96

Figure 4.41 SEM Micrograph of CT Specimen Used in $K_Q$ Evaluation at 100°C ........ 97
(a) Fatigue Failure (Striations) ........................................................................... 97
(b) Fast Fracture (Dimples) ................................................................................. 97

Figure 4.42 SEM Micrograph of CT Specimen Used in CGR Testing at Ambient Temperature .......................................................... 98
(a) Fatigue Failure (Striations) ........................................................................... 98
(b) Fast Fracture (Dimples) ................................................................................. 98

Figure 4.43 SEM Micrograph of CT Specimen Used in CGR Testing at 100°C .... 99
(a) Fatigue Failure (Striations) ........................................................................... 99
(b) Fast Fracture (Dimples) ................................................................................. 99

Figure 4.44 SEM Micrograph of CT Specimen Used in CGR Testing at 150°C .... 100
Figure 4.45 SEM Micrograph of CT Specimen Used in CGR Testing at 250°C .... 101
Figure 4.46 SEM Micrographs of a DCB Specimen Showing Three Types of Failure 102
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CHAPTER 1

INTRODUCTION

The consumption of fossil fuels, oil and gas in particular, has increased extensively over the years. Much to the displeasure of the consumers, there has also been a steady increase in the cost of these types of fuels. Their extensive use has contributed to undesirable levels of pollution leading to an increased amount of carbon dioxide emission to the atmosphere, thus causing the greenhouse effect. It has now been established that the increased concentration of greenhouse gases is the sole cause of global warming. The United States Department of Energy (USDOE) has been deeply concerned with the escalating costs and detrimental environmental effects related to the use of fossil fuels.\footnote{1} Therefore, USDOE has been exploring the possibility of developing alternate cost-effective and environment-friendly energy sources.

Hydrogen appears to be one such energy that could circumvent the problems associated with the usage of conventional fossil fuels. Numerous methods of hydrogen generation are currently being investigated. However, all of them have their own limitations. For example, solar hydrogen generation is limited to the availability of sufficient sunlight. Hydrogen is also known to be produced by the dissociation of water using conventional electrolytic processes. However, hydrogen generation by electrolysis
does not seem to be a viable option from the cost point of view.\cite{2} In view of these limitations, the USDOE has been considering hydrogen generation using nuclear heat that appears to be more viable.\cite{3} This approach enables the utilization of heat from the nations’ existing nuclear reactors to activate chemical reactions leading to the generation of hydrogen (H\textsubscript{2}) and oxygen (O\textsubscript{2}). Thermochemical processes based on this concept that include sulfur-iodine (S-I)\cite{4,5} and calcium-bromine (Ca-Br)\cite{6} cycles have been proposed in recent years. However, the S-I process is preferred to the Ca-Br cycle due to its greater efficiency in hydrogen generation.

The heat derived from a nuclear reactor will be transmitted through an intermediate heat-exchanger into the hydrogen generation plant, where thermochemical reactions will occur at different temperatures, as illustrated in Figure 1.1.\cite{7} The net result associated with the S-I process is the decomposition of water into H\textsubscript{2} and O\textsubscript{2} through a series of chemical reactions as shown in Figure 1.2. The first step associated with this process is the formation of sulfuric acid (H\textsubscript{2}SO\textsubscript{4}) and hydrogen iodide (HI) through reactions involving iodine (I\textsubscript{2}), sulfur dioxide (SO\textsubscript{2}) and water (H\textsubscript{2}O) at an approximate temperature of 120°C. Subsequently, H\textsubscript{2}SO\textsubscript{4} and HI will be allowed to undergo decomposition at temperatures up to 950 and 400°C, respectively. H\textsubscript{2} and O\textsubscript{2} generated from thermochemical reactions will subsequently be separated, and transferred to different storage tanks. I\textsubscript{2} and SO\textsubscript{2}, which are the resultant by-products, will be recycled to react with H\textsubscript{2}O, regenerating H\textsubscript{2}SO\textsubscript{4} and HI. In essence, I\textsubscript{2} and SO\textsubscript{2}, will thus, act as catalysts during chemical reactions. The overall reactions are shown below as reactions 1.1, 1.2 and 1.3, respectively.\cite{8,9}

\[
\text{I}_2 + \text{SO}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{HI} + \text{H}_2\text{SO}_4 \quad \text{(Temperature \sim 120°C)}
\]

Reaction 1.1
\[ \text{H}_2\text{SO}_4 \rightarrow \text{H}_2\text{O} + \text{SO}_2 + \frac{1}{2} \text{O}_2 \text{ (Temperature } \sim 950^\circ\text{C)} \]

\[ 2\text{HI} \rightarrow \text{H}_2 + \text{I}_2 \text{ (Temperature } \sim 400^\circ\text{C)} \]

Figure 1.1 Schematic View of the S-I Process

Figure 1.2 Reactions Involved in the S-I Process
It is obvious from the preceding discussion that the integrity of structural materials to withstand degradations resulting from the combined effect of aggressive chemical species and elevated temperatures will play an important role in successful implementation of the S-I process to generate hydrogen. Therefore, an extensive investigation was undertaken at the University of Nevada, Las Vegas (UNLV) to identify and characterize suitable structural materials having the desired resistance to both metallurgical and environmental degradations under conditions relevant to the S-I process.

More recently, the concept of the Next Generation Nuclear Plant (NGNP) has also been introduced within the charter of USDOE to foster more efficient utilization of nuclear heat to generate electricity in the twenty-first century. NGNP is aimed at using very high temperature gas-cooled reactor (VHTR) with an operational goal of producing helium (He) at temperatures up to 950°C and pressures up to 7 MPa for a design life of 60 years. Approximately 90% of the heat generated from the VHTR will be used to generate electricity while the remaining 10% will be employed to generate hydrogen. In essence, the nuclear reactors, envisioned within the NGNP program, will require high service temperatures and long design lives.\textsuperscript{10} Needless to say that a heat-exchanger will be needed to transmit heat to generate both electricity and hydrogen. Therefore, the structural materials to be used in heat-exchangers for applications in both NGNP and S-I process will be subjected to elevated temperatures approaching 1000°C.

At the onset of the materials research program related to nuclear hydrogen initiative (NHI) using the S-I process, several nickel (Ni)-base superalloys were identified by researchers at UNLV as candidate structural materials based on an extensive literature search.\textsuperscript{11-13} These efforts led to the identification of nickel (Ni)-base Alloy 617 having
excellent tensile properties at elevated temperatures, and superior corrosion resistance in
the presence of many hostile chemical species.\textsuperscript{[14-18]} Later, Alloy 617 was also identified
as a suitable heat-exchanger material for application in the NGNP program.\textsuperscript{[10, 19-21]}
Therefore, extensive metallurgical characterization including the evaluation of tensile
properties, fracture toughness and crack propagation of Alloy 617 at elevated
temperatures was deemed appropriate for both programs.

Ni-base Alloy 617 was developed during 1970’s as an advanced sheet material for
aerospace application. A combination of high strength and oxidation resistance at
temperatures up to 980°C makes Alloy 617 a suitable material for ducting, combustion
cans, and transition liners in both aircraft and land-based gas turbines. Alloy 617 has been
used in catalyst-grid support for production of nitric acid, heat-treating baskets and
reduction boats in the refining of molybdenum due to its high temperature corrosion
resistance.\textsuperscript{[14, 22]} The literature data \textsuperscript{[23]} indicate that this alloy has been used in the
fabrication of thermal energy storage capsules to contain eutectic fluoride mixtures of
sodium, magnesium, lithium and potassium at temperatures of 727°C. Alloy 617 has also
been used in the manufacturing of retort furnace for the tritium extraction facility \textsuperscript{[24]}, and
high temperature gas cooled reactors.\textsuperscript{[25,26]}

This material possesses exemplary resistance to creep deformation and ruptures at
temperatures up to 870°C. Further, it can maintain excellent metallurgical stability even
after its long-term exposure at elevated temperatures. Relatively lower coefficient of
thermal expansion of this alloy, compared to that of most austenitic stainless steels,
enables Alloy 617 to be used in conjunction with ferritic steels. Also, its low density
provides a high strength-to-weight ratio.\textsuperscript{[27]} Researchers \textsuperscript{[28]} have identified Alloy 617 as a
promising current-conducting material in solid oxide fuel cells since it can comply with the thermodynamic considerations required for such application. Alloy 617 has also been considered by NASA as a candidate material for heat-shields in space transportation systems. This material can be strengthened by the precipitation of metal carbonitrides M(C,N) and homogeneously distributed M_{23}C_{6} carbides resulting from a solution annealing treatment.

As discussed earlier, the structural materials to be used in both NHI and NGNP applications must possess superior resistance to metallurgical degradations under the influence of tensile and cyclic loading at elevated temperatures. Therefore, the determination of metallurgical properties such as the tensile strength and ductility, fracture toughness in the presence of a flaw, and crack propagation under loaded conditions was considered necessary for Alloy 617 to qualify as a candidate material for application in both programs. Simultaneously, moderate resistance to corrosive degradations in the presence of hostile chemical species at elevated temperatures is also a pre-requisite for this alloy to be identified as a suitable structural material for application in the S-I process. In view of these requirements, substantial efforts have been made in this investigation to evaluate numerous metallurgical properties of Alloy 617 including the tensile strength and ductility, fracture toughness, and crack propagation rate under conditions relevant to the actual applications. In addition, the susceptibility of this alloy to environment-assisted-cracking (EAC) in the presence of H_{2}SO_{4} has been investigated.

The tensile properties of Alloy 617 have been evaluated at temperatures up to 1000°C. A temperature of 1000°C was selected to comply with the American Society of Mechanical Engineers (ASME) pressure vessels code that requires an evaluation
temperature of 50°C above the desired operating temperature for a specific application. The tensile properties determined in this investigation include the yield strength (YS), the ultimate tensile strength (UTS) and ductility parameters such as the percent elongation (%El) and the percent reduction in area (%RA) as a function of the testing temperature. Previous studies \cite{31, 32} performed on other Ni-base alloys at UNLV did exhibit a phenomenon known as dynamic strain aging (DSA). Consistent with the prior observations, Alloy 617 also showed a tendency to DSA within a specified temperature range. Parameters such as activation energy (Q) for the formation of serrations and dislocation mobility, work hardening index (n), and dislocation density (ρ) related to the DSA phenomenon have been extensively studied in this investigation. Simultaneously, a concept known as yield strength anomaly (YSA) has also been observed during tensile testing of this alloy at very high temperatures. While the concept of DSA has been well-documented in many recent investigations \cite{33-39} performed at UNLV, a novel approach \cite{40} of characterizing YSA has also been presented later in this dissertation.

It has been well known \cite{41} that the presence of flaws in metals and alloys can impair their performance, leading to their premature failures resulting from the combined effect of stress (applied/residual) and defects. One way of characterizing a material's susceptibility to premature failure is to determine its fracture toughness (K_{IC}) using state-of-the-art fracture mechanics-based concepts. Simultaneously, the mode of loading can influence the structural stability of an alloy by demonstrating its resistance to crack propagation as functions of different metallurgical and environmental variables. In view of these considerations, Alloy 617 has also been studied to determine its K_{IC}, and crack propagation rate ($\frac{da}{dN}$) under a cyclic loading condition. An in-situ crack monitoring
device, known as the direct-current-potential-drop (DCPD), has been employed in this investigation to determine the $\frac{da}{dN}$ values as a function of stress intensity range $\Delta K = K_{\text{max}} - K_{\text{min}}$, where, $K_{\text{max}}$ and $K_{\text{min}}$ are the maximum and minimum stress intensity factors, respectively.

While numerous corrosion testing techniques prevail in the open literature with respect to the determination of EAC of structural materials, a sophisticated method based on the utilization of double-cantilever-beam (DCB) specimens has been adopted in this investigation to uniquely quantify the extent of environmental degradation as functions of metallurgical and experimental variables. The utilization of microscopic techniques to characterize degradations is very common with all metallurgical investigations. Therefore, in-depth characterization of metallurgical microstructures, and fractographic evaluations of the tested specimens have also been performed in this study. State-of-the-art techniques including optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) have, therefore, been employed to achieve these goals. Additionally, x-ray diffractometry (XRD) has been used to identify and characterize phase changes, if any, due to metallurgical transformation at elevated temperatures.
CHAPTER 2

TEST MATERIAL, SPECIMENS AND ENVIRONMENT

2.1 Test Material

As indicated in the previous chapter, Ni-base Alloy 617 has been identified to be a candidate structural material, which may satisfy the performance requirements for both NHI and NGNP programs. Alloy 617 is an austenitic precipitation-hardened and face-centered-cubic (FCC) nickel-chromium-cobalt-molybdenum (Ni-Cr-Co-Mo) alloy having a combination of excellent tensile strength at elevated temperatures and superior corrosion resistance in many hostile environments. The presence of high Ni content in this alloy enables significant plastic deformation in multiple slip planes and, can, thus, provide enhanced ductility under the influence of tensile loading. The high Ni and Cr contents make this alloy resistant to degradations while exposed to both oxidizing and reducing environments. A superior oxidation resistance of this alloy may be attributed to the presence of both Cr and aluminum (Al). Additionally, Co and Mo can induce significant strengthening resulting from solid-solution treatment. This alloy is easily weldable and can be readily cold-formed using conventional forming operations. However, sufficiently high forces are needed to cause plastic deformation due to its relatively high tensile strength even at elevated temperatures. The physical properties of this alloy are given in Table 2.1.
Table 2.1 Physical Properties of Alloy 617

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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<tbody>
<tr>
<td>Density</td>
<td>0.302 lb/in³</td>
</tr>
<tr>
<td></td>
<td>8.36 Mg/m³</td>
</tr>
<tr>
<td>Melting Temperature Range</td>
<td>2430-2510°F</td>
</tr>
<tr>
<td></td>
<td>1332-1380°C</td>
</tr>
<tr>
<td>Specific Heat at 78°F (26°C)</td>
<td>0.1 Btu/lb-°F</td>
</tr>
<tr>
<td></td>
<td>419 J/kg-°C</td>
</tr>
<tr>
<td>Electrical Resistivity at 78°F</td>
<td>736 ohm-circ mil/ft</td>
</tr>
<tr>
<td>(26°C)</td>
<td>1.22 µΩ-m</td>
</tr>
</tbody>
</table>

Two experimental heats of Alloy 617 were custom-melted at the Huntington Alloys Corporation, West Virginia using a vacuum-induction-melting (VIM) practice. These VIM heats were subsequently processed into rectangular and round bars of different dimensions using forging and hot-rolling. The hot-rolled rectangular bars were subsequently subjected to cold-rolling operation to reduce their thickness. Since both round and rectangular bars had substantial residual stresses resulting from cold and hot-rolling operations, these processed materials were thermally treated to relieve these internal stresses. This thermal treatment consisted of solution-annealing at 2150°F (1175°C) for variable time periods depending on the thickness of the processed bars. Such thermal treatment is known to produce large-sized austenitic grains with annealing twins in Ni-base alloys. The strengthening of Alloy 617 is known to be the result of precipitation of metal carbonitrides $M(C, N)$ and $M_23C_6$ carbides within the matrix of this alloy. The chemical compositions of both experimental heats of Alloy 617 are given in Table 2.2.
Table 2.2 Chemical Composition of Alloy 617 (wt%)

<table>
<thead>
<tr>
<th>Element</th>
<th>Heat No.</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>HV0860A</td>
<td>HV1160</td>
</tr>
<tr>
<td>C</td>
<td>0.010</td>
<td>0.060</td>
<td></td>
</tr>
<tr>
<td>Mn</td>
<td>&lt;0.001</td>
<td>0.121</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>0.05</td>
<td>0.002</td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>0.0013</td>
<td>0.0009</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>0.031</td>
<td>0.044</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>0.014</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>Ni</td>
<td>53.79</td>
<td>54.80</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>21.88</td>
<td>22.10</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>1.09</td>
<td>0.87</td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>0.57</td>
<td>0.29</td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>0.013</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Co</td>
<td>12.93</td>
<td>12.17</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>9.47</td>
<td>9.52</td>
<td></td>
</tr>
<tr>
<td>Nb</td>
<td>&lt;0.01</td>
<td>&lt;0.001</td>
<td></td>
</tr>
<tr>
<td>Ta</td>
<td>0.002</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>&lt;0.0001</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td></td>
</tr>
<tr>
<td>Ca</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td></td>
</tr>
<tr>
<td>N</td>
<td>0.001</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>0.007</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>0.004</td>
<td>0.002</td>
<td></td>
</tr>
<tr>
<td>Zr</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>-</td>
<td>&lt;0.009</td>
<td></td>
</tr>
</tbody>
</table>

2.2 Test Specimens

As discussed in the earlier chapter, Alloy 617 is being considered for both NHI and NGNP applications, requiring excellent metallurgical properties and superior corrosion resistance. Metallurgical properties such as high tensile strength and ductility, plane
strain fracture toughness ($K_{IC}$), and reduced crack propagation rates under different loading conditions are vital for Alloy 617 to be suitable for high temperature applications. Therefore, significant efforts have been made in this investigation to evaluate these properties using conventional and state-of-the-art techniques prescribed by the scientific and engineering communities. Smooth cylindrical specimens have been used for tensile properties evaluation at temperatures ranging from ambient to 1000°C. For $K_{IC}$ measurements, pre-cracked compact-tension (CT) specimens have been used to comply with the conventional fracture mechanics principles. CT specimens have also been used for determination of crack-growth-rate (CGR) at ambient and elevated temperatures. Wedge-loaded double-cantilever-beam (DCB) specimens, based on constant displacement theory of fracture mechanics, have been used to characterize the cracking susceptibility of Alloy 617 exposed to an acidic solution at an elevated temperature for variable time periods. An in-situ crack monitoring device, known as the direct-current-potential-drop (DCPD), has been utilized to determine CGR in CT specimens. The configuration and the dimensions of each type of specimens used in metallurgical and corrosion testing are described next in the following sub-sections.

2.2.1 Tensile Specimen

Smooth cylindrical specimens having an overall length of 101.6 mm (4 inches), 25.4 mm (1 inch) gage length and 6.35 mm (0.25 inch) gage diameter were machined from the round heat-treated bars of Alloy 617. These specimens were machined in such a way that their gage sections were parallel to the longitudinal rolling direction. A gage length ($l$) to gage diameter ($d$) ratio ($l/d$) of 4 was maintained to comply with the ASTM designation E
The specimen configuration with detailed dimensions and its pictorial view are illustrated in Figure 2.1.

Figure 2.1 Tensile Specimen

(a) Specimen Dimensions (All Dimensions are in Inches)

(b) Pictorial View

2.2.2 Compact Tension Specimens

Efforts were made to perform $K_{IC}$ measurements using CT specimens of two different dimensions. The first type of CT specimen had a length of 63.5 mm (2.5 inches), a width of 60.95 mm (2.4 inches) and a thickness of 25.4 mm (1 inch), as shown in Figure 2.2.
These specimens were machined according to the ASTM Designations E 399-1990, E 647-2000 and E 1820-2001. The intersection of the crack starter notch tip with the two specimen surfaces were 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 6.35 mm (0.125 inch) was provided at the tip of the notch for the straight-through slot terminating in the V-notch of the specimen, which facilitated fatigue pre-cracking at low stress intensity levels.

(a) Specimen Dimensions (All Dimensions are in Inches)

(b) Pictorial View

Figure 2.2 CT Specimens used in $K_{IC}$ Measurements
Conventionally, the CT specimens are tested in a pre-cracked condition. However, significant difficulty was encountered in pre-cracking these specimens even under cyclic loading. Such difficulty in pre-cracking of the CT specimens could be attributed to a low loading frequency (1 Hz) available with the current testing equipment. Therefore, CT specimens having a similar configuration but different dimension, shown in Figure 2.3, were used for evaluation of $K_{IC}$. A root radius of 0.25 mm (0.003 inch) was provided from the straight-through slot terminating in the V-notch of the specimen to facilitate fatigue pre-cracking at relatively low stress intensity levels. A width ($W$) to thickness ($B$) ratio of 4 was maintained to comply with the size requirements prescribed by the ASTM designations E 399-1990, E 647-2000 and E 1820-2001. A similar type of CT specimen was also used to determine CGR of Alloy 617 under the influence of cyclic loading at different temperatures.

(a) Specimen Dimensions (All Dimensions are in Inches)
2.2.3 DCB Specimen

The susceptibility of Alloy 617 to stress-corrosion-cracking (SCC) was determined using the fracture mechanics-based, wedge-loaded and pre-cracked DCB specimens. DCB specimens having a length of 101.6 mm (4 inches) width of 25.4 mm (1 inch) and a thickness of 9.525 mm (0.375 inch) with one slotted end for wedge-loading and V-shaped side grooves extended from the slot to the opposite end, were machined from rectangular bars of Alloy 617 according to the NACE Standard TM0177-1990. Side grooves were provided on either side of the DCB specimens to prevent branching of cracks, if any. The side grooves were machined as 20% of the wall thickness, thus, maintaining a web thickness \( B_w \) of 60% of the wall thickness (5.715 mm or 0.225 inch). Caution was exercised during machining to ensure that the crack plane was perpendicular to the short-transverse direction, and that the crack propagation would occur along the longitudinal rolling direction. Machining of the side grooves was done carefully to avoid overheating and cold-working. A total of 0.002 inch of metal was removed during the final two passes of machining. The DCB specimens were used to determine the average crack growth rate, when exposed to an aqueous solution under loading due to the insertion of wedges. The
configuration of the DCB specimen, loading-wedges, and their pictorial views are illustrated in Figures 2.4 and 2.5.

(a) Configuration of DCB Specimen (All Dimensions are in Inches)
Figure 2.4 Wedge-loaded DCB Specimen

Figure 2.5 Double-Taper Wedge

(b) Pictorial View of DCB Specimen

(a) Configuration of Double-Taper Wedge

(b) Pictorial View of Double-Taper Wedge
Wedges of different thickness were inserted into the specimen slots to apply the desired loads resulting from the separation of two arms. The thickness of the wedge was varied from 3.0036 mm (0.1182 inches) to 3.2156 mm (0.1265 inches) to provide arm-displacements ranging from 0.6436 mm (0.0253 inches) to 0.8556 mm (0.0336 inches), as given in Table 2.3.

Table 2.3 Wedge Thickness and Arm-Displacement

<table>
<thead>
<tr>
<th>Wedge Thickness mm (inch)</th>
<th>Arm-Displacement mm (inch)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.00 (0.11)</td>
<td>0.64 (0.02)</td>
</tr>
<tr>
<td>3.02 (0.11)</td>
<td>0.66 (0.02)</td>
</tr>
<tr>
<td>3.20 (0.12)</td>
<td>0.84 (0.03)</td>
</tr>
<tr>
<td>3.22 (0.12)</td>
<td>0.86 (0.03)</td>
</tr>
</tbody>
</table>

2.3 Test Environment

For Alloy 617 to be selected for application in the H₂SO₄ decomposition process under the S-I cycle, it is necessary to evaluate its cracking susceptibility in the presence of an acidic environment. Therefore, the pre-cracked and wedge-loaded DCB specimens were tested in presence of an acidic solution containing H₂SO₄ at 100°C. The testing environment was contained inside an autoclave, which accommodated the pre-cracked and wedge-loaded DCB specimens for evaluation of their K values as a function of duration. The composition of the acidic solution used in SCC testing involving these specimens is given in Table 2.4.
Table 2.4 Chemical Composition of the Test Solution

<table>
<thead>
<tr>
<th>Deionized Water (ml)</th>
<th>$\text{H}_2\text{SO}_4$ (95-98% Concentrated)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6000</td>
<td>Added to achieve a pH of 1.0</td>
</tr>
</tbody>
</table>
CHAPTER 3

EXPERIMENTAL PROCEDURES

As the title of this dissertation implies, this investigation is focused on the evaluation of the tensile properties of Alloy 617 at temperatures relevant to the $\text{H}_2\text{SO}_4$ decomposition process of the S-I cycle for generation of hydrogen using nuclear heat. Since the presence of minute flaws can influence the toughness of a metallic material under loaded condition, plane-strain fracture toughness ($K_{ic}$) of Alloy 617 has been determined at ambient and elevated temperatures using pre-cracked CT specimens. CT specimens of different dimensions were also used to evaluate the crack-growth behavior of this alloy using the DCPD in-situ crack monitoring device at different temperatures.

The structural material to be used in the $\text{H}_2\text{SO}_4$ decomposition process must also have adequate resistance to environment-induced degradation, such as SCC. Therefore, an extensive effort has been made to evaluate the susceptibility of Alloy 617 to SCC using pre-cracked and wedge-loaded DCB specimens immersed in an aqueous solution containing sulfuric acid for two different durations.

The extent and morphology of failure of all tested specimens have been determined using SEM. Further, TEM has been employed to characterize linear defects known as dislocations. Simultaneously, XRD has been utilized to verify phase changes, if any, resulting from metallurgical transformations at elevated temperatures. The use of TEM
and XRD enabled a development of a deformation mechanism as functions of temperature and other metallurgical variables, which will be presented in a later section. Optical microscopy was used to determine the metallurgical microstructures of two different heats of Alloy 617. The different experimental procedures used in this investigation are described in the following subsections.

3.1 Tensile Testing

A screw-driven, electro-mechanical Instron equipment (model 8862) was used to determine the tensile properties of Alloy 617 including the yield strength (YS), the ultimate tensile strength (UTS), the percent elongation (%E1) and the percent reduction in area (%RA) as a function of temperature. Initially, smooth cylindrical specimens were tested at a strain rate of $10^3$ sec$^{-1}$ in accordance with the ASTM Designation E 08-2004. Three specimens were tested at each temperature, and the average values of each parameter were recorded. The experimental data including load, displacement, engineering versus true stresses and strains were automatically recorded in a file. A software program (Bluehill 2) was used for data acquisition and generation of the engineering stress versus strain (s-e) diagrams at different temperatures. This program also automatically provided the magnitudes of YS and UTS. The magnitude of the %E1 based on the use of linear-variable-displacement-transducer also became available through use of this software. However, both ductility parameters (%E1 and %RA) were later determined using Equations 3.1 through 3.4.

\[
%E1 = \left[ \frac{L_f - L_o}{L_o} \right] \quad \text{Equation 3.1}
\]
\[
\% RA = \left[ \frac{A_o - A_f}{A_o} \right]
\]

Equation 3.2

\[
A_o = \frac{\pi \times D_o^2}{4}
\]

Equation 3.3

\[
A_f = \frac{\pi \times D_f^2}{4}
\]

Equation 3.4

where, \( A_o \) = Initial cross sectional area (inch\(^2\))

\( A_f \) = Cross sectional area at failure (inch\(^2\))

\( L_o \) = Initial overall length (inch)

\( L_f \) = Final overall length (inch)

\( L_g \) = Initial gage length (inch)

\( D_o \) = Initial gage diameter (inch)

\( D_f \) = Final gage diameter (inch)

A pictorial view of the tensile testing equipment is illustrated in Figure 3.1. This top-actuated testing system had an axial load capacity of 22.5 kip (100 kN) for both static and dynamic loading at variable speeds. This machine consisted of a large heavy-duty frame with an adjustable crosshead attached to the top grip, and a stationary actuator with another grip located at the bottom to enable loading and unloading of the test specimen. The axial motion was measured by means of force, displacement, or an external signal from the strain gage. The specimen was mounted between the two grips and pulled by the movable actuator. The load cell was used to measure the applied force on the tensile
specimen. The specifications for different components of this equipment are given in Table 3.1.\textsuperscript{[49]}

![Figure 3.1 Instron Testing Machine](image)

**Figure 3.1 Instron Testing 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 x 2</td>
<td>M30 x 2</td>
</tr>
</tbody>
</table>

Table 3.1 Specifications of Instron Machine
Testing was performed at temperatures ranging from ambient-1000°C. A ceramic-lined split furnace (model MDS1735A) was attached to the testing system for loading the smooth tensile specimen at elevated temperatures. Nitrogen was continuously purged through ceramic tubes located in the inner wall of this furnace during elevated temperature testing to prevent oxidation of the specimen surface. Two water-cooled stainless steel jackets surrounded the furnace to provide a safe and ergonomic outer surface for handling the grips during operation. This furnace contained six U-shaped molybdenum heating elements that provided the desired testing temperature. Three B-type thermocouples were used to monitor the testing temperature during tensile loading. An external control panel (model CU666F) was used to set the desired testing temperature, heating rate and dwell time. A heating rate of 8°C per minute was used to prevent any thermal shock of the critical testing components. MarM 246 was used as the specimen grip material. The use of this heat-resistant material ensured proper alignment of the test specimen without causing its distortion at elevated temperatures during tensile loading.

3.2 Parameters Related to Tensile Deformation

Plastic deformation under tensile loading can be influenced by both temperature and strain rate \((\varepsilon)\). Both factors can play important roles in characterizing deformation based on dislocation motion throughout the matrix and grain boundaries of metallic materials. Thus, an estimation of the dislocation density \((\rho)\), activation energy \((Q)\) and work hardening index \((n)\) is essential to develop a basic understanding of deformation.
mechanism under the influence of tensile loading. The following subsections are aimed at presenting different approaches for determination of \( \rho \), \( Q \) and \( n \).

### 3.2.1 Dislocation Density Calculation

The mobility of dislocations, generated during plastic deformation, can be influenced to a large extent by the diffusion of solute or solvent elements into the matrix of a material as functions of temperature and strain rate. The accumulation of such foreign elements around dislocations can impede their motion through the matrix as well as in the vicinity of the grain boundaries, thus, showing reduced plastic strain in the s-e diagrams. Therefore, efforts have been made to determine the magnitude of \( \rho \) of the tested tensile specimens using the TEM micrographs of their gage sections next to the primary fracture surfaces. A line intersection method \(^{[50]}\) was used to calculate the value of \( \rho \). According to this method, a grid containing horizontal and vertical lines was superimposed on the TEM micrographs of the tested specimens that contained dislocations, as shown in Figure 3.2.\(^{[31, 42, 50-53]}\) The magnitude of \( \rho \) was calculated using Equation 3.5. The average thickness (\( t \)) of the TEM sample was measured by electron-energy-loss-spectroscopy (EELS) connected to the Tecnai G² S-TWIN TEM using Equation 3.6.\(^{[54]}\)

\[
\rho = \frac{1}{t} \left[ \frac{\sum n_v}{\sum L_v} + \frac{\sum n_h}{\sum L_h} \right] \quad \text{Equation 3.5}
\]

\[
t = \lambda \ln \left( \frac{I_v}{I_h} \right) \quad \text{Equation 3.6}
\]
where,

\[ \sum n_v = \text{Number of intersections of vertical test lines with dislocations} \]

\[ \sum n_h = \text{Number of intersections of horizontal test lines with dislocations} \]

\[ \sum L_v = \text{Total length of vertical test lines (meters)} \]

\[ \sum L_h = \text{Total length of horizontal test lines (meters)} \]

\[ t = \text{Average thickness of the TEM sample,} \]

\[ \lambda = \text{Mean free path} \]

\[ I_t = \text{Total intensity reaching the spectrometer} \]

\[ I_\alpha = \text{Zero-loss intensity reaching the spectrometer} \]
3.2.2 Activation Energy Calculation

As indicated in the Introduction section, Alloy 617 exhibited reduced failure strain and serrated s-e curves generated at a strain rate of $10^{-3}$ sec$^{-1}$ within a certain temperature regime, which can be attributed to the occurrence of the DSA phenomenon. Since DSA can be influenced by both temperature and strain rate, tensile testing was later performed at two additional strain rates, above and below the initial strain rate ($10^{-2}$ and $10^{-4}$ sec$^{-1}$) at selected temperatures (100, 500 and 600°C). These temperatures represented the susceptible temperature regime and beyond, where serrations were predominant. A minimum of two tests was performed at both strain rates.

Literature $^{33-35}$ suggests that the occurrence of the DSA phenomenon is the result of the diffusion of interstitial and/or substitutional solute elements into the matrix of a susceptible material. Since diffusion is known to be a thermally-activated process, the determination of the activation energy based on the testing temperature and strain rate was performed in this investigation to develop a basic understanding of deformation mechanism. It has been suggested $^{33-35, 55-56}$ that the critical plastic strain ($\varepsilon_c$) needed to initiate the formation of serrations is a function of both the temperature and strain rate ($\dot{\varepsilon}$) according to Equation 3.7 $^{55-56}$, given below.

$$\varepsilon_c^{(m+\beta)} = K \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right)$$

Equation 3.7

where,

$Q$ = Activation energy at the onset of serration formation (KJ/mole)

$R$ = Universal gas Constant (0.83144 KJ/mol Kelvin)

$T$ = Absolute temperature (Kelvin)
m, β = Exponents related to the variation of vacancy concentration (Cv) and mobile dislocation density (ρm)

K = Constant

Researchers [33-34] have suggested that Cv and ρm are directly proportional to the true strain (ε) based on two empirical Equations 3.8 and 3.9, respectively, shown below.

Equation 3.8

\[ C_v \propto \varepsilon^m \]

Equation 3.9

\[ \rho_m \propto \varepsilon^\beta \]

Two methods have been used to calculate the magnitude of Q. One method is based on the use of Equation 3.7 that can be expressed in a modified form by taking natural logarithm on both sides and rearrangement, as shown in Equation 3.10. The slope of the line obtained by plotting \( \ln(\varepsilon_c) \) versus \( \frac{1}{T} \) is given by \( \frac{Q}{R(m+\beta)} \). The magnitude of \( (m+\beta) \) is unknown, which can be evaluated by rearranging Equation 3.7, as shown in Equation 3.11. Knowing the value of \( (m+\beta) \), Q can be determined from the slope \( \left( \frac{Q}{R(m+\beta)} \right) \). The different mathematical modifications mentioned above are given below.

Taking natural logarithm of Equation 3.7,

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

or, \( (m+\beta) \ln \varepsilon_c = \ln K + \ln \varepsilon_e + \frac{Q}{RT} \)
or,
\[
\ln \varepsilon_c = \frac{Q}{R(m + \beta)} \times \frac{1}{T} + \left( \frac{\ln K + \ln \varepsilon}{m + \beta} \right) \tag{Equation 3.10}
\]

\[
\ln (\varepsilon') = (m + \beta) \ln \varepsilon_c - \left[ \ln K + \frac{Q}{RT} \right] \tag{Equation 3.11}
\]

The other method used in Q calculation is based on McCormick’s strain aging model \(^{[33-34]}\), expressed as Equation 3.12.\(^{[57]}\). Rewriting this equation in a logarithmic form and simplifying, one can arrive at Equation 3.13, which represents a straight line.

Plotting \(\ln \left( \frac{\varepsilon_c^{(m+\beta)}}{T} \right)\) versus \(\frac{1}{T}\), Q can be determined from the slope \(\frac{Q}{k}\) at a constant \(\varepsilon\).

\[
\frac{\varepsilon_c^{(m+\beta)}}{T} = \left( \frac{C_1}{\phi C_0} \right)^{3/2} e^{(Q/kT)} \varepsilon \tag{Equation 3.12}
\]

\[
\ln \left( \frac{\varepsilon_c^{(m+\beta)}}{T} \right) = \frac{Q}{k} \left( \frac{1}{T} \right) + \ln \left( \frac{C_1}{\phi C_0} \right)^{3/2} \frac{kb}{LNU_m D_0} \varepsilon \tag{Equation 3.13}
\]

where,

- \(C_0\) = Initial concentration of solute in the alloy
- \(C_1\) = The local concentration of the solute at dislocation
- \(L\) = The obstacle spacing
- \(U_m\) = The maximum solute-dislocation interaction energy
- \(D_0\) = The frequency factor
- \(b\) = The Burger’s vector
k = Boltzman constant \((1.3807 \times 10^{-23} \text{ J/K})\)

\(N\) and \(\Phi\) = Constants

3.2.3 Computation of Strain Hardening Exponent

It is well established that metallic materials become work-hardened due to dynamic loading beyond the elastic limit.\(^{58}\) The extent of work hardening, commonly referred to as the strain hardening exponent \((n)\), is known to be related to both the true stress and true strain\(^{58,59}\) according to Holloman relationship, \(^{60,61}\) given by Equation 3.14.

\[
\sigma = K\varepsilon^n
\]

Equation 3.14

where,

\(\sigma\) = True stress (ksi or MPa)

\(\varepsilon\) = True strain

\(n\) = Strain-hardening exponent

\(K\) = Constant, known as strain hardening (strength) coefficient

Ideally, the value of \(n\) can be determined from the slope of a straight line obtained by plotting \(\log{\sigma}\) versus \(\log{\varepsilon}\) at a constant temperature. However, depending on the material under consideration, a linear relationship between \(\log{\sigma}\) and \(\log{\varepsilon}\) may not always be achieved.\(^{60,62-63}\) A non-linear relationship has previously been reported for several nickel-base alloys.\(^{64-65}\) An alternate relationship has been proposed by Ludwigson\(^{66}\) by adding a new term \((\Delta)\) to the ideal Hollomon equation, as shown in Equation 3.15. In this equation, \(\Delta = \exp(K_1 + n_1\varepsilon)\) where, \(K_1\) and \(n_1\) are the intercept and the slope of a line, respectively generated from the plot of \(\log(\Delta)\) vs. \(\log(\varepsilon)\).

\[
\sigma = K\varepsilon^n + \Delta
\]

Equation 3.15
For such a non-ideal situation, the magnitude of $n$ can be determined from the plot of $\log(\sigma)$ versus $\log(\varepsilon)$ using the best linear part of the resultant non-linear relationship. A superimposition of both linear and non-linear plots of $\log(\sigma)$ versus $\log(\varepsilon)$ is illustrated in Figure 3.3. An evaluation of both plots indicates that the linear portion of Ludwigson's curve coincided with the conventional linear part generated according to the Hollomon equation. It should also be noted that such coincidence of linear portions, based on both approaches, occurs at relatively higher $\varepsilon$ values. Therefore, the magnitudes of $n$ at different temperatures and strain rates were determined using the true strain values corresponding to the linear portion of the superimposed plots. It is obvious that a minimum value of $\Delta$ (approaching zero) was considered in the determination of $n$ using this approach.

Figure 3.3 Hollomon vs. Ludwigson Relationship
3.3 Fracture Toughness Measurement

Efforts have been made to determine the plane-strain fracture toughness (K\textsubscript{IC}) of Alloy 617 at ambient and elevated temperatures using CT specimens in an Instron testing equipment (model 8862) according to the ASTM Designation E 399-1990.\textsuperscript{44} The CT specimens were pre-cracked to a maximum length of 3 mm using a stress ratio \( R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} \) of 0.1 and a frequency of 1 Hz. The load applied during pre-cracking of the CT specimens was below the material’s yielding load. Testing was performed using a fracture toughness (K\textsubscript{IC}) software program provided by the Instron Corporation. Following pre-cracking, a graphite extensometer was attached to the test specimen during straining at a rate of 2 mm per minute to determine a conditional stress intensity factor (K\textsubscript{Q}) value.

Depending on the test material, three different types of load versus displacement (P-v) curves can be generated\textsuperscript{44,46}, as illustrated in Figure 3.4. Type I represents a classical P-v plot for common ductile materials. Alloy 617 is known\textsuperscript{42} to be a very ductile material by virtue of a large number of slip planes associated with its FCC structure. A similar type of plot (P-v) will be displayed in a later section that resulted from K\textsubscript{IC} testing involving CT specimens of Alloy 617. Type II plot is a representation of P-v curve generated on ductile materials that may exhibit yield point (upper and lower yield point) phenomenon\textsuperscript{67}, which can be attributed to the diffusion of interstitial elements such as carbon and nitrogen. Finally, Type III curve shows P-v plot for predominantly brittle materials that hardly exhibit any plasticity beyond YS.
In view of the fact that Alloy 617 displayed a Type I P-\(v\) plot, the magnitude of \(K_Q\) was determined using \(P_Q\) that was evaluated by drawing a 5\% secant line to intersect the P-\(v\) plot at a point \(P_5\) such that \(P_5\) becomes identical to \(P_Q\). The determination of \(P_Q\) value by this method was performed to comply with the related ASTM designations.\(^{[44, 46]}\) The magnitude of \(K_Q\) was then determined using Equation 3.16, which includes a dimensionless geometric factor, \(f\left(\frac{a}{W}\right)\) given by Equation 3.17.

\[
K_Q = \frac{P_Q}{B \sqrt{W} \cdot f\left(\frac{a}{W}\right)} \quad \text{Equation 3.16}
\]
\[
f\left(\frac{a}{W}\right) = \left[2 + \frac{a}{W}\right] \frac{0.886 + 4.64 \frac{a}{W} - 13.32 \left(\frac{a}{W}\right)^2 + 14.72 \left(\frac{a}{W}\right)^3 - 5.6 \left(\frac{a}{W}\right)^4}{\left(1 - \frac{a}{W}\right)^2}\]

Equation 3.17

where,

\[B = \text{Specimen thickness (mm)}\]
\[a = \text{Crack length (mm)}\]
\[W = \text{Specimen width (mm)}\]

3.4 Crack Propagation Study

Crack propagation study under cyclic loading was performed involving CT specimens of Alloy 617 that were pre-cracked in a manner similar to that of the ASTM designation E 399-1997. As in the case of \(K_{IC}\) measurements, a load ratio (R) of 0.1 and a frequency of 1 Hz were used to pre-crack the CT specimens. Similar values of \(R\) and frequency were used in crack propagation study under cyclic loading. However, the magnitude of \(\sigma_{\text{max}}\) and \(\sigma_{\text{min}}\) were varied to some extent even though the magnitude of \(R\) was maintained constant (0.1). The monitoring of crack length, and the determination of crack-growth-rate (CGR) and the resultant stress-intensity factor (\(K\)) were performed using an in-situ device known as DCPD. The DCPD method is based on the measurement of increase in potential drop due to an increase in electrical resistance resulting from the propagation of a pre-existing crack at the root of a notch contained in a CT specimen. During CGR testing, the magnitude of the current input was kept constant so that
Ohm's law could be satisfied showing an increase in both electrical resistance and potential drop spanning the crack opening mouth of the CT specimen. The crack length was determined using Johnson's equation, shown in Equation 3.18. Subsequently, CGR under cyclic loading \( \frac{da}{dN} \) was estimated using Paris law, given by Equation 3.19. This equation can be modified in a logarithmic form (Equation 3.20), giving a linear relationship between \( \frac{da}{dN} \) and \( \Delta K \), from which the slope (m) of the line could be estimated.

For \( 0 \leq 2a/W \leq 1 \)

where,

\( a = \) Crack length (as defined in Test Method E647)

\( a_r = \) Reference crack length from some other method

\( W = \) Specimen width

\( V = \) Measured PD voltage (volts)

\( V_r = \) Measured voltage corresponding to \( a_r \)
\[ \frac{da}{dN} = A(\Delta K)^m \]  \hspace{1cm} \text{Equation 3.19}

\[ \log\left(\frac{da}{dN}\right) = \log(A) + m \log(\Delta K) \]  \hspace{1cm} \text{Equation 3.20}

where,

\[ \frac{da}{dN} \] = CGR under cyclic loading, mm/cycle

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

\[ K_{\text{max}} \] = Maximum stress intensity factor, MPa \sqrt{m}

\[ K_{\text{min}} \] = Minimum stress intensity factor, MPa \sqrt{m}

\[ a \] = Crack length, mm

\[ N \] = Number of cycles

\[ A \] = Constant

\[ m \] = Slope of the linear portion of the plot

\[ R = \frac{K_{\text{min}}}{K_{\text{max}}} \]

3.5 SCC Testing using DCB Specimens

The DCB specimens were loaded by inserting double tapered wedges of different thickness into their slots using the prescribed method.\textsuperscript{47, 69} Prior to their loading, they were pre-cracked up to a length of 2 mm using an Instron equipment according to the ASTM Designation E 399-1990.\textsuperscript{44} A cyclic load with an R value of 0.1 and frequency of 1 Hz was also used to pre-crack the DCB specimens. For determination of wedge
thickness, a DCB specimen was subjected to tensile loading up to the yielding load ($P_Y$), generating a load versus displacement curve. Two sets of load below $P_Y$ were selected that corresponded to two levels of displacement. The magnitudes of these displacements were then taken into consideration to determine the wedge thickness using Equation 3.21.

$$W = (t + \delta)$$

Equation 3.21

where,

- $W$ = Wedge thickness
- $t$ = Gap between the two arms of the DCB specimen
- $\delta$ = Displacement corresponding to the desired load (from the elastic load-displacement curve)

The pre-cracked and wedge-loaded DCB specimens were subsequently immersed into an acidic solution at 100°C using an autoclave (Figure 3.5) for durations of 30 and 45 days. The specimens, upon completion of testing, were unloaded by removing the wedges through application of tensile loads in an Instron equipment. The tested specimens were then broken apart under tensile loading, and the final crack length was measured on the broken surface using SEM micrographs developed on them. A fractographic montage was subsequently prepared using SEM to estimate the approximate size of different cracked regions. This montage enabled the determination of approximate crack extension due to exposure into the testing environment under a wedge-loaded condition.
3.6 Metallographic Evaluations

The metallurgical microstructure plays an important role in differentiating properties of one material from other. Thus, metallographic analysis constitutes a significant step in characterizing the performance of the material of interest. A Leica optical microscope (Figure 3.6) was used for both microstructural analyses and examinations of the tested specimen surface. This microscope had a maximum resolution of 1000X. A digital camera with a resolution of 1 mega pixel enabled image capture on a computer screen through use of a Leica image acquisition software program.

For metallurgical evaluation, it is customary to prepare the specimen surface with a high precision. Thus, a conventional metallographic procedure was used to prepare the specimen surface for such evaluation. This procedure consisted of mounting a sample using an appropriate ratio of epoxy and hardener. The mounted specimens were subsequently ground using rotating discs containing abrasive papers. The grinding procedure involved several stages using finer grades of abrasive paper each time. The
polished sample was washed with deionized water to prevent any contamination and dried with acetone and ethanol. Finally, etching of the polished surface was done using Kalling’s reagent. This etchant contained 2 grams of cupric chloride (CuCl₂), 40 ml of hydrochloric acid (HCl) and 80ml of methanol (CH₃OH).[70-72]

![Leica Optical Microscope](image)

**Figure 3.6 Leica Optical Microscope**

3.7 Fractographic Evaluations

The extent and morphology of failure of the tested specimens were evaluated by scanning electron microscopy (SEM). The mode of failure of metallic material can be determined by analysis of the resultant micrographs. All tested specimens were sectioned into ½ to ¾ of an inch in length to accommodate them inside the vacuum chamber of the SEM. In general, failure can be classified into two major types, namely, ductile and brittle.[58] Ductile failures are characterized by dimples. Brittle failures can be of two types, namely, intergranular and transgranular. An intergranular failure is characterized
by crack propagation along grain boundaries, while a transgranular failure propagates through the grains.

A SEM uses electrons from a metal filament that are collected and focused into a narrow beam scanning across the specimen. This beam synchronizes with a spot on a computer screen for image acquisition. Electrons scattered from the subject are detected to create a current, the strength of which makes the spot on the computer brighter or darker. Thus, a photographic image with an exceptional depth of field is obtained. A JEOL-5600 SEM (Figure 3.7) having a maximum resolution of 100,000X was used in fractographic evaluations.

Figure 3.7 Jeol-5600 Scanning Electron Microscope

3.8 Transmission Electron Microscopic Evaluations

A Tecnai G² S-TWIN transmission electron microscope (TEM), illustrated in Figure 3.8, was used to characterize defects such as dislocations in the tested tensile specimens.
TEM operates at an extremely high acceleration voltage of around 300 kV that allows a point-to-point resolution of 0.2 nanometer and a magnification capability of 1,000,000 times of the actual sample size. This TEM used in this investigation was equipped with a high-angle-annular-dark-field (HAADF) detector, an X-ray energy dispersive spectrometer (EDS), and a Gatan image filter (GIF). Multiple TEM samples were prepared from tensile specimens tested at different temperatures. The sample preparation procedure is described in the next subsection.

Figure 3.8 Tecnai G² S-TWIN TEM

3.8.1 TEM Sample Preparation

Preparation of samples for TEM analyses involves the utilization of both conventional and state-of-the-art techniques. To ensure electron transparency, the
thickness of these samples was maintained between 50-100 µm. The overall procedure of sample preparation is described below.\cite{60,73-77}

- Multiple circular discs of 500-700 µm thickness were cut from the gage section of the tested tensile specimens using a precision cutter shown in Figure 3.9.

- These discs were then mechanically ground to about 100-150 µm using a Beuhler grinding equipment. 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 3.10.

- Finally, electron transparency was achieved by electropolishing these discs under an applied potential of 55 volts at cryogenic temperatures of around -10°C. A twin-jet TenuPol-5 electropolisher (Figure 3.11) was used for this purpose. The composition of the electrolyte used in electropolishing was 5% perchloric acid (HClO₄) in methanol (CH₃OH). 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.
3.9 Phase Characterization

X-ray diffractometry (XRD) was used to characterize phase changes, if any, in tensile specimens tested at ambient temperature, 100, 800 and 900°C. A Panalytical X'PERT Pro X-Ray diffraction spectrometer (Figure 3.12) with Copper (Cu) K$_\alpha$ 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 using Equation 3.22.\textsuperscript{[82]} Phase identification was
performed by comparing the resultant d-spacing values to those existing in available standards and open literature. [27, 78-81, 83-84]

\[ n\lambda = 2d \sin \theta \]  
Equation 3.22

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 3.12 Panalytical X'PERT Pro XRD Spectrometer
CHAPTER 4

RESULTS

This section presents the results of tensile testing, fracture toughness \((K_{IC})\) evaluations, crack propagation data under cyclic loading \(\left(\frac{da}{dN}\right)\) and stress corrosion cracking (SCC) susceptibility of Alloy 617 using numerous experimental techniques. Further, an in-depth analyses of metallographic and fractographic characteristics have been performed on the tested specimens to develop a basic understanding of plastic deformation and environmental degradation of Alloy 617 under different experimental conditions.

4.1 Microstructural Evaluations

The metallurgical microstructures of two heats of solution-annealed Alloy 617 are illustrated in Figure 4.1 in an etched condition. An evaluation of these optical micrographs revealed typical austenitic grains containing annealing twins, which are common characteristics of Ni-base superalloys. The annealing twins resulted from thermal treatment imparted to these materials. Carbide precipitates were also seen within grains.
Figure 4.1 Optical Micrographs, Kalling's Reagent

(a) Heat # HV0860A

(b) Heat # HV1160
4.2 Tensile Properties Evaluation

The results of tensile testing of Alloy 617, performed at temperatures up to 1000°C at a strain rate of $10^{-3}$ sec$^{-1}$, are illustrated in Figure 4.2 in the form of superimposed engineering stress versus engineering strain (s-e) diagrams. A cursory examination of these plots reveals that the tensile strength of this alloy in terms of YS and UTS was, in general, gradually reduced with an increase in the testing temperature. For most engineering metals and alloys, the reduced tensile strength with increasing temperature is usually associated with enhanced ductility. Interestingly, this alloy was capable of maintaining relatively high tensile strength, in terms of both parameters at temperatures up to 700°C. No distinction could, however, be made between the YS and UTS at temperatures of 900 and 1000°C. The fact that Alloy 617 maintained relatively high tensile strength up to a temperature of 700°C may suggest that this alloy may have adequate structural stability to provide the desired efficiency in hydrogen generation using the S-I cycle. It should, however, be noted that the critical design stress needed to achieve the maximum possible efficiency are yet to be established. The superior tensile strength of Alloy 617 at elevated temperatures may also be beneficial in transferring heat from nuclear reactors through the intermediate heat exchanger to be used in the NGNP program.

The magnitudes of different tensile properties determined from the s-e diagram and 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 enhancement at higher temperatures. The reduced failure strain ($e_\text{f}$) at 100°C may sound irrational or inconsistent from a basic metallurgical deformation
mechanism point of view. However, a similar phenomenon of reduced $e_f$ at higher temperatures has also been reported for other Ni-base superalloys (Alloys 276 and 22)$^{31-32}$, that have recently been tested at MPL.

![Figure 4.2 s-e Diagrams vs. Temperature](image)

Beyond 100°C, the magnitude of $e_f$ / $\%E_l$ was enhanced by an appreciable amount, which remained consistent up to 700°C. Simultaneously, serrations of varied heights were seen in the s-e diagrams corresponding to testing temperatures ranging from 100 to 700°C. The occurrence of reduced $e_f$ at 100°C, and the formation of serrations at different higher temperatures have been known $^{33-39}$ to be the result of a complex phenomenon, known as the dynamic strain aging (DSA) of metallic materials. The concept of DSA has been postulated to be associated with the diffusion of either interstitial or substitutional
solute elements into the metal matrix, which can impede the dislocation mobility through grains and past the grain boundaries. Since the plastic deformation in a metallic material is caused by the movement of dislocations through its lattice, a reduced dislocation mobility will, thus, lead to a lower plastic strain. A detailed analysis of the DSA phenomenon observed with Alloy 617, in terms of several related parameters, are presented later in appropriate locations of this dissertation. These parameters include the dislocation density ($\rho$), activation energy ($Q$), and work hardening index ($n$).

Table 4.1 Average Tensile Properties versus Temperature

<table>
<thead>
<tr>
<th>Temperature(°C)</th>
<th>YS ksi (MPa)</th>
<th>UTS Ksi (MPa)</th>
<th>%El</th>
<th>%RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>54 (371)</td>
<td>124 (856)</td>
<td>78.35</td>
<td>61.98</td>
</tr>
<tr>
<td>100</td>
<td>44 (306)</td>
<td>112 (774)</td>
<td>74.40</td>
<td>56.88</td>
</tr>
<tr>
<td>200</td>
<td>41 (283)</td>
<td>110 (761)</td>
<td>78.41</td>
<td>60.17</td>
</tr>
<tr>
<td>300</td>
<td>38 (265)</td>
<td>109 (752)</td>
<td>77.71</td>
<td>56.20</td>
</tr>
<tr>
<td>400</td>
<td>37 (254)</td>
<td>106 (728)</td>
<td>79.90</td>
<td>57.41</td>
</tr>
<tr>
<td>500</td>
<td>35 (244)</td>
<td>101 (697)</td>
<td>78.68</td>
<td>53.74</td>
</tr>
<tr>
<td>600</td>
<td>32 (221)</td>
<td>100 (688)</td>
<td>79.05</td>
<td>48.56</td>
</tr>
<tr>
<td>700</td>
<td>31 (211)</td>
<td>87 (598)</td>
<td>80.83</td>
<td>48.44</td>
</tr>
<tr>
<td>800</td>
<td>34 (234)</td>
<td>57 (392)</td>
<td>100.23</td>
<td>73.02</td>
</tr>
<tr>
<td>900</td>
<td>34 (237)</td>
<td>35 (240)</td>
<td>84.49</td>
<td>78.53</td>
</tr>
<tr>
<td>1000</td>
<td>19 (131)</td>
<td>19 (131)</td>
<td>88.23</td>
<td>72.07</td>
</tr>
</tbody>
</table>

An evaluation of the tensile data, shown in Table 4.1, reveals that the magnitude of YS was appreciably enhanced at temperatures of 800 and 900°C, followed by its substantial drop at 1000°C. The occurrence of enhanced strength, in terms of YS at relatively higher temperatures in Ni-base superalloys, has been termed as yield strength anomaly (YSA). Several mechanisms have been proposed by numerous investigators to explain the underlying principles of plastic deformation and YSA in austenitic metals and alloys at some critical temperatures. However, not a single
uniformed mechanism has yet been accepted by researchers investigating the YSA phenomenon. Such differences in mechanistic understanding stem from the fact that the compositions of the investigated materials vary significantly even though all of them may fall under the category of Ni-base superalloys. Further, the compositional variations may identify different temperature ranges at which these alloys may become susceptible to the YSA phenomenon.

It should be noted that the DSA / YSA phenomena of Alloy 617 has not yet been investigated by outside researchers prior to the present study performed at UNLV. Both XRD and TEM have been employed to determine phase changes, if any, and dislocations/precipitates interactions, respectively to develop a basic understanding of YSA in Alloy 617 within the temperature regime of 800-900°C. A mechanistic understanding of YSA, based on the current investigation, will be presented later in this section, and its rationalization be made in comparison to those postulated by other investigators. The variations of YS, UTS, %El and %RA with temperature are illustrated in Figures 4.3 through 4.6, respectively. The enhanced ductility, in terms of %El and %RA at 800 and 900°C despite high YS values, is unexplainable. However, a greater ductility at 1000°C can be attributed to the temperature-induced plastic deformation at this elevated temperature.
Figure 4.3 YS vs. Temperature

Figure 4.4 UTS vs. Temperature
Figure 4.5 %El vs. Temperature

Figure 4.6 %RA vs. Temperature
4.2.1 Dislocation Density Calculation

It has been postulated [55-56] that the reduced $\varepsilon_f$ and the formation of serrations in the s-e diagrams at different temperatures are the result of impaired dislocation mobility through the matrix and the grain boundaries of a structural material. Therefore, it is essential to calculate the dislocation density of specimens at and around the temperature, where a tendency to DSA was observed with Alloy 617. The average values of $\rho$ corresponding to these temperatures (room temperature, 100, 200 and 300°C), calculated through the placement of grids at ten (10) different locations of the respective TEM micrographs (Figures 4.7 to 4.10), are given in Table 4.2. An evaluation of these data clearly reveals that the average value of $\rho$ was higher at 100°C by an order of magnitude, compared to those at other temperatures. A maximum value of $\rho$, observed in the specimen tested at 100°C, is also consistent with the lowest $\varepsilon_f$ value seen in the s-e diagram, implying the occurrence of the DSA phenomenon at this temperature. The variation of $\rho$ with temperature is illustrated in Figure 4.11. Numerous studies [31-32, 92] involving other austenitic alloys, and martensitic T91 grade steels have also exhibited a similar pattern, showing a maximum dislocation density at a temperature, where a susceptibility to DSA was observed.
Figure 4.7 TEM Micrograph of Alloy 617 Tested at Ambient Temperature

Figure 4.8 TEM Micrograph of Alloy 617 Tested at 100°C
Figure 4.9 TEM Micrograph of Alloy 617 Tested at 200°C

Figure 4.10 TEM Micrograph of Alloy 617 Tested at 300°C
Table 4.2 $\rho$ versus Temperature

<table>
<thead>
<tr>
<th>Test temperature ($^\circ$C)</th>
<th>$\rho$ (No./m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>2.08E+17</td>
</tr>
<tr>
<td>100</td>
<td>1.42E+18</td>
</tr>
<tr>
<td>200</td>
<td>6.54E+17</td>
</tr>
<tr>
<td>300</td>
<td>8.95E+17</td>
</tr>
</tbody>
</table>

Figure 4.11 $\rho$ vs. Temperature
4.2.2 Activation Energy Calculation

As indicated earlier, the concept of DSA is known to be influenced by both temperature and strain rate. Therefore, a combination of different temperature and strain rate \( \varepsilon \) has been used to determine the magnitude of \( Q \) needed for the initiation of serrations in the s-e diagrams. At the onset of tensile properties evaluation, an \( \varepsilon \) value of \( 10^{-3} \) sec\(^{-1}\) was used. Subsequently, duplicate testing was performed at two additional strain rates \( (10^{-2} \) and \( 10^{-4} \) sec\(^{-1}\)), above and below this rate, to determine the average \( Q \) value at temperatures of 100, 500 and 600°C. The superimposed s-e diagrams generated at 100, 500 and 600°C using \( \varepsilon \) values of \( 10^{-2} \) and \( 10^{-4} \) sec\(^{-1}\) are illustrated in Figures 4.12 and 4.13, respectively. The critical plastic strain \( (\varepsilon_c) \) necessary to initiate serrations in the s-e diagram is known\(^{33}\) to be a function of both true strain rate \( (\varepsilon) \) and temperature. At the onset of serration, \( \dot{\varepsilon} \) can be taken as \( \varepsilon \) due to an insignificant variation between both parameters. The variation of \( \varepsilon_c \) with temperature at different strain rates is illustrated in Figure 4.14, showing a gradual drop in \( \varepsilon_c \) with temperature at a constant \( \varepsilon \). A similar pattern on the variation of \( \varepsilon_c \) with temperature at different strain rates has also been reported elsewhere.\(^{31-33}\)
Figure 4.12 s-e Diagram vs. Temperature

Figure 4.13 s-e Diagram vs. Temperature
As indicated in Chapter 3, two different methods have been used to determine the magnitude of Q. The first method of Q calculation was based on the slope \( \frac{Q}{R(m + \beta)} \) derived from the plot of \( \ln(e^c) \) versus \( \frac{1}{T} \), as illustrated in Figure 4.15. The magnitude of \((m + \beta)\) was determined from the plot of \( \ln(e^c) \) versus \( \ln(e^c) \), as shown in Figure 4.16. The average value of Q was then calculated by substituting value of \((m + \beta)\) in the \( \left(\frac{Q}{R(m + \beta)}\right) \) term. The measured values of \((m + \beta)\), as functions of temperature and strain rate, and the corresponding average Q values are given in Table 4.3. Thus, based on the first method, an average Q value of 29 KJ/mole can be approximated.
Figure 4.15 $\ln(\varepsilon^c)$ vs. $\frac{1}{T}$

Figure 4.16 $\ln \dot{\varepsilon}$ vs. $\ln \varepsilon^c$
Table 4.3 \((m + \beta)\) and Q Values (Method One)

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>((m + \beta)) (\dot{\varepsilon}) (sec(^{-1}))</th>
<th>Q (KJ/mole)</th>
<th>Average Q (KJ/mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(10^{-2})</td>
<td>(10^{-3})</td>
<td>(10^{-4})</td>
</tr>
<tr>
<td>100</td>
<td>3.75</td>
<td>3.75</td>
<td>3.75</td>
</tr>
<tr>
<td>500</td>
<td>7.61</td>
<td>7.61</td>
<td>7.61</td>
</tr>
<tr>
<td>600</td>
<td>7.76</td>
<td>7.76</td>
<td>7.76</td>
</tr>
</tbody>
</table>

The second method of Q calculation was based on the McCormick’s strain aging model \(^{33-34}\), in which \(\frac{Q}{k}\) represented the slope of a line when \(\ln \left[ \frac{\dot{\varepsilon}^{(m+\beta)}}{T} \right] \) was plotted as a function of \(\frac{1}{T}\) at a constant \(\dot{\varepsilon}\). The resultant linear relationship is shown in Figure 4.17. Substituting the value of \(k\) (Boltzmann’s constant) in this slope, the magnitude of Q was computed. The average value of Q, as a function of \(\dot{\varepsilon}\) was found to be approximately 94 KJ/mole, as given in Table 4.4. A comparison of the Q values determined by two methods indicates that there was a significant difference in activation energy (29 versus 94 KJ/mole) for initiation of serrations in Alloy 617 as functions of temperature and \(\dot{\varepsilon}\). However, a wide variation in Q value has also been reported by other investigators \(^{34, 93-94}\) using different methods. In view of such differences in the calculated Q values, an average value based on multiple methods has often been reported by these investigators.
Based on a same rationale, an average Q value of 62 KJ/mole can be approximated for the initiation of serrations in Alloy 617.

Figure 4.17 \( \ln \left( \frac{\varepsilon (\omega, \rho)}{T} \right) \) vs. \( \frac{1}{T} \)

Table 4.4 Q Value (Method Two)

<table>
<thead>
<tr>
<th>( \varepsilon ) (sec(^{-1}))</th>
<th>Q (KJ/mole)</th>
<th>Average Q (KJ/mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 10^{-2} )</td>
<td>93.31</td>
<td>93.74</td>
</tr>
<tr>
<td>( 10^{-3} )</td>
<td>94.57</td>
<td>93.74</td>
</tr>
<tr>
<td>( 10^{-4} )</td>
<td>93.35</td>
<td>93.74</td>
</tr>
</tbody>
</table>
4.2.3 Estimation of Work-Hardening Index

The average values of work-hardening index (n), based on triplicate tensile testing of Alloy 617 at temperatures ranging from ambient to 600°C using a strain rate of $10^{-3}$ sec$^{-1}$, are given in Table 4.5. These data suggest that the magnitude of n was not appreciably affected by the variation in temperature at a constant strain rate. Therefore, tensile testing was performed at three selected temperatures of 100, 500 and 600°C using strain rates of $10^{-2}$ and $10^{-4}$ sec$^{-1}$. A comparative analysis of the resultant n values, based on testing at three strain rates ($10^{-2}$, $10^{-3}$ and $10^{-4}$ sec$^{-1}$), is presented in Table 4.6. An evaluation of these data indicates that the magnitude of n was gradually enhanced at 500 and 600°C with slower strain rates. However, at 100°C, n value was increased to some extent at an intermediate strain rate of $10^{-3}$ sec$^{-1}$. A combined effect of slower strain rate and higher testing temperature could possibly account for greater plastic deformation over longer durations, thus, leading to higher n-values at elevated temperatures under gradually reduced strain rates.
Table 4.5 \( n \) vs. Temperature

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>( \varepsilon ) (sec(^{-1}))</th>
<th>Average ( n ) Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room Temperature</td>
<td>0.62</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td></td>
<td>0.65</td>
</tr>
<tr>
<td>200</td>
<td></td>
<td>0.69</td>
</tr>
<tr>
<td>300</td>
<td>(10^3)</td>
<td>0.7</td>
</tr>
<tr>
<td>400</td>
<td></td>
<td>0.7</td>
</tr>
<tr>
<td>500</td>
<td></td>
<td>0.68</td>
</tr>
<tr>
<td>600</td>
<td></td>
<td>0.68</td>
</tr>
</tbody>
</table>

Table 4.6 \( n \) vs. Temperatures 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 (°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

As indicated earlier in this section, Alloy 617 exhibited an anomalous increase in YS within a temperature range of 800 to 900°C. Such phenomenon of enhanced YS within a specific temperature range, known as yield strength anomaly (YSA), has been noted in many intermetallic alloys as well as in pure metals and disordered alloys. However, a generalized interpretation of this phenomenon is yet to be developed. YSA has been observed in intermetallic alloys on slip systems involving ordinary dislocations and super-dislocations. This phenomenon has also been linked to phase transitions in ordered and disordered alloys, where YS increases up to an order-disorder transition temperature and then sharply decreases.

The volume fraction and the morphology of precipitates formed at elevated temperatures, if any, depend on the composition and lattice mismatch parameters of alloys susceptible to the YSA phenomenon. Higher the volume percent of these precipitates, the more effective is their shear mechanism. Factors such as coherency strains at the matrix-precipitate interface, differences among their elastic modulii, lattice mismatch and long range order-disordering of the precipitates can significantly contribute to the strengthening mechanisms of different alloys due to YSA. Glatzel et. al. suggested that the misfit strains resulting from the differences in macroscopic thermal expansion and the compositional changes among the matrix and resultant precipitates within the susceptible temperature regime may also account for such anomalous behavior.

TEM has been employed to develop a mechanistic understanding of YSA observed with Alloy 617, when tested under tensile loading at 900°C. A TEM micrograph of a
sample, prepared from the gage section of the tested tensile specimen located away from the primary fracture surface, is shown in Figure 4.18. A cursory examination of this micrograph reveals a combination of precipitates, pinned dislocations and pile-up of dislocations in and around the grain boundaries of the tested specimen. In addition, the development of subgrains also occurred due to the formation and accumulation of dislocations oriented in different directions. These subgrains, displayed in Figure 4.19 in a magnified manner for clarity, are much smaller in size compared to those of typical austenitic grains. Some of the dislocations formed during plastic deformation could also be pinned and piled up in the vicinity of these subgrains and grain boundaries, causing strengthening of the material leading to the YSA phenomenon. Further, the formation of precipitates around subgrains and grain boundaries, shown in Figure 4.20, could also contribute to the enhanced YS at the tested temperature.

![TEM Micrograph of Specimen Tested at 900°C](image)

Figure 4.18 TEM Micrograph of Specimen Tested at 900°C
Figure 4.19 TEM Micrograph Showing Subgrains

Figure 4.20 TEM Micrograph Showing Precipitates
Scanning transmission electron microscopic (STEM) image mode, in conjunction with an energy dispersive spectrometry (EDS) was subsequently used to perform elemental analysis both at a precipitate and within the matrix. The resultant spectra and the chemical compositions of both precipitate and matrix are shown in Figure 4.21 and Table 4.7, respectively. As anticipated, both spectra exhibited elements that are commonly present in Ni-base austenitic alloys including Alloy 617. However, the concentrations of these elements, as detected by EDS, were different at the matrix and the precipitate due to metallurgical transformation that occurred at 900°C. An evaluation of the elemental analysis performed at both locations indicates that the concentrations of Cr, Mo and carbon (C) were increased, while the Ni content was substantially reduced in the precipitate. Simultaneously, line scanning was also performed across a precipitate using STEM image mode to differentiate the concentration of elements present in the matrix from that of the precipitate, as shown in Figure 4.22. Once again, the concentration of Cr and Mo was enhanced in the precipitate, verifying the observation made in the EDS study.
Figure 4.21 Spectra for Precipitate and Matrix

Table 4.7 Compositional Variation at Precipitate vs. Matrix

<table>
<thead>
<tr>
<th>Element</th>
<th>Precipitate (wt%)</th>
<th>Matrix (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>2.396</td>
<td>0.000</td>
</tr>
<tr>
<td>Al</td>
<td>0.541</td>
<td>0.315</td>
</tr>
<tr>
<td>S</td>
<td>3.503</td>
<td>2.386</td>
</tr>
<tr>
<td>Ti</td>
<td>1.397</td>
<td>1.230</td>
</tr>
<tr>
<td>Cr</td>
<td>25.882</td>
<td>20.444</td>
</tr>
<tr>
<td>Co</td>
<td>11.428</td>
<td>12.785</td>
</tr>
<tr>
<td>Ni</td>
<td>42.316</td>
<td>51.187</td>
</tr>
<tr>
<td>Mo</td>
<td>12.534</td>
<td>11.649</td>
</tr>
</tbody>
</table>
(a) STEM Image

(b) Line Scan Spectra

Figure 4.22 Line Scan Elemental Analyses
X-ray diffractometry (XRD) was also performed on specimens tested under tensile loading at 800 and 900°C to characterize the precipitates formed due to metallurgical transformation. The resultant spectra, showing intensity versus 2θ (based on Bragg's law \(^{[82]}\)) are shown in Figures 4.23 and 4.24, respectively. Efforts were made to identify the elements / precipitates corresponding to these peaks by comparing them to those with identical 2θ values cited in the open literature.\(^{[27]}\) The magnitudes of d-spacings, calculated from the 2θ values, were then compared to those existing in the literature, as shown in Table 4.8. These results confirm the formation of \(M_{23}C_6\) type of precipitates based on the calculated d-spacings corresponding to the 2θ values. The calculated d-spacings match well with the standard values prescribed by the International Center for Diffraction Data (ICDD)\(^{[83,84]}\) for \(M_{23}C_6\) type of precipitates.

![Figure 4.23 Intensity vs. 2θ at 800°C](image)

Figure 4.23 Intensity vs. 2θ at 800°C
Figure 4.24 Intensity vs. 2θ at 900°C

Table 4.8 Comparison of Calculated d-spacing to ICDD Standard.

<table>
<thead>
<tr>
<th>Calculated d-spacing</th>
<th>M$_{23}$C$_6$ d-spacing (ICDD Standard)</th>
</tr>
</thead>
<tbody>
<tr>
<td>800°C</td>
<td>900°C</td>
</tr>
<tr>
<td>2.076</td>
<td>2.074</td>
</tr>
<tr>
<td>1.802</td>
<td>1.801</td>
</tr>
<tr>
<td>1.268</td>
<td>1.270</td>
</tr>
<tr>
<td>1.084</td>
<td>1.084</td>
</tr>
</tbody>
</table>
4.3 $K_{IC}$ Evaluation

Duplicate testing was performed at ambient temperature and 100°C involving pre-cracked CT specimens having a thickness of 0.25 inch. As indicated in previous two chapters, a conditional stress intensity factor ($K_Q$) was determined from the load versus displacement ($P-v$) curve. $P-v$ curves, generated at ambient temperature and 100°C are illustrated in Figures 4.25 and 4.26, respectively. The magnitude of $P_Q$, determined according to the applicable ASTM Designations, is identified individually in each plot. $K_Q$ was then computed using Equations 3.16 and 3.17. The calculated values of $K_Q$, obtained from the $K_{IC}$ testing, are given in Table 4.9. These data suggest that consistent values of $K_Q$ were observed at each temperature. It was also interesting to note that, the magnitude of the average $K_Q$ was appreciably reduced (90 versus 76 MPa$\sqrt{m}$) due to an increase in temperature from ambient to 100°C. The detrimental effect of higher temperature on $K_{IC}$, as seen in this study, has also been observed by other investigators.\textsuperscript{[101-102]}

Figure 4.25 P- $v$ Curve at Ambient Temperature
Table 4.9 Results of $K_{IC}$ Measurements

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$P_0$ (N)</th>
<th>$K_Q$ MPa$\sqrt{m}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test 1</td>
<td>Test 2</td>
</tr>
<tr>
<td>Ambient</td>
<td>13.97</td>
<td>13.87</td>
</tr>
<tr>
<td>100</td>
<td>12.90</td>
<td>12.00</td>
</tr>
</tbody>
</table>

4.4 Determination of Crack-Growth-Rate

The structural materials to be used in both NHI and NGNP programs must withstand a maximum operating temperature of 950°C. However, temperatures less than 950°C may also be encountered from time to time during operations. Thus, variations in operating temperature may lead to the development of fluctuating thermal stresses. Based on this rationale, crack-growth-rate (CGR) studies were performed under cyclic loading at ambient and elevated temperatures involving CT specimens of Alloy 617. A decision
was made not to perform CGR testing at very high temperatures (above 200°C) due to a possibility of formation of blunted crack tip at elevated temperatures. A step-by-step approach was taken to progressively increase the testing temperature to accommodate CGR testing of this alloy. Triplicate testing was performed at ambient, 100, 150 and 200°C.

As indicated in the preceding chapter, the rate of crack propagation under cyclic loading \( \frac{da}{dN} \) is related to the stress intensity factor range \( \Delta K \) by an empirical relationship given by Equation 3.19. A logarithmic plot of \( \frac{da}{dN} \) versus \( \Delta K \) can generally be represented by Figure 4.27 \cite{103}, showing a sigmoidal shape that can be classified into three regions. Region I is bounded by a threshold value \( \Delta K_{TH} \), below which no appreciable crack-growth is commonly observed. In essence, at stress intensity ranges below \( \Delta K_{TH} \), cracks behave as non-propagating cracks. Region II represents a linear relationship between \( \log \left( \frac{da}{dN} \right) \) and \( \log(\Delta K) \). Finally region III is a region of accelerated crack growth, at which \( K_{max} \) approaches fracture toughness \( (K_{IC}) \) of a material of interest. The CGR data in terms of \( \log \left( \frac{da}{dN} \right) \) versus \( \log(\Delta K) \) as a function of temperature are illustrated in Figures 4.28 through 4.31. It should be noted that the plots, shown in these figures, were generated using a load ratio (R) and a loading frequency of 0.1 and 1 Hz, respectively. An examination of these figures clearly reveals three distinct regions, as identified in Figure 4.27. The linear portions of these plots are reproduced in Figures 4.32 through 4.35, showing the magnitude of m as a function of temperature.
Figure 4.27 log\( \frac{da}{dN} \) vs. log(\( \Delta K \)) \( \frac{da}{dN} = A(\Delta K)^m \)
Figure 4.28 CGR Data at Ambient Temperature

Figure 4.29 CGR Data at 100°C
Figure 4.30 CGR Data at 150°C

Figure 4.31 CGR Data at 200°C
Figure 4.32 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region

Figure 4.33 $\log \left( \frac{da}{dN} \right)$ vs. $\log(\Delta K)$, Linear Region
Figure 4.34 $\log\left(\frac{da}{dN}\right)$ vs. $\log(\Delta K)$, Linear Region

Figure 4.35 $\log\left(\frac{da}{dN}\right)$ vs. $\log(\Delta K)$, Linear Region
The overall CGR data of Alloy 617 are given in Table 4.10, showing the average values of all relevant parameters. The resultant number of cycles (N) generated from the CGR testing is plotted as a function of temperature, as shown in Figure 4.36. An evaluation of Figure 4.36 indicates that the number of cycles needed to achieve comparable crack-growth was drastically reduced with an increase in testing temperature from ambient to 100°C, suggesting that crack propagation might be significantly enhanced at this temperature even though the other experimental variables were kept constant. It is however, interesting to note that the magnitude of N needed for crack extension at 150°C was not significantly different from that at 100°C. Beyond 150°C, a relatively greater reduction in N value was noted, possibly due to the blunting of the crack-tip resulting from enhanced plastic deformation at this elevated temperature. The variation of N with temperature is shown in Figure 4.36.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>( a_i ) (mm)</th>
<th>( a_f ) (mm)</th>
<th>( \Delta a ) (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>15.24</td>
<td>10.16</td>
<td>2.54</td>
<td>6.60</td>
<td>4.99</td>
<td>128725</td>
</tr>
<tr>
<td>100</td>
<td>5.08</td>
<td>15.01</td>
<td>9.93</td>
<td>2.51</td>
<td>6.21</td>
<td>6.09</td>
<td>94559</td>
</tr>
<tr>
<td>150</td>
<td>5.08</td>
<td>15.10</td>
<td>10.02</td>
<td>2.47</td>
<td>6.54</td>
<td>6.05</td>
<td>91040</td>
</tr>
<tr>
<td>200</td>
<td>5.08</td>
<td>14.27</td>
<td>9.19</td>
<td>2.51</td>
<td>5.80</td>
<td>6.31</td>
<td>85068</td>
</tr>
</tbody>
</table>
An evaluation of the resultant data, shown in Table 4.10, indicates that the average value of m within the linear region ranged between 4.99 and 6.31. Literature \cite{104, 105} suggests that the magnitude of m can vary between 2.5 and 7 depending on the type of a material of interest. Thus, the resultant m values match well with those cited in the open literature. Efforts have been made to relate the $\Delta K_{(final)}$ to the overall CGR in terms of $\left(\frac{da}{dN}\right)$, as shown in Table 4.11 as a function of the testing temperature. An evaluation of these data indicates that, while Alloy 617 exhibited a reduced $\Delta K_{(final)}$ value at 100°C, its CGR was enhanced at this temperature. Thus, it is obvious that this alloy might undergo enhanced crack extension at a temperature approaching 100°C. It is, however, interesting to note that neither $\Delta K_{(final)}$ nor $\left(\frac{da}{dN}\right)$ was appreciably changed due to an change in
temperature from 100 to 150°C, suggesting a threshold temperature range (100-150°C) within which the CGR may not be influenced by such variation in temperature. Consistent with the lowest N value, observed at 200°C (Table 4.10), both $\Delta K_{(\text{final})}$ and $\left(\frac{da}{dN}\right)$ values were also appreciably reduced at this temperature, possibly due to the blunting of crack-tip resulting from enhanced plastic deformation at a higher temperature.

Table 4.11 $\Delta K_{(\text{final})}$ 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>59.12</td>
<td>2.08E-2</td>
</tr>
<tr>
<td>100</td>
<td>56.02</td>
<td>4.93E-2</td>
</tr>
<tr>
<td>150</td>
<td>57.32</td>
<td>4.24E-2</td>
</tr>
<tr>
<td>200</td>
<td>52.12</td>
<td>1.63E-2</td>
</tr>
</tbody>
</table>

4.5 DCB Test Results

The results of SCC testing involving pre-cracked and wedge-loaded DCB specimens of Alloy 617 are given in Table 4.12, showing initial and final crack lengths ($a_i$ and $a_f$, respectively), change in crack length ($\Delta a$), and average CGR based on testing durations of 30 and 45 days. Specimens 1 and 2 were tested under lower initial loads imparted by
the wedges, while specimens 3 and 4 were subjected to higher wedge loads. The resultant data indicate that at the lower load, the DCB specimen did not exhibit any crack extension after 30 day's of exposure into the 100°C acidic solution. However, a negligible crack growth (0.221 mm) was observed with the specimen exposed to the same environment for 45 days. On the other hand, specimens subjected to higher initial load exhibited measurable crack extensions at the end of both 30 and 45 days of testing. Thus, these results suggest a synergistic effect of a higher applied load and a longer test duration in causing a greater CGR value, when immersed in an identical aqueous solution.

Table 4.12 Results of DCB Testing *

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>ai (mm)</th>
<th>ar (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>30.7949</td>
<td>0.221</td>
<td>45</td>
<td>5.68E-8</td>
</tr>
<tr>
<td>3</td>
<td>30.5739</td>
<td>30.9727</td>
<td>0.3988</td>
<td>30</td>
<td>1.53E-7</td>
</tr>
<tr>
<td>4</td>
<td>30.5739</td>
<td>31.2394</td>
<td>0.6655</td>
<td>45</td>
<td>1.71E-7</td>
</tr>
</tbody>
</table>

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

4.6 Fractographic Evaluations

The results of fractographic evaluation involving cylindrical specimens of Alloy 617 strained under tensile loading at a rate of $10^{-3} \text{ sec}^{-1}$ within a temperature range of ambient-1000°C, are illustrated in Figure 4.37. An evaluation of these SEM micrographs
indicates that Alloy 617 suffered from intergranular brittle failure at temperatures ranging from ambient-300°C (Figures 4.37a through 4.37d). However, the severity of cracking was more pronounced with the specimen tested at 100°C, possibly due to its lowest ductility in terms of εf. Between 400 and 600°C, the morphology of failure was predominantly ductile characterized by dimpled microstructures (Figures 4.37e through 4.37g). At temperatures above 600°C, the SEM micrographs revealed a combination of ductile and brittle failures, characterized by dimples and intergranular cracking, respectively. As indicated earlier, the susceptibility of Alloy 617 to the YSA phenomenon was observed at temperatures of 800 and 900°C, at which an anomalous increase in YS was seen.
(b) 100°C

(c) 200°C
(d) 300°C

(e) 400°C
(h) 700°C

(i) 800°C
Figure 4.37 SEM Micrographs of Alloy 617 Specimens Tested at $10^{-3}$ sec$^{-1}$
The phenomenon of DSA, as described before, is known to be influenced by both temperature and strain rate. Therefore, tensile testing was performed at strain rates of $10^{-2}$ and $10^{-4}$ sec$^{-1}$ at temperatures of 100, 500 and 600°C. The fracture morphologies of these specimens are shown in SEM micrographs, identified by Figures 4.38 and 4.39, respectively. It is interesting to note that Alloy 617 exhibited intergranular brittle failure (Figures 4.37b, 4.38a and 4.39a) at 100°C irrespective of the strain rate applied during tensile testing. On the contrary, predominantly ductile failures, characterized by dimpled microstructures, were seen at 500 and 600°C at all strain rates ($10^{-2}$, $10^{-3}$ and $10^{-4}$ sec$^{-1}$).
Figure 4.38 SEM Micrographs of Alloy 617 Specimens Tested at $10^2$ sec$^{-1}$
Figure 4.39 SEM Micrographs of Alloy 617 Specimens Tested at $10^{-4}$ sec$^{-1}$

The SEM micrographs of the CT specimens used in the determination of stress-intensity-factor at ambient temperature and 100°C are illustrated in Figures 4.40 and 4.41. The failure modes of the CT specimens, used in CGR testing under cyclic loading at ambient temperature, 100, 150 and 200°C, were also analyzed by SEM. The resultant fracture morphologies of these specimens are shown in Figures 4.42 through 4.45. As expected, all CT specimens exhibited a combination of striations and dimples on the broken surfaces, which could be the result of cyclic loading and fast fracture, respectively.
Figure 4.40 SEM Micrographs of CT Specimen used in $K_Q$ Evaluation at Ambient Temperature

(a) Fatigue Failure (Striations)

(b) Fast Fracture (Dimples)
Figure 4.41 SEM Micrographs of CT Specimen used in $K_Q$ Evaluation at 100°C
Figure 4.42 SEM Micrographs of CT Specimen used in CGR Testing at Ambient Temperature

(a) Fatigue Failure (Striations)

(b) Fast Fracture (Dimples)
(a) Fatigue Failure (Striations)

(b) Fast Fracture (Dimples)

Figure 4.43 SEM Micrographs of CT Specimen used in CGR Testing at 100°C
Figure 4.44 SEM Micrographs of CT Specimen used in CGR Testing at 150°C
(a) Fatigue Failure (Striations)

(b) Fast Fracture (Dimples)

Figure 4.45 SEM Micrographs of CT Specimen used in CGR Testing at 200°C
The SEM micrographs taken at three regions of a wedge-loaded and fatigue pre-cracked DCB specimen, tested in a 100°C acidic solution, are illustrated in Figure 4.46. As anticipated, striations were seen in this micrograph from the tip of the notch up to the region of environment-assisted-cracking (SCC). The SCC morphology was characterized by transgranular brittle failure identified by cleavage. Beyond the SCC region, dimpled microstructures, indicating ductile failures, were observed that resulted from fast fracture of the DCB specimens under tensile loading.

Figure 4.46 SEM Micrographs of a DCB Specimen Showing Three Types of Failure
CHAPTER 5

DISCUSSION

Ni-base austenitic Alloy 617 has been tested for evaluation of its tensile properties at temperatures relevant to both NHI and NGNP programs. This alloy may undergo flow-induced plastic deformation due to the fluctuation in temperature during the \( \text{H}_2\text{SO}_4 \) decomposition process related to hydrogen generation using a thermochemical (S-I) cycle. Therefore, the susceptibility of this alloy to plastic deformation in the presence of flaws, characterized by stress intensity factor (\( K_{1C} \)), has been evaluated at ambient and elevated temperature under tensile loading. Simultaneously, the crack-growth-rates (CGR) of Alloy 617 have been determined at ambient and elevated temperatures using mechanical variables having values identical to those used in \( K_{1C} \) measurements. A limited number of testing has also been performed to evaluate the average crack-extension-rate of this alloy due to a synergistic effect of wedge-load and an acidic environment. SEM and TEM have been extensively employed to characterize the morphology of failure of the tested specimens, and to develop a mechanistic understanding of dynamic strain aging (DSA) and yield strength anomaly (YSA) phenomena, respectively. XRD has also been used to characterize precipitates related to the occurrence of YSA in Alloy 617.

The results of tensile testing indicate that Alloy 617 became susceptible to two important metallurgical phenomena, namely DSA and YSA, respectively, when the
specimens were strained in tension at an intermediate rate of $10^3$ sec$^{-1}$. The concept of DSA is usually manifested by the occurrence of reduced plastic strain ($\epsilon_f$) and formation of serrations within different ranges of temperature. Alloy 617 exhibited the lowest $\epsilon_f$ value at 100°C, followed by its enhancement at 200°C and beyond. The onset of serrations was also initiated at 100°C, followed by their continuation at temperatures up to 700°C. As postulated earlier, DSA of susceptible materials is known to be associated with the diffusion of solute or solvent elements through their matrices and in the vicinity of their grain boundaries, thus, minimizing or preventing the mobility of dislocations generated during plastic deformation at elevated temperatures. Such incidents may reduce the magnitude of $\epsilon_f$, as observed in the present study. The formation of serrations of different heights is also a function of the precipitation of diffused elements within the metal matrix at elevated temperatures, thus, requiring progressively higher and lower stresses to cause continued plastic deformation.

Obviously, the interaction of the diffused elements and precipitates with dislocations can play an important role in causing DSA of susceptible material. Accordingly, the magnitude of dislocation density ($\rho$) was computed from the TEM micrographs of the specimens tested within a critical temperature range and beyond. Interestingly, the highest value of $\rho$ was observed with the specimen tested at 100°C, which also exhibited the lowest $\epsilon_f$ value. Thus, the resultant data are consistent in that, they verified the underlying mechanism of the DSA phenomenon observed with Alloy 617. Since the diffusion of solute/solvent elements is a thermally-activated process, and DSA is known to be influenced by both temperature and strain rate, extensive efforts have been made to determine the magnitude of the activation energy ($Q$) necessary for the initiation of
serrations. An average Q value of 62 KJ/mole was estimated based on the analyses of the tensile data obtained at different temperatures and strain rates. Even though literature data on Q are not available for Alloy 617, the average Q value estimated from the present study matches well with those obtained by recent MPL investigators on other austenitic alloys. As to the effect of temperature and strain rate on the work-hardening index (n), its value was gradually enhanced at 500 and 600°C at lower strain rates (10^{-3} and 10^{-4} sec^{-1}).

A unified mechanism of YSA phenomenon is yet to be established. While numerous theories have been proposed, none of them has been accepted beyond a doubt. However, the analyses of the TEM micrographs of a specimen tested under tensile loading at 900°C exhibited a combination of precipitates, pinned dislocations and pile-up of dislocations in the vicinity of its grain boundaries. Fine subgrains were also formed due to the accumulation of dislocations, which could account for the anomalous increase in YS of Alloy 617, when tested in tension within a temperature range of 800 to 900°C. Both TEM and XRD data confirmed the formation of $M_{23}C_6$ type of precipitates at these temperatures.

Consistent with the observation made by other investigators, the magnitude of $K_Q$ was appreciably reduced at an elevated temperature. It should be noted that, for practical applications, $K_Q$ can be taken as $K_{IC}$. As anticipated, the plots of $\log\left(\frac{da}{dN}\right)$ versus $\log(\Delta K)$ exhibited three stages at all tested temperatures. The second stage of these plots was characterized by a linear relationship, satisfying the Paris equation. The number of cycles (N) necessary for comparable crack growth of Alloy 617 was gradually reduced with increasing temperature. However, a maximum crack growth rate was observed at
100°C, which was also accompanied by an appreciable reduction in $\Delta K_{(final)}$. At 200°C, this alloy, once again, showed a significant drop in $\Delta K_{(final)}$ value corresponding to the lowest CGR, possibly due to the blunting of the crack-tip. As to the cracking susceptibility of Alloy 617 in a 100°C acidic solution, a synergistic effect of higher wedge-load and longer exposure time on average CGR was observed.

Fractographic evaluations of the tested tensile specimens revealed combined intergranular brittle and ductile failures at temperatures up to 300°C. Between 400 and 600°C, Alloy 617 exhibited ductile failures characterized by dimples. However, predominantly brittle failures were seen with specimens tested between 700 and 1000°C. The variation in strain rate did not change the morphology of the tested tensile specimens. As anticipated, the CT specimens tested under cyclic loading showed striations and dimples indicating brittle and ductile failures, respectively. Finally, the DCB specimens exhibited three regions showing striations, cleavage and dimples resulting from fatigue pre-cracking, SCC and fast fracture, respectively.
CHAPTER 6

SUMMARY AND CONCLUSIONS

The tensile properties, toughness and crack-growth-rate (CGR) of Alloy 617 have been evaluated as functions of different metallurgical and mechanical variables using different techniques. The microstructural and fractographic evaluations of the tested materials and specimens have been performed by optical microscopy and scanning electron microscopy, respectively. Transmission electron microscopy (TEM) has been employed to analyze the tensile data for development of a mechanistic understanding of plastic deformation as functions of temperature and strain rate. The key results and significant conclusions derived from this investigation are given below.

- Austenitic grains with annealing twins were observed in optical micrographs of the thermally-treated materials.

- Reduced failure strain ($e_f$) and initiation of serrations were noted in the s-e diagrams at 100°C, suggesting the occurrence of dynamic strain aging (DSA).

- DSA is known to be the result of diffusion of solute-solvent elements into the metal matrix, causing impaired dislocation motion due to their precipitation, thus, leading to reduced plastic flow. The results indicate that the dislocation density ($\rho$), determined from the TEM micrographs, was maximum at 100°C at which the $e_f$ value was also minimum.
• The activation energy (Q) for the onset of serrations was approximately 62 K_kjole, which is comparable to those of Alloy 22 and Alloy 276, that were also recently investigated.

• The work-hardening index (n), which is a measure of plasticity, was enhanced at 500 and 600°C at strain rates of 10^{-3} and 10^{-4} sec^{-1}. However, the magnitude of n was not appreciably affected by the variation in temperature (ambient-600°C) at a strain rate of 10^{-3} sec^{-1}.

• Enhanced yield strength at 800 and 900°C was associated with the occurrence of yield strength anomaly (YSA) phenomenon, which could be attributed to the combined effect of precipitates (M_{23}C_{6}), pinned dislocations, dislocation pile-ups and formation of subgrains, as verified by TEM and XRD.

• CT specimens with 1-inch thickness could not be tested for K_{1C} evaluation due to a prohibitively long pre-cracking time. The average K_Q value, using thinner CT specimens, was appreciably reduced due to a change in temperature from ambient to 100°C. Conventionally, the magnitude of K_Q can be taken as K_{1C}.

• The number of cycles needed to achieve a comparable crack extension was gradually reduced with increasing temperature. Minimum values of \( \Delta K_{(final)} \) and CGR were observed at 200°C, possibly due to crack-tip blunting.

• A combination of greater wedge-load and longer duration resulted in enhanced cracking susceptibility of Alloy 617 in the 100°C solution in terms of average CGR. At lower wedge-load, the DCB specimen did not exhibit any crack extension following shorter exposure period.
• Depending on the testing temperature, the tensile specimens showed two major types of failure morphologies including intergranular brittle and ductile failures, irrespective of the applied strain rate.

• The CT specimens used in $K_{1C}$ and $\frac{da}{dN}$ experiments exhibited striations and dimpled microstructures resulting from cyclic loading and ductile tearing, respectively.

• Three types of failure modes were seen on the fractured surface of the DCB specimens used in SCC testing. These failures correspond to cyclic loading used in pre-cracking (striations), SCC (cleavage) and fast fracture (dimples).
CHAPTER 7

SUGGESTED FUTURE WORK

Stress intensity factor evaluation using CT specimens with a thickness greater than 0.25-inch could not be accomplished due to prohibitively long durations needed to pre-crack them under cyclic loading. This difficulty resulted from a limiting loading frequency of 1 Hz associated with the existing Instron testing equipment. It is, however, anticipated that such task could be accomplished in future allowing cyclic loading of the CT specimen for extended period. The unusually longer time needed to pre-crack the thicker CT specimens simply implies that this material should possess high toughness by virtue of its excellent ductility.
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 where ever applicable)

A1.1 Stress-Strain Diagrams at Room Temperature

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

Sample 1

Sample 2
A1.3 Stress-Strain Diagrams at 200°C

Sample 3

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 3

Sample 1
Sample 2

Sample 3
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

121
A1.8 Stress-Strain Diagrams at 700°C

Sample 1

Sample 2
A1.9 Stress-Strain Diagrams at 800°C
Sample 2

Sample 3
A1.10 Stress-Strain Diagrams at 900°C

Sample 1

Sample 2
A1.11 Stress-Strain Diagrams at 1000°C
Sample 2

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

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Sample #</th>
<th>YS (ksi)</th>
<th>UTS (ksi)</th>
<th>%E1</th>
<th>%RA</th>
</tr>
</thead>
<tbody>
<tr>
<td>RT</td>
<td>Sample 1</td>
<td>54</td>
<td>124</td>
<td>78.68</td>
<td>61.37</td>
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<td></td>
<td>Sample 2</td>
<td>53</td>
<td>123</td>
<td>78.53</td>
<td>62.01</td>
</tr>
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<td>Sample 3</td>
<td>54</td>
<td>125</td>
<td>77.84</td>
<td>62.55</td>
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<tr>
<td>100</td>
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<td>44</td>
<td>111</td>
<td>73.40</td>
<td>55.64</td>
</tr>
<tr>
<td></td>
<td>Sample 2</td>
<td>45</td>
<td>113</td>
<td>75.18</td>
<td>58.80</td>
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<tr>
<td></td>
<td>Sample 3</td>
<td>45</td>
<td>113</td>
<td>74.63</td>
<td>56.20</td>
</tr>
<tr>
<td>200</td>
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<td>42</td>
<td>111</td>
<td>78.74</td>
<td>65.23</td>
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<td>Sample 2</td>
<td>41</td>
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<td>78.05</td>
<td>55.48</td>
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<td></td>
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<td>40</td>
<td>109</td>
<td>78.44</td>
<td>59.82</td>
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<tr>
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<td>Sample 1</td>
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<td>109</td>
<td>76.29</td>
<td>54.24</td>
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<tr>
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<td>Sample 2</td>
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<td>55.73</td>
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<tr>
<td></td>
<td>Sample 3</td>
<td>38</td>
<td>109</td>
<td>78.34</td>
<td>58.62</td>
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<tr>
<td>400</td>
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<td>37</td>
<td>106</td>
<td>81.32</td>
<td>54.55</td>
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<tr>
<td></td>
<td>Sample 2</td>
<td>37</td>
<td>105</td>
<td>78.35</td>
<td>56.20</td>
</tr>
<tr>
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<td>Sample 3</td>
<td>37</td>
<td>106</td>
<td>80.02</td>
<td>61.46</td>
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<tr>
<td>500</td>
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<td>103</td>
<td>76.35</td>
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<td>600</td>
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<td>76.73</td>
<td>55.50</td>
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<td>Sample 2</td>
<td>32</td>
<td>100</td>
<td>80.99</td>
<td>45.69</td>
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<td>99</td>
<td>79.43</td>
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<tr>
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<td>Sample 3</td>
<td>32</td>
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<td>87.64</td>
<td>47.99</td>
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<tr>
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<td>72.59</td>
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<td>33</td>
<td>56</td>
<td>100.12</td>
<td>73.46</td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td>35</td>
<td>58</td>
<td>98.26</td>
<td>73.79</td>
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<tr>
<td>900</td>
<td>Sample 1</td>
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<td>35</td>
<td>113.60</td>
<td>77.43</td>
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<td>Sample 2</td>
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<td>35</td>
<td>115.11</td>
<td>79.64</td>
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<td>Sample 3</td>
<td>35</td>
<td>36</td>
<td>73.14</td>
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<td>1000</td>
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<td>19</td>
<td>19</td>
<td>109.57</td>
<td>72.07</td>
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<tr>
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<td>Sample 2</td>
<td>19</td>
<td>19</td>
<td>104.09</td>
<td>65.79</td>
</tr>
<tr>
<td></td>
<td>Sample 3</td>
<td>20</td>
<td>21</td>
<td>51.02</td>
<td>67.63</td>
</tr>
</tbody>
</table>
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

[Graph showing stress-strain relationship]

Sample 1
A2.2 Stress-Strain Diagrams at 500°C

Sample 2

Sample 1
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 wherever applicable)

A3.1 Stress-Strain Diagrams at 100°C

![Stress-Strain Diagram](image)

Sample 1
A3.2 Stress-Strain Diagrams at 500°C
A3.3 Stress-Strain Diagrams at 600°C

Sample 1

Sample 2
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

138
APPENDIX B

RESULTS OF $K_{IC}$ TESTING

Room Temperature

![Graph for Room Temperature](image1)

100°C

![Graph for 100°C](image2)
APPENDIX C

RESULTS OF CGR TESTING

Ambient Temperature Set 1:

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

Ambient Temperature Set2:

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

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
**Alloy 617**

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

150°C Set 2:

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

200°C Set 1:

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

200°C Set 2:

CGR Data at 200°C


\[ \log \left( \frac{da}{dN} \right) \text{ vs. } \log(\Delta K) \text{, Linear Region} \]
APPENDIX D

SCANNING ELECTRON MICROGRAPHS 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)
TRANSMISSION ELECTRON MICROGRAPHS

C1 TEM Micrographs of Specimens Tested at Room Temperature

35000X

35000X

C2 TEM Micrographs of Specimen Tested at 100°C

97000X

97000X
C3 TEM Micrographs of Specimen Tested at 200°C

C4 TEM Micrographs of Specimen Tested at 200°C
C5 TEM Micrographs of Specimen Tested at 900°C

22500X

115000X
APPENDIX F

UNCERTAINTY ANALYSES OF EXPERIMENTAL RESULTS

Physical parameters such as length, time, force, stress etc. are directly measured in any experiment. The instruments used to determine these parameters may vary in accuracy every time they are being used. Also, such parameters that are out of the control of the researcher might cause undesired variations in these measurements. Efforts were made to replicate all experiments to determine an average value of each parameter. However, the undesired variations might have caused deviations in the derived results. These variations caused by the machines used in experiments can be reduced by calibrating them on a timely basis. However the variations caused due to human errors and environmental factors are unavoidable. 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.

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

Equation A1

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

D.1 Uncertainty Calculation in Instron Results

The results generated from the Instron testing equipment are stress (\( \sigma \)), percentage elongation (\%El), and percentage reduction in area (\%RA). The stress is based on the load (\( P \)) and the initial cross-sectional area (\( A_i \)) of the tested specimen. The \%El is based on the change in length (\( \Delta l \)) during the testing and the \%RA is based on the initial and final cross-sectional areas (\( A_i \) and \( A_f \)). The magnitude of \( P \) was obtained from the load-cell of the 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.

\[
\sigma = \frac{P}{A}\quad \text{Equation A2}
\]

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

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

\[
U_{Ai} = (U_{Di})^2
\]

Uncertainty in load-cell = ± 0.03% lb

Uncertainty in caliper = ± 0.001 inch.

Sample calculation:

For yield stress (YS) = 53.78 ksi

The measured load (P) = 2661.14 lbf

Uncertainty in load (U_P) = 6321 \times 0.0003

= ± 0.79834

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

Initial Diameter (\(D_i\)) = 0.2510 inch.

Uncertainty in diameter (\(U_{Di}\)) = ± 0.001 inch.

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

\[
= 0.0494 \text{ inch}^2
\]

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

\[ = 0.394 \times 0.001 \]

\[ = \pm 0.000394 \]

Uncertainty in stress, \( U_{\sigma} = \left( \left( \frac{\partial \sigma}{\partial P} \cdot U_P \right)^2 + \left( \frac{\partial \sigma}{\partial A_i} \cdot U_{A_i} \right)^2 \right)^{\frac{1}{2}} \)

\[ = 20.242 \]

\[ = -1090470.258 \]

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

\[ U_{\sigma} = \left( (20.242 \cdot 0.79834)^2 + (1090470.258 \cdot 0.000394)^2 \right)^{\frac{1}{2}} \]

\[ = 429.94 \text{ psi} = \pm 0.42 \text{ ksi} \]

One example of the use of the uncertainty analysis is shown in this section. This can be implemented to all experimental results discussed in this dissertation.
D.1.2 Calculation of Uncertainty in Percentage Elongation ($U_{\%El}$)

Sample calculation:

Change in length ($\Delta l$) = 0.758 inch.

Gage length ($l$) = 1 inch.

$$\%El = \frac{\Delta l}{l} \cdot 100$$

Uncertainty in $\Delta l$ ($U_{\Delta l}$) = ± 0.001

Uncertainty in $\%El$ ($U_{\%El}$),

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

Equation A4

$$\frac{d^2\%El}{d\Delta l} = 100$$

$$= 100$$

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

$$U_{\%El} = \left[ (100 \cdot 0.001)^2 \right]^{1/2}$$

$$U_{\%El} = ± 0.1$$

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.

D.1.3 Calculation of Uncertainty in Percentage Reduction in Area ($U_{\%RA}$)

Sample calculation:

$$\%RA = 61.37\%$$

Uncertainty in initial cross-sectional area ($U_{Al}$) for the smooth specimen:

Initial Diameter ($D_i$) = 0.2510 inch.
Uncertainty in initial diameter, 

\[(U_{D_i}) = \pm 0.001 \text{ in}\]

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

\[= 0.0494 \text{ inch}^2\]

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

\[= 0.394\]

Uncertainty in initial cross-sectional area, 

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

\[= 0.394 \times 0.001\]

\[= \pm 0.000394\]

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

Final Diameter \((D_f) = 0.1560 \text{ inch}\).

Uncertainty in final diameter \((U_{D_f})\),

\[= \pm 0.001 \text{ inch}\].

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

\[= 0.01911 \text{ inch}^2\]

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

\[= 0.2450\]
Uncertainty in final cross-sectional area,

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

\[= 0.2450 \times 0.001 \]
\[= 0.0002450\]

Uncertainty in \(\%RA\),

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

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

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

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

\[= 783.08\]

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

\[= -2024.29\]

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

\[
U_{\%RA} = \left[ (783.08 \times 0.000394)^2 + (-2024.29 \times 0.0002450)^2 \right]^{1/2}
\]

\[= 0.584\]

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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Graduate College
University of Nevada, Las Vegas

Vikram Marthandam

Address:
4280 Escondido Street, Apt#214
Las Vegas, NV 89119

Degrees:
Bachelor of Engineering, Mechanical Engineering, 2002
University of Madras, India

Master of Science, Mechanical Engineering, 2004
University of Nevada, Las Vegas, USA

Special Honors and Awards:
Awarded graduate research assistantship at UNLV to pursue M.S. and Ph.D. in Mechanical Engineering

President, Material Advantage, UNLV Student Chapter, TMS, 2007-Present

Secretary, American Nuclear Society, UNLV Student Chapter, 2005-2006

Selected Publications:


Dissertation Title: Tensile Deformation, Toughness and Crack Propagation Studies of Alloy 617
Dissertation Examination Committee:
Chairperson, Dr. Ajit K. Roy, Ph.D.
Committee Member, Dr. Anthony E. Hechanova, Ph.D.
Committee Member, Dr. Brendan J. O’Toole, Ph.D.
Committee Member, Dr. Daniel P. Cook, Ph.D.
Graduate College Representative, Dr. Edward S. Neumann, Ph.D.