Identifying and Understanding Environment-Induced Crack Propagation Behavior in Solid Strengthened Ni-Based Superalloys

Reactor Concepts RD&D

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ABSTRACT

In this project, four major tasks have been conducted to identify and understand the environment-induced crack propagation behavior of two solid solution-strengthened Ni-based superalloys, INCONEL 617® and HAYNES 230®, both of which are major candidate and back-up material for intermediate heat exchanger (IHX) in NGNP (Next Generation Nuclear Plant) program.

In Task 1, the fatigue crack propagation (FCP) as well as sustained loading crack growth (SLCG) behavior of INCONEL 617 and HAYNES 230 were studied at elevated temperatures ranging from 600-800°C in laboratory air under constant stress-intensity-factor (K) condition. The crack propagation tests were conducted using a baseline cyclic triangular waveform with a frequency of 1/3 Hz. Various hold times were imposed at the maximum load of a fatigue cycle to study the hold time effect. The results show that a linear elastic fracture mechanics (LEFM) parameter, stress intensity factor (K), is sufficient to describe the FCP and SLCG behavior at the testing temperatures. The results showed that both INCONEL 617 and HAYNES 230 exhibited the time-dependent FCP, steady SLCG behavior and existence of a damage zone ahead of crack tip. A thermodynamic equation was adapted to correlate the SLCG rates to determine thermal activation energy. The fracture modes associated with crack propagation behavior were discussed, and the mechanism of time-dependent FCP as well as SLCG was identified. Compared to INCONEL 617, the lower crack propagation rates of HAYNES 230 under time-dependent condition were ascribed to different fracture mode and presence of numerous W-rich M₆C-type and Cr-rich M₇₃C₆-type carbides. Toward the end, a phenomenological model was employed to correlate the FCP rates at cycle/ time-dependent FCP domain.

In Task 2, a nano-indentation technique has been conducted to understand the cracking mechanism under cycle-dependent FCP and SLCG condition. TEM samples of crack tip have been fabricated using FIB (focus ion beam) technique. TEM analysis and examination have been conducted to explore the key crack propagation mechanism under different conditions. The results show that oxygen is a major detrimental element to cause intergranular cracking of specimen under sustained loading cracking condition. TEM BF (bright field) and EELS (electron energy loss spectra) mapping have been performed.

In Task 3, effects of load ratio, R-ratio, on FCP behavior INCONEL 617 and HAYNES 230 were studied simultaneously in laboratory air using a constant stress intensity factor (K)-controlled mode with different load ratios (R-ratio) at 700°C. The FCP tests were performed in both cycle and time-dependent FCP domains to examine the effect of R-ratio on the FCP rate, da/dn. For cycle-dependent FCP test, a 1-second sinusoidal fatigue was applied for the compact tension (CT) specimens to measure their FCP rates. For time-dependent FCP test, a 3-second sinusoidal fatigue with a hold time of 300 seconds at maximum load was applied. Both cycle/time-dependent FCP behaviors were characterized and analyzed. The results showed that increasing R-ratio would introduce the fatigue incubation and decrease the FCP rates at cycle-dependent FCP tests. On the contrary, fatigue incubation was not observed at time-dependent FCP tests for both INCONEL 617 and HAYNES 230 at each tested R-ratio, suggesting that association of maximum load (K_max) with crack tip open displacement (CTOD) and environmental factor governed the FCP process. Also, for time-dependent FCP, HAYNES 230 showed lower FCP rates than INCONEL 617 regardless of R-ratio. However, for cycle-
dependent FCP, HAYNES 230 showed the lower FCP rates only at high $R$-ratios. Fracture surface of specimens were examined using SEM to investigate the cracking mechanism under cycle/time-dependent FCP condition with various $R$-ratios.

In Task 4, FCP tests have been conducted on INCONEL 617 and HAYNES 230 obtained from Idaho National Laboratory (INL) and vendors at 700°C at cyclic and hold time fatigue and constant $K_{\text{max}}$-controlling. In this task, INCONEL 617 from INL and Special Metal Co. have slight difference in carbon content, and HAYNES from INL and Haynes International Inc. have same carbon content but slight difference in grain size. Microstructure evaluation has been conducted. FCP tests of HAYNES 230 have been completed, and both materials from INL and vendor displayed identical FCP rates at cycle/time dependent condition. For INCLONE 617 from INL and vendor, cycle-dependent FCP test has been completed and both materials displayed identical cycle-dependent FCP rates. However, for hold-time FCP tests, creeping during FCP test have occurred and resulted in crack growth retardation and abnormal FCP behavior. The reason for creeping during FCP may be from lower strength for these materials.

In summary, both INCONEL 617 and HAYNES 230 are susceptible to, environmental-induced cracking, i.e. SAGBOE-induced cracking at elevated temperature. However HAYNES 230 appears to display higher cracking resistance than INCONEL 617. When both alloys are utilized in highly oxidizing environment such as NGNP system, the environment-induced degradation should be considered.

@ INCONEL and HAYNES are trade mark of Special Metal Co. and Haynes International Inc. respectively.
# TABLE OF CONTENTS

COVER PAGE ............................................................................................................. 1  
ABSTRACT .................................................................................................................. 2  
TABLE OF CONTENTS ............................................................................................. 4  
LIST OF FIGURES ..................................................................................................... 6  
LIST OF TABLES ....................................................................................................... 9  
1. INTRODUCTION .................................................................................................... 10  
2. MATERIALS AND EXPERIMENTS ....................................................................... 12  
   2.1 Materials and Specimens .................................................................................. 12  
   2.2 FCP Test .......................................................................................................... 13  
   2.3 Microstructure and Fracture Analysis ............................................................... 14  
      2.3.1 Optical Microstructure .............................................................................. 14  
      2.3.2 Fractography ............................................................................................. 14  
   2.4 Nano-indentation Examination ....................................................................... 14  
   2.5 TEM sample Preparation and TEM Analysis ............................................... 14  
3. RESULTS AND DISCUSSIONS .......................................................................... 22  
   3.1 Results and Discussions of Task 1 ................................................................. 22  
      3.1.1 Microstructure and Tensile properties ...................................................... 22  
      3.1.2 Fatigue Crack Propagation (FCP) .............................................................. 22  
      3.1.3 Sustained loading crack growth (SLCG) .................................................. 23  
      3.1.4 Fractography .............................................................................................. 25  
      3.1.5 Time-dependent FCP of INCONEL 617 and HAYNES 230 ...................... 26  
      3.1.6 Crack Propagation Mechanism ................................................................. 26  
      3.1.7 Modeling Characterization of Time-dependent FCP ................................. 28  
      3.1.8 Summary of Task 1 .................................................................................. 29  
   3.2 Results and Discussions of Task 2 ................................................................ 43  
      3.2.1 Nano-indentation Test of INCONEL 617 ................................................... 43  
      3.2.2 Summary of Nano-indentation ................................................................. 44  
      3.2.3 TEM studying on Crack Tip Area ............................................................. 44  
   3.3 Results and Discussions of Task 3 ................................................................ 50
3.3.1 FCP Test at 1-second Fatigue ................................................................. 50
3.3.2 FCP Test at 300-second Hold-time Fatigue .............................................. 50
3.3.3 Fractography .......................................................................................... 50
3.3.4 Model of Load Ratio Effect .................................................................. 51
3.3.5 Summary of Task 3 .............................................................................. 54
3.4 Results and Discussions of Task 4 .............................................................. 63
  3.4.1 Microstructure .................................................................................. 63
  3.4.2 FCP Test ................................................................................................ 63
  3.4.3 Summary ............................................................................................ 64
4. Conclusions ........................................................................................................ 64
5. RECOMMENDATIONS .................................................................................... 64
6. List of Publication, Thesis and Presentation .................................................... 64
7. ACKNOWLEDGEMENT ................................................................................. 64
  8. , for helpful discussions.
  8.
PARTICIPANTS .................................................................................................. 64
9. REFERENCES .................................................................................................. 64
10. COST DATA .................................................................................................. 75
## LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figures</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 1</td>
<td>Geometry of a compact-tension specimen (mm)</td>
<td>16</td>
</tr>
<tr>
<td>Figure 2</td>
<td>Instron Model 8862 and Experimental Setting-up</td>
<td>17</td>
</tr>
<tr>
<td>Figure 3</td>
<td>Schematic illustration of FCP testing procedure</td>
<td>17</td>
</tr>
<tr>
<td>Figure 4</td>
<td>Schematic illustration of sustained loading crack growth (SLCG) testing procedure</td>
<td>18</td>
</tr>
<tr>
<td>Figure 5</td>
<td>Schematic illustration of FCP testing procedure. (a) FCP test at 1-second fatigue</td>
<td>19</td>
</tr>
<tr>
<td>Figure 6</td>
<td>A Hysitron TI 950 TriboIndente System for Nano-indentation</td>
<td>20</td>
</tr>
<tr>
<td>Figure 7</td>
<td>Schematic of TEM sample location at front of crack tip and FEI NOVA 200 Focused Ion Beam (FIB) System</td>
<td>21</td>
</tr>
<tr>
<td>Figure 8</td>
<td>A TECNAI-F30 Transmission Electron Microscope (TEM) in UNLV</td>
<td>22</td>
</tr>
<tr>
<td>Figure 9</td>
<td>Microstructures of as-received materials: (a) optical micrograph of INCONEL 617; (b) optical micrograph of HAYNES 230; (c). SEM micrograph of INCONEL 617; (d). SEM micrograph of HAYNES 230</td>
<td>31</td>
</tr>
<tr>
<td>Figure 10</td>
<td>Constant ΔK FCP curves for different hold times tested at 600°C in air: (a) INCONEL 617 and (b) HAYNES 230</td>
<td>32</td>
</tr>
<tr>
<td>Figure 11</td>
<td>FCP rates as a function of period at various temperatures</td>
<td>33</td>
</tr>
<tr>
<td>Figure 12</td>
<td>SLCG of INCONEL 617 and HAYNES 230 in air at constant K equal to 27.75 MPa√m and 700°C</td>
<td>33</td>
</tr>
<tr>
<td>Figure 13</td>
<td>FCP curves of INCONEL 617 and HAYNES 230 at $K_{max} = 27.75$ MPa√m, $R=0.1$, and room temperature after SLCG testing at 700°C</td>
<td>34</td>
</tr>
<tr>
<td>Figure 14</td>
<td>Damage zone size measurement</td>
<td>34</td>
</tr>
<tr>
<td>Figure 15</td>
<td>Incubation vs. temperature (K)</td>
<td>35</td>
</tr>
<tr>
<td>Figure 16</td>
<td>SLCG rate, $da/dt$, vs. $I/T$</td>
<td>35</td>
</tr>
</tbody>
</table>
Figure 17  SEM fracture surface micrographs of INCONEL 617 specimen subjected to FCP tests at 700°C: (a) at 3s; (b) at 3+300s; (c) at 3+1000s………………………………36

Figure 18  SEM fracture surface micrographs of INCONEL 617 specimen subjected to SLCG tests: (a) at 600 °C; (b) at 700 °C; (c) at 800 °C…………………………..37

Figure 19  SEM fracture surface micrographs of HAYNES 230 specimen subjected to FCP tests at 700°C: (a) at 3s; (b) at 3+300s; (c) Close-view of (b)………………..38

Figure 20  SEM fracture surface micrographs of HAYNES 230 specimen subjected to SLCG tests: (a) at 600 °C; (b) at 700 °C; (c) at 800 °C……………………………….39

Figure 21  Static crack growth, $da/dt$, vs. period at $K_{max} =27.75$ MPa$\sqrt{m}$: (a) INCONEL 617 and (b) HAYNES 230……………………………………………………40

Figure 22  Damage zone examination of INCONEL 617: (a) FCP curve at room temperature after SLCG test at 800°C; (b) Corresponding SEM micrograph of fracture surface. Arrow indicates the crack growth direction…………………………..40

Figure 23  Damage zone examination of HAYNES 230: (a) FCP curve at room temperature after SLCG test at 800°C; (b) Corresponding SEM micrograph of fracture surface. Arrow indicates the crack growth direction……………………………41

Figure 24  Normalized FCP rates, $da/dn$, of INCONEL 617 by the time-dependent factor, $\alpha (t,T)$ at $K_{max} =27.75$ MPa$\sqrt{m}$……………………………………………………..41

Figure 25  Characteristic curves to describe FCP behavior of INCONEL 617 and HAYNES 230………………………………………………………………………………..42

Figure 26  An example of nano-indentation specimen; (a) Nano-indentation location; (b) loading vs. displacement during indentation……………………………………..45

Figure 27  Map of hardness, deduced modulus and location ahead of crack tip for INCONEL 617 specimen subjected to SLCG test at 800°C…………………………..45

Figure 28  Map of hardness, deduced modulus and location ahead of crack tip for INCONEL 617 specimen subjected to FCP test at 6s and room temperature…………..46

Figure 29  Preparation of TEM sample ahead of crack tip by FIB, (a) sample location; (b) FIB lift-out technique for cross-section TEM sample……………………………..46
Figure 30 TEM sample prepared by FIB lift-out procedure. (a). Sample was lift out; (b). A TEM sample ready for observation.

Figure 31 TEM images of INCONEL specimen tested at 6s-FCP and room temperature with different magnification along [011].

Figure 32 TEM images of INCONEL specimen subjected to SLCG at 800°C with different magnification along [011].

Figure 33 FETEM mode image and EELS spectrum of specimen tested at SLCG and 800°C. (a). Zero-loss image; (b). O Mapping; (c). O-edge EELS spectrum; (d) Cr-mapping.

Figure 34 Constant-ΔK controlled FCP curves with different R-ratios at 1-second and 700°C in air; (a) INCONEL 617 and (b) HAYNES 230.

Figure 35 FCP rates as a function of R-ratio at Kmax=27.75 MPa√m.

Figure 36 Comparison of fatigue incubation at different R-ratios.

Figure 37 Constant-ΔK controlled FCP curves with different R-ratios at 300-second hold time and 700°C in air; (a) INCONEL 617 and (b) HAYNES 230.

Figure 38 Summary of FCP rates as a function of R-ratio at Kmax=27.75 MPa√m.

Figure 39 Fracture surfaces of INCONEL 617 specimen tested at 700°C, 1s and different R. Note that hollow arrow indicates the FCP direction and solid arrow indicates the border line of different R-ratios. (a) R = 0.05 and 0.1, (b) R = 0.1 and 0.3, (c) R=0.3 and 0.5, and (d) R=0.5 and 0.7.

Figure 40 Fracture surfaces of HAYNES 230 specimen tested at 700°C, 1s and different R. Note that the hollow arrow indicates the FCP direction and solid arrow indicates the border line of different R-ratios. (a) R = 0.05 and 0.1, (b) R = 0.1 and 0.3, (c) R=0.3 and 0.5, and (d) R=0.5 and 0.7.

Figure 41 Fracture surfaces of INCONEL 617 specimen tested at 700°C, 300s hold time and different R-ratios. Note that the hollow arrow indicates the FCP direction. (a) R = 0.05, and (b) R = 0.5.

Figure 42 Fracture surfaces of HAYNES 230 specimen tested at 700°C, 300s hold time and different R-ratios. Note that the hollow arrow indicates the FCP direction. (a) R = 0.05, and (b) R = 0.5.
Figure 43  \(\frac{da}{dn}\) vs. \(\Delta K_{app}\)…………………………………………………………………...

Figure 44  \(\frac{da}{dt}\) vs. \(R\)………………………………………………………………………..

Figure 45  Microstructure of INCONEL 617 INL; (a) Optical Microstructure; (b) SEM BSD Microstructure…………………………………………………………………………65

Figure 46  Microstructure of INCONEL 617 SM; (a) Optical Microstructure; (b) SEM BSD Microstructure……………………………………………………………………66

Figure 47  Microstructure of HAYNES 230 H; (a) Optical Microstructure; (b) SEM BSD Microstructure……………………………………………………………………66

Figure 48  Microstructure of HAYNES 230 INL; (a) Optical Microstructure; (b) SEM BSD Microstructure…………………………………………………………………..67

Figure 49  Constant \(\Delta K\)-controlled FCP curves of Alloy 230-HA tested at different hold time and 700°C……………………………………………………………………67

Figure 50  Constant \(\Delta K\)-controlled FCP curves of Alloy 230-HA tested at different hold time and 700°C…………………………………………………………………68

Figure 51  FCP rate of HAYNES 230 as a function of period at 700 °C…………………69

Figure 52  Crack length vs. cycle number of alloy 617-SM at 700°C…………………..70

LIST OF TABLES

<table>
<thead>
<tr>
<th>Tables</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 1</td>
<td>Chemical Composition of Experimental Alloys Used in Different Tasks</td>
<td>s</td>
</tr>
</tbody>
</table>
1. INTRODUCTION

Solid solution strengthened Ni-based superalloys, INCONEL 617 and HAYNES 230, have been extensively used as heat exchange tubing materials in the power generation industry and petrochemical field, due to their exceptional combination of high temperature strength and oxidation resistance, and excellent resistance to a wide range of corrosive environments. Presently, INCONEL 617 has been considered as a candidate for intermediate heat exchanger (IHX) material in the VHTR (very high temperature reactor) of the NGNP (next generation nuclear plant) program conducted by the US DOE. The IHX requires the candidate material to operate at temperature ranging from 650-850°C, and to be exposed to thermal static/cyclic (creep and fatigue) stress impact. In NGNP program, the high temperature design methodology (HTDM) considering environment-induced crack initiation and crack growth based on fracture mechanics will be employed to design and assess the system life expectancy [1-9]. Although INCONEL 617 is a very mature high temperature alloy, the premature failure procedure under condition of complicated thermal stress impact and aggressive environment interaction above 800 °C is far from understanding. As backup candidates, HAYENS 230 have not been characterized in aspects of fatigue crack growth, time-dependent fatigue crack growth, and environment-induced cracking [3, 8-9].

As well documented [10-12], Ni-based superalloys such as INCONEL 718 and 783 usually show time-dependent fatigue crack propagation (FCP) behavior at intermediate temperature (550-650 °C) in air, when the fatigue frequency is decreased to a certain value or a sufficient holding period is imposed on the cyclic loading peak. The intermediate temperature allows the stress status of crack tip to be defined by a parameter of linear fracture mechanics, stress intensity factor (K). The hold time fatigue is also called creep-fatigue conventionally. At such time-dependent stage, FCP process is associated with the accumulation of mechanical and environmental degradation [13-14]. Experimental results demonstrate that the SAGBOE (stress accelerated grain boundary oxygen embrittlement) plays a predominade role for determining the time-dependent FCP rate in air. The mechanistic parameter only plays a negligible role for the time-dependent FCP process. The conventional mechanistic creep crack propagation (CFCP) mode, which simply superposes the cyclic (fatigue) and static (creep) components of crack increment as the total crack growth in one cycle, is unable to represent the time-dependent FCP rate [15-21]. The conventional term creep-fatigue crack propagation seems misleading for the high strength Ni-based precipitation strengthened superalloys such as INCOENL 718, 783 or
Wasploy. A full time-dependent FCP model considering oxygen grain boundary diffusion kinetics, oxygen thermal activation energy and linear fracture mechanics has been experimentally established to characterize the SAGBOE-induced time-dependent FCP of INCONEL 718 and 783 [10-12]. According to the SAGBOE induced full time-dependent FCP theory, if the FCP rate \((da/dn)\) at the full time dependent condition can be correlated with the sustained loading crack growth (SLCG) rate \((da/dt)\), the environmental effect, or SAGBO mechanism, is thought to be responsible for the crack propagation process. The SCLG rate, \(da/dt\), is able to characterized by a thermal activation Arrhenius equation, wherein the thermal activation energy is a characteristic value to represent the involvement of oxygen. If the stress status of crack tip no longer can be defined using the linear fracture mechanics owing to high temperature, the crack may grow under condition of interaction of environment and creep damage.

The accelerated crack growth under hold time fatigue condition is a major concern to employ HTDM (high temperature design methodology) for NGNP program [6, 9]. Identification and understanding of environment or SABGO-induced cracking at elevated temperature will ensure the reliability and service life expectance for IHX system. So far, most work only focuses on the creep-fatigue life cycle testing and thermal exposure testing for INCONEL 617, and HAYNES 230 [6, 9]. The data on crack growth behaviors of those alloys at hold time fatigue and static loading is scanty. The proposed work is expected to explore the insights into the fundamental damage mechanism under interaction of static-fatigue oxidation and environment condition as well as distinguish the environment and mechanistic effects for the cracking procedure. Furthermore, understanding the crack growth process of those solid-strengthening alloys at elevated temperature in air will strongly assist the next phase studies on the crack growth process of these candidate alloys in helium environment, which will be considered as the coolant media in the IHX of NGNP. In addition, effects of minor element such as carbon on FCP, in particular, hold time fatigue of Alloys 617 and 230 will also be performed for quality control of material fabrication.

Therefore, the project is aimed at studying the crack growth procedure of Alloys 617 and 230 at temperature of 600-800°C in air under static/cyclic loading condition to identify the environmental and mechanical damage components, and to understand in-depth the failure mechanism. The objectives of the project are listed as follows:

- To identify the FCP mode of the proposed materials, INCONEL 617 and HAYNES 230 under cyclic and hold time fatigue conditions at temperature of 600-800 °C in air.

- To measure the SLCG rate, \(da/dt\), at temperature of 600-800 °C in air.

- To correlate the SLCG process with a rate-controlled thermal activation equation to calculate the oxygen thermal activation energy.

- To correlate the full time-dependent FCP rate, \(da/dn\), with the SLCG rate, \(da/dt\), identifying the cracking mechanism.

- To analyze the structure of fracture surface and crack tip area using SEM, TEM and FIB to understand the cracking mechanism.
To study the effects of carbon addition on FCP behavior, in particular, time-dependent FCP behavior.

To study the effects of load ratio, $R$-ratio, on FCP behavior under cycle/time-dependent condition to simulate the operation condition.

To accomplish the proposed objectives, the project consisted of four tasks as follows:

(1). **Task 1**: FCP and sustained loading measurement, and time-dependent FCP model development.

(2). **Task 2**: Microstructure and nano-mechanical characterization of crack tip.

(3). **Task 3**: Effects of load ratio, $R$-ratio, on FCP behavior.

(4). **Task 4**: Effects of carbon content on FCP behavior.

### 2. MATERIALS AND EXPERIMENTS

#### 2.1 Materials and Specimens

Four heats of the experimental alloys, INCONEL 617 and HAYNES 230, were procured and obtained from the Huntington Alloys Corporation, West Virginia, Haynes International, Inc., Indiana, and Idaho National Laboratory, Idaho Falls, ID, USA, respectively. These materials will be used in proposed tasks accordingly. All alloy bars were subjected to the standard solid solution treatment at a temperature of 1175°C for INCONEL 617 and 1237°C for HAYNES 230 followed by rapid cooling. Such thermal treatment is known to produce isotropic austenitic grains with annealed twins in Ni-based superalloys [7-8, 20, 22]. The chemical composition of these alloys is given in Table 1.

The CT specimens used in the crack-propagation-rate (CPR) test were fabricated from the as-received bar materials in such a way that the crack plane was perpendicular to the short-transverse direction. The American Society for Testing and Materials (ASTM) Designation E 647-2000 standard was followed to prepare the CT specimens. The dimensions of a CT specimen are illustrated in Figure 1. Prior to the CPR test, the CT specimens were precracked up to a length of 2.00 mm in an Instron servo-controlled hydraulic test machine at room temperature at a load ratio ($R = \frac{K_{\text{min}}}{K_{\text{max}}}$), a frequency, and a maximum stress intensity factor of 0.1, 1 Hz, and 22 MPa/√m respectively. A direct-current-potential-drop (DCPD) technique was used to continuously monitor the crack length of the CT specimens during both precrack and CPR tests. The DCPD method involved passing a constant current of due to the extension of crack length using Johnson’s equation [23], given by **Equation 1**.

**Equation 1**

where $V_o$ and $a_o$ are the initial crack mouth potential and crack length, $V_i$ and $a_i$ are the instantaneous crack mouth potential and crack length, $y$ is the half of the distance between the
two points for which the crack mouth potential is measured, and \(W\) is the specimen width. The stress intensity factor, \(K\), was obtained using Tada’s equation [24]:

\[
K = \frac{P}{B \sqrt{W}} \left[ \left( \frac{2 + a/W}{1 - a/W} \right)^{3/2} \right] \left[ 0.886 + 4.64(a/W) - 13.32(a/W)^2 + 14.72(a/W)^3 - 5.6(a/W)^4 \right] \tag{Equation 2}
\]

where \(P\) is applied load, \(B\) is specimen thickness, \(a\) is crack size for a CT specimen, and \(W\) is specimen width. Also, \(\Delta K = K_{\text{max}} - K_{\text{min}}\), and load ratio, \(R = K_{\text{max}}/K_{\text{min}}\), where \(K_{\text{max}}\) and \(K_{\text{min}}\) are the maximum and minimum stress intensity factor respectively. All tests of crack propagation were conducted in an Instron Model 8862 system in which a conduct heating furnace was attached to provide high temperature. Figure 2 shows the pictures of experimental setting-up.

### 2.2 FCP Test

For Task 1, FCP and SLCG tests were performed at temperatures of 600, 700 and 800\(^\circ\)C, respectively. A ceramic-lined split furnace, attached to an Instron test machine, was used to heat the CT specimens to the desired testing temperatures. Figure 3 illustrates the FCP testing procedure. At testing temperature, a precracked CT specimen was firstly subjected to a triangular 3-second fatigue allowing a crack length growth of minimum 2.00 mm so that a steady FCP rate could be determined. Following the 3s fatigue test, a series of hold time FCP tests were performed by superimposing various hold times of 60, 120, 300, 600 and 1000 seconds at maximum load of the triangular waveform. Crack extensions ranging from 1.50 to 2.00 mm between each hold time was selected to monitor the instantaneous crack length as a function of the loading cycle at each testing temperature to measure FCP rate, \(da/dn\). During FCP testing, the maximum stress intensity factor \((K_{\text{max}})\) and load ratio \((R)\) were kept at 27.75 MPa\(\sqrt{m}\) and 0.1 respectively so that fatigue crack propagated at a constant \(\Delta K\) with value of 25 MPa\(\sqrt{m}\), which is less than one quarter of fracture toughness of INCONEL 617 and HAYNES 230 at room temperature [7-9, 20, 22, 25].

In addition to triangular and hold time FCP tests, SLCG tests were also conducted. Figure 4 shows the testing procedure, involving loading a precracked specimen at a constant \(K\) with a value of 27.75 MPa\(\sqrt{m}\), equal to the \(K_{\text{max}}\) of the FCP test, for a sufficient period at respective elevated temperatures to measure the SLCG rate, \(da/dt\). After the steady SLCG test, the tested specimen was cooled to room temperature and a FCP test with a load ratio \((R)\) of 0.1, a cyclic period of 6s, and \(K_{\text{max}}\) of 27.75 MPa\(\sqrt{m}\) was subsequently carried on the specimen again to identify and measure the damaged zone caused by the previous SLCG. The damage zone concept and the measurement procedure have been reported elsewhere [10-12]. Mathematically, the sustained loading (or called infinite holding) status can be considered as a specific hold time fatigue with only one cycle, wherein the hold time is sufficiently long. A software program, provided by the Fracture Technology Associates, Bethlehem, PA, USA, was used to continuously monitor cracking and record data, which led to the development of crack length versus number of loading cycles, and \(\Delta K\) controlling.

For Task 3, the FCP tests were conducted at 700\(^\circ\)C in laboratory air. Two types of fatigue waveform, a sinusoidal fatigue cycle with a period of 1.0 second and a trapezoidal fatigue cycle composed of a 3-second baseline sinusoidal cycle and 300-second holding period at maximum...
load, were employed, respectively. It was thought that these two types of fatigue, 1-second sinusoidal and 300-second hold-time fatigue, allowed both materials to display the cycle-dependent and time-dependent FCP behavior, respectively. For all FCP tests, the maximum stress intensity factor \((K_{\text{max}})\) was kept at 27.75 MPa\(\sqrt{\text{m}}\). A series of load ratio, \(R\)-ratio, ranging from 0.05 to 1.0 were chosen during FCP test to examine the effects of \(R\)-ratio on FCP behavior. Schematic illustrations of the FCP testing procedure were presented in Figure 5, showing that, after pre-cracked, a CT specimen was heated to 700°C, and a fatigue cycle, 1-second fatigue or 300-second hold-time fatigue, was introduced with \(R\) equal to 0.05 firstly, allowing the crack to grow to a length of 1.50 to 2.00 mm so that a steady FCP rate could be measured. Given a fatigue load with sinusoidal or trapezoidal waveform and testing temperature at 700°C, the FCP test was performed from one to the next with increase of \(R\)-ratio.

For Task 4, FCP tests for the materials with different carbon and from different heats were conducted at 3-second sinusoidal and 3-second baseline with different hold-time waveform, \(R\) equal 0.1 and 700°C. The testing procedure followed the procedure shown in Figure 3. The effects of carbon and different heats would be evaluated in terms of cycle/time-dependent FCP rates.

After FCP tests, specimens were broken into two halves, and measurement of beach marks associated with various testing conditions was conducted for the crack length and \(K\)-value calibration. The DCDP monitor system in this study has a measurement error of 5-8%.

2.3 Microstructure and Fracture Surface Analysis

2.3.1 Optical Microstructure

Optical microstructures of the as-received materials, INCONEL 617 and HAYNES 230, were examined in a polished and etched condition by using a Leica optical microscope. The etchant used was Kalling’s reagent, which was a mixture of 40 ml of distilled water (H\(_2\)O), 40 ml of hydrochloric acid (HCl), 40 ml of ethanol (CH\(_3\)COOH), and 2 g of cupric chloride (CuCl\(_2\)). For optical microstructure, average grain size and grain morphology would be evaluated [22, 27-28].

2.3.2 Fractography

After crack propagation testing, specimens were broken into two halves to check fracture surface. Fractographic evaluations, showing the morphology of failures and cracking of the tested specimens, were performed by using a JEOL-5610 SEM.

For alloy 230, some fracture surfaces of tested specimens were cleaned if fracture surface were covered by numerous oxides to block the fracture details. The cleaning process involved boiling the specimen for about one hour in a mixture solution with 150g NaOH, 100 g KMnO\(_4\), and 1 L water at 70°C [27-28]. Fracture surface of each tested specimen was observed and analyzed by using a JEOL-5610 scanning electron microscope (SEM) to evaluate the fracture mode.

2.4 Nano-indentation Examination
A Hysitron TI 950 TriboIndenter™ was used to perform the nano-indentation tests on INCONEL 617 and HAYNES samples subjected to SLCG test at 800°C and 6s-FCP test at room temperature, respectively. Nano-indentation tests at room temperature were first performed in a grid around the crack to determine if the hardness and reduced modulus values of this region as a function of x/y coordinates would help identify the cracking mechanism. Two more grids of indents (closer spacing and lower force) were then performed at other crack locations to verify that the area near a crack contains higher hardness values than the areas further away from a crack. Figure 6 show the picture of TI 950 TriboIndenter nano-indentation system.

2.5 TEM sample Preparation and TEM Analysis

After crack growth testing, cracked specimen was used to TEM analysis to investigate the microstructure of crack tip. In this project, after sustained loading crack growth test at 800°C, specimen of INCONEL 617 was FIBed for TEM analysis. It is expected to find the cracking mechanism through carbide and dislocation analysis. The TEM sample was taken at front of crack tip, the sample had with a thickness of 50-80 nm. A FEI Nova 200 FIB (focus ion beam) system, which combines the FIB technology with SEM in a single tool, was used. This system can provide SEM imaging in order to monitor the whole process during FIB milling. A FIB cross-section lift-out technique based on a TEM-wizard program in FIB system was used to obtain the cross-sectioned TEM sample of crack tip area. The preparation of TEM cross-sectioned samples by FIB was conducted in Arizona State University, Tempe, AZ. Figure 7 show the schematic location of TEM sample and a NOVA 200 FIB system.

A TECNAI-G2-F30 transmission electron microscope with a 300 keV field emission gun was used to characterize the FIBed samples. Samples were analyzed using the conventional bright field (BF), selected-area diffraction (SAD), EELS (electron energy loss spectrum) -mapping and high-angle annular dark-field scanning-transmission electron microscopy (HAADF-STEM) mode for defects, diffraction and Z-contrasting imaging, respectively. All TEM images were recorded using a Gatan SC 200 CCD camera with resolution of 2kx2k. The elemental distribution of each sample was also determined using the corresponding X-ray energy dispersive spectrometry (EDX) under the STEM mode. For STEM/EDX mode, the electron probe with a size of 0.2 nm was used to examine the dedicated area of sample. Figure 8 show the picture of TECNAI-F30 TEM system used in this project.

Table 1 Chemical Composition of Experimental Alloys Used in Different Tasks

<table>
<thead>
<tr>
<th>Materials</th>
<th>C</th>
<th>Mn</th>
<th>Fe</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Al</th>
<th>Ti</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
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</thead>
<tbody>
<tr>
<td>617 for Task 1,3</td>
<td>0.06</td>
<td>0.12</td>
<td>0.002</td>
<td>-</td>
<td>22.10</td>
<td>54.80</td>
<td>0.87</td>
<td>0.29</td>
<td>12.17</td>
<td>9.52</td>
<td>0.001</td>
</tr>
<tr>
<td>230 for Task 1,3</td>
<td>0.11</td>
<td>0.50</td>
<td>0.42</td>
<td>0.39</td>
<td>22.01</td>
<td>60.76</td>
<td>0.40</td>
<td>0.29</td>
<td>0.10</td>
<td>1.25</td>
<td>14.06</td>
</tr>
<tr>
<td>617-SM for Task 4</td>
<td>0.09</td>
<td>0.04</td>
<td>1.34</td>
<td>-</td>
<td>22.10</td>
<td>52.16</td>
<td>1.1</td>
<td>0.45</td>
<td>12.50</td>
<td>9.63</td>
<td></td>
</tr>
<tr>
<td>617-INL for Task 4</td>
<td>0.05</td>
<td>0.1</td>
<td>1.6</td>
<td>0.1</td>
<td>22.2</td>
<td>54.10</td>
<td>1.1</td>
<td>0.40</td>
<td>11.60</td>
<td>8.6</td>
<td></td>
</tr>
<tr>
<td>230-INL for Task 4</td>
<td>0.11</td>
<td>0.11</td>
<td>0.42</td>
<td>0.39</td>
<td>21.77</td>
<td>60.76</td>
<td>0.40</td>
<td>0.29</td>
<td>0.10</td>
<td>1.25</td>
<td>14.06</td>
</tr>
</tbody>
</table>
Figure 1 Geometry of a compact-tension specimen (mm)
Figure 2  Intron Model 8862 and Experimental Setting-up

Figure 3 Schematic illustration of FCP testing procedure
Figure 4 Schematic illustration of sustained loading crack growth (SLCG) testing procedure
Figure 5  Schematic illustration of FCP testing procedure. (a). FCP test at 1-second fatigue
(b) FCP test at 300-second hold time fatigue
Figure 6 A Hysitron TI 950 TriboIndente System for Nano-indentation

Figure 7 Schematic of TEM sample location at front of crack tip and FEI NOVA 200 Focused Ion Beam (FIB) System
Figure 8  A TECNAI-F30 Transmission Electron Microscope (TEM) in UNLV
3. RESULTS AND DISCUSSIONS

3.1 Results and Discussions of Task 1

3.1.1 Microstructure and Tensile properties

The microstructures of as-received INCONEL 617 and HAYNES 230 are presented in Figure 9. Optical micrographs of both materials exhibited isotropic microstructure including a few annealed twin structures due to solid-solution treatment and lots of carbides distributed along the inter/intra-granular areas, Figure 9 (a) and (b). The grain size measurement indicated that INCONEL 617 and HAYNES 230 had average grain size of 97 (ASTM No. 4) and 55 microns (ASTM No. 5.5) respectively. Both INCONEL 617 and HAYNES 230 are composed of a face center cubic (fcc) austenite with different types of carbides, which usually forms during thermo-mechanical processing. The major carbide in INCONEL 617 is the Cr-rich M23C6-type nano-scaled carbides. Relative to INCONEL 617, HAYNES 230 has higher carbon content (0.11 wt%) in this study and includes two types of carbides, the primary W-rich M6C-type and the secondary Cr-rich M23C6-type carbides. In general, M6C-type carbide is micron-sized and M23C6-type carbide is nano-sized in solid-solution treated HAYNES 230 [29-31]. The SEM examination of only polished as-received materials can reveal the morphology of M6C-type carbide. Figure 9 (c) and (c) presented the SEM micrographs of polished as-received materials, showing that HAYNES 230 includes numerous micron-sized M6C-type carbides (5-6 microns), which, however, are absent in INCONEL 617. The tensile properties and hardness of both materials at room temperature are given in Table 2, indicating that INCONEL 617 has slightly higher tensile strength and elongation than HAYNES 230 at room temperature.

3.1.2 Fatigue Crack Propagation (FCP)

As examples, the constant \( \Delta K \)-controlled FCP curves of INCONEL 617 and HAYNES 230 tested at 600°C for 3s-cyclic fatigue and hold time fatigue with various holding periods are presented in Figure 10 (a) and (b), respectively. The variations of crack length \( (a) \) with number of fatigue cycles \( (n) \) under all tested conditions exhibit a linear relationship, indicating that the crack propagated at a constant rate under constant \( \Delta K \)-controlled mode. The slope of crack growth curve represents the FCP rate, \( da/dn \). Among the hold times with sufficient period, Fig. 6 indicates that the FCP rates of both INCONEL 617 and HAYNES 230 increase with the hold time periods. The enhancement of FCP rates of INCONEL 617 are not pronounced for the short hold times such as 60, 120 and 300s, whereas HAYNES 230 shows significant increase in FCP rates at those hold time periods. All data of crack growth length vs. fatigue cycle under different temperature and hold time conditions were processed using linear regression analysis to calculate the FCP rates, which were plotted in Figure 11 as a function of fatigue period. Both INCONEL 617 and HAYNES 230 exhibit the time-dependent behavior, i.e. the FCP rate, \( da/dn \), is a function of the hold time length. Such time-dependent FCP behaviors have been also observed in many Ni-based superalloys such as INCONEL 718, 783 and Waspaloy [5-7, 11]. Considering experimental data scattering, the degree of time-dependence at a given temperature increases gradually with the length of hold time, though it may not be significant for INCONEL 617 at short hold time period. Theoretically, a linear relationship between the FCP rate and hold time length can be established when the hold time exceeds a critical value, i.e. FCP becomes fully time-dependent [10-11, 27]. Temperature is an important parameter to affect time-dependent
FCP process. Enhancement of temperature allows the time-dependent FCP to start at a shorter hold time or period, resulting in higher FCP rate. For both INCONEL 617 and HAYNES 230 at 800°C, the time-dependent FCP behavior already started at hold time of 60s. However, for INCONEL 617 at 700°C, the time-dependent FCP behavior has not been observed until the length of hold time was 120s. All data of crack length vs. cycle at different temperatures display the linear relationship, suggesting that the LEFM parameter, $K$, is sufficient to define the stress state of crack tip at the experimental temperatures [5-7, 22]. It should be noted that at the highest old time 300 and 1000s, since crack advancement was too fast to be recorded by the system. Another interesting observation in Figure 11 is that, at 3s cyclic fatigue without holding, INCONEL 617 and HAYNES 230 have comparable FCP rates at temperatures of 600 and 700°C, suggesting that 3s triangular fatigue at those temperatures allows the crack to propagate in cycle-dependent mode, in which the FCP rates are mainly determined by the mechanistic parameter, $\Delta K$. With introduction of hold time, fatigue crack propagates in time-dependent mode, in which the FCP rate becomes a function of $\Delta K$, temperature, and hold time. In summary, the all above experimental results demonstrate that the FCP rates of INCONEL 617 are almost 1-2 magnitudes higher than HAYNES 230 in time-dependent FCP stage, as well as INCONEL 617 starts the time-dependent FCP at shorter hold time than HAYNES 230, suggesting that HAYNES 230 has higher resistance to time-dependent FCP than INCONEL 617.

3.1.3 Sustained loading crack growth (SLCG)

The SLCG rate, $da/dt$, was measured at temperatures of 600, 700 and 800°C following the testing procedure shown in Figure 4. As examples, the SLCG curves for INCONEL 617 and HAYNES 230 at 700°C are shown in Figure 12. For INCONEL 617, initially there is no crack extension for about 30,000 seconds after a precracked specimen received a static load, $K_{max}$. At about 40,000 seconds, the crack started to initiate and grow. It took about 350,000 seconds to reach the steady crack growth. As the sustained load $K_{max}$ was kept constant during crack growth, the crack length has a linear relationship with time. The slope of the linear portion of crack growth curve in Figure 4 represents the SLCG rate, $da/dt$. As seen in Figure 12, the intersection between crack growth line and horizontal line represents the incubation, $t_i$, wherein the crack starts to propagate stably. Figure 12 also shows that HAYNES 230 displays a lower crack growth rate than INCONEL 617. After the completion of SLCG test, the damage zone identification test was conducted subsequently involving a FCP test with the same specimen at room temperature with same $K_{max}$ as SLCG, 6s period and R value of 0.1. The FCP curves of both INCONEL 617 and HAYNES 230 for damage zone tests were plotted as shown in Figure 13, where the FCP exhibits considerably different crack propagation behavior compared to the normal linear steady FCP behavior shown in Figure 10. Since $K_{max}$ was kept the same as the SLCG test, the fatigue crack propagated immediately without any fatigue incubation caused by the plastic zone. The zero cycle refers to the end of the SLCG test. The crack length represents the crack increment at 6s fatigue. Initially, fatigue crack grew at a very fast rate, and then the growth rate gradually slowed down with the increase of crack length. Eventually, the crack growth rate returned to a stable value and the crack propagated again showing a linear relationship between the crack length and number of cycles. The distance from the end of SLCG test to the beginning of a normal crack growth rate is defined as damage zone, inside which the FCP rate was accelerated. As shown in Figure 13 for INCONEL 617, the intersection between the fitting lines of accelerated and normal FCP portion could determine the size of damage zone formed during SLCG. For SLCG at 700°C, both INCONEL 617 and HAYNES 230 produced the
damage zone with size of 85.79 and 19.01 microns respectively. The existence of a damage zone ahead of crack tip was also observed in other Ni-based precipitation-strengthened superalloys INCONEL 718 and 783, and was thought to be the essential mechanism for the time-dependent FCP behavior of those alloys [10-12, 26].

All data of SLCG tests at different temperatures were processed according to the methods illustrated in Figure 12 and Figure 13 to calculate $da/dt$, $t_i$, and damage zone size. Figure 14 compares the damage zone size of INCONEL 617 and HAYNES 230 after SLCG tests. Early studies indicated that the damage zone is a function of holding period, temperature and applied load [10-12, 26]. Given the testing temperature, Figure 14 shows HAYNES 230 has smaller damage zone size than INCONEL 617. Figure 15 and Figure 16 illustrate the plots of the incubation ($t_i$) vs. temperature, and SLCG rate ($da/dt$) as a function of absolute temperature inverse (1/$T$), exhibiting that a high value of $da/dt$ and low value $t_i$ are observed at a higher temperature. For $da/dt$ vs. 1/$T$ in Figure 16, the well-defined linear relationship was noticed. Therefore, a thermodynamic equation, Arrhenius equation, was employed to correlate the $da/dt$ and temperature as shown in equation (3). Figure 16 also indicates that HAYNES 230 has one order magnitude lower $da/dt$ than INCONEL 617, suggesting HAYNES 230 has higher resistance to SLCG. The rate of SLCG can be expressed as:

$$\frac{da}{dt} = C \cdot e^{\left(-\frac{Q}{RT}\right)}$$

(3)

where $C$ is a constant, $T$ is the absolute temperature, $R$ is the gas constant, and $Q$ is a type of thermal activation energy. Based on Figure 16, the $Q$ value can be calculated as 222.15 kJ/mole for INCONEL 617 and 221.75 kJ/mole for HAYNES 230 respectively. For precipitation strengthened Ni-based superalloys INCONEL 718 and 783 at $K_{max}$ equal to 26.5 and 35 MPa$\sqrt{m}$, the Q values have been reported to be 255 and 242 kJ/mole for SLCG in air respectively [10-12, 26]. The activation energy of 232 kJ/mole has been reported for oxidation of HAYNES 230 in stress-free state at high temperatures [32]. The measured $Q$ value in Figure 16 suggests the SLCG process of INCONEL 617 and HAYNES 230 is a rate-controlling process including oxygen thermal interaction with materials. It was thought that the applied stress at crack tip could assist oxygen interaction with material of crack area through reducing the effective activation energy [33]. Therefore, it is not surprising that the activation energy measured from SLCG of HAYNES 230 is about 10 kJ/mole lower than that measured from high temperature oxidation in stress-free state.

### 3.1.4 Fractography

As illustrated above, the experimental results indicated that both INCONEL 617 and HAYNES 230 displayed the time-dependent FCP behavior under hold time fatigue condition. The time-dependent FCP rate increased with period of hold time. Under sustained loading condition, both INCONEL 617 and HAYNES 230 exhibited the rate-controlling crack growth process. Another interesting result is that HAYNES 230 displayed lower time-dependent FCP rates and SLCG rates than INCONEL 617 in Figure 11 and Figure 16, whereas HAYNES 230 has smaller grain size (55 microns) than INCONEL 617 (97 microns), as shown in Figure 9 (a) and (b). Usually, the fine grain size facilitates the time-dependent FCP and SLCG of
superalloys, and consequently reduces the cracking resistance under time-dependent condition [34-35]. To understand these testing results, all specimens were broken into two halves after surfaces indicate that the accelerated time-dependent FCP rates due to introduction of hold time are associated with fracture mode change. Figure 17 shows SEM micrographs of the fracture surfaces for specimen of INCONEL 617 tested at 700°C under 3s, 3+300s and 3+1000s fatigue. At 3s fatigue (cycle-dependent FCP domain), the crack propagated in a typical transgranular fracture mode and the well-defined fatigue striation structures were observed. Figure 17 (a). When a hold time with period of 300s or 1000s was imposed at $K_{max}$, the FCP displayed the time-dependent behavior, resulting in acceleration of FCP rate. The corresponding fracture appearances showed the intergranular fracture feature in Figure 17 (b) and (c). The grain boundary splitting was evident and no fatigue striations appeared on the fracture facets. A few nano-sized voids associated with intergranular M23C6 carbides were observed, suggesting that carbides were pulled out during fracture. Under sustained loading condition, the crack propagation became fully time-dependent in all testing temperatures, and the crack propagated in fully intergranular fracture mode, Figure 18. Facet grain boundaries were split during crack propagation, and some scattered carbides and voids were still found on grain boundaries. A few oxidized features appeared on the grain boundaries and slightly increased with temperature, resulting in rough grain boundary surfaces. The intergranular fracture is the brittle failure mode, suggesting very little grain boundary cohesion during fracture.

Compared with INCONEL 617, the fracture surface of HAYNES 230 tested at high temperature was usually covered by dense oxides and carbides, which somewhat suppressed the details of fracture features. Figure 19 shows SEM micrographs of HAYNES 230 tested at 700°C at 3s and 3+300s fatigue. Due to the presence of two types of carbides, micron-sized $M_6C$ and nano-sized M23C6 carbides, the fracture features of HAYNES 230 are remarkably different from that of INCONEL 617. Fracture appearance at 3s fatigue showed the fatigue striations in Figure 19 (a), whereas some areas were covered by oxides. In addition, lots of cleaved micron-sized $M_6C$-type carbides were noticed, showing the flat and featureless fracture facets as indicated in Figure 18 (a). When crack propagated at 3+300s fatigue, the FCP was time-dependent and the FCP rate increased. The corresponding fracture surface was relatively rough, and cleaved $M_6C$ facets were still visible, however the fatigue striations disappear and some secondary cracks of the cleaved $M_6C$-carbides were observed in Figure 19 (b). Figure 19 (c) shows the close-view of highlighted area in Figure 19 (b), exhibiting that the rough surface is due to the secondary carbides, M23C6-type carbides, which are usually in nano-scale size and distributed along grain boundaries in HAYNES 230 [27-32]. So, these dimple-enriched rough surfaces are the intergranular fracture surface indeed. The nano-sized intergranular M23C6 particles could act as nucleation sites of micro-voids during grain boundary splitting, producing the dimple rupture (or called microvoid coalescence fracture) features. The micron-sized $M_6C$-type carbides ahead of crack were passed by forming the cleavage surface during crack propagation, which sometimes leads to the secondary cracks that may reduce the crack growth rate [27-32]. Therefore, the fracture mode of HAYNES 230 under time-dependent FCP condition is combined with dimple-included intergranular fracture together with $M_6C$-type carbide cleavage and secondary cracks. Figure 20 indicates that lots of cleaved carbides, secondary cracks and intergranular dimples are present under sustained loading condition at 600, 700 and 800°C, suggesting the similar fracture mode as time-dependent fatigue. Therefore, the combination of dimple-included intergranular fracture accompanying with carbide cleavage and secondary cracks are responsible for the lower
crack growth rates of HAYNES 230 compared to INCONEL 617 at condition of time-dependent FCP and SLCG.

3.1.5 Time-dependent FCP of INCONEL 617 and HAYNES 230

As illustrated in Figure 11, the FCP rates of INCONEL 617 and HAYNES 230 increase with period of hold time at elevated temperature under time-dependent condition. At fully time-dependent stage, da/dn displays the linear relationship with period of fatigue. Mathematically, the SLCG, da/dt, can be correlated with the time-dependent FCP rate, da/dn, at fully time-dependent stage, since the sustained loading can be considered as a specific hold time fatigue with only one cycle and prolonged holding period. To identify the fully time-dependent FCP process, the FCP rate, da/dn, was converted into the static crack growth rate, da/dt, by using

$$\frac{da}{dt} = \frac{1}{t} \cdot \frac{da}{dn} \quad \text{Equation 3}$$

where $t$ is fatigue period.

Figure 21 shows the plots of static crack growth rate, da/dt, converted by equation (4) using the data in Figure 11, as a function of fatigue period, $t$. In order to correlate the SLCG process with the FCP, the infinite period, $t_\infty$, was employed to represent the period of SLCG condition. Considering the experimental scattering, Figure 21 shows the static crack growth rate, da/dt, moronically decreases with period when fatigue crack grows in the triangular fatigue with 3s period or hold time fatigue with the short holding periods. This stage is called cycle-dependent FCP domain, wherein da/dt decreases with period. In contrast, when fatigue period increase to a certain value, the static crack growth rate, da/dt, is a constant and equal to the SLCG rate, i.e. da/dt at period of $t_\infty$. This stage is called fully time-dependent FCP domain, wherein da/dt is independent of fatigue period and the SLCG rate can be related to the FCP rate. As expected, high temperature usually causes the higher crack growth rate in cycle / time-dependent FCP stage. It should be noted that the various minimum holding periods are required in order to approach the fully time-dependent FCP at different temperatures. Lowering temperature requires longer hold time to reach the fully time-dependent FCP stage. For example, HAYNES 230 needs at least approximately 20,000 seconds hold to reach the fully time-dependent FCP stage at 600°C, Figure 21(b). Therefore, the results of Figure 21 as well as presence of incubation for SLCG suggest that the conventional concept, which considers the hold time FCP rate as superposition of fatigue and creep crack growth rate, appears to be incorrect for the experimental alloys, INCONEL 617 and HAYNES 230, in the testing condition where $K$ can define the stress state at crack tip.

3.1.6 Crack Propagation Mechanism

It has been well documented that the accelerated time-dependent FCP rate of precipitate-strengthened Ni-based superalloys caused by hold time is associated with SAGBOE (stress...
accelerated grain boundary oxygen embrittlement), which allows crack to propagate in intergranular fracture mode [10-12, 26]. A damage zone model based on SAGBOE mechanism has been proposed to characterize the time-dependent FCP and SLCG process of INCONEL 718 and 783, and the detailed damage zone mechanism can be found in references [10-12, 26]. In this study, the presence of damage zone has been confirmed in Figure 13, and the measured thermal activation energy for SLCG is relatively lower than that for stress-free high-temperature oxidation of HAYNES 230. Therefore, the time-dependent FCP behavior of INCONEL 617 and HAYNES 230 can also be characterized using the damage zone model. Accordingly, when the specimens of INCONEL 617 and HAYNES 230 were subjected to hold time fatigue, oxygen in air was thought to penetrate along the grain boundary at crack tip and interact with grain boundary materials during holding. As a result, a damage zone ahead of crack tip was formed. Inside the damage zone, materials have been embrittled due to oxygen interaction at grain boundaries. After holding, the following cyclic loading (unloading and reloading) permitted the crack to advance at a fast rate within the damage zone. The crack advancement rate was related to the degree of the damage and damage-zone size, which were a function of applied loading, temperature, and hold time length. Thus, a high temperature and long holding period usually produce the high FCP rate and promote the time-dependent FCP occurrence. For SLCG as shown in Figure 12, the holding period was extended to a prolonged period. When a precracked specimen was subjected to a sustained loading, K_{max}, at the testing temperature, the crack could not grow immediately. As time elapsed, oxygen in air was believed to penetrate along grain boundary to form a damage zone ahead of crack tip. The damage degree and damage zone size increased with holding time until to a critical value, wherein crack initiated and then propagated. The duration of damage zone establishment corresponded to the incubation. The steady SLCG rate, da/dt, was equal to the propagation rate of damage zone as well as was associated with damage zone size and damage degree [10-12, 26]. When the consumption and formation of the damage zone ahead of crack tip approached the dynamic balance status, the sustained loading crack propagated at a steady rate. Therefore, as displayed in Figure 14, the fact that HAYNES 230 has smaller damage zone size than INCONEL 617 is thought to be associated with the lower SLCG rates. Figure 22 shows the FCP curve of damage zone identification test and corresponding SEM micrograph of fracture surface for INCONEL 617 specimen tested at room temperature and 6s fatigue subsequently following the completion of SLCG at 800°C. Figure 22 (a) demonstrates that INCONEL 617 has an accelerated FCP rate inside the damage zone with size of about 201 microns. The corresponding SEM micrograph of fracture morphology exhibits a boundary between SLCG at 800°C and 6s-FCP at room temperature. In the region of SLCG at 800°C, the fracture feature shows the typical intergranular fracture. In the region of FCP at 6s and room temperature, the fracture mode shows intergranular fracture firstly, and then gradually transforms to the conventional fatigue fracture with transgranular striations. The intergranular fracture area in the 6s-FCP region represents the damage zone formed during the SLCG at 800°C. The FCP rate dramatically increased within the damage zone. Figure 22(a). As crack propagated further, the fracture mode gradually transformed to trangranular fracture accompanying with the decreasing of FCP rate. As crack passed the damage zone, the FCP rate resumed the normal and fracture mode became fully transgranular mode, Figure 22(b). The width of intergranular fracture domain in 6s FCP region is in agreement with the damage zone size measured in Figure 22(a). For HAYNES 230, the damage zone size is only about 21 microns, Figure 23. The fracture feature of damage zone is not very clear because of the presence of many cleaved M_{6}C carbides and oxides. Nonetheless, the transgranular feature due
to FCP outside the damage zone, and dimple-included intergranular fracture feature due to FCP inside the damage zone, and SLCG at 800°C are still visible, Figure 23(b). Lots of intergranular dimples are noticed, indicating the grain boundary damage due to SLCG. In contrast to INCONEL 617, numerous intergranular carbides in HAYNES 230 may block the oxygen penetration along grain boundaries, resulting in forming a smaller damage zone.

### 3.1.7 Modeling Characterization of Time-dependent FCP

According to the above discussion, time-dependent FCP and SLCG process are essentially rate-controlling processes associated with the formation and propagation of damage zone ahead of crack tip. Therefore, a time-dependent factor, \( \alpha(t, T) \), including parameter of thermal activation energy \( (Q) \), fatigue period \( (t) \), and temperature \( (T) \) can be adapted to normalize the all measured FCP rates. The time-dependent factor is defined as:

\[
\alpha(t, T) = \ln(t) - \frac{Q}{RT}
\]

Equation 4

where \( Q \) is the thermal activation energy for SLCG, \( t \) is the period of fatigue, \( R \) is gas constant, and \( T \) is the absolute temperature. As indicated in Figure 16, since INCONEL 617 and HAYNES 230 have identical \( Q \) value, the average value of 222.00 kJ/mole based on the two experimental alloys was used in the equation (5). Figure 24 re-plots the all measured FCP rates of INCONEL 617 in Figure 11 against the time-dependent factor, \( \alpha(t, T) \), showing that all data fall in one curve that consists of two straight lines. The horizontal line with a constant FCP rate, \( \frac{da}{dn} \), represents the cycle-dependent FCP stage, where the FCP rate is the pure mechanical behavior associated with material Young’s modulus and cyclic stress intensity factor, \( \Delta K \). The other line obtained from the SLCG data represents the time-dependent FCP stage. The crack growth rate is controlled by the static stress intensity factor, \( K_{max} \). The empirical expression of the time-dependent FCP of INCONEL 617 in Figure 24 can be obtained as follows:

\[
\ln\left(\frac{da}{dn}\right) = k\alpha(t, T) + B
\]

Equation 5

where \( k \) and \( B \) are constants respectively.

Similarly, the data of FCP rates for HAYNES 230 were indexed using \( \alpha(t, T) \). The characteristics curves of FCP for INCONEL 617 and HAYNES 230 are exhibited in Figure 25. In cycle-dependent FCP domain, INCONEL 617 and HAYNES 230 have the similar FCP rates. Since INCONEL 617 and HAYNES 230 have identical Young’s modulus from room temperature to high temperature [7-8], it is not surprising that FCP rates are identical at same \( \Delta K \). At cycle-dependent FCP stage, Paris law can be used to describe the FCP rate as follows:

\[
\frac{da}{dn} = A \cdot (\Delta K)^n
\]

Equation 6

where \( A \) is a constant, \( \Delta K \) is the cyclic stress intensity factor and \( n \) is a material constant.

For time-dependent FCP of INCONEL 617, the empirical expression is:
\[
\ln\left( \frac{da}{dn} \right) = 1.08 \cdot \alpha(t, T) + 19.01 \quad \text{Equation 7}
\]

For time-dependent FCP of HAYNES 230, the FCP rate is

\[
\ln\left( \frac{da}{dn} \right) = 0.96 \cdot \alpha(t, T) + 14.46 \quad \text{Equation 8}
\]

The semi-empirical time-dependent FCP model provides a simple and straightforward method to characterize the complete FCP behavior of INCONEL 617 and HAYNES 230 under both pure fatigue and hold time fatigue conditions. In Figure 25 and Equation 5, there are two interesting parameters at cycle/time-dependent FCP stage. One is the offset value of the time-dependent factor for the beginning of time-dependent FCP. The other is the degree of time-dependence, i.e. the slope \(k\) in Equation 5, when crack propagation becomes time-dependent. If the absolute value of time-dependent factor offset and \(k\) are lower, the resistance to time-dependent FCP is higher. In Figure 25, HAYNES 230 has higher resistance to time-dependent FCP than INCONEL 617. Both time-dependent factor offset and \(k\) value are parameters of the material property and can be controlled through modification of alloy chemical composition and microstructures. The characteristics curve indexes the resistance of the alloy to time-dependent FCP. When time-dependent factor is small, the crack propagates at cycle-dependent mode and fracture mode is transgranular. The FCP behavior is related to mechanical damage process associated with material Young’s modulus and \(\Delta K\). Hence the FCP rate is constant under constant-\(\Delta K\) condition. As time-dependent factor increases, the fatigue propagates at time-dependent mode. The FCP rate no longer remains constant, increases with the time-dependent factor, and is determined by the maximum loading, \(K_{\text{max}}\), hold time period, and temperature. The intergranular fracture is associated with the crack propagation at time-dependent domain.

### 3.1.8 Summary of Task 1

The effects of temperature and hold time on FCP behavior as well as SLCG of INCONEL 617 and HAYNES 230 have been extensively studied using precracked CT specimens under a constant K-controlled mode. The fracture mode was discussed. At fully time-dependent domain, the FCP rate was able to be related to the SLCG rate. Toward this, a characteristic curve is able to characterize all the data. The key results suggest the following conclusions:

1. Both INCONEL 617 and HAYNES 230 can display fully time-dependent FCP behavior at elevated temperatures ranging from 600°C to 800°C if fatigue period or hold time becomes sufficiently long. Crack propagation rate under fully time-dependent condition is determined by the static stress intensity factor, \(K_{\text{max}}\).

2. Both INCONEL 617 and HAYNES 230 display the rate-controlling SLCG process, and a thermodynamic equation is able to correlate the crack growth rates. Both alloys show the identical thermal activation energy during crack growth.
3. The fracture mode of INCONEL 617 at time-dependent FCP and SLCG is fully intergranular fracture. Relative to INCONEL 617, the fracture mode of HAYNES 230 under time-dependent condition is combination of dimple-included intergranular fracture accompanying with carbide cleavage and secondary cracks. Numerous primary $M_6C$ carbides and secondary intergranular $M_{23}C_6$ in HAYNES 230 are responsible for the mixed fracture modes and lower crack propagation rates.

4. A time-dependent factor including thermal activation energy, period and temperature can be employed to index all data of the experimental alloys. Two characteristic curves are obtained to characterize the FCP process of INCONEL 617 and HAYNES 230 at cycle and time-dependent FCP domain. HAYNES 230 displays lower time-dependent FCP rates than INCONEL 617.

5. In the experimental temperatures, 873K-1073K (600°C-800°C), the LEFM parameter, $K$, is sufficient to characterize the crack tip stress state. The conventional creep-fatigue mechanism induced time-dependent FCP appears to be invalid in such temperature ranges, and the hold time fatigue should be a proper term instead of creep-fatigue.

6. Both INCONEL 617 and HAYNES 230 are susceptible to SAGBOE-induced cracking at elevated temperature. However HAYNES 230 appears to display higher cracking resistance than INCONEL 617. When both alloys are utilized in highly oxidizing environment such as NGNP system, the SAGBOE-induced degradation should be considered.
Figure 9  Microstructures of as-received materials: (a) optical micrograph of INCONEL 617; (b) optical micrograph of HAYNES 230; (c). SEM micrograph of INCONEL 617; (d). SEM micrograph of HAYNES 230

<table>
<thead>
<tr>
<th>Materials</th>
<th>YS (MPa)</th>
<th>UTS (MPa)</th>
<th>EL (%)</th>
<th>RA (%)</th>
<th>Hardness (RB)</th>
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<tr>
<td>Alloy 617</td>
<td>371</td>
<td>855</td>
<td>78.4</td>
<td>62.0</td>
<td>86.8</td>
</tr>
<tr>
<td>Alloy 230</td>
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<td>820</td>
<td>46.0</td>
<td>43.0</td>
<td>92.0</td>
</tr>
</tbody>
</table>
Figure 10  Constant $\Delta K$ FCP curves for different hold times tested at 600°C in air: (a) INCONEL 617 and (b) HAYNES 230
Figure 11 FCP rates as a function of period at various temperatures

Figure 12 SLCG of INCONEL 617 and HAYNES 230 in air at constant $K$ equal to 27.75 MPa$\sqrt{m}$ and 700°C
Figure 13  FCP curves of INCONEL 617 and HAYNES 230 at \( K_{\text{max}} = 27.75 \text{ MPa}\sqrt{\text{m}} \), \( R=0.1 \), and room temperature after SLCG testing at 700\(^\circ\)C.

Figure 14  Damage zone size measurement.
Figure 15 Incubation vs. temperature (K)

Figure 16 SLCG rate, $da/dt$, vs. $1/T$
Figure 17  SEM fracture surface micrographs of INCONEL 617 specimen subjected to FCP tests at 700°C: (a). at 3s; (b). at 3+300s; (c) at 3+1000s
Figure 18  SEM fracture surface micrographs of INCONEL 617 specimen subjected to SLCG tests: (a). at 600 °C; (b). at 700 °C; (c) at 800 °C
Figure 19  SEM fracture surface micrographs of HAYNES 230 specimen subjected to FCP tests at 700°C: (a) at 3s; (b) at 3+300s; (c) Close-view of (b)
Figure 20  SEM fracture surface micrographs of HAYNES 230 specimen subjected to SLCG tests: (a) at 600 °C; (b) at 700 °C; (c) at 800 °C
Figure 21 Static crack growth, $da/dt$, vs. period at $K_{max} = 27.75$ MPa√m: (a) INCONEL 617 and (b) HAYNES 230

Figure 22 Damage zone examination of INCONEL 617: (a). FCP curve at room temperature after SLCG test at 800°C; (b). Corresponding SEM micrograph of fracture surface. Arrow indicates the crack growth direction.
Figure 23 Damage zone examination of HAYNES 230: (a). FCP curve at room temperature after SLCG test at 800°C; (b). Corresponding SEM micrograph of fracture surface. Arrow indicates the crack growth direction.

Figure 24 Normalized FCP rates, $da/dn$, of INCONEL 617 by the time-dependent factor, $\alpha(t,T)$ at $K_{max} = 27.75$ MPa/$\sqrt{m}$.
Characteristic curves to describe FCP behavior of INCONEL 617 and HAYNES 230

**Figure 25** Characteristic curves to describe FCP behavior of INCONEL 617 and HAYNES 230
3.2 Results and Discussions of Task 2

3.2.1 Nano-indentation Test of INCONEL 617

A Hysitron Ti 950 TriboIndente as shown in Figure 6 was used to perform the nano-indentation tests on INCONEL 617 samples subjected to SLCG test at 800°C and 6s-FCP test at room temperature respectively. Nano-indentation tests at room temperature were first performed in a grid around the crack to determine if the hardness and reduced modulus values of this region as a function of x/y coordinates would help identify the cracking mechanism. Two more grids of indents (closer spacing and lower force) were then performed at other crack locations to verify that the area near a crack contains higher hardness values than the areas further away from a crack.

A diamond Berkovich probe was used to perform nano-indentation tests on the samples. The sample surface was polished, and cleaned with acetone and a cotton swab. All tests within the First Region were performed in load-controlled feedback mode to a peak force of 7,000 µN. All tests within the Second and Third Test Regions were performed in load-controlled feedback mode to a peak force of 3,000 µN. A load function consisting of a 5-second loading to peak force segment, followed by a 2-second hold segment and a 5-second unloading segment was used. Figure 26 shows an example of location for nano-indentation test and force versus displacement. For the First Region a total of 153 indents were performed in a 17 x 9 grid extending 100 µm left of the crack tip, 20 µm right of the crack tip, and 30 µm above and below the crack tip. These indents were spaced 7.5 µm apart. For the Second Test Region 10 indents were performed from click-script Piezo Automation in a T-pattern approximately 4 µm apart. For the Third Test Region 49 indents were performed from Piezo Automation in a 7 x 7 grid with 3 µm spacing.

For specimen subjected to SLCG at 800°C, Figure 27 show the map of hardness and reduced modulus values from the First Test Region as a function of x/y coordinates, respectively. The results show that hardness and reduced modulus value are high around crack tip area and decrease with increasing position from the crack tip, suggesting materials around crack tip have been embrittled during SLCG at 800°C.

For specimen subjected to 6s-FCP at room temperature, Figure 28 displays the map of hardness and reduced modulus as a function of position around testing regions, respectively. The indents that indicate very soft material (shown as red points) are recording effects based on the proximity to the cracks. These indents show low hardness and reduced modulus because the sample can easily be deformed directly near the crack edge. Aside from those indents, the hardness values near the crack tip are consistently slightly higher than away from the cracks. Another aspect seen in the hardness plots is that the hardness appears to be its highest directly in front of the crack propagation direction. The reduced modulus plots show the reduced modulus values slightly decreasing as the indents are performed closer to the cracks. Comparing the values of hardness and reduced modulus for specimens subjected to SLCG at 800°C and 6s-FCP at room temperature, it was found that sample subjected to SLCG at 800°C had slighter higher hardness and much higher reduced modulus. Thus, materials of crack tip at 6s-FCP still keep relatively higher stiffness compared to sample after SLCG test 800°C. 6s-FCP test at room
temperature didn’t cause much embrittlement of materials ahead of crack tip, though work hardening occurred in small area ahead of crack tip.

3.2.2 Summary of Nano-indentation

Nano-indentation examination of crack tip area was extensively conducted on INCONEL 617 specimens subjected to SLCG at 800°C and 6s-FCP test at room temperature. The results show that for sample after SLCG test, the hardness and reduced modulus values decrease with increasing position from the crack tip, and for sample after 6s-FCP at room temperature, low hardness was found around crack edge and high hardness and slight decreasing of modulus appeared in the front crack area. SLCG produced higher hardness and reduced modulus ahead of crack tip than 6s-FCP, suggesting environment factor cause embrittlement and increasing of hardness and reduced modulus.

3.2.3 TEM studying on Crack Tip Area

TEM sample was taken in front of crack tip area from for alloy 617 specimen subjected to SLCG test at 800°C. FIB (focus ion beam) cross-section lift-out procedure was used for TEM sample preparation. Utilization of FIB allows the sample to be accurately taken in specific site with minimizing introduction of artifacts, which may exist when using grinding method to prepare sample. The TEM sample was conducted analysis to examine the local microstructure and chemical profile just ahead of crack tip area. Figure 29 (a) shows TEM sample was located at front of crack tip, and Figure 29 (b) shows that sample area was coated first and then the trenches around the sample were milled using FIB for the cross-section lift-out sample preparation. Figure 30 (a) shows a TEM sample was lift out using a probe when thickness was approached. Figure 30 (b) shows a FIB prepared TEM sample ready for observation, and the thickness of sample is about 50-60nm ready.

Figure 31 shows TEM images of crack tip of INCONEL 617 subjected to 6s fatigue test and room temperature. The testing condition is typical cycle-dependent FCP condition. As image shown, numerous dislocation networks were observed in [110] zone axis direction, suggesting lots of deformation and slip occurred during FCP test. FCP is pure mechanistic damage behavior. In contrast, Figure 32 shows that TEM images of specimen subjected to 800°C SLCG exhibited well organized dislocation network and less dislocation density than specimen tested at 6s FCP and room temperature. The specimen subjected to SLCG at 800°C was also examined using FETEM (filter energy TEM) mode to identify chemical information at front of crack tip area. Figure 33 show FETEM mode images. Figure 33 (a) is zero-loss image, showing the subgrain structure and well-organized dislocation lines, and indicating that no strong deformation occurred in the crack front areas. Energy loss mapping image of same area and EELS (electron energy loss spectrum) as seen in Figure 33 (b) and (d) show that there is oxygen and Cr segregation in the crack front region, suggesting that oxygen penetrated into crack front area and interacted with materials forming Cr-contained oxides. Figure 33 (c) shows EELS spectrum of O-edge.

Based on TEM analysis, crack propagation at cycle-dependent condition is pure mechanistic damage process, and sustained loaded crack growth is primarily related oxygen interaction with grain boundary of crack tip area, i.e. environmental factor.
Figure 26  An example of nano-indentation specimen; (a) Nano-indentation location; (b) loading vs. displacement during indentation.

Figure 27  Map of hardness, deduced modulus and location ahead of crack tip for INCONEL 617 specimen subjected to SLCG test at 800°C
Figure 28 Map of hardness, deduced modulus and location ahead of crack tip for INCONEL 617 specimen subjected to FCP test at 6s and room temperature.

Figure 29 Preparation of TEM sample ahead of crack tip by FIB, (a) sample location; (b) FIB lift-out technique for cross-section TEM sample.
Figure 30 TEM sample prepared by FIB lift-out procedure. (a). Sample was lift out; (b). A TEM sample ready for observation.

Figure 31 TEM images of INCONEL specimen tested at 6s-FCP and room temperature with different magnification along [011]
Figure 32  TEM images of INCONEL specimen subjected to SLCG at 800°C with different magnification along [011]
Figure 33  FETEM mode image and EELS spectrum of specimen tested at SLCG and 800°C. (a). Zero-loss image; (b). O Mapping; (c). O-edge EELS spectrum; (d) Cr-mapping
3.3 Results and Discussions of Task 3

3.3.1 FCP Test at 1-second Fatigue

As for cycle-dependent FCP test, Figure 34 plots the constant $K_{\text{max}}$-controlled FCP results of INCONEL 617 and HAYNES 230 tested at 700°C, 1-second period and various $R$-ratios. When $R$-ratio was low such as $R$ equal to 0.05, 0.1 and 0.3, the instant FCP was observed. The variation of crack length ($a$) with number of fatigue cycle ($n$) exhibited a linear relationship, indicating that the crack propagated at a constant rate. Again, the slope of crack growth line represents the FCP rate, $da/dn$. However with the increase of $R$-ratio such as $R$ equal to 0.5 and 0.7, retardation of crack growth appeared at FCP curves, and there existed a certain number of fatigue cycles before reaching a steady FCP rate, indicating the presence of fatigue incubation at $R$ equal to 0.5 and 0.7. The value of incubation cycle could be determined by calculating the intersection point between the extended steady FCP line and horizontal axis. When the FCP reached a steady rate, Figure 34 also showed that the FCP rate generally decreased with the increase of $R$-ratio. Figure 35 summarizes FCP rates of INCONEL 617 and HAYNES 230 as a function of $R$-ratio, showing that the FCP rates of both materials decreases with increase of $R$. When $R$-ratio is low such as $R$ equal to 0.05 or 0.1, INCONEL 617 and HAYNES 230 have identical FCP rates. With the increase of $R$-ratio, the gap between FCP rates increased. Haynes 230 displays slower FCP rate at higher $R$-ratio than INCONEL 617. Figure 36 compared the incubation value of both materials, showing that HAYNES 230 has much longer incubation than INCONEL 617 at $R$ equal to 0.7.

3.3.2 FCP Test at 300-second Hold-time Fatigue

As for time-dependent FCP tests, Figure 37 represents the constant $K_{\text{max}}$-controlled FCP results of both INCONEL 617 and HAYNES 230 tested at 700°C, 300-second hold-time fatigue and various $R$-ratios. In contrast to the cycle-dependent FCP tests, the instant FCP without incubation was observed at time-dependent FCP tests for each $R$-ratio. For INCONEL 617, Figure 37(a) shows the FCP rate slightly decreased with increase of $R$-ratio. The instant and steady FCP was evident at each $R$-ratio, even at $R$ equal to 1.0. As for HAYNES 230, only partial FCP data were obtained and valid FCP test at $R$ equal to 1.0 didn’t complete due to unexpected failure of clamp pins during testing. Nonetheless, Figure 37(b) shows the results of FCP tested at $R$ equal to 0.05, 0.1 and 0.5, indicating similar results to INCONEL 617, i.e. the instant FCP at $R$ equal to 0.5 and reduced FCP rates with increase of $R$-ratio. Figure 38 presents the FCP rate of both materials as a function of $R$-ratio tested at both cycle/time dependent domain and 700°C. As expected, the FCP rate could be accelerated at time-dependent domain. Figure 38 shows that both materials have about one order magnitude higher FCP rates at condition of 300-second hold-time fatigue than that at 1-second cyclic fatigue. Relative to INCONEL 617, HAYNES 230 displayed lower FCP rates at time-dependent FCP region at each $R$-ratio. These results were true only when $R$-ratio was increased from one test sequence to the next. Also, the linear relationship between variation of crack length ($a$) and number of cycle ($n$) in Figure 37(a) and (b) suggests that the LEFM parameter, stress intensity factor ($K$), is suitable to characterize FCP behavior at testing temperature.

3.3.3 Fractography
After FCP testing, fracture surfaces of HAYNES 230 specimen subjected to oxide-layer cleaning process and as-tested INCONEL 617 specimen were examined using SEM. Figure 39 and Figure 40 presents SEM images of fracture surfaces for specimens tested under 1-second-period fatigue condition, respectively. As expected, the fracture mode for the cycle-dependent FCP was transgranular. The border lines at different R-ratios were observed as indicated in images. Typical striations were observed at different R-ratios for both INCONEL 617 and HAYNES 230. The insets in Figure 39 (b) and Figure 40 (b) show the striation structures, where spacing are related to the distance of crack advancement per cycle. As shown in Figure 34, when R-ratio was small such as 0.05, 0.1 or 0.3, FCP was instant. Continuous striations across the border line at different R-ratios were observed in Figure 39 (a)-(b) and Figure 40 (a)-(b), suggesting crack grew immediately as fatigue was applied. In contrast, FCP of both alloys showed incubation or retardation of crack growth, when R-ratio was equal to 0.5 or 0.7. For the FCP with incubation or retardation, the striation features didn’t appear in the next crack growth region next to border line, when R-ratio was switched from one to the next. Numerous cleavage-like features were observed in the crack growth region after retardation. As crack grew away from the border line and the steady FCP was resumed, the fracture surface was found containing the striations and a little intergranular feature. The insets in Figure 39 (c)-(d) and Figure 40 (c)-(d) showed the striations and facet intergranular features containing lots of oxides of intergranular nano-sized M23C6 carbides. The cleavage-like feature was associated with incubation and new crack growth.

When FCP tests were conducted at condition of 300-second hold-time fatigue, FCP of both materials occurred at time-dependent domain as previously described. The time-dependent FCP rates have been enhanced to at least one order of magnitude relative to cycle-dependent FCP rate as shown in Figure 37. Figure 41 and Figure 42 shows SEM images of fracture surface for specimens tested at 300-second hold-time fatigue and various R-ratios for INCONEL 617 and HAYNES 230, respectively. It is shown that all fracture surfaces display the intergranular fracture mode at each R-ratio. For HAYNES 230, fracture surface contained numerous oxides related to intergranular nano-scaled Cr-enriched M23C6 carbides and ductile features, indicating a complex fracture mode including intergranular fracture, carbide cleavage, and dimple fracture. The inset in Figure 42 (a) and highlighted region in Figure 42 (b) indicate intergranular facets including numerous oxides. Detailed analysis of fracture mode of HAYNES 230 at time-dependent FCP domain has been conducted and reported previously, suggesting that the combined fracture modes were responsible for the lower FCP rates than INCONEL 617 [37-38].

3.3.4 Model of Load Ratio Effect

FCP process of Ni-based superalloys usually shows the cycle-dependent behavior at ambient temperature or high frequency. The cycle-dependent FCP is an accumulation mechanistic damage process, and the fracture mode is transgranular [39-41]. Numerous studies have shown that, for cycle-dependent FCP, R-ratio has significant influence on FCP rate in both threshold and Paris regions. For FCP process at constant-load-controlled mode, the FCP rate usually increases and range of threshold stress-intensity-factor (∆Kth) decreases with R-ratio [39-41]. The effects of R-ratio on FCP rates, described in term of stress intensity factor range (∆K), is attributed to crack closure behavior due to fracture surface, roughness, build-up of oxide scale on fracture surface, and development of plastic deformation at the crack tip. In intermediate region
or Paris region of FCP at constant-load-controlled mode, the FCP rate, \( da/dn \), can be correlated with range of stress intensity factor, \( \Delta K \), by the Paris Law [39-41]:

\[
\frac{da}{dn} = C(\Delta K)^m \tag{Equation 9}
\]

where both \( C \) and \( m \) are constants.

For INCONEL 617, S.-S Hsu has showed that, at cycle-dependent domain, constant-load-controlled condition and 650 °C, an increase in the \( R \)-ratio could significantly increase the FCP rate by means of primarily increasing the proportionality factor \( C \) in Eq. (10) [20].

In contrast, in this study, Figure 35 demonstrated that FCP rate decreased with increase in \( R \) at cycle-dependent stage and constant \( K_{\text{max}} \)-controlled condition. These results are consistent with that of similar studies by Dimopulous, et al [42]. By means of manipulation of the terms \( K_{\text{max}} \) and \( \Delta K \), Walker [41] proposed the total applied load due to variation of \( R \)-ratio could be expressed as:

\[
\Delta K_{\text{app}} = (1 - R)^\alpha \cdot K_{\text{max}} \tag{Equation 10}
\]

where \( \alpha \) is a constant.

Then, at cycle-dependent FCP at condition of \( K_{\text{max}} \)-controlled mode, FCP rate can be expressed as:

\[
\frac{da}{dn} = C_1 \cdot (\Delta K_{\text{app}})^{m_1} \tag{Equation 11}
\]

where both \( C_1 \) and \( m_1 \) are experimentally determined constants.

Using data of Figure 35, Figure 43 plots the FCP rate as a function of \( \Delta K_{\text{app}} \) converted using Eq. (11) and \( \alpha \) equal to 1.0-1.5, showing a well-defined linear dependence between FCP rates and \( \Delta K_{\text{app}} \). The linear regression fitting analysis of Figure 43 gave a coefficient of determination (\( R^2 \)-value) of 0.978 for INCONEL 617, and 0.993 for HAYNES 230, respectively. Also, the experimental constant, \( m_1 \), was calculated to be 0.95 for INCONEL 617, and 1.47 for HAYNES 230, respectively.

Another interesting finding at cycle-dependent FCP tests was appearance of incubation and retardation of crack growth, when \( R \) was increased to 0.5 and 0.7. This observation could be related to FCP at near threshold region and crack closure. As shown in Equation 10, \( K_{\text{app}} \) decreases with increase of \( R \)-ratio at condition of constant \( K_{\text{max}} \)-controlled mode. In general, as \( K_{\text{app}} \) decreases near to or less than threshold of stress intensity factor range (\( \Delta K_{\text{th}} \)), FCP retardation will occur and crack growth will be delayed or terminated if crack closure, which might be caused by oxidation or roughness of fracture, occurs [40-43]. In this study, since the cycle-dependent FCP was conducted at constant \( K_{\text{max}} \)-controlled mode, there always existed a crack tip open displacement (CTOD) even at very high \( R \)-ratio. Presumably, CTOD allowed oxygen in air to interact with the local material ahead of crack tip at high temperature even during crack closure or retardation, and then weaken the crack tip area producing intergranular
damage and cleavage fracture. As a result, the crack growth could start again after specimen received sufficient fatigue cycles. In crack initiation or restarting stage, the fracture surface showed the cleavage-like feature. As long as the steady FCP was resumed, FCP was predominantly controlled by $\Delta K_{\text{app}}$ and Equation 11 was still valid to describe the steady FCP rates as shown in Figure 43. During FCP, CTOD would allow environmental effect to be involved, producing mixed fracture modes with striation and intergranular features as shown in Figure 39-Figure 40 (c) and (d).

Comparing to the cycle-dependent FCP tests at period of 1-second and different $R$-ratios, the results of time-dependent FCP tests at a fatigue with 300-second hold-time show the enhanced FCP rates and instant FCP behavior without incubation and retardation of crack growth at each $R$-ratio. For the solid-solution-strengthened Ni-based superalloys such as INCONEL 617, HAYNES 230 and HAYENS 188, the time-dependence of FCP rate at elevated temperature could be ascribed to environmental attack and creep process, both of which competitively affect the FCP process. When creeping is predominant during hold time FCP process at high temperature such as above 800°C or high applied load, the creep fracture mechanics parameter, $C^*$, at the maximum load of a cycle is suitable to correlate with the time-dependent FCP rate [22, 42]. In this study, the fact that the linear crack growth behavior shown in Figure 37 suggests the stress intensity factor ($K$), a LEFM parameter, is valid still to describe the FCP process. Indeed, previous task studies have indicated that the environmental attack was primarily responsible for the time-dependent FCP behavior of INCONEL 617 and HAYNES 230 at intermediate testing temperature (600-800°C) in air [37-38]. According to task 1 results, when a specimen was subjected to a hold-time fatigue at intermediate temperature, a damage zone due to oxygen interaction and embrittlement could be formed at the front of crack tip during holding. After holding, the following cyclic portion of fatigue, unloading and loading, would cause the substantial crack extension within the damage zone, resulting in the accelerated crack growth behavior. Experimental results showed that formation of damage zone of INCONEL 617 and HAYNES 230 during holding was related to SAGBOE mechanism [37-38]. The crack growth at time-dependent condition actually involves a thermodynamic process of damage zone formation and propagation. At fully time-dependent FCP domain, $K_{\text{max}}$ associated with CTOD is thought to be a major mechanistic parameter to govern the FCP process, and the cyclic portion of a fatigue cycle only played a negligible role in determining the FCP rate. The detailed damage zone mode for time-dependent FCP of Ni-based superalloys has been discussed in Task 1. In this task, the FCP tests were performed starting at $R$ equal 0.05 and $R$-ratio was increased from one test to the next as illustrated in Figure 5. Since the constant $K_{\text{max}}$-controlled mode and 300-second hold-time fatigue were applied, a CTOD always existed and a damage zone could be formed during holding regardless of the values of $R$-ratio and $\Delta K_{\text{app}}$. Given a cyclic loading, $\Delta K_{\text{app}}$, with a value either low or high, the damage zone due to oxygen embrittlement could be cracked, resulting in the instant crack growth. Therefore, the incubation or retardation of crack growth was not observed during the FCP tests with 300-second hold-time at each $R$-ratio. Mathematically, at fully time-dependent FCP domain, a pure time-dependent parameter, sustained loading crack growth rate ($da/dt$), could be employed to correlate FCP rates at different $R$-ratios. The cyclic crack growth rate ($da/dt$) to sustained loading crack growth rate ($da/dt$) can be correlated by the following equation [37-38]:

53
Equation 12

\[
d\frac{a}{d} = \frac{1}{t} \cdot \frac{d}{d}n
\]

where \(t\) is period of fatigue

Note that when \(R\) is equal to 1, the fatigue loading becomes a sustained loading as illustrated in Figure 5 (b). Figure 44 re-plots \(da/dt\) converted from data of Figure 37 using Equation 12 as a function of \(R\)-ratio value, and the data of sustained loading crack growth rates produced in the previous study were also included in figure to correlate with \(da/dn\) [37-38]. Within a scattered range of one order magnitude, \(da/dt\) converted by Equation 12 could be correlated with FCP rate, \(da/dn\), regardless of \(R\)-ratio. Previous study indicated that the sustained loading crack growth was a thermally activated process, where \(K_{\text{max}}\) is a major mechanistic parameter to determine \(da/dt\). Therefore, at time-dependent FCP domain, FCP is mainly governed by \(K_{\text{max}}\), and FCP occurred instantly at each \(R\)-ratio. The cyclic mechanistic parameter such as \(R\)-ratio plays a minor role in affecting the fully time-dependent FCP behavior.

3.3.5 Summary of Task 3

The effect of load ratio on FCP of INCONEL 617 and HAYNES 230 was studied under cycle and time-dependent FCP condition at 700°C using a constant \(K_{\text{max}}\)-controlled mode. Experimental and analytical results suggest the following conclusions:

1. For cycle-dependent FCP tests at a 1-second cyclic fatigue, increasing load ratio results in decreasing FCP rate, \(da/dn\), as well as introduction of incubation and retardation of crack growth. It has been shown that the effect of load ratio can be accounted for by total applied stress intensity factor range, \(\Delta K_{\text{app}}\), and concept of FCP at near threshold region. After incubation or retardation of crack growth, the restarting of cracking and resuming of FCP are related to environmental effect and the presence of CTOD due to constant \(K_{\text{max}}\)-controlled-mode.

2. For time-dependent FCP tests at a fatigue with a period of 300-second hold-time, instant FCP without remarkable incubation or retardation of crack growth was observed at each \(R\)-ratio as well as FCP rate decreased slightly with increasing \(R\)-ratio. The formation and cracking of a damage zone ahead of crack tip is thought to be related to the instant FCP behavior. The FCP process at each \(R\)-ratio is governed by \(K_{\text{max}}\), and a sustained loading crack growth rate, \(da/dt\), is able to be employed to correlate with the FCP rates.

3. When FCP behavior is related to the environmental effect such as cycle-dependent FCP at \(R\) equal to 0.5 or 0.7, or time-dependent FCP, HAYNES 230 displayed lower FCP rate than INCONEL 617. The combined fracture mode is responsible for such results.

4. The instant FCP behavior and enhanced FCP rates at time-dependent condition is independent of \(R\)-ratio. This finding is important and instructive for component design and maintenance.
Figure 34  Constant-ΔK controlled FCP curves with different R-ratios at 1-second and 700°C in air; (a) INCONEL 617 and (b) HAYNES 230.
Figure 35 FCP rates as a function of $R$-ratio at $K_{\text{max}}=27.75$ MPa$\sqrt{\text{m}}$

Figure 36 Comparison of fatigue incubation at different $R$-ratios
Figure 37 Constant-$\Delta K$ controlled FCP curves with different $R$-ratios at 300-second hold time and 700°C in air; (a) INCONEL 617 and (b) HAYNES 230
Figure 38 Summary of FCP rates as a function of $R$-ratio at $K_{\text{max}}=27.75$ MPa$\sqrt{\text{m}}$
Figure 39 Fracture surfaces of INCONEL 617 specimen tested at 700°C, 1s and different R. Note that hollow arrow indicates the FCP direction and solid arrow indicates the border line of different R-ratios. (a) $R = 0.05$ and 0.1, (b) $R = 0.1$ and 0.3, (c) $R=0.3$ and 0.5, and (d) $R=0.5$ and 0.7.
Figure 40 Fracture surfaces of HAYNES 230 specimen tested at 700°C, 1s and different $R$. Note that the hollow arrow indicates the FCP direction and solid arrow indicates the border line of different $R$-ratios. (a) $R = 0.05$ and 0.1, (b) $R = 0.1$ and 0.3, (c) $R = 0.3$ and 0.5, and (d) $R = 0.5$ and 0.7.

Figure 41 Fracture surfaces of INCONEL 617 specimen tested at 700°C, 300s hold time and different $R$-ratios. Note that the hollow arrow indicates the FCP direction. (a) $R = 0.05$, and (b) $R = 0.5$. 
Figure 42 Fracture surfaces of HAYNES 230 specimen tested at 700°C, 300s hold time and different R-ratios. Note that the hollow arrow indicates the FCP direction. (a) $R = 0.05$, and (b) $R = 0.5$.

Figure 43 $da/dn$ vs. $\Delta K_{app}$
Figure 44 \( \frac{da}{dt} \) vs. \( R \)

\[ K_{\text{max}} = 27.75 \text{ MPa} \sqrt{\text{m}} \text{ and } 700^\circ \text{C} \]
3.4 Results and Discussions of Task 4

3.4.1 Microstructure

The composition of Alloys 617 and 230 used for Task 4 is shown in Table 1. The microstructure analyses of Alloys 230 and 617 for Task 4 have been extensively evaluated using SEM and optical microscopy. Figure 45-48 list the micrographs of optical microstructure and SEM BS (back scatter) images, showing grain and carbide morphology, respectively. As described previously, after annealing, both alloys show isotropic microstructure and numerous carbides. For INCONEL 617 used for Task 4, the carbon addition has a little difference, and for HAYNES 230, the carbon content is same but grain size is slightly different. Grain size measurement and carbide fraction measurement have been conducted. Table 2 summarizes the evaluation results for property and microstructure analysis.

3.4.2 FCP Test

FCP tests of both INCONEL 617 and HAYNES 230 with different heats were conducted at cycle/time dependent condition, respectively. Same as testing procedure in Task 1 as shown in Figure 3, $K_{\text{max}}$ was kept constant at 27.27 MPa/$\sqrt{\text{m}}$, and load ratio, R equal to 0.1, at 700°C for all tests. For cycle-dependent FCP test, a period of 3s was chosen. For time-dependent, a hold time fatigue with various holdings ranging from 10, 60…. to1000s were chosen.

FCP testing of HAYNES 230 obtained from INL and Haynes, 230-HA and 230-INL, was conducted at 700°C, R equal 0.1 and different hold time. Figure 49 and Figure 50 showed that the crack grew linearly under constant $K$ condition. Increasing hold time would cause FCP rate increasing, i.e. both specimens showed time-dependent FCP process. Figure 51 summaries the FCP rates of HAYNES 230 with different heats at different period. At cycle-dependent, alloy 230 used in Task 1 shows a little higher FCP rate than other two 230 alloys. With increasing of hold time period, the FCP rates of all Alloys 230 are identical, suggesting that same carbon, slight difference in and other element contents, and grain size did considerably affect FCP rates in time-dependent FCP domain.

For Alloys 617 FCP testing, it was found that both alloys display linear crack growth at 3s cycle-dependent FCP test, and the FCP curves of hold time tests were very scattered. Both alloys, 617-SM and 617-INL, didn’t display time-dependent FCP behavior. The FCP rates under longer holding periods were even lower than that under shorter holding period. The abnormal results were different to the previous results of alloy 617, and produced confusion to us. The testing system problem or materials creeping may cause such discrepant results. To identify which cause the discrepancy, system was re-calibrated and trial testing at 3s and 3+60s were conducted.

Figure 52 shows the results of alloy 617-SM tested at 3s and twice tests at 3+60s. The abnormal FCP behavior re-occurred, and FCP rate of 3s was higher than that of 3+60s. The step-like crack growth was noticed for both 3+60s test, however the well-defined linear behavior was observed for 3s. The step-like curve for 3+60s FCP behavior suggests creeping occurred during FCP testing. The linear fracture mechanics parameter, stress intensity factor, was no longer
defined at crack tip. Since creeping released stress field of crack tip, crack growth retardation would take place.

**3.4.3 Summary**

Alloys 230 of different heats show identical time-dependent FCP rates. For Alloys 617, creeping significantly affected FCP results and caused abnormal FCP behavior. Future study should reduce $K_{\text{max}}$ so that LEFM parameter, $K$, will be valid. Since the project is ended on Sept. 30, 2012, the extra tests will depend on future funding. The creeping phenomenon was new discovering for Alloy s617 with composition shown in Table 1. Previous tests were not such behavior and further study is necessary to fully understand this alloy.

**4. CONCLUSIONS**

In this project, the crack propagation behavior of INCONEL 617 and HAYNES 230 has been extensively studied at elevated temperature. The key results suggest the following conclusions:

1) Both INCONEL617 and HAYNES 230 display fully time-dependent FCP behavior at elevated temperatures at condition of hold time fatigue.

2) Load ratio significantly affects time-dependent FCP behavior, causing instantly crack growth.

3) Numerous $M_6C$ and intergranular $M_{23}C_6$ in Haynes 230 are responsible for the mixed fracture modes and higher cracking resistance.

4) TEM and nano-indentation analysis of crack tip area indicated that oxygen interaction is a major factor responsible for time-dependent FCP behavior.

5) A phenomenological model, based on thermodynamic theory, was developed to index all FCP data. Two characteristic curves are obtained to describe the FCP process at cycle and time-dependent FCP domain.

6) Both Alloys 617 and 230 are susceptible to environment-induced cracking. When both alloys are utilized in oxidizing environment such as NGNP system, the environmental factor should be considered.

7) Carbon content and creeping during test affect FCP behavior.

**5. RECOMMENDATIONS**

This project has developed a time-dependent model based on thermodynamic equation to characterize the FCP behavior of Alloys 617 and 230. Since all tests were conducted in laboratory air, the tests in environment including He and steam are necessary to simulate to operating situation of NGNP system. Also effect of carbon on FCP behavior of Alloys 617 and 230 should be further studied to improve materials resistance to FCP.
Table 3: Summary of Microstructure evaluation and Properties of As-receive materials

<table>
<thead>
<tr>
<th>Material ID</th>
<th>C, wt%</th>
<th>ASTM #</th>
<th>Ave. grain Size (um)</th>
<th>M6C AF %</th>
<th>M23C6 AF %</th>
<th>Yield St. (MPa)</th>
<th>Ulti. St. (MPa)</th>
<th>Elong. (%)</th>
<th>Hardness (RB)</th>
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<tbody>
<tr>
<td>617-SM</td>
<td>0.09</td>
<td>2.55</td>
<td>205</td>
<td>None</td>
<td>1.1</td>
<td>323.33</td>
<td>772.2</td>
<td>63</td>
<td>86</td>
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<td>617-INL</td>
<td>0.05</td>
<td>2.8</td>
<td>191</td>
<td>None</td>
<td>0.82</td>
<td>333.01</td>
<td>772.2</td>
<td>60</td>
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<td>230-HA</td>
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<tr>
<td>230-INL</td>
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<td>361.97</td>
<td>820.47</td>
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<td>92</td>
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Figure 45  Microstructure of INCONEL 617 INL; (a) Optical Microstructure; (b) SEM BSD Microstructure.
Figure 46 Microstructure of INCONEL 617 SM; (a) Optical Microstructure; (b) SEM BSD Microstructure

Figure 47 Microstructure of HAYNES 230 H; (a) Optical Microstructure; (b) SEM BSD Microstructure.
Figure 48 Microstructure of HAYNES 230 INL; (a) Optical Microstructure; (b) SEM BSD Microstructure

Figure 49 Constant $\Delta K$-controlled FCP curves of Alloy 230-HA tested at different hold time and 700°C
Figure 50  Constant $\Delta K$-controlled FCP curves of Alloy 230-HA tested at different hold time and 700°C
Figure 51 FCP rate of HAYNES 230 as a function of period at 700 °C
Figure 52 Crack length vs. cycle number of alloy 617-SM at 700°C
6. LIST OF PUBLICATION, THESIS AND PRESENTATION


2) Sudin Chatterjee, Mechanical Behavior of Alloy 230 at Temperature Relevant to NGNP Program, PhD. Dissertation, University of Nevada Las Vegas, NV, May 2010.


7. ACKNOWLEDGEMENT

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8. PARTICIPANTS

1). Shawoon K. Roy, PhD student, Department of Mechanical Engineering, University of Nevada Las Vegas, supported by project from July 2010-Sept. 2012.

2). Muhammad H. Hasan, formerly PhD. student, Department of Mechanical Engineering, University of Nevada Las Vegas, supported by project from Sept. 2009-July 2010.

3). Joydeep Pal, formerly PhD student, Department of Mechanical Engineering, University of Nevada Las Vegas, supported by project from Sept. 2009- July 2010.

4). Sudin Chatterjee, formerly PhD student, Department of Mechanical Engineering, University of Nevada Las Vegas, supported by project from Sept. 2009-Dec. 2010.

9. REFERENCES


10. COST DATA

A quarterly spending plan was developed by the PI and approved by NEUP project sponsor. The actual costs reflect all money actually spent by the project in the corresponding period plus and estimated accrual amount for work performed. Table 4 show the budget data and actual cost data throughout three years period.

Table 4: Cost Data by Quarter

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<th>To</th>
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<th>Actual Spending</th>
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