CHARACTERIZATION OF FATIGUE MECHANISMS IN NI-BASED SUPERALLOYS

DISSERTATION

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By

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ABSTRACT

Ni-based superalloys are important for turbine engine airfoil applications. Historically, creep has been the main failure mode and thus creep mechanisms have been the subject of numerous studies. However, modern airfoil designs maintain cooler temperatures, and consequently creep is no longer the primary failure mode. Rather, in the cooled components, experience and experimental studies have shown that fatigue is the life-limiting factor. The changing cause of failure highlighted the need for a comprehensive study of fatigue deformation mechanisms. Information about crack propagation and the associated deformation mechanisms has allowed appropriate design changes based on fatigue as a life-limiting factor.

The focus of the study will be on a monocrystalline Ni-based superalloy, René N5, which is currently used for airfoils. Compact tension specimens were tested under cyclic loading conditions to determine the influence of microstructure and material properties on crack propagation and fatigue failure. The crack growth rate as a function of temperature, environment, frequency, and crystallographic orientation was determined. High resolution scanning electron microscopy was used to examine the
fracture surface on length scales from nano to macro. Deformation mechanisms in the plastic zone ahead of the crack tip and within the plastic wake of the crack were studied using TEM and FIB techniques.

Environment and frequency seem to have a larger effect on fatigue crack growth rates and threshold stress intensity factor ranges, while temperature and orientation effects are present, but not as dramatic. In the normal blade orientation, (001)[100], mode I crack propagation was prevalent, with mode II crack propagation found at higher $\Delta K$ values. Interdendritic particles appear to be slowing crack growth rates in the threshold region of specimens tested in air. Microstructural analysis showed no change in $\gamma'$ precipitate size or morphology with temperature or stress. From TEM investigations, it is theorized that a combination of mechanisms is occurring during testing, which is the reason there is no universal trend with temperature for the threshold stress intensity factor ranges. The mechanisms discussed include Kear-Wilsdorf locking, oxide-induced crack closure, and crack tip softening due to $\gamma'$ depletion.
Dedicated to my loving and supportive parents,
Debra and Felix Yablinsky
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CHAPTER 1

Introduction

Ni-based superalloys are used in turbine aircraft engines for vanes, disks, and blades. In particular, monocrystalline Ni-based superalloys are used in blades, which are the focus of this research. These components are used in the combustion and turbine sections of an engine. Within the combustion section, incoming compressed air is mixed with fuel and ignited. The ignited air then expands through the turbine section, extracting mechanical work, which drives earlier stages of the engine. Engine efficiency has been increased by increasing the operating temperature, with some current engines reaching up to 1400°C entering the turbine section [1]. With the melting temperature of nickel at 1455°C, modifications in blade design were a necessity in order to decrease the in-service temperature of the components. Modern designs have cooling channels in order to keep the blade cooler than a maximum acceptable temperature. The cooling channels cause a dramatic thermal gradient across the blade, with the root being as cool as 760°C. Because of service conditions, many different types of failure can occur, including creep, creep-fatigue, thermal fatigue, low cycle fatigue, high cycle fatigue, and
thermal-mechanical fatigue. Therefore, the blade components must have excellent high temperature properties, such as superb creep resistance and superb fatigue resistance.

One way to increase creep and fatigue resistance is to increase grain size [1-3]. Early turbine blades were wrought alloys, and therefore had a polycrystalline microstructure. These blades could only be used at limited temperatures because grain boundary sliding and creep contributed to failure at elevated temperatures. To reduce the number of grain boundaries, directionally solidified blades were produced using a conventional Bridgman method, which produces a columnar microstructure, but does not entirely eliminate grain boundaries. To acquire a monocrystalline structure, a modified Bridgman technique is utilized, which employs a seeding method that produces a single crystal. These types of blades exhibit better creep and fatigue resistance and are therefore optimal for turbine blade components.

Another way to increase creep and fatigue resistance is through alloying. Since the 1980s, alloying has been a highly studied area of superalloys in an attempt to add elements that will contribute to better properties. The resulting alloys are grouped into generations, which have the same base elements, with the latter generations making both Re and Ru additions. The higher the generation, the better creep resistance and fatigue resistance.

Other physical properties that are desired for blade materials are high strength, ductility, low density, low thermal expansion coefficient, high thermal conductivity, and phase stability. Low density keeps the total engine weight at a minimum while
decreasing the stress contribution of the centrifugal motion. A low thermal expansion coefficient provides excellent resistance to thermal fatigue by minimizing stresses from thermal expansion. High thermal conductivity dissipates heat faster, which minimizes temperature gradients and provides resistance to thermal fatigue. At high temperatures, the equilibrium microstructures will be most stable, so components must be designed so that structure and properties can be maintained over time [4].

Environmental effects also contribute to high temperature component failure. During operation, components are exposed to a mixture of air and fuel, and in this environment, oxidation and hot corrosion can occur. Hot corrosion is an oxidation reaction with the contaminants found in jet fuel. More recently, thermal barrier coatings have been applied in order to protect against oxidation and hot corrosion. However, these coatings are brittle and crack during service, which can initiate a crack that eventually will propagate into the blade.

The properties of Ni-based superalloys are close to the desired properties for a turbine blade, and are therefore used commercially as a high-temperature material. Until recently, creep failures have been the main life-limiting factor for turbine blades, and while many creep studies have been performed, more fundamental fatigue research has not been done. The following chapters focus on Ni-based superalloys and their elevated temperature mechanical behavior in order to gain a comprehensive characterization of deformation mechanisms in different operating regimes.
CHAPTER 2

Background

2.1. Ni-based Superalloys

2.1.1. Alloying

One reason that Ni-based superalloys possess such excellent high temperature properties is their high tolerance for alloying additions. This tolerance decreases the amount of detrimental phases that form during casting and in service operation. Ni-based superalloys can be comprised of upwards of ten alloying elements. Many additions are made to improve specific properties that, in turn, help maintain component integrity at higher temperatures. For example, chromium, a solid solution strengthener, is added in order to increase hot corrosion resistance [1]. Aluminum and titanium have been added in order to precipitation harden the material [1, 4], while other elements, such as rhenium and ruthenium, increase oxidation resistance [1, 5, 6].
Large amounts of alloying additions have lead to different superalloy classes, or generations. The first monocrystalline Ni-based superalloys are considered the first generation and contain 10 wt% chromium. In the later generations, the wt% of Cr is decreased and rhenium and ruthenium are added in place of the Cr. The wt% for Cr, Re, and Ru are seen in Table 2.1., and the total wt% of all three elements combined is approximately 10 wt% for each generation.

**Table 2.1.** Selected list of components for Ni-based superalloys which determine the generation.

<table>
<thead>
<tr>
<th>Generation</th>
<th>Cr (wt%)</th>
<th>Re (wt%)</th>
<th>Ru (wt%)</th>
</tr>
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<tr>
<td>1st</td>
<td>10</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2nd</td>
<td>6</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>3rd</td>
<td>3</td>
<td>6</td>
<td>0</td>
</tr>
<tr>
<td>4th</td>
<td>2</td>
<td>5-6</td>
<td>3</td>
</tr>
</tbody>
</table>

2.1.2. Microstructure

Ni-based superalloys have a two phase microstructure. The matrix phase, or \( \gamma \) phase, has an fcc lattice and is commonly made up of nickel, cobalt, chromium, molybdenum, ruthenium, rhenium, and tungsten. Most of the alloying additions have similar atomic radii to nickel and can easily substitute for a nickel atom on the fcc lattice. The \( \gamma \) phase is the continuous phase in the microstructure and must therefore be stable
throughout the lifetime of the component or else the high temperature properties will diminish. The second phase, which forms a coherent precipitate, is called the $\gamma'$ phase. This ordered L1$_2$ structure is mostly comprised of nickel and aluminum in the form of Ni$_3$Al, however, other elements such as niobium and tantalum also segregate to the $\gamma'$ phase. This phase forms as cuboidal $\gamma'$ precipitates that often align along the [100] direction. The $\gamma/\gamma'$ microstructure can be seen in Figure 2.1. In monocrystalline Ni-based superalloys, the volume fraction of the $\gamma'$ phase is about 70%. Even though the precipitates are coherent, they have a different lattice parameter than the matrix phase, causing a small degree of misfit, which will be discussed shortly. Most commercially available monocrystalline alloys have negative misfit ($a_{\gamma'} < a_{\gamma}$) [1].

![Figure 2.1](image.png)

**Figure 2.1.** Two phase microstructure common to Ni-based superalloys. The $\gamma$ phase is light gray while the $\gamma'$ phase is darker gray. Note the cuboidal $\gamma'$ aligns with the cube directions.
Other microstructural features also form, such as carbides, borides, and topologically close packed (TCP) phases. Carbides and borides, which contribute strength and high temperature stability, can be found throughout the alloy. Some of the metals that promote carbide formation are chromium, molybdenum, titanium, tantalum, and hafnium. The crystallographic structure, volume percent, and position of the carbides and borides depends on the processing conditions. On the other hand, TCP phases, which are comprised of elements such as rhenium, molybdenum, tungsten, and chromium, are detrimental to Ni-based superalloys. In order to minimize TCP formation the alloying additions that form them must be kept to limited quantities [1], which is unfortunate because in most cases the solubility of these beneficial elements is higher than the amount actually put into the alloy. In the end, though, the negative effect of the formation of the TCP phases outweighs the additional strengthening benefits given by a higher concentration of the solid solution elements.

2.1.3. Misfit

As mentioned earlier, the $\gamma$ and $\gamma'$ phases have slightly different lattice parameters. The misfit between the phases is defined as

$$\delta = 2 \left[ \frac{a_{\gamma'} - a_{\gamma}}{(a_{\gamma'} + a_{\gamma})} \right]$$  \hspace{1cm} (2.1)
where \( a_\gamma \) and \( a_\gamma' \) are the lattice parameters for the respective phases, and the sign can be either negative or positive. In order to maintain the coherent nature of the microstructure, the magnitude of the lattice misfit must be low. The magnitude and sign of the misfit determines the \( \gamma' \) precipitate morphology. Cuboidal \( \gamma' \) is found in both negative and positive misfit alloys, but the smaller the magnitude of the misfit, the larger the precipitates must be in order to form the cuboidal morphology [1]. Lattice misfit also causes coherency strains, which can impede dislocation motion and precipitation harden the alloy. The amount, size, and morphology of the precipitates will therefore play a role in the mechanical behavior of the material [7].

In Ni-based superalloys, the lattice misfit will change with temperature, and it has been shown that \( \delta(T) \) increases slightly with temperature until around 625°C, then decreases. Gornostyrev investigated this phenomenon and attributed it to the following: 1. the difference in thermal expansion coefficients of the phases, and 2. the changes in alloy composition due to the redistribution of alloying components with temperature. Experiments have shown that the thermal expansion coefficient of \( \gamma \) is greater than that of \( \gamma' \). The difference in thermal expansion coefficients play a role at temperatures less than 0.6\( T_m \), while compositional changes will not affect \( \delta(T) \) because diffusion rates at these temperatures are very slow. Above 0.6\( T_m \), the redistribution of alloying components becomes the dominant factor, and \( \delta(T) \) is defined by the shape of the \( \gamma/\gamma' \) gap in the Ni-Al phase diagram [8, 9].
In monocrystalline alloys, the magnitude of the $\gamma/\gamma'$ misfit must be small in order to minimize interfacial energy so that coarsening is restricted. Precipitate coarsening decreases the strength of the material, while directional coarsening can cause a detrimental rafted microstructure. The rafted structure, which has been discussed in literature [3, 10-15], will not be considered in this study because it was not observed.

2.1.4. Dislocation Motion

In fcc alloys, the preferred slip systems are $\{111\}<110>$ type, and dislocation movement on the $\{111\}$ planes is termed octahedral slip. A perfect dislocation moving through the $\gamma$ phase will encounter $\gamma'$ particles, and in order for the dislocation to continue moving it must transfer to the $\gamma'$ phase. In particular, as an $a/2<110>$ type dislocation moves into the $\gamma'$, an anti-phase boundary (APB) is formed in the wake. An APB is an unfavorable high-energy structure that is formed when the $\gamma'$ ordering is destroyed along the glide plane, forming a boundary between two otherwise perfect crystals. Since the APB is unfavorable, further dislocation motion is hindered. In order to continue deformation, a second dislocation must also pass through the precipitate. The dislocation-APB-dislocation structure must move through the $\gamma'$ phase in order to deform the $\gamma'$ lattice back to a properly ordered structure [1, 16-18]. Within the $\gamma'$ phase, the perfect $\gamma$ dislocation is called a superpartial, while the dislocation pair is called a superdislocation.
If the superpartial dislocations further dissociate in the \( \gamma' \) phase, other types of faults can form. A complex stacking fault (CSF) can form by \( a/6\langle112\rangle\{111\} \) shear displacement, which changes nearest-neighbor configurations. Other formations are the superlattice intrinsic stacking fault (SISF) or the superlattice extrinsic stacking fault (SESF), which are analogous with common intrinsic and extrinsic stacking faults found in fcc materials. The SISF and SESF are produced by shear displacements on \{111\} planes of type \( 1/3\langle112\rangle \) and do not change the nearest-neighbor configuration [1, 19].

2.1.5. Yield Strength Phenomenon

The formation of \( \gamma' \) by a precipitation hardening heat treatment is crucial for attaining the high temperature strength needed for Ni-based superalloy applications. During mechanical deformation, strength increases with temperature to a maximum (around 800°C) and then decreases. Common to all \( L1_2 \) ordered materials, this trend is called the yield stress phenomenon [20]. The reason for the anomalous yield stress behavior is as follows.

In general, dislocations move on the planes with the lowest critical resolved shear stress, and in fcc materials with (001) as the tensile direction, the \{111\} planes fit this requirement. At low temperatures, dislocations will move between particles or cut through the particles by shearing. In both cases, the stresses needed to continue dislocation motion are increased because of the formation of an APB. When stress is
applied to the material, the anisotropy of the APB energy and the elastic anisotropy associated with the dislocations promote the cross-slip of some segments of the superpartial dislocations from the \{111\} slip plane to the \{100\} slip plane. Additionally, part of the APB cross-slips with the segment, which reduces the overall energy of the defect configuration because APB energy on \{100\} planes is lower than on \{111\} planes. Even though this configuration is favorable, with further applied stress the dislocation segment becomes pinned in what are called Kear-Wilsdorf locks. This is because the critical resolved shear stress on \{100\} planes is very high and the dislocation segments can no longer shear the precipitate. Until the point of maximum strength, this cross-slip mechanism increases the strength of the material by making dislocation motion more difficult. After the point of maximum strength in the materials, which is usually at elevated temperatures, the material can use some of the thermal energy to activate dislocation motion. The energy is used for either of the following: cross-slip of the dislocations back onto the \{111\} planes (where the APB energy is higher), or drag of the APB through the \(\gamma'\) phase. Another observation at higher temperatures is that the critical resolved shear stress for all sets of planes decreases with temperature, so the energy needed to slip along \{100\} planes will decrease, making dislocation motion easier. Once the material starts to deform by cuboidal slip (along \{100\} planes), the locking mechanism can no longer contribute to the hardening of the material, and therefore the yield stress will decrease [1].
2.1.6. Precipitation Hardening

Precipitation hardening also contributes to the strength of Ni-based superalloys, where the \( \gamma' \) precipitates act as the hardening phase. As discussed in Section 2.1.4., dislocations that shear particles couple into a dislocation-APB-dislocation configuration [1, 16-18]. Forces on the dislocations will control the onset of particle shearing and consist of 1. forces due to applied shear stress, 2. forces from dislocation-dislocation interaction, and 3. pinning forces that are a consequence of APB energy. The first dislocation must bow between the precipitates in order to counteract the pinning forces by increasing the line tension. The second dislocation will be straight since it will be easier to move through the precipitates based on the fact that the dislocation is removing the APB and lowering the overall energy [16, 18]. For small precipitate sizes, the dislocations can be spaced further apart, or weakly coupled, and an increase in precipitate size yields an increase in strength. When the particle size equals \( \frac{2T'}{\gamma_{APB}} \), where \( T \) is line tension and \( \gamma_{APB} \) is the anti-phase boundary energy, the dislocations can cut through the particle at a closer spacing, and are termed strongly coupled dislocations [1, 17]. At a critical diameter, the dislocations stop shearing through the particles and the Orowan looping mechanism is favored [17]. In this mechanism, the dislocation forms expanding loops around the particles. The dislocation eventually annihilates as it continues to bow around the particle, which leaves a dislocation loop.
around the precipitate and a dislocation that continues through the matrix. The resulting dislocation loop around the particle will impede further dislocation movement around the particle (ignoring any cross-slip), by repelling other approaching dislocations, adding another strengthening mechanism [7]. All of these strengthening mechanisms have been observed experimentally [16, 18].

2.2. Fatigue Crack Growth

2.2.1. Fatigue Crack Propagation

Crack propagation is important in low cycle fatigue because it is the dominant portion of the fatigue life. The focus of crack growth is on long cracks, where crack growth rates are independent of crack size and linear elastic fracture mechanics can be applied. In general, the Paris law gives the fatigue crack growth increment (change in crack length per cycle) as

$$\frac{da}{dN} = C(\Delta K)^m$$  \hspace{1cm} (2.2)

where C and m (m=2-4 for ductile materials) are scaling constants influenced by material, microstructure, temperature, load ratio, and environment. $\Delta K$ is the stress intensity factor range and is defined as
\[ \Delta K = K_{\text{max}} - K_{\text{min}} = Y\Delta\sigma \sqrt{a} \]

where \( Y \) is a geometrical factor based on the ratio of crack length to the width of the specimen, \( \Delta\sigma = \sigma_{\text{max}} - \sigma_{\text{min}} \), and \( a \) is the crack length. Note that this is the most general form of the equation, and for compact tension specimens, which are used in this study, the stress intensity factor range equation is much more complex. The Paris law is applicable under single mode far-field loading with a constant load ratio, \( R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}} \).

Stable crack growth occurs when \( k_{\text{max}} \) (\( k_{\text{max}} = \frac{\Delta K}{(1-R)} \)) is less than the fracture toughness of the material, \( k_{\text{IC}} \). Total fatigue life can be derived from the Paris law and takes the final form of

\[ N_f = \frac{1}{CY^2(\Delta\sigma)^2} \pi \ln \left( \frac{a_f}{a_o} \right) \]

where \( a_f \) is the crack length critical to failure and \( a_o \) is the initial crack length. Again, this is the simplest form of the equation and is much more complicated for specific cases.

The fatigue crack path is categorized by the type of loading: mode I, which is planar crack growth, often including fatigue striations, and mode II, which is crystallographic fatigue crack growth. In mode I, crack growth consists of simultaneous or alternating flow along two slip systems on a small scale, resulting in a macroscopically planar crack path that is perpendicular to the loading axis. Mode I is also the type of crack growth where striations can be seen. Striations are formed during cycling as the crack opens and blunts. Upon closure, the compressive forces are not high enough to
plastically deform the crack tip back to the original formation, so the crack advances a distance. The striations are seen as parallel ridges that are perpendicular to the crack growth direction, with the striation spacing directly relating to the distance the crack has advanced during that cycle. Not all fatigued samples will exhibit striations, since striation development is strongly influenced by the value of $\Delta K$, stress state, environment, and alloy composition. In mode II, the crack extends by single shear in the direction of the primary slip system. With a loading axis of $<001>$ in fcc materials, the single shear happens on $\{111\}$ planes, which leads to a macroscopically zigzag crack path.

For stable growth propagation, a schematic of $\log da/dN$ vs $\log \Delta K$ is shown in Figure 2.2. Region A exhibits a slow crack growth rate. The value of $\Delta K$ where the crack growth rate decreases to a negligible level is called the fatigue threshold, $\Delta K_o$, and is $R$ dependant. Below this $\Delta K_o$, the crack will not grow or grows at undetectable rates. $\Delta K_o$ will decrease with increasing $R$ until a critical $R$ value when $\Delta K_o$ becomes independent of $R$. When $\Delta K_o$ is independent of $R$, $\Delta K_o=\Delta K_{th}$. Region B exhibits intermediate crack growth rates, and since the Paris law describes this region well, it is often called the Paris Region. Region C has high growth rates and leads to catastrophic failure at a high $\Delta K$. Here, static fracture modes, such as cleavage or fibrous failure, occur in addition to striation growth. The $K$ at failure is termed $K_c$, which is the dynamic fracture toughness of the material.
Figure 2.2. Schematic of fatigue crack growth rate vs stress intensity factor range. Plot is shown as log $da/dN$ vs log $\Delta K$.

Crack closure can affect the material response during fatigue crack growth. Closure can be caused by roughness-induced crack closure, cyclic plasticity-induced crack closure, or oxide-induced crack closure [21]. Region A will be most affected by closure, since loads are small and the crack tip opening displacement is not large. If one of these closure mechanisms is operative, it will decrease the effective stress intensity factor range, $\Delta K_{\text{eff}}$, which is defined as:
\[ \Delta K^{\text{eff}} = K_{\text{max}} - K_{\text{cl}} \]  

where \( K_{\text{cl}} \) is the \( K \) associated with the start of closure. \( K_{\text{cl}} \) is the stress intensity factor where closure begins and will be greater than \( K_{\min} \). Thus, \( \Delta K^{\text{eff}} \) shifts the fatigue crack growth plot to smaller \( \Delta K \) values, with a larger shift near threshold.

2.3. Fatigue in Ni-based Superalloys

2.3.1. Influence of Temperature

At nominal temperatures, a decrease in temperature will decrease ductility and therefore decrease overall fatigue life [1, 22]. Fracture toughness decreases with decreasing temperature, and therefore catastrophic failure of a specimen will occur at a lower \( \Delta K \). The decreased plasticity also affects the fatigue crack propagation rate, since decreasing the temperature will decrease the plastic zone size as long as the ductile mechanism for growth is still active. A decrease in plastic zone size will make it difficult to advance cracks, and therefore the threshold value will increase [21].

While effects at nominal temperatures are fairly straightforward, the effect of elevated temperatures on fatigue crack growth is quite complicated. At high temperatures, time-dependent processes, like creep and oxidation, will contribute to crack propagation. These processes will be affected by cycle frequency and load ratio,
with lower frequency being closer to monotonic loading, and load ratio influencing crack oxidation [21]. The effect of oxidation will be discussed in the next section as an environmental factor.

At elevated temperature, a number of studies found that decreasing the temperature will reduce the threshold value for fatigue crack propagation [23-25], which contrasts the trend seen at nominal temperatures. A few authors have justified the difference by noting that there is less oxide at the lower temperatures, while at higher temperatures, the growing oxide scale is filling the crack tip and thus reducing the effective stress intensity factor [23-25].

Previous monocrystalline Ni-based superalloy studies on Rene N4 and Mar-M200 found that at intermediate temperatures (~750°C) cracks propagated in mode II, while at higher temperatures (~950°C), crack propagation occurred in mode I [26, 27]. A different study on Mar-M200, which tested over a larger number of temperatures, narrows down the crack growth mechanism change to somewhere between 760°C and 850°C [28]. However, all of these studies were low cycle fatigue (LCF) studies. In studies where fatigue crack growth (FCG) tests were performed, all specimens tested at a temperature at or above 550°C had crack propagation in mode I [23-25, 29, 30]. These authors additionally note that a transition in crack propagation to the octahedral, mode II type crack growth happens in the high ΔK region. It is possible that even though the first set of authors saw octahedral facets in their intermediate temperature tests, they were pushed into the mode II cracking regime because they were at high stresses. LCF
testing done by Chieragatti on round bar specimens of Mar-M200 at 650°C found planar cracking, arguing that this type of fatigue crack growth should be prevalent during mode I loading. The author also noted that mode II cracking will only occur in the last stage of fatigue crack growth, referring to the higher $\Delta K$ region [31].

Additionally, Henderson and Martin found that in FCG tests, the $\Delta K$ where the crack propagation changed from mode I to mode II was the same for 650°C and 850°C [25]. It can therefore be concluded that the crack path depends more on the loading than the temperature, at least at temperatures above 650°C.

A summary of the literature on temperature can be found at the end of the chapter in Table 2.2., which was created as a quick reference guide.

### 2.3.2. Influence of Environment

At elevated temperatures oxide layers can form on air exposed surfaces of Ni-based superalloys, which include the wake of the crack. The oxide thickness varies along the wake of the crack with exposure time, and the extent of oxidation is influenced by alloy composition, microstructure, environment, $\Delta K$, and load ratio. The oxide layer plays a significant role in oxide-induced closure in near-threshold crack propagation response. The relationships are complicated, but oxide-induced crack closure is promoted by moisture containing environments, elevated temperatures, low
load ratios, low $\Delta K$ levels, low cyclic frequencies, high strain ranges, and lower strength microstructures [21].

Specifically, at low $\Delta K$ levels and because of the nature of fatigue testing, the oxidized surfaces will repeatedly contact. In some cases, the oxide can break and the exposed surface can again oxidize. This mechanism, termed fretting, can cause a thicker oxide layer to form than would occur when leaving a portion of un-oxidized material in air for the same length of time. The oxide acts as a wedge that has been inserted into the crack and causes crack tip blunting, which decreases the stresses seen at the crack tip, reducing crack growth rates. Therefore, the oxide-induced closure mechanism has a larger effect near threshold and will cause the $\Delta K_{th}$ value to be higher than if closure was not a working mechanism [21].

In contrast, oxides have also been found to decrease the fatigue lifetimes of components because of increased crack growth rates. Oxide layers are complex structures commonly made with aluminum, which diffuses to the surface from $\gamma'$ particles. The Al diffusion creates a problem at high temperatures, because a region depleted of $\gamma'$ can form around the crack. The crack will therefore have little resistance to crack propagation since the material ahead of the crack tip decreases in strength. When this occurs, crack propagation rates drastically increase [32, 33].

During low cycle fatigue testing, Defresne and Remy found that fatigue lives of specimens decrease when cracks initiate at surface porosity in comparison with sub-surface porosity [34]. The sub-surface initiation sites will behave like a vacuum initiated
crack since these pores are not linked with the surface and therefore will not form oxide layers. The conclude that because there are no oxide effects in vacuum the crack propagation rate is slower than in air [34], implying that the oxide increases the crack growth rate. No investigation was done in order to confirm that there were no environmentally induced changes to the microstructure, mainly $\gamma'$ depletion, which would be expected in specimens where the crack growth rate was increased in air.

A summary of the literature on environment can be found at the end of the chapter in Table 2.2., which was created as a quick reference guide.

2.3.3. Influence of Frequency

Studies have shown that frequency can affect crack growth rates in the threshold region [23, 25]. A decrease in frequency reduces crack growth rates, therefore increasing the stress intensity factor at threshold. Lower frequencies will be closer to monotonic loading conditions than higher frequencies. Henderson and Marin propose that because there is more time in tension during a cycle, this gives dislocations more time to cross-slip into the Kear-Wilsdorf configuration [25]. Since cross-slip and subsequent movement on cube planes is a slow process [35], this theory makes some sense. However, this mechanism could breakdown at higher temperatures, given the added thermal energy as discussed in Section 2.1.5. Additionally, at lower frequencies, there is more time for oxide to form on the surface. As discussed previously, thicker
oxide will decrease the effective stress intensity factor, causing lower crack growth rates at increased ΔK values.

A low cycle fatigue study by Leverant et al. tested round bar specimens over a frequency range of 0.033Hz to 1058Hz [28]. The results led them to conclude that higher frequencies and lower temperatures lead to more octahedral cracking, while lower frequencies and higher temperatures lead to more planar cracking. The effect of temperature on crack path has been discussed previously, so only the effect of frequency will be discussed here. In the study, fatigue life (in hours) decreased by four orders of magnitude when increasing frequency through the range tested. However, the results were on the same magnitude until around 8Hz, where the specimen life-time began decreasing steadily. This means that a continued increase in frequency leads to an increased crack growth rate. Additionally, TEM micrographs of the dislocation substructure were shown for a single temperature, 600°C. At low frequencies, a homogeneous distribution of dislocations was seen. As frequency increased, the dislocation content was reduced, and eventually was confined to what appeared to be planar bands. From these dislocation structures, they conclude that the planar bands will lead to octahedral cracking and the homogeneous substructure leads to planar cracking. Therefore, lower frequencies lead to mode I fracture, while higher frequencies lead to mode II fracture [28].

A summary of the literature on frequency can be found at the end of the chapter in Table 2.2., which was created as a quick reference guide.
2.3.4. Influence of Orientation

In monocrystalline form, nickel is elastically anisotropic and the stiffness of the material depends on crystallographic orientation with respect to the loading direction. Reed determines the stiffness along some directions for pure nickel, which will be reflected in the Ni-based superalloys. Calculations showed that the <100> directions have the smallest stiffness, the <111> directions have the largest stiffness [1]. Low cycle fatigue testing on monocrystalline Ni-based superalloys showed that fatigue life will depend on the orientation of the loading axis with respect to the crystallographic orientation. The elastically soft directions, <100>, displayed the best fatigue life, while the <111> directions had the worst fatigue life [1, 20, 25, 34, 36].

A study by Müller et al. showed that a change in load axis from <001> to <011>, having crack propagation direction <010> in both cases, did not influence fatigue crack growth rate [24]. This observation can be explained using the work by Wen and Yue [37]. Their study investigated the differences in crack propagation path when varying the loading axis. With a change only in the crack loading direction ([001] to [011]) with a propagation direction of <100>, both specimens failed in stage I on crystallographic slip planes. However, the orientation differences resulted in different orientations of the active slip planes to the loading axis, and therefore the crack path was along different angles to the loading axis all though the fracture mode was the same. Because the
fracture modes were the same and along the same family of planes the fatigue crack growth rates were similar [37].

A study by Anton tested cylindrical specimens with a loading direction of [001] at 650°C. Results showed that crack growth from a pore was faster along the <100> directions than along the <110> directions. This difference was attributed to the differences in elastic modulus between the two directions. The stiffness of the [110] direction is higher than in the [100] direction, and therefore the crack propagation in the [110] direction will be slower. Furthermore, the modulus in the direction of the crack growth influences the crack tip opening displacement, since the material in front of the crack will be in compression when the crack opens [20].

Secondary orientation was also investigated by Ai et al using single edge notch specimens. The [110] direction had the slowest fatigue crack propagation rate, while the [100] direction had a much faster propagation rate [36], which agrees with the study by Anton. This orientation effect however was less obvious than the same results seen at a lower temperature, where the same orientation dependence was seen to a higher degree by Defresne and Remy. In this study, the difference in crack propagation rates was attributed to crack closure mechanisms [38]. The authors mention several types of crack closure mechanisms, but do not go into great detail as to which mechanisms they think played a role in their study. This is most likely because each mechanism is complex and multiple mechanisms may be working at the same time.
A summary of the literature on orientation can be found at the end of the chapter in Table 2.2., which was created as a quick reference guide.

2.3.5. Deformation Mechanisms

During fatigue, dislocations form in both horizontal and vertical $\gamma$ channels due to tensile and compressive stresses imposed by the cyclic nature of testing [39]. A study done by Ott et. al., studied dislocation movement through the microstructure ahead of the crack tip [22]. In specimens with cuboidal $\gamma'$ the crack propagated through the horizontal $\gamma$ channels and along $\gamma/\gamma'$ interfaces. The authors found that most of the dislocations remain confined within the $\gamma$ phase because shearing of the $\gamma'$ phase forms a high energy APB. Therefore, in this microstructure, the crack follows the $\gamma$ channels and can propagate rather fast since there are minimal interruptions by the $\gamma'$ phase. In the cuboidal morphology the crack also branched onto other parallel planes since there are also vertical $\gamma$ channels and there isn’t a perfect arrangement of cuboidal particles [22, 36].

In general, dislocation structures seen under low cycle fatigue conditions have consisted of dislocations contained within the $\gamma$ channels, with minimal shearing of the $\gamma'$ particles. The dislocations that do shear the $\gamma'$ particles are those formed in order to relieve misfit. During cyclic loading, these dislocations will travel and and slip bands can be formed that shear the $\gamma'$ particles. \{111\}<101> type superdislocations were found in
the $\gamma'$ phase on octahedral planes after a large number of cycles [40, 41]. In some cases, 
{100}<011> type dislocations were found with the same burgers vector as the superdislocations, indicating that cross slip had occurred [40].

Dislocation movement is also affected by temperature. At low temperatures, dislocations are expected to shear through particles, and coarsening should not be observed [39, 42]. At high temperatures, diffusion plays a larger role, and the climb of dislocations will be easier. Leverant et. al. have observed dislocation structures over a range of temperatures [28]. At low temperatures, dislocations form shear bands along the slip planes that the crack path likes to follow, causing octahedral fracture. At high temperatures, dislocations can climb and slip more easily and form a homogeneous substructure, leading to a crack path that is perpendicular to the loading axis [28].

2.4. Literature Summary

A reference table for the literature is presented in Table 2.2. LCF is the acronym for low cycle fatigue; FCG for fatigue crack growth. This information is taken from references [1, 20-34, 36-38].
Table 2.2. Summary of the literature survey on fatigue in Ni-based superalloys and the effect of temperature, environment, frequency, and orientation.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>↑T ↑ΔK_{th}</th>
<th>LCF: ≥ 850°C mode I ≤ 750°C mode II</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>FCG: ≥ 550°C mode I 25°C mode II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Note: higher ΔK = mode I</td>
</tr>
<tr>
<td>Environment</td>
<td>Air: ↑oxidation with ↑T</td>
<td>Oxide-induced closure ↓stresses ↓crack growth rate</td>
</tr>
<tr>
<td></td>
<td>Vacuum: No oxidation</td>
<td>Denuded zone (γ' depletion) ahead of crack tip ↑ crack growth rates</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Crack propagation slower than air with presence of denuded zone</td>
</tr>
<tr>
<td>Frequency</td>
<td>↓freq ↑ΔK_{th}</td>
<td>↓freq and ↑T = mode I</td>
</tr>
<tr>
<td></td>
<td>↓freq ↑oxide</td>
<td>↑freq and ↓T = mode II</td>
</tr>
<tr>
<td></td>
<td></td>
<td>↑freq ↑crack growth rate because there is less time for cross-slip</td>
</tr>
<tr>
<td>Orientation</td>
<td>Changing load axis</td>
<td>No influence on crack growth</td>
</tr>
<tr>
<td></td>
<td>Changing crack growth direction</td>
<td>Crack growth rates highest in [100] directions</td>
</tr>
</tbody>
</table>
CHAPTER 3

Materials and Experimental Procedure

3.1. Material

3.1.1. Alloy

The material used for this work was monocrystalline Ni-based superalloy René N5. The nominal composition of the alloy can be seen in Table 1 [43]. Monocrystalline slabs of René N5 were made at GE Aviation using the modified Bridgman technique. A schematic of a slab is in Figure 3.1. Slabs were grown so that the z-directions were near the [001] and [011] directions. Slabs were selected so that the deviation of the actual orientation from the loading axis would be no greater than 10 degrees, a standard deviation used within the industry. The slabs were given an industry standard heat treatment before being acquired by Ohio State University.
Table 3.1. Nominal composition in weight percent of Ni-based superalloy René N5 [43].

<table>
<thead>
<tr>
<th>Alloying Addition</th>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ta</th>
<th>Re</th>
<th>Hf</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition in wt%</td>
<td>61.8</td>
<td>7.0</td>
<td>8.0</td>
<td>2.0</td>
<td>5.0</td>
<td>6.2</td>
<td>7.0</td>
<td>3.0</td>
<td>0.2</td>
</tr>
</tbody>
</table>

3.1.2. Slab Orientation

The slab orientations were confirmed in both the x-direction and z-direction, as defined in Figure 3.1. To do this, a small section of each slab was cut parallel to the yz plane. These sections were cut a second time, parallel to the xy plane. The pieces were mounted in a parallel polishing machine and polished through successively higher SiC grit paper, from 400 through 1200 grit. The sections were final polished using 0.02μm colloidal silica on a polishing cloth and mounted onto stubs using Leitseiber silver paint for observation in the scanning electron microscope (SEM). Orientation image microscopy was performed to get the orientation of the slab. The misorientation with respect to the z direction and the x direction are seen in Table 3.2.
Figure 3.1. Schematic of as-received Ni-based superalloy slabs. The coordinate system referred to in the text is shown on the slab.

Table 3.2. Slab misorientations with respect to the z direction and x direction, defined in Figure 3.1.

<table>
<thead>
<tr>
<th>Slab ID#</th>
<th>Misorientation from z</th>
<th>Misorientation from x</th>
</tr>
</thead>
<tbody>
<tr>
<td>B2211</td>
<td>9.4° from (001)</td>
<td>10.0° from [100]</td>
</tr>
<tr>
<td>122</td>
<td>10.0° from (001)</td>
<td>7.3° from [100]</td>
</tr>
<tr>
<td>B2231</td>
<td>2.4° from (001)</td>
<td>4.5° from [100]</td>
</tr>
<tr>
<td>3-024</td>
<td>5.1° from (001)</td>
<td>8.6° from [100]</td>
</tr>
<tr>
<td>3-023</td>
<td>2.4° from (001)</td>
<td>3.4° from [110]</td>
</tr>
</tbody>
</table>
3.2. Mechanical Testing

3.2.1. Specimen Design and Preparation

Compact tension, or C(T), specimens were machined from the slabs following ASTM Standard E 647[44]. Two sizes of specimens were made, $W = 40$ mm and $W = 20$ mm, the dimensions of the latter being shown in Figure 3.2. $W$ is the distance from the load line to the back face of the specimen. The notch was made to be the width of the wire used during electrical discharge machining (EDM), approximately 0.5mm. Specimens were machined so that testing could be done in the following orientations, given as (loading axis)[crack growth direction]: (001)[100], (001)[110], (011)[100], (011)[110]. The sides of the specimens were polished using successively higher SiC grit paper, from 320 to 1200 grit, for in-situ crack growth observations. The samples were then final polished using 0.02μm colloidal silica until a mirror finish was established. Specimens dimensions were then measured (in mm) for use in the mechanical testing software.
3.2.2. Direct Current Potential Drop Crack Growth Measurements

The direct current potential drop (DCPD) method is used to measure crack length during fatigue crack growth testing. This method is used for conductive specimens, where resistance, which changes with crack length, can be measured. It is used to provide an accurate average through-thickness crack length in real time over the course of the test. However, this technique is sensitive to lead placement, and electric noise can add scatter to the measured crack length. The measured specimen dimensions were used to calculate precise lead placement for DCPD leads in accordance with ASTM Standard E 647 [44]. Before welding, specimens were final cleaned using acetone, and
were handled with gloves afterward to prevent contamination with oils. Wires for DCPD, along with thermocouples for temperature control, were spot welded on each specimen as shown in Figure 3.3. For the placement of the voltage measurement leads, \(Z=5\text{mm}\) for \(C(T)\) specimens with \(B=10\text{mm}\), and \(Z=3\text{mm}\) for specimens with \(B=5\text{mm}\). Types of wire used for each application can be seen in Table 3.3. During testing, a current of 5 amps was run through the specimen. A reference voltage was determined and compared to the voltage measured across the crack to determine real time crack length, \(a\), using ASTM Standard E 647 [44]. Note that the DCPD technique is valid for cracks growing perpendicular to the loading axis, and a deviation greater than 20° will cause the measurements to be invalid.

### Table 3.3. Types of wires used for DCPD.

<table>
<thead>
<tr>
<th>Lead</th>
<th>Type of Wire</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current</td>
<td>Chromel 0.035”</td>
</tr>
<tr>
<td>Measured</td>
<td>Chromel 0.010”; 30 gauge (Air), 24 gauge (Vacuum)</td>
</tr>
<tr>
<td>Reference</td>
<td>Chromel 0.010”; 30 gauge (Air), 24 gauge (Vacuum)</td>
</tr>
<tr>
<td>Thermocouple</td>
<td>K-type; 30 gauge (Air), 24 gage (Vacuum)</td>
</tr>
</tbody>
</table>
Figure 3.3. Welding locations for the DCPD leads. 1: Voltage Measurement location. 2: Input Current Location. 3: Reference Lead #1 Location. 4: Reference Lead #2 or Thermocouple Location.

3.2.3. Fatigue Crack Growth Testing

Table 3.4. shows the test matrix utilized to characterize the effects of temperature, environment, frequency, and orientation on fatigue crack growth. An intermediate range of temperatures was chosen in order to try to separate the effects of fatigue from effects of creep, and coincidentally are the temperatures that the
Table 3.4. Specimen matrix. Test conditions for each specimen are listed.

<table>
<thead>
<tr>
<th>Specimen #</th>
<th>Temp (°C)</th>
<th>Freq (Hz)</th>
<th>Environment</th>
<th>Orientation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>550</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>2</td>
<td>650</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>3</td>
<td>750</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>4</td>
<td>850</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>5</td>
<td>550</td>
<td>10</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>6</td>
<td>650</td>
<td>10</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>7</td>
<td>750</td>
<td>10</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>8</td>
<td>850</td>
<td>10</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>9</td>
<td>550</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>10</td>
<td>650</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>11</td>
<td>750</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>12</td>
<td>850</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>13</td>
<td>550</td>
<td>10</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>14</td>
<td>650</td>
<td>10</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>15</td>
<td>750</td>
<td>10</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>16</td>
<td>850</td>
<td>10</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>17</td>
<td>550</td>
<td>10</td>
<td>Air</td>
<td>(001)[110]</td>
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<tr>
<td>18</td>
<td>750</td>
<td>10</td>
<td>Air</td>
<td>(001)[110]</td>
</tr>
<tr>
<td>19</td>
<td>550</td>
<td>10</td>
<td>Air</td>
<td>(011)[100]</td>
</tr>
<tr>
<td>20</td>
<td>750</td>
<td>10</td>
<td>Air</td>
<td>(011)[100]</td>
</tr>
<tr>
<td>21</td>
<td>550</td>
<td>10</td>
<td>Air</td>
<td>(011)[110]</td>
</tr>
<tr>
<td>22</td>
<td>750</td>
<td>10</td>
<td>Air</td>
<td>(011)[110]</td>
</tr>
<tr>
<td>23</td>
<td>550-Interrupt</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>24</td>
<td>750-Interrupt</td>
<td>0.5</td>
<td>Vacuum</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>25</td>
<td>550-Interrupt</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
<tr>
<td>26</td>
<td>750-Interrupt</td>
<td>0.5</td>
<td>Air</td>
<td>(001)[100]</td>
</tr>
</tbody>
</table>
dovetail region of the blade experiences during service. Environments of air and vacuum were selected to reflect in-service crack growth conditions for surface initiated and internal initiated cracks, respectively. Frequencies and orientations were also chosen to reflect in-service conditions.

A load frame with a vacuum furnace and 25 kN load cell limit was used for vacuum testing. An air furnace frame similar to an MTS 810 system was used for air testing. The software used to control the tests is WinMate 1.0, by UDRI, Dayton, OH. The specimens underwent three different loading regimes, ending in a portion that was precracked (part I), a portion that was allowed to run to threshold (part II), and a portion that was run to high ΔK (part III). Figure 3.4. shows an image of a fracture surface, with the crack front lines from the different parts of the test highlighted. All tests were run using a sinusoidal waveform and an R- ratio (R = σ_{min}/σ_{max}) of 0.1. The real time crack length, taken by DCPD, was used by the software to determine K values using:

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

(3.1)

where P is the load, B is the specimen thickness, and \( \alpha = a/W \).

Specimens were precracked at temperature at a frequency of 20Hz in load control until a sharp crack was established. According to ASTM E 647 [44], this is when the precrack length is 0.1B, the width of the notch, or 1.0mm, whichever is the largest. Given the specimen geometry, the largest in this case will be 1.0mm. The fatigue crack
growth tests were done in load control, utilizing a load shedding technique described in [44], so that the specimen would see a normalized K gradient, C, of:

$$C = \left( \frac{1}{K} \right) \left( \frac{dK}{da} \right) \geq -0.08 \text{mm}^{-1}$$

(3.2.)

over part II of the test. The test was stopped when the crack growth rate was under $1 \times 10^{-9}$ mm/cycle. The test was restarted at a similar $\Delta K$ to part II of the test, but this time the crack was allowed to grow under constant load range conditions until ~80-90% of the specimen width was cracked. The specimen was then unloaded and brought to room temperature before again monotonically loading to final failure.

**Figure 3.4.** Image of a fracture surface with the crack front lines overlayed. Crack growth direction is left to right and the loading direction is normal to the plane.
3.2.4. Interrupted Fatigue Crack Growth Testing

Interrupted tests were also performed under the conditions described in Table 3.4. These tests were stopped after reaching threshold for crack tip deformation investigations. Specimens were sliced down the middle so that one specimen became two, each with a thickness of approximately 2.5mm. One half specimen was again fatigued under the same frequency as the test, except at room temperature to prevent additional oxidation along the crack wake. The crack was grown approximately 1 mm away from the threshold crack front before loading monotonically to final fracture. The other half specimen was characterized as follows, with microscopy details discussed later in Section 3.4. The center face of the specimen was parallel polished through 1200 grit SiC paper and then final polished using 0.2μm colloidal silica. Both faces of the specimen were examined in a scanning electron microscope (SEM). The crack tip and plastic zone on the center face was characterized using orientation image microscopy. The same face was then etched and examined in an SEM to look at the microstructure. Site-specific transmission electron microscopy foils were extracted using focused ion beam techniques, which are discussed in Section 3.3.4.
3.2.5. Crack Tip Opening Displacement Measurement

Crack tip opening displacement (CTOD) data was obtained during testing with an MTS 632.05B-04 high temperature axial extensometer with alumina extension rods that contacted the specimen above and below the notch. A picture of the wired specimen in the air test frame with the extensometer attached is shown in Figure 3.5. CTOD data was taken for 10 cycles at a time at random intervals throughout the test. Displacement data was plotted as a function of force in order to identify changes in stiffness due to crack closure. Low and high load portions of the data were fit linearly, and a closure force ($P_{cl}$) was determined from the value where the linear fits crossed. The force at closure was used to calculate the effective stress intensity factor, $\Delta K_{eff}$, using:

$$K_{cl} = \frac{P_{cl}}{B \sqrt{W}} \left[ \frac{(2 + \alpha)}{(1 - \alpha)^{3/2}} \right] (0.886 + 4.64\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4)$$

(3.3.)

$$\Delta K_{eff} = K_{max} - K_{cl}$$

(3.4.)

Where $K_{cl}$ is the stress intensity factor at closure, and $K_{max}$ is the max stress intensity factor at the crack length where the CTOD data was obtained.
3.2.6. Data Analysis

Following ASTM Standard E 647 [44], the DCPD crack length must be adjusted to agree with the actual measured crack length. The crack length at the lines ending the precrack, threshold, and large ΔK regions was optically measured across the specimen at 5 points. The measurements were used to determine a weighted average:

\[ a_{\text{meas}} = \frac{[a_1 + a_2 + 2(a_3 + a_4 + a_5)]}{8} \]  

(3.5.)
where $a_1$ and $a_2$ are the crack lengths measured on the edges, and $a_3$ through $a_5$ are the crack lengths measured across the width of the specimen at even intervals. This correction is done to take the curvature of the crack into account, and crack lengths 3 through 5 are given additional weight because they are the crack lengths that are in plane strain. The DCPD crack length was then shifted so that the crack measurement at the start of the test section matched the actual crack length using:

$$a_{shift} = a_{DCPD} - (a_{start}^{DCPD} - a_{meas})$$  \(3.6\)

where $a_{shift}$ is the new crack length, $a_{DCPD}$ is the crack length measured by DCPD, and $a_{meas}$ is the crack length measured at the crack front line, that starts the test section. Next, the data was adjusted so that the last DCPD data point would match the actual measured crack length at the end of the test section using:

$$a_{adj} = a_{shift} + \left[\frac{(a_{end}^{meas} - a_{end}^{start})}{a_{end}^{shift}/a_{start}^{shift}}\right](a_{start} - a_{start}^{shift})$$  \(3.7\)

where $a_{adj}$ is the adjusted crack length. The final data for crack length was then referred to as adjusted crack length. The adjusted crack lengths were, on average, within 0.40mm ±0.24mm of the DCPD crack length measurements.

The cycle count, max load, and adjusted crack length was input into a smoothing program that calculates $da/dN$ and $\Delta K$. The program is derived from a method documented in [45]. The program fit used a minimum of 7 points along an interval
determined by the smoothness of the data. The final data set was plotted as $\frac{da}{dN}$ vs $\Delta K$ for comparison.

3.3. Characterization

3.3.1. Orientation Imaging Microscopy

Orientation imaging microscopy (OIM) was done on the sections of the slabs cut as described in Section 3.1.1. OIM was done at 25kV, with a spot size of 5 and a working distance of 24 or 25mm. The scan size was roughly 1mm by 1mm with a step size of 0.01mm. Data analysis software was used to plot the pole figure for each scan and determine the angular distance from the axis.

OIM was also performed on the interrupted specimens. These scans were also done at 25kV, a spot size of 5, and a working distance of 24mm. The scans were large enough to encompass the area around the crack tip in order to view any lattice rotation due to strains imposed during testing.
3.3.2. Optical Microscopy

Images of the fracture surfaces were taken with a Canon PowerShot SX200IS digital camera so that the loading axis was normal to the plane of the image. Additionally, images were taken of the side of the fractured halves so that the crack growth direction was left to right, and the crack profile could be viewed. Specimens were examined using a Keyence VHX-600 light microscope. Images were taken so that the fracture surface was perpendicular to the camera. For some images the use of the Keyence z-direction stitching software was used to form images that had all areas in focus. Distortions from changing focal lengths were accounted for in the software. A step size of 10μm to 25μm was used to form the images.

3.3.3. Scanning Electron Microscopy

Fractography was done using an FEI Sirion SEM. A back scatter detector was used to look at the dendritic macrostructure that formed when the slabs were directionally solidified. Specimens were then sectioned along the plane that included both the crack growth direction and loading axis in order to examine the fracture surface profile and the microstructure along the crack wake. The sections were polished with SiC paper through 1200grit, and subsequently polished using 0.02μm
colloidal silica. The specimens were examined in the microscope before etching. The $\gamma'$
etchant is comprised of 30mL nitric acid, 50mL lactic acid, and 2mL hydrofluoric acid.
The samples were etched between 5-15 seconds, depending on the strength of the
etchant (which grows stronger with time). The sections were again examined using an
SEM.

3.3.4. Focused Ion Beam Techniques

Site-specific TEM foils were made at the crack front in the fully tested specimens,
and ahead of the crack tip in the interrupted tests, using a focused ion beam (FIB). For
all foils, an FEI Nova was used for the initial thinning and an FEI Helios was used for
plucking and final thinning of the foil. The foils taken from the interrupted test
specimens were plucked perpendicular to the crack propagation direction,
approximately 5μm away from the crack tip. The procedure for making a site specific
foil follows.

Platinum was placed over the area of interest in order to protect the surface.
Trenches on either side of the foils were milled using a 30kV ion beam with a current of
6.8nA. Foils were thinned to approximately 1μm with a 2.8nA ion beam current. The
foils were then plucked using an Omniprobe. This procedure begins with an undercut
and a side cut, so that the foil is attached to the specimen on one side only. Then a
tungsten needle is attached to the foil with platinum. When the needle is secure, the
third side of the foil is cut free and the foil is slowly lifted from the trench. The specimen is then removed from the FIB and a copper grid is inserted into the microscope. The foil is aligned with the grid and attached with platinum. Once attached, the tungsten needle is cut from the foil and removed from the working area. Foils are then slowly milled using decreasing ion beam current, ranging from 2.8nA down to 100pA. Finally, a 5kV ion beam with a current of 47pA is used to remove any damage induced by the higher voltage and higher currents.

3.3.5. Transmission Electron Microscopy

Transmission electron microscopy was performed using both an FEI CM200 and an FEI Tecnai F20. Images in the CM200 were taken using bright field, and tilting experiments were done using multiple zone axes and two beam conditions. Dislocation analysis was performed using the g·b method. Images acquired using the Tecnai were taken using the scanning transmission electron microscopy (STEM) mode. Bright field and dark field images were taken simultaneously over a range of two beam conditions. Electron dispersive spectroscopy (EDS) spot analysis was performed on any included particles to find the component elements.
CHAPTER 4
Fatigue Crack Growth as a Function of Temperature, Environment, and Frequency

4.1. Effect of Temperature on Crack Growth Behavior

4.1.1. Fatigue Crack Growth Results for Vacuum Testing at 10Hz

The da/dN vs. δK graph for all temperatures in vacuum at a frequency of 10Hz and an orientation of (001)[010] is shown in Figure 4.1. The value for the stress intensity range at threshold for all temperatures is shown in Table 4.1. δK_{th} varies with temperature, however a clear trend cannot be determined. Additionally, crack growth rate changes with temperature, shown by comparing da/dN values for temperatures at a given δK, but again, there is no clear trend. It is interesting to note that the highest δK_{th} for the vacuum specimens is at 650°C. This result does not directly compare to the trends found in literature, which find a direct proportionality between temperature and both the threshold stress intensity factor, and the crack growth rate [23-25]. This is
probably due to the reported trends being for air tested specimens, which are discussed in Section 4.2.1.

**Table 4.1.** List of all $\Delta K_{th}$ values and $da/dn$ (cycles/mm) @ $\Delta K = 10$ MPa$\sqrt{m}$ for specimens tested at 10Hz in vacuum with an orientation of (001)[100].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\Delta K_{th}$ (MPa$\sqrt{m}$)</th>
<th>da/dn (cycles/mm) @ $\Delta K = 10$ MPa$\sqrt{m}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>4.84</td>
<td>2.18E-8</td>
</tr>
<tr>
<td>650</td>
<td>6.82</td>
<td>1.04E-8</td>
</tr>
<tr>
<td>750</td>
<td>5.93</td>
<td>1.85E-8</td>
</tr>
<tr>
<td>850</td>
<td>5.93</td>
<td>2.23E-8</td>
</tr>
</tbody>
</table>
Figure 4.1. Crack growth rate vs stress intensity factor for data taken at all temperatures in vacuum at 10Hz having an orientation of (001)[100].

4.1.2. Fractography of 10Hz/Vacuum Specimens

Figure 4.2. shows the macroscopic fracture surfaces in the top section of the image and the crack profile in the bottom section of the image. The approximate
orientation of the specimens is shown in Figure 4.2.a. For all of the specimens the fatigue crack grew perpendicular to the loading axis.

![Image](image_url) **Figure 4.2.** Macroscopic view of post-failure specimens tested in vacuum, at 10Hz, with an orientation of (001)[100] and a temperature of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The top image shows the fracture surface while the bottom image shows the crack profile. The orientation of all specimens is shown in a).

The fracture surfaces from part II of the test, where ΔK decreased to threshold, were macroscopically flat with surface roughness. These fractography results are similar to
those seen in other fatigue crack growth studies reported in the literature [23-25, 29, 30].

Optical microscopy images of the fracture surfaces taken near the threshold crack front line within a dendrite core are seen in Figure 4.3. In these images, the crack growth direction is from left to right and the loading direction is out of the paper. The specimens have rough fracture surfaces, where the fracture of the interdendritic region is different than within the dendrites, leading to the macroscopic dendritic structure being seen on the fracture surfaces. The appearance of the dendrite structure on the fracture surface has been seen in other second generation alloys as well [46]. Closer investigation of the interdendritic region, seen in Figure 4.4., shows that the particles are faceted and smooth compared to the surrounding area, and the particles fractured similarly at all temperatures.
Figure 4.3. Optical microscope images of the fracture surfaces of the specimens tested in vacuum at 10Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation for all specimens is shown in a).
Figure 4.4. SEM image of interdendritic particle taken near the threshold crack front from the specimen tested at 750°C in vacuum at 10Hz with a (001)[100] orientation. Note: This is a representative particle for all temperatures tested.

Optical microscopy images of the fracture surface at the threshold crack front for all temperatures are shown in Figure 4.5. In these images, the crack growth direction is bottom to top and the loading direction is normal to the image. There is a noticeable trend toward a smoother crack front as temperature increases. In the 550°C specimen, the crack front is jagged and the change in crack length over the thickness of the specimen varies by over 1mm, with local oscillations on the order of hundreds of microns. The 650°C specimen also has a jagged crack front, but the difference in crack length over the thickness of the specimen is around 1mm and the jaggedness is reduced...
to oscillating within a hundred microns. For 750°C and 850°C, the crack front shows no sharp turns, but is slightly wavy. Over the width of the specimens, the crack length only changes on the order of hundreds of microns. It is interesting that the 550°C and 650°C crack fronts would end along octahedral planes since the part II crack path was not on octahedral planes. However, as noted by [23-25, 29, 30], there crack path will be along octahedral planes at higher ΔK. In part III of the test, where ΔK levels were increasing, faceted octahedral growth is seen, with the facets having smooth surfaces. The portion of the fracture surface after the threshold crack front line was tested at higher ΔK during part III of the testing, and the threshold crack front must be jagged because of part III. Another observation is that the region of the fracture surface relating to part III was smooth in the 750°C and 850°C tests. From these results, there is definitely an increase in octahedral fracture at high ΔK values, however the crack path changes again to planar at higher temperatures. The amount of faceted crack growth decreased with increasing temperature. Additionally, the decrease in octahedral fracture with increasing temperature was seen by Leverant et. al. [28].
Figure 4.5. Optical microscopy images of the crack fronts at threshold for the specimens tested in vacuum at 10Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation of all specimens is shown in a).
High resolution SEM was used to examine microscale features of the fracture surface near threshold. Images from each specimen are shown in Figure 4.6. The 550°C, 650°C, and 750°C surface features are similar and will be described as a “terraced” structure, owing to the parallel contour features in the images. Upon studying the images more closely, the terraced features of the 550°C and 650°C specimens are aligned at ~45° to the crack growth direction, which implies that some deformation was due to slip along the octahedral planes. To the contrary, the terraces in the 750°C specimen are not well aligned with any particular direction.

In the 850°C specimen, an outlier at this scale, there are islands of material which do not have the “terraced” structure, but instead appear to be smooth with no obvious crystallographic alignment. In between the islands there are nanoscale octahedral facets. It is clear that the crack is growing by a different mechanism than the other temperatures.
Figure 4.6. High resolution scanning electron microscope images of the fracture surfaces near threshold for the specimens tested in vacuum at 10Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation of all specimens is shown in a), and the terrace features are highlighted by the black lines in b).
4.2. Effect of Environment on Crack Growth Behavior

4.2.1. Fatigue Crack Growth Results for Air Testing at 10Hz

Figure 4.7. shows the da/dN vs. ΔK graph for all temperatures in air at a frequency of 10Hz and an orientation of (001)[100]. The ΔKth values are compared with those measured in vacuum in Table 4.2., and the da/dN values at ΔK=10MPa√m are compared to those in vacuum in Table 4.3. In air, ΔKth and crack growth rates increase with increasing temperature. Note, that in contrast to the specimens tested at 10Hz in vacuum, the 850°C test yielded the highest ΔKth. This increasing ΔKth trend is attributed to the formation of an oxidation layer which caused crack closure. This matches trends seen in the literature for the effects of oxide-induced crack closure [23].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>ΔKth (MPa√m)</th>
<th>Vacuum/10Hz/(001)[100]</th>
<th>Air/10Hz/(001)[100]</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>4.84</td>
<td>6.03</td>
<td></td>
</tr>
<tr>
<td>650</td>
<td>6.82</td>
<td>6.29</td>
<td></td>
</tr>
<tr>
<td>750</td>
<td>5.93</td>
<td>8.97</td>
<td></td>
</tr>
<tr>
<td>850</td>
<td>5.93</td>
<td>14.06</td>
<td></td>
</tr>
</tbody>
</table>
Table 4.3. List of da/dN values @ $\Delta K=10\text{MPa}\sqrt{m}$ for specimens tested at 10Hz with an orientation of (001)[100].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>da/dN (cycles/mm)</th>
<th>da/dN (cycles/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Vacuum/10Hz/(001)[100]</td>
<td>Air/10Hz/(001)[100]</td>
</tr>
<tr>
<td>550</td>
<td>2.18E-8</td>
<td>2.18E-8</td>
</tr>
<tr>
<td>650</td>
<td>1.04E-8</td>
<td>3.04E-8</td>
</tr>
<tr>
<td>750</td>
<td>1.85E-8</td>
<td>3.92E-8</td>
</tr>
<tr>
<td>850</td>
<td>2.23E-8</td>
<td>n/a</td>
</tr>
</tbody>
</table>

Compared to the vacuum tests, the crack growth rate decreases to a threshold value abruptly, which is in contrast to the smooth transition seen between the Paris region and the threshold region seen in vacuum data. This abrupt decrease is reflected in the graph as a sharp knee in the data followed by the dotted line down to the threshold data. The 850°C data does not have a Paris region because the $\Delta K$ that the test was started at was not high enough, and therefore it is uncertain whether or not the data would exhibit a knee. However, the data does show large decreases in crack growth rate with small decreases in $\Delta K$, which is similar to the lower temperature tests.

The “sharpness” of the knee was quantified by measuring the slopes of the threshold region and Paris region for both the vacuum specimens and air specimens. For both vacuum and air specimens, the Paris region slope was approximately 2-6. The threshold region slopes for vacuum were ~20-70, while the threshold region slopes for air were ~60-260. Comparing these values, it is determined that the transition from Paris region to threshold region is definitely sharper for air.
Figure 4.7. Crack growth rate vs. stress intensity factor for data taken at all temperatures in air at 10Hz having an orientation of (001)[100].
4.2.2. Fractography of 10Hz/Air Specimens

Figures 4.8 shows images of the fracture surfaces and the fracture profile, arranged similarly to the macroscopic images of the specimens tested in vacuum. All fracture surfaces were macroscopically flat with smaller scale surface roughness for all parts of the test. In contrast to the vacuum tests, there was no transition to octahedreal cracking at higher $\Delta K$ values for any of the temperatures tested. In the 750°C and 850°C images, there is evidence of octahedral fracture, but this has occurred after part III of the test, when the specimen loaded monotonically to failure at room temperature.

Figure 4.9 shows optical microscopy images of the fracture surfaces from the air test specimens taken near the threshold crack front line within a dendrite core. In all cases, the fracture surfaces were discolored from oxidation. The fractography indicates that they have fatigued similarly to the vacuum specimens, where the dendritic macrostructure can be seen on the fracture surfaces. Figure 4.10 shows an image of an interdendritic particle near the threshold crack front line. The crack propagated through the particle and the oxidation of the particle is different than the surrounding region. The image shown is representative of interdendritic particles for all temperatures.
Figure 4.8. Macroscopic view of specimens tested in vacuum, at 10Hz, with an orientation of (001)[100] and a temperature of  a) 550°C. b) 650°C. c) 750°C. d) 850°C. The top image shows the fracture surface while the bottom image shows the fracture profile. The orientation of all specimens is shown in a).
Figure 4.9. Optical microscope images of the fracture surfaces of the specimens tested in air at 10Hz, an orientation of (001)[100], and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The approximate orientation of all specimens is shown in a). Note: the threshold line in d) is to the far right, with the area immediately to the left of the line, in light gray, being the threshold region. Other lines are from precracking.
Figure 4.10. SEM image of an interdendritic particle near the threshold crack front from the specimen tested at 850°C in air at 10Hz with a (001)[100] orientation. Note: This is a representative particle for all temperatures tested.

Figure 4.11. presents images of the fracture surface at the threshold crack front for each test temperature. In all images the crack length varies by about 1mm across the width of the specimen. The crack front is pinned by the particles in the interdendritic region for all of the temperatures tested, with some particles having a greater interaction with the crack front than others. Since the test was conducted in air, oxide formation may be more chemically favorable for the interdendritic particles,
forming a structure which impedes crack growth. This pinning of the crack front may contribute to the abrupt crack growth rate decrease seen in the fatigue crack growth data for the air tests. However, it is unclear why pinning suddenly takes effect at this $\Delta K$.

The composition of the interdendritic particles was examined using electron dispersive spectroscopy (EDS). Figure 4.12. has images of both large pinning interaction and small pinning interaction, with arrows indicating the spot where EDS was performed. It was found that the particles that have a larger interaction are rich in Ni and Co, while the particles that have a smaller interaction are Ta rich.
Figure 4.11. Images of the crack fronts for the specimens tested in air at 10Hz at test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation of all specimens is shown in a).
crack growth direction \[ \rightarrow \]

a) \hspace{1cm} b)

![SEM images](image)

**Figure 4.12.** SEM images of the threshold crack front where pinning is occurring with a a) large interaction. b) small interaction. The arrows point to the spots EDS was performed. The threshold crack front line is highlighted by the dotted line.

High resolution scanning electron microscopy images of the fracture surfaces from the 10Hz air specimens are shown in Figure 4.13. Optical observations of oxidation on the surface were confirmed, and the amount of oxidation on the surface varies depending on the length of the test. In the 550°C and 650°C images, it is apparent that the oxide preferentially forms on the gamma channels. In the 750°C and 850°C
specimens, the oxide has grown so that there appears to be a homogeneous layer covering the surface.

Figure 4.13. High resolution scanning electron microscope images of the fracture surfaces near threshold for the specimens tested in air at 10Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation of all specimens is shown in a).
4.3. **Effect of Frequency on Crack Growth Behavior**

4.3.1. **Fatigue Crack Growth Results for Air Testing at 0.5Hz**

Fatigue crack growth data for specimens tested at all temperatures in air at a frequency of 0.5Hz and an orientation of (001)[100] is seen in Figure 4.14. As reported in Table 4.4., the threshold stress intensity factor range peaks at 750°C. When comparing $\Delta K_{th}$ values for 10Hz and 0.5Hz in air, the 0.5Hz values are larger for all but 850°C. It is probable that the oxide-induced crack closure is enhanced at lower frequency. Previous studies of frequency effects found that lowering frequency will increase the $\Delta K_{th}$ [23, 25]. This is because at the lower frequency, the crack is exposed to air longer, causing a thicker oxide layer. Also note that interrupted test data is also shown in Figure 4.14., and the $\Delta K_{th}$ values are reported in Table 4.4. For the 550°C and 750°C tests, the $\Delta K_{th}$ value was within 0.5MPa√m of the tests that underwent all test parts. From this result, it is concluded that differences in $\Delta K_{th}$ that are less than 0.5MPa√m are within the error of the test.
Table 4.4. List of all $\Delta K_{th}$ values for specimens tested at in air with an orientation of (001)[100].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Air/10Hz/(001)[100]</th>
<th>Air/0.5Hz/(001)[100]</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>6.03</td>
<td>6.54</td>
</tr>
<tr>
<td>550 Interrupted</td>
<td>n/a</td>
<td>6.82</td>
</tr>
<tr>
<td>650</td>
<td>6.29</td>
<td>8.53</td>
</tr>
<tr>
<td>750</td>
<td>8.97</td>
<td>14.38</td>
</tr>
<tr>
<td>750 Interrupted</td>
<td>n/a</td>
<td>14.81</td>
</tr>
<tr>
<td>850</td>
<td>14.06</td>
<td>12.85</td>
</tr>
</tbody>
</table>

Crack growth rates at $\Delta K=10$MPaVm for 0.5Hz are compared to $da/dN$ values at 10Hz in Table 4.5., and like the 10Hz specimens, there is an increase in crack growth rate with temperature. Similar to the 10Hz air tests, the crack abruptly arrests to a threshold crack growth rate at some point in the test. Again, the 850°C test does not show the same feature because part II of the test started at a $\Delta K$ lower than the transition from the Paris region to the threshold region.

Table 4.5. List of $da/dN$ values @ $\Delta K=10$MPaVm for specimens tested in air with an orientation of (001)[100].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>da/dN (cycles/mm)</th>
<th>da/dN (cycles/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Air/10Hz/(001)[100]</td>
<td>Air/0.5Hz/(001)[100]</td>
</tr>
<tr>
<td>550</td>
<td>2.18E-8</td>
<td>3.52E-8</td>
</tr>
<tr>
<td>650</td>
<td>3.04E-8</td>
<td>8.82E-8</td>
</tr>
<tr>
<td>750</td>
<td>3.92E-8</td>
<td>n/a</td>
</tr>
<tr>
<td>850</td>
<td>n/a</td>
<td>n/a</td>
</tr>
</tbody>
</table>
4.3.2. Effect of Crack Closure at 0.5Hz

Figure 4.15. compares the measured fatigue crack growth data with data plotted against the effective stress intensity factor range, $\Delta K^{\text{eff}}$. The $\Delta K^{\text{eff}}$ was calculated using the acquired crack opening displacement data. The threshold stress intensity factor
range values are reported in Table 4.6. The values of $\Delta K_{th}^{\text{eff}}$ are smaller than the $\Delta K_{th}$ values, which is the expected trend when an oxide causes premature contact of the crack faces. The effect for each specimen is only on the order of ~1-2 MPa$m^{\frac{1}{2}}$, which means the crack closure effect is active, but may not be the most prevalent influence on crack growth.

Table 4.6. List of all $\Delta K_{th}$ and $\Delta K_{th}^{\text{eff}}$ values for specimens tested at 0.5Hz in air with an orientation of (001)[100].

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\Delta K_{th}$ (MPa$m^{\frac{1}{2}}$)</th>
<th>$\Delta K_{th}^{\text{eff}}$ (MPa$m^{\frac{1}{2}}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>6.54</td>
<td>5.33</td>
</tr>
<tr>
<td>650</td>
<td>8.53</td>
<td>6.29</td>
</tr>
<tr>
<td>750</td>
<td>14.38</td>
<td>12.25</td>
</tr>
<tr>
<td>850</td>
<td>12.85</td>
<td>11.11</td>
</tr>
</tbody>
</table>
Figure 4.15. Fatigue crack growth data for specimens tested in air at 0.5Hz with an orientation of (001)[100]. Crack tip opening displacement results are plotted with the adjusted DCPD data.
4.3.3. Fractography of 0.5Hz/Air Specimens

The fracture surface and profile of the specimens tested at 0.5Hz in air at all temperatures is seen in Figure 4.16. The fracture profiles for all specimens show that each failed perpendicular to the loading axis. The fracture surfaces and profiles of the interrupted test specimens were comparable to the full tests.

Optical microscopy images were taken of the fracture surfaces and are seen in Figure 4.17. Like the 10Hz specimens, the dendritic macrostructure is noticeable on the fracture surface. While not shown, note that the fracture surfaces of the interrupted tests looked similar to the fracture surfaces from the full tests at the same temperature. Closer investigation of the threshold region in the SEM showed the surfaces were oxidized with the $\gamma$ channels preferentially oxidizing compared to the $\gamma'$ precipitates. Images are similar to those seen for the 10Hz tests and are therefore not shown here. Figure 4.18 shows an image of the interdendritic region near the threshold crack front of the 850°C specimen. At this frequency, the crack path of the 850°C specimen grows around the particles. The interdendritic particles for all other temperatures fatigued similarly to the particles at 10Hz.
**Figure 4.16.** Macroscopic view of specimens tested in air, at 0.5Hz, with an orientation of (001)[100] and a temperature of a)550°C. b)650°C. c)750°C. d)850°C. The top image shows the fracture surface while the bottom image shows the fracture profile. The orientation for all specimens in shown in a).
Figure 4.17. Optical microscope images of the fracture surfaces near the threshold crack front of the specimens tested in air at 0.5Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. The orientation for all images is shown in a). Note: the threshold line in d) is to the far right, with the area immediately to the left of the line being the threshold region. The line on the left of the image is from precracking.
Upon investigation of the crack front, pinning is again prevalent in the specimens tested at 550°C, 650°C and 750°C, shown in Figure 4.19.a. through 4.19.c. At 850°C, Figure 4.19.d., we see no pinning along the crack front due to the crack growing around the interdendritic particles. Like the 10Hz tests in air, no octahedral fracture is seen at higher ΔK values.

**Figure 4.18.** SEM image of the interdendritic region near the threshold crack front line of the specimen tested at 850°C in air at 0.5Hz with a (001)[100] orientation. Note: This is a representative particle for all temperatures tested.
Figure 4.19. Images of the threshold crack fronts for the specimens tested in air at 0.5Hz with an orientation of (001)[100] and test temperatures of a) 550°C. b) 650°C. c) 750°C. d) 850°C. For all images, the orientation is shown in a).
4.4. Microstructure Analysis

4.4.1. Microstructure of 10Hz/Vacuum and 10Hz/Air Specimens

Changes in precipitate size and morphology between the as received slabs and the specimens after testing at 10Hz were examined. Figure 4.20. shows the as-received microstructure compared to the microstructures of thermally exposed, un-stressed regions of the specimens after testing. Despite the elongated temperature hold during testing, the γ’ size and morphology did not change in any of the specimens.

The microstructures at the fracture surface for two different crack growth rates, 1E-8 mm/cycle and near threshold, are compared for both the 750°C vacuum and 750°C air specimens, in Figures 4.21. and 4.22., respectively. In both specimens, there does not appear to be any microstructural changes due to the addition of fatigue loading. In the air specimen, one can see the oxidation layer along the fracture surface, which is at the top in Figures 4.22.a. and 4.22.b. The oxide has formed a diffuse layer, forming in both γ and γ’.
Figure 4.20. a) As-Received slab. b) 550°C Vacuum. c) 750°C Vacuum. d) 550°C Air. e) 750°C Air. Images b-e are from thermally exposed/no stress regions of the specimens. The orientation of all specimens is shown in a).
Figure 4.21. Microstructures from the 750°C Vacuum 10Hz (001)[100] test. a) thermally exposed. b) $da/dN= 1E^{-8}$ mm/cycle. c) $da/dN= 1E^{-10}$ (threshold). Approximate crystallographic directions for all images are indicated in a).
Figure 4.22. Microstructures from the 750°C Air 10Hz (001)[100] test. a) thermally exposed. b) $\frac{da}{dN} = 1E^{-8}$ mm/cycle. c) $\frac{da}{dN} = 1E^{-10}$ mm/cycle (threshold). Approximate crystallographic directions for all images are indicated in a).
4.4.2. Microstructure of 0.5Hz/Air Specimens

The oxidation along the crack wake and at the crack tip was examined in the interrupted tests. The crack wake and crack tip for the 550°C test is shown in Figure 4.23. There is a small amount of oxidation, approximately 0.1mm thick around the crack tip and along the crack wake. The crack propagated through both γ and γ', and similar to observations of the fracture surface, it appears that the oxide is thicker near the γ channels and thinner near the γ' precipitates.

![crack growth direction →](image)

**Figure 4.23.** Crack profile for the interrupted specimen tested at 550°C. The crack propagates through both γ and γ' with oxide preferentially forming on the γ channels.
The crack wake and crack tip for the 750°C test is shown in Figure 4.24. Because of the higher test temperature, there was more oxidation along the wake and a large portion of oxidized material around the crack tip. For this test temperature, a zone denuded of $\gamma'$ precipitates was seen along the crack wake and ahead of the crack tip that ranged in size from 0.2$\mu$m to 0.5$\mu$m. Since there was not a denuded zone for the 10Hz specimens tested in air, the appearance of the denuded zone can be contributed to the decrease in frequency. Additionally, this zone should become larger at higher temperatures where diffusion is faster. As reported in literature, depleting $\gamma'$ precipitates from the region immediately ahead of the crack tip will cause a softening that allows faster crack propagation rates [32].

![Crack profile for the interrupted specimen tested at 750°C. A denuded zone is present along the crack wake and ahead of the crack tip.](image)

**Figure 4.24.** Crack profile for the interrupted specimen tested at 750°C. A denuded zone is present along the crack wake and ahead of the crack tip.
4.5. **Substructure Analysis**

4.5.1. **Dislocation Structure in the Paris Region**

A foil was selectively plucked from the fracture surface the 750°C, vacuum, 10Hz, (001)[100] specimen after fracture at a point along the crack wake where the crack growth rate was approximately to 1E-8 mm/cycle. Figure 4.25. shows that there is a large amount of dislocations within the γ channels and low dislocation content within the γ’ precipitates. This is attributed to the strains at this ΔK, along with the cyclic nature of fatigue causing the dislocations to become tangled. Preliminary g·b dislocation analysis found that the dislocations are ½{110} type.
Figure 4.25. Bright field STEM image from within a dendrite. The foil was plucked from the Paris region of the crack wake and the foil orientation is shown in the image.

4.5.2. Dislocation Structure Near the Threshold Crack Front

Foils were also selectively plucked from the 750°C, vacuum, 10Hz, (001)[100] specimen from two areas near threshold: one area within a dendrite and one area within the interdendritic region over one of the included particles. Both foils were approximately 10μm from the crack front. Figure 4.26. shows images of the substructure present in each area. In both cases, there are regions of dislocations within γ channels, with little dislocation content within the γ' precipitates. In both foils
there is a smaller amount of dislocation content than seen in the Paris region. This is due to smaller strains near threshold. Again, a preliminary g·b analysis found that the dislocations are ½{110} type.

In Figure 4.26.b., the area around the included particle does not appear rich in dislocations, as was expected since dislocations normally accommodate misfit between particles and the matrix. Additionally, it does not appear that the particle is acting as a dislocation source or sink. Electron dispersive spectroscopy (EDS), using the Tecnai, was performed on the particle found in the interdendritic region foil. EDS analysis showed that the particle contained a large wt% of tantalum.
Figure 4.26. Bright field STEM images from within the a) dendrite and b) interdendritic region near a carbide particle. Both foils were plucked near the threshold crack front and the foil orientation is shown in each image.
4.5.3. Deformation Mechanisms Ahead of the Crack Tip

From the interrupted specimens tested at 550°C and 750°C at 0.5Hz in air, site-specific foils were plucked from the region ahead of the crack tip, approximately 10μm from the tip of the crack. The foils were oriented so that the foil was perpendicular to the crack tip and one edge of the foil was directly ahead of the crack tip. This configuration allows a larger viewing area of deformation assuming the plastic zone ahead of the crack is symmetric above and below the plane of the crack.

Figure 4.27. shows an image of the substructure in the foil plucked from the specimen tested at 550°C. Within the γ channels and γ’ precipitates there is an intense amount of dislocations, which are taking three different configurations. The dislocations that are at 45° angles to the channels have, by preliminary investigation, been found to be $\frac{1}{2}$\{110\} type. Some of these dislocations appear to also be bowing, most likely coming to this configuration by cross slip onto cube planes. Additionally, there are dislocations that appear parallel to the vertical channels, which have burgers vectors of \{100\} type.
Figure 4.27. Bright field STEM of site-specific foil plucked from ahead of the crack tip in the 550°C Air 0.5Hz test that was interrupted at threshold. The orientation of the foil is shown.

Figure 4.28. shows an image of the substructure in the foil plucked from the specimen tested at 750°C. At this temperature, the dislocation structure is similar to 550°C and includes straight dislocations that are at 45° angles to the γ channels, some looping dislocations, and some dislocations parallel to the vertical channels. Dislocations in the γ channels are more confined to the interfaces, though there is
content within the channel also. For both temperatures, the distribution of dislocations seems homogeneous throughout the foils.

These TEM results are similar to those seen by Leverant et. al. In the study by Leverant, a homogeneous distribution of dislocations was seen at low frequencies which lead to mode I cracking, i.e. a flat crack plane perpendicular to the loading direction [28]. This seems to describe the results of the present study well.

Figure 4.28. Bright field STEM of site-specific foil plucked from ahead of the crack tip in the 750°C Air 0.5Hz (001)[100] test that was interrupted at threshold.
4.6. Effect of Environment and Frequency on $\Delta K_{th}$ and $da/dN$ vs. Temperature

The threshold stress intensity factor range for all tests versus temperature are shown graphically in Figure 4.29. From the graph we can see that testing in air at 10Hz and 0.5Hz has a more dramatic effect on $\Delta K_{th}$, meaning the change in threshold stress intensity factor changes more with temperature in air than in vacuum. This increase in $\Delta K_{th}$ is attributed to the contributions of environmental effects near the threshold region. Contributions from both pinning and oxide-induced closure increase $\Delta K_{th}$ values at 10Hz and 0.5Hz in air compared to vacuum.

When decreasing frequency, the value of $\Delta K_{th}$ increased compared to 10Hz for all temperatures except 850°C. In a study by Henderson et al., the authors compare the dislocation movement at multiple frequencies and attribute an increase in $\Delta K_{th}$ at low frequency to the Kear-Wilsdorf locking mechanism [25]. In the TEM foils from the 550°C and 750°C specimens tested at 0.5Hz there was evidence of cross-slip, while in the foils from the 750°C specimen at 10Hz there was no apparent cross-slip. The idea that lower frequencies allow more time for cross slip [25] is a viable explanation for trends in our study the 0.5Hz values are higher than the 10Hz values. However, as discussed in Section 2.3.3., the cross-slip locking mechanism will only increase $\Delta K_{th}$ until the temperature is high enough for either cross-slip back to octahedral planes or APB dragging. The approximate temperature for this transition is 800°C, and after this temperature, the $\Delta K_{th}$ should start decreasing again. For this study, the result is a peak
Figure 4.29. Dependence of \( \Delta K_{th} \) on temperature.

Figure 4.30. Dependence of crack growth rate at \( \Delta K = 10 \text{MPa}\sqrt{\text{m}} \) on temperature.
$\Delta K_{th}$ value at 750°C, after which the locking mechanism breaks down and the $\Delta K_{th}$ value decreases. Additionally, the $\Delta K_{th}$ value at 850°C could be decreasing because pinning is not a factor at 0.5Hz and 850°C. The crack grows around the interdendritic particles, therefore negating any added strength the particle gives to the microstructure.

The crack growth rate versus temperature for tests where there was a value at $\Delta K=10$ MPa√m are plotted in Figure 4.30. In general, the crack growth rate increases in air compared to tests at the same temperature in vacuum. Additionally, higher crack growth rates are seen in specimens with a lower frequency, given the same test environment. The increase in crack growth rates is attributed to the formation of a denuded zone ahead of the crack tip. In the 10Hz specimens tested in air there was a region next to the oxide layer where the $\gamma'$ particles were beginning to be dissolved. This small decrease in strength contributed to increased crack growth rates compared to vacuum. In the 0.5Hz specimens, where environmental effects are greater than at 10Hz, the $\gamma'$ particles were completely dissolved next to the oxide layer forming a denuded zone. The further decrease in the material strength increased crack growth rates compared to 10Hz.
CHAPTER 5
Fatigue Crack Growth as a Function of Orientation

5.1. Effect of Orientation on Crack Growth Behavior

5.1.1. Fatigue Crack Growth Results for Varying Orientation

Fatigue crack growth data for specimens tested in air at 0.5Hz with different orientations is shown in Figure 5.1. Data shown are for specimens where the DCPD crack length measurement system is valid. Figure 5.2. shows the crack profile and fracture surface for all specimens. If the crack path deviates from perpendicular to the loading axis by more than 20 degrees, the data is invalid. Therefore, useful data was only obtained from the tests at 550°C with orientations of (001)[110] and (011)[100] and a single test at 750°C with an orientation of (011)[100]. Table 5.1. shows the $\Delta K_{th}$ values for the valid crack growth tests and Table 5.2. shows the $da/dN$ values at $\Delta K=10\text{MPa} \sqrt{\text{m}}$ for valid tests where the information was available. Compared to the original test orientation of (001)[100], all of the threshold values increased when changing
orientation. The $\Delta K_{th}$ values for the 750°C specimens are larger than the 550°C specimens, similar to the tests in the (001)[100] orientation. For 550°C, changing the crack growth direction to [110] increases the $\Delta K_{th}$ value more than changing the loading axis to (011).

Table 5.1. List of all $\Delta K_{th}$ values for all specimens tested at 0.5Hz in air.

<table>
<thead>
<tr>
<th>Temperature (°C) / Orientation</th>
<th>$\Delta K_{th}$ (MPa(\sqrt{m}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air / 0.5Hz</td>
<td></td>
</tr>
<tr>
<td>550 / (001)[100]</td>
<td>6.54</td>
</tr>
<tr>
<td>550 / (011)[100]</td>
<td>7.88</td>
</tr>
<tr>
<td>550 / (001)[110]</td>
<td>8.15</td>
</tr>
<tr>
<td>750 / (001)[100]</td>
<td>14.38</td>
</tr>
<tr>
<td>750 / (011)[110]</td>
<td>15.00</td>
</tr>
</tbody>
</table>

Table 5.2 List of $da/dN$ values at $\Delta K=10\text{MPa}\sqrt{m}$ for specimens tested at 0.5Hz in air.

<table>
<thead>
<tr>
<th>Temperature (°C) / Orientation</th>
<th>$da/dN$ (cycles/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air / 0.5Hz</td>
<td></td>
</tr>
<tr>
<td>550 / (001)[100]</td>
<td>3.52E-8</td>
</tr>
<tr>
<td>550 / (011)[100]</td>
<td>2.44E-8</td>
</tr>
<tr>
<td>550 / (001)[110]</td>
<td>2.07E-8</td>
</tr>
<tr>
<td>750 / (001)[100]</td>
<td>n/a</td>
</tr>
<tr>
<td>750 / (011)[110]</td>
<td>n/a</td>
</tr>
</tbody>
</table>
In the 550°C specimens, the slope of the Paris region in the original orientation of (001)[100] is approximately 3.9, while for the different orientations, (011)[100] and (001)[110], the slope of the Paris region is approximately 2.75, both values within the normal range of slopes in ductile materials. Additionally, there does not appear to be a knee in the data at 550°C with an orientation of (001)[110]. The crack growth rate in
the Paris region also decreases in the (001)[110] and (011)[100] specimens compared to (001)[100]. The decrease in crack growth rates were seen in literature for a change in crack growth direction [20, 36, 38], but there was no apparent effect on crack growth rate from a change in loading direction [24, 37].

For the 750°C specimens, both loading direction and crack growth direction changed in the only valid test, so that the orientation was (011)[110]. The Paris region slope is smaller for the (001)[100] specimen than for the (011)[110] specimen, ~2 and ~5.3 respectively, meaning that small increases in ΔK, the crack growth rate increases more in the (011)[110] specimen than the original orientation. Additionally, there is not as abrupt a knee in the (011)[110] oriented specimen since the Paris slope is large, and the knee happens at a smaller crack growth rate. Larger Paris region slopes are associated with brittle materials. While the (011)[110] specimen is not failing by brittle fracture, the larger slope is probably due to a smaller number of slip planes available in this orientation. Similarly, in other studies where the crack growth direction was varied, it was found that the [110] direction propagated slower than the [100] direction [20, 36]. In this study, the curves cross each other in the Paris region, however, in the threshold region, the trend for crack growth rate is the same as that seen in literature.
Figure 5.2. Macroscopic view of specimens tested in air at 0.5Hz with a temperature of a) 550°C, b) 750°C, c) 550°C, d) 750°C, e) 750°C. The orientations are listed in the image. Top image is the fracture surface; bottom image is the crack profile. Note: useful data was only obtained from a, c, and e.
5.1.2. Fractography of Differently Orientation Specimens

As noted previously, macroscopic images of all specimens tested in different orientations is presented in Figure 5.2. Table 5.3. lists the specimens, the validity of the result, the angle the crack path takes from the crack growth direction, and the closest plane to the crack path.

<table>
<thead>
<tr>
<th>Specimen Temp.(C) / Orientation</th>
<th>Valid</th>
<th>Angle of crack path from growth direction</th>
<th>Approximate plane of crack path</th>
</tr>
</thead>
<tbody>
<tr>
<td>550 / (001)[110]</td>
<td>Yes</td>
<td>19°</td>
<td>(310)</td>
</tr>
<tr>
<td>550 / (011)[100]</td>
<td>Yes</td>
<td>5°</td>
<td>(011)</td>
</tr>
<tr>
<td>750 / (001)[110]</td>
<td>No</td>
<td>55°</td>
<td>(111)</td>
</tr>
<tr>
<td>750 / (011)[100]</td>
<td>No</td>
<td>25°</td>
<td>(310)</td>
</tr>
<tr>
<td>750 / (011)[110]</td>
<td>Yes</td>
<td>10°</td>
<td>(320)</td>
</tr>
</tbody>
</table>

Optical images of the fracture surfaces of all specimens tested with a change in orientation are presented in Figure 5.3. Specimens tested at 550°C and 750°C with an orientation of (001)[110] did not fracture similarly to each other or the previous sets of tests. In the case of 550°C, topological lines at 45° angle to the crack growth direction can be seen in Figure 5.3.a. At 750°C, the fracture surface has failed on multiple parallel planes with ligaments connecting them. The ligaments, which are large transitions
between the planes, are seen in Figure 5.3.b. as the features that are nominally parallel to the crack growth direction. Some cracking along the ligaments can also be seen in the image.

The fracture surfaces of the specimens with an orientation of (011)[100] were again different from each other. In Figure 5.3.c., the fracture surface of the 550°C specimen, the dendritic macrostructure is apparent at the right side of the image. In the areas that match with the core of the dendrite, the surface does not have large topological changes, however in the areas that match with the interdendritic areas, there is a rougher surface than the core areas. The fracture is similar to the original orientation (001)[100] where the interdendritic regions are rougher. For the specimen tested at 750°C, the fracture surface is shown in Figure 5.3.d. Like the specimen tested at 750°C / (001)[110] this fracture surface also has ridges running parallel to the crack growth direction in order to accommodate changes in crack growth plane. However, in this specimen the ridges are much more numerous and not as large.
Figure 5.3. Optical microscope images of the fracture surfaces near the threshold crack front line from the specimens tested in air at 0.5Hz at a) 550°C. b) 750°C. c) 550°C. d) 750°C. e) 750°C. The orientations are listed in the image. Note: useful data was only obtained from a, c, and e. Note: The threshold crack front line in e is on the right; precrack on the left.
Last, the fracture surface of the specimen tested at 750°C with an orientation of (011)[110] is shown in Figure 5.3.e. In the image, the threshold crack front is the approximately vertical slightly curved line on the right of the image, and the threshold region precedes it on to the left. The dendritic macrostructure can be seen on the fracture surface, with the core regions of the dendrite cracking smoother than the interdendritic regions.

Optical microscopy images of the crack front are shown in Figure 5.4. For the tests performed with the orientation of (001)[110] the crack changes in length by 500μm or less over the width of the specimen. The crack front of the specimen tested at 550°C, Figure 5.4.a., is fairly smooth, while the crack front of the specimen tested at 750°C, Figure 5.4.b., has pinning points. Figures 5.4.c. and 5.4.d. show the crack fronts of the tests performed with the orientation of (011)[100]. The crack front of both temperatures tested showed pinning and the crack length changes in length by 1mm or less over the width of the specimen. For the test performed at 750°C with the orientation of (011)[110], Figure 5.4.e., the crack changes in length by less than 500μm and has pinning points along the crack front.

Because the specimens were tested in air, closer SEM investigation showed oxidation on the surface, with the γ channels preferentially oxidizing compared to the γ’ precipitates. The images are not shown since they look comparable to the specimens tested at 10Hz in air.
Figure 5.4. Optical microscope images of the crack fronts for the specimens tested in air at 0.5Hz at a)550°C. b)750°C. c)550°C. d)750°C. e) 750°C. Orientations are listed with the image.
5.2. Effect of Orientation on $\Delta K_{th}$ and $da/dN$ vs. Temperature

Figure 5.5. presents the $\Delta K_{th}$ values versus temperature and Figure 5.6. presents the crack growth rate at $\Delta K=10\text{MPa}\sqrt{\text{m}}$ when available for all specimens tested at 0.5Hz. Changing the crack growth direction from [100] to [110] increases $\Delta K_{th}$ and decreases crack growth rates, both with a larger magnitude than changing the loading axis. In Chapter 4, an increase in $\Delta K_{th}$ values was attributed to environmental effects of oxide-induced closure and pinning. For the 550°C specimen in the (001)[110] orientation, there was no pinning prevalent on the fracture surface, yet this threshold value increased the most compared to the (001)[100] orientation. Other studies [20, 36] have also found that changing the crack growth direction from [100] to [110] was found to decrease crack growth rates. Therefore changing crack growth direction will increase threshold values due to the decrease in crack growth rates due to specimen orientation. Additionally, there is probably a contribution to the increased $\Delta K_{th}$ from oxide-induced closure, since the specimen was tested in air and this was found to be a valid mechanism at this frequency.

When changing the loading axis only, no change in crack propagation rate was seen in literature [24, 37]. Here, the results of the test differ from the literature. In the present study, changing the loading axis alone from the (001) to the (011) orientation increases $\Delta K_{th}$ and decreases crack growth rates. The 550°C specimen in the (011)[100] orientation fatigued similarly to the (001)[100] specimens, where the dendritic
**Figure 5.5.** Dependence $\Delta K_{th}$ on temperature.

**Figure 5.6.** Dependence of crack growth rate at $\Delta K=10$MPaV$m$ on temperature.
macrostructure and pinning were apparent on the fracture surface. However, the fracture surface had more topological difference because the crack path was traveling through more of the interdendritic region. Because of the constant change in crack path, the crack growth rates were decreased compared to the (001)[100] orientation, which also results in an increase in the threshold value. The threshold value is additionally affected by oxide-induced crack closure and pinning, as discussed in Chapter 4.

A change in both the loading axis from (001) to (011) and crack growth direction from [100] to [110] increases ΔK_th and changes the crack growth rate. The crack growth rate trend changes within the test because the slopes within the Paris region are different and the curves cross at a ΔK higher than the transition from threshold region to Paris region. Additionally, the decrease in crack growth rates is not as abrupt at this different orientation, but still present. The differences in Paris region slopes is attributed to the changes in slip plane orientation when the specimen orientation changes. The threshold value is additionally affected by oxide-induced crack closure and pinning, as discussed in Chapter 4.
CHAPTER 6

Conclusions and Future Research

6.1. Conclusions

Experience and experimental studies have historically found that creep is the life-limiting factor in turbine blades. With recent design changes, fatigue has become a more prominent life-limiting factor for in service conditions. Previous studies have characterized creep mechanisms in monocry stalline Ni-based superalloys for blade applications. When fatigue is studied, most often the studies use a lifetime approach to study fatigue initiation and overall fatigue life. However, for this investigation, a damage tolerant approach is used to study fatigue propagation behavior and crack growth mechanisms.

In this study, monocry stalline Ni-based superalloy René N5 was studied under cyclic loading conditions in order to gain a comprehensive characterization of crack propagation behavior. Compact tension specimens were systematically tested varying temperature, environment, frequency, and orientation. Fractography on failed
specimens was done on a macro to nano-scale. SEM and TEM investigations were done to quantify effects of microstructural and substructural deformation on fatigue crack propagation behavior.

Environment and frequency seem to have a larger effect on fatigue crack growth rates and threshold stress intensity factor ranges, while temperature and orientation effects are present, but not as dramatic. Crack growth rates for crack propagation in vacuum are slower than for air, so in service, internal crack initiation will not propagate as fast as a surface initiated crack. However, since the $\Delta K_{th}$ values are lower for vacuum, the cracks would begin to propagate at lower stress levels. During service, turbine blades can see a range of frequencies. From this study, it is clear that at a lower frequency cracks will begin to grow at higher stresses than cracks at higher frequencies, because of higher $\Delta K_{th}$ value, but propagate faster, because of higher $da/dN$.

In the normal blade orientation, (001)[100], mode I crack propagation was prevalent, with mode II crack propagation found at higher $\Delta K$ values. The dendritic macrostructure plays a role in the fatigue crack growth of specimens, since interdendritic particles appear to be slowing crack growth rates in the threshold region of specimens tested in air. Site-specific foils taken along the crack wake from the Paris and threshold regions showed no change in deformation mechanism, adding some value to the theory that the interdendritic particles are the cause of the abrupt decrease in crack growth rate for air specimens.
Microstructural analysis showed no change in $\gamma'$ precipitate size or morphology with temperature or stress. In the lower frequency specimens, a denuded zone was apparent around the crack wake and in front of the crack tip. TEM foils taken from ahead of the crack tip showed a homogeneous distribution of dislocations, with more dislocations in cube orientations at lower temperatures. It is theorized that a combination of mechanisms is occurring during testing, which is the reason there is no universal trend with temperature for threshold stress intensity factors. The mechanisms include Kear-Wilsdorf locking, oxide-induced crack closure, and crack tip softening due to $\gamma'$ depletion. Figure 6.1 is a graphical representation of each mechanism on fatigue crack growth.

Orientation also has an effect on the threshold stress intensity factor range and crack growth rate, but the trends are not as clear as the effects of environment and frequency, and are therefore not represented in Figure 6.1. In service blades have a loading direction of (001) but the secondary orientation is random. In this study, the crack growth rate was decreased for both a change in loading direction and a change in crack growth direction. However, the change in crack growth direction had a larger effect on the crack growth rate, and therefore increased $\Delta K_{th}$ more than a change in loading axis. Because of this, there would be no reason for the industry standard of a (001) loading axis to change.
Figure 6.1. Generalized $da/dN$ vs $\Delta K$ curves for specimens tested. The causes of the shifts in threshold and crack growth rates are labeled.
6.2. Unresolved Issues and Suggestions for Further Research

While much knowledge was gained during this study, there are still more investigations that can be done in order to have a more comprehensive knowledge of fatigue in monocristalline Ni-based superalloys. The author submits the following for further research:

1. Pinning of interdendritic particles causes an abrupt decrease in crack growth rate during the transition from the Paris region to the threshold region. Performing a multi-specimen study of interrupted tests at a single temperature to determine when particles begin pinning will aid in investigating why pinning is a prevalent mechanism. The study would include substructural investigation with site-specific foils taken from pinning sites when possible and in the free area of the crack front.

2. Because of time constraints, the substructures of specimens tested at 550°C and 750°C were characterized, and conclusions about deformation mechanism at all temperatures were drawn from these images only. Substructural investigations ahead of the crack tip in interrupted tests at 650°C and 850°C are necessary to confirm theories on crack growth mechanisms.

3. For specimens tested in (001)[110], (011)[001], and (011)[110] a closer microstructural investigation to confirm fracture planes and confirm a contribution from oxide-induced closure. A substructural investigation should be
done in order to examine the active slip planes and determine the effect of available slip planes on crack growth rates.

4. Since vacuum initiation is an issue in turbine engines, and there is no secondary orientation for in service blades, further investigation of the effect of orientation on crack growth in vacuum would be interesting. There could be a need to re-orient the blades if crack growth rates were slower for a different loading axis than (001).
REFERENCES


APPENDIX A

Specimen Size Effect on Fatigue Crack Growth

As stated in the experimental procedure, two different sizes of C(T) specimens were used for this study. Four specimens with a W=40 and an orientation of (001)[100] were tested in vacuum at 10Hz. The specimens tested at 650°C to 850°C yielded accurate data since the fracture plane was nominally perpendicular to the loading axis. However, the 550°C specimen fractured on what appear to be octahedral planes. Because the crack grew off of the plane normal to the loading axis by more than 12 degrees, this specimen was not considered in the main thesis. However, the change of crack path between the smaller specimen and larger specimen is interesting since a number of authors [26-28] have reported octahedral growth for temperatures in the area of 550°C. In Figure A.1.a., the 550°C, W=40 specimen is shown, which appears to have fractured on octahedral planes. The surface is macroscopically jagged, but each facet is smooth and mirror like. The facets can be seen on a smaller length scale in Figure A.1.b. and Figure A.1.c.

As discussed in Section 2.3.1., octahedral fracture was seen in round bar specimens, where ΔK levels are not measured. This is important to note since a large
**Figure A.1.** Images of the 550°C, W=40 specimen.  

a) Optical microscopy image of the fracture surface (top) and crack profile (bottom). 

b) Optical microscopy image of the large, smooth, mirror-like facets. 

c) High-resolution SEM image showing the smoothness of the fracture, even on the nanoscale.
number of authors [23-25, 29-31] contribute the appearance of octahedral fracture to high $\Delta K$ values and not to a temperature effect, at least in this temperature regime ($>550^\circ$C). In fact, in a study by Müller et. al., both round bar and C(T) specimens were tested. The authors found that at 550°C, the round bar specimens preferred octahedral growth, while the C(T) specimens fractured perpendicular to the loading axis. They compare the results by saying that the LCF crack growth rates for {111} planes are proportional to crack growth rates at very high $\Delta K$ values during fatigue crack growth tests.

In this study, both specimens, W=40 and W=20, tested at 550°C at 10Hz in Vacuum were precracked and then began loading in part II of the test so that the starting $\Delta K$ was 13 MPa$\sqrt{m}$. Because the starting $\Delta K$ was similar, the change in crack growth is attributed to the sample size. In the larger sample there may have been residual stresses that increased the $\Delta K_{eff}$ to a value greater than the $\Delta K$ aimed for, and therefore the crack grew on octahedral planes instead of perpendicular to the loading axis as was expected.

Additionally, since this specimen was tests at 10Hz, there may be an effect from the frequency on the crack propagation. Studies have shown that at higher frequencies the dislocation structure is confined to slip bands [28]. Furthermore, at 550°C, there may not be enough time for the dislocations that form on the slip plane to recover. Because of this, any crack advancement will be on octahedral planes. However, in Leverant’s study, the difference in crack growth rates between 0.05Hz and 10Hz were
insignificant, suggesting to this author that the crack paths would have had to of been fairly similar. Figure A.2. shows the da/dN vs ΔK data for the 550°C specimens in vacuum. Even though the data is for the same frequency, 10Hz, there is a significant difference in crack growth rates between the two specimens, with the crack growth rate of the octahedrally fractured specimen two orders of magnitude lower.

**Figure A.2.** Crack growth rate vs. stress intensity factor for test conditions of 550°C, Vacuum, 10Hz, (001)[100]. Specimen sizes are shown in the legend.
Because the crack growth rate is so much lower, and in Leverant’s study the crack growth rates were similar in this frequency range, we can assume there was no effect of frequency on crack path.

From the discussion it is clear that the fracture of the specimens seem to have been purely a sample size effect. The larger specimen, W=40, cracked on octahedral planes because of the increased stress on the specimen caused by residual stresses.
APPENDIX B

Characterization of Low Cycle Fatigue in a Ni-based Turbine Disk Alloy

Abstract

Better microstructure and deformation mechanism-sensitive models for fatigue damage accumulation in Ni-based superalloys will allow more accurate prediction of engine component lifetime. In this study, low cycle fatigue tests were performed on a polycrystalline Ni-based superalloy used in turbine disks. Test specimens were subjected to different cooling rates to vary the size distribution of γ' precipitates, then tested in strain control at temperatures from 205-705°C. Following testing, γ' size and distribution were characterized using high resolution scanning electron microscopy (SEM). Fracture surfaces and deformation mechanisms operating in the bulk material were examined using transmission electron microscopy (TEM) and SEM. Failure was characterized by the formation of single or multifaceted initiation sites on the fracture surface. The local deformation mechanisms operative at the initiation sites were
studied by TEM analysis of thin foils selectively extracted from these facets using focused ion beam (FIB) techniques.

Introduction

Ni-based superalloys are used in the aircraft engine industry because of their excellent high temperature properties, which include good ductility, oxidation and hot corrosion resistance, and microstructural stability [4]. They are used in the hottest sections of the turbine engines for both disk and blade applications. Turbine disks, which operate at mid range temperatures (up to 800°C), are designed to be fatigue limited. Stresses due to centrifugal motion and forces from the attached blade cause mechanical fatigue in the bore (center) region of disks.

In the case of low cycle fatigue, we know that most of the lifetime is taken up by crack propagation, and that it is the initiation of a crack that will tend to limit the life of a part. Lifetime and fatigue mechanism studies have been done on disks [47-51]; however, the micromechanics of cyclic damage are not well understood [52], especially at the initiation site. In this study, the use of focused ion beam techniques will be utilized to observe dislocation structures at initiation sites. Dislocation structures present are similar to bulk deformation, with the exception of a dense dislocation region near the surface. The results will be discussed and conclusions about the appearance of the dislocation region will be made.
Experimental Procedure

In this study, uncoated René 104 specimens were cooled from the supersolvus temperature at different cooling rates in the range of 0.8°C/s to 1.4°C/s in order to produce a range of microstructures representative of in-service materials. Dogbone specimens with a gauge length of 6.5cm and a diameter of 1cm were used. Strain controlled tension-zero low cycle fatigue (LCF) tests were performed in air at 205-705°C. The mean strain, ε_mean, ranged from 0.2% to 1.26%. Specimens were tested at Metcut, and were given to OSU for further investigation after failure.

Samples were sectioned, polished and etched using a solution of 50mL lactic acid, 30mL nitric acid, and 2mL hydrofluoric acid. The size, distribution, and volume fraction of γ′ precipitates were characterized using a FEI Sirion SEM, and quantified using Fovea Pro image analysis software. Fractography was characterized using a FEI Quanta scanning electron microscope (SEM). Fracture surfaces were observed parallel to the loading axis and were characterized based on type and location of initiation. Tilt fractography [53-57], was used to identify the fracture plane of the facets of interest. Transmission electron microscope (TEM) foils were prepared conventionally and by using a focused ion beam (FIB). Using a FEI DB235, TEM foils were milled from internal and surface initiation sites, plucked in situ using an Omniprobe, and attached to a copper grid before final thinning. Deformation mechanisms working at the initiation
site and in the bulk material were observed using a FEI Tecnai TEM in scanning TEM (STEM) mode.

Plastic zone size was calculated using linear elastic fracture mechanics [58], while accounting for the test conditions. The plastic zone for a round bar with an internal initiation site was calculated. Equations for $K_i$ were found in Tada and Paris [59]. The crack length assumed for the calculations was 1μm, which was based on the observed initiation sites. Stresses used to calculate were the mid-life max stress, which is a reasonable estimate given the crack does not initiate immediately on the start of the test, but more likely, closer to mid-life.

Results and Discussion

Because of the different cooling rates, $\gamma'$ size and distribution varied at each temperature. No correlation was found between cooling rate and $\gamma'$ size. The volume fraction of $\gamma'$ was 50-60%, which is normal for wrought and powder metallurgy Ni-based superalloys of comparable strengths [43]. Specimens that had higher cooling rates (0.8-1.4°C/s) are investigated for the remainder of the study in order to better compare findings. A cooling rate of 1.4°C/s resulted in a microstructure with evenly sized secondary $\gamma'$ particles and a low tertiary $\gamma'$ content, and can be seen in Figure B.1.
Transgranular (faceted) grain initiation was found to dominate at all temperatures. Figure B.2. shows initiation sites seen at 538°C at 0.43% and 0.87% total strain range. Figure B.2.a. shows an internal initiation site, while Figure B.2.b. shows a surface initiation site. At all temperatures, the initiation site changed from multifaceted internal initiation to single facet surface initiation with increasing strain range. The trend toward surface initiation is well known and has been previously observed in many disk alloys [51, 60-62].
Figure B.2. Samples tested at 538°C. a) Internal initiation site seen at 0.43% total strain range. b) Surface initiation site seen at 0.87% total strain range.

The resulting overall dislocation structures from the internal initiation site, found in the specimen tested at 538°C to a total strain range of 0.43%, can be seen in Figure B.3.a. In Figure B.3.a. there is a dense dislocation region near the surface ~2μm in width, with multiple slip systems active in the area under the surface. Also observed are dislocation bands running into the foil from the surface, which appear to have emanated from the dense dislocation region. Tilt fractography studies indicate that the facet fractured along a \{111\} type plane. The substructure at the initiation site can be compared to the substructure away from the initiation site, or in the bulk deformed material, seen in Figure B.3.b. The same overall dislocation mechanisms are at work: slip on multiple planes as well as dislocation movement within bands.
Figure B.3. Samples tested at 538°C, 0.43% total strain range. a) Bright-field STEM micrograph of the substructure seen in the foil extracted from the surface initiation site. b) Bright-field STEM micrograph of the substructure seen in the bulk, away from the initiation site.

The substructure seen in the foil taken from the surface initiation site, found in the specimen tested at 538°C to a total strain range of 0.87%, can be seen in Figure B.4.a. A region of dense dislocations is seen directly near the fracture surface which extends into the foil \( \sim 1.5\mu m \). In the same foil, further away from the fracture surface, many active slip systems can be seen by an array of stacking faults. A comparison to bulk mechanisms, seen in Figure B.4.b., show that the same type of deformation mechanism is at work: slip on multiple planes. However, in the bulk dislocation bands are observed, which are not seen in the foil taken from the initiation site. The
occurrence of dislocation bands near the initiation site is not discounted, but the small region of the material observed may simply not contain a dislocation band.

Figure B.4. Samples tested at 538°C, 0.87% total strain range. (a) Bright-field STEM micrograph of the substructure seen in the foil extracted from the surface initiation site. (b) Bright-field STEM micrograph of the substructure seen in the bulk, away from the initiation site.

For both initiation sites, facets fractured along a {111} type plane, and a dense dislocation region was seen at the surface. We know that dislocations slip along {111} type planes in [110] directions. This dense dislocation region could have come to be next to the fracture surface in multiple ways. A dislocation band may have already been present in the material and the fracture may have occurred parallel to and along one
edge of the band. From observation of the dislocation structures within the bulk material, the dense dislocation region found near the surface is thinner than the observed bulk dislocation bands. Other reasoning for the dislocation bands suggests that the region may be the remnant plastic zone which was formed by the crack tip during crack propagation. The crack tip plastic zone calculation yielded a result of $\sim 1.2 \mu m$ for a plane stress condition and $\sim 0.7 \mu m$ for a plane strain condition. The result is on the length scale of the observed dislocation bands. Therefore, the most likely explanation for the appearance of a dense dislocation region next to the fracture surface is that it is the dislocation structure left from the plastic zone as the crack propagated.

**Conclusions**

The use of focused ion beam techniques to examine the substructure near the fracture surface is still a novel technique that helps researchers to better understand deformation mechanisms. In this study we concluded the following:

1. Initiation site changed from multifaceted internal initiation to single facet surface initiation with increasing strain range.

2. Using FIB techniques to study deformation near the crack initiation sites, it was found that the internal initiation site (low strain range) showed a dense dislocation region near the surface as well as dislocation bands and multiple slip
systems below the surface. The surface initiation site (high strain range) showed a region of dense dislocations near the surface and multiple slip systems elsewhere.

3. Initiation site deformation structures were similar to those seen in the bulk of the same specimen.

4. Plastic zone calculations were compared to the observed dislocation region next to the fracture surface. Calculations indicate that the dense dislocation region is caused by the plastic zone during crack propagation.

The authors gratefully acknowledge NASA for providing funding through the Propulsion 21 project.
APPENDIX C

Fatigue Behavior in Monocrystalline Ni-Based Superalloys for Blade Applications


Abstract

Ni-based superalloys are used for turbine airfoil applications due to their excellent high temperature properties. Alloy design has historically focused on creep resistance as the critical design limiting parameter. Recently, the focus has shifted to include fatigue resistance, resulting in the need to better understand the effects of alloy microstructure on fatigue crack initiation and propagation.

In this study, sustained peak low cycle fatigue tests of coated monocrystalline René N5 specimens oriented with the loading axis along [001] were conducted in strain control in the temperature range of 980-1100°C. Tests were performed with either a tensile dwell (hold) time or a compressive dwell time. Fracture surfaces were characterized via scanning electron microscopy (SEM). Microstructural analysis was
performed using a high resolution SEM. Far field deformation mechanisms were examined perpendicular to the loading axis and parallel to the loading axis using transmission electron microscopy. The occurrence of creep-fatigue interaction was characterized by the different types of rafting seen, and the deformation mechanisms varied between specimens with compressive hold time and tensile hold time.

**Introduction**

Nickel based superalloys have been used historically in the aerospace industry for turbine airfoil applications. The alloys are useful at high temperatures because of their creep resistance, along with hot corrosion and oxidation resistance [3, 13]. Until recently, creep has been the limiting factor for turbine airfoils, and thus creep mechanisms have been the subject of numerous studies [3, 13, 63-66]. Early generation alloying of monocrystalline alloys was developed to improve creep resistance by adding slower diffusing elements, such as rhenium and ruthenium. Rhenium additions improve creep rupture life and oxidation resistance [5, 67], while ruthenium decreases overall density compared to rhenium, stabilizes the microstructure, and increases oxidation resistance [5, 6]. Additionally, creep resistance increases with a decreasing number of grain boundaries, and therefore the invention of the monocrystalline blade increased creep resistance. Coincidentally, fatigue resistance has tended to increase with
improvements in creep resistance. However, more recently fatigue failures have been experienced. Consequently, fatigue mechanisms have become of interest.

It is known that engine efficiency increases with increasing temperature, and therefore the need for alloys that can withstand higher temperatures is necessary. Past engine designs have been limited by the melting temperature of the blade material, and ceramic systems are not feasible due to fracture issues. However, more recent changes in modern high pressure turbine (HPT) blade designs incorporate cooling channels, which allow an increase in the engine operating temperature while keeping the blade at an acceptable maximum temperature. The introduction of cooling channels has caused local stresses, especially within the thin regions between the channels, and temperature gradients across the blade to increase. In these HPT components, experience and experimental studies have shown that fatigue can be a life-limiting factor. The changing limitation from creep to fatigue has highlighted the need for a comprehensive study of fatigue deformation mechanisms, as well as creep-fatigue mechanisms, since the engine operates at high temperature where creep occurs.

Previously, creep-fatigue studies have observed that cyclic creep decreases lifetime compared to creep alone [68]. A study by Zrnik observed fracture surfaces and dislocation structures in specimens that were subjected to static loading, cyclic loading and cyclic loading with a hold at maximum load. Static tests were performed in tension while the cyclic tests were tension-tension, with an R-ratio ($P_{\text{min}}/P_{\text{max}}$) of 0.0125. After static loading, tangles of dislocations were present in both the $\gamma$ and $\gamma'$ phases and the
fracture surface was indicative of pure creep. Under cyclic loading conditions, as the hold time decreased to 0 (purely cyclic loading), the dislocations became more confined to the matrix phase alone. The fracture surfaces of specimens with longer hold times were similar to specimens failed in pure creep, while specimens with shorter hold times had fatigue striations, like the fracture surfaces seen in the pure fatigue case [69]. While these observations provide insight into the transition from creep to creep-fatigue to fatigue deformation, the results are only representative of the tension case. Furthermore, no observations of the microstructural changes with deformation were noted.

The object of this study is to characterize the sustained peak low cycle fatigue behavior of René N5, a second-generation single crystal nickel based superalloy containing 3% rhenium. Tests with tension and compression dwell were performed. We observe that fracture modes and microstructural evolution differ between compression and tension dwell. The deformation character for each of the test types was found to be dislocation network formation and γ’ shearing, respectively. Creep-fatigue interactions influence the deformation behavior more than either creep or fatigue alone.
Experimental Procedure

For this study, the material of focus was monocry stalline nickel-based superalloy René N5. Specimens were prepared by GE Aviation and tested by Metcut (Cincinnati, OH). Coated, cylindrical specimens oriented with the [001] axis parallel to the loading direction were tested in air at 980-1100°C (1800-2000°F). Sustained peak low cycle fatigue (SPLCF) tests were performed in total strain control, with a total strain range between 0.4 and 1.2 percent, and a 2 minute dwell (hold) time in either compression or tension, as illustrated in the strain vs. time schematics in Figures C.1 and C.2, respectively. The corresponding stress vs. time behavior is also shown schematically. During each cycle, the material yields during loading to peak strain. During the hold time, stress relaxation occurs which coincides with creep in the material. Over the course of the compression hold experiment, the mean stress increases and a positive mean stress develops by mid-life, while in the tension hold experiment, the mean stress decreases but stays positive. The peak stress stabilized after about 10-20% of the total life, and was less than 350 MPa for all specimens. All specimens failed under 20,000 cycles. The fractured specimens were provided to OSU for analysis.
Figure C.1. Schematic of applied strain during cyclic loading with compression hold time and the resulting stress response evolution with time.

Figure C.2. Schematic of applied strain during cyclic loading with tension hold time and the resulting stress response evolution with time.
Fracture surfaces were characterized using an FEI Quanta scanning electron microscope (SEM). The fracture surfaces were characterized parallel to loading axis. The specimens were then sectioned for both SEM and transmission electron microscopy (TEM) analysis. Microstructures and substructures were investigated parallel and perpendicular to the loading axis, having normal directions of [100] and [001] respectively. Samples with a [100] normal were oriented using orientation image mapping techniques. Specimens for SEM were mounted in conductive Bakelite and etched with a solution of 50mL lactic acid, 30mL nitric acid, and 2mL hydrofluoric acid. The two phase $\gamma/\gamma'$ microstructure was characterized using a high resolution FEI Sirion SEM. TEM foils were made using conventional preparation techniques. All foils were jet polished using a solution of 10% hydrochloric acid and 90% methanol at -20°C and 13V. The substructures were characterized with an FEI CM200 TEM. Burgers vectors were determined by $g \cdot b$ analysis. Additionally, the thermally exposed but undeformed material obtained from the grip ends of a specimen was examined for comparison purposes.

**Results and Discussion**

A comparison of the fracture surfaces in specimens with compressive and tensile dwells shows different failure mechanisms operating under each condition. Fractography indicated that specimens with compression hold cycles failed similarly at
all temperatures surveyed. A representative fracture surface can be seen in Figure C.3.a., which has multiple crack initiation sites at the surface (A and B), with only one of the initiation sites (A) growing to form the main crack. Multiple surface initiation sites are common at high temperatures for coated specimens. Fatigue striations were observed, and a representative section of a fracture surface is shown in Figure C.3.b. The crack extension per cycle decreased during testing as would be expected in a total strain control test since the peak applied stress intensity will decrease as the specimen becomes more compliant.

![Fracture surface of specimen with compression dwell. Arrows indicate surface initiation sites.](image1)

![Striations on the fracture surface of a compression dwell specimen. Arrow indicates direction of crack propagation.](image2)

**Figure C.3.** a) Fracture surface of specimen with compression dwell. Arrows indicate surface initiation sites. b) Striations on the fracture surface of a compression dwell specimen. Arrow indicates direction of crack propagation.
The decrease in crack growth rate became more dramatic in specimens tested at higher temperatures. This was determined by the closer spacing of fatigue striations at longer crack lengths. The crack wake of each specimen was oxidized. Oxidation, which is prevalent at high temperatures, can increase crack closure and could be a contributing factor to the slowing crack propagation. Also, the plane of crack propagation changes from perpendicular to the loading direction during initiation to steep angles to the loading axis during propagation. Assuming the fatigue crack growth process is dominated by mode I loading, this transition from mode I to mixed mode loading may reduce the driving force for crack growth.

In the case of the tension hold, crack extension by creep deformation is observed in contrast to typical cyclic propagation. In this type of failure, seen in Figures C.4.a. and C.4.b., there are flat, shallow dimples with evidence of casting pores. As these voids grow, they coalesce and the specimen finally fails. The fracture surfaces of the specimens were macroscopically flat compared to the compression hold specimens. In the tension dwell case, oxidation is seen over the entire surface, but does not appear to be as thick as the oxidation observed on the compression dwell tests. This oxidation most likely occurred when the samples were finally fractured at temperature, since ductile failure happens by the coalescence of voids that may not be exposed to the air during the SPLCF tests.
Figure C.4. a) Fracture surface of specimen with tension dwell. No obvious initiations sites at surface are observed. b) Fracture surface of specimen with tension dwell. Shallow dimples can be seen, with casting pores throughout the surface.

The $\gamma/\gamma'$ microstructures in the SPLCF specimens with compression and tension dwell time were characterized and compared with the microstructure of the undeformed thermally exposed material. In the case of the thermally exposed material, cuboidal $\gamma'$ dominated the structure, with no preferential alignment of the $\gamma'$ phase (i.e. rafting). The microstructure on the (001) and (100) crystallographic planes can be seen in Figures C.5.a. and C.5.b., which are images taken parallel and perpendicular to the loading axis, respectively. In all figures, the $\gamma$ phase is a light gray while the $\gamma'$ phase is a dark gray.
In the compression dwell tests, rafts formed parallel to the loading axis, referred to as “p-type” rafting. The flat sides of the rafts can be seen in Figure C.6.a. Characterization of this structure transverse to the loading axis shows that the rafts formed in orthogonal orientations, both parallel to the loading axis, seen in Figure C.6.b. In the case of the tension dwell tests, there was “n-type” rafting, or rafts perpendicular to the loading axis. Figure C.7.a. shows the rafting parallel to the loading axis, while Figure C.7.b. shows the flat sides of the rafts, which are perpendicular to the loading direction. Both types of rafting are well documented for nickel-based superalloys with a negative $\gamma/\gamma'$ misfit [3, 13-15].
Figure C.6. Microstructure of specimen with compression dwell. P-type rafting is observed. A) Parallel to load- [100] normal, loading axis is vertical. B) Perpendicular to load- [001] normal- specimen axis is orthogonal to image.

Figure C.7. Microstructure of specimen with tension dwell. N-type rafting is observed. a) Parallel to load- [100] normal, loading axis is vertical. b) Perpendicular to load- [001] normal- specimen axis orthogonal to image.
In both tension and compression dwell specimens, the $\gamma'$ phase became the continuous phase after loading, in contrast to the thermally exposed material where $\gamma$ is continuous. Additionally, a topological inversion has taken place, where the $\gamma'$ phase has a larger area fraction after deformation. From the figures, it can be observed that the $\gamma$ phase is altered compared to specimens tested under creep alone. In creep, long continuous $\gamma$ and $\gamma'$ channels are developed [3, 65], but in the SPLCF specimens, the $\gamma$ phase has shorter sections, as seen in Figure C.7.a. Here the $\gamma$ channels are interrupted by the $\gamma'$ phase coalescing with neighboring rafts. This is attributed to the creep-fatigue interaction, where the fatigue causes additional deformation that creep alone could not accomplish. Because interrupted tests were not done, it is not clear whether the rafts were ever long and continuous as expected during monotonic creep. It is known that rafts are caused by the superposition of the external stress and the coherency stress, which results in an unequal stress state developing in the $\gamma$ channels during deformation [1, 3, 10, 13, 70]. During tensile loading, the external stress reduces the compressive strain energy in the vertical channels relative to the horizontal channels. Stresses in the horizontal channels are relieved by the formation of dislocation networks. Because the stresses are not relieved in all directions, a gradient in elastic strain energy is formed between the horizontal and vertical channels. This gradient is the driving force for diffusional mass transport, and the rafted structure forms [70, 71]. As seen in the schematics in Figures C.1. and C.2., the cyclic loading causes both compressive and tensile stresses in the specimen. Therefore the rafts do not fully develop because, in the
case of cyclic fatigue, dislocations are forming in both horizontal and vertical channels, reducing the driving force for diffusional mass transport, and therefore rafting.

Even in the thermally exposed material, in the absence of applied stress, dislocations are present at $\gamma/\gamma'$ interfaces, probably to accommodate the negative misfit of the $\gamma'$ precipitates due to the rhenium additions [72]. Figure C.8 shows that these dislocations travel through the $\gamma$ channels and loop around the $\gamma'$ precipitates. The dislocations have burgers vectors of $a/2<110>$ and $a/2<T10>$. Intersecting dislocations form nodes and dislocation networks. No rafting was observed, however, after thermal exposure. In the absence of externally applied stresses, the dislocations move because of a combination of thermal gradient stresses and the stresses associated with the negative misfit of the $\gamma/\gamma'$ interface. The dislocations achieve lower energy configuration by arranging into the network formation.
**Figure C.8.** Bright field TEM image of the thermally exposed material. Dislocation networks form at $\gamma/\gamma'$ interfaces to accommodate the negative misfit. Loading axis is normal to image.

In the compression hold specimens, extensive dislocation networks formed in the $\gamma$ phase, as seen in Figure C.9. Investigation of a large section of the sample revealed that dislocation networks formed in both vertical and horizontal $\gamma$ channels, along with $\gamma/\gamma'$ interfaces. The dislocations in the networks have burgers vectors of $a/2<011>$, $a/2<011>$, and $a/2<110>$. An isolated dislocation with a burgers vector of $a/2<011>$ was found within the $\gamma'$ phase that connected to the dislocations in the matrix networks. This indicates that the applied stress was large enough to cause dislocations to shear the $\gamma'$ phase. While there were isolated dislocations shearing the $\gamma'$ phase, most of the deformation appears to be confined to the $\gamma$ phase. Therefore, the
dominant deformation mode was dislocation movement within the $\gamma$ channels and not by a $\gamma'$ shearing mechanism.

![Image](image.png)

**Figure C.9.** Bright field TEM image of specimen with compression dwell. Dislocation motion in the $\gamma$ channels dominates the deformation. Loading axis is nearly horizontal.

In the tension hold specimens, the dislocation networks were confined to the $\gamma'/\gamma'$ interfaces, shown in Figure C.10. The dislocation networks were again seen in both horizontal and vertical channels. Multiple dislocations with burgers vectors of $<101>$ were observed in the $\gamma'$ phase. Screw dislocations were observed shearing through multiple $\gamma'$ particles at once, while others were bowing within the $\gamma'$ phase. Since the dislocation networks were confined to the interface, the $\gamma'$ appears to be an important
deformation mode, suggesting that at least at this stage of deformation, the \( \gamma' \) phase is acting as the continuous “matrix” phase.

Figure C.10. Bright field TEM image of specimen with tension dwell. Shearing of \( \gamma' \) particles was the primary deformation mode. Loading axis is nearly horizontal.

TEM observations from both samples reveal that dislocation networks have formed in both vertical and horizontal \( \gamma \) channels and along all \( \gamma/\gamma' \) interfaces. As stated earlier, the formation of dislocations along both types of channels decreases the driving energy for diffusional mass transport. Therefore it is evident through the TEM studies that the cyclic loading is deterring raft formation.
Conclusions

In this work, the microstructural changes occurring in SPLCF specimens with tension and compression dwell times were compared with thermally exposed material. It was observed that the combination of creep and fatigue loading caused short, discontinuous rafts, in comparison to rafts observed in monotonic creep. TEM analysis confirmed that dislocations were moving in both horizontal and vertical channels, and therefore deterring raft formation because the driving force for diffusional mass transport is being reduced by the strain accommodation. The reduced driving force does not allow long continuous rafts to form as easily, and the short, discontinuous rafting was observed. Dislocation network formation and $\gamma'$ shearing appears to be the main deformation mode for compression dwell and tension dwell, respectively. For each of the SPLCF specimens it is difficult to distinguish what deformation is due to monotonic loading or cyclic loading. This highlights the need for a separate fatigue study, where the fatigue mechanisms can be described before attempting to analyze the combined, and probably synergistic, effect of creep and fatigue.

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