Quantitative Metallurgy of Anisotropic Nickel-Base Superalloys Under Tensile and Fatigue Loading

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QUANTITATIVE METALLURGY OF ANISOTROPIC NICKEL-BASE SUPERALLOYS UNDER TENSILE AND FATIGUE LOADING

by

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ABSTRACT

Nickel-base superalloys are ideal materials for application in high temperature and high stress environments. Their resistance to both heat and corrosion makes these metals well-suited for use as components in engines and turbines. There has been much interest in characterizing the mechanical properties of Ni-base superalloys under severe conditions. Either large monotonic loads or cyclic loads are the most investigated. Also, research efforts tend to focus on the influence of microstructural features of fatigue life, and they accomplish this through qualitative observation. Presented here is both quantitative and qualitative analysis done on Ni-base superalloy specimens that have been subjected to multiple types and degrees of tensile and fatigue loading. The quantitative fracture features referred to as the fracture length deviation and surface roughness are the focus of the analysis. The method of quantifying these features is a focal point of this work and is described in detail with the intention that others will be able to apply it in future research. The research presented here also catalogues an extensive collection of microscopic-level images obtained with a specific optical microscope that allows a topographical view to be taken of the fracture surface. This type of analysis allows comparisons to be drawn across multiple samples of both the directionally solidified and the single crystal variety of the superalloy, in addition to distinguishing the different effects of the material orientation.
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CHAPTER 1:
INTRODUCTION

Since turbomachinery operates at high temperatures for long durations of time, the systems and the materials that comprise them are susceptible to fatigue. In turbomachinery, superalloys are the desired materials given their high tolerances for extreme heat and fatigue. In gas-powered engines specifically, Nickel-base superalloys are often the metal of choice when casting the blades of the turbine. The purpose of this research is to demonstrate a novel approach that quantifies the texture and orientation of the fractured test specimens. This thesis extends from research recently conducted on the same materials [1] [2]. The previous research dealt with modeling the performance of DS and SX type samples under various fatigue conditions, this research focuses on developing a straightforward approach to characterize and compare the fracture behaviors that resulted from failure under fatigue. The comparison will be done both quantitatively and qualitatively, using the macroscale and microscale topographical images of the tested specimens. The fragments of specimens being analyzed were subject to either tensile stress or low cycle fatigue (LCF).
2.1 Nickel-Base Superalloys

A Nickel-base superalloy is the subject material of this thesis. The material is similar to the Inconel 792 (IN792) superalloy, but has a slightly different composition (the exact composition is proprietary information). Nickel-base (Ni-base) superalloys are resistant to both corrosion and heat, often being capable of withstanding temperatures up to 1000°C [3]. Even at elevated temperatures, Inconel superalloys have been shown to have high tensile and creep strength [4] as well as low cycle fatigue resistance [5]. Also, Ni-base superalloys are relatively inexpensive to manufacture [6]. These qualities make it an ideal choice for machine components that are subjected to high temperatures and stresses. This is why the main application of Ni-base superalloys is using them to manufacture the rotating parts of land-based turbines and gas powered aircraft engines [5] [7].

There are a variety of Ni-base superalloys, with each different type varying in composition, grain size, and heat treatment. Inconel 792 is composed of different quantities of the following metals: Ni, Cr, Co, Mo, W, Ta, Al, Ti, Hf, Nb [8]. Its mechanical properties can vary slightly depending on the particular ratio of element composition and the processing method. Generally, IN792 can withstand temperatures from 600°C to 900°C depending on its exact composition [6]. This makes it an attractive alloy for components in extreme temperature environments. Provided for reference, Table 2-1 lists common types of this superalloy along with the ratio of elements in their compositions.
Nickel-base superalloys like IN792 possess the previously mentioned desirable properties as a result of their composition. These properties can be further improved depending on how the metal is processed. The traditional process involves melting the metal into liquid form and pouring it into a mold so the resulting superalloy cools to a desired shape. This is called conventional casting (CC). Direct solidification (DS) is a variant method of casting molten metals in a mold in such a way that cavities are prevented from forming as it cools. The DS method allows a lattice pattern of crystals to form within the superalloy. The process involves using a temperature gradient to slowly cool the molten metal, which gives the superalloy a columnar grain structure [8]. This, for example, can increase the creep rupture resistance of the metal [8]. Superalloys formed like this are called “DS superalloys.” A similar but different solidification process is used to forge single-crystal metals or “SX superalloys.” For these metals, the cooling technique only allows the propagation of one crystalline pattern throughout the material, making the microstructure more ordered and consistent. The benefits of this include more resistance to certain types of deformation and increased temperature tolerance [8]. The crystalline structure also makes the superalloy have anisotropic properties, meaning that the
behavior is dependent on the direction of application. In Figure 2.1, turbine blades are presented with their microstructures magnified to show a comparison of the grain structures of the three different manufacturing methods described. This study focuses on comparing the DS and SX forms of the superalloy.

![Grain structures of SX, DS, and CC turbine blades respectively](image)

**Figure 2-1:** Grain structures of SX, DS, and CC turbine blades respectively [8].

As a disclaimer, the samples used in this study were provided by a private turbomachinery company. Therefore, the results of the tests done on the specimens are their proprietary property and as such cannot be revealed here. The specific composition of the superalloy is also proprietary. However, the general results and normalized finding can be discussed.

### 2.2 Mechanisms Deformation, Crack Initiation, and Rupture

Nickel-base superalloys have high yield and ultimate tensile strengths that remain consistent until exposed to much higher temperature. This is the case for IN792 as well; however, at approximately 750°C the strength of the metal decreases, and it becomes much more ductile [6].
Figure 2-2: Tensile results from previous study of polycrystalline IN792: (a) yield strength ($\sigma_s$) and ultimate strength ($\sigma_b$) and (b) elongation and reduction of area [6].

It is worth noting that the ductility starts to decrease after 850°C. This temperature-dependent property influences how IN792 deforms. The more ductile a metal is, the harder it is for stress to cause rupture. Rupture is reached when part of a material completely fails under stress.

The deformation behavior of superalloys is largely dependent on their microstructure. The grain boundaries that result from the different kinds of processes affect the mechanical properties of the resulting metal. For example, creep deformation is initiated in polycrystalline materials when their grain boundaries slip across each other [8]. Creep is time dependent, which is why Ni-base superalloys such as IN792 are so resistant to them since their microstructures are designed to resist deformation under long periods of time under stress and high temperatures. This is the result of the precipitates (hardened particles dispersed in the metal as a result of heat treatment processes) between the grain boundaries of the superalloy being exceptionally resistant to shearing [8][9]. The grain boundaries themselves provide resistance to dislocation movement [9].

Shear stress tends to affect the deformation behavior in two different ways depending on the magnitude of the strain rate and temperature, though it has been observed that temperature is a
much greater factor than strain rate [7]. At lower temperatures and higher strain rates, sections of the test material are subjected to strain hardening before fracturing occurs. At higher temperatures and lower strain rates, sections of the material instead softened and become easier to elongate [10]. This gave way to recrystallization (the process of defect-free grains growing to entirely replace the deformed grains). Observations showed that at the fracture regions the grains were elongated in both cases. However, the elongation was much more prevalent at the higher temperatures and lower strain rates [10]. Comparatively, the grains in the fractured regions of the samples tested at lower temperatures and higher strain rates mostly kept their original dimensions with only a little elongation in the tensile direction [10].

The fatigue properties of Ni-base superalloys depend on both the microstructure and the cyclic variations in temperature and stress that are being experienced. Turbomachinery, like aircraft engines, alternate between high and low speed rotations. This causes fluctuations in the frequency that the blades experience, which in turn cause fatigue and potentially deformation over time [8]. Precipitates can shear or slip throughout the microstructure, which can lead to crack initiation at the surface of the superalloy. Cracks tend to initiate at locations in the microstructure that are variations or defects resulting from the processing of the superalloy [8]. Fatigue cracks will often propagate along the same plane that the crack initiated in, and generally starts at the surface and then grows inward. This can lead to rupture.

Under fatigue cycling, it has been previously observed that the effect of temperature on DS specimens tends to be reliant on material orientation. Longitudinal cracks (cracks that propagate with grains that are in the stress direction) seem to have no dependence on the temperature conditions [11]. Transverse cracks (cracks that propagate in grains that are perpendicular to the
stress direction) do seem to rely a little on the testing temperature [11]. Diagonal cracks (cracks that propagate in grains diagonal to the stress direction) appear to largely depend on the testing temperature, or at the very least more than the other two type of cracks [11]. Another study discovered that a main crack starts at the edge of a center-through-hole (in that study, a small hole purposely placed to guarantee crack initiation in the middle of the specimen) and subcracks are created at high angle grain boundaries that are about perpendicular to the load axis [12]. These two types of cracks propagate and combine and form a “network of crack paths” [12].

Single crystal specimens have shown to have good creep resistance as a result of their lack of grain boundaries [13]. While their fracture behavior is not influenced by grain boundaries like the DS specimens, SX specimens rupture in similar ways. Their crystallographic structure affects how stress is distributed and this stress distribution can become more complex when the crystals are not aligned perfectly [14][15]. Previous studies show that cracks initiate on the surface of SX specimens, possibly due to micro-pores on the surface serving as points for stress concentration [16][13]. These cracks propagate perpendicular to the load direction or along slip planes [13].

The texture and size of cleavages (the separation of the crystal lattice planes) can be influenced by the loading temperature. It was observed that a specimen tested at a lower temperature had larger and less textures cleavage facets [13].

2.3 Mechanical Testing of DS and SX Nickel-base Superalloy Specimens

The Ni-base superalloy material studied here is taken from a previous study conducted at UCF [1] [2]. The samples underwent two different types of tests. Tensile tests were performed on fourteen specimens, and fatigue tests were performed on the other twenty-three. An example of the dog-bone shaped specimens that are used is included in Figure 2-3 with its dimensions for
reference. The specimens were cut from slabs of the superalloy at different angles. The slab depicted in Figure 2-4 is an example of the specimens being cut out at a 45°.

![Dimensions of undeformed specimen](image)

**Figure 2-3:** Dimensions of undeformed specimen [1].

![Specimens](image)

**Figure 2-4:** a) Nickel-base superalloy SX slab. b) Slab with specimens cut out. c) Undeformed specimen.

Half of these slabs were fabricated using direct solidification and the other half were done using the single crystal cooling technique. Figure 2-5 illustrates the difference in the crystal grain structures when the specimens are cut at different angles in both the DS and SX slabs.
It should be noted that the [001] SX and [010] SX specimens are microstructurally identical because of the nature of single crystal alloys. Since specimens are grouped by their microstructures in the analysis section of this thesis, [001] SX and [010] SX specimens are in the same group. However, the naming designation of each specimen is consistently based on the angle it was cut out of its respective slab, regardless of the groups they are put into for analysis. This is to ensure the naming conventions are consistent throughout the thesis.

2.3.1 Tensile Testing
The tensile tests were uniaxial, conducted using a load-cell and a direct contact extensometer. The set-up of the testing apparatus is shown in Figure 2-6. Computer application software was used to run and record the experiments. Linear variable differential transformers, LVDTs, were used to measure and convert mechanical deformation into electrical voltage. This change in voltage could then be used to calculate the displacement of both ends of the gauge section. All tensile tests were strain-controlled under isothermal conditions.
2.3.2 Fatigue Testing
A servo-hydraulic axial fatigue machine was used for low cycle fatigue (LCF) experiments. This machine applies uniform strain through the cross section of a test specimen. The device is shown in Figure 2-7. A computer program controlled these tests as well. Like the tensile testing, linear differential variable transformers are used to convert mechanical deformation into electrical voltage for data recording.
Fractography of DS and SX Materials

Fractographic examination is important. When similar materials are tested under similar conditions, the resulting fractures will always be slightly different from one another. No fracture surface can be exactly identical to another one due to tiny variations in the individual mechanical properties, microstructure, and even the environmental interactions of a sample [18].

To ensure consistency, this thesis references the objectives of fractographic examination described in literature [18] for guidance, as well as using the terms it defines to describe fracture
elements in the qualitative analysis section. To start, the general objectives to adhere to when studying a fractograph are:

1. Identify the type of fracture: brittle or ductile (see Figure 2-8) [18]
2. Determine the crack path [18]
3. Identify the zones corresponding to the three stages of fracture, which are: crack nucleation, crack growth, and final separation [18]
4. Identify the operating fracture mechanisms [18]
5. Identify the factors which influence the fracture process [18]

![Fracture classification and macroscopic characteristics](image)

**Figure 2-8:** Schematic representation of (a) the fracture classification according to amount of plastic deformation and (b) typical macroscopic characteristics of a fatigue fracture [18].

The following is a list of the terms used to distinguish different kinds of fracture lines, with definitions of them taken from the second chapter of the *Fractography and Failure Analysis* textbook [18].
Chevrons: V-shaped ridges that point towards the crack initiation location; formation is due to curved crack propagation [18].

Beach Marks: long, well-defined arch-shaped concentric lines; the arch origin point corresponds to the crack initiation site and the propagation direction of the arches corresponds to the crack propagation; usually formed when a crack stops and re-starts its propagation [18].

Ratchet Marks and Ridges: multiple cracks that start on different planes and then interconnect with each other through secondary cracks that spread perpendicular to the fracture plane; their orientation is parallel to the direction of the crack growth [18].

Shear Lips: protrusions formed on the outer edges caused by high shear stresses and abrupt alterations in the state of stress towards the end of fracture; allows for identification of the final fracture zone; usually shows a neck [18].

Cleavages: macroscopic cleavages are brittle fractures on a single facet; often cracks appear as ranges in a “river-like” pattern; propagation direction corresponds to the direction that the ranges align in [18].

Multiple Cracking: cracks that branch out occur frequently in brittle materials; commonly a ‘Y’ or ‘T’ configuration will be created; in the case of the Y configuration, the crack propagates in the direction of the branches [18].

Previous analysis has been conducted on mechanically tested DS and SX Ni-base superalloys. Other researchers have used fractography to obtain a better understanding of fracture behaviors of this material. By analyzing SEM images, like the ones in Figure 2-9, and comparing them with experimental data, the researchers of this study were able to draw useful conclusions. It was determined that the lack of grain boundaries in SX materials did grant good creep resistance [13].
as observed in other research. The only visible deformation bands that were observed were in the [011] orientation [13]. The researchers also linked the cleavage texture and size of fractures to the test temperature [13].

![Fracture Images](image)

**Figure 2-9:** Fractographs of fractured specimens: (a) [001] orientation at 825 °C; (b) [111] orientation at 825 °C; (c) [001] orientation at room temperature [13].

One study that analyzed specimens of a IN792 superalloy used fractography to determine fracture characteristics that correspond to testing temperatures. Observations in the fracture surface of the specimens tested at 550°C, 600°C, 650°C, 700°C, 850°C, and 900°C revealed the cracks initiated at the center of the specimen, which then propagate in a branching-like pattern [6]. Both dimple and transgranular cleavage can be seen, indicating the tensile crack propagates
slowly at first as plastic deformation before fracturing quickly [6]. For the specimen tested at 760°C, the fracture surface has several more visible crystal planes, meaning the fracture mode is predominantly cleavage [6]. Images of the tensile tested specimens from this study are provided in Figure 2-10.

![Figure 2-10](image)

Figure 2-10: The fracture surface SEM images of an IN792 superalloy. Tensile tested specimen at: (a) 550°C, (b) 600°C, (c) 650°C, (d) 700°C, (e) 760°C, (f) 850°C, and (g) 900°C [6].

Another effort that involved fractography studied the crack growth of a different directionally solidified superalloy, CM247LC [19]. The purpose of these tests was to observe crack initiation behavior at a pre-machined notch in the test samples [19]. Macroscopic images allowed the authors to observe where the crack initiated in the notch and how it propagated under creep fatigue. The authors determined transgranular cracks initiate in the notch direction and does not grow until brittle-like fracture occurs [19]. The displacement of the notch caused by fatigue can be seen in Figure 2-11 [19].
Figure 2-11: Observational results of crack growth behaviors under fatigue conditions for CM247LC [19].
(a) 930°C, 0.01 Hz, $t_f = 165$ h [19]. (b) 930°C, 1.0 Hz, $t_f = 35$ h [19].

Referencing previous research, such as this, provides a beneficial starting point to continue similar research. For example, because of the conclusions reached by Zhang et al. [13], it is known that micro-pores, crack initiation points, dislocation lines, and cleavage facets are important fracture features.

Additional fractographs from previous research are included in Figures 2-12, 2-13, and 2-14 with brief descriptions for more reference of previous fractography that emphasized the qualitative features of specimens.
**Figure 2-12**: Typical fracture surfaces (a,b,c,e,f,g,i,j,k) and longitudinal sections (d,h,l) of M951G alloy specimens subjected to tensile creep tests at different conditions. (a)-(d), (e)-(h), (i)-(l) specimens tested at 900°C under 270, 360, and 400 MPa respectively [20].

**Figure 2-13**: The SEM micrographs of the tensile fracture surface of DD407 SCNBS with a strain rate of 0.001/s and the temperature of (a, b) 293 K and (c, d) 873 K [21].
Figure 2-14: Fracture surface of a single crystal Ni-base superalloy specimen with two distinct cracking modes [22].

The cases presented are good examples of qualitative analysis; defining features non-numerically based on observation. This is more commonly practiced than using quantitative metrics to define features, such as roughness. However, roughness calculations are not absent from research efforts [23], but it does require extra steps to be taken in fractography. When the objective is to find the roughness of a fractured specimen, it is common to section the test specimens first and then use a scanning electron microscope to obtain images [23]. With those images, the profile lengths can be measures and used to calculate the linear roughness [23]. Previous research has made good use of this method to record roughness values on the microscale and studying how the loading factors affect them [23]. An example of this method of application is shown in Figure 2-15. The fractographs show the relationship of the stress intensity applied to the specimens and the resulting roughness: higher stress intensities yield lower roughness [23].
2.5 Knowledge Gaps

Previous research has utilized fractographs to observe deformation behavior in Ni-base superalloys. Often the principal focus is on microscopic analysis. Many studies researched the effects that mechanical properties have on cycle life through fatigue tests and then qualitatively make observations at the resulting microstructures. Comparatively, there are fewer current studies that choose to focus on features such as fracture surface roughness and fracture angle. These types of failure qualities may offer some interesting insights about the mechanical behavior of Ni-base superalloys when taken into consideration alongside what is already known about their microstructures and mechanical properties. Naturally, some studies have commented on how factors such as temperature have influenced the roughness of a fractured sample [24]
[25] [26] [27], but often it is not the focus of research. Usually it is observation-based with no measurable metric attached. In instances where roughness is numerically defined [23], it is common to section the test specimens and use a scanning electron microscope to obtain images and measurements. Alternatively, serial sections can be taken in order to reconstruct the fracture surface three-dimensionally, and then the profile lengths can be measured in the 3-D image [23].

A method of quantitatively measuring the fracture roughness will be presented here with the intention of developing a more convenient approach for research in future works. This method will allow the roughness parameter to be calculated using measurements obtained from images taken with powerful optical microscopes, without needing to section the sample. Quantitative analysis will also be used to draw comparisons across multiple samples of both the DS and SX variety of the material, as well as distinguishing the different effects of material orientation. For this reason, multiple specimens are used for analysis. A collection of all the images taken of the specimens are included for reference, presented alongside their test conditions and calculated roughness values.
CHAPTER 3: EXPERIMENTAL RESULTS AND ANALYTICAL APPROACH

3.1 Introduction

As previously discussed, the specimens involved in this research underwent two different types of tests, tensile and low cycle fatigue, at temperatures ranging from 752°F to 1832°F. The tensile tests were conducted in order to characterize the mechanical properties of the superalloy and how those properties can differ depending on the material orientation. The LCF tests were used to determine how those properties affect the behavior and performance of the superalloy under stress. It is always important to determine such attributes in order to achieve a more rounded perspective of what is being worked with, which in this case is a Ni-base superalloy. With a well-rounded view of the material, analyzing multiple facets of the subject material is not only easier to accomplish, but can also provide insights to achieve a higher level of analysis. In this chapter, the general results of each test type is discussed, as well as the analysis techniques that will be employed later. Note that since the specimens were lent to the university for research, the specific numerical results cannot be revealed as they are propriety data. Only general observations in the trends are discussed. Whereas life prediction modeling was the goal of the experiments, the auxiliary goal addressed here is analyzing mechanisms of failure.

3.2 Overview of the Tensile Test Results

In total, there fourteen samples underwent tensile testing. Seven of the samples were cut from directional solidified superalloy (DS) and seven were from single-crystal superalloy (SX). The loading conditions used for the tensile tests are provided in Table 3-1 for the DS samples and
Table 3-2 for the SX samples. Specimen number, material orientation, strain rate, and the temperature of the experiments are presented in these tables.

Table 3-1 Tensile test matrix for the DS specimens.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Tensile Temperature (°F)</th>
<th>Tensile Temperature (°C)</th>
<th>Orientation</th>
</tr>
</thead>
<tbody>
<tr>
<td>011-DS-5</td>
<td>0.01</td>
<td>1562</td>
<td>850</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>011-DS-6</td>
<td>0.01</td>
<td>1652</td>
<td>900</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>011-DS-7</td>
<td>0.01</td>
<td>1742</td>
<td>950</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>011-DS-8</td>
<td>0.01</td>
<td>1832</td>
<td>1000</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>010-DS-6</td>
<td>0.01</td>
<td>1652</td>
<td>900</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-7</td>
<td>0.01</td>
<td>1742</td>
<td>950</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-8</td>
<td>0.01</td>
<td>1832</td>
<td>1000</td>
<td>[ 0 1 0 ]</td>
</tr>
</tbody>
</table>

Table 3-2 Tensile test matrix for the SX specimens.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Tensile Temperature (°F)</th>
<th>Tensile Temperature (°C)</th>
<th>Orientation</th>
</tr>
</thead>
<tbody>
<tr>
<td>011-SX-8</td>
<td>0.001</td>
<td>1652</td>
<td>900</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>011-SX-7</td>
<td>0.001</td>
<td>1832</td>
<td>1000</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>011-SX-9</td>
<td>0.01</td>
<td>1652</td>
<td>900</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>011-SX-6</td>
<td>0.01</td>
<td>1832</td>
<td>1000</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>111-SX-5</td>
<td>0.01</td>
<td>1652</td>
<td>900</td>
<td>[ 0 1 1.45 ]</td>
</tr>
<tr>
<td>111-SX-4</td>
<td>0.01</td>
<td>1742</td>
<td>950</td>
<td>[ 0 1 1.45 ]</td>
</tr>
<tr>
<td>111-SX-3</td>
<td>0.01</td>
<td>1832</td>
<td>1000</td>
<td>[ 0 0 1.45 ]</td>
</tr>
</tbody>
</table>

The results from these tests are consistent with previously published findings on Ni-base superalloys, showing that these specimens have high yield and ultimate tensile strength.
However, when tested at the highest temperature, both the DS and SX specimens yielded at a lower stress but the trend was only observed for the specimens cut in the [001] orientation [1] [2].

![Figure 3-1: a) Variation of UTS with temperature for [001], [010], and [011] oriented DS specimens[1]. b) Variation of UTS with temperature for [001],[011],[111] oriented SX specimens [2].](image)

The strength of the superalloy appears to be greater when tested at medium temperatures, but reduces at higher temperatures (illustrated in Figure 3-1). This is consistent with previously published research shows that Ni-base superalloys exhibit increased strength at intermediate temperatures before decaying at very high temperatures [8] [10]. This affects the softening and hardening process the metal goes through before rupturing, which will in turn affect the deformation and fracture behaviors.

Each specimen was loaded until rupture. The fragments of the specimens were collected and carefully labeled so that proper analysis could be conducted later. The fractured specimens are laid out in Figure 3-2. They are grouped according to their material type (DS or SX) and orientation. Each group organizes the specimens by the temperature they were tested at. The low and high temperature test specimens are shown from left to right respectively.
While true that tensile tests are most often used for determining the mechanical properties of a substance, microstructural study has also been done on tensile tested specimens. Though previous research tends to focus only on the fracture behavior on the microscopic level as it relates to grain boundaries and phase shifts. This thesis focuses more on other fracture surface features as well as determine any trends in the behavior of fracturing as it relates to the different conditions in which the specimens were tested.

3.3 Overview of the Fatigue Test Results

Twenty-three specimens underwent fatigue testing: twelve DS samples and eleven SX samples. The test matrixes that were used for the fatigue experiments are provided in Table 3-3 for the DS
samples and Table 3-4 for the SX samples. Specimen number, strain range, strain rate, temperature of the experiments, and amplitude ratios are presented in these tables.

**Table 3-3**: LCF test matrix for the DS specimens.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Strain Range, (in/in)</th>
<th>Temperature, (°F)</th>
<th>Temperature, (°C)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Inv. Fat. Amp. Ratio, R</th>
<th>Direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>001-DS-1</td>
<td>0.012</td>
<td>1292</td>
<td>700</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-DS-2</td>
<td>0.016</td>
<td>1292</td>
<td>700</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-DS-3</td>
<td>0.016</td>
<td>932</td>
<td>500</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-DS-4</td>
<td>0.016</td>
<td>752</td>
<td>400</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-DS-5</td>
<td>0.016</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>011-DS-1</td>
<td>0.016</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>011-DS-2</td>
<td>0.014</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 1 ]</td>
</tr>
<tr>
<td>010-DS-1</td>
<td>0.014</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-2</td>
<td>0.014</td>
<td>932</td>
<td>500</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-3</td>
<td>0.012</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-4</td>
<td>0.016</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
<td>010-DS-5</td>
<td>0.014</td>
<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
</tbody>
</table>

**Table 3-4**: LCF test matrix for the SX specimens.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Strain Range, (in/in)</th>
<th>Temperature, (°F)</th>
<th>Temperature, (°C)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Inv. Fat. Amp. Ratio, R</th>
<th>Direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>001-SX-1</td>
<td>0.01</td>
<td>1832</td>
<td>1000</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-SX-2</td>
<td>0.012</td>
<td>1832</td>
<td>1000</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-SX-3</td>
<td>0.012</td>
<td>1832</td>
<td>1000</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>001-SX-4</td>
<td>0.012</td>
<td>1652</td>
<td>900</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 0 1 ]</td>
</tr>
<tr>
<td>011-SX-1</td>
<td>0.012</td>
<td>1832</td>
<td>1000</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
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<td>0.012</td>
<td>1652</td>
<td>900</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
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<td>0.012</td>
<td>1472</td>
<td>800</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
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<td>1292</td>
<td>700</td>
<td>0.001</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
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<td>752</td>
<td>400</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 1 0 ]</td>
</tr>
<tr>
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<td>0.018</td>
<td>1292</td>
<td>700</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 1 1 47]</td>
</tr>
<tr>
<td>111-SX-2</td>
<td>0.018</td>
<td>1832</td>
<td>400</td>
<td>0.01</td>
<td>-1</td>
<td>[ 0 1 1 47]</td>
</tr>
</tbody>
</table>
The material orientation affects the DS specimens more than the SX specimens, which makes sense considering the SX alloy has a more consistent microstructure. This is shown in the following graph in Figure 3-3.

![Graph comparing the number of cycles until failure to the test temperature.](image)

**Figure 3-3:** Graph comparing the number of cycles until failure to the test temperature.

It is noteworthy that the DS specimens lasted for the most life cycles when tested at intermediate high temperatures compared to the lower temperatures but did not last as long at the highest temperatures. Comparatively, the SX specimens endured more cycles consistently and at even higher temperatures. However, the SX specimens still did not withstand the fatigue for as many cycles when tested at the highest temperature. This is consistent with previous research that showed the fatigue life is higher in SX Ni-base superalloys at lower temperatures [16].

When each specimen failed, the fragments were collected and carefully labeled just like the tensile specimens. The fractured LCF specimens are laid out in Figure 3-4. They are grouped according to their material type (DS or SX) and orientation. Each group organizes the specimens by the temperature they were tested at, corresponding the low and high temperature test specimens from left to right respectively.
3.4 Fracture Analysis Methods

The samples are observed and photographed using two different imaging devices: a Dino-Lite Digital Microscope Premier AM5018MZTL microscope camera and a Keyence VHX-900F digital microscope. The fracture features in the images obtained using the Dino-Lite microscope camera and the accompanying DinoXcope imaging software (Version 1.7.3). The method by which the fracture features are measured is taken from the guidance of a published source [28].

Figure 3-4: Deformed specimens from the LCF tests.
The fracture surface is treated as a topographical map, with the peaks and valleys measured from a chosen reference plane.

Two different roughness values shall be determined for each specimen. Roughness is calculated by averaging the peak and valley values. For the sake of accuracy, two different calculations will be made. One value using the Absolute Average Roughness formula:

$$\bar{R}_A = \frac{1}{n} \sum_{i=1}^{n} |d_i|$$ (3.4.1)

and the other value using the Root Mean Square Roughness formula:

$$\bar{R}_{RMS}^2 = \frac{1}{n} \sum_{i=1}^{n} d_i^2$$ (3.4.2)

The way the fracture lengths (the peaks and valleys on the fracture surface) are measured is illustrated in the diagram in Figure 3-5, with the $d_i$ value being the deviation from the “flatness” represented by the reference plane. The reference plane is established to approximate where the end boundary of the sample was before it was deformed, which is accomplished by placing the plane on the vertical axis where it would have an equal amount of area (the total amount of material, not the vertical deviation of the peaks and valleys) both above and below it. The absolute average roughness is used to quantify the roughness value. The root mean square roughness is calculated as a comparison factor to account for the variability of the measurements (if variability is minimal, the root mean calculation is not included in tables or graphs).
Figure 3-5: Schematic depicting how to take topographical measurements on a fractured specimen.

The same procedure is applied to two different topographical images obtained from the two different imaging devices: the Dino-Lite microscope and the Keyence microscope. The Dino-Lite (shown in Figure 3-6) will be used to obtain a large-scale image of the profile of the deformed specimen, which will represent the *macrotopography* of the specimen. The “profile” of the specimen refers to the image captured from a side-view, which is illustrated in Figure 3-5.

The measurement method is applied using ImageJ [29] software with the images taken with the Dino-Lite. Since this microscope captures images on a larger scale (less magnification power), the value determined using peaks and valleys will not be termed “roughness”. More accurately, the value will describe how irregular (jagged) the shape of the resulting fracture is. This will be termed the *fracture length deviation* in this thesis.
Figure 3-6: (a) Dino-Lite microscope and (b) image taken of sample 011-DS-8, with added reference lines to show topographical measurements of vertical deviations.

The Keyence microscope (shown in Figure 3-7), which can obtain topographical images at very small scales (high magnifications), will be used to obtain values that would represent what is actually considered the “roughness” feature, since the microscale of the measurements can more accurately describe the texture of the fracture surface. The feature this measurement represents will be referred to as the *surface roughness* in this thesis.

Figure 3-7: Keyence VHX-900F digital microscope
The Keyence microscope is able to create three-dimensional topographical surface images, like the one shown in Figure 3-8, by using built-in software to render together a series of images at different levels of magnification. When each layer comes into focus, the software calculates the height differentials of each image and combines all together to construct one three-dimensional image. The Keyence software always allows the user to take measurements directly on the 3-D image, which is how the previously described procedure was carried out. The method remains consistent across the two types of images.

**Figure 3-8:** Topographical rendering of the surface of sample 011-DS-7 taken on the Keyence microscope, with added reference lines to show topographical measurements of vertical deviations.
CHAPTER 4: METALLURGICAL ANALYSIS AND RESULTS

The fractographs are presented alongside analytic reasoning. Microscopy is accompanied by macroscopy to fully describe all aspects of the quantitative analysis. Comparing the fractographs of different specimens with similar test conditions provides a visual component to the analysis of fracture behavior trends in the material. The visual comparison serves as a backdrop for the numerical and graphical data presented. The main purpose of this work is to make quantitative connections between the different factors that can affect the fracture response of the material. It is also the hope that by providing the methodology, others can use it to advance the research of any material.

Figure 4-1: Flowchart depicting the methodology of the analysis process.

4.1 Fractographic and Microstructural Analysis

Each sample was imaged with both of the previously described microscopes. Analysis was first performed on the macroscopic images. Observations of the larger features could 1) provide
insight on the fracture behavior and 2) determine which area of the surface would be ideal to look at closer with the more powerful microscope. The benefit of using the macroscopic images as a starting point for analysis is to allow some insight of what kind of deformation the samples undergo.

The main purpose of macroscopy is to take measurements of the fractured surface to determine the fracture length deviation. The numerical analysis represents how evenly, or flat, a sample breaks. The irregularity, or lack thereof, of a fracture shape gives clues as to why and how a sample fails.

For the purposes of this research, macroscopic images will be used to draw physical comparisons between different samples and quantifying them. For example, Figure 4-2 shows the sample from this set with the lowest fracture length deviation of 0 mm. It can be observed that the failure of this sample left a uniform surface. The inner material was not displaced very much during the stress failure, leaving the sample looking almost like it was purposely cut. Some initial
assumptions can be made on how this failure occurred. Since the material type is SX, it is possible that planar slip occurred so neatly that the resulting surface is mostly smooth. The material orientation [ 0 1 1 ] and its corresponding angle of the slip planes would account for why the sample has this fracture angle. It is also possible that this particular sample had an inner defect in its microstructure which allowed such a perfect fracture to propagate.

Contrastingly, the sample shown in Figure 4-3 does not appear as if it was cut. It failed in such a way that it looks as though shards are protruding through the surface. The fracture length deviation for this sample was calculated to be 2.0665 mm. The reason the fracture length deviation of this sample is so high could be because of its material type (DS) and/or its material orientation. The cause of torn looking fracture shape is likely the result of the nature of the tensile test; an inference from how predominate the necking is in this sample. However, not all tensile samples have fracture surfaces that are this jagged (all fractographs are compiled and labeled in Appendix A for convenient reference).
It is worth noting that since these two samples underwent two different kinds of loading conditions, the types of stress and fatigue they underwent would affect their fracture behavior differently. The difference in results of the two test types is discussed in the analysis section. Spotlighting these two samples in this section is not meant to compare tensile and LCF tests directly, but rather to illustrate the two ends of the fracture spectrum that resulted from the experiments.

After macroscopic analysis, the Keyence device is used for microscopic analysis. The technique for measuring and calculating the surface roughness is identical to the fracture length deviation technique, but since the magnification is significantly greater, the profile represented by the number is more accurate to the roughness of the actual surface. The magnified images simplify the identification of structural elements that affect the fracture process, and the topographical rendering makes it easy to measure the peaks and valleys of the surface.

![Figure 4-4: (a) Microscopic optical image of sample 111-SX-1. (b) Topographical rendering of surface.](image)
Reviewing sample 111-SX-1 in Figure 4-4, this time with the Keyence microscope, it is observed that the angled surface of the specimen is not completely flat. Micropores can be observed in the magnified image, which have been previously observed to be potential points of stress concentration [13]. It is possible due to the many pores in this sample that the stress concentrations were uniformly dispersed, which encouraged a clean breakage.

Despite the presence of micropores, which would technically count as a valley according to previously defined criteria, the surface roughness value for this sample is less than 1 µm. The reason for this is for the sake of consistency. Since the Keyence microscope cannot detect noticeable height changes from the surface, then the analysis treats the surface as flat even though a more sensitive microscope might be able to distinguish the smaller height changes. This allows all the data collected from all the samples to be compared against the same metric.

![Micro-pores](image1)

![Topographical rendering](image2)

**Figure 4-5:** (a) Microscopic optical image of sample 011-DS-6 and (b) topographical rendering of surface.
Sample 011-DS-6 photographed with the Keyence microscope in Figure 4-5 is a good example of a non-flat surface. Its topographical rendering shows a steep valley and a fairly tall peak (assuming the yellow level terrain is the “base”). The sample has a surface roughness value of 576.075 µm, which is the highest roughness value of the tensile tests. The microscopic optical image shows that the interior of this specimen has noticeably less micropores than sample 111-SX-1, which could explain why the failure was so much less clean.

![Sample images](image)

**Figure 4-6**: Comparison of DS samples all LCF tested at 1292°F with a strain range of 0.016 in/in. Macroscopic fractographs make it easy to compare fractured samples side by side. In Figure 4-6, three LCF tested samples are shown next to each other to compare how the material orientation affects the fracture behavior. The material orientations are different, but all of the specimens are made of the DS metal and tested with the same conditions. Even though the test conditions were the same, the resulting fracture surfaces look different. The [001] oriented sample appears the most flat compared to the rest, as reflected by its fracture length deviation value of 0.538 mm. The fracture length deviation values for the [010] and [011] oriented specimens are 0.7398 mm and 2.0345 mm respectively. These values show a 37% increase in the variation of the fracture
surfaces between the [001] and [010] samples, and then a 278% increase between the [001] and [011] samples.

The effect grain boundaries has on fracture behavior is well illustrated in Figure 4-6. The [001] specimen has its crystal grains running parallel to the line of tensile stress, the [010] specimen has its crystal grains running perpendicular to the line of tensile stress, and the [011] specimen has its crystal grains running 45° to the line of tensile stress.

The fracture of the [010] specimen is predictable considering the grain are aligned in a stacked way that would make it easy for tensile stress to pull apart the layers, a concept that is proven since there is a visible second crack initiation below where the specimen has already failed. The fracture of the [011] specimen is interesting because the angle of the jagged sharp edges are in the neighborhood of a 45° angle. The [001] specimen is less predictable based on grain boundaries since the tensile stress would pull on all them more or less evenly. It would be more accurate to ascribe a different factor to why the [001] specimen failed the way it did (compared to the effect the grains had on the other two specimens).
Another fractographic comparison is illustrated in Figure 4-7. Four samples, two of DS material and two of SX material, have the same material orientations and were tested at the same strain rates. This shows the fracture behavior differences between DS and SX samples and the effect temperature has on the fracture shape. The DS material appears to be more rugged and uneven than the SX, and the small increase in temperature does not appear to have too great an impact. The corresponding fracture length deviation measurements are as follows. For the DS samples, the values are 1.084 mm and 1.7893 mm from left to right respectively. For the SX samples, the values are 0.7568 mm and 1.4952 mm from left to right respectively. Therefore, there is about 30% less variation in the fracture surfaces between the DS and SX samples tested at 1742°F and 16% less variation in the ones tested at 1832°F. This trend is consistent with the initial prediction.
4.2 Data Analysis

After taking measurements from all the samples, the data was compiled into tables and analyzed.

The tables are included here so the reader can reference the raw data if desired.

Table 4-1: Tensile experiments: test conditions and fracture data values.

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
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<td>0.01</td>
<td>0.7570</td>
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<td></td>
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<td>0.3555</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>1832</td>
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<td>1.5035</td>
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</tr>
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<td>1652</td>
<td>0.01</td>
<td>1.0633</td>
<td>186.0750</td>
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</table>

Figure 4-8: Tensile experiments results: fracture length deviation vs. temperature.
The graph of the data in Figure 4-8 is separated by material type and orientation and plots the calculated fracture length deviation values versus the testing temperature. This highlights some apparent patterns in the fracture behavior of the specimens. The DS [011] samples have the highest fracture length deviations of any of the other type sample types, with no obvious relationship between the fracture behavior and the temperature. The DS [010] samples had significantly lower deviations than the DS [011] samples, but also had no evident relationship with temperature. However, the SX [001] samples clearly show an increase in their deviation values as the temperature increases. The SX [011] samples have more deviation in their surfaces than the SX [001] samples. It is unclear if there is a direct relationship between fracture length deviation and temperature in the case of the SX [011] samples, however, the observed trend in the available data appears to show an increase in their deviation values as the temperature increases.

Figure 4-9: Tensile experiments results: surface roughness vs. temperature.
The graph of the data in Figure 4-9 is separated by material type and orientation and plots the calculated surface roughness values versus the testing temperature. Much like the fracture length deviation graph, this graph shows that the DS [011] samples have some of the highest values with no clear relationship between the roughness and the temperature. Unlike the fracture length deviation values, the surface roughness value trends of the DS [010], SX [001], and SX [011] samples showed an obvious increase as the test temperature was increased. Consistent with the deviation values, the surface roughness values of the SX [011] samples are greater than the values of the SX [001] samples.

Table 4-2 LCF experiments: test conditions and fracture data values.

<table>
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<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Range (in/in)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
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<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>932 0.016 0.01</td>
<td>1.2478</td>
<td>123.0500</td>
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<tr>
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<td>1832 0.018 0.01</td>
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<td>~~~</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
In Table 4-2, the three samples without calculated roughness values did not rupture during the tests. Since they did not fracture, they were not included in the analysis nor were they taken into account when considering possible trends in the data.

![Graph](image.png)

**Figure 4-10:** LCF experiments results: fracture length deviation vs. temperature vs. strain range.

The graph in Figure 4-10 separates the test samples into groups by material type and orientation and plots the calculated fracture length deviation values versus the testing temperature and the strain range. The purpose of using a three-dimensional graph is so that both contributing factors of the LCF tests can be viewed simultaneously.

At greater strain ranges, the DS specimens showed an increase shift in their fracture length deviation values. The DS specimens tested at higher temperatures tended to have lower deviation values, but it is worth noting that the specimens that were tested at lower temperature also had greater strain ranges. In fact, the of the DS specimens that were tested at the same temperature,
the ones with greater strain ranges appeared to have greater deviation values. It can be assumed that in LCF tests, strain range is the more important factor when dealing with DS material.

There are less discernable patterns in the fracture behavior of the LCF tested SX specimens since their data is spread quite uniformly. However, it does seem that SX [001] specimens tend to yield smaller fracture length deviation values when tested at higher temperatures. The plot also reveals that the majority of SX specimens have smaller deviation values than the DS specimens.

Material orientation seems to be a weaker factor for the LCF tests than it was for the tensile tests.

![Figure 4-11: LCF experiments results: surface roughness vs. temperature vs. strain range.](image)

The graph in Figure 4-11 separates the test samples into groups by material type and orientation and plots the calculated surface roughness values versus the testing temperature and the strain range. Similar to the pattern observed in the fracture length deviations, DS samples tend to have
higher surface roughness values at greater strain ranges. Interestingly, DS [001] specimens tend to yield larger surface roughness values when tested at higher temperatures, but DS [010] specimens tend to yield smaller surface roughness values.

Similar to the trend in the fracture length deviation data, SX [011] specimens yield larger surface roughness values than the SX [001] specimens. Also, it can be noted that the majority of SX specimens tested at high temperatures along with a low strain range had small surface roughness values. Additionally, the surfaces of the SX specimens tend to be smoother than the DS specimens.

Material type appears to be a strong factor of fracture behavior. The DS specimens tend to yield higher deviation and roughness values. On average, the fracture length deviation of SX specimens was about 43% less than DS specimens from the tensile tests and about 52% less for the ones from the LCF tests. For surface roughness, the average for the SX specimens was about 33% less than the DS specimens from the tensile tests and about 20% less for the ones from the LCF tests.

For convenience, the qualitative analysis for each sample has been listed in table format so that the features and test conditions for each sample can be easily reference. The tables also list the page number where each samples’ collection of photographs can be found in the appendix. The tables are separated by test type (tensile or LCF) and material type (DS or SX).
Table 4-3 Qualitative analysis and descriptions of visual features for the tensile tested DS samples.

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<tr>
<th>Orientation</th>
<th>Specimen ID</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
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<td>[0 1 0]</td>
<td>010-DS-6</td>
<td>1652</td>
<td>0.01</td>
<td>ductile fracture; crescent-shape cup necking, implies crack initiated at center and propagated outward; visible chevron on edge of surface confirms propagation direction; possible crystallographic crack on profile surface; large micropores</td>
<td>54</td>
</tr>
<tr>
<td></td>
<td>010-DS-7</td>
<td>1742</td>
<td>0.01</td>
<td>ductile fracture; mostly uniform fracture surface, prominent shear lip protrusion; cleavages in grain direction; multiple small cracks on profile surface</td>
<td>56</td>
</tr>
<tr>
<td></td>
<td>010-DS-8</td>
<td>1832</td>
<td>0.01</td>
<td>necking visible, but some brittle fracture evident; 40° ridge on fracture surface; cleavages in grain direction; large micropores</td>
<td>58</td>
</tr>
<tr>
<td>[0 1 1]</td>
<td>011-DS-5</td>
<td>1562</td>
<td>0.01</td>
<td>ductile fracture; cup-shape necking, implies crack initiated at center and propagated outward; shear lips around edge of fracture surface; multiple cracks in Y configuration confirm propagation direction; micropores visible</td>
<td>60</td>
</tr>
<tr>
<td></td>
<td>011-DS-6</td>
<td>1652</td>
<td>0.01</td>
<td>ductile fracture; cup-shape necking; 60° prominent shear lip; small, sharp protrusions in fracture surface</td>
<td>62</td>
</tr>
<tr>
<td></td>
<td>011-DS-7</td>
<td>1742</td>
<td>0.01</td>
<td>ductile fracture; cup-shape necking; 60° prominent shear lip and 50° smaller one; large micropores</td>
<td>64</td>
</tr>
<tr>
<td></td>
<td>011-DS-8</td>
<td>1832</td>
<td>0.01</td>
<td>ductile fracture; cup-shape necking; 55° prominent shear lip; large micropores</td>
<td>66</td>
</tr>
</tbody>
</table>
Table 4-4 Qualitative analysis and descriptions of visual features for the tensile tested SX samples.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Specimen ID</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>[0 0 1]</td>
<td>011-SX-6</td>
<td>1832</td>
<td>0.01</td>
<td>necking visible, but some brittle fracture evident; 30° ridge that rises to 90° on fracture edge; cleavages in grain direction; many large micropores</td>
<td>68</td>
</tr>
<tr>
<td>[0 0 1]</td>
<td>011-SX-7</td>
<td>1832</td>
<td>0.001</td>
<td>ductile fracture with prominent necking; 40° shear lip; many large micropores</td>
<td>70</td>
</tr>
<tr>
<td>[0 0 1]</td>
<td>011-SX-8</td>
<td>1652</td>
<td>0.001</td>
<td>ductile fracture; uniform fracture surface with 15° offset; small secondary cracks visible on profile surface; large micropores</td>
<td>72</td>
</tr>
<tr>
<td>[0 0 1]</td>
<td>011-SX-9</td>
<td>1652</td>
<td>0.01</td>
<td>ductile fracture; mostly uniform surface; secondary crack branching in T configuration visible on profile surface; large micropores</td>
<td>74</td>
</tr>
<tr>
<td>[0 1 1]</td>
<td>111-SX-3</td>
<td>1832</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is offset by 30°; prominent shear lip that connects to ridge-like protrusion; large micropores</td>
<td>76</td>
</tr>
<tr>
<td>[0 1 1]</td>
<td>111-SX-4</td>
<td>1742</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is offset by 35°; large micropores evenly distributed</td>
<td>78</td>
</tr>
<tr>
<td>[0 1 1]</td>
<td>111-SX-5</td>
<td>1652</td>
<td>0.01</td>
<td>ductile fracture; 50° ridge on fracture surface; ratchet marks and micropores visible</td>
<td>80</td>
</tr>
</tbody>
</table>
Table 4-5 Qualitative analysis and descriptions of visual features for the LCF tested DS samples.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Specimen ID</th>
<th>Temperature (°F)</th>
<th>Strain Range (in/in)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>[0 0 1]</td>
<td>001-D5-1</td>
<td>1292</td>
<td>0.012</td>
<td>0.01</td>
<td>Specimen did not fracture</td>
<td>~~~</td>
</tr>
<tr>
<td></td>
<td>001-D5-2</td>
<td>1292</td>
<td>0.016</td>
<td>0.01</td>
<td>ductile fracture; lower plane offset by 35° and higher plane by 60° on fracture surface; visible ridge on fracture surface</td>
<td>82</td>
</tr>
<tr>
<td></td>
<td>001-D5-3</td>
<td>932</td>
<td>0.016</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is offset by 30°; ratchet marks visible</td>
<td>84</td>
</tr>
<tr>
<td></td>
<td>001-D5-4</td>
<td>752</td>
<td>0.016</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is half offset by 15° and half by 45°; ratchet marks visible; micropores visible</td>
<td>86</td>
</tr>
<tr>
<td></td>
<td>001-D5-5</td>
<td>1292</td>
<td>0.016</td>
<td>0.001</td>
<td>ductile fracture; mostly uniform fracture surface with 25° offset; visible shear lip; micropores visible</td>
<td>88</td>
</tr>
<tr>
<td>[0 1 0]</td>
<td>010-D5-1</td>
<td>1292</td>
<td>0.014</td>
<td>0.001</td>
<td>ductile fracture with prominent necking; visible ridges, ratchet marks, and micropores</td>
<td>90</td>
</tr>
<tr>
<td></td>
<td>010-D5-2</td>
<td>932</td>
<td>0.014</td>
<td>0.001</td>
<td>ductile fracture; two separate 50° ridges on fracture surface; noticeable cracks branching from the middle</td>
<td>92</td>
</tr>
<tr>
<td></td>
<td>010-D5-3</td>
<td>1292</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; crescent-shape cup necking, implies crack initiated at center and propagated outward; small ridges and beach marks are visible</td>
<td>94</td>
</tr>
<tr>
<td></td>
<td>010-D5-4</td>
<td>1292</td>
<td>0.016</td>
<td>0.001</td>
<td>ductile fracture; cup necking, implies crack initiated at center and propagated outward; multiple shear lips are visible; ridges and micropores are visible on fracture surface; possible crystallographic crack on profile surface</td>
<td>96</td>
</tr>
<tr>
<td></td>
<td>010-D5-5</td>
<td>1292</td>
<td>0.014</td>
<td>0.001</td>
<td>Specimen did not fracture</td>
<td>~~~</td>
</tr>
<tr>
<td>[0 1 1]</td>
<td>011-D5-1</td>
<td>1292</td>
<td>0.016</td>
<td>0.001</td>
<td>brittle fracture; three sharp 60° protrusions from fracture surface; micropores visible</td>
<td>98</td>
</tr>
<tr>
<td></td>
<td>011-D5-2</td>
<td>1292</td>
<td>0.014</td>
<td>0.001</td>
<td>ductile fracture; 30° shear lip; sharp 40° protrusion from fracture surface; micropores visible</td>
<td>100</td>
</tr>
</tbody>
</table>
Table 4-6 Qualitative analysis and descriptions of visual features for the LCF tested SX samples.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Specimen ID</th>
<th>Temperature (°F)</th>
<th>Strain Range (in/in)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>001-SX-1</td>
<td>1832</td>
<td>0.01</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is offset by 45°; visible chevrons on fracture surface suggest initiation started at edge and propagated inward; micropores visible; visible ratchet marks on profile surface</td>
<td>102</td>
</tr>
<tr>
<td></td>
<td>001-SX-2</td>
<td>1832</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; mostly uniform fracture surface; visible chevrons on fracture surface suggest initiation started at edge and propagated inward; visible ratchet marks on profile surface</td>
<td>104</td>
</tr>
<tr>
<td></td>
<td>001-SX-3</td>
<td>1832</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; fracture surface is offset by 20°; large ratchet marks on profile surface; few micropores visible</td>
<td>106</td>
</tr>
<tr>
<td>[ 0 0 1 ]</td>
<td>001-SX-4</td>
<td>1652</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; fracture surface is offset by 20°; small ratchet marks on profile surface; few ridges visible on fracture surface</td>
<td>108</td>
</tr>
<tr>
<td></td>
<td>011-SX-1</td>
<td>1832</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; mostly uniform fracture surface; small ridges on fracture surface; visible ratchet marks on profile surface</td>
<td>110</td>
</tr>
<tr>
<td></td>
<td>011-SX-2</td>
<td>1652</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; mostly uniform fracture surface; small shear lips on edges; visible ridges on fracture surface; micropores visible</td>
<td>112</td>
</tr>
<tr>
<td></td>
<td>011-SX-3</td>
<td>1472</td>
<td>0.012</td>
<td>0.001</td>
<td>ductile fracture; very uniform fracture surface</td>
<td>114</td>
</tr>
<tr>
<td></td>
<td>011-SX-4</td>
<td>1292</td>
<td>0.018</td>
<td>0.001</td>
<td>ductile fracture; fracture surface is offset by 50°; visible ridges on fracture surface; micropores visible</td>
<td>116</td>
</tr>
<tr>
<td></td>
<td>011-SX-5</td>
<td>752</td>
<td>0.018</td>
<td>0.01</td>
<td>ductile fracture; fracture surface is offset by 35°; visible ridges on fracture surface; few micropores visible</td>
<td>118</td>
</tr>
<tr>
<td>[ 0 1 1 ]</td>
<td>111-SX-1</td>
<td>1292</td>
<td>0.018</td>
<td>0.01</td>
<td>ductile fracture; very uniform fracture surface offset by 45°; micropores visible</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>111-SX-2</td>
<td>1832</td>
<td>0.018</td>
<td>0.01</td>
<td>Specimen did not fracture</td>
<td>~~~</td>
</tr>
</tbody>
</table>
4.3 Evaluation and Discussion

The main purpose of this analysis was to determine the microstructural strengths and weaknesses of the Ni-base superalloy. The data obtained accomplished that and provided some valuable insight into the fracture behavior of such metals. Though it should be acknowledged that this insight is based on a relatively small sample size. For future research, larger sets of experimental data would be needed to make more accurate evaluations of the trends that the fracture behavior follows. More samples should be included with a test matrix that ensures that each type of sample is tested at all the different temperatures and other test conditions. The variety of specimens must have corresponding test conditions to obtain more direct comparisons and establish patterns.
CHAPTER 5: CONCLUSION

A quantitative method of analyzing the fracture features of deformed metal specimens was presented in this thesis. The main benefit of the presented method is the fact that sectioning the desired specimen is not required for analysis, which makes the process simpler and more convenient. This methodology was applied to specimens made from a Nickel-base superalloy that were experimented with various loading conditions. However, the method is valid for any fractured material. The subject material of this work is a variation of the IN792 superalloy, provided a turbomachinery company. The specimens of this material underwent either tensile testing or low cycle fatigue testing. After the rounds of testing, two different microscopes were used to obtain both macro and microscale images of the specimens. Quantitative analysis was carried out after the photography was completed to determine two different roughness values for each specimen. These two values were termed the fracture length deviation and the surface roughness. The distinction between the two is necessary to provide a wider perspective of how the material is deformed after failure. The values were used to establish the strength of influence certain factors had on fracture behavior trends. The most notable of these factors is the material type; the SX specimens tended to yield smoother fracture surfaces. A comprehensive description of the qualitative features observed across all the fractured specimens is also provided, as is the complete macroscopy and microscopy images of all the fractured specimens.

In future work, the macroscopy and/or microscopy could benefit from being conducted at the time of testing. A microscope set up to record the crack growth like in previous research [19] would allow an even more consistent way to determine crack initiation and propagation. Furthermore, the development and use of a crack propagation mathematical model [30] could
provide interesting insights into the fracture behavior, especially if the model could be used to predict the resulting roughness of specimens [23] based on initial conditions.
APPENDIX A:
MICROSCOPY IMAGES
A.1 Images of Fractured Samples from the Tensile Tests

Sample ID: 010-DS-6

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[ 0  1  0 ]</td>
<td>1652</td>
<td>0.01</td>
<td>0.7570</td>
<td>90.5867</td>
</tr>
</tbody>
</table>

Figure A-1: 010-DS-6 imaged with the Dino-Lite microscope (surface and profile views respectively).

Figure A-2: Optical image of 010-DS-6 taken with the Keyence microscope.
Figure A-3: Microscopic optical image of 010-DS-6 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-4: Topographical rendering of the surface of 010-DS-6 made with the Keyence software.
Sample ID: 010-DS-7

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[ 0 1 0 ]</td>
<td>1742</td>
<td>0.01</td>
<td>0.3555</td>
<td>136.2283</td>
</tr>
</tbody>
</table>

**Figure A-5:** 010-DS-7 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-6:** Optical image of 010-DS-7 taken with the Keyence microscope.
Figure A-7: Microscopic optical image of 010-DS-7 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-8: Topographical rendering of the surface of 010-DS-7 made with the Keyence software.
Sample ID: 010-DS-8

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[ 0 1 0 ]</td>
<td>1832</td>
<td>0.01</td>
<td>0.7640</td>
<td>286.4833</td>
</tr>
</tbody>
</table>

**Figure A-9**: 010-DS-8 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-10**: Optical image of 010-DS-8 taken with the Keyence microscope.
Figure A-11: Microscopic optical image of 010-DS-8 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-12: Topographical rendering of the surface of 010-DS-8 made with the Keyence software.
Sample ID: 011-DS-5

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[0 1 1]</td>
<td>1562</td>
<td>0.01</td>
<td>1.5035</td>
<td>305.4067</td>
</tr>
</tbody>
</table>

**Figure A-13:** 011-DS-5 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-14:** Optical image of 011-DS-5 taken with the Keyence microscope.
Figure A-15: Microscopic optical image of 011-DS-5 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-16: Topographical rendering of the surface of 011-DS-5 made with the Keyence software.
Sample ID: 011-DS-6

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[0 1 1]</td>
<td>1652</td>
<td>0.01</td>
<td>2.0665</td>
<td>576.0750</td>
</tr>
</tbody>
</table>

**Figure A-17:** 011-DS-6 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-18:** Optical image of 011-DS-6 taken with the Keyence microscope.
Figure A-19: Microscopic optical image of 011-DS-6 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-20: Topographical rendering of the surface of 011-DS-6 made with the Keyence software.
Sample ID: 011-DS-7

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
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<td>1742</td>
<td>0.01</td>
<td>0.8638</td>
<td>171.4250</td>
</tr>
</tbody>
</table>

**Figure A-21:** 011-DS-7 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-22:** Optical image of 011-DS-7 taken with the Keyence microscope.
Figure A-23: Microscopic optical image of 011-DS-7 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-24: Topographical rendering of the surface of 011-DS-7 made with the Keyence software.
Sample ID: 011-DS-8

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS</td>
<td>[0 1 1]</td>
<td>1832</td>
<td>0.01</td>
<td>1.7893</td>
<td>519.8183</td>
</tr>
</tbody>
</table>

**Figure A-25**: 011-DS-8 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-26**: Optical image of 011-DS-8 taken with the Keyence microscope.
Figure A-27: Microscopic optical image of 011-DS-8 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-28: Topographical rendering of the surface of 011-DS-8 made with the Keyence software.
Sample ID: 011-SX-6

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SX</td>
<td>[ 0  0  1 ]</td>
<td>1832</td>
<td>0.01</td>
<td>0.4212</td>
<td>202.1500</td>
</tr>
</tbody>
</table>

**Figure A-29:** 011-SX-6 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-30:** Optical image of 011-SX-6 taken with the Keyence microscope.
Figure A-31: Microscopic optical image of 011-SX-6 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-32: Topographical rendering of the surface of 011-SX-6 made with the Keyence software.
Sample ID: 011-SX-7

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SX</td>
<td>[ 0  0  1 ]</td>
<td>1832</td>
<td>0.001</td>
<td>0.4670</td>
<td>208.3317</td>
</tr>
</tbody>
</table>

**Figure A-33:** 011-SX-7 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-34:** Optical image of 011-SX-7 taken with the Keyence microscope.
Figure A-35: Microscopic optical image of 011-SX-7 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-36: Topographical rendering of the surface of 011-SX-7 made with the Keyence software.
Sample ID: 011-SX-8

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SX</td>
<td>[0 0 1]</td>
<td>1652</td>
<td>0.001</td>
<td>0.2705</td>
<td>146.4783</td>
</tr>
</tbody>
</table>

**Figure A-37:** 011-SX-8 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-38:** Optical image of 011-SX-8 taken with the Keyence microscope.
Figure A-39: Microscopic optical image of 011-SX-8 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-40: Topographical rendering of the surface of 011-SX-8 made with the Keyence software.
Sample ID: 011-SX-9

Material Type, Test Conditions, and Results:

<table>
<thead>
<tr>
<th>Metal Type</th>
<th>Orientation</th>
<th>Temperature (°F)</th>
<th>Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SX</td>
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<td>1652</td>
<td>0.01</td>
<td>0.2165</td>
<td>112.5183</td>
</tr>
</tbody>
</table>

Figure A-41: 011-SX-9 imaged with the Dino-Lite microscope (surface and profile views respectively).

Figure A-42: Optical image of 011-SX-9 taken with the Keyence microscope.
Figure A-43: Microscopic optical image of 011-SX-9 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-44: Topographical rendering of the surface of 011-SX-9 made with the Keyence software.
Sample ID: 111-SX-3

Material Type, Test Conditions, and Results:

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**Figure A-45:** 111-SX-3 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-46:** Optical image of 111-SX-3 taken with the Keyence microscope.
Figure A-47: Microscopic optical image of 111-SX-3 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-48: Topographical rendering of the surface of 111-SX-3 made with the Keyence software.
Sample ID: 111-SX-4

Material Type, Test Conditions, and Results:

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**Figure A-49:** 111-SX-4 imaged with the Dino-Lite microscope (surface and profile views respectively).

**Figure A-50:** Optical image of 111-SX-4 taken with the Keyence microscope.
Figure A-51: Microscopic optical image of 111-SX-4 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-52: Topographical rendering of the surface of 111-SX-4 made with the Keyence software.
Sample ID: 111-SX-5

Material Type, Test Conditions, and Results:

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Figure A-53: 111-SX-5 imaged with the Dino-Lite microscope (surface and profile views respectively).

Figure A-54: Optical image of 111-SX-5 taken with the Keyence microscope.
Figure A-55: Microscopic optical image of 111-SX-5 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-56: Topographical rendering of the surface of 111-SX-5 made with the Keyence software.
A.2 Images of Fractured Samples from the LCF Tests

Sample ID: 001-DS-2

Material Type, Test Conditions, and Results:

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**Figure A-57:** Sample 001-DS-2 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-58:** Optical image of 001-DS-2 taken with the Keyence microscope.
**Figure A-59:** Microscopic optical image of 001-DS-2 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

**Figure A-60:** Topographical rendering of the surface of 001-DS-2 made with the Keyence software.
Sample ID: 001-DS-3

Material Type, Test Conditions, and Results:

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**Figure A-61:** Sample 001-DS-3 imaged with Dino-Lite microscope (surface and profile views respectively).

![Surface and Profile Views](image)

**Figure A-62:** Optical image of 001-DS-3 taken with the Keyence microscope.

![Optical Image](image)
Figure A-63: Microscopic optical image of 001-DS-3 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-64: Topographical rendering of the surface of 001-DS-3 made with the Keyence software.
Sample ID: 001-DS-4

Material Type, Test Conditions, and Results:

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**Figure A-65:** Profile view of sample 001-DS-4 imaged with Dino-Lite microscope.

**Figure A-66:** Optical image of 001-DS-4 taken with the Keyence microscope.
Figure A-67: Microscopic optical image of 001-DS-4 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-68: Topographical rendering of the surface of 001-DS-4 made with the Keyence software.
Sample ID: 001-DS-5

Material Type, Test Conditions, and Results:

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Figure A-69: Sample 001-DS-5 imaged with Dino-Lite microscope (surface and profile views respectively).

Figure A-70: Optical image of 001-DS-5 taken with the Keyence microscope.
Figure A-71: Microscopic optical image of 001-DS-5 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-72: Topographical rendering of the surface of 001-DS-5 made with the Keyence software.
Sample ID: 010-DS-1

Material Type, Test Conditions, and Results:

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**Figure A-73:** Profile view of sample 010-DS-1 imaged with Dino-Lite microscope.

**Figure A-74:** Optical image of 010-DS-1 taken with the Keyence microscope.
Figure A-75: Microscopic optical image of 010-DS-1 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-76: Topographical rendering of the surface of 010-DS-1 made with the Keyence software.
Sample ID: 010-DS-2

Material Type, Test Conditions, and Results:

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**Figure A-77:** Profile view of sample 010-DS-2 imaged with Dino-Lite microscope.

**Figure A-78:** Optical image of 010-DS-2 taken with the Keyence microscope.
Figure A-79: Microscopic optical image of 010-DS-2 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-80: Topographical rendering of the surface of 010-DS-2 made with the Keyence software.
Sample ID: 010-DS-3

Material Type, Test Conditions, and Results:

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**Figure A-81:** Profile view of sample 010-DS-3 imaged with Dino-Lite microscope.

**Figure A-82:** Optical image of 010-DS-3 taken with the Keyence microscope.
Figure A-83: Microscopic optical image of 010-DS-3 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-84: Topographical rendering of the surface of 010-DS-3 made with the Keyence software.
Sample ID: 010-DS-4

Material Type, Test Conditions, and Results:

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**Figure A-85:** Profile view of sample 010-DS-4 imaged with Dino-Lite microscope.

**Figure A-86:** Optical image of 010-DS-4 taken with the Keyence microscope.
Figure A-87: Microscopic optical image of 010-DS-4 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-88: Topographical rendering of the surface of 010-DS-4 made with the Keyence software.
Sample ID: 011-DS-1

Material Type, Test Conditions, and Results:

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**Figure A-89:** Sample 011-DS-1 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-90:** Optical image of 011-DS-1 taken with the Keyence microscope.
**Figure A-91:** Microscopic optical image of 011-DS-1 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

**Figure A-92:** Topographical rendering of the surface of 011-DS-1 made with the Keyence software.
Sample ID: 011-DS-2

Material Type, Test Conditions, and Results:

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**Figure A-93:** Profile view of sample 011-DS-2 imaged with Dino-Lite microscope.

**Figure A-94:** Optical image of 011-DS-2 taken with the Keyence microscope.
Figure A-95: Microscopic optical image of 011-DS-2 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-96: Topographical rendering of the surface of 011-DS-2 made with the Keyence software.
Sample ID: 001-SX-1

Material Type, Test Conditions, and Results:

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**Figure A-97**: Sample 001-SX-1 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-98**: Optical image of 001-SX-1 taken with the Keyence microscope.
Figure A-99: Microscopic optical image of 001-SX-1 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-100: Topographical rendering of the surface of 001-SX-1 made with the Keyence software.
Sample ID: 001-SX-2

Material Type, Test Conditions, and Results:

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**Figure A-101:** Sample 001-SX-2 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-102:** Optical image of 001-SX-2 taken with the Keyence microscope.
Figure A-103: Microscopic optical image of 001-SX-2 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-104: Topographical rendering of the surface of 001-SX-2 made with the Keyence software.
Sample ID: 001-SX-3

Material Type, Test Conditions, and Results:

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**Figure A-105:** Sample 001-SX-3 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-106:** Optical image of 001-SX-3 taken with the Keyence microscope.
Figure A-107: Microscopic optical image of 001-SX-3 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-108: Topographical rendering of the surface of 001-SX-3 made with the Keyence software.
Sample ID: 001-SX-4

Material Type, Test Conditions, and Results:

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**Figure A-109:** Sample 001-SX-4 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-110:** Optical image of 001-SX-4 taken with the Keyence microscope.
Figure A-111: Microscopic optical image of 001-SX-4 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-112: Topographical rendering of the surface of 001-SX-4 made with the Keyence software.
Sample ID: 011-SX-1

Material Type, Test Conditions, and Results:

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Figure A-113: Sample 011-SX-1 imaged with Dino-Lite microscope (surface and profile views respectively).

Figure A-114: Optical image of 011-SX-1 taken with the Keyence microscope.
Figure A-115: Microscopic optical image of 011-SX-1 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-116: Topographical rendering of the surface of 011-SX-1 made with the Keyence software.
Sample ID: 011-SX-2

Material Type, Test Conditions, and Results:

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**Figure A-117**: Sample 011-SX-2 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-118**: Optical image of 011-SX-2 taken with the Keyence microscope.
Figure A-119: Microscopic optical image of 011-SX-2 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-120: Topographical rendering of the surface of 011-SX-2 made with the Keyence software.
Sample ID: 011-SX-3

Material Type, Test Conditions, and Results:

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**Figure A-121:** Sample 011-SX-3 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-122:** Optical image of 011-SX-3 taken with the Keyence microscope.
Figure A-123: Microscopic optical image of 011-SX-3 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-124: Topographical rendering of the surface of 011-SX-3 made with the Keyence software.
Sample ID: 011-SX-4

Material Type, Test Conditions, and Results:

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**Figure A-125:** Sample 011-SX-4 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-126:** Optical image of 011-SX-4 taken with the Keyence microscope.
Figure A-127: Microscopic optical image of 011-SX-4 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-128: Topographical rendering of the surface of 011-SX-4 made with the Keyence software.
Sample ID: 011-SX-5

Material Type, Test Conditions, and Results:

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Figure A-129: Sample 011-SX-5 imaged with Dino-Lite microscope (surface and profile views respectively).

Figure A-130: Optical image of 011-SX-5 taken with the Keyence microscope.
Figure A-131: Microscopic optical image of 011-SX-5 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-132: Topographical rendering of the surface of 011-SX-5 made with the Keyence software.
Sample ID: 111-SX-1

Material Type, Test Conditions, and Results:

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<th>Strain Range (in/in)</th>
<th>Isothermal Strain Rate (1/s)</th>
<th>Fracture Length Deviation (mm)</th>
<th>Surface Roughness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SX</td>
<td>[ 0 1 1 ]</td>
<td>1292</td>
<td>0.018</td>
<td>0.01</td>
<td>0.0000</td>
<td>327.7733</td>
</tr>
</tbody>
</table>

**Figure A-133:** Sample 111-SX-1 imaged with Dino-Lite microscope (surface and profile views respectively).

**Figure A-134:** Optical image of 111-SX-1 taken with the Keyence microscope.
Figure A-135: Microscopic optical image of 111-SX-1 taken with Keyence microscope. The second image shows the microscopic image with a topographical color scheme.

Figure A-136: Topographical rendering of the surface of 111-SX-1 made with the Keyence software.
REFERENCES


