To the Graduate Council:

I am submitting herewith a dissertation written by Michael Lee Benson entitled “Strain-Induced Phase Transformation and Anisotropic Lattice-Strain Development in a Cobalt-Based Superalloy.” I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Materials Science and Engineering.

__________________________
Peter K. Liaw, Major Professor

We have read this dissertation and recommend its acceptance:

Hahn Choo

John D. Landes

Joseph E. Spruiell

Accepted for the Council:

Carolyn R. Hodges
Vice Provost and Dean of the Graduate Council

(Original signatures are on file with official student records.)
ACKNOWLEDGEMENTS

The completion of this work would not be possible without the support of numerous people. I would like to thank my primary advisors, Profs. Peter Liaw and Hahn Choo, for constantly challenging me to grow in my professional life. Sincere thanks go out to Prof. Walter Riemers at Technical University Berlin for his kind help with the peak-profile analysis. Also, a large portion of this dissertation would not have been possible without the labor of Dr. Alexandru Stoica, with whom it has been a privilege to work. I would like to thank the members of Cornerstone Church of Knoxville for constantly challenging me to grow spiritually and as a person. I cannot fail to mention my parents, Kurt and Sandra Benson, and my sister, Andrea Linville, whose unwavering care and support are of more value than precious metals. Most of all, I wish to acknowledge a sovereign and kind God who has watched over my life.
ABSTRACT

This dissertation addresses two issues concerning the deformation behavior of a cobalt-based superalloy: 1) the strain-induced face-centered-cubic to hexagonal-close-packed phase transformation and 2) the anisotropic lattice-strain development. Cobalt-based alloys are known to exhibit a metastable face-centered-cubic crystal structure at room temperature. This structure can transform to the thermodynamically-stable hexagonal-close-packed phase upon application of a load. While this phenomenon has been studied and documented before, this work offers a more in-depth study on this issue, as it addresses the phase transformation under several loading modes: monotonic tension, monotonic compression, stress-controlled high-cycle fatigue, and strain-controlled low-cycle fatigue. The experimental technique utilized in this research is neutron diffraction. This nondestructive method is particularly well suited to the problem because it can be used to study the transformation with the mechanical load applied in-situ.

Anisotropic lattice strain development is a natural problem to study, as neutron diffraction is also well suited for this measurement. Individual crystallites in a polycrystalline matrix by nature have directionally-dependent responses to the applied stress. When neighboring anisotropic grains interact, inhomogeneities known as intergranular strains develop. This dissertation assumes that the total lattice strain is the sum of two contributions: the linear-elastic contribution and the plasticity-induced intergranular contribution. Within the context of this analysis, the effect of the phase transformation on the deformation behavior is also inferred.
TABLE OF CONTENTS

Chapter 1: Introduction ................................................................................................... 1

1.1. Cobalt-Based Superalloys .................................................................................... 1

1.2. Strain-Induced Phase Transformation ................................................................. 4

1.3. Neutron Diffraction as a Characterization Tool .................................................... 8

1.4. Lattice-Strain Development and Anisotropy ....................................................... 10

1.5. Motivation and Outline of the Research ............................................................. 15

Chapter 2: Literature Review ......................................................................................... 19

2.1. Aspects of the Phase Transformation ................................................................... 19

2.2. Anisotropic Lattice-Strain Development in Cubic Materials ............................... 26

Chapter 3: Phase Transformation under Different Loading Conditions ..................... 35

3.1. Introduction .......................................................................................................... 35

3.2. Experimental Procedure ...................................................................................... 35

3.3. Results .................................................................................................................. 46

3.4. Discussion ............................................................................................................ 64

3.5. Summary .............................................................................................................. 78

Chapter 4: Stacking-Fault Evolution and the Phase Transformation ............................ 79

4.1. Introduction .......................................................................................................... 79

4.2. Experimental Procedure ...................................................................................... 79

4.3. Results .................................................................................................................. 85

4.4. Discussion ............................................................................................................ 91
4.5. Summary .................................................................................................................. 93

Chapter 5: Lattice-Strain Distribution and the Phase Transformation .................. 96
5.1. Introduction ............................................................................................................. 96
5.2. Experimental Procedure ....................................................................................... 96
5.3. Theory ..................................................................................................................... 97
5.4. Results .................................................................................................................... 105
5.5. Discussion ............................................................................................................. 152
5.6. Summary ................................................................................................................ 167

Chapter 6: Conclusions .............................................................................................. 168

Chapter 7: Future Work ............................................................................................. 171

References .................................................................................................................... 173

Appendices .................................................................................................................. 180

Vita .................................................................................................................................. 193
LIST OF TABLES

Table 1-1: The nominal composition of ULTIMET® alloy in weight percent. .................. 2

Table 3-1: List of stress levels at which diffraction patterns were obtained for the monotonic-loading experiments................................................................. 42

Table 3-2: Calculation of the work done on or by the transformation according to Patel and Cohen [50]................................................................................................................... 69

Table 4-1: Ex-situ specimens for the Synchrotron-radiation experiments. ....................... 81

Table 5-1: Measured and calculated compliances for the tension experiment. ............... 115

Table 5-2: Measured and calculated compliances for the compression experiment. ..... 118
LIST OF FIGURES

Figure 1-1: Tensile properties of ULTIMET® alloy as a function of temperature. .......... 3

Figure 1-2: Low-cycle fatigue behavior of ULTIMET® alloy at three temperatures. ....... 5

Figure 1-3: High-cycle fatigue behavior of ULTIMET® alloy........................................... 6

Figure 1-4: Equilibrium phase diagram of the binary Co-Cr system. ............................... 7

Figure 1-5: Experimental setup utilized for this work.......................................................... 9

Figure 1-6: Shift of a diffraction peak as a load is applied in-situ..................................... 11

Figure 1-7: Lattice strain with a component parallel to the tensile axis for various \( hkl \) in three engineering materials. (a) aluminum, (b) copper, and (c) stainless steel......... 13

Figure 1-8: Rietveld refinement on data taken from stainless steel (a) in the reference state and (b) after 2% plastic strain................................................................. 14

Figure 1-9: Summary of the research................................................................................ 17

Figure 2-1: Shearing involved in FCC twinning............................................................... 20

Figure 2-2: Dependence of the martensite-start temperature on stress for three cases..... 23

Figure 2-3: Phase transformation after high-cycle fatigue.............................................. 24

Figure 2-4: Phase transformation after low-cycle fatigue................................................. 25

Figure 2-5: A description of anisotropic lattice-strain development.................................. 27

Figure 2-6: A demonstration of intergranular strains through Eshelby theory............... 29

Figure 2-7: Strain pole figure of cold-rolled interstitial-free steel................................. 31

Figure 2-8: Inverse strain pole figure of 316 stainless steel after 5,159 fatigue cycles.... 33
Figure 2-9: (a) Measured strain pole figure and (b) SODF-calculated strain pole figure determined from a spherical-harmonics fit to the measured data. .............................................. 34

Figure 3-1: Specimen geometry for the tensile and high-cycle fatigue experiments. .... 36

Figure 3-2: Specimen geometry for the low-cycle fatigue experiment. ..................... 37

Figure 3-3: Engineering stress-strain curve obtained during the monotonic tension experiment. ......................................................................................................................... 40

Figure 3-4: Engineering stress-strain curve obtained during the in-situ monotonic compression experiment. ........................................................................................................ 41

Figure 3-5: Stress-strain hysteresis loops from the in-situ low-cycle fatigue experiment. Diffraction patterns were measured at the six labeled points. ................................................. 43

Figure 3-6: Overlay of diffraction patterns obtained during monotonic tensile loading. .... 47

Figure 3-7: Overlay of diffraction patterns obtained during the monotonic compression experiment: (a) axial data and (b) transverse data. ................................................................. 48

Figure 3-8: Overlay of diffraction patterns obtained during the high-cycle fatigue experiment: (a) axial data and (b) transverse data. ................................................................. 49

Figure 3-9: Overlay of diffraction patterns obtained during the room-temperature low-cycle fatigue experiment with a 2.5% total strain range. .................................................. 50

Figure 3-10: Overlay of diffraction patterns obtained during the room-temperature low-cycle fatigue experiment with a total strain range of 2.0%. .............................................. 52

Figure 3-11: Overlay of diffraction patterns obtained from the 873 K low-cycle fatigue experiment: (a) axial and (b) transverse data. ................................................................. 53

Figure 3-12: Overlay of diffraction patterns obtained from the 1,148 K low-cycle fatigue experiment. .............................................................................................................. 54

Figure 3-13: Rietveld fit for the case of uniaxial tension. ................................................. 55

Figure 3-14: Weight fraction of HCP phase as a function of tensile stress. .................. 56
Figure 3-15: Example Rietveld fit for the monotonic compression data................. 58

Figure 3-16: Weight fraction of HCP phase as a function of compressive stress......... 59

Figure 3-17: Example Rietveld fit for the high-cycle fatigue data........................... 61

Figure 3-18: Weight fraction of HCP as a function of fatigue cycles for stress-controlled high-cycle fatigue .............................................................................................................. 62

Figure 3-19: Accumulation rate as a function of fatigue cycles for the case of high-cycle fatigue. .................................................................................................................................................. 63

Figure 3-20: Example Rietveld fit for the case of low-cycle fatigue.......................... 65

Figure 3-21: Weight fraction of HCP as a function of fatigue cycles during low-cycle fatigue. .................................................................................................................................................. 66

Figure 3-22: Accumulation rate of HCP phase during low-cycle fatigue. .................... 67

Figure 3-23: Data from the literature illustrating the decreased plastic work that occurs during high-cycle fatigue. .................................................................................................................................................. 71

Figure 3-24: Measured and predicted temperature during (a) high-cycle fatigue and (b) low-cycle fatigue.................................................................................................................................................. 73

Figure 3-25: Intensity variation of the 111 and 200 diffraction peaks in the axial data.. 75

Figure 3-26: Intensity variations of the 220 diffraction peaks during compressive deformation.............................................................................................................................................. 77

Figure 4-1: Experimental setup for beamline G3 at the HASYLAB synchrotron......... 82

Figure 4-2: Mathematical fit of the peak profile using two methods. ......................... 84

Figure 4-3: The stacking-fault probability as a function of tensile plastic strain. ........ 86

Figure 4-4: The twinning probability as a function of tensile plastic strain. ............... 87

Figure 4-5: Domain size as a function of tensile plastic strain................................. 88
Figure 4-6: Stacking-fault probability as a function of compressive strain................. 89

Figure 4-7: Twinning probability as a function of compressive plastic strain. ............ 90

Figure 4-8: Domain size as a function of compressive plastic strain. ......................... 92

Figure 4-9: Demonstration of the preferred shear direction for the cases of (a) tensile deformation and (b) compressive deformation................................................................. 94

Figure 5-1: The fundamental triangle demonstrating that $A_{hkl}$ varies from 0 to 1/3 along two paths. ......................................................................................................................... 99

Figure 5-2: One-dimensional representation of the lattice strains of ULTIMET® alloy at tensile stresses of 300 and 890 MPa. ................................................................................. 100

Figure 5-3: Axial lattice strains from the monotonic tension experiment...................... 106

Figure 5-4: Transverse lattice strains from the monotonic tensile experiment.............. 107

Figure 5-5: Axial lattice strains from the monotonic compression experiment............. 108

Figure 5-6: Transverse lattice strains from the monotonic compression experiment...... 109

Figure 5-7: Axial lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422............... 111

Figure 5-8: Transverse lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.............. 112

Figure 5-9: Axial lattice strains obtained at the maximum strain from the low-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422........... 113

Figure 5-10: Transverse lattice strains obtained at the maximum tensile strain during low-cycle fatigue: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422......... 114

Figure 5-11: Measured compliances and the curve generated from the modified Kröner model......................................................................................................................... 116

Figure 5-12: Measured compliances and the calculated curve from the modified Kröner model for the compression data................................................................. 119
Figure 5-13: Measured and calculated lattice strains for the tensile experiment........... 120

Figure 5-14: The effective stresses from the strain analysis........................................... 121

Figure 5-15: The intergranular strain amplitude from the tensile experiment.............. 122

Figure 5-16: Measured and calculated lattice strains from the compression experiment: (a) axial data and (b) transverse data. ................................................................. 124

Figure 5-17: The effective stresses from the compression experiment. .................... 125

Figure 5-18: The intergranular-strain amplitude from the compression experiment..... 126

Figure 5-19: Measured and calculated lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data...................... 127

Figure 5-20: Measured and calculated lattice strains at the minimum stress. .......... 128

Figure 5-21: Effective stresses at the maximum stress from the high-cycle fatigue experiment....................................................................................................................... 129

Figure 5-22: Effective stresses at the minimum stress from the high-cycle fatigue experiment....................................................................................................................... 130

Figure 5-23: The intergranular-strain amplitudes at the maximum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data...................... 131

Figure 5-24: The intergranular-strain amplitude at the minimum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data...................... 132

Figure 5-25: Measured and calculated lattice strains at the maximum tensile strain for low-cycle fatigue: (a) axial data and (b) transverse data. ........................................ 134

Figure 5-26: Measured and calculated lattice strains at tensile zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data...................... 135

Figure 5-27: Measured and calculated lattice strains at compressive zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. .................. 136
Figure 5-28: Measured and calculated lattice strains at the maximum compressive stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. .......................... 137

Figure 5-29: Measured and calculated lattice strains at compressive zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. ................................. 138

Figure 5-30: Measured and calculated lattice strains at tensile zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. ........................................ 139

Figure 5-31: Effectives stresses at the maximum tensile strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{1,\text{eff}}$ ..................................................... 140

Figure 5-32: Effective stresses at tensile zero stress from the low-cycle fatigue experiment....................................................................................................................... 141

Figure 5-33: Effective stresses at compressive zero strain from the low-cycle fatigue experiment....................................................................................................................... 142

Figure 5-34: Effective stresses at the maximum compressive strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{1,\text{eff}}$ ................. 143

Figure 5-35: Effective stresses at compressive zero stress from the low-cycle fatigue experiment....................................................................................................................... 144

Figure 5-36: Effective stresses at tensile zero strain from the low-cycle fatigue experiment....................................................................................................................... 145

Figure 5-37: Intergranular-strain amplitude at the maximum tensile strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. ................................. 146

Figure 5-38: Intergranular-strain amplitude at tensile zero stress from the low-cycle fatigue experiment. ........................................................................................................ 147

Figure 5-39: Intergranular-strain amplitude at compressive zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. ................................. 148

Figure 5-40: Intergranular-strain amplitude at the maximum compressive strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. ................................. 149
Figure 5-41: Intergranular-strain amplitude at compressive zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. 150

Figure 5-42: Intergranular-strain amplitude at tensile zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data. 151

Figure 5-43: The intergranular-strain amplitudes during fatigue cycle 75. 154

Figure 5-44: Stress partitioning for tensile deformation. 156

Figure 5-45: Stress partitioning at the maximum stress from the high-cycle fatigue experiment. 158

Figure 5-46: Stress partitioning at the minimum stress for the high-cycle fatigue experiment. 159

Figure 5-47: Stress partitioning at the maximum tensile strain from the low-cycle fatigue experiment. 160

Figure 5-48: Stress partitioning at tensile zero stress from the high-cycle fatigue experiment. 161

Figure 5-49: Stress partitioning at compressive zero strain from the low-cycle fatigue experiment. 162

Figure 5-50: Stress partitioning at the maximum compressive strain from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 163

Figure 5-51: Stress partitioning at compressive zero stress for low-cycle fatigue. 164

Figure 5-52: Stress partitioning at tensile zero strain from the low-cycle fatigue experiment. 165
Chapter 1: Introduction

1.1. Cobalt-Based Superalloys

Superalloys, in general, were developed in order to obtain high-strength materials at temperatures above 650°C. Common superalloys include: austenitic Fe-based systems, Cr-Ni-Co-Fe systems, Ni-based systems, and Co-based systems [1]. The first cobalt-based superalloys were developed around the turn of the 20th century by Elwood Haynes for high-temperature, high-strength service [2-4]. By World War II, these materials were being used in the turbosupercharger for aircraft engines [1].

Cobalt alloys are available in wrought, cast, weld-overlay, and powder-metallurgy forms. While cobalt is expensive, this system of alloys exhibits desirable characteristics, such as corrosion/wear resistance and high-temperature strength. Cobalt alloys can be classified according to their uses into four categories, as discussed in [2].

1. Wear
2. High temperature
3. Biomedical
4. Aqueous corrosion

The material that is the subject of this work is known as ULTIMET® alloy and is manufactured by Haynes International, Inc. This commercial cobalt-based superalloy is suitable for high-temperature and aqueous-corrosion uses. The nominal composition of this cobalt-based superalloy is given in Table 1-1 [5]. Mechanical properties of ULTIMET® alloy have been characterized in the literature. For instance, Figure 1-1 [6] shows that three regimes are observed in the tensile properties as a function of
Table 1-1: The nominal composition of ULTIMET® alloy in weight percent.

<table>
<thead>
<tr>
<th>Co</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Fe</th>
<th>W</th>
<th>Mn</th>
<th>Si</th>
<th>N</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>54a</td>
<td>26</td>
<td>9</td>
<td>5</td>
<td>3</td>
<td>2</td>
<td>0.8</td>
<td>0.3</td>
<td>0.08</td>
<td>0.06</td>
</tr>
</tbody>
</table>

a As balance

(ULTIMET Alloy Product Brochure, Haynes International, Kokomo, IN, p. 3 [5].)
Figure 1-1: Tensile properties of ULTIMET® alloy as a function of temperature.

temperature: I. relatively mild changes of tensile properties with increasing temperature, II. stable tensile properties with increasing temperature, and III. relatively rapid changes of tensile properties with increasing temperature. Based upon this observation, Jiang et al. [6] performed low-cycle-fatigue experiments at one temperature in each of the three regimes, and his results are shown in Figure 1-2. The fatigue life of the alloy increased when the temperature increased from 293 to 873 K, but the fatigue life decreased when the temperature increased from 873 to 1,173 K. Jiang et al. [7] also performed high-cycle fatigue experiments on this material, as shown in Figure 1-3 [8]. The material exhibited a fatigue endurance limit of about 500 MPa during high-cycle fatigue. Example applications of ULTIMET® alloy include electrogalvanizing rolls, weld overlays, nozzles, and shielding for fan blades [5]. In the context of this work, high-cycle fatigue is defined as stress-controlled fatigue with tension-tension loading (i.e., no reversal of the load). Low-cycle fatigue, on the other hand, is defined as strain-controlled fatigue with fully-reversed tension-compression loading.

1.2. Strain-Induced Phase Transformation

Figure 1-4 [9] shows the equilibrium phase diagram for the binary Co-Cr system, with the approximate composition of ULTIMET® alloy denoted on the Figure by a dashed line. During processing, the material was (a) hot rolled at a temperature of 1,204°C, (b) annealed at 1,121°C in air, and (c) water quenched to room temperature. This process is paramount to a solution heat treatment in the face-centered-cubic (FCC) region of the phase diagram and a fast quench to the hexagonal-close-packed (HCP) region of the phase diagram. The allotropic FCC $\rightarrow$ HCP phase transformation is
Figure 1-2: Low-cycle fatigue behavior of ULTIMET\textsuperscript{®} alloy at three temperatures.

Figure 1-3: High-cycle fatigue behavior of ULTIMET alloy.

Figure 1-4: Equilibrium phase diagram of the binary Co-Cr system.

kinetically sluggish because of the very low free-energy change associated with the transition. Thus, the FCC phase is held in a metastable state after the fast quench [1].

The transition can occur when deformation is applied: a phenomenon known as a strain-induced phase transformation. The strain-induced FCC → HCP phase transformation is known to occur in ULTIMET® superalloy, and the first major objective of this work is to provide a complete characterization of the phase transformation under different loading conditions [6-8].

1.3. **Neutron Diffraction as a Characterization Tool**

Neutrons have many properties that make neutron diffraction an important tool for characterizing materials behavior [10]. For the purposes of this work, neutrons penetrate deeply into most engineering materials, such that the information obtained is averaged over a bulk sampling volume. Neutron diffraction is a nondestructive technique, and diffraction experiments involving external fields applied in-situ are feasible. This potential for neutron diffraction has been realized through the development of engineering diffractometers at pulsed neutron sources, including the Spectrometer for Materials Research and Stress (SMARTS) [11] at Los Alamos National Laboratory in the United States, the ENGIN-X spectrometer [12] at Rutherford-Appleton Laboratory in the United Kingdom, and the up-and-coming VULCAN spectrometer [13] at Oak Ridge National Laboratory in the United States. The scattering geometry of SMARTS and ENGIN-X is shown schematically in Figure 1-5. The specimen is placed 45° to the polychromatic incident beam. Detector banks are fixed at scattering angles of $2\theta = \pm 90^\circ$, where $\theta$ is the Bragg angle.
Figure 1-5: Experimental setup utilized for this work.
Neutron diffraction is governed by Bragg’s Law, which is shown in Equation 1–1:

\[ n\lambda = 2d_{\text{hkl}} \sin \theta , \]  

\text{Equation 1-1}

where \( n \) is the order of diffraction, \( \lambda \) is the wavelength of the radiation, and \( d_{\text{hkl}} \) is the spacing between the diffracting atomic planes defined by Miller indices \( hkl \) [14,15].

Time-of-flight techniques are utilized to measure the diffraction patterns. Hence, the entire spectrum of the polychromatic beam is analyzed with the Bragg angle held fixed [16]. The wavelengths of diffracted neutrons are calculated from the measured times-of-flight over the known moderator-to-sample (the moderator controls the wavelength spectrum of the incident beam) and sample-to-detector distances. These wavelengths are related to \( d \)-spacings through Bragg’s Law. The main advantage of time-of-flight neutron diffraction for this work is the ability to measure multiple diffraction peaks simultaneously without the need to scan through the Bragg angle. This technique will be used to increase the current understanding of the strain-induced phase transformation in ULTIMET® alloy under different loading conditions.

1.4. Lattice-Strain Development and Anisotropy

Diffraction peaks shift position when a load is applied, as shown in Figure 1-6. This peak shift can be used to calculate lattice strain, \( \varepsilon_{\text{hkl}} \), with Equation 1–2:

\[ \varepsilon_{\text{hkl}} = \frac{d_{\text{hkl}} - d_{\text{hkl,0}}}{d_{\text{hkl,0}}} , \]  

\text{Equation 1-2}

where \( d_{\text{hkl}} \) is the position of the \( hkl \) diffraction peak measured at a given load and \( d_{\text{hkl,0}} \) is the position of the \( hkl \) diffraction peak measured at a suitable, experiment-dependent reference state. The lattice strain is dependent upon \( hkl \), as the positions of several
Figure 1-6: Shift of a diffraction peak as a load is applied in-situ.
diffraction peaks can be monitored during an in-situ loading neutron-diffraction experiment. It is measured relative to a given reference state that must be stated when reporting such data. Since this strain is based upon changes in lattice spacing (i.e., the stretching of atomic bonds), it is elastic in nature.

Figure 1-7 shows lattice strains with a component parallel to the tensile axis measured in three FCC engineering materials. The curves in Figure 1-7 represent the behavior of six hkl: 111, 200, 220, 311, 331, and 531. Because of the nature of diffraction, the behavior of a particular diffraction peak represents an average of a certain subset of grains within the polycrystal. For instance, the strain measured from the shift of the 200 diffraction peak represents the behavior of the subset of grains that have {200} planes oriented for diffraction (i.e., the <200> plane normal vector is parallel to the loading axis in Figure 1-5). While the polycrystal may be isotropic on the macroscopic level, the individual crystallites on the grain level are certainly anisotropic. Therefore, it is the local mechanical anisotropy that gives rise to the observed differences in lattice strain measured from the various diffraction peaks, as evident in Figure 1-7.

Rietveld refinement uses a perfect crystal structure to mathematically fit a curve to an entire diffraction pattern. Software packages, such as the General Structure Analysis System (GSAS), are used to accomplish this refinement [17,18]. Figure 1-8(a) [19] shows a Rietveld refinement on stainless steel in the reference state before deformation was applied. A Rietveld refinement on a diffraction pattern from the same specimen after 2% plastic strain is shown in Figure 1-8(b). The difference between the measured and calculated values is shown in the curve at the bottom of each Figure. The difference curve for the strained specimen shows sinusoidal behavior, which is an
Figure 1-7: Lattice strain with a component parallel to the tensile axis for various $hkl$ in three engineering materials. (a) aluminum, (b) copper, and (c) stainless steel.

Figure 1-8: Rietveld refinement on data taken from stainless steel (a) in the reference state and (b) after 2% plastic strain.

indication that the peak position is not modeled well. The sinusoidal behavior is not observed in the specimen at the reference state. The difficulty in fitting the peak positions stems from the fact that the peaks shift at different rates relative to the applied load. Indeed, a distorted crystal structure is needed in order to accurately describe the peak positions when local mechanical anisotropy is present. The second major issue of this work is to propose a mathematical description of the anisotropic displacement of diffraction peaks from their expected positions due to the application of stress.

1.5. Motivation and Outline of the Research

While Jiang et al. [6-8] did address the phase transformation during their characterization of the fatigue properties, they only used ex-situ laboratory X-ray diffraction. Therefore, important details concerning the structural transition, such as the onset of the transformation and the accumulation rate of the developing phase during different loading conditions, were not elucidated. Characterization of the phase transformation under different loading conditions using neutron diffraction with in-situ applied load will offer a more complete understanding of how the new phase develops. The phase transformation should also be associated with stacking-fault development in the FCC matrix. This phenomenon can be characterized with a technique that is complementary to the neutron diffraction: ex-situ synchrotron X-ray diffraction. Analysis of the peak widths will help identify how subgrain-deformation characteristics, such as stacking-fault development, are related to the phase transformation.

The phase transformation is only one aspect of the overall deformation behavior of the alloy, however. Some description of the deformation in the FCC phase should be
present in the proposed work. Therefore, this work also includes a new method to
describe the anisotropic lattice-strain development in an FCC polycrystal during elastic
and plastic deformation. Within the context of this methodology, an approach to infer
load-sharing characteristics between the FCC phase and the developing HCP phase will
be proposed and applied.

The overall goal of the present work is to provide a comprehensive description of
the deformation behavior of a cobalt-based superalloy, including the phase
transformation, stacking-fault evolution, and lattice-strain development. The specific
objectives of this research include: (a) describing the onset of the phase transformation
under different loading modes, (b) characterizing the accumulation rate of the HCP phase
under different loading modes, (c) describing stacking-fault evolution in the material as a
result of deformation and its relation to the phase transformation, (d) developing and
applying a method to describe the lattice-strain distribution in reciprocal space in both the
elastic and plastic regimes of deformation, and (e) proposing a method to infer load-
sharing characteristics between the FCC and HCP phases through analysis of the lattice
strains of the FCC phase.

The work found herein is summarized in Figure 1-9. The experimental portion of
this work is found in the cross-hatched fields. They include (a) neutron-diffraction
experiments with in-situ monotonic tension, monotonic compression, high-cycle fatigue,
and low cycle fatigue and (b) line-profile analysis of specimens deformed by monotonic
tension and monotonic compression. These experiments contribute to the understanding
of the phase transformation, including the onset of the transformation, accumulation rate
of the HCP phase, crystallography of the HCP phase, and stacking-fault evolution. The
Figure 1-9: Summary of the research.
newly-developed analysis technique for the lattice strains involves Hooke’s Law in three dimensions, a modified version of the Kröner model, and a polynomial expansion of spherical harmonics. This method will be applied to each loading mode studied in the neutron-diffraction work. Finally, the phase transformation and lattice strains are related through the load-sharing characteristics between the FCC and HCP phases.
Chapter 2: Literature Review

2.1. Aspects of the Phase Transformation

2.1.1. Overview

Little free energy change (i.e., ~100 cal-g\(^{-1}\)) is associated with the allotropic FCC → HCP phase transformation [1]. The transformation is often classified as martensitic, although some debate remains on that issue [20]. Most of the experimental work in the literature focuses on the thermally-induced phase transformation [20-34], as opposed to the strain-induced phase transformation [35-39].

The references primarily discuss the formation of stacking faults in the FCC matrix as a pre-transformation event. The existence of stacking faults requires the creation of Shockley partial dislocations. Nishiyama [40] was the first to propose a mechanism for the FCC → HCP phase transformation in cobalt: the passage of coupled Shockley partial dislocations over every other \{111\} plane in the FCC matrix. The transformation occurs, therefore, via shearing of the lattice, which is similar in nature to FCC twinning (Figure 2-1) [41]. The shear associated with this deformation is derived by Hosford [41] as 0.707. Therefore, the transformation is considered largely to be dislocation driven, which will be the view adopted in this work.

2.1.2. Effect on Physical and Mechanical Properties

The two allotropic forms of cobalt exhibit very similar densities, and little volume change is expected during the phase transformation [1]. The elastic properties of the FCC and HCP phases show some variation, however, with modulus values reported to
Figure 2-1: Shearing involved in FCC twinning.

vary by 5% in pure cobalt at the transformation temperature \[1,42]\). This effect can be enhanced when anisotropy is considered, since the modulus is a strong function of the angle between the loading direction and the \([001]\) lattice vector for a hexagonal crystal \[41]\).

The literature indicates that the FCC phase is the plastically hard phase, with the ultimate tensile strength being higher in the FCC form than the HCP form \[1]\). Recent work by Tao et al. \[43,44]\) on austenitic stainless steel at cryogenic temperatures confirms this idea. Transmission electron microscopy work by Rajan et al. \[27-30]\) showed that the hardening mechanism of cobalt-based superalloys is actually stacking-fault intersections, as opposed to the formation of the HCP phase. Lu et al. \[45]\), Ishmaku and Han \[46]\), and Singh and Doherty \[47]\) agree with their conclusions. However, Singh and Doherty \[48]\), in another paper, suggest that load sharing between the FCC and HCP phases may attribute to the work hardening, as well.

The influence of the HCP phase on the low-cycle fatigue behavior has been studied by Chalant and Remy \[35]\). In these studies, planar deformation mechanisms, such as twinning and the phase transformation, promoted a longer fatigue life. This observation was attributed to an increased crack-initiation period along the HCP platelet boundaries. Farhangi \[49]\) later correlated the formation of the HCP phase with crack propagation, as well.

2.1.3. The Effect of Applied Stress on Martensitic Transformations

Patel and Cohen \[50]\) considered the transformation strains and their relation to the effect of applied stress on a martensitic phase transformation. If indeed the shear
strain along the habit plane is the only parameter that is important, then the effect of applied stress would be the same regardless of its direction (i.e., tensile or compressive). However, they showed that the martensite-start temperature demonstrated different dependencies on applied stress for the cases of uniaxial tension, uniaxial compression, and hydrostatic compression, as in Figure 2-2 [50]. Because of this observed effect, they reasoned that the transformation normal strain resolved on the habit plane must also play a role in the transformation.

2.1.4. Transformation in ULTIMET® Superalloy

The fatigue behavior of the cobalt-based ULTIMET® alloy has been extensively characterized by Jiang et al. [6-8,51,52]. They performed X-ray experiments on the fracture surfaces of fatigued specimens in order to determine the phase-transformation properties of the material under cyclic-loading conditions. In the case of high-cycle fatigue (Figure 2-3), the HCP 101 diffraction peak was not observed until a maximum stress level of 762 MPa was applied [8]. For low-cycle fatigue (Figure 2-4), the HCP diffraction peak was observed at a total strain range of 0.6%. The intensity of the HCP peak increased as the total strain range was increased up to 2.5%, indicating that a larger total strain range resulted in a larger extent of transformation. Thus, Jiang et al.’s [6-8,51,52] work showed that the phase transformation occurred under cyclic loading.

2.1.5. Critical Issues

While the work of Jiang et al. [6-8,51,52] represents an excellent characterization of the fatigue properties of ULTIMET® superalloy, his description of the strain-induced FCC → HCP phase transformation left two major questions unanswered. First, the onset
Figure 2-2: Dependence of the martensite-start temperature on stress for three cases.

(From J. R. Patel and M. Cohen: Acta Metall., 1953, 1, pp. 531-537 [50].)
Figure 2-3: Phase transformation after high-cycle fatigue.

Figure 2-4: Phase transformation after low-cycle fatigue.

of the phase transformation was not specified for the cyclic loading conditions. Second, the rate of accumulation of the new phase was not characterized. Answering these questions for different loading conditions, which is a major objective of this work, will offer valuable scientific insight into the nature of the phase transformation.

2.2. Anisotropic Lattice-Strain Development in Cubic Materials

2.2.1. Elastic Anisotropy

The effective Young’s modulus, $E_{\text{hkl}}$, in a cubic crystal along a certain lattice vector $\langle hkl \rangle$ is described by Equation 2–1 [41].

$$\frac{1}{E_{\text{hkl}}} = s_{11} + (2s_{12} - 2s_{11} + s_{44})A_{\text{hkl}} \quad \text{Equation 2-1}$$

The orientation parameter, $A_{\text{hkl}}$, in Equation 2–1 is defined in Equation 2–2 [19].

$$A_{\text{hkl}} = \frac{h^2k^2 + h^2l^2 + k^2l^2}{(h^2 + k^2 + l^2)^2} \quad \text{Equation 2-2}$$

Daymond et al. [19] incorporated $A_{\text{hkl}}$ into the Rietveld refinement code found in the General Structure Analysis System (GSAS) software package in order to mathematically describe the anisotropic lattice-strain development evident in Figure 1-7 [53]. His approach shifted the position of the calculated diffraction peak by an amount proportional to $A_{\text{hkl}}$. His results from the modified Rietveld fit are compared with the strains calculated from isolated diffraction peaks in Figure 2-5 [19] for polycrystalline stainless steel undergoing uniaxial tension. This Figure shows that his approach was successful below the elastic limit of 200 MPa. However, in the plastic regime the strains calculated from the modified-Rietveld and single-peak methods show significant deviations.
Figure 2-5: A description of anisotropic lattice-strain development.

The major limitation of Daymond et al.’s approach is that the method is physically significant only during the macroscopically elastic regime of deformation.

2.2.2. Intergranular Strains

Local mechanical anisotropy in a polycrystal leads to grain-to-grain interaction strains upon the application of a far-field load [54]. These strains, which result from stress/strain incompatibilities among neighboring grains, are termed intergranular strains in this work. Intergranular strains can be the result of elastic and plastic effects.

Intergranular strains resulting from elasticity can be understood by considering Eshelby theory [55] in Figure 2-6. Figure 2-6(a) shows an infinite, homogeneous, elastic medium with an ellipsoidal inclusion embedded in the medium. The elastic properties of the inclusion are distinguishable from those of the medium. The inclusion is removed from the medium and independently deformed in Figure 2-6(b). In order to insert the inclusion back into the hole in the matrix, a traction force must be applied to the surface of the inclusion, as in Figure 2-6(c). The traction force is analogous to intergranular stress that develops among neighboring grains of differing crystallographic orientation upon the application of load to a polycrystal. Intergranular strains result from the stress. The intergranular strains due to elastic effects are small relative to the total lattice strain and relax to zero upon release of the far-field load.

Plasticity-induced intergranular strains are similar in concept to the intergranular strains resulting from stretching of atomic bonds, except that they are caused by grain deformation due to dislocation glide. Since certain grain orientations are more favorable for slip-system activation than others, a large potential exists for inhomogeneities among
Figure 2-6: A demonstration of intergranular strains through Eshelby theory. (a) an infinite, homogeneous, elastic medium with an ellipsoidal inclusion, (b) the inclusion is removed from the matrix and independently deformed, (c) the deformed inclusion is inserted into the matrix with a traction force, which is analogous to intergranular stress due elastic effects.
neighboring grains in a random polycrystal. Indeed, any shape change of a given grain must be accommodated by its neighboring grains [41]. Continuity among neighboring grains can be maintained by the development of elastic strains, which are commonly referred to as simply “intergranular strains” in the literature [56]. These plasticity-induced intergranular strains are larger in magnitude than their elastic counterparts, and they significantly impact the measured lattice strain. Also, plasticity-induced intergranular strains do not relax to zero upon unloading.

Understanding plasticity-induced intergranular strains is important because they add to the strains caused by macroscopic stress fields in, for instance, welded joints and parts formed by plastic deformation [54,57]. Brown et al. [58], in fact, describe the total lattice strain as the sum of two contributions: the linear-elastic contribution and the plasticity-induced intergranular contribution. This description tacitly states that all deviations from linearity in Figure 1-7 are due to plasticity-induced intergranular strains.

2.2.3. Stress/Strain Orientation Distribution Function

The Stress/Strain Orientation Distribution Function (SODF) has been introduced in order to describe the distribution of residual intergranular strains in plastically-deformed polycrystals [59-64]. A comprehensive measurement of the distribution of intergranular strains in both reciprocal space (i.e., the various \( hkl \)) and sample-orientation space (i.e., the various components of strain relative to the sample coordinate system) is necessary in order determine the SODF.

Two representations of the lattice-strain distribution are commonly used in work involving the SODF: strain pole figures [62] (Figure 2-7) and inverse strain pole figures.
Figure 2-7: Strain pole figure of cold-rolled interstitial-free steel.

The strain pole figure is a two-dimensional representation of the lattice strain with a given crystal orientation (i.e., <110> for Figure 2-7) in sample-orientation space. The inverse strain pole figure, therefore, is a two-dimensional representation of a given lattice-strain component relative to the specimen coordinate system (i.e., parallel to the long axis of a cylindrical fatigue specimen in Figure 2-8) in reciprocal space. The inverse strain pole figure utilizes the fundamental triangle to represent the lattice strain of the various hkl for cubic polycrystals.

In the determination of the SODF, coefficients of spherical harmonics are fit to the measured data. The solution to the SODF is not unique (even with the comprehensive measurement), and different methods are used to stabilize the result [59-64]. Figure 2-9 shows measured and calculated strain pole figures from the SODF, as discussed by Wang et al. [60]. The spherical-harmonics approach accurately describes the distribution of residual intergranular strains in a plastically-deformed polycrystal.

2.2.4. Critical Issues

The main objective is to offer a description of elastic and plastic anisotropic lattice-strain development in cubic materials, as a description of only elastic anisotropy is currently available in the literature [19]. The approach developed in this work will assume that the total lattice strain is the sum of the linear-elastic contribution and the plasticity-induced intergranular contribution. Hooke’s Law and a modified version of the Kröner model will mathematically account for the linear-elastic contribution. The plasticity-induced intergranular contribution will be described by a polynomial expansion of spherical harmonics.
Figure 2-8: Inverse strain pole figure of 316 stainless steel after 5,159 fatigue cycles.

Figure 2-9: (a) Measured strain pole figure and (b) SODF-calculated strain pole figure determined from a spherical-harmonics fit to the measured data.

Chapter 3: Phase Transformation under Different Loading Conditions

3.1. Introduction

This chapter deals with the characterization of the phase-transformation characteristics under a variety of loading conditions, including monotonic tension and compression, stress-controlled high-cycle fatigue, strain-controlled low-cycle fatigue, and low-cycle fatigue at elevated temperatures. The onset of the transformation and the accumulation rate of the new phase during the various deformation conditions are characterized. This information is then used to draw conclusions about the nature of the transformation and its relationship to plasticity.

3.2. Experimental Procedure

3.2.1. Material

ULTIMET® alloy has a nominal composition in weight percent of 26Cr-9Ni-5Mo-3Fe-2W, with as-balance Co. The stock material was processed by (a) hot-rolling at a temperature of 1,204°C, (b) annealing at 1,121°C in air, and (c) water quenching to room temperature. Cylindrical, threaded-end specimens for the monotonic tension and high-cycle fatigue experiments were machined from plate stock with a 6 mm gauge diameter and a 31.6 mm gauge length, as shown in Figure 3-1. The specimens for the monotonic compression experiment were cylinders 12 mm in height and 6 mm in diameter. The cylindrical, threaded-end specimens for the low-cycle fatigue experiment were 8 mm in diameter with a 24 mm gauge length, as shown in Figure 3-2. The
Figure 3-1: Specimen geometry for the tensile and high-cycle fatigue experiments.
Figure 3-2: Specimen geometry for the low-cycle fatigue experiment.
macroscopic modulus and 0.2 \% yield stress obtained from the in-situ tension diffraction experiment were 256 GPa and 525 MPa, respectively.

3.2.2. In-Situ Neutron Diffraction

The characterization tool for this work was time-of-flight neutron diffraction. This technique utilizes a polychromatic beam with a fixed scattering angle to measure diffraction patterns. The advantage of time-of-flight diffraction in this case was that Bragg reflections of a large range of d-spacings were measured simultaneously without the need to scan through the Bragg angle. Therefore, the development of the HCP phase was monitored as a function of the deformation.

Two time-of-flight spectrometers were utilized for this work: 1) the Spectrometer for MAterials Research at Temperature and Stress (SMARTS) at Los Alamos Neutron Science Center, US [11] and 2) the ENGIN-X instrument at the ISIS Pulsed Neutron and Muon Source, UK [12]. The SMARTS instrument was used for the monotonic experiments and the high-cycle fatigue experiment, with neutron count times of one hour. The ENGIN-X instrument was used for the low-cycle fatigue experiments, with neutron count times of forty-five minutes.

The SMARTS experimental setup is shown in Figure 1-5(a). The specimen was oriented 45° to the polychromatic incident neutron beam with the scattering angle fixed at \(2\theta = \pm 90^\circ\) for two detector banks [11,65]. The direction of the scattering vectors, \(\vec{q}_r\) and \(\vec{q}_\perp\), which bisected the incident and diffracted wave vectors of the neutron beam, were constant for a given detector bank. The scattering vector was oriented parallel to the loading direction of the specimen for \(2\theta = +90^\circ\) (i.e., the axial data) and
perpendicular to the loading direction of the specimen for $\theta = -90^\circ$ (i.e., the transverse data). Figure 1-5(b) shows the experimental setup for ENGIN-X, which was similar to SMARTS except the specimen was rotated $90^\circ$.

3.2.3. Room-Temperature Mechanical Deformation

The deformation modes studied in this work included monotonic tension, monotonic compression, high-cycle fatigue, and low-cycle fatigue. The monotonic-loading experiments were performed at the SMARTS instrument. The engineering stress-strain curves obtained during the tension and compression experiments are shown in Figure 3-3 and Figure 3-4, respectively. For both monotonic experiments, diffraction patterns were obtained at roughly every 25 MPa, up to a maximum tensile or compressive stress of 890 MPa. A complete list of stress levels at which diffraction patterns were obtained is shown in Table 3-1. The creep deformation resulting from the hold times has been subtracted out of the stress-strain curves presented in Figure 3-3 and Figure 3-4.

The high-cycle fatigue experiment was performed at SMARTS in stress-controlled mode. The maximum and minimum stresses were $\sigma_{\text{max}} = 810$ MPa and $\sigma_{\text{min}} = 81$ MPa, respectively. Diffraction patterns were obtained at $\sigma_{\text{max}}$ and $\sigma_{\text{min}}$ for a number of fatigue cycles: 1, 5, 14, 55, 105, 505, 1,005, 5,005, 10,007, 11,008, 12,009, 13,009, 14,009, 15,009, 16,009, and 18,009. The specimen failed at 21,337 cycles.

The low-cycle fatigue experiment was conducted at the ENGIN-X instrument. This experiment was performed in strain-controlled mode with maximum and minimum strains of $\varepsilon_{\text{max}} = 1.25\%$ and $\varepsilon_{\text{min}} = -1.25\%$, respectively. Diffraction patterns were measured at six points along the stress-strain hysteresis loop, as shown in Figure 3-5, for
Figure 3-3: Engineering stress-strain curve obtained during the monotonic tension experiment. Creep deformation resulting from the neutron hold times has been subtracted.
Figure 3-4: Engineering stress-strain curve obtained during the in-situ monotonic compression experiment. Creep deformation resulting from the neutron hold times has been subtracted.
Table 3-1: List of stress levels at which diffraction patterns were obtained for the monotonic-loading experiments.

<table>
<thead>
<tr>
<th>Tensile Stress Levels (MPa)</th>
<th>Compressive Stress Levels (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>-10</td>
</tr>
<tr>
<td>120</td>
<td>-60</td>
</tr>
<tr>
<td>220</td>
<td>-120</td>
</tr>
<tr>
<td>250</td>
<td>-170</td>
</tr>
<tr>
<td>275</td>
<td>-220</td>
</tr>
<tr>
<td>300</td>
<td>-250</td>
</tr>
<tr>
<td>325</td>
<td>-275</td>
</tr>
<tr>
<td>350</td>
<td>-300</td>
</tr>
<tr>
<td>375</td>
<td>-325</td>
</tr>
<tr>
<td>400</td>
<td>-350</td>
</tr>
<tr>
<td>425</td>
<td>-375</td>
</tr>
<tr>
<td>450</td>
<td>-400</td>
</tr>
<tr>
<td>475</td>
<td>-425</td>
</tr>
<tr>
<td>500</td>
<td>-450</td>
</tr>
<tr>
<td>525</td>
<td>-475</td>
</tr>
<tr>
<td>550</td>
<td>-500</td>
</tr>
<tr>
<td>575</td>
<td>-525</td>
</tr>
<tr>
<td>600</td>
<td>-550</td>
</tr>
<tr>
<td>625</td>
<td>-575</td>
</tr>
<tr>
<td>720</td>
<td>-600</td>
</tr>
<tr>
<td>810</td>
<td>-625</td>
</tr>
<tr>
<td>890</td>
<td>-720</td>
</tr>
<tr>
<td>600</td>
<td>-510</td>
</tr>
<tr>
<td>300</td>
<td>-250</td>
</tr>
<tr>
<td>20</td>
<td>-20</td>
</tr>
<tr>
<td>600</td>
<td>-720</td>
</tr>
<tr>
<td>810</td>
<td>-810</td>
</tr>
<tr>
<td>890</td>
<td>-830</td>
</tr>
<tr>
<td>830</td>
<td>-850</td>
</tr>
<tr>
<td>890</td>
<td>-890</td>
</tr>
<tr>
<td>900</td>
<td>-900</td>
</tr>
<tr>
<td>600</td>
<td>-600</td>
</tr>
<tr>
<td>300</td>
<td>-300</td>
</tr>
<tr>
<td>20</td>
<td>-20</td>
</tr>
</tbody>
</table>
Figure 3-5: Stress-strain hysteresis loops from the in-situ low-cycle fatigue experiment. Diffraction patterns were measured at the six labeled points.
a number of fatigue cycles: 1, 4, 8, 12, 30, 50, 75, 100, 250, and 500 cycles [66]. The six measurement points corresponded to: the maximum tensile strain (1), the unload point after the tensile half cycle (2), compression at 0 strain (3), the maximum compressive strain (4), the unload point after the compressive half cycle (5), and tension at 0 strain (6), as labeled in Figure 3-5. The specimen failed at 950 fatigue cycles.

3.2.4. Elevated-Temperature Mechanical Deformation

The low-cycle fatigue behavior was also studied under elevated-temperature conditions at maximum and minimum strains of $\varepsilon_{\text{max}} = 1.0\%$ and $\varepsilon_{\text{min}} = -1.0\%$, respectively. The temperatures chosen were 873 K and 1,148 K, along with a second room-temperature experiment for a baseline at the decreased strain amplitude. The temperature was controlled by a thermocouple wrapped around the specimen and a parabolically-focused optical furnace. The same six measuring points as described in Figure 3-5 were used for these experiments. Diffraction patterns were obtained at (a) cycles 12, 30, 50, and 75 at room temperature; (b) cycles 1, 12, 30, 50, 100, 250, and 500 at 873 K; and (c) cycles 1, 4, 8, 12, 30, 50, 75, and 100 at 1,148 K.

3.2.4. Rietveld Refinement for Weight Fraction

Diffraction patterns were recorded in two detector banks: one with the scattering vector parallel to the loading axis and one with the scattering vector perpendicular to the loading axis. These patterns, therefore, represent two texture components. A simultaneous Rietveld fit to the data can be used to determine the weight fraction of HCP phase with the General Structure Analysis System (GSAS) software package [17,67]. The phase fraction in GSAS is determined by a phase-specific scale factor through:
\[ W_p = \frac{S_{ph} m_p}{\sum_{p=1}^{N_p} S_{ph} m_p}, \]  

Equation 3-1

where \( W_p \) is the weight fraction of the \( p^{th} \) phase, \( S_{ph} \) is the scale factor for the \( p^{th} \) phase in the \( h^{th} \) histogram, \( m_p \) is the mass of the unit cell for the \( p^{th} \) phase, and \( N_p \) is the total number of phases present [17].

The advantage of this approach is the ability to obtain the weight fraction information in-situ along with the application of the load. However, two main factors increase the uncertainty in the weight-fraction information obtained in this manner: (a) the measurement of only two texture components, while numerous texture components would be desirable and (b) the large amount of plastic deformation led to significant texture development and large anisotropic peak shifts, both of which are difficult to model. Since the weight-fraction values reported here are subject to these limitations, the main conclusions are based upon the determined accumulation rates (i.e., the trends).

The space groups used in the refinement were \( \text{Fm\overline{3}m} \) for the FCC phase and \( \text{P6}_3/\text{mmc} \) for the HCP phase [68]. The atoms were placed at 0,0,0 and face-centering translations, with fractional occupancies of the constituent elements equal to the alloy composition in atomic percent. The peak positions were modeled by Daymond et al.’s [19] approach to cubic anisotropy. The texture development in both phases was modeled with \( 8^{th} \) order spherical harmonics, with the assumption of cylindrical symmetry of the texture.
3.3. Results

3.3.1. Overview of the Phase Transformation

The diffraction spectra obtained as a function of tensile load are shown in Figure 3-6. The 101 HCP diffraction peak was first observed in the axial data [Figure 3-6(a)] at a macroscopic stress of 720 MPa. The transverse data [Figure 3-6(b)] show that the 103 HCP peak was resolved in addition to the 101 HCP Bragg reflection. The intensity of the HCP peaks increased with increasing tensile stress.

The diffraction spectra obtained during uniaxial compressive loading are shown in Figure 3-7. The axial data [Figure 3-7(a)] show no sign of the HCP phase. However, the transverse data [Figure 3-7(b)] indicate that the 101 HCP peak first appeared at a compressive stress of 830 MPa. The intensity of this peak slightly increased with increasing compressive stress.

The overlay of diffraction patterns from the high-cycle fatigue experiment is shown in Figure 3-8. The axial data [Figure 3-8(a)] demonstrate that the 101 HCP peak was observed upon the first fatigue cycle. The 101 and 103 HCP peaks were observed in the transverse data [Figure 3-8(b)], which is similar to the case in Figure 3-6. The striking feature of this data set is the fact that the HCP peaks did not increase in intensity as fatigue progressed.

The results from the room-temperature low-cycle fatigue experiment with a total strain range of 2.5% are shown in Figure 3-9. The first observation of the HCP phase during low-cycle fatigue occurred at fatigue cycle 8, where the 101 HCP peak was observed in both the axial [Figure 3-9(a)] and transverse [Figure 3-9(b)] data. The
Figure 3-6: Overlay of diffraction patterns obtained during monotonic tensile loading: (a) axial data and (b) transverse data.
Figure 3-7: Overlay of diffraction patterns obtained during the monotonic compression experiment: (a) axial data and (b) transverse data.
Figure 3-8: Overlay of diffraction patterns obtained during the high-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 3-9: Overlay of diffraction patterns obtained during the room-temperature low-cycle fatigue experiment with a 2.5% total strain range: (a) axial data and (b) transverse data.
intensity of this peak increased dramatically with increasing fatigue cycles. Additionally, the 102 and 103 HCP peaks were resolved in the axial and transverse data at 250 and 500 cycles. Finally, the 100 HCP diffraction peak was observed at 500 cycles in the transverse data. The results were similar for the room-temperature low-cycle fatigue experiment with a total strain range of 2%, as shown in Figure 3-10. The first observation of the 101 HCP peak was 30 fatigue cycles.

The results from the low-cycle fatigue experiment at 873 K are found in Figure 3-11. This experiment showed that the material remained FCC for a majority of the fatigue test at 873K. In fact, the 101 HCP peak was not observed until 500 fatigue cycles in the axial data. The specimen failed before a diffraction pattern could be obtained after 500 fatigue cycles. Finally, the diffraction patterns obtained from the low-cycle fatigue experiment at 1,148 K are presented in Figure 3-12. This data shows only the typical FCC diffraction peaks.

3.3.2. Weight Fraction during Monotonic Tension

Figure 3-13 illustrates an example Rietveld fit at 890 MPa for determining weight fraction in the tension data. The diffraction pattern included the typical 111, 200, 220, and 311 FCC peaks in the high-intensity portion of the spectrum. The 101 HCP peak was observed in the axial data, while the 101, 102, and 103 HCP peaks were observed in the transverse data. All of these peaks represent pyramidal-type planes in the hexagonal system. The weight fraction of the HCP phase is shown as a function of tensile stress in Figure 3-14. While no HCP peak was observed below 720 MPa, the weight fraction increased with increasing tensile stress from 720 MPa to 890 MPa. Upon unloading, the
Figure 3-10: Overlay of diffraction patterns obtained during the room-temperature low-cycle fatigue experiment with a total strain range of 2.0%. 

ULTIMET® Superalloy
Low-Cycle Fatigue: 298 K

Normalized Intensity

d-spacing (nm)

0 cycles
30 cycles
75 cycles
Figure 3-11: Overlay of diffraction patterns obtained from the 873 K low-cycle fatigue experiment: (a) axial and (b) transverse data.
Figure 3-12: Overlay of diffraction patterns obtained from the 1,148 K low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 3-13: Rietveld fit for the case of uniaxial tension: (a) axial data and (b) transverse data.
Figure 3-14: Weight fraction of HCP phase as a function of tensile stress.

\[ W_{f,\text{tension}} = 0.001\sigma - 0.714 \]
volume fraction remained roughly constant. The weight fraction demonstrated a linear dependence on tensile stress for the stress range of 720 MPa to 890 MPa, as in:

\[ W_{f,\text{tension}} = a\sigma + b, \quad \text{Equation 3-2} \]

where \( W_{f,\text{tension}} \) is the weight fraction of HCP during monotonic tension, \( \sigma \) is the stress, \( a \) is the slope, and \( b \) is the intercept. The accumulation rate of the HCP phase with respect to tensile stress, \( R_{\text{tension}} \), is defined as:

\[ R_{\text{tension}} = \frac{dW_{f,\text{tension}}}{d\sigma} = a. \quad \text{Equation 3-3} \]

The onset of the transformation during tension, \( \sigma_{0,\text{tension}} \), is estimated by the stress that satisfies \( W_{f,\text{tension}} = 0 \), which is:

\[ \sigma_{0,\text{tension}} = -\frac{b}{a}. \quad \text{Equation 3-4} \]

For this case, \( a = 0.00104 \text{ MPa}^{-1} \) and \( b = -0.714 \), which means that \( R_{\text{tension}} = 0.1 \text{ g-MPa}^{-1} \) and \( \sigma_{0,\text{tension}} = 685 \text{ MPa} \). All accumulation rates reported in this work were determined assuming 100 g of material.

3.3.3. Weight Fraction during Monotonic Compression

The example Rietveld fit at 890 MPa for the compression data is shown in Figure 3-15. Overall, the intensity of the HCP peaks for the compression experiment was very low relative to background. Figure 3-16 shows the weight fraction of HCP versus compressive stress. The weight fraction during compression, \( W_{f,\text{compression}} \), demonstrated a linear dependence on stress just as in Equation 3-2. The parameters for the compressive case were \( a = 0.000467 \text{ MPa}^{-1} \ b = -0.326 \), such that the accumulation rate and transformation onset were \( R_{\text{compression}} = 0.05 \text{ g-MPa}^{-1} \) and \( \sigma_{0,\text{compression}} = 698 \text{ MPa} \),
Figure 3-15: Example Rietveld fit for the monotonic compression data: (a) axial data and (b) transverse data.
Figure 3-16: Weight fraction of HCP phase as a function of compressive stress.

\[ W_{f, \text{compression}} = 0.000467\sigma - 0.326 \]
respectively.

3.3.4. Weight Fraction during High-Cycle Fatigue

The example Rietveld fit at cycle 1 for high-cycle fatigue is shown in Figure 3-17. The weight fraction of HCP at $\sigma_{\text{min}}$ is shown versus fatigue cycles in Figure 3-18. The data at $\sigma_{\text{min}}$ are presented because less scatter was observed as compared to the data at $\sigma_{\text{max}}$. The HCP phase developed quickly during the first few fatigue cycles, and then the weight fraction saturated. The weight fraction of HCP phase during high-cycle fatigue, $W_{f,HCF}$, demonstrated a logarithmic dependence on fatigue cycles, as in:

$$W_{f,HCF} = a \log N + b,$$

Equation 3-5

where $a$ and $b$ are the slope and intercept, respectively, on a semilog scale and $N$ is the number of fatigue cycles. The accumulation rate of the HCP phase during high-cycle fatigue, $R_{HCF}$, is defined by:

$$R_{HCF} = \frac{dW_{f,HCF}}{dN} = \frac{a}{N \ln 10}.$$

Equation 3-6

The onset of the transformation during high-cycle fatigue, $N_{0,HCF}$, is estimated by the fatigue cycle at which $W_{f,HCF} = 0$, as in:

$$N_{0,HCF} = 10 \frac{b}{a}.$$

Equation 3-7

For HCF, $a$ and $b$ were determined as $a = 0.00415$ and $b = 0.177$ at $\sigma_{\text{min}}$. The accumulation rate and transformation onset during high-cycle fatigue were $R_{HCF} = 0.18(1/N)$ and $N_{0,HCF} = 0$, respectively. $R_{HCF}$ is shown as a function of $N$ in Figure 3-19. The accumulation rate starts quickly drops to 0 within the first 50 cycles. For a majority of the high-cycle fatigue life, no HCP phase is formed.
Figure 3-17: Example Rietveld fit for the high-cycle fatigue data: (a) axial data and (b) transverse data.
Figure 3-18: Weight fraction of HCP as a function of fatigue cycles for stress-controlled high-cycle fatigue.

\[ W_{f,HCF} = 0.004 \ln N + 0.177 \]
Figure 3-19: Accumulation rate as a function of fatigue cycles for the case of high-cycle fatigue.

**ULTIMET® Superalloy**
High-Cycle Fatigue

\[ R_{HCF} = 100 \left( \frac{0.0018}{N} \right) \]
3.3.5. Weight Fraction during Room-Temperature Low-Cycle Fatigue

The example Rietveld fit at 500 fatigue cycles for low-cycle fatigue is presented in Figure 3-20. The weight fraction of HCP phase is shown as a function of fatigue cycles in Figure 3-21. The weight fraction of HCP during low-cycle fatigue demonstrated a logarithmic dependence on fatigue cycles, as in Equation 3–5. For low-cycle fatigue, \( a = 0.121 \) and \( b = -0.0661 \). The accumulation rate and transformation onset during low-cycle fatigue were \( R_{LCF} = 5.3(1/N) \) and \( N_{0,LCF} = 3 \) cycles, respectively. \( R_{LCF} \) is shown as a function of \( N \) in Figure 3-22. While the accumulation rate certainly decreases with fatigue cycles, it is still appreciable up to 100 cycles.

3.4. Discussion

3.4.1. Phase Transformation Mechanism

Nishiyama [40] proposed that the formation of the HCP phase should occur by the passage of a stacking fault over every second \{111\} plane. Since then, the accumulation and coalescence of stacking faults has become a well-studied mechanism of the FCC → HCP phase transformation in cobalt [20,31,35,37,39]. This mechanisms leads to the observed orientation relationship between the parent and daughter phases: \((111)_{\text{FCC}} \parallel (001)_{\text{HCP}}\), which is know as the Shoji-Nishiyama relationship [20,40]. The existence of stacking faults implies the creation of Shockley partial dislocations in the FCC matrix, so the transformation is dislocation driven. Plastic deformation is, therefore, an important prerequisite for this particular transformation. The observations of this experimental work should all be tied to plastic deformation as a primary driving force for the transformation.
Figure 3-20: Example Rietveld fit for the case of low-cycle fatigue: (a) axial data and (b) transverse data.
Figure 3-21: Weight fraction of HCP as a function of fatigue cycles during low-cycle fatigue.

\[ W_{f, LCF} = 0.121 \ln N - 0.066 \]
Figure 3-22: Accumulation rate of HCP phase during low-cycle fatigue.

\[ R_{LCF} = 100 \left( \frac{0.053}{N} \right) \]

ULTIMET® Superalloy
Low-Cycle Fatigue
3.4.2. Tension-Compression Asymmetry

According to the results of this study, the phase transformation is favored under tensile deformation as compared to compression. This observation is in accordance with the work of Patel and Cohen [50], who observed that the martensite-start temperature in Fe alloys increased at a greater rate under tensile stress than under compressive stress. They argue that the direction of the applied stress (i.e., tensile or compressive) would not have any affect on the transformation if it was sensitive only to the resolved shear stress. Since an effect was observed experimentally (Figure 2-2), they showed with a thermodynamic argument that the dilatational component was also significant, as in:

\[
U = \frac{1}{2} \gamma_0 \sigma_1 \sin 2\theta ± \frac{1}{2} \epsilon_0 \sigma_1 (1 + \cos 2\theta),
\]

Equation 3-8

where, \( U \) is the work done on or by the transformation, \( \gamma_0 \) is the shear strain of the transformation, \( \sigma_1 \) is the absolute value of the applied stress, \( \theta \) is the angle between the loading axis and the habit-plane normal vector, and \( \epsilon_0 \) is the normal strain of the transformation [50].

Table 3-2 shows the relevant parameters of Equation 3-8, along with the resulting maximum \( U \), for Patel and Cohen’s [50] work and the transformation considered here. For both cases, the value of \( U \) for tension is greater than that for compression. Since \( U \) adds to the thermodynamic driving force [50], tensile deformation is considered to be more favorable than compressive deformation. The dilatational strain for this case is an order of magnitude smaller than that considered in the previous work. Even so, the applied stress is 700 MPa for this case, as compared to only 7 MPa in the case of Patel and Cohen [50]. So, the applied stress was large enough in this case to compensate for
Table 3-2: Calculation of the work done on or by the transformation according to Patel and Cohen [50].

<table>
<thead>
<tr>
<th></th>
<th>Patel and Cohen</th>
<th>Present Work</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\gamma_0$</td>
<td>0.2</td>
<td>0.3535</td>
</tr>
<tr>
<td>$\varepsilon_0$</td>
<td>0.04</td>
<td>0.008</td>
</tr>
<tr>
<td>$\sigma_1$</td>
<td>7 MPa</td>
<td>700 MPa</td>
</tr>
<tr>
<td>$U_{max}$ Tension</td>
<td>6 J/mol</td>
<td>864 J/mol</td>
</tr>
<tr>
<td>$U_{max}$ Compression</td>
<td>4 J/mol</td>
<td>825 J/mol</td>
</tr>
</tbody>
</table>
the rather small volume change associated with the FCC → HCP transformation.

3.4.3. Phase Transformation under Cyclic Loading

The transformation immediately occurred during the first cycle with no accumulation with further cycling for the case of high-cycle fatigue. This observation is in stark contrast with the case of low-cycle fatigue, where the transformation accumulated gradually with increasing cycles. This contrast is due to the differences in the way plasticity accumulates during the two cases. For high-cycle fatigue, some plastic strain does occur with each fatigue cycle. This phenomenon is commonly termed strain ratcheting and is believed to be related to the Baushinger effect [69]. Even so, the amount of plastic strain per fatigue cycle drastically reduces after the initial loading period, as demonstrated in Figure 3-23 [8]. For low-cycle fatigue, on the other hand, reverse plasticity occurs during each cycle. This compressive half-cycle allows for a large (1~2% for this case) accumulation of tensile plastic strain during each fatigue cycle. Considering the discussion of Section 3.4.2, most of the HCP-phase accumulation during low-cycle fatigue likely occurs during this tensile half-cycle, beginning with point 5 in Figure 3-5. In short, the contrast in the transformation characteristics during the two fatigue cases demonstrates the connection between (tensile) plasticity and the phase transformation.

3.4.4. Tensile Plastic Work Criterion

A hysteresis energy criterion for a strain-induced phase transformation has been considered before in the literature [70]. Because of the effect of the transformation dilatational strain (see Section 3.4.2), the criterion must be restricted to tensile plastic
Figure 3-23: Data from the literature illustrating the decreased plastic work that occurs during high-cycle fatigue.

work. This explanation is consistent with the discussion in Section 4.3.3, since the area under the hysteresis loops decreases with increasing fatigue cycles for high-cycle fatigue. In contrast, the area under the hysteresis loops remains roughly constant for low-cycle fatigue, allowing for the observed accumulation of the HCP phase.

Furthermore, the temperature evolution of this material during high-cycle and low-cycle fatigue has been characterized by infrared thermography and thermodynamic modeling [51,52]. The results showed that the temperature predictions were more accurate for high-cycle fatigue than for low-cycle fatigue, as shown in Figure 3-24 [51,52]. The model neglected the energy dissipation due to microstructural changes, so all the hysteresis energy was assumed to be released as heat. For the high-cycle fatigue case, Figure 3-24(a) [51], the maximum stress of 703 MPa is likely too small to generate an appreciable amount of HCP phase, considering the onset of 685 MPa as suggested by the neutron results. With no transformation to dissipate the hysteresis energy, the model captured the temperature evolution very well. For the low-cycle fatigue case, Figure 3-24(b) [52], the total strain range of 2.5%, which is identical to the fatigue conditions of the present neutron experiment, was indeed sufficient for the HCP phase to form and accumulate. So, the same model consistently over-predicted the temperature. The energy dissipation due to the transformation is indeed small, since the difference between the predicted and measured temperatures in Figure 3-24(b) is only 4°C. Nevertheless, the energy dissipation due to microstructural changes is not necessarily negligible for the case of ULTIMET® superalloy.
Figure 3-24: Measured and predicted temperature during (a) high-cycle fatigue and (b) low-cycle fatigue.

3.4.5. Transformation Texture under Tension

FCC metals undergoing uniaxial tension typically exhibit two texture components: <200> and <111> parallel to the loading axis [41]. This texture development would require an increase in the intensity of the 111 and 200 peaks in the axial data. Figure 3-25 shows that only the 111 peak increased in intensity in the axial detector data, while the 200 peak actually decreased in intensity after an initial increase. This observation suggests that grains with <200> oriented parallel to the loading axis are participating in the phase transformation, with a competition between deformation texture and transformation causing the non-monotonic behavior. Non-monotonicity is also seen in the 111 intensity after 810 MPa, since the intensity decreased after an initial increase with increasing applied stress.

Typical HCP texture under uniaxial tension involves the c-axis aligning perpendicular to the loading axis [41]. The 001 HCP peak is masked by the 111 FCC peak, but the (100) prismatic atomic plane is normal to the (001) basal plane in the hexagonal system. Therefore, the typical HCP fiber texture would exhibit itself as the 100 reflection in the axial data. This diffraction peak would appear in Figure 3-6 at a d-spacing of 0.22 nm, a position which is not masked by any FCC peak. In fact, Figure 3-6 shows that only the pyramidal reflections of the type $10k$ were observed.

The transformation exhibits the well-known $\{001\}_\text{HCP} \parallel \{111\}_\text{FCC}$ orientation relationship between the parent and daughter phases. Shear deformation is required along the {111} planes in order for the transformation to occur. A transformation texture of 001 parallel to the loading axis would imply that a {111} plane with no resolved shear transformed, which is not likely given the nature of the transformation. Therefore,
Figure 3-25: Intensity variation of the 111 and 200 diffraction peaks in the axial data.
transformation texture, not deformation texture, dominates the HCP phase [44]. In fact, the c-axis seems to form at angles centered around 45° to the loading axis. This transformation texture ensures that shear deformation can occur along the close-packed planes in the FCC lattice.

3.4.6. Transformation Texture under Compression

Typical FCC texture under compressive deformation involves <110> aligning parallel to the load [41]. Figure 3-26 shows that the 220 peak indeed increases in intensity during compressive loading. The HCP texture under compressive-loading conditions involves the c-axis aligning parallel to the loading axis. As explained in Section 3.3.4, this orientation would imply observation of the 100 reflection in the transverse data at a d-spacing of 0.22 nm, and the data in Figure 3-7 does not meet that condition. Again, even though the transformation is less favorable under compressive loading conditions, the transformation texture dominates the HCP phase over the expected deformation texture. Even in the compressive case, shear deformation must be maintained along the close-packed planes in order to develop the HCP phase.

3.4.7. The Transformation at Different Temperatures

The diffraction data obtained at 873 K (Figure 3-11) showed that the transformation did occur during low-cycle fatigue, but it was greatly retarded compared to the room-temperature results (Figure 3-10). The FCC phase is beginning to stabilize as the temperature approaches the FCC-HCP boundary in the equilibrium phase diagram (Figure 1-4), which is about 1,173 K for the binary Co-Cr system. These results are in agreement with Jiang et. al’s [6] work, where the HCP phase was observed at this
Figure 3-26: Intensity variations of the 220 diffraction peak during compressive deformation.
temperature. At the temperature of 1,148 K, no phase transformation was observed. These results also confirm Jiang et. al’s [6] work at 1,173 K. The Ni present in this alloy is an FCC stabilizer [35], so the actual boundary between the FCC and HCP regions is likely less than 1,173 K as shown in Figure 1-4. Since the phase transformation is affected by the temperature, it likely plays a role in the anomalous fatigue life observed in Figure 1-2. To completely understand the effect the transformation has on the fatigue life, however, the lattice strains must be considered. This analysis is discussed in Sections 5.5.2 and 5.5.3.

3.5. Summary

This chapter focused on the phase-transformation characteristics of a cobalt-based superalloy. The HCP phase demonstrated a linear dependence on applied stress and a logarithmic dependence on fatigue cycles. The transformation onsets at room temperature during tension, compression, high-cycle fatigue, and low-cycle fatigue were characterized as 685 MPa, 698 MPa, the first fatigue cycle, and the third fatigue cycle, respectively. The accumulation rates during monotonic tension and compression were reported as 0.1 g-MPa$^{-1}$ and 0.05 g-MPa$^{-1}$, respectively. The accumulation rates during the fatigue cases were inversely proportional to the cycle number. A criterion of tensile plastic work consistently explains the observed results. The observed texture of the HCP phase is consistent with an orientation that maintains a shear deformation on close-packed planes in the FCC matrix. At 873 K, the HCP phase was found to form at a much later fatigue cycle than at room temperature. At 1,148K, the FCC phase was stable with no observed transformation to the HCP phase.
Chapter 4: Stacking-Fault Evolution and the Phase Transformation

4.1. Introduction

This chapter is concerned with subgrain deformation characteristics of ULTIMET® superalloy. The information is obtained via peak-profile analysis of synchrotron X-ray data. This data is complementary to the neutron-diffraction data discussed in Chapter 3, since this method probes information on a more local level inside the material. The work of this chapter revealed deformation characteristics on a smaller scale than the neutron diffraction, so additional insight into the overall deformation behavior is gained.

4.2. Experimental Procedure

4.2.1. Material

ULTIMET® alloy is a commercial superalloy with a nominal composition of 26Cr-9Ni-5Mo-3Fe-2W and balance Co (in weight percent). The material for the present study was supplied by Haynes International, Inc. Before specimens were machined, the stock material was processed by (a) hot rolling at a temperature of 1,204°C, (b) annealing at 1,121°C in air, and (c) water quenching to room temperature. The fast quench created a metastable FCC structure with less than 5% (by volume) carbides. The range of grain diameters in the material was 50 - 250 μm [8].
4.2.2. Synchrotron X-Ray Diffraction

The specimens for the synchrotron X-ray studies were deformed separately for the diffraction measurements. All ex-situ deformation was performed on the same servohydraulic Materials Test System, MTS model 810. Individual specimens were deformed to 0, 0.6, 2.5, 7, and 10% plastic strains for the tensile case and 0, -3.3, -5.6, and -7.9% for the compressive case, as shown in Table 4-1. Table 4-1 also gives the corresponding stress values for each specimen. Small specimens, 3 - 5 mm high, were cut from the centers of the gauge sections of the deformed material. These specimens were mechanically polished to a surface finish of 0.5 μm and electrolytically polished in a solution of perchloric acid in ethyl alcohol for 10 seconds at 5 V.

The experiment for the X-ray line-profile analysis was performed at Hasylab beamline G3 in Hamburg, Germany [71], where monochromatic X-ray synchrotron radiation with a wavelength of 0.179 nm was used. While beamline G3 contains a four-circle diffractometer [71], this experiment only utilized a scan through the Bragg angle, as demonstrated schematically in Figure 4-1. The scattering vector was oriented parallel to the loading axis for all measurements. The beam size was defined by a 2 mm x 2 mm slit collimator, and a soller inside the secondary beam confined the acceptance to 0.15°. A scintillation counter was used to measure the diffraction pattern with the diffraction angle, 2θ, ranging from 2θ = 48° to 2θ = 128° and a step size of Δ2θ = 0.016° for ψ = 0° (ψ = the inclination angle) and a data-acquisition time of about 2 seconds per step. The instrumental resolution was determined with Si powder.
Table 4-1: Ex-situ specimens for the Synchrotron-radiation experiments.

<table>
<thead>
<tr>
<th>Plastic Strain (%)</th>
<th>Stress (MPa)</th>
<th>Plastic Strain (%)</th>
<th>Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>440</td>
<td>0</td>
<td>-100</td>
</tr>
<tr>
<td>0.6</td>
<td>680</td>
<td>-3.3</td>
<td>-790</td>
</tr>
<tr>
<td>2.5</td>
<td>740</td>
<td>-5.6</td>
<td>-930</td>
</tr>
<tr>
<td>7</td>
<td>830</td>
<td>-7.9</td>
<td>-990</td>
</tr>
<tr>
<td>10</td>
<td>880</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 4-1: Experimental setup for beamline G3 at the HASYLAB synchrotron.
4.2.3. Line Profile Analysis

The peak-profile analysis was performed using a Rietveld method with the Materials Analysis Using Diffraction (MAUD) software package [72]. Lutterotti and Gialanella [73] discuss the technique, which is founded upon the famous work of Warren [74]. The program receives the diffraction patterns from the standard (for the instrumental broadening) and the deformed specimen as inputs to perform the analysis. The method used here involved three parameters: the effective domain size, the sum of the intrinsic and extrinsic stacking-fault probabilities (SFP), and the twinning probability (TP). The primary relationship among these quantities is shown in Equation 4-1 [73].

$$\frac{1}{D_{\text{eff}}} = \frac{1}{D} + \left[1.5(\alpha' + \alpha''') + \beta'\right] \sum_b |L_0|, \quad \text{Equation 4-1}$$

where $D_{\text{eff}}$ is the effective domain size, $\alpha'$ is the intrinsic stacking-fault probability, $\alpha'''$ is the extrinsic stacking-fault probability, $h_0 = \sqrt{h^2 + k^2 + l^2}$, $u$ is the number of unbroadened components, $b$ is the number of broadened components, $L_0 = h + k + l$, $D$ is the true crystallite dimension, and $\beta'$ is the twinning probability.

The grain size in this case is large enough that $1/D$ is insignificant. On the other hand, $D_{\text{eff}}$ contains contributions from faulting, which can have a particle-size effect on the peak width [74]. Figure 4-2 shows that this approach improved upon the fit obtained using a Gaussian profile function. One goodness of fit criterion is

$$R_{wp} = \sqrt{\frac{(I_o - I_c)^2}{\sum I_o^2}}$$

(\text{where } I_o \text{ and } I_c \text{ are the observed and calculated intensities, respectively}), with a smaller value of $R_{wp}$ representing a better fit. For Figure 4-2, $R_{wp} = 0.03$ with MAUD and $R_{wp} = 0.06$ with a typical Gaussian fit. Specifically, the asymmetry
Figure 4-2: Mathematical fit of the peak profile using two methods.
in the peak was captured by the procedure in MAUD. The calibration sample gave a peak with a full-width-at-half-maximum (FWHM) of 0.059° 2θ, as indicated in Figure 4-2.

4.3. Results

4.3.1. Monotonic Tension

The line-profile analysis revealed subgrain deformation characteristics, such as SFP, TP, and $D_{\text{eff}}$. The SFP, which is presented in Figure 4-3, appreciably increased in the deformed specimens as compared to the undeformed specimen in the case of tensile deformation. No significant change in SFP was observed in the 0.6 and 2.5% specimens. A decrease in SFP from 0.087 ± 0.03 in the 2.5% specimen to 0.04 ± 0.01 in the 7% specimen was observed. The SFP then remained constant in the 10% specimen. The TP under tension, as shown in Figure 4-4, demonstrated no significant dependence on the plastic strain. Figure 4-5 indicates that a significant decrease in domain size was observed from 23 ± 6.9 nm in the reference specimen to 12 ± 3.6 nm in the specimen deformed to 0.6% tensile plastic strain. No significant change in domain size outside of the error bars was observed in subsequent specimens with increased plastic strains.

4.3.2. Monotonic Compression

Figure 4-6 shows that, during compressive deformation, the SFP increased from 0.01 ± 0.003 in the undeformed specimen to 0.04 ± 0.001 in the 3.3% specimen. The SFP, however, remained constant to within the uncertainty with further increase of compressive plastic strain. Figure 4-7 demonstrates that the TP appreciably increased
Figure 4-3: The stacking-fault probability as a function of tensile plastic strain.
Figure 4-4: The twinning probability as a function of tensile plastic strain.
Figure 4-5: Domain size as a function of tensile plastic strain.
Figure 4-6: Stacking-fault probability as a function of compressive strain.
Figure 4-7: Twinning probability as a function of compressive plastic strain.
from $0.023 \pm 0.008$ in the undeformed specimen to $0.055 \pm 0.02$ in the specimen deformed to 7.9% compressive plastic strain. $D_{\text{eff}}$ in Figure 4-8 11 showed no significant dependence upon compressive plastic strain.

4.4. Discussion

4.4.1. Subgrain Deformation Characteristics during Tensile Loading

Under tensile loading, small plastic strains (up to about 2%) were characterized by a large (relative to the undeformed state) stacking-fault density (Figure 4-3). However, the stacking-fault density decreased and subsequently remained constant during large plastic strains (~ 7% and greater). The transformation onset, as discussed in Section 3.2, is roughly at 2% plastic strain. Therefore, two regimes of plastic deformation can be identified based upon the knowledge gained from these experiments. Plastic strains up to 2% are accommodated by partial dislocation slip, which creates stacking faults in the FCC matrix. However, plastic strains over 2% are accommodated by the phase transformation, which consumes the stacking faults [37]

4.4.2. Subgrain Deformation Characteristics during Compressive Loading

The line-profile analysis in this work showed that the twinning probability increased during compressive deformation (Figure 4-7) but not tensile deformation (Figure 4-4). This observation can be explained by a careful consideration of deformation twinning. While slip can occur along either [ $\overline{1}10$ ] or [ $1\overline{1}0$ ], twining is a directional phenomenon. In fact, Hosford [41] showed that the twinning shear along [ $11\overline{2}$ ] is not equivalent to the shear along [ $\overline{1}\overline{1}2$ ]. Indeed, twinning in FCC crystals
Figure 4-8: Domain size as a function of compressive plastic strain.
involves a sudden shearing of the lattice on \{111\} planes in \(\langle 1 \overline{1} 2 \rangle\) directions and never in the opposite direction.

Figure 4-9 illustrates an FCC crystal undergoing tensile deformation and compressive deformation with the load applied along the \([001]\) direction. Figure 4-9(a) demonstrates that, during tension, the crystal must shear such that the top half displaces towards the left and the bottom half displaces to the right. This type of shear requires selection of the \([\overline{1} \overline{1} 2]\) direction. The situation changes when compression is considered [Figure 4-9(b)], since the top half of the crystal must now displace to the right and the required shear direction is \([1 1 \overline{2}]\). Therefore, the inherent directionality associated with twinning explains the observation that twinning is more favorable under compression than under tension.

4.5. Summary

This chapter focused on subgrain deformation characteristics studied by line-profile analysis. An interplay between stacking-fault accumulation and phase transformation was found to exist during monotonic tensile deformation. Plastic strains below 2~3\% were found to be accommodated by stacking-fault accumulation, while plastic strains above this value were characterized by the phase transformation. The phase transformation consumed the stacking faults, according to the results obtained here. The line-profile analysis showed that twinning was more probable during compressive deformation than in tensile deformation. Twinning has an inherently directional quality, as the twinning shear can only occur along the \([1 1 \overline{2}]\) directions. So, the twinning
Figure 4-9: Demonstration of the preferred shear direction for the cases of (a) tensile deformation and (b) compressive deformation.
shear is more favorable under compressive deformation. As reported in Chapter 3, the phase transformation was not favored under compressive deformation, so no dramatic interplay between faulting and the transformation was observed.
Chapter 5: Lattice-Strain Distribution and the Phase Transformation

5.1. Introduction

This chapter deals with the lattice-strain development of the FCC phase in a cobalt-based superalloy. A method is introduced for mathematically capturing the distribution of the strains in reciprocal space. As described in Section 2.2, the lattice strain is described as the sum of two contributions: the linear-elastic contribution and the intergranular contribution. The development of the intergranular strains can be considered in detail through the use of this analysis technique. While only the lattice strains of the FCC phase are measured, the analysis technique offers a method by which the load-sharing characteristics of the FCC and HCP phases can be inferred. The load partitioning was found to have consequences for the fatigue life of the alloy as a function of temperature. This analysis can be applied to any cubic material.

5.2. Experimental Procedure

5.2.1. Material

ULTIMET® alloy has a nominal composition in weight percent of 26Cr-9Ni-5Mo-3Fe-2W, with as-balance Co. The specimens used for the experiments considered in this chapter are shown in Figure 3-1 and Figure 3-2. The macroscopic modulus and 0.2% yield stress obtained from the in-situ loading diffraction experiment were 256 GPa and 525 MPa, respectively.
5.2.2. Lattice-Strain Measurement

The experimental data for this Chapter came from the in-situ loading experiments described in Section 3.2. Seven diffraction peaks were chosen for the calculation of strain: 111, 200, 220, 311, 331, 420, and 422. The scattering vectors of the two detector banks defined the two measured components of lattice strain (see Figure 1-5): (a) $\varepsilon_{33, hkl}$, parallel to the loading direction and (b) $\varepsilon_{11, hkl}$, perpendicular to the loading direction.

The positions of the peaks were determined with a Gaussian fit to the peak profile for the tensile, compressive, and high-cycle fatigue data. The reference d-spacing for the tensile and high-cycle fatigue experiments was obtained at a 20 MPa load before the specimen experienced any deformation. For the compression experiment, the reference was obtained at a 10 MPa compressive stress. The peak profiles of the low-cycle fatigue data sets were fit with a pseudo-Voigt function. The reference d-spacing for the low-cycle fatigue was obtained at a load of 0.

5.3. Theory

5.3.1. One-Dimensional Representation of the Strain

Current methods of presenting the distribution of lattice strains include a strain pole figure (where the strain of a given $hkl$ is represented in sample-orientation space, as in Figure 2-7) [60] and an inverse strain pole figure (where the strain component in a fixed specimen axis is represented in crystal-orientation space, as in Figure 2-8) [63]. The inverse strain pole figure is a two-dimensional representation of the lattice-strain distribution. Measurement of a complete inverse strain pole figure is impossible in this case, since the scattering vectors are fixed at 0 and 90° relative to the loading axis (see
Figure 1-5). Therefore, a simplified one-dimensional representation of the lattice-strain distribution was used for this work.

On the fundamental triangle, which is shown in Figure 5-1, \( A_{hkl} \) (see Equation 2-2) varies from 0 to 1/3 along two paths. In the elastic regime of deformation, these paths are equivalent (i.e., the dependency of \( E_{hkl} \) on \( A_{hkl} \) along the two paths is the same). In general, however, the two paths are not necessarily equivalent. Therefore, the quantity \( A_{hkl}^* \), which is formally defined in Equation 5-1, accounts for the general case by assigning \( + A_{hkl} \) to Path 1 and \( - A_{hkl} \) to Path 2 in Figure 5-1:

\[
A_{hkl}^* = \begin{cases} 
A_{hkl} \; & \text{if } [hkl] \in [11n] \\
- A_{hkl} \; & \text{if } [hkl] \in [10n] \text{ or } [h1n] 
\end{cases}
\]

Equation 5-1

Axial lattice strains measured at tensile macroscopic loads of 300 MPa and 890 MPa (before and after yielding, respectively) are plotted in this manner in Figure 5-2. The distribution is symmetric about \( A_{hkl}^* = 0 \) in the elastic regime, but the symmetry clearly ceases after the onset of macroscopic yielding.

5.3.2. The Elastic Compliances and the Modified Kröner Model

The first step in determining the linear-elastic contribution was to identify the appropriate \( hkl \)-dependent compliances to use in the calculations. In the macroscopically elastic regime of deformation, a uniaxial stress state is a valid assumption for the in-situ diffraction experiment. Therefore, the two components of the linear-elastic contribution (\( \varepsilon_{33,hkl}^e \) and \( \varepsilon_{11,hkl}^e \), respectively) depend upon the applied macroscopic load, \( \sigma^M \), as follows [75]:

\[
\]
Figure 5-1: The fundamental triangle demonstrating that $A_{hkl}$ varies from 0 to 1/3 along two paths.
Figure 5-2: One-dimensional representation of the lattice strains of ULTIMET® alloy at tensile stresses of 300 and 890 MPa.
\[ e^e_{33,\text{hkl}} = \sigma^M / E_{\text{hkl}} \]  \hspace{1cm} \text{Equation 5-2}

\[ e^e_{11,\text{hkl}} = -\left(v_{\text{hkl}} / E_{\text{hkl}}\right)\sigma^M . \]  \hspace{1cm} \text{Equation 5-3}

Where \( E_{\text{hkl}} \) and \( \nu_{\text{hkl}} \) are the diffraction elastic modulus and diffraction Poisson’s ratio, respectively. \( 1/E_{\text{hkl}} \) and \( \nu_{\text{hkl}} / E_{\text{hkl}} \) are measured from the slopes of the hkl-dependent lattice-strain vs. applied-stress curves. Further, they are related to the diffraction elastic constants, \( s_{1,\text{hkl}} \) and \( s_{2,\text{hkl}} \), through Equations 5-4 and 5-5 [76]:

\[ 1/E_{\text{hkl}} = s_{1,\text{hkl}} + \frac{1}{2} s_{2,\text{hkl}} \]  \hspace{1cm} \text{Equation 5-4}

\[ -\nu_{\text{hkl}} / E_{\text{hkl}} = s_{1,\text{hkl}} . \]  \hspace{1cm} \text{Equation 5-5}

The measured compliances (i.e., \( 1/E_{\text{hkl}} \) and \( \nu_{\text{hkl}} / E_{\text{hkl}} \)) correspond to only seven specific values of \( A_{\text{hkl}} \), which is defined in Equation 2-2. For the analysis it is necessary to obtain the compliance values as a continuous function of \( A_{\text{hkl}} \). This continuous function is obtained through consideration of DeWit’s [77] modification of the Kröner model. The original Kröner model was developed to determine the isotropic modulus of a polycrystal from the single-crystal elastic constants [78]. His method was initially based upon Eshelby theory [55], and the result obtained from his method is commonly termed the “self-consistent” value.

According to DeWit [77], the diffraction elastic constants are related to the bulk modulus, \( K \), and the diffraction shear modulus, \( G_{\text{hkl}} \), through Equations 5-6 and 5-7:

\[ s_{1,\text{hkl}} = 1/(9K) - 1/(6G_{\text{hkl}}) \]  \hspace{1cm} \text{Equation 5-6}

\[ \frac{1}{2} s_{2,\text{hkl}} = 1/(2G_{\text{hkl}}) . \]  \hspace{1cm} \text{Equation 5-7}
Finally, DeWit’s [77] modified version of the Kröner model relates $s_{1,hkl}$ and $s_{2,hkl}$ from powder data to the single-crystal elastic constants, $c_{ij}$, as shown in Equation 5-8:

$$G_{hkl}^3 + \alpha_{hkl}G_{hkl}^2 + \beta_{hkl}G_{hkl} + \gamma = 0.$$  
Equation 5-8

The coefficients $\alpha_{hkl}$, $\beta_{hkl}$, and $\gamma$ are defined in Equations 5-9 through 5-11:

$$\alpha_{hkl} = (3/8)[3K + 4(\mu + 3(\eta - \mu)A_{hkl})] - (1/5)(2\eta + 3\mu)$$  
Equation 5-9

$$\beta_{hkl} = (3/4)K[\mu + 3(\eta - \mu)A_{hkl}] - (3/40)(6K\eta + 9K\mu + 20\eta\mu)$$  
Equation 5-10

$$\gamma = -3K\mu\eta/4.$$  
Equation 5-11

where $2\eta = c_{11} - c_{12}$, $3K = c_{11} + 2c_{12}$, and $\mu = c_{44}$. The orientation parameter, $A_{hkl}$, is defined by Equation 2-2. Now, through De Wit’s [77] modified Kröner model, one can obtain the elastic compliances as a continuous function of $A_{hkl}$. This method is also discussed by Gnäupel-Herold et. al [76].

5.3.3. The Linear-Elastic Contribution

Inhomogeneities, other than intergranular strains, that bias the lattice strains can develop during deformation. One example would be a fatigue crack. The effect of these additional inhomogeneities on the lattice strains can be captured by relaxing the assumption of a uniaxial stress state. Therefore, this analysis method utilized Hooke’s law with a triaxial stress state and cylindrical symmetry (i.e., $\sigma_{11} = \sigma_{22} \neq 0$ and $\sigma_{33} \neq 0$), as in:

$$\varepsilon_{33,hkl} = \frac{1}{E_{hkl}}[(\sigma_{33} - \sigma_{\text{ref}}) - 2\nu_{hkl}\sigma_{11}].$$  
Equation 5-12

$$\varepsilon_{11,hkl} = \varepsilon_{22,hkl} = \frac{1}{E_{hkl}}[(1 - \nu_{hkl})\sigma_{11} - \nu_{hkl}(\sigma_{33} - \sigma_{\text{ref}})].$$  
Equation 5-13
where \( \sigma_{\text{ref}} \), the stress at which the reference d-spacing was determined, must be subtracted from \( \sigma_{33} \).

The values of \( E_{\text{hkl}} \) and \( \nu_{\text{hkl}} \) used in Equations 5-12 and 5-13 are those determined from the modified Kröner model, as discussed in Section 5.3.2. In order to capture the effect of the aforementioned inhomogeneities, values of the \( \sigma_{33} \) and \( \sigma_{11} \) are fit to the data within the context of the overall strain analysis. These values are termed the “effective stresses”, \( \sigma_{33,\text{eff}} \) and \( \sigma_{11,\text{eff}} \).

5.3.4. Plasticity-Induced Intergranular Contribution

Bunge [79] showed that the even-order spherical harmonics can be expressed in terms of linear combinations of homogeneous polynomials of fourth and sixth degree. The fourth degree polynomial is the familiar \( A_{\text{hkl}} \) (Equation 2-2), while the sixth degree polynomial is \( C_{\text{hkl}} \) in Equation 5-14:

\[
C_{\text{hkl}} = \frac{h^2 k^2 l^2}{(h^2 + k^2 + l^2)^3}
\]  

Equation 5-14

The linear combinations of the fourth and sixth degree polynomials result in the octahedral harmonic polynomials, \( k_{2j+2} \) (where \( j \) is an integer such that \( 1 \leq j \leq 4 \)), which are shown in Equations 5-15 through 5-18 [79]:

\[
k_4 = 0.64636 \times (5A_{\text{hkl}} - 1)
\]  

Equation 5-15

\[
k_6 = 0.359601 \times (231C_{\text{hkl}} - 21A_{\text{hkl}} + 2)
\]  

Equation 5-16

\[
k_8 = 0.835193 \times (52C_{\text{hkl}} - 65A_{\text{hkl}}^2 + 18A_{\text{hkl}} - 1)
\]  

Equation 5-17

\[
k_{10} = -0.265929 \times (3553A_{\text{hkl}}C_{\text{hkl}} - 979C_{\text{hkl}} - 187A_{\text{hkl}}^2 + 55A_{\text{hkl}} - 2)
\]  

Equation 5-18
The intergranular contribution to the lattice strain, \( \varepsilon_{33,hl}^I \), can be represented by the \( k_{2j+2} \) according to Equation 5-19:

\[
\varepsilon_{33,hl}^I = \varepsilon_a^I P(A_{hkl}, C_{hkl}) = \varepsilon_a^I \sum_j \beta_j k_{2j+2} (A_{hkl}, C_{hkl}). \tag{5-19}
\]

Where \( \varepsilon_a^I \) is the *intergranular-strain amplitude*, \( P(A_{hkl}, C_{hkl}) \) is a polynomial representing the sum of the \( k_{2j+2} \), and \( \beta_j \) are fit to the experimental lattice-strain data. The intergranular-strain amplitude, which in fact corresponds to the intergranular strain along <100>, is a parameter used to scale the intergranular strains from one load level to the next. The expression in Equation 5-19 can also be used to describe \( \varepsilon_{11,hl}^I \).

5.3.5. Overview of the Parameters

The final fitting routine involved a total of \( 4n+6 \) fitting parameters for \( 14n \) data points, where \( n \) is the number of load levels (or, fatigue cycles) considered in the measurement. There were \( n \) values each for \( \sigma_{11,\text{eff}} \) and \( \sigma_{33,\text{eff}} \) from the linear-elastic contribution. The intergranular contribution was described by four coefficients \( \beta_j \): \( \beta_1 \) through \( \beta_3 \) were free parameters. \( \beta_4 \) was constrained by the condition \( P(0,0) = 1 \), as in Equation 5-20:

\[
\beta_4 = (1 + 0.64636 \beta_1 - 0.719202 \beta_2 + 0.835193 \beta_3) / 0.531858. \tag{5-20}
\]

While the \( k_{2j+2} \) accurately described the shape of the intergranular contribution, the scale factor \( \varepsilon_a^I \) accounted for the fact that the strains were increasing with the applied load. In summary, there were \( 2n \) effective stresses, six \( \beta_j \) (one set of three for each strain component), and \( 2n \) intergranular-strain amplitudes (one set of \( n \) for each strain component) that were fit to the entire lattice-strain data set (including each load level or
fatigue cycle, each $hkl$, and each strain component).

5.4. Results

5.4.1. Measured Lattice Strain

The axial lattice strains measured during the monotonic tension experiment are shown against the applied stress in Figure 5-3 with the commonly-used representation of $hkl$-dependent stress-strain curves. Figure 5-3(a) shows the lattice strains for the 111, 200, 220, and 311 diffraction peaks, while Figure 5-3(b) shows the strains for the 331, 420, and 422 diffraction peaks (the lattice strains for the remaining data sets will be presented in this manner). The axial lattice strains were tensile, with the strain along $\langle 200 \rangle$ exhibiting the largest magnitude. The transverse strains for the tensile experiment are shown in Figure 5-4. These strains were compressive, reflecting the expected Poisson contraction. Elastic and plastic anisotropy was evident in both the axial and transverse strains, since strains of different $hkl$ exhibited different behavior with respect to the applied load. The also exhibited intergranular-strain development, since significant deviations from linearity were observed.

The axial lattice strains from the monotonic compression test are shown in Figure 5-5. The axial strains were compressive, with the strain along $\langle 200 \rangle$ exhibiting the largest magnitude. The transverse lattice strains are displayed in Figure 5-6. These strains were tensile because of the expansion in the Poisson direction. Elastic anisotropy was evident, since the strains of different $hkl$ exhibited different behavior with respect to the applied load. The deviations from linearity were not as demonstrative during compression as they were during tension.
Figure 5-3: Measured axial lattice strains from the monotonic tension experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.
Figure 5-4: Transverse lattice strains from the monotonic tensile experiment: (a) for 111, 200, 220, and 311 and (b) 331, 420, 422.
Figure 5-5: Axial lattice strains from the monotonic compression experiment: (a) 111, 200, 220, and 311 and (b) 331, 420, and 422.
Figure 5-6: Transverse lattice strains from the monotonic compression experiment: (a) 111, 200, 220, and 311 and (b) 331, 420, and 422.
Figure 5-7 displays the axial lattice strains from the high-cycle fatigue experiment. These strains were obtained at the maximum stress and are shown as a function of fatigue cycles. The lattice strains obtained at the minimum stress of each fatigue cycle are shown in Appendix A. The transverse lattice strains obtained at the maximum stress are displayed in Figure 5-8. The lattice strains quickly saturated to an equilibrium value during high-cycle fatigue, with the strain along <200> exhibiting the largest magnitude.

Figure 5-9 shows the axial lattice strains obtained at the maximum tensile strain during low-cycle fatigue. The corresponding transverse lattice strains are presented in Figure 5-10. As indicated in Figure 3-5, lattice strain data is also available for the maximum compressive strain (point 4), two zero-stress points (points 2 and 5), and two zero-strain points (points 3 and 6). The data for these cases are shown in Appendix B. The lattice strains during low-cycle fatigue showed some small increase in first 12 fatigue cycles, but they subsequently saturated to an equilibrium value. Again, the strain along <200> demonstrated the highest magnitude.

5.4.2. Elastic Compliances

The measured elastic compliances were analyzed according to the method described in Section 5.3.2. For the tension experiment, the resulting best-fit values for the single-crystal elastic stiffnesses were $c_{11} = 0.257 \times 10^{12}$, $c_{12} = 0.155 \times 10^{12}$, and $c_{44} = 0.147 \times 10^{12}$ Pa. Table 5-1 shows the measured compliances and the compliances back-calculated from the best-fit $c_{ij}$ for the tension experiment. Figure 5-11 shows the measured compliances together with the calculated curve from the modified Kröner model. The corresponding table and figure for the compression case are presented in
Figure 5-7: Axial lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.
Figure 5-8: Transverse lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.
Figure 5-9: Axial lattice strains obtained at the maximum strain from the low-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.
Figure 5-10: Transverse lattice strains obtained at the maximum tensile strain from the low-cycle fatigue experiment: (a) for 111, 200, 220, and 311 and (b) for 331, 420, and 422.
Table 5-1: Measured and calculated compliances for the tension experiment.

<table>
<thead>
<tr>
<th>hkl</th>
<th>Ahkl</th>
<th>measured</th>
<th>error</th>
<th>calculated</th>
<th>measured</th>
<th>error</th>
<th>calculated</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>0.000</td>
<td>5.26</td>
<td>0.06</td>
<td>5.25</td>
<td>-1.64</td>
<td>0.06</td>
<td>-1.74</td>
</tr>
<tr>
<td>311</td>
<td>0.157</td>
<td>4.34</td>
<td>0.08</td>
<td>4.26</td>
<td>-1.29</td>
<td>0.04</td>
<td>-1.25</td>
</tr>
<tr>
<td>420</td>
<td>0.160</td>
<td>4.34</td>
<td>0.20</td>
<td>4.24</td>
<td>-1.21</td>
<td>0.11</td>
<td>-1.24</td>
</tr>
<tr>
<td>220</td>
<td>0.250</td>
<td>3.72</td>
<td>0.06</td>
<td>3.76</td>
<td>-1.05</td>
<td>0.04</td>
<td>-1.00</td>
</tr>
<tr>
<td>422</td>
<td>0.250</td>
<td>3.60</td>
<td>0.18</td>
<td>3.76</td>
<td>-0.88</td>
<td>0.18</td>
<td>-1.00</td>
</tr>
<tr>
<td>331</td>
<td>0.274</td>
<td>3.58</td>
<td>0.11</td>
<td>3.64</td>
<td>-0.97</td>
<td>0.09</td>
<td>-0.94</td>
</tr>
<tr>
<td>111</td>
<td>0.333</td>
<td>3.39</td>
<td>0.07</td>
<td>3.37</td>
<td>-0.73</td>
<td>0.05</td>
<td>-0.80</td>
</tr>
</tbody>
</table>
Figure 5-11: Measured compliances and the curve generated from the modified Kröner model.
Table 5-2 and Figure 5-12, respectively. The calculated values matched measured values quite well for both cases.

5.4.3. Strain Analysis

The analysis set forth in Sections 5.3.3 and 5.3.4 was applied to each of the data sets discussed in Section 5.4.1. Figure 5-13 shows the measured and calculated lattice strains at 300 MPa, 890 MPa, and after unloading for the tensile experiment. The calculated curves match the measured data well in both the elastic and plastic regimes of deformation and upon releasing the stress.

The difference between the axial effective stress and the applied stress was a useful quantity, since this difference revealed an effect of the phase transformation on the lattice strains. The transverse effective stress was considered on its own, since the applied transverse stress was zero. Hence, the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ is shown in Figure 5-14(a), and $\sigma_{11,\text{eff}}$ is shown in Figure 5-14(b). The axial effective stress agreed with the applied stress until 720 MPa was reached. Then, it increased up to 205 MPa, with a decrease upon unloading to 74 MPa. The transverse effective stress was zero until 720 MPa, after which it increased to 130 MPa. The transverse effective stress remained constant at roughly 130 MPa as the stress was released. The intergranular-strain amplitude is shown versus plastic strain in Figure 5-15. The amplitude demonstrated a power-law dependence upon plastic strain in two regimes. The first regime was between 0.0013 and 0.0019 mm/mm, and the second regime was between 0.02 and 0.08 mm/mm. The transverse amplitude increased with plastic strain as a power law until 0.0019 mm/mm. Subsequently, it saturated at a value of approximately 500 µε. Upon
Table 5-2: Measured and calculated compliances for the compression experiment.

<table>
<thead>
<tr>
<th>hkl</th>
<th>Ahkl</th>
<th>1/E (10^{12} \text{ Pa}^{-1})</th>
<th>\text{measured}</th>
<th>\text{error}</th>
<th>\text{calculated}</th>
<th>-\nu/E (10^{12} \text{ Pa}^{-1})</th>
<th>\text{measured}</th>
<th>\text{error}</th>
<th>\text{calculated}</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>0.000</td>
<td>5.33</td>
<td>0.06</td>
<td>5.42</td>
<td>-1.84</td>
<td>0.06</td>
<td>-1.88</td>
<td></td>
<td></td>
</tr>
<tr>
<td>311</td>
<td>0.157</td>
<td>4.39</td>
<td>0.08</td>
<td>4.28</td>
<td>-1.29</td>
<td>0.04</td>
<td>-1.31</td>
<td></td>
<td></td>
</tr>
<tr>
<td>420</td>
<td>0.160</td>
<td>4.37</td>
<td>0.20</td>
<td>4.26</td>
<td>-1.26</td>
<td>0.11</td>
<td>-1.30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>220</td>
<td>0.250</td>
<td>3.69</td>
<td>0.06</td>
<td>3.72</td>
<td>-1.07</td>
<td>0.04</td>
<td>-1.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>422</td>
<td>0.250</td>
<td>3.83</td>
<td>0.18</td>
<td>3.72</td>
<td>-0.96</td>
<td>0.18</td>
<td>-1.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>331</td>
<td>0.274</td>
<td>3.67</td>
<td>0.11</td>
<td>3.58</td>
<td>-1.06</td>
<td>0.09</td>
<td>-0.96</td>
<td></td>
<td></td>
</tr>
<tr>
<td>111</td>
<td>0.333</td>
<td>3.25</td>
<td>0.07</td>
<td>3.29</td>
<td>-0.82</td>
<td>0.05</td>
<td>-0.82</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Figure 5-12: Measured compliances and the calculated curve from the modified Kröner model for the compression data.
Figure 5-13: Measured and calculated lattice strains for the tensile experiment: (a) axial data and (b) transverse data.
Figure 5-14: The effective stresses from the strain analysis: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma_M$ and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-15: The intergranular strain amplitude from the tensile experiment: (a) axial data and (b) transverse data.
unloading, the intergranular-strain amplitude decreased only slightly.

For the compression experiment, material flow beyond the compression platens caused the lattice strains to be invalid at high compressive stresses. Therefore, the data was only considered up to -600 MPa. The measured and calculated lattice strains at -300 MPa, -500 MPa, and -600 MPa for the compression experiment are shown in Figure 5-16. For the case of compressive loading, the analysis method fit the data well in all regimes of deformation. The appropriate effective stress quantities are presented in Figure 5-17. The axial effective stress and the applied stress closely agreed with each other, within the uncertainty of the calculation. The transverse effective stress remained at roughly zero up to -600 MPa. The intergranular-strain amplitude from the compression test is shown as a function of plastic strain in Figure 5-18. The axial amplitude demonstrated a linear dependence on plastic strain. The transverse amplitude, however, remained constant at roughly zero.

The measured and calculated lattice strains during high-cycle fatigue for cycles 1, 1,005, and 18,009 are shown in Figure 5-19 (at the maximum stress) and Figure 5-20 (at the minimum stress). The calculated curves fit the data well for the case of high-cycle fatigue. The appropriate effective stress quantities are shown as a function of fatigue cycles in Figure 5-21 (at the maximum stress) and Figure 5-22 (at the minimum stress). At the maximum stress, the axial effective stress deviated from the applied stress by about 150 MPa throughout the fatigue life. The magnitude of the transverse effective stress was around 75 MPa for the entire fatigue life. At the minimum stress, these respective values were 0 and 45 MPa. The intergranular-strain amplitudes are shown in Figure 5-23 (at the maximum stress) and Figure 5-24 (at the minimum stress). The
Figure 5-16: Measured and calculated lattice strains from the compression experiment: (a) axial data and (b) transverse data.
Figure 5-17: The effective stresses from the compression experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and sM and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-18: The intergranular-strain amplitude from the compression experiment: (a) axial data and (b) transverse data.
Figure 5-19: Measured and calculated lattice strains at the maximum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-20: Measured and calculated lattice strains at the minimum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-21: Effective stresses at the maximum stress from the high-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $s_M$ and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-22: Effective stresses at the minimum stress from the high-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $s_M$ and (b) $\sigma_{1,\text{eff}}$. 

Figure (a) shows the difference between $\sigma_{33,\text{eff}}$ and $s_M$ for ULTIMET® Alloy high-cycle fatigue. The plot displays a trend where the effective stress decreases as the applied stress increases, indicating a decrease in fatigue resistance with increasing stress levels. The error bars suggest a degree of variability in the measurements.

Figure (b) presents $\sigma_{33,\text{eff}}$ for the same alloy and fatigue condition. The plot similarly shows a decrease in effective stress with increasing applied stress, with error bars indicating variability. The high-cycle fatigue experiment highlights the importance of stress levels in determining the material's fatigue life.
Figure 5-23: The intergranular-strain amplitudes at the maximum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-24: The intergranular-strain amplitude at the minimum stress from the high-cycle fatigue experiment: (a) axial data and (b) transverse data.
amplitudes for all cases saturated quickly as a function of fatigue cycles. At the maximum stress, the axial and transverse amplitudes saturated at 1,200 and 900 µε, respectively. At the minimum stress, the axial and transverse amplitudes saturated at 500 and 400 µε, respectively.

The measured and calculated lattice strains during low-cycle fatigue for cycles 1, 75, and 500 are shown in Figure 5-25 through Figure 5-30. These figures respectively correspond to points 1 through 6 in Figure 3-5: i.e., the maximum tensile strain (point 1), the tensile zero-strain point (point 2), the compressive zero-stress point (point 3), the maximum compressive strain (point 4), the compressive zero-strain point (point 5), and the tensile zero-stress point (point 6). The calculated curves fit the data well for all six of the cases considered. The appropriate effective stress quantities are respectively shown against fatigue cycles in Figure 5-31 through Figure 5-36 for the six cases. The quantities \( \sigma_{33,\text{eff}} - \sigma^M \) and \( \sigma_{11,\text{eff}} \) increased with fatigue cycles until cycle 100 and then saturated. The saturation values for the six respective cases were \( \sigma_{33,\text{eff}} - \sigma^M = 200 \text{ MPa and } \sigma_{11,\text{eff}} = 180 \text{ MPa} \), \( \sigma_{33,\text{eff}} - \sigma^M = 150 \text{ MPa and } \sigma_{11,\text{eff}} = 225 \text{ MPa} \), \( \sigma_{33,\text{eff}} - \sigma^M = 100 \text{ MPa and } \sigma_{11,\text{eff}} = 175 \text{ MPa} \). The intergranular-strain amplitudes are respectively presented in Figure 5-37 through Figure 5-42 for the six cases. Perhaps the most notable trend in these Figures is the fact that the transverse amplitude was zero for the compressive and tensile zero-strain points. In general, however, a slight dependence of the amplitude on fatigue cycles was notice for the first 100 cycles, after which a saturation value was reached. The saturation values for the axial amplitudes
Figure 5-25: Measured and calculated lattice strains at the maximum tensile strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-26: Measured and calculated lattice strains at tensile zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-27: Measured and calculated lattice strains at compressive zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-28: Measured and calculated lattice strains at the maximum compressive stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-29: Measured and calculated lattice strains at compressive zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-30: Measured and calculated lattice strains at tensile zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-31: Effectives stresses at the maximum tensile strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-32: Effective stresses at tensile zero stress from the low-cycle fatigue experiment: (a) the difference between $\sigma_{3,\text{eff}}$ and $\sigma_M$ and (b) $\sigma_{1,\text{eff}}$. 
Figure 5-33: Effective stresses at compressive zero strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{1,\text{eff}}$. 
Figure 5-34: Effective stresses at the maximum compressive strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-35: Effective stresses at compressive zero stress from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33,\text{eff}}$ and $\sigma^M$ and (b) $\sigma_{11,\text{eff}}$. 
Figure 5-36: Effective stresses at tensile zero strain from the low-cycle fatigue experiment: (a) the difference between $\sigma_{33, eff}$ and $\sigma^M$ and (b) $\sigma_{1, eff}$.
Figure 5-37: Intergranular-strain amplitude at the maximum tensile strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-38: Intergranular-strain amplitude at tensile zero stress from the low-cycle fatigue: (a) axial data and (b) transverse data.
Figure 5-39: Intergranular-strain amplitude at compressive zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-40: Intergranular-strain amplitude at the maximum compressive strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-41: Intergranular-strain amplitude at compressive zero stress from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
Figure 5-42: Intergranular-strain amplitude at tensile zero strain from the low-cycle fatigue experiment: (a) axial data and (b) transverse data.
were 700, 500, -200, -400, -300, and 500 µε for the six respective cases. The saturation values for the transverse amplitudes were 450, 400, 0, -200, 0, and 300 µε for the six respective cases.

5.5. Discussion

5.5.1. Intergranular Strains and Plastic Deformation

Figure 5-15 indicates that the intergranular strains were correlated with the tensile plastic deformation. The two regimes noted in Figure 5-15 correspond to two stages of hardening that are commonly observed in materials that follow a power law. The first stage is characterized by large changes in stress and small changes in strain, and the second stage is characterized by small changes in stress and large changes in strain. The intergranular strains show very sharp changes with plastic strain during the first regime and more gradual changes with plastic strain during the second regime, as Figure 5-15 indicates. For this particular material, these two regimes of plastic deformation are also separated by stacking-fault accumulation and phase transformation, as discussed in Section 4.4.1 (also see Figure 4-3). The phase transformation accommodates the plastic deformation during the second regime, when strain accumulates most rapidly with applied stress. This fact could attribute to the decreased dependency of the intergranular strains on plastic strain noted in Figure 5-15 at plastic strains greater than 0.02 mm/mm.

For the case of compressive deformation, the intergranular strains seem to develop more slowly when compared to the tension case. For example, the magnitudes of the axial amplitudes at 600 MPa were 542 µε for the tension case and 378 µε for the compression case. The respective magnitudes of the transverse amplitudes were 352 µε
and 0. However, the plastic strain developed more slowly during the compressive case, as well. So, regardless of whether tensile or compressive deformation is applied, the intergranular strains are inherently dependent upon the plastic strain.

The dependence of the intergranular strains on plastic strain should not change for the fatigue cases. Indeed, most of the plastic strain occurs during the first fatigue cycle during high-cycle fatigue with little accumulation during subsequent fatigue cycles, as discussed in light of the phase-transformation characteristics in Section 3.4.3. So, the intergranular strains quickly reach a saturation value during high-cycle fatigue, as observed in Figure 5-23 and Figure 5-24.

For the case of low-cycle fatigue, the biggest changes to the intergranular strains occur within the fatigue cycle. It is within a fatigue cycle, in fact, that the most noticeable changes in plastic strain are occurring, especially when one considers reverse plasticity during the compressive half-cycle. This fact is demonstrated in Figure 5-43, where the amplitude has been plotted against strain during fatigue cycle 75. The axial amplitude started at 650 µε at the maximum tensile strain. It decreased to -400 µε at the maximum compressive strain. Finally, the axial amplitude increased to 500 µε as the fatigue cycle ended. The transverse amplitude also demonstrated a similar loop.

Even though most of the changes in the amplitude are expected within a fatigue cycle, some dependence of the intergranular strains on stress was noted in Figure 5-15. So, cyclic hardening, which occurs during the first 12 fatigue cycles [6], could cause some dependence of the intergranular strains on fatigue cycles. By and large, however, the intergranular strains should saturate as a function of fatigue cycles, since the strain is controlled during low-cycle fatigue.
Figure 5-43: The intergranular-strain amplitudes during fatigue cycle 75: (a) axial data and (b) transverse data.
5.5.2 Load Sharing between the FCC and HCP Phases

The effective stresses, $\sigma_{33,\text{eff}}$ and $\sigma_{11,\text{eff}}$, represent the stress on the FCC phase required to correctly describe the measured lattice strain, after accounting for intergranular strains. If the effective stresses do not match the applied values (i.e., $\sigma_{33,\text{eff}} \neq \sigma^M$ or $\sigma_{11,\text{eff}} \neq 0$), then loading sharing is occurring and the phase transformation is biasing the measured lattice strain. The load partitioning between the FCC and HCP phases can be determined by considering stress-balance requirements, as in:

$$f_{\text{HCP}}\sigma_{\text{HCP}} + f_{\text{FCC}}\sigma_{\text{FCC}} = \sigma_{\text{applied}} \quad \text{Equation 5-21}$$

where $f_{\text{HCP}}$ is the weight fraction of HCP, $\sigma_{\text{HCP}}$ is the stress in the HCP phase, $f_{\text{FCC}}$ is the weight fraction of FCC, $\sigma_{\text{FCC}}$ is the stress in the FCC phase, and $\sigma_{\text{applied}}$ is the applied stress [75,80]. The applied stress is known, the stress experienced by the FCC phase is the effective stress, and the phase fractions are known. Therefore, the stress experienced by the HCP phase can be calculated. Equation 5-21 can be applied to the axial and transverse stress components.

The stress partitioning during monotonic tensile deformation is shown in Figure 5-44. The stress in the HCP is negative for both the axial and transverse components at the first observation of the HCP peak. When the stress increases from 720 to 890 MPa, the axial stress increases to around +100 MPa. Upon release of the macroscopic load, the axial stress becomes compressive again. The transverse stress in the HCP phase, however, seems to remain roughly constant at around -600 to -700 MPa, considering the confidence bounds of the calculation. When both stress components are compressive, the HCP phase is under hydrostatic compression. This is an indication that the HCP phase
Figure 5-44: Stress partitioning for tensile deformation: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
is constrained on all sides by the FCC matrix. In return, the FCC phase experiences an additional tensile stress.

The stress partitioning during high-cycle fatigue is shown in Figure 5-45 and Figure 5-46 for the maximum and minimum stresses, respectively. At the maximum stress, the axial HCP stress begins at -100 MPa and quickly increases with fatigue cycles to 200 MPa. The transverse stress in the HCP stays constant at roughly -400 MPa with increasing fatigue cycles. At the minimum stress, the stress in HCP is more or less zero, although the trends are noisy. The transverse stress remains constant at -200 MPa with increasing fatigue cycles. So, the hydrostatic stress state observed during the tensile case seems to disappear during high-cycle fatigue.

The stress partitioning during low-cycle fatigue is shown in Figure 5-47 through Figure 5-52 for the six respective cases noted in Figure 3-5. The FCC stress in all six cases show a dependency on cycles for the first 100 fatigue cycles and reaches a saturation value. The saturation values of the FCC stress for the six cases are: \( \sigma_{33,FCC} = 750 \) MPa and \( \sigma_{11,FCC} = 20 \) MPa, \( \sigma_{33,FCC} = 100 \) MPa and \( \sigma_{11,FCC} = 250 \) MPa, \( \sigma_{33,FCC} = -250 \) MPa and \( \sigma_{11,FCC} = 250 \) MPa, \( \sigma_{33,FCC} = -500 \) MPa and \( \sigma_{11,FCC} = 250 \) MPa, \( \sigma_{33,FCC} = 250 \) MPa and \( \sigma_{11,FCC} = 250 \) MPa, \( \sigma_{33,FCC} = 250 \) MPa and \( \sigma_{11,FCC} = 200 \) MPa, respectively.

The saturation values of the HCP stress for the six cases are: \( \sigma_{33,HCP} = -250 \) MPa and \( \sigma_{11,HCP} = -750 \) MPa, \( \sigma_{33,HCP} = -500 \) MPa and \( \sigma_{11,HCP} = -750 \) MPa, \( \sigma_{33,HCP} = -1,000 \) MPa and \( \sigma_{11,HCP} = -750 \) MPa, \( \sigma_{33,HCP} = -1,250 \) MPa and \( \sigma_{11,HCP} = -750 \) MPa, \( \sigma_{33,HCP} = -750 \) MPa and \( \sigma_{11,HCP} = -500 \) MPa, respectively. Therefore, for the case of low cycle fatigue, the hydrostatic stress on the HCP phase
Figure 5-45: Stress partitioning at the maximum stress from the high-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
Figure 5-46: Stress partitioning at the minimum stress from the high-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
Figure 5-47: Stress partitioning at the maximum tensile strain from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
Figure 5-48: Stress partitioning at tensile zero stress from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
Figure 5-49: Stress partitioning at compressive zero strain from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$.
Figure 5-50: Stress partitioning at the maximum compressive strain from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 

([Graph showing stress partitioning])
Figure 5-51: Stress partitioning at compressive zero stress from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
Figure 5-52: Stress partitioning at tensile zero strain from the low-cycle fatigue experiment: (a) $\sigma_{33}$ and (b) $\sigma_{11}$. 
never relaxes. During tension, the hydrostatic stress state relaxed after the stress increased to 890 MPa. The macroscopic stress never exceeds 700 MPa, so the hydrostatic stress state cannot relax during low-cycle fatigue. As a consequence, the FCC matrix experienced an additional tensile stress.

5.5.3. Consequences for the Fatigue Life

One interesting aspect of ULTIMET<sup>®</sup> superalloy is the anomalous fatigue life as a function of temperature. Figure 1-2 shows that the fatigue life at 873 K is longer than at room temperature. Generally, the fatigue life is expected to decrease with increasing temperature, as environmental effects become significant. Jiang et al. [6] note that the HCP phase forms at both 298 K and 873 K, which affirms the results discussed in Section 3.4.5. They suggest that the difference in the fatigue life at 298 and 873 K is due to the fact that the ductility of the material is much greater at the higher temperature, as shown in Figure 1-1. The results of this analysis, however, offer more insight into this issue. The introduction of the HCP phase is detrimental to the fatigue life, since it introduces a tensile stress on the FCC matrix. The tensile stress is favorable conditions for fatigue-crack growth. The HCP phase forms later at 873 K than at room temperature, as illustrated by Figure 3-10 and Figure 3-11. So, the fatigue life is longer at 873 K than room temperature. The fatigue behavior at 1,173 K decreases because of the expected environmental effects, as discussed by Jiang et al. [6].
5.6. Summary

This chapter focused on the lattice-strain development in the cobalt-based superalloy. A method was described by which the anisotropic distribution of the lattice strains in reciprocal space can be mathematically described. The total lattice strain was considered to be the sum of two contributions: the linear-elastic contribution and the intergranular contribution. The linear-elastic contribution was described by Hooke’s law, and the intergranular contribution was described by a polynomial expansion of spherical harmonics. The calculations from the analysis method accurately fit the lattice-strain data measured during monotonic tension and compression, as well as during high-cycle and low-cycle fatigue.

The intergranular-strain development during the different loading modes was considered by consideration of the intergranular-strain amplitudes. In general, the intergranular strains were found to be dependent upon the plastic strain. The load partitioning between the FCC and HCP phases was calculated through stress-balance requirements and the effective stresses. The HCP phase was found to form under a state of hydrostatic compression, which placed the FCC matrix in a state of tension above and beyond the applied macroscopic load. The difference of fatigue life at 298 and 1,173 K was attributed to additional tensile stress that was experienced by the FCC matrix at 298 K, since the phase transformation occurred much later during low-cycle fatigue at the higher temperature.
Chapter 6: Conclusions

1. The onset of the strain-induced FCC $\rightarrow$ HCP phase transformation was 685 MPa for tensile deformation, 698 MPa for compressive deformation, cycle 1 for high-cycle fatigue, and cycle 3 for low-cycle fatigue.

2. The accumulation rate of the HCP phase was 0.1 g-MPa$^{-1}$ for tensile deformation, 0.05 g-MPa$^{-1}$ for compressive deformation, and proportional to $1/N$ (where $N$ is the number of fatigue cycles) for high- and low-cycle fatigue.

3. The FCC $\rightarrow$ HCP transformation was more favorable under tensile deformation than compressive deformation. This observation was consistent with a thermodynamic argument based upon the shear and dilatational transformation strains. The applied stress was large enough to compensate for the fact that the volume change was small for this phase transition, and the dilatational transformation strain opposed the transformation for the case of compressive deformation.

4. During fatigue, the HCP accumulated according to a tensile plastic work criterion. For high-cycle fatigue, the tensile plastic work decreased dramatically with fatigue cycles. Hence, the HCP phase formed immediately on the first fatigue cycle with no further accumulation during subsequent fatigue cycles. For low-cycle fatigue, reverse plasticity during the compressive half-cycle allowed for a continual input of tensile plastic work throughout the fatigue life. Therefore, the HCP phase accumulated during low-cycle fatigue.
5. The structural transition was greatly delayed during low-cycle fatigue upon increasing the temperature to 873 K. No transformation was observed at 1148 K.

6. The tensile plastic work criterion was consistent with temperature predictions performed in previous work on ULTIMET® superalloy. The temperature predictions agreed with the measurement when no transformation was expected, but a discrepancy of 4°C was noted when the transformation occurred. The energy dissipated by microstructural changes in this case may not be negligible, as was assumed in the thermodynamic model.

7. The HCP phase forms with a texture that allows for shear deformation along the close-packed {111} planes in the FCC matrix. Since stacking-fault (and, hence, partial dislocation) accumulation was a necessary prerequisite for this transformation, the shear deformation was a requirement.

8. Plastic strains below 2% were accommodated by stacking-fault accumulation. Plastic strains above 2% were accommodated by the phase transformation. These two regimes correspond to the two regimes of power-law hardening for this alloy: (a) a regime characterized by a large increase in stress and a small increase in strain and (b) a regime characterized by a small increase in stress and a small increase in strain.

9. Deformation by twinning was found to be more probable under compressive loading.

10. The distribution of lattice strains in reciprocal space can be mathematically captured by considering the strain as the sum of two contributions: the linear-elastic contribution and the intergranular contribution.
11. The intergranular strains bias the measured lattice strains and lead to the observed deviations from linearity in the \( hkl \)-dependent stress-strain curves.

12. The intergranular strains showed a power-law dependence upon plastic strain during tensile deformation. As in the case of Conclusion #8, this dependency was separated into the two regimes of power-law hardening: (a) plastic strains less than 2\% and (b) plastic strains greater than 2\%.

13. Inhomogeneities in the deforming material, such as a fatigue crack or a new phase, further bias the lattice strains. This effect was accounted for within the linear-elastic contribution discussed in Conclusion #10.

14. The load-sharing characteristics among two phases can be calculated from the analysis method of Chapter 5.

15. The HCP phase was found to form in a state of hydrostatic compression. This stress state, in turn, caused the FCC phase to experience an additional tensile stress. The additional tensile stress in the FCC phase was detrimental to the fatigue life. Since the phase transformation was delayed at 873 K as compared to room temperature, the fatigue life was longer at the higher temperature.
Chapter 7: Future Work

1. A more sensitive technique for detecting the phase transformation should be applied to verify/refine the characterization presented in this work. An excellent option would be Resonant Ultrasound Spectroscopy (RUS), since the elastic properties of the bulk will change as the phase transformation occurs. However, completing in-situ deformation experiments with RUS, as was done here with neutron diffraction, may prove difficult. A very fine increment of plastic strain should be considered, since RUS is a very sensitive technique.

2. The modeling efforts presented in Figure 3-24 should be revisited in order to verify if the temperature predictions can be improved by including the energy dissipated by the phase transformation into the calculations.

3. The texture development of this material during deformation should be characterized by measuring complete pole figure for both the FCC and HCP phases during monotonic tension and compression, high-cycle fatigue, and low-cycle fatigue.

4. The neutron measurements discussed in Chapter 3 should be repeated with synchrotron X-Ray experiments. The data-collection times are smaller for synchrotron X-Ray diffraction than for neutron diffraction, so creep deformation would be eliminated. Finer deformation increments would be possible, as well. Furthermore, synchrotron experiments allow for the measurement of the complete lattice-strain and intensity distributions in both reciprocal and sample-orientation
space. The complete strain orientation and crystal orientation distribution functions could, therefore, be characterized as a function of deformation.

5. The strain analysis discussed in Chapter 5 should be applied to many data sets, including: single-phase materials, dual-phase materials, transforming materials, and materials exhibiting a body-centered cubic crystal structure.

6. The strain analysis of Chapter 5 should be incorporated into a Rietveld refinement to capture the peak positions of deforming cubic polycrystals.

7. Texture development is important when considering lattice strains measured by diffraction. A method to account for texture should be incorporated into the strain analysis developed in Chapter 5.

8. Potentially, the concepts of Chapter 5 to capture the intergranular-strain distribution for cubic materials could be extended to the case of hexagonal materials.


Appendices
Figure A-1: Measured axial lattice strains at the minimum stress for high-cycle fatigue: 
(a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure A-2: Measured transverse lattice strains at the minimum stress for high-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Appendix B: Measured Lattice Strains at the Five Positions around the Stress-Strain Hysteresis Loop for Low-Cycle Fatigue

Figure B-1: Measured axial lattice strains at tensile zero stress for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.

![Graph showing lattice strain vs fatigue cycles for different crystallographic planes.](image)
Figure B-2: Measured transverse lattice strains at tensile zero stress for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-3: Measured axial lattice strains at compressive zero strain for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-4: Measured transverse lattice strains at compressive zero strain for low-cycle fatigue: (a) 111, 200, 220, and 311 and (b) 331, 420, and 422.
Figure B-5: Measured axial lattice strains at the maximum compressive strain for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-6: Measured transverse lattice strains at the maximum compressive strain for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-7: Measured axial lattice strains at compressive zero stress for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-8: Measured transverse lattice strains at compressive zero stress for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-9: Measured axial lattice strains at tensile zero strain for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Figure B-10: Measured transverse lattice strains at tensile zero strain for low-cycle fatigue: (a) 111, 200, 220, and 331 and (b) 331, 420, and 422.
Vita

Michael Lee Benson was born on Barksdale Air Force Base near Shreveport, LA. He was raised in Maryville, TN and graduated from Heritage High School. He entered The University of Tennessee (UT) as an undergraduate in 1997 and graduated with a Bachelor of Science in Materials Science and Engineering in 2002. Michael subsequently entered graduate school at UT under Prof. Peter K. Liaw. Upon finishing his doctorate in Materials Science and Engineering (with a Metallurgy concentration), he joined the United States Nuclear Regulatory Commission.