ON THE CREEP DEFORMATION MECHANISMS
OF AN ADVANCED DISK NI-BASE SUPERALLOY

DISSERTATION

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ABSTRACT

Ni-base superalloys are an important class of high temperature structural materials that are used in the hot section of aircraft gas turbine engines. These materials possess the inherent capability to retain strength and resistance to creep, fatigue, and oxidation at elevated temperature, which makes them suitable for such applications. With the development of newer generation turbine disk alloys, it is highly imperative for aircraft engine manufactures to substantiate the use of the material by conducting a thorough examination of the materials mechanical properties. During service conditions, these components are subjected to elevated temperatures and complex stress states where time dependent creep deformation is of utmost concern and needs to be accounted for from a design criteria point of view. Creep deformation can be a performance and life-limiting factor of the component and therefore the relationship between microstructure, creep behavior, and underlying creep deformation mechanisms must be fully understood in order to design alloys and tailor microstructures for improved creep resistance.

The main objective of this research was therefore aimed at investigating the fundamental relationship between microstructure and creep deformation mechanisms using a variety of electron microscopy characterization techniques. The alloy used in this research, Rene 104, is a newer generation powder metallurgy Ni-base superalloy that was developed specifically for aircraft gas turbine disk applications with extended service durability at temperatures exceeding 650°C. The influence of stress and temperature was
studied first and it was found that during creep deformation at temperatures between 677-815°C and stresses between 345-724MPa a variety of distinctly different creep deformation mechanisms were operative. In addition to identifying the creep deformation mechanisms an attempt was made to determine the creep rate limiting process so that an improved understanding of the fundamental processes that control deformation can be better understood.

Microtwinning was found to the dominant deformation mechanism following creep at 677°C/690MPa and 704°C/724MPa. Microtwins form by the motion of paired a/6<112> Shockley partial dislocations that shear both the γ matrix and γ′ precipitates. The rate limiting process in this mechanism is diffusion mediated atomic reordering that occurs in the wake of the shearing, twinning partial dislocations in order to maintain the ordered L12 structure of the γ′ precipitates. This reordering process helps to fundamentally explain the temperature and rate dependence of microtwinning under creep conditions within this temperature and stress regime. At a slightly higher temperature but lower stress (760°C and 345MPa), a stacking fault related shearing mechanism, which typically spanned only a few micrometers in length, was the principle deformation mode. The faults left behind in the γ′ precipitates determined to be extrinsic in nature. During creep at the highest temperature and lowest stress (815°C and 345MPa) a thermally activated climb/bypass mechanism of a/2<110> dislocations were found to be the dominant deformation mechanism. In this mechanism, the γ′ precipitates were not
sheared but instead were bypassed by a/2<110> matrix dislocations. In addition to the identification of creep deformation mechanisms as a function of stress and temperature, characterization of the post creep γ′ precipitate microstructure revealed that microstructural evolution of the γ′ precipitates has occurred during creep at the higher test temperatures where the secondary γ′ precipitates have coarsened and the tertiary γ′ precipitates have dissolved. In combination with creep at low stress and high temperature, the microstructural evolution may have contributed to the transition from one deformation mode to another.

In an attempt to link the influence of microstructure (γ′ precipitate size scale, distribution, volume fraction, and γ channel width spacing) on creep deformation behavior and creep deformation mechanisms, specimens with different size scaled microstructural features were crept at the same temperature and stress (677°C and 724MPa) in order to provide a direct comparison between differences in microstructure. It was found that a microstructure consisting of a bimodal distribution of γ′ precipitates with coarse secondary γ′ precipitates, a high volume fraction of tertiary γ′ precipitates and a wide γ channel width spacing results in a less creep resistance microstructure that deformed primary by a/2<110> dislocation activity in the γ matrix at small strain and secondary γ′ shearing via superlattice intrinsic stacking faults at higher strains. The more creep resistant microstructure consisted of a bimodal distribution of γ′ precipitates with a finer secondary γ′ precipitate size, low volume fraction of γ′ and narrow γ channel width spacing.
spacing. The combination of these microstructural features promoted \( \frac{a}{2}<110> \) dislocation dissociation and decorrelation of \( \frac{a}{6}<112> \) Shockley partial dislocations that deformed the \( \gamma \) matrix at low strains, which then led to microtwinning at higher strain. These results suggest that in order to improve the creep resistance of this alloy the material needs to be heat treated such that it will develop into a microstructure with fine-scaled microstructural features.

Owing to the importance of microtwinning as principal deformation mode during creep in Ni-base superalloys, a further investigation as to what the microstructural features are that contribute to the formation of microtwins during creep deformation was studied. Twinning is generally considered a low temperature, high strain rate deformation mode in materials that possess a face centered cubic (fcc) crystal structure and so it was quite surprising to observe microtwinning as a dominant deformation mode in a \( \gamma' \) strengthened superalloy whose microstructure consists of a fcc \( \gamma \) matrix and L1\(_2\) structured \( \gamma' \) precipitates. Experimentally, microtwin nucleation sources have been identified and their evolution into fully developed microtwins as a function of increasing plastic deformation has been explored. At the earliest stages of deformation and for the microstructure that promoted microtwinning at larger strains, it was found that grain boundary carbides/borides and/or intragranular carbides act as dislocation sources that emit \( \frac{a}{2}<110> \) dislocations in the \( \gamma \) matrix. Due to the combined effects of narrow \( \gamma \) channel width spacing, stacking fault energy, and resolved shear stress the \( \frac{a}{2}<110> \)
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Dedicated to
Kinga and Miki
Miłość na zawsze!
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CHAPTER 1

INTRODUCTION

Powder metallurgy produced Ni-based superalloys are an important class of high temperature structural materials, which have been developed specifically to meet the demands of aircraft gas turbine engines where they are subjected to temperatures in excess of 600ºC and operate under a complex stress state. Creep deformation is of concern for materials used in such applications since at high temperature, deformation may occur over a period of time. For this reason, it is important to have a fundamental understanding of the mechanisms that contribute to creep deformation.

The purpose of this research is aimed at exploring the relationship between microstructure, stress, and temperature on the rate limiting process that control creep deformation. This dissertation is divided into chapters that address each of these issues. In Chapter 2, a literature review is presented which introduces the basic physical and mechanical metallurgy principals as it relates to Ni-base superalloys used in high temperature applications. The goal of reviewing this technical content is to provide a detailed explanation as to what the microstructural constituents that are commonly present in these alloys and how they can be altered to attain the desired mechanical
properties, which ultimately justifies the use of these materials in high temperature structural applications such as in a aircraft gas turbine engine. Following this section of the review, the current state of knowledge concerning creep deformation mechanisms in Ni-base superalloys and what implications they have on creep rate-limiting processes is reviewed.

The research presented in this dissertation is centered around the use of advanced materials characterization techniques to identify creep deformation mechanisms so that the results can be correlated with the overall macroscopic deformation response of the material under a range of test conditions and incorporated in existing and future physically based creep models. In Chapter 3, the effect of stress and temperature on creep deformation mechanisms is presented. A detailed characterization of the deformation substructure has revealed a variety of different deformations mechanisms have become operative. A rather interesting deformation mechanism was uncovered during creep at an intermediate stress and temperature. Microtwinning was found to be the principal deformation mode during creep within this regime which was quite surprising to observe since twinning in general is considered a low temperature, high strain rate mode of deformation.

In Chapter 4, a more in depth study was conducted in order to determine the salient microstructural features that control deformation and favor one deformation mode vs. another. The influence of microstructure size scale (γ′ precipitate size and interparticle spacing) was determined by conducting creep experiments at the same stress and temperature on specimens that received different heat treatments. The results of this chapter reveals that a more creep resistance microstructure is one that has a combination
of fine scaled secondary γ’ precipitates, low volume fraction of tertiary γ’ precipitates, and a fine γ channel width spacing. This microstructure also led to microtwinning at the later stages of deformation. The less creep resistant microstructure has coarse secondary γ’ precipitates, high volume fraction of tertiary γ’ precipitates, and a wide γ channel width spacing.

In Chapter 5, microtwin nucleation sources have been identified and their evolution into fully developed microtwins were tracked by examining samples that were interrupted at varying stages of creep deformation. A mechanism for microtwin nucleation was proposed based on experimental post mortem and in-situ TEM characterization. The results of this study show that the factors which control creep deformation is highly influenced by internal factors such as the microstructure and external factors such as stress and temperature. It is highly imperative that the detailed deformation processes characterized in this study be account for in the development of sound, physically based models for creep in Rene 104 and other similar PM Ni-base disk superalloys.
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The goal of this chapter is to review the physical and mechanical metallurgy of Ni-base superalloys and to present a basic understanding of why superalloys are suitable for high temperature applications through an interpretation of physical and mechanical metallurgy principles. The approach that will be used to evaluate the relationship between microstructure and properties will be subdivided into three fundamental concentration areas. First, the physical metallurgy of superalloys will be reviewed with attention given to specific phases that exist in these alloys that are known to contribute to the materials unique mechanical properties. In particular, the $\gamma'$ precipitate phase will be discussed in detail since it is this phase, which acts as effective barriers to dislocation motion. The second thrust will be to examine the mechanical behavior of superalloys as it relates to the strengthening mechanisms outlined in the preceding section. In particular, the mechanical behavior of Ni$_3$Al, when considered alone, exhibits profound deformation characteristics in that an anomalous yield stress behavior is observed when deformed at increasingly higher temperatures. This behavior will be reviewed then further extended to
when Ni$_3$Al is present as a precipitate phase in a nickel rich solid solution. Lastly, it is essential to establish a link between the microscopic deformation characteristics and the macroscopic creep response; therefore, a detailed understanding of the mechanisms that control creep deformation modes are needed. Deformation modes such as APB hardening, γ′ shearing/superlattice stacking fault formation, microtwinning, and Orowan looping will be discussed. For the γ′ shearing mechanism and microtwinning, a number of contradictory theories have been proposed throughout literature in an attempt to explain the shearing process by dislocations (matrix, superpartial, Shockley partial, and superlattice Shockley partials) and subsequent superlattice intrinsic/extrinsic stacking fault formation. Such controversies will be addressed as well as an identification of areas that seem to be lacking in the literature in this realm of research.

Nickel based superalloys are an important class of engineered materials in that they were developed specifically for industrial and aircraft gas turbine engines whose harsh service conditions necessitates the use of sophisticated structural materials that possess a stringent combination of material property attributes such as high temperature strength and resistance to creep, thermal/mechanical fatigue, oxidation, and corrosion [1]. The deformation behavior of superalloys is a multi-faceted subject matter given that the system is comprised of a number of elemental constituents with each being intentionally added to enhance and balance the aforementioned material properties. A compiled list of the common alloying elements and their role in terms of physical metallurgy and strengthening mechanisms is presented in Table 2.1.

From this table it can easily be deduced that there are a number of strengthening mechanisms that are inherently present in superalloys, which makes this alloy system the
premier material of choice for high temperature structural applications such as in the
turbine section of aircraft engines. An investigation into the structure, processing, and
property relationships of existing and newly developed superalloy chemistries is required
to address the ever increasing demand for increased engine operating temperature
capabilities, engine efficiency, and performance.

2.2 Physical Metallurgy of Ni₃Al

Superalloys attain a significant portion of high temperature strength and creep resistance
from the presence of coherent, ordered, L1₂ crystal structured γ′ Ni₃Al precipitates that
are embedded in a disordered γ (F.C.C.) matrix phase. The L1₂ crystal structure can be
evisaged as having a F.C.C. lattice with Al atoms occupying corner lattice positions and
Ni atoms occupying the face-centered lattice positions. Ni₃Al is considered to be a long
ranged order structured in that there is a strong tendency for atoms to be surrounded by
unlike atoms arranged in a periodic ordered fashion [2]. Ni₃Al is formed by a disorder-
order phase transformation from a peritectic reaction between the disordered nickel rich
solid solution and the liquid. Upon heating, the ordered structure is maintained up to a
melting temperature of ~1383°C as shown in the binary Ni-Al phase diagram presented
in Figure 2.1.

2.3 Mechanical Metallurgy of Ni₃Al

The mechanical behavior of the γ′ phase (Ni₃Al) alone has been the subject of numerous
studies and is now well documented [3, 4]. Davies et al [5] and Thornton et al [6, 7] were
among the first to study the anomalous flow stress behavior of Ni₃Al as a function of
temperature. The behavior was noted to be temperature-path history independent. With
increasing temperature, it was observed that the flow stress showed a corresponding increase as well but is sensitive to strain rate. (Figure 2.2) This phenomena is quite different from the behavior of other materials in that when deformed, a decrease in the flow stress with temperature would be expected since dislocations become more mobile as a result of thermally activated climb or cross slip mechanisms. Theories as to the anomalous yield stress behavior of Ni₃Al share the common theme in that thermally activated screw dislocations that are traveling on octahedral {111} glide planes cross slip onto {010} cube glide planes where they are immobilized [8-10]. This theory was originally proposed by Kear and Wilsdorf [11] in the treatment of plastically deformed Cu₃Au alloys and has justly been termed the Kear-Wilsdorf locking mechanism. Cross slipping in this manner is plausible since the APB energy on the {010} plane is less than the APB energy on the {111} [12]. As temperature increases, thermally activated cross slipping of screw dislocations onto this plane are promoted, locking ensues, and a concomitant increase in flow stress results. After the yield stress peak, the yield stress drops when the critically resolved shear stress is reached to activate an alternative slip system [13].

2.4 Mechanical Behavior of Ni-based Superalloys

2.4.1 Strengthening Mechanisms

There are a number of strengthening mechanisms that are inherent to Ni-based superalloys. In a simplistic interpretation, plastic deformation in crystalline materials is associated with the movement of dislocations through the lattice and barriers to dislocation motion will give rise to a strengthening contribution. In superalloys, these
impediments can take on the form of grain boundaries, solid solution elements, precipitates, dispersoids, carbides and other dislocations.

### 2.4.2 Oxide Dispersion Strengthening

Stable, high melting, oxides are often mechanically alloyed to superalloys through powder metallurgy processing techniques to produce a fine dispersion of oxide particles that are stable at elevated temperatures (ThO₂, Y₂O₃, and Al₂O₃). Strengthening results since oxides by their nature are hard, brittle ceramic materials where they act as hard obstacles that effectively obstruct dislocation motion. An example of creep and stress rupture benefits was reported by Howson et al in MA 6000E [14]. In this specific alloy, γ′ strengthening and oxide dispersion strengthening are both synergistic; however, at higher test temperatures (1093°C) creep strength was attributed mostly to the oxide dispersoids as seen in post mortem TEM images where the dislocations were effectively pinned by the oxides whereas dislocations were observed to bypass γ′ precipitates. What this indicates is that there is a temperature regime wherein strengthening is benefited by oxide dispersions.

### 2.4.3 Solid Solution Strengthening

When solute elements are present in a solid solution, they impart local lattice distortions. Dislocations will encounter these strain fields and can hinder dislocation motion to some degree. Elements such as W, Mo, Co Cr are intentionally added for this reasoning. At elevated temperatures (>0.6Tm), where diffusion and thermally activated cross-slip processes dominate creep, slower diffusing solute elements such as W and Mo become increasingly important in terms of providing solid solution strengthening [15]. The
strength contribution of solid solution strengthening is notable; however, it is significantly small when compared to other strengthening mechanisms.

2.4.4 Grain Boundary Strengthening

Grain boundaries act as effective barriers to dislocation slip. As an example, for a given grain orientated with a high Schmid factor, a dislocation source may be initiated within the grain interior. With further applied stress, dislocations will be emitted from this source and begin to propagate through the microstructure until they are either effectively impeded by other features in the microstructure or they make their way to grain boundaries. For the latter case, two events may then occur: pileup of dislocations at the grain boundaries or dislocation transmission across the boundary. Macroscopic yielding would result when the dislocations slip onto adjacent grains. Depending upon alloy chemistry and thermal processing, metal carbides may precipitate within grain interiors and/or along grain boundary. Those present along the boundaries act to stabilize the grain boundaries and increase the creep resistance since their presence provides an additional resistance to dislocation transmission across grains. Furthermore, they act to restrict grain boundary sliding, and thereby increasing creep rupture life [16].

Carbide precipitation along grain boundaries has been shown to have the effect of creating a precipitate free zone on either side of the grain boundary, wherein the regions surrounding the grain boundary carbides are free of $\gamma'$ precipitates. The effect of carbide precipitation and $\gamma'$ precipitate free zones on grain boundary strength/dislocation transmission has been the subject of work by Baither et al in Nimonic PE16 [17-19]. In this alloy system a $\gamma'$ precipitate free zone developed in regions adjacent to the grain
boundaries that were adorned with $\text{M}_{23}\text{C}_6$ type Cr-carbides. In-situ TEM studies show that dislocation transmission is easily facilitated through the PFZ since there is virtually no resistance to motion. This example shows a negative effect of grain boundary carbide precipitation.

A relationship between the yield strength of a material as a function of grain size has been proposed by Hall [20] and Petch [21]:

$$\sigma_y = \sigma_o + k_y \sqrt{d}$$

Where $\sigma_y$ is the yield strength, $\sigma_o$ is the flow stress, $k_y$ is a locking parameter, and $d$ is the grain size. This simple relationship shows that the yield strength is proportional to the square root of grain size. Moreover, the finer the grain size, the more resistant the material is to deformation because there are more obstacles (boundaries) that effectively resist dislocation motion and must be overcome in order for yielding to continue. Stoloff and Liu [22] noted that the Hall-Petch behavior is obeyed for polycrystalline Ni$_3$Al. On the influence of boron segregation to grain boundaries, Liu stated that boron additions promoted grain boundary cohesiveness yet still follows the Hall-Petch relationship [23]. In addition, Wallow et al [24] has also shown that there is a synergistic relationship between grain boundary strengthening and $\gamma'$ precipitation strengthening, where the Hall-Petch relationship was upheld; however, a change in $\gamma'$ volume fraction and radius of precipitate curvature altered only the Hall-Petch slope ($k_y$).
2.4.5 Precipitation Strengthening

The prime strengthening agent in Ni-based superalloys is the $\gamma'$ precipitate. The strength resulting from the presence of these precipitates are a function of size, volume fraction, distribution, and nature of the precipitate interface. As precipitate size and volume fraction increase, it is expected that strength would increase as well. Now add into the factor that the precipitates are an ordered structure that is stable at elevated temperatures. Dislocations that come in contact with the precipitates must bypass or shear through it. If by shear, a high-energy antiphase boundary (APB) or superlattice stacking fault structure would result. The role of APB energy and stacking fault energy on strength and deformation modes will be discussed in a later section. There are classical reviews published in the literature that describes the strength contribution of ordered precipitates such as these [25, 26]. In addition to the factors described above, Decker [27] outlined several other key factors that contribute to strengthening by $\gamma'$ precipitates alone: APB energy, $\gamma'$ fault energy (CSF, SISF, and SESF), coherency strain, and $\gamma/\gamma'$ modulus mismatch.

2.5 Deformation Modes

The identification of deformation mechanisms in $\gamma'$ strengthened superalloys have transpired from direct in-situ and post mortem transmission electron microscopy (TEM) imaging of dislocations and their interaction with $\gamma'$ precipitates. Such interaction phenomenon includes precipitate shearing, climb, cross-slip, and Orowan looping. Commonly observed defect structures present in creep-deformed superalloys that give rise to deformation are dislocations, stacking faults, and twins. Techniques for imaging
and characterizing such structures are available by means of diffraction contrast, weak beam, and high-resolution TEM imaging. Much of the detailed mechanistic understanding and theoretical modeling of creep in superalloys is based on these experimental TEM observations. Nabarro and Villiers [28] have compiled a collection of commonly observed dislocation precipitate shearing reactions in single crystal and polycrystalline superalloys. As evident, there are a number of different ways in which a superalloy can deform plastically by way of dislocation motion and $\gamma'$ precipitate shear. The motivation behind this section of the literature review is to provide an in depth analysis of the observed deformation modes and to interpret proposed $\gamma'$ shearing mechanism.

2.5.1 Slip systems and dislocations in FCC and L1$_2$ Structures

To begin an analysis on creep deformation modes, it is important to review several important concepts that will aide in the discussion of slip, precipitate shear, and stacking fault formation. Superalloys consist of a two-phase $\gamma/\gamma'$ structure. The crystallography of slip in the FCC matrix differs from slip in the L1$_2$ $\gamma'$ phase. For the case of FCC metals, slip generally occurs by the glissile motion of perfect dislocations on closed packed planes and in close packed directions which are represented by the twelve $\{111\}<110>$ slip systems. The matrix dislocation that slips on the close packed octahedral plane possesses the burgers vector $a/2<110>$, which represents a perfect lattice translation, along close packed directions. Matrix dislocations can further dissociate into Shockley partials possessing a burgers vector of $a/6<112>$ which are still glissile on the $\{111\}$ plane. This occurs by the following dissociation reaction:
Dislocation dissociation of this form is energetically favorable based on strain energy considerations. Associated with this dissociation leads to a disruption in the stacking sequence of the atomic planes, i.e. a stacking fault is produced. A number of mechanical characteristics can be related to the magnitude of the fault energy, i.e. cross slip. Stacking faults in F.C.C. materials are considered to be intrinsic or extrinsic stacking fault in nature. An intrinsic stacking fault can be produced when an atomic plane is removed. Conversely an extrinsic stacking fault is produced by the insertion of an additional atomic plane. These stacking faults can either be formed by the collapse of vacancies, condensation of interstitials, or by shear displacements of atomic planes. For example, if the crystal is sheared by $a/6<112>$ displacements, an intrinsic stacking fault if sheared by a single Shockley partial and an extrinsic stacking fault is produced if sheared by two Shockley partials on adjacent atomic planes.

The geometry of and dissociation of dislocations in the intermetallic Ni$_3$Al phase is more complicated than in F.C.C. structures. Dislocations in such structures are given the prefix “super” as this designates to dislocations in an ordered “super” lattice crystal structure. In this case, dislocations and partial dislocations are of the form: $a<101>$ (superdislocation), $a/2<101>$ (superpartial), $a/6<112>$ (Shockley partial), and $a/3<112>$ (superlattice Shockley partial). Several commonly observed dislocation dissociation reactions are presented below as reported by Sun et al [29].
A geometrical method of envisioning the formation of APB and superlattice stacking faults (S-SF) in the L1$_2$ structure is presented in Figure 2.3 a-d [3]. These figures depict the stacking sequence of three adjacent (111) close packed planes along with important crystallographic slip directions. In Figure 2.3a, shear displacement of one atomic plane in the $a/2[\bar{1}01]$ direction will result in an APB with incorrect nearest neighbor bonds. A SISF (Figure 2.3c) is formed by the shear displacement of $a/3[\bar{2}11]$, which results in like atoms lying atop one another but no wrong nearest neighbor bond violation. A SESF can be created by shear displacements of $a/3<112>$ on two adjacent planes. Furthermore a complex stacking fault (CSF), shown in Figure 2.3d, is formed by the shear displacement of $a/6[\bar{1}12]$. This produces a fault in the stacking sequence as well as creates wrong nearest neighbor atomic bonding. From a nearest neighbor bond violation criterion, a ranking of the relative fault and APB energies would appear as follows: CSF>APB>SISF~SESF. It appears to be logical that the energies associated with producing such a structure will have a large impact on and will dictate the manner in which precipitates are sheared and thus would in turn, have an affect on the macroscopic creep deformation behavior.
2.5.2 APB Shearing Mechanism

APB strengthening provides a significant role in alloy systems that have coherent, ordered precipitates. Assuming that climb and cross slip mechanisms are delayed or prohibited by temperature and stress, then precipitate shear would be the factor controlling creep deformation. The movement of paired dislocations (weak or strong paired coupled) is necessitated by the fact that upon shearing, the leading dislocation (D1) creates a high-energy antiphase boundary (cross-hatched), the order of which is restored upon subsequent shearing by the trailing dislocation (D2). As such, dislocations generally travel in pairs for this mechanism. This phenomenon can be seen schematically in Figure 2.4.

2.5.3 Orowan Looping Mechanism

Orowan looping is a deformation mode that is likely to occur in superalloys possessing an array of precipitates that are widely spaced apart from one another. Dislocations have the propensity to bypass coherent as well as incoherent particles in a process designated as Orowan looping. Orowan looping occurs when there is an array of impenetrable obstacles that are widely spaced apart from one another. A moving dislocation interacts with the particles and instead of shearing; the dislocation bows between them, is pinched off, and finally loops around the particles. A succeeding dislocation gliding on the same slip plane would then experience a repulsive force given off by the dislocation loop and would thus contribute to strength by a form of work hardening. The stress required for looping is inversely proportional to the interparticle spacing. The looping stress decreases as the interparticle spacing increases and vice versa for a decrease in interparticle spacing.
2.5.4 Precipitate Shearing vs. Orowan Looping

Present in the literature are particle-hardening theories that provide quantitative descriptions of discrete obstacle/dislocation interactions as a material tends to plastic deformation [25, 26, 30]. In alloy systems that inherently possess coherent, ordered precipitates; a transition between pairwise dislocation cutting (i.e. APB hardening mechanism as discussed in previous sections) and Orowan looping has been envisaged to exist.

For the case of pairwise cutting of $\gamma'$ precipitates in superalloys, dislocations have been observed to travel in pairs of matrix type dislocations ($b=a/2<110>$). As a result, an increase in the yield stress is required to propagate the dislocation through these discrete obstacles. Gleiter and Hornbogen is accredited with providing the first quantitative theory of order strengthening [31, 32]. The works of Brown and Ham [33], and Raynor and Silcock [34], further advanced this original theory. An acceptable form of the critical resolved increase in shear stress required to shear a coherent particles as published by Raynor and Silcock is as follows:

\[
\Delta \tau_o = \frac{1}{2} \left( \frac{\gamma_{APB}}{b} \right)^{\frac{3}{2}} \left( \frac{bdf}{T} \right)^{\frac{1}{2}} A \frac{1}{2} \left( \frac{\gamma_{APB}}{b} \right)^{\frac{1}{2}} f
\]  

Where $\gamma_{APB}$ is the APB energy, $b$ is the burgers vector, $d$ is particle diameter, $f$ is volume fraction, $T$ is the line tension of the dislocation, and $A$ is the particle shape factor.

Through a collection of particle hardening theories that are present in the literature, Reppich [35] has provided a schematic illustration of the transition from weak to strong-
paired particle cutting as well as the transition from cutting to Orowan looping as shown in Figure 2.5. Curve 1 depicts the increase in CRSS from Equation 6. Curve 2 shows a decrease in the cutting stress as particle size decreases. Cutting is still operable in this regime but a transition is marked from weak paired to strong paired coupling. Curves 3 and 4 correspond to when the Orowan looping mechanisms is favored beyond a critical particle diameter when the Orowan stress is less than the cutting stress. A simple observation drawn from this schematic is that there seems to be an optimal particle size that exists between cutting and looping that provides a high value of yield strength.

Historically this yield stress increase has been depicted as a function of the average particle size, volume fraction, and APB energy only. This is to assume that order strengthening would be the only impediment to dislocation motion while neglecting coherency strain, γ/γ’ modulus differences, and γ/γ’ stacking fault energy differences as pointed out by Huether and Reppich [36]. Another discrepancy in the model is that it does not account for the effect of bi-modal and tri-modal precipitate size scale distributions as found in some newer polycrystalline disk alloys. A modification to this form would be required to account for this. Another factor that needs to be mentioned is that the model assumes a constant dislocation line tension. This may be a good approximation; however, physically is not a valid assumption since the line tensions of screw and edge dislocations differ by a factor of 4 [37]. Despite these notable inconsistencies, the form of this equation does follow experimental results quite adequately as compared experimentally by Martens et al [38] in monomodal γ’ strengthened Nimonic PE 16 and Melander et al [37] in Nimonic 80A.
On a further note, much of these theories have been for Nimonic superalloys in which there are a low volume fraction of $\gamma'$ and that the morphology of $\gamma'$ precipitates are roughly spherical. This leads to the question of this model’s validity for high volume fraction of precipitates with a cuboidal morphology. To the authors knowledge and present literature search there have been no published findings that would suggest Orowan looping as a probable strengthening mechanism in superalloys containing a high volume fraction of cuboidal shaped $\gamma'$ precipitates such as those present in single crystal turbine blade materials.

With reference to the experimental details of particle shearing vs. Orowan looping theory, a significant factor that needs to be addressed is the ability to precisely and routinely measure the $\gamma'$ volume fraction and $\gamma'$ diameter. From this there is some skepticism concerning the validity of such models. At the time when much of this theory was being developed, transmission electron microscopes did not have the resolution power as compared to modern advanced microscopes of today. Dark field images, utilizing a superlattice reflection, were the norm when acquiring images of $\gamma'$ precipitates; however, advanced imaging techniques such as energy filtered TEM (EFTEM) are now available and have been proven to be a superior method for rapidly acquiring high resolution images of $\gamma'$ precipitates in Ni-based superalloys down to a size scale of $\sim$3nm [39]. From EFTEM images, accurate characterization of $\gamma'$ size, volume fraction, and distribution can be conducted using quantitative image analysis techniques.
2.5.5 Superlattice Stacking Fault Shear Mechanism

Precipitate shear of ordered precipitates by a/2<110> type dislocations and APB formation does not represent the major deformation mode in superalloys. In fact, precipitate shear and superlattice stacking fault formation has constituted a significant portion of experimental observations in published work on creep in Ni-based superalloys. Models have been proposed in literature, which describes dislocation shearing configurations by a/2<110> F.C.C. matrix dislocations, a/6<112> Shockley partials, a/3<112> superlattice Shockley partials, and the formation of superlattice extrinsic (SESF) and superlattice intrinsic (SISF) stacking faults resulting thereof.

2.5.5.1 Mechanism 1

A model of precipitate shear and superlattice stacking fault formation has been proposed by Condat and Decamps [40]. Based on experimental weak beam image observations, Condat and Decamps proposed a shearing mechanism by a single a/2<110> matrix dislocations. The hypothesized mechanism is centered on the following theme: 1) a single a/2<110> matrix dislocation (BD) enters and shears the γ’ precipitate 2) creating an APB 3) a Shockley partial (αC) nucleates on the APB plane and 4) αC removes the APB and creates a lower energy SISF (or SESF if the Shockley partial is Cα) and 5) leaving behind a Shockley partial at the interface. (Figure 2.6)

The theory of Shockley partial dislocation spontaneously nucleating on an APB and sweeping across the APB plane to restore the order of the structure and thereby producing a lower energy-stacking fault seems quite perplexing and leaves some ambiguity to this interpretation. Research conducted by Suzuki et al [41] have described
that a conversion of an APB into an SISF by the nucleation of a Shockley on the APB plane is probable and energetically favorable. A schematic illustration depicting a Shockley nucleation event on an APB can be seen in Figure 2.7. Suzuki merely suggested that a nucleation event can occur but does not offer any scientific reasoning as to the mechanism by which a Shockley partial nucleates in their writings. Although not explicitly written, their schematic does indicate that an additional dissociation reaction takes place from the \( \frac{a}{2}<110> \) while inside the precipitate. A dissociation reaction occurs from the \( a/2<110> \) creating both a Shockley partial and a superlattice Shockley partial. Shockley partial nucleation was studied by Chiba and Hanada [42]. (Figure 2.8) They continued this argument of a local dissociation of the matrix dislocation dissociating into a Shockley partial and superlattice Shockley partial but added that after the event has occurred, the SISF must be bounded by the superlattice Shockley partials \( a/3<112> \) during this transformation.

### 2.5.5.2 Mechanism 2

In addition to the work on SISF loop nucleation on an APB plane, Caron et al [43] reported on another scheme that would create an SISF. This time, a \( a/2<110> \) matrix dislocation directly dissociates at the \( \gamma/\gamma' \) interface, creating a superlattice Shockley partial that would then shear the precipitate according to the following reaction:

\[
\frac{a}{2} < 110 > \rightarrow \frac{a}{3} < 121 > + SISF + \frac{a}{6} < 112 > \tag{7}
\]
In this case, the superlattice Shockley partial enters and shears the precipitate, creating the SISF and leaving at the interface a \(a/6<112>\) Shockley partial. Based on Frank’s rule of dislocation dissociation \((b_1^2 + b_2^2 + b_3^2)\) it seems that this dissociation mechanism is energetically unfavorable as noted by Zhang et al [44] Link and Feller-Kniepmeier [45] addressed this issue by stating that the Shockley partial relaxes the coherency stresses at the \(\gamma/\gamma'\) interface which reduces the overall energy of the configuration so that the reaction can proceed at the coherent interface. Milligan and Antolovich [46] have reported experimental evidence of this shearing mechanism in single crystal PWA 1480 when crept at 700°C and 900°C, where it was mentioned that the Shockley partial left at the \(\gamma/\gamma'\) interface is immobilized since further propagation through the ordered precipitate would create an APB. The microscopy does clearly show a \(a/2<110>\) matrix dislocation within the \(\gamma\) matrix channel between two \(\gamma'\) precipitates and a \(a/3<112>\) superlattice Shockley partial leading the shearing action within the precipitate itself; however, there was no additional image or dislocation analysis in this specimen that would indicate a Shockley partial present at that interface. If this were unambiguously identified, then this mechanism could conclusively be substantiated from their work. Veld et al [47] on the other hand did find this mechanism to be operative in superalloy MA 6000 crept at 760°C and 790°C and identified a \(a/6<112>\) Shockley partial that looped the precipitate at the \(\gamma/\gamma'\) interface through g dot b analysis. It seems from this theory and experimental evidence that this is a valid shearing mechanism.
2.5.5.3 Mechanism 3

Decamps et al [48] proposed yet another mechanism by which a SISF or SESF can form within a sheared $\gamma'$ precipitate. Instead of their original mechanism of shear by a single matrix dislocation and nucleation of a Shockley partial to wipe out the APB, the newly proposed mechanism consists of shear by a dissociated matrix dislocation. Schematic illustrations of the proposed shearing mechanisms are located in Figure 2.9a-b for SISF and SESF formation, respectively.

The illustrations indicate that a single matrix dislocation is dissociated into two Shockley partial while still present in the $\gamma$ matrix. If this were true, a stacking fault should be present between the Shockley partials. On an aside note, this discrepancy is not shown in the illustrations or eluded to in their report. The next stage in the shearing process is that the leading Shockley partial enters the precipitate where it begins the shearing process. For the SESF case (Figure 2.9b), they assume that there is a high enough force for the partial dislocation to cross the interface; however, no further analysis or quantitative description of the force or energy barrier required to do so were suggested.

This is an interesting topic in and of itself but should further be mentioned that there is a present lack of research in the literature on this subject. Eventually, the trailing partial enters the precipitate and leaves an APB in its wake. A narrow CSF would be formed between the two Shockley partials that are causing the shearing action and the idea of an additional Shockley partial nucleating on the APB plane to create a SESF was again utilized to account for the experimentally observed stacking faults within the precipitates. For the SISF case (Figure a), the shearing process is of the same form but
now there is an inversion on the Shockley partials where the leading partial becomes the trailing and vice versa. An additional Shockley then nucleates on the now trailing partial, removing the APB, and creating the SISF. The idea of the partial dislocation inversion process while inside the precipitate is still unclear after reviewing this manuscript.

In a further study on the same subject matter, high resolution imaging was conducted by Decamps et al [49] to determine if the Shockley partial nucleates on APB plane itself or on an adjacent \{111\} plane above or below the fault. Conventional TEM studies cannot determine this; therefore high resolution TEM is needed since it has the capability of imaging atomic planes. The analysis was carried out by tilting the stacking fault on edge so that a side view of the stacking sequence of atomic planes can be seen. It was determined that in the SISF case, the fault occurred on a single plane. The SESF results on the other hand showed a two-layer stacking fault configuration, which would indicate that the Shockley nucleated above or below the APB plane.

2.5.5.4 Mechanism 4

The final stacking fault shearing mechanism that will be discussed in this review was prompted by experimental evidence that depicts a shearing configuration consisting of a continuous stacking fault contrast through the \(\gamma\) matrix as well as the \(\gamma'\) precipitates (Figure 2.9a) as opposed to stacking fault contrast from within the \(\gamma'\) precipitates alone. The superalloy consisted of a bi-modal distribution of \(\gamma'\) precipitates instead of a monomodal distribution as was present in the previous scenarios. The proposed shearing mechanism is represented in Figure 2.9b. The mechanism involves the decorrelated movement of Shockley partials that shear the matrix as well as the precipitate. The
movement of Shockley partials in the F.C.C. matrix does create a stacking fault, which
does account for the fault presence in the matrix phase. The trailing Shockley partial must
be pinned at some point in the microstructure and the shearing action is caused by the
leading partial. The proposed faulting in the precipitate themselves is less intuitive. As a
Shockley partial enters the γ’ precipitate, a CSF is formed, which as noted previously is
an extremely high-energy configuration. After shearing, it is proposed that Shockley
partial of opposite sign nucleates on a plane above or below the CSF, converting the
configuration to a lower energy SESF configuration. If this were the case, then the
nucleated Shockley does not effectively alter the structure of the fault and thus SESF
formation is not possible. Therefore, this mechanism does not seem plausible based on
these grounds.

2.5.6 Mechanism for Microtwinning

Deformation twinning is known to occur in metals possessing B.C.C. and H.C.P. crystal
structures due to the limited number of available slips systems [50]. However, in F.C.C.
metals that possess a greater number of slip systems, deformation by twinning has not
been considered to readily occur except under low temperature, high strain rate
conditions, or when the twinning stress is less than the stress required to initiate slip [50].
Microtwinning in Ni-based superalloys has recently been identified as yet another
deformation mode that is possible during high temperature creep. Microtwinning has
been observed in single crystal alloys SRR99 [51] and CMSX-4 [52, 53] and more
recently in polycrystalline disk alloys René 88DT [54].
The works of Ardakani et al [51, 55] revealed that microtwins formed in SRR99 and CMSX-4. The twinned structure was identified through diffraction contrast TEM imaging and selected area diffraction, whose patterns were acquired along a beam direction of [011]. The presence of fundamental, superlattice, and twinned reflections in the diffraction pattern clearly reveals a twinned structure and that the twinning planes are of the \{111\} type and the twinning direction is of the <112> type. Ardakani and coworkers experimental observations are valid; however, the authors did not attempt to provide any suggestion for microtwin formation mechanisms in their analysis.

Kakehi [56] on the other hand proposed a mechanism that may lead to the formation of microtwins by superpartial dislocation shear and superlattice stacking fault formation. He proposed that $\gamma'$ precipitates are sheared by a/3<112> superlattice Shockley partials on adjacent \{111\} planes, which would result in SISF formation on the first layer, SESF on the second layer, and a twin on successive layers. Veld et al [47] also briefly mentioned that microtwins are formed by stacking faults on successive planes. Gunturi [57] and Knowles [52] have presented some contradictory findings that dispute Kakehi’s proposal. They observed microtwins to be present in CMSX-4 only when SESF formation is predominate and not by shear on adjacent \{111\} close packed planes by identical a/3<112> superpartial dislocations. Their conclusion was that the microtwinning process is somehow related to shear and SESF formation.

Kakehi attempted to justify his proposal by stating that shear by a/6<112> Shockley partials is highly unlikely because the ordered L1_2 structure would be destroyed as this would result in a higher energy configuration with incorrect nearest neighbor atomic bonds. The scientific reasoning behind this is valid, since shear by successive
a/6<112> Shockley partials does indeed create a CSF with wrong nearest neighbor bond configurations. The passage of a/3<112> partials would however preserve the L12 structure. In a review by Christian and Mahajan [58], deformation induced twinning in L12 superlattice structures would result in the production of a pseudotwin, that possesses an imperfect superlattice with incorrect atomic ordering. Atomic re-shuffling would thus be required to convert the pseudotwin structure to that of a true twinned structure with correct nearest neighbor atomic bonds.

Kolbe [59] was the first to propose a diffusion based crystallographic model that accounts for thermally activated atomic reordering to produce a true twin structure from a CSF in the γ′ phase in Ni superalloys. Assuming the microtwins formed by successive shear of a/6<112> Shockley partials along adjacent (111) planes, a CSF (pseudotwin) would result in the γ′ phase possessing a lower symmetry orthorhombic phase and a true twin structure in the F.C.C. γ matrix phase. The atomic reshuffling steps required to convert the pseudotwin to a true twin in L12 can be seen in a series of schematic illustrations in Figure 2.10a-d, and it can be argued that this diffusion process can be facilitate at elevated temperatures such as those incurred during creep.

Viswanathan et al [54, 60] were the first to provide experimental evidence of the microtwin formation process through diffraction contrast (BF and WB) and high resolution imaging experiments on crept René 88DT at 650°C/838MPa/0.5% strain. (Refer to Figure 2.11) BF and WB microscopy techniques were utilized to determine the sign and nature of partial dislocations, which were determined to be a/6<112> partials of the same sign. High resolution imaging conclusively illustrated a twinned structure that transcended both the matrix and γ′ precipitate phase. Their results convincingly show that
microtwin formation is the results of successive shear by a/6<112> partials on adjacent {111} planes. To reiterate, this would produce a pseudotwin in the L1₂ structure as indicated above but can be converted to a true twinned structure if atomic rearrangement were allowed to take place. By accepting Kolbe’s [59] proposed mechanism of atomic rearrangement, Viswanathan et al [54] validated their experimental findings and their proposed mechanism of microtwin formation.

2.6 Summary

This survey of the current literature reviewed a critical niche in the superalloy research community by reviewing factors that contribute significantly to strengthening such as grain boundary, solid solution, dispersion, and precipitate strengthening. For each strengthening mechanism the major factors that influence the strengthening behavior was presented in a qualitative manner. An assortment of γ’ shearing mechanisms are present in the literature that propose theories as to how γ’ precipitates are sheared by dislocations and how stacking faults (SISF, SESF, and CSF) are formed as a result. These theories were primarily based on experimental TEM results of crept superalloy materials.

Strengthening mechanisms in superalloys is a well-documented subject matter. What requires more attention is the interpretation of these deformations mechanisms as it relates to the creep rate limiting processes that control deformation. Although defect structure analysis is present in the literature for the mechanisms reported, there seems to be a lack of completeness in the characterization sense. For each mechanism observed in a crept deformed specimen, a complete experimental TEM analysis should be conducted to absolutely determine the type and nature of stacking faults that are responsible for
matrix and precipitate shearing configurations as well as the dislocations and partial
dislocation responsible for creating the chemical and structural faults. A thorough
analysis should include: conventional two-beam BF imaging, weak beam imaging, and
high-resolution imaging. Each of the recorded images should then be matched with
computer simulated dislocation and stacking faults for unambiguous identification.
Furthermore, there seems to be a need for in-situ TEM deformation studies so that
deformation mechanisms can be observed as they are occurring over real time.

2.7 Research Motivation

The motivation behind the research presented in this dissertation is aimed at exploring the
creep deformation mechanisms in a newer generation Ni-base superalloy that was
specifically developed for turbine disk applications in aircraft gas turbine engines where
it is expected to have the capability of operating at higher temperatures, while
maintaining structural integrity, than currently used turbine disk alloys. Although there
has been a significant amount of research that has been dedicated to understanding the
mechanical properties of this alloy, a fundamental understanding of the mechanisms
responsible for creep deformation has yet to be fully elucidated.
Table 2.1: List of alloying additions and their expected effect in terms of strengthening mechanisms and physical metallurgy of Ni-based superalloys [28, 61, 62].

<table>
<thead>
<tr>
<th>Expected Effect</th>
<th>Alloying Elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solid Solution Strengthening ($\gamma$)</td>
<td>Co, Cr, Fe, W, Mo, V, Ta</td>
</tr>
<tr>
<td>Dispersion Strengthening</td>
<td>ThO$_2$, Al$_2$O$_3$, Y$_2$O$_3$, MgO</td>
</tr>
<tr>
<td>Precipitation Strengthening ($\gamma'$, $\gamma''$)</td>
<td>(Co, Ni)$_3$(Al, Ti, Ta, Nb)</td>
</tr>
<tr>
<td>Grain Boundary Strengthening</td>
<td>Ti, Cr, W, Mo, V, Co, Ta, Zr, Hf</td>
</tr>
<tr>
<td>Oxidation Resistance</td>
<td>Al, Cr, Ta, La, Th</td>
</tr>
<tr>
<td>Grain Boundary Refinement</td>
<td>B, C, Zr, Hf</td>
</tr>
<tr>
<td>Carbides MC</td>
<td>W, Ta, Ti, Mo, Nb</td>
</tr>
<tr>
<td>Carbides $M_2$C$_3$</td>
<td>Cr</td>
</tr>
<tr>
<td>Carbides $M_2$C$_6$</td>
<td>Cr, Mo, W</td>
</tr>
<tr>
<td>Carbides $M_6$C</td>
<td>Mo, W</td>
</tr>
</tbody>
</table>
Figure 2.1: Binary Ni-Al phase diagram [63].

Figure 2.2: Anomalous yield stress behavior of Ni$_3$Al as a function of temperature from Thornton et al [7].
Figure 2.3: Illustration showing a) three \{111\} close packed planes in the L1_2 structure and the effect of shear displacements on b) APB formation c) SISF formation, and d) CSF formation [3].
Figure 2.4: Illustration depicting the shearing action from an array of ordered precipitates by paired dislocation [30].

Figure 2.5: Critical resolved shear stress increase as a function of particle diameter for particles shearing vs. Orowan looping [35].
Figure 2.6: Mechanism of γ′ precipitate shear by a single matrix dislocation and the creation of a) SISF and b) SESF within the γ′ precipitate [40].

Figure 2.7: Mechanism of γ′ precipitate shear by a dissociated matrix dislocation and the creation of a) SISF and b) SESF within the γ′ precipitate [48].
**Figure 2.8:** Schematic illustration depicting the conversion of an APB to a SISF by the nucleation of a Shockley partial on the APB plane [41].

**Figure 2.9:** a) Experimental BF TEM image showing continuous stacking fault contrast in the matrix and b) proposed shearing mechanism that would account for this [64].
Figure 2.10: Schematic of the microtwin formation mechanism a) projection of the (1\bar{1}0) plane of the L1₂ structure b) being sheared by a/6[11\bar{2}] Shockley partials shear on successive \{\bar{1}11\} planes creating a pseudo twin c) atomic reordering and d) resultant true twinned structure [59].
Figure 2.11: BF TEM diffraction contrast and HRTEM imaging used to identify microtwinning and the nature of the twinning partial dislocations during creep of Ni-base superalloy René 88DT at 650°C/838MPa/0.5% strain by Viswanathan et al [54].
CHAPTER 3

A TEM STUDY OF CREEP DEFORMATION MECHANISMS IN ADVANCED DISK NI-BASE SUPERALLOY RENE 104

Abstract

In this study the elevated temperature creep deformation mechanisms of a newer generation polycrystalline Ni-base disk superalloy (Rene 104) was investigated through conventional and high-resolution transmission electron microscopy characterization techniques. It was found that there are distinctly different deformation mechanisms that become operative during creep at temperatures between 677-815ºC and at stresses ranging from 345-724MPa. During creep at 690 MPa and 677ºC, microtwinning was observed to be the prime deformation mechanism. At a slightly higher stress of 724MPa and temperature 704ºC, a mixture of microtwinning and dislocation activity was observed which caused the material to creep more rapidly. With increasing test temperature (760ºC) shorter stacking faults were observed to be a prime deformation mechanism, which was similar to the microtwinning mode in that the faults were determined to be predominately extrinsic in nature. These faults were present in a small amount at 760ºC and were virtually nonexistent at 815ºC. The main creep rate controlling mechanism at 815ºC was determined to be dislocation climb bypass. In addition to the correlation of creep behavior with the deformation mechanisms it was also observed that microstructural evolution occurs rapidly during creep at the highest test temperatures where the secondary γ' has coarsened and the tertiary γ' precipitates have dissolved.
3.1 Introduction

Ni-base disk superalloys are an important class of high temperature structural materials that are most often used in the aerospace industry in the manufacturing of critical hot section engine components in aircraft gas turbine engines [65]. The reason why these alloys are chosen over other candidate materials is mainly because of their inherent capacity to retain strength and resistance to creep, fatigue, and oxidation during service conditions at high homologous temperatures. Such unique properties are achieved through its microstructure which consists primarily of coherent, ordered, L1₂ structured, Ni₃Al based intermetallic γ' precipitates that are embedded in a disordered, face centered cubic (fcc), solid solution (Co, Cr, W, Mo, etc.) γ matrix,

The precipitates themselves are unique in that during high temperature deformation, the γ' precipitates impart resistance to plastic deformation since they act as effective barriers to dislocation motion as high energy atomic configurations would develop if the precipitates were to be sheared by perfect or partial dislocations operating on <110>{111} or <112>{111} slip systems, respectively. Examples of these structural and chemical defects include antiphase boundaries (APB), complex stacking faults (CSF), superlattice intrinsic stacking faults (SISF), or superlattice extrinsic stacking faults (SESF). Much of the detailed mechanistic understanding and interpretation of rate controlling time dependent creep deformations of Ni-base Superalloys have been determined directly from in-situ and/or post mortem transmission electron microscopy (TEM) observations of dislocations and their interaction with the γ' precipitates.

In light of the many creep deformation mechanisms that have been reported during creep of Ni-base superalloys, it is the purpose of this study to perform an in depth
TEM characterization study to determine the deformation mechanisms responsible for creep for a newly developed, powder metallurgy (PM) produced, turbine disk alloy René 104 as a function of stress and temperature. Although there has been a substantial amount of unpublished proprietary research already performed on this alloy, a fundamental investigation of the mechanisms responsible for creep deformation has not yet been fully explored. Knowledge of these deformation mechanisms are highly imperative for aircraft gas turbine material development programs when detailed representation of the fundamental mechanism controlling creep is to be accounted for in creep deformation modeling and disk life prediction studies.

3.2 Materials and Experimental Methods

Advanced disk Ni-base Superalloy ME3 (also known as René 104) is a newer generation powder metallurgy (PM) produced alloy that is intended for use as a turbine disk in aircraft gas turbine engines. Initial alloy design and development commenced in the mid 1990’s as part of NASA’s High Speed Research/Enabling Propulsion Materials (HSR/EPM) program with GE Aircraft Engines and Pratt & Whitney as industrial collaborators [66, 67]. The goal of the HSR/EPM program was to develop an advanced turbine disk alloy that can be utilized in future supersonic aircraft gas turbine engine applications with the capability of extended service durability at temperatures up to 650°C and/or in current gas turbine engines as a replacement turbine disk material of which can outperform existing gas turbine disk alloys such as IN100 and René 88DT in terms of disk life. It is expected that with the combination of powder metallurgy
processing, increased additions of refractory elements and a multimodal $\gamma'$ precipitate size distribution René 104 can be utilized at higher temperatures.

3.2.1 Supersolvus Heat Treatment

The materials used for this study were extracted from scaled up turbine disk forgings that had previously been subjected to a proprietary supersolvus heat treatment. A generalized schematic illustrating a typical supervolvus heat treatment schedule is presented in Figure 3.1. In a typical supersolvus heat treatment, the material is held for a finite period of time at a temperature above the $\gamma'$ solvus then cooled to room temperature, which is then followed by an intermediate stabilization treatment.

The purpose of applying such a heat treatment is to resolutionize all of the non-uniform $\gamma'$ precipitates that are present during the powder consolidation, billet extrusion, forging, and heat treatment operations so that a more uniform dispersion of $\gamma'$ precipitates can form within the grain interiors. A bimodal $\gamma'$ size distribution occurs as a result of the supersolvus heat treatment, where upon the $\gamma'$ precipitates that were initially present during the powder consolidation and hot forging operations (primary $\gamma'$) are resolutionized back into the matrix when given a sufficient period of time at temperature. (Segment 1) The time maintained above supersolvus is however minimized since there can be appreciable grain growth that can occur at these elevated temperatures which can have an affect on the mechanical properties of the material.

After the hold time above the solvus temperature, the material can either be air, fan, or oil cooled in order to tailor the size and distribution of the $\gamma'$ precipitates. (Segment 2) The rate at which the material cools during this segment of the heat
treatment protocol is crucial in that it has a direct effect on the nucleation and growth kinetics of the coarser secondary $\gamma'$ and finer tertiary $\gamma'$ precipitates [68, 69]. Following this step, an intermediate aging heat treatment is generally applied to stabilize the microstructure and also to bring out more tertiary $\gamma'$ precipitates out of the supersaturated solid solution $\gamma$ matrix and/or to coarsen the newly formed tertiary $\gamma'$ precipitates. (Segment 3) Often, the tertiary $\gamma'$ has been designated as aging $\gamma'$ since they can nucleate and grow during the second aging heat treatment.

The actual creep specimens used in this study were extracted from different scaled up turbine disk forgings whereby the locations from which they were taken possessed slightly different cooling rates. Each specimen had an average ASTM grain size of 6.5. From validated finite element modeling (FEM) of the disk forging thermal heat treatments, the range of cooling rates experienced from these creep specimens were from 60-82°C/min. Due to the narrow range in cooling rates, the starting microstructure (i.e. $\gamma'$ precipitate morphology and grain size) for each of the creep experiments were nearly identical. A representative SEM micrograph depicting the bimodal $\gamma'$ precipitate size distribution is presented in Figure 3.2.

3.2.2 Creep Testing

Samples that were extracted from the turbine disk forgings and that were used in this study were provided by GE Aircraft Engines (Evendale, OH). The actual creep testing was performed by MetCut Research Inc. (Cincinnati, OH). After a predetermined level of strain had been accumulated, testing was interrupted in order to evaluate the deformation substructure using TEM characterization techniques. The details of the creep tests along
with some microstructure and processing information is presented in Table 3.1. Following creep deformation, the experimental creep data and specimens were sent to The Ohio State University for TEM deformation substructure characterization. In this study only four specimens out of a larger quantity of creep tests were further down selected for the more in depth characterization studies.

3.2.3 Microstructural Characterization

A variety of electron microscopy characterization techniques were employed to characterize the general microstructural features and creep deformation substructure. Scanning electron microscopy (SEM) was used to characterize the $\gamma'$ precipitate size and volume fraction. Samples were metallographically prepared from the grip ends of the creep specimens and an etchant consisting of a mixture of lactic, nitric, and hydrofluoric acid was applied to selectively etch the $\gamma'$ phase. The remnant structures were, in turn, imaged then analyzed by quantitative image analysis using Adobe Photoshop CS and Fovea Pro 3.0 plug-in. To characterize the post creep deformation substructure, TEM foils were prepared from the gauge length by sectioning the specimens $\sim 45^\circ$ with respect to the tensile axis to make it possible to view deformation activity along crystallographic planes that experience maximum shear stresses. After sectioning, the foils were then ground to a thickness of $\sim 100 \mu m$, slurry drilled, then jet polished using an electrolyte consisting of 10% HClO4 and 90% Methanol at -45°C/15V or an electrolyte consisting of 5% HClO4, 35% 2-n butoxyethanol and 60% Methanol at -45°C/15V. The deformation substructures that formed under the aforementioned creep test conditions were then
characterized using a Philips CM200 TEM for defect analysis, a FEI TF20 Tecnai TEM for bright field STEM imaging, and a FEI Titan 80-300 TEM for HRTEM imaging.

3.3 Results

3.3.1 Creep Deformation Behavior

For this study creep testing parameters (stress and temperature) were chosen so that a preliminary evaluation of the elevated temperature creep response of this alloy can be assessed. Specimens were subjected to test temperatures between 677-815°C, which are above the expected operating temperatures for an actual turbine disk during service. The results of the creep tests are presented in Figure 3.3a-b in plots of plastic strain versus time. The time required to reach the different levels of creep strain were dependent upon the temperature and stress that the specimens were subjected to. For example, when Specimen I was crept at 677°C at 690MPa it took nearly 3750 hours to reach 2.0% creep strain. By raising the test temperature and stress slightly as in the case of Specimen II (704°C and 724MPa) deformation has occurred much more rapidly and had reached 0.4% strain in only 160 hours. Thus is indicative of the strong temperature dependence of creep deformation in this temperature regime. A similar comparison can be made by examining the creep response of Specimen III and IV which was crept to the same stress but at different temperatures, 760°C and 815°C, respectively. Creep at the higher temperature resulted in a much more rapid rate of strain accumulation, particularly beyond several hundred hours. These differences in macroscopic creep behavior can better be explained through correlation with the creep deformation mechanisms, which have been identified using TEM characterization techniques and is presented in the following section.
3.3.2 Microstructural Characterization

3.3.2.1 Microstructural Evolution

A representative micrograph of the pre-crept microstructure was previously presented in Figure 3.1. As can be seen in the starting microstructure, there is a bimodal distribution of \(\gamma'\) precipitates in which there exists a finer tertiary \(\gamma'\) precipitate population that is situated in the \(\gamma\) matrix channels and between the much coarser secondary \(\gamma'\) precipitates. Upon inspection of the \(\gamma'\) precipitate microstructure for the specimens that were crept at 677 and 704ºC, the bimodal \(\gamma'\) precipitate size distribution is still retained following exposure at temperature for times in excess of 3000hrs and 160hrs, respectively. (Figure 3.4a-b, respectively) These results show that the microstructure is relatively stable especially for these two test temperatures, which is approximately 25-50ºC greater than the current temperatures that these materials would experience during actual service conditions.

On the other hand, following the duration of creep at higher temperatures (760ºC and 815ºC) it is quite apparent that exposure at these temperature and for a prolong period of time has led to microstructural evolution of the \(\gamma'\) precipitates whereby slight coarsening of the larger secondary \(\gamma'\) precipitates has ensued while complete (or nearly complete) dissolution of tertiary \(\gamma'\) precipitates has occurred. The bimodal size distribution in this case is however not preserved at 760ºC and 815ºC for 3365 and 140hrs, Figure 3.4c-d, respectively. The clusters of tertiary \(\gamma'\) precipitates that once were situated between the larger secondary \(\gamma'\) are no longer present. Although slight coarsening of the secondary \(\gamma'\) is noticeable, the microstructure also exhibits a much lower density of the finer \(\gamma'\) that once may have been part of the tertiary \(\gamma'\) population, but now have
coarsened to a stable precipitate size. As the tertiary $\gamma'$ begins to dissolve into the matrix, the $\gamma$ channel width and effective spacing between secondary $\gamma'$ precipitates increases. As a result, it should be much easier for dislocations to bow through and/or circumvent the $\gamma'$ precipitate field rather than being forced to cut precipitates in a planar shearing configuration.

The above microstructural observations are important since aircraft engine manufactures would like to increase the temperature capabilities of these alloys in order to improve both engine performance and efficiency. Although the test temperatures selected in this study are much greater than the expected operating temperatures of an actual aircraft engine during service, it is nevertheless necessary to have some basic knowledge concerning the stability of the microstructure. The structural integrity of these components may be compromised if these were to operate under conditions where microstructural evolution is taking place over the life of the engine component. What these results show is that an upper bound service temperature should be placed for these alloys which needs to be accounted for if there were to be a major engine design change that would allow for such materials to run at higher temperatures.

3.3.3 Creep Deformation Structure Characterization

3.3.3.1 Microtwinning

Specimen I (Creep at 677°C and 690MPa)

Diffraction contrast TEM and high resolution TEM (HRTEM) characterization techniques were employed to identify the post creep deformation mechanism of Specimen I that was crept to 2.0% plastic strain at 677°C and 690MPa in 3750hrs. When
the deformation substructure is viewed using conventional diffraction contrast imaging, a homogeneous distribution and a high number density of continuous planar deformation features that shear through both the $\gamma$ matrix as well as the $\gamma'$ precipitates are observed of which is shown in Figure 3.5. These deformation structures do traverse the entire length of grain, from grain boundary to grain boundary, and then slip onto adjacent grains or across annealing twins as in the grain shown in this image. It is evident that these are microtwins when characterized using high-resolution transmission electron microscopy (HRTEM). Figure 3.6 shows a series of HRTEM images of microtwins of varying thickness that are present in the $\gamma$ matrix phase. The twins are referred to as microtwins since these twin displacements are on the order of several atomic layers thick.

More conclusive evidence, proving that these faulted structures are microtwins, was determined by selected area electron diffraction analysis. In this tilting experiment a grain was selected that was not too far from the [011] zone axis and had a number of microtwins present that were oriented edge on. A selected area aperture was placed over the area and an electron diffraction pattern was acquired. There were actually two slip systems that were active in this particular grain. An image of the microtwins viewed in this orientation and a selected area diffraction pattern acquired from the [011] zone axis is presented in Figure 3.7a-b, respectively. When the reflections are indexed, fundamental reflections corresponding to the FCC $\gamma$ matrix and superlattice reflections corresponding to the $\gamma'$ precipitates are clearly recognizable. Aside from the expected (200), (111), and (220) type fundamental and (100) type superlattice reflections, there are additional twin reflections that are present, which gives further conformation that these planar faulted structures are microtwins.
In addition to conventional diffraction contrast imaging and electron diffraction analysis, HRTEM was used to study the microtwins as it passes through the ordered, L1$_2$ structured $\gamma'$ precipitate. An unfiltered HRTEM image of a 15 atomic layer thick microtwin present within a coarse secondary $\gamma'$ precipitate and viewed along a [011] orientation is presented in Figure 3.8. A fast fourier transform (FFT) of the HRTEM image, which is analogous to a diffraction pattern (inset), shows that there are reflections that correspond to the expected fundamental and twin-related reflections in the same manner as the SADP pattern and electron diffraction analysis presented in Figure 3.8. Superlattice reflections of the (100) and (110) type are also present in the FFT which gives an indication that the $\gamma'$ precipitate possess the ordered L1$_2$ structure. Furthermore, it is apparent that there is faint ordering contrast along the (100) subplanes. The significance of these observations in relation to how microtwins can form in $\gamma'$ strengthened Ni-base superalloys will be explained further in more detail in a later section.

Microtwinning is the prevalent deformation mechanism in all grains examined, as it was rare to observe any other defect structures such as dislocation activity within the matrix or near grain boundaries. Since this particular creep specimen was carried out to such high strain and that the twins extend across individual grains and propagate through to adjacent ones, it was extremely difficult to find discrete locations where the microtwins have initiated and/or terminated. This is the focus of an additional study aimed at tracking the nucleation and evolution of microtwins with increasing plastic deformation later Chapter 5.
Specimen II (Creep at 704°C and 724MPa)

When crept at a slightly higher temperature and stress, as in the case of Specimen II at 704°C and and 724MPa, several deformation mechanisms were observed to be operating simultaneously: microtwins emanating from grain boundaries and dislocations near grain boundaries. What can be seen when viewed using conventional BF TEM imaging are highly planar, continuous stacking fault shearing configurations, extending through $\gamma$ and $\gamma'$ in the same manner in which microtwins were observed in the previous case with the exception that they are not present to a high degree and they do not extend across entire grains. (Refer to Figure 3.9) Along the length of the continuous stacking faults it can be seen that there are changes in contrast due to the presence of partial dislocations that are on the same octahedral \{111\} type glide planes or on parallel 111 planes. The primary deformation mechanism therefore appears to be microtwinning just as in the case of the previous specimen, which was crept at a lower temperature and stress.

A higher magnification view and analysis of the stacking faults comprising a select microtwin is presented in complimentary BF/DF TEM images using the (±200) reflection. (Figure 3.10) The partial dislocations that are responsible for creating the faulted structures in the matrix and $\gamma'$ precipitates are marked by arrows. In the wake of the partials, layered stacking faults are clearly distinguishable by the change in contrast of the fault fringes. Also apparent are regions where there is a periodicity in stacking fault contrast, which is indicative of the microtwinning mechanisms.

The nature of the stacking fault was determined by the asymmetric contrast in BF and DF imaging conditions. In the DF $g\overline{2}00$ image (Figure 3.10) when the diffraction vector is placed at the center of the stacking fault, the fault is extrinsic in nature when it
points towards a bright outer fringe and intrinsic in nature when it points toward a dark outer fringe [70]. In regions where the stacking fault is continuous through $\gamma$ and $\gamma'$, the fault was determined to be extrinsic; a detail that has been reported as a characteristic of a microtwin whose twinning partial dislocations are of the same type (a/6<112> Shockley partials) and are on adjacent {111} planes [54].

When the stacking faults were tilted in the TEM to edge on where the feature was parallel to the incident beam and on a [011] zone axis, fundamental and superlattice reflections were present; however, twin reflections could not be seen. A primary reason why twin reflections could not be observed for this case is that the microtwins were in the early stages of formation and have not thickened substantially such that diffraction from the twinned regions could be detected. Nevertheless, considering all this data enables us to concluded that microtwinning is one of the operative mechanisms for Specimen II as well.

Aside from microtwinning, there also exists a high density of dislocation activity in the $\gamma$ matrix that appear not to be shearing the $\gamma'$ precipitates. These dislocations are found throughout grain interiors but are mainly concentrated along grain boundaries. (Refer to Figure 3.9) The combination of the thermally activated microtwinning mechanism in conjunction with dislocation activity help to explain the faster creep rates when compared to Specimen I, where microtwinning alone was observed.
3.3.3.2 Extended Stacking Fault Shear

Specimen III (Creep at 760°C and 345 MPa)

The deformation substructures that formed during creep at 760°C and 345MPa are different from the microtwins described above. A representative TEM image of the common defect structures that are observed can be seen in Figure 3.11. The image depicts shearing configuration in which planar continuous faults are observed to traverse both matrix and precipitates. The stacking faults do not transcend across entire grains such as in the case of microtwinning. These deformation features are much shorter in length and are heterogeneously distributed within grains. They typically span only a few micrometers in width. In addition to these stacking fault shearing configurations, a/2<110> dislocations tend to coexist and are present within the matrix where there are often observations that these dislocations are locally dissociating into partial dislocations. Complementary BF and DF TEM analysis of the stacking faults indicate that most that the faults present with the γ′ precipitates are superlattice extrinsic stacking faults (SESF). (Refer to Figure 3.12a-b)

3.3.3.3 Thermally Activated Climb/Bypass

Specimen IV (Creep at 815°C and 345 MPa)

A different deformation mechanism was observed in the specimen crept at the highest test temperature. There are a few occurrences of the stacking fault related precipitate shearing mechanism; however, the prominent deformation feature is now due to dislocation activity, which is shown in Figure 3.13. The dislocations appear to be in climb/bypass mode whereby the dislocations are bypassing the γ′ precipitates by a
thermally activated climb mechanism. Diffraction contrast TEM analysis was performed on a select area that showed unpaired dislocations that are clearly present in the $\gamma$ matrix, unpaired dislocations that seem to be situated above or below $\gamma'$, and dislocation loops that appear to be encircling the $\gamma'$ precipitates. (The dislocations analyzed are highlighted with a box.) The dislocations themselves were identified as $a/2\langle011\rangle$ type dislocations using the $g \cdot b$ invisibility criteria as shown Figure 3.14. The dislocations are visible for $g=\overline{111}$ and $g=\overline{111}$ but are invisible for $g=\overline{311}$ and $g=200$ and so based on the invisibility the Burgers vectors of these dislocations were determined to be $a/2[011]$.

3.4 Discussion

3.4.1 Deformation Mechanism Mapping

In the creep deformation literature for superalloys, there has not yet been a systematic study of the effects of microstructure, stress, and temperature on the underlying creep deformation mechanisms for a range of microstructures and test conditions. There is a particular deficiency of this information for newly developed Ni-base superalloys. Stemming from the identification of creep mechanisms presented in this study, as well as in other creep deformation mechanism studies reported in literature, a deformation mechanisms map was constructed which shows the operative deformation mechanisms as a function of stress and temperature. The deformation mechanism map, presented in Figure 3.15, presents the post creep deformation mechanisms that have been identified in this study, as well as similar disk alloys studies on Rene 88DT [54]. It does not capture the influence of grain size, precipitate size/distribution, alloy chemistry nor the effect of strain rate even though such factors known to be important and contributing factors that
control deformation behavior. The map was constructed to provide a qualitative representation of the operative creep mechanisms as a function of stress and temperature.

At low temperature and high stress it is expected that dislocation coupled \(a/2<110>\) APB shearing of the \(\gamma'\) precipitates is the dominant deformation mechanism. In this mechanism, the dislocations travel in pairs since shearing of the \(\gamma'\) precipitates by the first \(a/2<110>\) dislocation would cause an APB to form. The order would be restored however, when the secondary \(a/2<110>\) dislocation shears the precipitates. Since the \(a/2<110>\) dislocations are perfect dislocations that are in the \(\gamma\) matrix, they do not leave a fault behind in the matrix or in the precipitate. It is noted that the APB shearing mechanism has been accounted for in many dislocation based models of particle shearing of ordered precipitates [31-33, 35, 36].

This mechanism has not been experimentally observed in this study since it is likely that creep experiments were not performed in the temperature and stress regime where this mode of deformation can occur. Moreover, this mechanism may not occur for a bimodal \(\gamma'\) precipitate size distribution. At high temperatures, thermally activated dislocation bypass by unpaired \(a/2<110>\) dislocations is occurring. Dislocation climb is therefore the likely creep rate controlling deformation mechanisms provided that enough thermal activation is available to allow dislocations to find alternative pathways to circumvent the \(\gamma'\) precipitates. It should be noted that the temperature at which this mechanism is observed to occur is well beyond conditions in which these materials will be expected to perform.

It is however within the intermediate temperature and stress regime where there are deformation modes that are not presently well understood. These mechanisms result
in matrix and precipitate shearing modes, which leave stacking faults and microtwins in their wake. In the following sections, the creep rate limiting processes will be described in more detail for these creep mechanisms.

3.4.2 Microtwinning Mechanism

Honeycombe [71] describes twinning as the cooperative movement of atoms by macroscopic shear with a crystallographic shift of the lattice in the twinned region, across the twinning plane, being a mirror image of the parent lattice. The packing sequence in an F.C.C. structure on the octahedral plane 111 has the packing sequence of ABCABCABC and when the lattice is sheared in the <112> direction the structure now possess ABCABACBA stacking. Deformation twinning is known to occur in metals possessing bcc and hcp crystal structures due to the limited number of available slips systems. However, in fcc metals that posses a greater number of slip systems, deformation by twinning has not been considered to readily occur except under low temperature, high strain rate conditions, or when the twinning stress is less than the stress required to initiate slip [50].

Nonetheless, twinning has been observed in Ni-base superalloys during high temperature creep deformation whereby the strain rate under these conditions are orders of magnitude lower. Guimier and Strudel have reported that microtwins were present after at temperatures above 500°C in a low volume fraction Waspaloy [72]. It was suggested that microtwins form by the motion of a/3<112> type superlattice Shockley partial dislocations that are operating on adjacent {111} glide planes by a pole
mechanism since the ordered L1₂ structure of the γ′ precipitates was preserved even after twinning.

Ardakani et al [51] have shown that microtwins form in single crystal Ni-base Superalloy SRR99 when crept in tension at 750ºC and 850MPa along the [011] crystallographic orientations. Although they did not provide a mechanism for twin formation, they did mention that it is likely that the twins formed by the dissociation of a a/2[01T] dislocation into a a/6[112] Shockley partial dislocation and a a/3[12T] superlattice Shockley partial dislocation where the a/3[12T] is responsible for creating the twin.

Kakehi [56] also observed microtwinning during creep at 700ºC and 820MPa on an experimental Ni-base Superalloy with a multimodal γ′ precipitate size distribution. Microtwins were reported to nucleate with the larger γ′ precipitates. Kakehi adopted the mechanism proposed by Guimier and Strudel stating that shear by a/6<112> Shockley partials is highly unlikely because the ordered L1₂ structure would be destroyed as this would result in a higher energy configuration with incorrect nearest neighbor atomic bonds. The scientific reasoning behind this is valid, since shear by successive a/6<112> Shockley partials does indeed create a CSF with wrong nearest neighbor bond violations. The passage of a/3<112> partials would however preserve the L1₂ structure.

In contrast, Knowles and Chen [52] have shown through detailed TEM work on creep mechanisms of CSMX-4 that twins are almost always associated with superlattice extrinsic stacking faults (SESFs) which suggested that microtwins may be produced by an entirely different mechanism than the mechanisms presented above. In a review by Christian and Mahajan [58] deformation twinning in L1₂ superlattice structures would
result in the production of a pseudotwin that possesses an imperfect superlattice with incorrect atomic ordering. Atomic re-shuffling would thus be required to convert the pseudotwin structure to that of a true twinned structure with correct nearest neighbor atomic bonds.

Kolbe was the first to propose a diffusion-based crystallographic model that accounts for thermally activated atomic reordering to produce a true twin structure from a CSF in the $\gamma'$ phase in Ni superalloys. Assuming the microtwins formed by successive shear of $a/6<112>$ Shockley partials along adjacent $\{111\}$ planes, a CSF (pseudotwin) would result in the $\gamma'$ phase possessing a lower symmetry orthorhombic phase and a true twin structure in the fcc $\gamma$ matrix phase. The atomic reshuffling steps are required to convert the pseudotwin to a true twin and it can be argued that this diffusion process can be facilitated at elevated temperatures such as those incurred during high temperature creep. Although not thoroughly substantiated through direct TEM evidence, Viswanathan et al [54] provided experimental evidence of the microtwin formation process through diffraction contrast (BF and WB) and high resolution imaging experiments on crept René 88DT at $650^\circ$C/838MPa/0.5% strain. BF and WB microscopy techniques were utilized to determine the sign and nature of partial dislocations, which were determined to be $a/6<112>$ partials of the same sign. High-resolution TEM imaging shows a twinned structure that transcended through the $\gamma$ matrix and $\gamma'$ precipitate phases. Their results convincingly show that microtwin propagate as result of successive shear by $a/6<112>$ partials on adjacent $\{111\}$ planes.
3.4.3 Modeling Microtwinning

Based on experimental observations and detailed TEM characterization of the microtwinning deformation mechanisms that was reported for Ni-base superalloys Rene 88DT during creep at 650ºC and 838MPa [54] and Ni-base superalloy Rene 104 during creep at 704ºC and 724MPa [73], a quantitative creep model was developed by Karthikeyan et al [74]. The model takes into account that microtwins formed by the cooperative motion of paired a/6<112> Shockley partial dislocations, which shear both \( \gamma \) and \( \gamma' \) on \{111\} type close packed planes. Due to the ordered nature of the L1_2 structure \( \gamma' \) precipitates, initially the order is destroyed and a high-energy 2-layer complex stacking fault (pseudotwin) is produced as the precipitates are sheared by these paired Shockley partial dislocations.

Facilitated by creep at elevated temperatures, atomic reordering in the wake of the leading partials would revert the pseudotwin to that of a true twinned structure with rearrangement of Ni and Al atoms to the ordered L1_2 arrangement as originally proposed by Kolbe [59] It can be argued that this diffusion based reordering process is the creep rate limited process and has such formed the basis of the microtwinning model proposed by Kathikeyan et al [54]. The following paragraphs summarize the essence of this microtwinning creep model with an emphasis on how microstructural characterization techniques were used to provide direct inputs into the model.

The basis of the microtwinning creep model utilizes the Orowan expression, which relates strain rate to the magnitude, velocity, and mobile dislocation density of the twinning partial dislocations according to the following expression:
\[ \varphi = \rho_{tp} \cdot b_{tp} \cdot \nu \]  

where \( \rho_{tp} \) is the density of mobile twinning partials, \( b_{tp} \) is the Burgers vector of the twinning partial dislocation \((a/6<112>)\), and \( \nu \) is the dislocation velocity.

The main factor in the development of the microtwinning model rests upon an understanding of the reordering kinetics of the process, which ultimately limits the mobility, or the velocity, of the twinning partial dislocations. Considering that a two-layer fault (or pseudotwin with incorrect nearest neighbor bonds) formed in the wake of the first two Shockley partials, the fault energy of is \( \Gamma_{pt} \). Reordering reduces the energy of the two layered fault. If the Shockley partials were to shear the \( \gamma' \) slowly, then there is sufficient time for reordering to take place, eventually to that of a true twin, \( \Gamma_{tt} \).

Karthikeyan assumed that the energy drop takes place exponentially with time such that:

\[ \Gamma(t) = (\Gamma_{pt} - \Gamma_{tt}) \exp(-Kt) + \Gamma_{tt} \]  

[2]

where \( K \) is a constant determining the reordering rate and \( t \) is the time. From a work balance approach Karthikeyan developed an expression for the velocity of the twinning partial dislocations that takes the reordering kinetics into account:

\[ \nu = \frac{D_{ord} \cdot (b_{tp} / x^2)}{\ln \left[ \frac{f_2 \cdot \Delta \Gamma}{2 \cdot \tau_{eff} \cdot b_{tp} - f_2 \cdot \Gamma_{tt}} \right]} \]  

[3]
When substituted into the Orowan strain rate equation the following expressing results is given by:

\[
\varphi = \rho_{tp} \cdot b_{tp} \cdot \nu = \rho_{tp} \cdot b_{tp} \frac{D_{ord} \cdot (b_{tp} / x^2)}{\ln \left[ \frac{f_2 \cdot \Delta \Gamma / (2 \cdot \tau_{eff} \cdot b_{tp} - f_2 \cdot \Gamma_n)}{2} \right]}
\]

where \( \rho_{tp} \) is the density of mobile twinning partials, \( b_{tp} \) is the Burgers vector of the twinning partial, \( D_{ord} \) is the diffusion coefficient for ordering and \( x \) is the short range diffusion length (assumed to be several nearest neighbor distances, or \( \sim 2 \cdot b \)), and \( f_2 \) is the volume fraction of the secondary \( \gamma' \) precipitates. The effective stress, \( \tau_{eff} \), in the presence of tertiary \( \gamma' \) precipitates, is given by:

\[
\tau_{eff} = \tau - \frac{f_2 \cdot \Gamma_{pt}}{2 \cdot b_{tp}}
\]

The experimental values of parameters such as dislocation density \( \rho_{tp} \), volume fraction of the secondary \( \gamma' \) precipitate were determined directly from TEM observations.

The twinning partial dislocation density was determined through diffraction contact TEM images from Specimen II, which was crept at 724MPa and 704ºC. The measured number of twinning partial dislocations per length of the microtwin was \( 3.8 \times 10^5 \) m\(^{-1}\) and the number of microtwins per cross section height was \( 4.4 \times 10^5 \) m\(^{-1}\). To
obtain the density of twinning partial dislocations the number of twinning partials per microtwin was multiplied by the total number of microtwins analyzed per cross section height which turned out to be $1.66 \times 10^{11} \text{ m}^{-2}$. Additional parameters that were experimentally measured were the change in volume fraction of secondary and tertiary $\gamma'$ precipitates from the starting microstructure to the final microstructure following creep deformation. The fraction of secondary $\gamma'$ varied from 0.34-0.38 while the fraction of tertiary $\gamma'$ varied from 0.06-0.02 from the initial and final microstructures analyzed. The creep model predictions are in reasonable agreement with experimental results. Published analysis of the results can be seen in a paper published by Karthikeyan et al [74].

These results suggest that the reordering kinetics that is associated with the twinning partial velocity is reasonable. Insight from more recent findings however shows that there may be additional factors that potentially could influence the reordering kinetics. HRSTEM imaging of microtwins formed in the sample crept to 2.0% strain at 677ºC and 690MPa reveals that there is segregation of heavy elements taking place at the microtwin interface [75]. This is of course would have an effect on the mobility of the twinning partial dislocations and the reordering process that occurs in the wake of the shearing partial dislocations whereby, the heavier elements have slower diffusion kinetics and will ultimately lead to longer times for the reordering process to occur.

3.4.4 Extended Faulting

The shorter stacking faults that were observed during creep at higher temperature and low stress (760ºC and 345MPa) were determined to be extrinsic stacking faults and are
strikingly similar to the planar faulted structures characterized by Zhang et al [76] and Decamps et al [64]. The Ni-base superalloy studied by Zhang et al (Haynes C263) had been crept at 800°C and stress between 120-250MPa. Their detailed TEM analysis showed that the planar stacking fault related shearing of the γ matrix and the γ' precipitates were a direct result of a dissociation of a a/2[011] dislocation into a a/3[112] and a/6[211] partial dislocations. It was suggested that the leading a/3[112] partial dislocation sheared both the γ matrix and the γ' precipitate, which left the extrinsic stacking fault in its wake. As the trailing a/6[211] Shockley partial dislocation attempts to keep up with the leading a/3[112] superlattice Shockley partial dislocation it would remove the stacking fault in the γ matrix since the passage of a a/3[112] and a/6[211] on the same glide plane is basically the same as passing a a/2[011] dislocation is a perfect dislocation in the γ matrix and would leave no stacking fault as it deforms the matrix. The trailing a/6[211] dislocation does not shear the γ' precipitates since it would create a high energy APB. Instead the trailing Shockley partial dislocation would leave a Shockley partial loop around the precipitates. The total length of the stacking fault shearing configuration would thus be related to the mobility of the leading and trailing partial dislocations. This mechanism provides a reasonable explanation for the formation of stacking fault structures observed in Specimen III, which was characterized in this study, since there are plenty of a/2<110> type matrix dislocations present within the local vicinity of the stacking faults.

Another possible mechanism for SESF formation in the γ' precipitates has been proposed by Decamps et al [64] in which SESFs are formed in the γ' precipitates themselves or continuous stacking faults transcends both γ and γ'. This work was
stimulated by experimental observations of the creep deformation substructure of superalloy NR3 following creep at 700°C and 650MPa. The authors proposed that the SESF’s are formed by the decorrelated motion of Shockley partial dislocations that are dissociated from a single $a/2<110>$ dislocation. The leading Shockley partial dislocation is proposed to shear the $\gamma'$ precipitates and create a complex stacking fault. Due to the relatively high energy of the CSF, the authors proposed that it would be energetically favorable to nucleate a Shockley partial dislocation a plane above or below the CSF. The main concern with this model the nucleation of the Shockley partial dislocation would not maintain the correct atomic ordering. A two layer CSF would be created in the $\gamma'$ precipitates and only after atomic reordering process has occurred then the fault can be converted to a SESF. This reordering hypothesis appears to have been overlooked by Decamps et al [64] This leading shearing configuration involving a two layer fault is similar to the microtwinning mechanism as proposed by Kolbe in which an atomic reordering process was realized [59].

3.4.5 Effect of Microstructural Evolution on Creep Behavior

As discussed in the preceding section, dissolution of the finer tertiary $\gamma'$ precipitates had occurred for the duration of the creep tests at 760 and 815°C. With the microstructural evolution that is taking place, the benefits of having the bimodal $\gamma'$ size distribution for improved creep resistance dissipates. It has been shown by Locq et al that the presence of the finer scaled tertiary $\gamma'$ precipitates play an essential part in governing the creep resistance and underlying deformation mode [77]. When the polycrystalline PM disk alloy NR3 is given a standard supersolvus heat treatment which results initially in a
bimodal $\gamma'$ microstructure, planar \{111\}<112> slip involving shearing of the $\gamma'$ precipitates occurs and tends to be the predominate deformation mode during creep at 700°C and at two different stress levels (500 and 650MPa).

A dramatic difference in deformation behavior was noticed when the material is given an additional age at 800°C for 500 hrs, during which the secondary $\gamma'$ were reported to coarsen by approximately 30% while the tertiary $\gamma'$ has completely dissolved back into the matrix and most likely contributed to coarsening of the secondary $\gamma'$ by an Oswald ripening mechanisms. Following creep for the monomodal $\gamma'$ precipitate distribution at the same conditions of temperature (700°C) and stress (500 and 650MPa), the creep rate is much more rapid where plastic deformation is governed by a/2<110> dislocation activity that deform only in the matrix by climb and glide processes. In their research, the more creep resistant microstructure is thus a structure that has a bimodal $\gamma'$ distribution and their interpretation was that if the tertiary $\gamma'$ precipitates were present then they would offer an addition impediment to dislocation motion and actually force the dislocations to shear the precipitates in a stacking fault related shearing mechanism as originally proposed by Decamps et al [64].

3.5 Conclusion

A TEM investigation aimed at exploring the effect of stress and temperature on the creep deformation mechanisms of a newer generation advanced Ni-base disk superalloy was performed. To evaluate the high temperature capabilities of this alloy, creep experiments were conducted at stresses and temperatures that are above the normal conditions experience during service. Transmission electron microscopy characterization methods
were used to identity and correlate the deformation mechanisms to the macroscopic creep behavior. The following conclusions can be drawn from this study:

1) A variety of different deformation mechanisms were found to be operative during creep deformation at temperatures and stress ranging between 677-815°C and 345-724MPa. The results were used to create a creep deformation map that presents the creep mechanism as a function of stress and temperature.

2) Microtwinning was found to be a dominant deformation mechanism during creep at an intermediate temperature and stress. The microtwins propagate by the motion of paired and identical a/6<112> Shockley partial dislocations and the creep rate limited process is presumed to be a diffusion based atomic reordering process that restores the order of the L12 structured γ’ precipitates.

3) The combination of high temperature creep and low stress resulted in planar stacking fault shearing of γ and γ’ which left behind a SESF in the γ’ precipitates. The results were compared to several models proposed in literature and it seems likely that the SESF were created by the dissociation of a a/2<110> dislocation into a a/3<112> and a/6<112> Shockley partial dislocations.

4) Thermally activated climb/bypass was observed at the highest creep temperature where unpaired a/2<110> dislocations were observed to be bypassing the γ’ precipitates by a climb mechanism.

5) Microstructural evolution has taken place during creep at test temperatures of 760°C and 815°C whereby the larger secondary γ’ precipitates have coarsened and the finer tertiary γ’ precipitates have dissolved.

6) This work suggests that in order to develop sound, physically based models that are to be used to predict the creep behavior of Ni-base disk superalloys will require multiple deformation mechanism to be operative in parallel and can be activated through changes in stress and temperature. In addition, the models must include and account for microstructural evolution (coarsening and/or dissolution) of the γ’ precipitates.
Table 3.1: Creep test specimen I.D. and experimental creep testing conditions.

<table>
<thead>
<tr>
<th>Sample I.D.</th>
<th>Cooling Rate (°C/min)</th>
<th>Temperature (°C)</th>
<th>Stress (MPa)</th>
<th>Creep Strain (%)</th>
<th>Time (hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen I</td>
<td>62</td>
<td>677</td>
<td>690</td>
<td>2.0</td>
<td>3730</td>
</tr>
<tr>
<td>Specimen II</td>
<td>86</td>
<td>704</td>
<td>724</td>
<td>0.4</td>
<td>160</td>
</tr>
<tr>
<td>Specimen III</td>
<td>60</td>
<td>760</td>
<td>345</td>
<td>0.36</td>
<td>3365</td>
</tr>
<tr>
<td>Specimen IV</td>
<td>45</td>
<td>815</td>
<td>345</td>
<td>0.42</td>
<td>142</td>
</tr>
</tbody>
</table>
Figure 3.1: Schematic illustration of a typical supersolvus heat treatment that is applied to turbine disk alloys in order to develop a multimodal distribution of γ′ precipitates.

Figure 3.2: Representative SEM micrograph depicting the bimodal γ′ precipitate size distribution that develops following a supersolvus heat treatment in Ni-base disk superalloy Rene 104.
Figure 3.3: a) Experimental creep curves of plastic creep strain vs. time plotted for specimens crept under the following conditions: i) Specimen I (677°C/690MPa) ii) Specimen II (704°C/724MPa) iii) Specimen III (760°C/345MPa) and iv) Specimen IV (815°C/345MPa). Figure 3.3b scaled up view showing the creep curves at the earlier stages of creep deformation.
Figure 3.4: Representative SEM micrographs of the post creep $\gamma'$ precipitate microstructure for specimens crept at a) 677°C/690MPa/2.0% strain (in 3730hrs), b) 704°C/724MPa/0.4% strain (in 160hrs), c) 760°C 345MPa/0.36% strain (in 3365hrs) and d) 815°C/345MPa/0.42% strain (in 142hrs) which shows an shift in the $\gamma'$ precipitate size distribution particularly during creep at the higher test temperatures (760 and 815°C).
Figure 3.5: BFSTEM image of the creep deformation microstructure from Sample I crept at 677°C and 690MPa to 2.0% showing planar faulted structures that shear through both γ matrix and γ’ precipitates.
Figure 3.6: HRTEM images of microtwins in the γ matrix of Sample I crept at 677°C and 690MPa to 2.0% at varying thickness. (Images courtesy of L. Kovarik)
Figure 3.7: a) BF TEM image of the microtwins viewed nearly edge on and oriented about the [011] zone axis and b) indexed SADP showing the presence of twin reflections and confirmation that the observed planar deformation structures are microtwins.
**Figure 3.8:** HRTEM image of a microtwin in a secondary $\gamma'$ precipitate as viewed along the [011] zone axis. The insert fast fourier transform shows the presence of fundamental, twin, and superlattice reflections of the $\gamma'$. (Image courtesy of L. Kovarik)
**Figure 3.9:** BF TEM micrograph showing the deformation substructure of the specimen crept to 0.4% strain at 704°C and 724MPa/0.4% strain. Continuous stacking faults transcending $\gamma/\gamma'$ are emanating from grain boundaries and dislocations are concentrated near grain boundaries.

**Figure 3.10:** Bright field and dark field TEM micrographs showing the stacking fault contrast of a microtwin a) BF $g200$ b) DF $g200$ c) BF $g200$ and d) DF $g200$. 
Figure 3.11: BF TEM micrograph of the creep deformation substructure following creep at 760°C and 345MPa showing mixture of a/2<110> dislocations and γ' precipitate shearing configurations involving stacking faults.
Figure 3.12: a) BF and b) CDF TEM micrographs of the stacking faults in the γ′ precipitates for the sample crept at 760°C and 345MPa imaged with g=111. Note: the direction of the g vector when placed at the center of the stacking fault points towards the bright outer fringe, indicating the faults are extrinsic in nature.
**Figure 3.13:** BF TEM micrograph showing the typical deformation substructure formed during creep deformation at 815°C and 345MPa where the primary deformation mechanism is a/2<110> dislocation climb/bypass.
Figure 3.14: Series of BF TEM micrographs of the dislocation structure following creep at 815°C and 345MPa that were used to identify the Burgers vector of the dislocations: a) \( g = \{111 \} \), \( B = [1\bar{1}2] \) b) \( g = 1\{1\bar{1}\} \), \( B = [01\bar{1}] \) c) \( g = 3\{1\bar{1}1\} \), \( B = [11\bar{2}] \) and d) \( g = 200 \), \( B = [01\bar{1}] \).
Figure 3.15: Ni-base disk superalloy creep deformation mechanism map created by the results found in this study of Rene 104 and by Viswanathan et al [54] in Rene 88DT.
CHAPTER 4

EFFECT OF MICROSTRUCTURE ON CREEP DEFORMATION
MECHANISMS AND SUBSTRUCTURE EVOLUTION

Abstract
The influence of microstructure on creep rate controlling deformation mechanisms has been investigated through a combination of creep experiments and TEM deformation mechanism characterization on a newer generation Ni-base disk Superalloy (Rene 104) with particular emphasis placed on the role of secondary and tertiary γ' precipitate size and γ channel width. A direct comparison was made by testing specimens with the different microstructures at the same stress (724MPa) and temperature (677ºC). Furthermore, the evolution of the deformation substructure was studied by interrupting creep tests at 0.05, 0.2 and 1.0% strain then analyzing the deformation microstructure. It was found that the less creep resistant microstructure possessed a greater secondary γ' size, wider γ channel width, and higher volume fraction of tertiary γ'. Deformation in this microstructure commences by way of a/2<110> dislocations that are concentrated in the γ matrix at lower strains, which then transition to a SISF related precipitate shearing mode at larger strains. The more creep resistant microstructure possessed a finer γ channel width spacing, which promoted a/2<110> dislocation dissociation into a/6<112> Shockley partials at lower strains and microtwinning at higher strains.
4.1 Introduction

The structural components within the hot section of aircraft gas turbine engines, such as turbine disks, must be able retain structural integrity during extreme operating conditions where they are subjected to temperatures in excess of 600ºC and complex stress states throughout the disk cross section and where the disk is attached to other components such as turbine blades. For this reason, Ni-base superalloys are the material selected for such applications since they possess the inherent capacity to retain strength and resistance to creep and fatigue particularly at high homologous temperatures. These unique material properties are attained primarily through precipitation strengthening by coherent, ordered, Ni$_3$Al based intermetallic γ’ precipitates that are embedded in a disordered, solid solution, face centered cubic (fcc) γ matrix. Since these turbine engine components operate in a temperature and stress regime where creep deformation is of utmost concern, there have been a number of detailed TEM characterization studies aimed at identifying creep deformation mechanisms and the implications that they may have on the macroscopic creep deformation response of the alloy for a range of microstructures, test conditions, and for different single crystal blade and polycrystalline disk alloy chemistries.

Alloy chemistry and microstructure are two important factors that govern the creep resistance of a polycrystalline Ni-base disk superalloy. Aside from alloy chemistry, much of the desired mechanical properties are attributed to the size scale and distribution of the γ’ precipitates, which are the prime strengthening constituent in this class of materials. The macroscopic creep response and underlying creep rate controlling deformation mechanism can be altered considerably simply through modification of the γ’ precipitate microstructure, which can be tailored through heat treatments. During the
final heat treatment operation in turbine disk alloys the material is given a supersolvus heat treatment that will yield a bimodal size distribution of $\gamma'$ precipitates. During his treatment the material is heat treated above the $\gamma'$ solvus for a predetermined amount of time that is sufficient to dissolve the primary $\gamma'$ precipitates that are present during the hot forging operations. It has been well established that the rate at which the material cools from the $\gamma'$ solvus hold period has a direct effect on the $\gamma'$ precipitate size scale, distribution, and morphology [69]. A slower cooling rate will result in a much coarser secondary and tertiary $\gamma'$ precipitates and vice versa for a faster cooling rate.

There have been reports on the effect of processing conditions on creep deformation behavior in Ni-base disk superalloys where it has been shown that a direct relationship exists between $\gamma'$ size scale and distribution (influenced by the heat treatment), creep behavior, and operative deformation mechanism. In creep studies of Rene 88DT by Viswanathan et al [54], it was shown that during creep at the same test condition (650ºC and 838MPa), a slow cooled microstructure, consisting of a coarser secondary and tertiary $\gamma'$ precipitates resulted in an isolated faulting deformation mode where the secondary $\gamma'$ precipitates were sheared in manner which resulted in superlattice extrinsic stacking faults in the precipitates and corresponded to the lesser creep resistance microstructure and deformation mode [54]. The faster cooled microstructure, consisting of finer secondary and tertiary $\gamma'$ precipitates, resulted in microtwinning and corresponded to a much more creep resistant microstructure and deformation mode.

Dubiez-Le Goff has reported on the effect of subsolvus vs supersolvus heat treatment on creep behavior and creep deformation mechanisms for Udimet 720 during creep deformation at 650ºC and for varied levels of stress (600-900MPa) [78]. Depending
on how the material is heat treated, the microstructure can be tailored for strength and low cycle fatigue or creep resistance. The subsolvus heat-treated material consisted of fine grained microstructure with primary, secondary, and tertiary $\gamma'$ precipitates while the supersolvus heat treated microstructure consisted of a coarse grained microstructure with only secondary and tertiary $\gamma'$ precipitates. In all test conditions the more creep resistant microstructure corresponded to the subsolvus heat treated material.

The precipitate size distribution also has an effect on the creep behavior. During creep of NR3 at 700°C and 500, 650MPa by Locq et al [77], it was shown that a monomodal $\gamma'$ size distribution creeps much more rapidly and it deforms by way of a/2<110> dislocations in a climb/bypass mechanism. In this case, the $\gamma'$ precipitates were not sheared but rather deformation is localized in the $\gamma$ matrix and the precipitates themselves are circumvented. The bimodal $\gamma'$ microstructure on the other hand is more creep resistant and it deforms through precipitate shearing in a manner, which leaves stacking faults in the matrix and/or precipitates.

Aside from the studies summarized above, surprisingly there have been a limited number of systematic studies in Ni-base disk superalloys that have been conducted which specifically address the role of microstructure on creep deformation mechanisms even though the microstructure plays a major role in determining the mechanical properties materials. The purpose of this study therefore is to investigate the influence of microstructure ($\gamma'$ precipitate size scale, size distribution, volume fraction and interparticle spacing (i.e. g channel width spacing) on creep deformation mechanisms through detailed TEM characterization of crept specimens that possessed different starting microstructure features but at the same stress and temperature. Furthermore, in
order to track the deformation substructure as it evolves with increasing deformation specimens were interrupted at varying levels of plastic deformation.

4.2 Materials and Experimental Procedure

A newer generation advanced disk Ni-base superalloy ME3/Rene 104, that was recently co-developed by NASA, GE Aircraft Engines, and Pratt & Whitney was the material chosen for this study [67]. The material was supplied in the form of a turbine disk forging that had been processed using a powder metallurgy route. A final supersolvus heat treatment procedure was applied to the forging, which resulted in the development of a bimodal distribution of γ′ precipitates. In disk alloys, it is known that during a supersolvus heat treatment the rate at which the material cools from the γ′ solvus temperature has a direct effect on the microstructural development of γ′ precipitates, which will ultimately affect precipitate size scale, distribution, and morphology [68, 69, 79]. Due to the differences in cooling rate experienced throughout the cross section of the turbine disk following this heat treatment, different size scale and volume fraction of the coarser secondary and finer tertiary γ′ precipitates have been developed in the outer rim and inner bore locations.

Samples for microstructural analysis were extracted from these regions, mounted in conductive Bakelite, metallographically prepared using progressively finer SiC paper to 1200 grit then given a final 0.05µm colloidal silica polish. Orientation imaging microscopy (OIM) was performed with a Phillips XL-30 ESEM equipped with a TSL EBSD system to measure grain size. Following OIM, samples were subsequently etched using a solution consisting of 2ml HF, 30ml HNO₃, and 50ml lactic acid. This solution
preferentially etches away the $\gamma'$ and accordingly the remnant structures were then imaged with a FEI Sirion SEM using an ultra-high resolution through lens detector. Afterwards, quantitative image analysis using Fovea Pro software was used to measure salient microstructural features (such as precipitate size, volume fraction, and $\gamma$ channel width spacing) that are known to have an influence on creep resistance.

To investigate the influence of $\gamma'$ precipitate morphology of these two different starting microstructures on creep behavior and to characterize the deformation substructure that evolves at different stages of deformation, constant load tensile creep experiments were performed at the same test conditions (677°C, 724MPa, and interrupted at three levels of strain 0.05, 0.2 and 1.0%). Creep specimens, having a cylindrical gauge section measuring 4mm in diameter and 16mm in length, were extracted from the rim and bore regions and were machined using low stress grinding procedures by Metcut Research Inc. The amount of creep strain was measured using an averaging extensometer connected to two linear variable displacement transducers. Temperature was monitored and controlled using a type K thermocouple that was attached to the specimen.

Following creep testing, thin foils for TEM analysis were prepared by extracting sections from the gauge at a 45° angle with respect to the tensile axis. This was done in order to view deformation activity along slip systems that experience maximum shear stress. The foils were mechanically thinned to a thickness of 100µm then slurry drilled to obtain 3mm disks. Finally thinning was conducted on a Struers Tenupol 5 twin jet polishing unit using an electrolyte consisting of 60% methanol, 35% 2-n-butoxyethanol, and 5% perchloric acid at a temperature of -45°C and with an applied voltage of 15V.
Analysis of the post creep deformation structure was performed on a Phillips CM200 operating at 200kV.

4.3 Results

4.3.1 Microstructural Characterization

4.3.1.1 Grain Size Characterization

Orientation imaging microscopy (OIM) was used to characterize the difference in grain size between the rim and bore microstructures. OIM maps for the rim and bore microstructure are presented in Figure 4.1 and Figure 4.2 where it can be seen that there is a difference in grain size. The average grain size for the rim and bore microstructures is 50 and 35 microns in diameter, respectively. The differences in grain size between these regions of the turbine disk are to be expected since these locations experienced different thermal histories during the hot forging and the final supersolvus heat treatment. Furthermore, there is no texture in these specimens as can be seen in the IPF presented in part b in each of the image sets. In addition to grain size measurements the fraction of $\Sigma 3$ twin boundaries was determined. The bore microstructure possessed a greater number of these special grain boundaries. Even though the effect of $\Sigma 3$ twin boundaries on creep deformation behavior was not a focal point in this study it is known that they do have an influence on the high cycle fatigue resistance of this alloy as reported by Gao et al [80]. In their study, thermomechanical processing was used to modify the microstructure by increasing the number of $\Sigma 3$ grain boundaries to enhance resistance to intergranular cracking.
4.3.1.2 $\gamma'$ Precipitate Characterization

Representative SEM images of the precipitate microstructure depicting the $\gamma'$ size scale and bimodal distribution can be seen in Figure 4.3a-f for the rim and bore microstructures at different levels of magnification. As can be seen in this series of SEM micrographs the primary microstructural differences are in the secondary $\gamma'$ precipitate size, $\gamma$ channel width and tertiary $\gamma'$ volume fraction. Quantitative image analysis techniques were used to measure the average secondary and tertiary $\gamma'$ precipitate size and volume fraction, which are on average 186.6nm and 25.5nm for the rim microstructure and 321.4nm and 26.2nm for the bore microstructure. The volume fractions of the secondary $\gamma'$ and tertiary $\gamma'$ precipitates for the rim structure were 48.4% and 2.7% for the rim microstructure. The volume fractions of the secondary and tertiary $\gamma'$ precipitates were 46.1% and 8.2% for the bore microstructure.

Due to the faster cooling rate experienced in the rim section, the average size of the secondary $\gamma'$ are finer in size and the tertiary $\gamma'$ are present to a much lesser degree when compared to the microstructure from the bore section, which experienced a slower cooling rate. While the tertiary $\gamma'$ precipitate size is roughly identical when the two microstructures are compared, the volume fraction is much higher in the bore microstructure as can be seen in when the microstructures are imaged at 60 and 240kx magnification.

The minimum separation distance between the coarser secondary $\gamma'$ precipitates ($\gamma$ channel width) was measured using a line intercept method. It was found to be larger for the bore microstructure as compared to the rim microstructure, on average $159.4\pm43.1$nm.
versus 63.5±10.2nm, respectively. All of these salient microstructural features that were analyzed (grain size, secondary γ’ precipitate size, tertiary γ’ volume fraction, and γ channel width) are expected to control the deformation behavior during creep at elevated temperatures.

4.3.2 Creep Testing

A comparison between the creep behavior of the rim and the bore microstructure can be seen in Figure 4.4 on the tests that were carried out to approximately 1.0% plastic strain. Since stress and temperature were identical for these test cases, it can easily be deduced that slight differences in the size scale of microstructural features can have a large effect of the creep deformation response of the material. From multiple tests of these microstructures crept under the same test conditions but interrupted at different levels of strain, the normal primary transients and general form of the creep curve are reproducible. The three interrupted creep tests from the rim microstructure are shown in Figure 4.5. Upon examination of the early stages of deformation both microstructures exhibit a normal primary creep transient period; however, the bore microstructure initially deforms more rapidly than the rim microstructure. There is no pronounced steady state creep regime but rather there is a transition from primary to tertiary creep. Overall, the rim microstructure is significantly more creep resistant than the bore microstructure.

4.3.3 Deformation Mechanism Identification

In order to account for the difference in creep behavior between the two different microstructures, a detailed TEM characterization study of the deformation structures that developed during the course of creep deformation was performed for specimens
interrupted at approximately 0.05%, 0.2% and 1.0% plastic strain. The results of the TEM characterization studies from the rim microstructure will be presented first which will then be followed by the TEM characterization results from the bore microstructure.

4.3.3.1 Rim Microstructure

4.3.3.1.1 (0.05 % Plastic Strain)

TEM analysis of the creep deformation substructures from the rim microstructure at the earliest stage of deformation reveals that deformation is occurring predominately in the γ matrix as little or no evidence of γ' precipitate shearing events were observed. Figure 4.6 shows the typical deformation structure that is observed using conventional diffraction contrast TEM imaging, which consists primarily of highly planar stacking faults that tend to be concentrated in the γ matrix phase. The stacking faults appear in this image as extended alternating bright and dark fringes in the matrix. The partial dislocations appear to have "percolated" between the secondary γ' precipitates, leaving them unsheared. The stacking faults are single layer faults and are most likely created by the dissociation of a prefect a/2<110> matrix dislocation into two a/6<112> Shockley partial dislocations on {111} type close packed glide planes. A decorrelated movement of the two Shockley partials dislocations must be occurring whereby the leading partial dislocation is more mobile than the trailing partial dislocation and as a consequence, an intrinsic stacking fault is created in the matrix. As the leading partial dislocation propagates, it does not appear to shear through the γ' precipitates since this would create a high energy complex stacking fault. Instead the leading Shockley partial dislocation loops around the precipitate and then continues along its deforming path in the matrix phase.
4.3.3.1.2 (0.2% Plastic Strain)

With increasing creep deformation to 0.2% strain, the same stacking fault structures are observed; however, they are present in a much higher density as shown in Figure 4.7. In addition, nearly all of the grains examined possessed these deformation features. When the faults are imaged inclined (Figure 4.7.) and nearly edge on (Figure 4.8) it can be noticed that a majority of the faults again do not shear through the $\gamma'$ precipitates. As mentioned in the previous section, this would indicate that the decorrelated Shockley partial dislocation, which is laying out the stacking fault, does not shear through the precipitates themselves since this would leave a complex stacking fault. Instead, $a/6<112>$ Shockley partial dislocation loops that are left around the precipitates that have the same Burgers vector as the leading Shockley partial dislocation, and an intrinsic stacking fault is left within the matrix.

The nature of the stacking faults were determined through complementary BF/DF TEM imaging pairs under a two beam diffraction condition with the deviation parameter set close to $s=0$ and the operative diffraction vector $g_{111}$. As expected the outer fringes in the BF image is symmetric and the outer fringes in the centered DF image is asymmetric. The stacking faults that were used in this analysis are the two stacking faults that are in between two secondary $\gamma'$ precipitates in the center of the image. To determine whether the stacking faults are intrinsic or extrinsic in nature, the $g$ vector is placed in the center of the stacking fault in the centered DF image. (Figure 4.9b) When this is done, the $g$ vector points away from the bright outer fringe, which indicates that the stacking faults are intrinsic, based on the two-beam dynamical theory [70].
In addition to identifying the intrinsic nature of the matrix stacking faults and explaining how the long planar faulted structures arise, another interesting observation was noted. With an increase in plastic deformation there are also occurrences where the precipitates are being sheared as well. This can be seen in Figure 4.9a where there appears to be overlapping stacking faults that traverse both the $\gamma$ and $\gamma'$ phases. A better representation of the shearing can be observed in Figure 4.10a-b in tilting experiments for which the faults. An arrow in the image is pointing to the same carbide, which can act as a point of reference when comparing the two images. In Figure 4.10b, the faults are inclined so the stacking faults are It appears that the single layer faults have become faintly visible if not invisible when they are viewed edge on since they are now parallel with the electron beam of the microscope. There are however faulted structures that are still visible which indicates that they must be overlapping stacking faults or microtwins. When these structures were tilted edge on and oriented along the [011] zone axis in order acquire a selected area diffraction pattern there were no twin reflections that were present in the diffraction pattern. While this does not necessarily rule-out that they are microtwins, both the thickness and density of these features are smaller such that twin reflections may not be visible in the SADP.

4.3.3.1.3 (1.0 % Plastic Strain)

For the rim microstructure that was crept to 0.98% plastic strain in 2725 hrs, microtwinning was found to be the dominant mode of deformation. Figure 4.11 displays a BF TEM micrograph of the microtwins viewed nearly edge on and oriented along the [011] zone axis along with its corresponding selected area diffraction pattern. The
diffraction pattern shows fundamental reflections from the $\gamma$ matrix, superlattice reflections from the $\gamma'$ precipitates, and twin reflections. The presence of these twin reflections indicates that these planar fault structures are in fact microtwins. The microtwins appear to be present in relatively the same density as the long planar stacking faults that were observed during creep to 0.2% strain. Characteristic of the microtwinning mechanism are highly planar shearing configurations that shear through both $\gamma$ and $\gamma'$ precipitates by identical $a/6<112>$ Shockley partial dislocations in adjacent $\{111\}$ glide planes [54]. The remarkable observation that microtwinning is a principal creep process occurring at high temperature and low strain rate is the subject of further discussion in a following section.

4.3.3.2 Bore Microstructure

4.3.3.2.1 (0.05% Plastic Strain)

TEM deformation substructure characterization of the bore microstructure that was crept to 0.05% strain differs from the rim microstructure in that there is a homogeneous distribution of dislocations that are present in the $\gamma$ matrix. What sets this deformation mechanism for the bore microstructure apart from the rim microstructure is that the dislocations are not dissociated into Shockley partial dislocations, leaving behind long planar matrix intrinsic stacking faults. A representative BF TEM micrograph showing the typical deformation substructure can be seen in Figure 4.12. When viewed at a higher magnification, it appears that the finer tertiary $\gamma'$ precipitates are resisting dislocation motion as can be seen by the many pinning points along the dislocation line length as can be seen in Figure 4.13. The Burgers vectors of these dislocations were determined using
the \( g \) dot \( b = 0 \) invisibility criterion. A series of two-beam BF TEM micrographs that were acquired with different imaging conditions is presented in Figure 4.14. Dislocation marked A is visible when imaged under \( g=1\bar{1}1 \) and \( g=\bar{2}00 \) but invisible when imaged with \( g=\bar{1}1\bar{1} \), \( g=3\bar{1}1 \), and \( g=0\bar{2}2 \). Based the invisibility criteria the dislocation has a Burgers vector of \( a/2[011] \). There are other dislocations that can be determined in this manner and based on this it is noted that there are many \( a/2<110> \) type dislocations that are present within the deformed microstructure, each having have different Burgers vectors. Additional observations are needed to determine how the dislocations are interacting with the finer tertiary \( \gamma' \) precipitates.

### 4.3.3.2.2 (1.0% Plastic Strain)

With an increase in plastic deformation there is also an increase in the amount of matrix dislocation activity. Furthermore, there is also evidence of an additional deformation process that has commenced at this later stage of creep in which single layer stacking faults are operative on multiple slip planes and have formed in the coarser secondary \( \gamma' \) precipitates. When the stacking faults are present within the precipitates it indicates that shearing of \( \gamma' \) is taking place. Figure 4.15 shows a representative TEM micrograph of the dislocation structure for the bore microstructure crept to 1.0% plastic strain in 1175 hrs. The increased dislocation activity that is present in the \( \gamma \) matrix can be seen BF and WBDF TEM images presented in Figure 4.16, respectively. The dislocations are present in such a high density that it is only with the WBDF imaging technique that the dislocations can be resolved.
Since the precipitate shearing mode appears to be different from the microtwinning mechanism, as observed in the rim microstructure, an analysis of the dislocations responsible for creating the single layer stacking faults as well as the nature of the stacking faults was conducted. The stacking faults were identified by complementary BF/DF imaging pairs that were imaged using the g020 reflection. When the g vector is placed in the center of the stacking faults in the centered DF image (Figure 4.17b), it points away the bright outer fringe, which indicates that the stacking fault is intrinsic in nature based on the imaging rules set forth for the {200} type reflection that was used as the imaging condition [70]. Since the intrinsic stacking faults are within the $\gamma'$ precipitates they are superlattice intrinsic stacking faults (SISF). There is an unlikely possibility that these faults could be complex stacking faults; however, additional characterization needs to be conducted to prove or disprove this.

4.4 Discussion

4.4.1 Effect of Microstructure on Creep Deformation Mechanisms

To interpret and account for the differences in creep response it is important to focus on the main microstructural differences that set these structures apart from one another, namely: size scale and volume fraction of the secondary $\gamma'$ precipitates, size scale and volume fraction of the tertiary $\gamma'$ precipitates, and $\gamma$ channel width spacing. It is the combination of these microstructural features that will ultimately govern how the material will deform under high temperature creep conditions; however, an examination of each of them will be discussed in the proceeding section.
4.4.2 Effect of $\gamma'$ Precipitate Size and Volume Fraction

The finer tertiary $\gamma'$ precipitates are situated in between the coarser secondary $\gamma'$ precipitates and are present to a much higher degree in the bore microstructure, which is directly related to the cooling of the material within this region of the turbine disk, following the supersolvus heat treatment. As shown in the TEM micrographs presented in Section 4.3.3.2.1, the early stages of creep deformation in the bore microstructure are dominated by $a/2<110>$ dislocation activity. Due to the high volume fraction of tertiary $\gamma'$ precipitates that are present in the $\gamma$ channels between the secondary $\gamma'$ precipitates, they provide resistance to dislocation motion that can ultimately affect the overall creep behavior as shown by Locq et al [77].

There are several ways in which the material can continue to deform by $a/2<110>$ dislocations in the matrix: by shearing, looping or bypassing. Shearing of these fine precipitates by $a/2<110>$ dislocations will create anti-phase boundaries (APBs) in the precipitates. This process is possible since dislocation debris loops are not observed surrounding this fine population of particles. Since there is significantly less tertiary $\gamma'$ precipitates in the rim microstructure it is presumed that they will contribute less to strengthening the $\gamma$ matrix.

4.4.3 Effect of $\gamma$ Channel Width

While the volume fraction of tertiary $\gamma'$ precipitates may play a major role in affecting creep strength in the bore structure, especially at lower strain, it appears that the $\gamma$ channel width is controlling deformation for the rim structure and may also be a precursor to microtwinning at higher strain. There have been a limited number of studies, which have
investigated the effect of γ channel width on dislocation dissociation and decorrelation. Raujol et al have experimentally observed this process occurring during creep deformation in a similar PM Ni-base disk alloy NR3 at 650MPa and 700ºC as well as during in-situ TEM straining of the same alloy at room temperature. Raujol et al have experimentally observed this process occurring during creep deformation in a similar PM Ni-base disk alloy NR3 at 650MPa and 700ºC as well as during in-situ TEM straining of the same alloy at room temperature [81]. Their studies have shown that when a perfect a/2<110> dislocation attempts to glide through a precipitate field with a fine spacing between precipitates, the dislocation dissociates into leading and trailing Shockley partial dislocations with a stacking fault in between. They have suggested that this decorrelation is responsible for the shearing mechanism proposed by Decamps et al [64] in which the leading Shockley partial dislocation shears through both the precipitate and matrix and leaves behind a continuous stacking fault.

Of course there are a combination of factors that would determine whether dislocation dissociation will occur or not and they are based upon microstructural features (such as γ′ precipitate size, volume fraction and interparticle spacing), alloy chemistry (which influences stacking fault energy), stress, and temperature. Douin et al has investigated the dislocation dissociation process through dislocation dynamic simulations which has provided new insights as to the role microstructure plays on promoting or inhibiting dissociation of matrix dislocations [82]. Through simulation results, it was determined that the dissociation and decorrelation process is a function of the effective shear stress oriented with respect to the direction of the leading and/or trailing partial dislocations.
4.4.4 Dislocation Dissociation and Decorrelation

In addition to the $\gamma$ channel width affecting dislocation dissociation and decorrelation of the Shockley partials, Suzuki segregation (which is the segregation of solute elements to stacking faults) might also aide in the dislocation dissociation process as the stacking energy may locally decrease, thereby increasing the separation distance between the leading and trailing partial dislocations. Segregation of solute elements that are known to lower the stacking fault energy, such as Co and Cr in Ni, has been reported in Ni-base superalloys [83]. It is quite probable that Suzuki segregation can take place during high temperature creep conditions, which can facilitate diffusion of solute elements to stacking faults and assist in the dissociation and decorrelation process. Han et al has provided experimental evidence of Suzuki segregation in a Co-base superalloy through EDX line profile analysis [84]. Experimental energy dispersive X-ray spectroscopy (EDX) line profiling of the stacking faults in this study has provided preliminary evidence of segregation of Co and Cr to the matrix stacking faults, but ongoing analytical electron microscopy investigations are being directed towards validating these findings.

4.4.5 Mechanism for Microtwinning

Microtwins have been observed in during creep deformation for several different Ni-base superalloys [51, 52, 54, 73]. Kolbe has provided a hypothesis for the formation of microtwins in $\gamma'$ strengthened Ni-base Superalloys by the passage of identical a/6<112> Shockley partial dislocations that shear $\gamma$ and $\gamma'$ on sequential {111} glide planes [59]. Although no direct TEM evidence was provided by Kolbe to substantiate his hypothesis, further work by Viswanathan et al [54] on creep of Rene 88DT at 650°C and 838MPa
proved through detailed dislocation analysis that microtwins form by motion of identical Shockley partials. The present study of creep in Rene 104 at interrupted levels of strain provides experimental evidence that a necessary precursor to the microtwinning mechanisms is the decorrelation of a/2<110> dislocations. The decorrelation occurs as a result of the fine γ channel widths in the rim microstructure. When the decorrelation event happens, the leading partial dislocation sweeps out and deforms the γ matrix, leaving behind intrinsic stacking faults. It is not entirely clear how identical Shockley partial dislocations on adjacent glide planes subsequently form; however, possible theories for microtwin nucleation will be presented in the following chapter.

4.4.6 Mechanisms for SISF formation in γ’

During prolonged time under creep conditions, the creep rate increase coincides with a large increase in the dislocation density within the γ matrix. The enhanced dislocation activity in the matrix should be accommodated by precipitate shearing and shearing of the coarser secondary γ’ precipitates. This present study has shown that the precipitates are sheared by a a/3<112> type superlattice Shockley partial dislocations that leave SISF in the precipitate. Milligan et al has shown that SISF can be created as a result of a a/2<110> dissociation event that occurs at the γ/γ’ interface and produces a/3<211> which shears the γ’, creates a SISF and leaves behind a a/6<21> Shockley partial at the interface during creep of single crystal PWA 1480 at 760ºC [46]. Chen and Knowles have studied precipitate shearing and SISF formation as well. During creep of CMSX-4 at 750ºC and 750MPa they have shown that the reaction and dissociation of two intersecting a/2<110> matrix dislocations with different Burgers vectors can initiate a/3<112> slip
which would create an SISF in the precipitate as it is sheared by the $a/3\langle 112 \rangle$ super-Shockley partial dislocation [85].

Although direct evidence of the dislocations present at the $\gamma/\gamma'$ interface has not been obtained in this study because of the difficulty to resolve individual dislocations, it seems quite plausible that mechanisms proposed by Chen and Knowles may be occurring since there are many $a/2\langle 110 \rangle$ dislocations in the matrix that can intersect with one another to create this shearing configuration. Interestingly, at the smaller strain level examined ($0.05\%$), no SISF configurations were observed while only a single $a/2\langle 110 \rangle$ slip system appears to be active in the grain analyzed in Figures 4.13-4.15. Hence, initiation of dislocation activity on a secondary slip system may be an important precursor to $\gamma'$ shearing by SISF formation. Additionally, there are clearly multiple stacking fault configurations present, indicating that multiple slip systems have also been activated. The shearing of the secondary $\gamma'$ may therefore be an important recovery process that helps relieve the strain hardening in the matrix, enabling continued dislocation activity in the matrix.

4.4.7 Comparing Creep Rate Limiting Process (Microtwinning vs SISF Shearing Mechanisms)

It has been shown that the creep rate limiting process in the microtwinning mode is related to the atomic reordering processes that must occur when the matrix and precipitate are sheared by paired $a/6\langle 112 \rangle$ Shockley partial dislocations that operative on adjacent $\{111\}$ glide planes [60, 74]. At the present time it is not possible to provide a complete explanation for the differences in creep-rates for the two different
microstructures since the rate limiting process associated with SISF formation in the bore structure is not presently identified. One possibility is that the reaction between a/2<110> matrix dislocations, which may be a precursor to SISF formation, is thermally activated. In this case, there may indeed be an activation barrier to the reaction. It is also possible that subsequent movement of the a/3<112> super-Shockley partial into the secondary γ’, thereby creating the SISF, may be a difficult thermally-activated process due to the complex core structure expected for this partial in the L1₂ structure [29]. More recently, it has been reported by Kovarik et al that atomic reordering, much like the atomic reordering processes of microtwinning, is also possible when SISF related stacking fault configurations are created during creep of Ni-base superalloys [75].

4.4.8 Incorporating Microstructural Effects into Deformation Mechanism Mapping

The main theme of this Chapter has been the effect of microstructure on deformation mechanisms for creep at the same stress and temperature. The microstructural and TEM deformation mechanism characterization results reveal that depending on the scale of the microstructural features it is possible to dramatically alter the creep deformation response of the material and favor one deformation mode versus another. From these results it is possible to modify the creep deformation map that was previously reported in Chapter 3 which incorporates the results of the detailed TEM characterization study of both Rene 104 and Rene 88DT. Note: Yield strength as a function of temperature for the rim and bore microstructures has been experimentally determined using the ETMT machine, details of which are presented in Appendix B. There was one outlier for the rim microstructure that was tested at 649°C which does need to be replicated. If this outlier is
disregarded then a plot of yield strength vs. temperature can be plotted to show the
difference in yield strength between the rim and bore microstructures.

Creep experiments were conducted at 677°C and at a yield stress of 724MPa. The ETMT measured yield strengths at this temperature are 1045 and 1025MPa for the rim and bore microstructures, respectively. Therefore, the percentage at which the creep tests were carried out was 69.3 and 70.6% for the rim and bore microstructures, respectively. A plot of yield strength vs. temperature including this data and depicting the creep test conditions for which the rim microstructure showed a/2<110> dislocation dissociation at low strains, microtwinning at the larger strains and the bore microstructure which showed a/2<110> dislocation activity at low strains and γ’ precipitate shearing at larger strains is presented in Figure 4.18. For the rim and bore microstructures the strain rate that was measured after the initially primary creep transient were 2.77 x10^{-8} s^{-1} and 5.56 x 10^{-8} s^{-1}, respectively which is significantly lower than the 1.8 x10^{-3} strain rate that was used for the tensile tests. Based on these findings, the creep deformation map presented in Section 3.4.1 needs to be modified to account for the microstructural effect and strain rate effects.

4.5 Conclusions

The focus of this study was to explore the effects of microstructure on the creep deformation mechanisms in Ni-base disk superalloy Rene 104 using transmission electron microscopy characterization techniques. Samples with different starting microstructures were crept at the same stress and temperature and therefore it was possible to identify key microstructural features that influence modes of deformation,
which were then identified using the TEM then related the macroscopic creep deformation behavior. The following conclusions can be drawn from this work:

1) The creep deformation response of Ni-base disk superalloy Rene 104 was found to be highly dependent upon $\gamma'$ precipitate size scale, volume fraction, and $\gamma$ channel width spacing.

2) The more creep resistant “rim” microstructure consisted of finer scaled secondary $\gamma'$ precipitates, low volume fraction of tertiary $\gamma'$, and finer $\gamma$ channel width spacing. This combination promoted deformation via $a/2<110>$ dislocation dissociation and $a/6<112>$ Shockley partial decorrelation which resulted in matrix intrinsic stacking faults at low strain which then transitioned into a microtwinning deformation mode at higher strains.

3) It was apparent that the finer $\gamma$ channel width of the rim microstructure promoted $a/2<110>$ dislocation dissociation and decorrelation, which may be a possible precursor to the microtwinning mechanism.

4) The less creep resistant “bore” microstructure consisted of coarser secondary $\gamma'$ precipitates, a higher volume fraction of tertiary precipitates and a wider $\gamma$ channel width. This combination allowed for more deformation to occur in the $\gamma$ matrix via $a/2<110>$ dislocation activity which then transitioned into precipitate shearing by an SISF related $\gamma'$ shearing mechanism.

5) This work suggests that there is connection between microstructure scale and deformation mechanisms and that the microstructure can be tailored for optimal creep resistance.
Figure 4.1: OIM results from the rim microstructure: a) color coded grain orientation map b) inverse pole figure depicting the random orientation of the grains and c) grain orientation map with Σ3 twin boundaries are highlighted in red.
Figure 4.2. OIM results from the bore microstructure: a) color coded grain orientation map b) inverse pole figure depicting the random orientation of the grains and c) grain orientation map with Σ3 twin boundaries are highlighted in red.
Figure 4.3: Representative SEM micrographs revealing the microstructural differences in γ’ precipitate size, distribution, and morphology between the rim a) 15kx b) 60kx c) 240kx and bore d) 15kx e) 60kx f) 240kx sections of the turbine disk used in this study at different magnifications.
Figure 4.4: Experimental creep curves for the bore and rim microstructures tested at 724MPa and 677°C showing the difference in creep response.
Figure 4.5: Experimental creep curves for the rim microstructure crept in tension at 724MPa and 677ºC for 0.05, 0.2 and 1.0% strain.
Figure 4.6: BF TEM micrograph showing the development of highly planar matrix stacking faults during the early stages of creep deformation of the rim microstructure at 677°C and 724MPa.
**Figure 4.7:** BF TEM micrograph of stacking faults in the $\gamma$ matrix following creep deformation at 677°C and 724MPa of the rim microstructure.

**Figure 4.8:** BF TEM micrograph of the stacking faults tilted nearly edge showing how a majority of the faults are not shearing through the $\gamma'$ precipitates. Creep at 677°C and 724MPa of the rim microstructure.
Figure 4.9: a) BF and b) centered DF TEM micrographs of stacking faults in the \( \gamma \) matrix during creep deformation to 0.2\% strain at 677\(^\circ\)C and 724MPa.
**Figure 4.10:** BF TEM images of the intrinsic stacking faults that are imaged a) inclined to reveal that stacking fault fringes and b) edge which (arrows are pointing to the carbides as a reference location between the two images).
Figure 4.11: a) BF TEM micrograph of microtwins formed following creep deformation to 0.98% strain at 677°C and 724MPa. b) SADP showing fundamental, superlattice and twin reflections (marked by arrows). B=[011]
**Figure 4.12:** Representative BF TEM micrograph of the creep deformation substructure found in the bore microstructure following creep to 0.05% strain at 677°C and 724MPa.

**Figure 4.13:** Representative BF TEM micrograph of the creep deformation substructure found in the bore microstructure following creep to 0.05% strain at 677°C and 724MPa at a higher magnification showing the tertiary $\gamma'$ precipitates resisting dislocation motion.
Figure 4.14: Series of BF TEM micrographs from the bore microstructure of the a/2<110> type dislocation structure that developed in the bore microstructure following creep at 677°C and 724MPa to 0.05% strain.
Figure 4.15: Post creep dislocation substructure of the bore microstructure interrupted after 1.0% strain at 677°C and 724MPa which shows increased dislocation content in the γ matrix and precipitate shearing as marked by the stacking faults present in the secondary γ' precipitates.

Figure 4.16: High magnification TEM images of the dislocation activity in the γ matrix following creep at 677°C and 724MPa for the bore microstructure. a) BF and b) WBDF TEM images.
**Figure 4.17:** TEM analysis on the nature of the stacking fault present in the secondary γ’ precipitate a) BF TEM micrograph imaged with g020 and b) centered DF TEM micrograph imaged with g020. The vector when placed at the center of the CDF image points towards the bright outer fringe indicating that they stacking faults are superlattice intrinsic stacking faults.
Figure 4.18: Plot of yield strength vs. temperature for the rim and bore microstructures. Included within this plot are experimental observations of creep mechanisms for the rim and bore microstructures crept at 677°C and 724MPa.
CHAPTER 5

ON THE NUCLEATION OF MICROTWINs
DURING CREEP DEFORMATION

Abstract
During high temperature creep deformation in Ni-base disk superalloys, microtwinning has been found to be a principle deformation mode that is highly dependent upon internal microstructural details such as γ’ size, distribution, morphology, and γ channel width, as well as external factors such as temperature and stress. Creep at 677ºC and 724MPa for a microstructure that consists of a bimodal γ’ precipitate size distribution with a fine-scale secondary γ’, a low volume fraction of tertiary γ’, and a fine γ channel width promoted microtwin development at the later stages of creep. In this TEM-based deformation mechanism study, the aim was to identify microtwin nucleation sources and track their evolution into fully developed microtwins as a function of increasing plastic deformation. Creep specimens were interrupted at different levels of plastic deformation. At the earliest stages of creep, deformation is highly localized around carbides, borides and serrated grain boundaries, which act as sources of a/2<110> matrix dislocations. Due to microstructural effects and low matrix stacking fault energies, the dislocations readily dissociate into Shockley partial dislocations. The detailed process by which these partials cooperatively shear both γ and γ’ phases during the course of deformation have been accounted for in the development of microtwin nucleation models.
5.1 Introduction

Ni-base superalloys are a class of materials that are predominately used in aircraft gas turbine engines in some of the most demanding applications where they must provide structural integrity while operating at extreme conditions of stress and temperature such as in aircraft gas turbine engines. During the study of high temperature creep deformation of these materials there have been a variety of deformation mechanisms that have been reported during creep of Ni-base superalloys [64, 73, 86]. Among the more recent deformation mechanisms that have been experimentally observed during high temperature creep is microtwinning [51, 72, 87].

Previous research on the creep deformation behavior and creep deformation mechanism identification of Ni-base Superalloy ME3 (Rene 104) has shown that microtwinning is a dominant deformation mechanism that exists during creep deformation when crept at a test temperature of 677°C and a stress level of 690MPa. (Refer to Chapter 3) Deformation in that case was carried out to such a high level of plastic deformation (2.0% strain) that it was difficult to capture discrete locations where microtwins may have initiated or terminated. To determine the salient microstructural features that promote microtwinning, creep experiments were conducted at 677°C and 724MPa creep at 677°C on different starting microstructures and it was found that a fine secondary $\gamma'$ precipitate size, low volume fraction of tertiary $\gamma'$, and fine $\gamma$ channel width results favored microtwinning during creep in this intermediate temperature regime. (Refer to Chapter 4) The evolution of the deformation substructure was tracked in this microstructure by interrupting creep tests at varying levels of plastic strain.
The objective of this research is to further explore and interpret the microtwin nucleation process by taking a deeper look into the deformation substructure at the earliest stages of deformation. In addition, in-situ TEM straining experiments were conducted in an attempt to capture a glimpse of the nucleation events that lead into microtwinning in real time.

5.2 Materials and Experimental Procedure

The material used in this study was extracted from the rim section of turbine disk forging that was previously subjected to a proprietary supersolvus heat treatment. The microstructural characterization details can be found in Chapter 4. Creep experiments were conducted on this microstructure at 677°C and 724MPa and interrupted at 0.05, 0.2, and 1.0% strain. It was found that this microstructure promoted a/2<110> dislocation dissociation at the early stages of creep which then transitioned to microtwinning at the later stages of creep. It was for this reason that additional research was conducted on these specimens in order to determine how microtwins nucleate in this material and for this particular microstructure. Following creep experiments foils for TEM deformation substructure characterization studies were extracted from the gauge at a 45° angle with respect to the tensile axis in order to view deformation activity along slip planes that experience maximum shear stress. The foils were mechanically thinned using progressively finer SiC paper to a thickness of 150µm then slurry drilled. Finally thinning was conducted on a Struers twin jet polishing unit using an electrolyte consisting of 60% methanol, 35% 2-n-butoxyethanol, and 5% perchloric acid at a temperature of -45°C and
an applied voltage of 15V. Conventional diffraction contrast TEM analysis was performed on a Phillips CM200 TEM operating at 200kV.

In addition to post mortem creep deformation characterization analysis, in-situ TEM straining was performed on the pre-crept specimens. The in-situ TEM studies were conducted at CEMES CNRS with Dr. Marc Legros. Specimens were sectioned from the gauge and cut into 1mm by 3mm rectangular specimens that were subsequently mechanically thinned then electropolished to electron transparency. TEM in-situ straining experiments were performed on a JEOL 2100 TEM operating at 200kV. The goal of this was to part of the study was to take the pre-deformed specimens at the varying stages of deformation and deform them in the microscope in order to experimentally observe the deformation processes as they are occurring.

5.3 Results

5.3.1 Creep Deformation Substructure Characterization

In order to find nucleation sources of microtwins during creep deformation of Ni-base superalloy Rene 104 it was important to examine the deformation substructure at the earliest stages of deformation. It was experimentally determined, and presented in the previous chapter, that the microstructure which favored the microtwinning deformation mode was the “rim” microstructure which had on average a secondary γ′ precipitate size of 186.6nm, tertiary γ′ size of 25.5nm and γ channel width spacing of 64nm. A closer look into the deformation activity of this microstructure following 0.05% plastic strain revealed a particular grain of interest, which allowed for a unique view of the deformation activity as viewed down the (111) glide plane. A low magnification BF
STEM imaging of the grain is presented in Figure 5.1. The grain consists of numerous minor carbide and/or boride phases that are present along the grain boundaries and or within the grain interiors. Upon closer examination, the minor carbide or boride phases and grain boundaries appear to be discrete sources of deformation.

5.3.2 Identification of Deformation Sources

A series of BF TEM images of deformation sources are presented in Figure 5.2a-d. In all cases deformation commences either at carbide/boride particles that are within the grains or along grain boundaries. The particles act as stress concentrators where dislocation emission apparently occurs. The $\gamma$ matrix phase is being deformed and it can be seen that the dislocations are emitted as perfect dislocations, but then immediately dissociate into partial dislocations that are bounding matrix stacking faults. The stacking faults when viewed in this orientation, with the fault plane nearly parallel to the foil plane, do not show the alternating bright and dark fringes that one would normally expect to see when characterizing stacking faults. The stacking faults instead appear as dark grey regions that are bound by partial dislocations and are present in the $\gamma$ matrix phase. The partial dislocations do not appear to be shearing the secondary $\gamma'$ or tertiary $\gamma'$ precipitates. Instead, they appear to be percolating throughout the $\gamma$ matrix where they are observed to loop around the coarser $\gamma'$ precipitates.

5.3.3 AEM Characterization of Carbides/Borides

A TEM EDX analysis of several particles that were observed to be dislocations sources were performed. In Figure 5.3a, a BF TEM image of an intragranular carbide is shown along with its corresponding spot EDX spectrum. Based on the chemical composition of
the EDX spectrum (Figure 5.3b), the carbide is enriched in Ti, Ta, Co, Cr, Mo, and Nb, which is a clear indication that this is most likely a MC type carbide. When the same type of analysis was done on a grain boundary particle, as shown in Figure 5.4a, the EDX spectrum (Figure 5.4b) showed that the particle was enriched in Co, Cr, Mo, C, and B which gives an indication that this again is likely to be an M\(_{23}C_6\) carbide; however, the presence of the boron reveals that boron may be enriched at the grain boundary or there are borides present.

5.3.4 Detailed TEM Deformation Substructure Analysis

Perhaps the most striking deformation features that were observed during TEM characterization is that the dislocations emitted from the grain boundary carbides/borides, or intragranular carbide sources readily dissociate into Shockley partial dislocations. Figure 5.3 shows a/2<110> dislocations being emitted from the same intragranular MC carbide source in which the spot EDX analysis was performed in Figure 5.3a-b. When the dislocation is emitted from the carbide source, it is evident that it has dissociated and the leading partial dislocation has "percolated" between the secondary γ' precipitates, leaving them unsheared. The finer tertiary γ' precipitates are not sheared but they are looped. This can be seen in the +/-g imaging condition where there is the expected inside/outside contrast of dislocation of a dislocation loop surrounding the precipitate.

A detailed TEM analysis of the perfect and partial dislocations as well as the matrix stacking faults was conducted. BF TEM diffraction contrast images, that were acquired using different imaging conditions is presented in Figure 5.5. The leading Shockley partial dislocation and the stacking fault is visible in Figure 5.5a while the
trailing Shockley partial dislocation is visible in Figure 5.5j. The Shockley partial dislocations were determined to be on the (111) glide plane based on the g dot R invisibility criterion for planar faults. The dislocations and stacking faults cannot be operating on the (111) glide plane since the stacking fault is invisible for \( \pm g \bar{2}02 \) and \( \pm g0\bar{2}2 \) for which g dot R does not equal zero. For the same reason they also cannot be on the (1\( \bar{1} \)1) glide plane \( \pm g\bar{2}20 \) and \( \pm g2\bar{0}2 \) since g dot R does not equal zero. This leaves either the (1\( \bar{1} \)1) or the (111) glide plane. The stacking fault cannot be on the (1\( \bar{1} \)1) glide plane since g dot R does not equal zero for imaging with \( \pm g\bar{2}20 \) and \( \pm g\bar{2}02 \) for which the fault is invisible. Therefore the stacking fault and dislocations must be operating on the (111) glide plane.

In the analysis of the partial dislocations that bound the matrix intrinsic stacking fault, the trailing partial dislocation is completely invisible when imaged with \( g\bar{2}20 \), \( g2\bar{0}2 \). It is and completely visible with \( g\bar{1}1\bar{1} \) and shows weak contrast with \( g\bar{2}20 \). The leading partial dislocation is visible when imaged with \( g\bar{1}1\bar{1} \) and \( g\bar{2}20 \) and invisible with \( g0\bar{2}2 \) and \( g1\bar{1}1 \). Based on this analysis, the leading partial dislocation has a Burgers vector of \( a/6[\bar{2}11] \) and the trailing partial dislocation has a Burgers vector of \( a/6[\bar{1}\bar{1}2] \), which has dissociated from a perfect \( a/2[\bar{1}01] \) matrix dislocation. The dislocations are operating on the (111) glide plane and bound a matrix intrinsic stacking fault. In addition to determining the Burgers vectors of the dislocations, it is also evident that the perfect \( a/2<110> \) type dislocations all have the same Burgers vector and that they have dissociated into Shockley partial dislocations that have a Burgers vector of the type \( a/6<112> \). Moreover, it was shown in the previous chapter that this process is likely to be the precursor to microtwinning since at larger strain levels, for the same microstructure...
and test conditions, fully developed microtwins have developed. The following discussion section attempts to provide an interpretation as to how microtwins can form based on this experimental TEM evidence.

5.3.5 In-situ TEM Straining Experiments

In order to experimentally observe how microtwins form during deformation, in-situ straining of pre-crept samples was performed. Unfortunately, during high temperature in-situ straining, all of the specimens developed cracks along grain boundaries that were oriented perpendicular with respect to the straining axis. Therefore only room temperature in-situ straining experiments provided any tangible results that are worth reporting here. A series of BF TEM micrographs of the deformation activity during straining is presented in Figure 5.6. An interpretation of the results is as follows: in the first image shown in Figure 5.6a, several dislocation and stacking faults that were active on several slip systems. The stacking faults were only observed to be operative on parallel slip systems. When the material was strained, it was apparent that there was some cross slip activity that had occurred from one stacking fault system to the next.

Overlapping stacking faults, seen in the right hand micrograph in Figure 5.6b, were most likely formed by a/2<110> dislocations splitting into Shockley partial dislocations on parallel glide planes. A decorrelated movement of the partial dislocations results where the leading partial dislocation becomes more mobile than the trailing dislocation. As the leading Shockley sweeps out and deforms the γ matrix it is pinned by the γ’ precipitate since if sheared, a high energy complex stacking fault would be created in the precipitates. A pileup of dislocations is formed at a γ’ precipitate. Multiple partial
dislocations on parallel planes come in contact with the same barrier. The leading partial dislocations then become immobile and the trailing partial dislocations have a chance to reunite with the leading partial, which then recombines with the leading partial to form a perfect a/2<110> dislocation which can then cross slip onto different {111} glide planes. They appear to cross slip on a slip system which is parallel to the one with which they once were present on. Which then undergo a dissociation event as well. (Figure 5.6b)

Additional cross slip events occur that deposit dislocations atop the stacking fault created below which then dissociate as well, creating a series of overlapping stacking fault configuration. (Figure 5.6c) This overlapping of stacking faults in the γ matrix may be a clear indication that a microtwin is being formed since one the characteristic feature of a microtwin is the overlapping stacking faults on adjacent {111} planes. These characteristics closely resemble to experimental results acquired during TEM in-situ straining of nickel by Robertson [88].

5.4 Discussion

Microtwinning is quite a surprising mechanism to observe under low strain rate creep conditions since in general, twinning is often regarded as a high strain rate deformation process. Even so, there have been attempts to determine both theoretically and experimentally how microtwins actually form during creep in γ' strengthened Ni-base superalloys. Presently, in the literature there are opposing views as to how microtwins can form in a two-phase γ/γ' superalloy microstructure. The following sections attempt to show how microtwins can form in first in the fcc γ matrix and then in L1₂ structure γ' precipitates.
5.4.1 Twinning in the FCC Crystal Structure

If one considers the fcc structure of the $\gamma$ matrix phase alone, a microtwin can form by the passage $a/6<112>$ Shockley partial dislocations that are gliding on successive $\{111\}$ close packed planes. It can easily be envisioned how twins can form in fcc crystals by constructing a simple model of the ABC stacking sequence as viewed perpendicular to the $(111)$ plane as shown in Figure 5.7. The twin is created by the passage of $a/6<112>$ Shockley partial dislocations on adjacent $\{111\}$ glide planes. After the passage of the first and second partial dislocations an intrinsic stacking fault is created (Figure 5.7b) then a 2-layer extrinsic stacking fault is created. (Figure 5.7c) Finally after the passage of the third partial dislocation a 3-layer fault, or a twin is created. (Figure 5.7d)

5.4.2 Twinning in the L1$_2$ Crystal Structure

5.4.2.1 Twin Formation via $a/3<112>$ Superlattice Shockley Partial Dislocations

A perfect true twinned structure can be formed if instead of shear by identical $a/6<112>$ Shockley partial dislocations, the crystal is sheared by $a/3<112>$ superlattice Shockley partial dislocations since the ordered would be preserved in the $\gamma'$ precipitates. As shown in Figure 5.8b when the first $a/3<112>$ partial dislocation passes, a superlattice intrinsic stacking fault is created. When the second partial shears the crystal a superlattice extrinsic stacking fault is created. (Figure 5.8c) Finally, as the third superlattice Shockley partial dislocation passes a twin structure is created which does not have any nearest neighbor bond violations and the ordered structure is maintained across the twinning plane. (Figure 5.8d)
5.4.2.2 Twin formation via a/6<112> Shockley Partial Dislocations

If the same methodology is applied to a model of the L1\textsubscript{2} crystal structure where the crystal is displaced, or sheared, by identical a/6<112> Shockley partial dislocation on adjacent {111} close packed glide planes, complications arise due to the fact the ordered structure would be destroyed if this were to occur. The prefect L1\textsubscript{2} crystal is displayed in Figure 5.9a and when it is sheared by the passage of a a/6<112> Shockley partial dislocation, a high energy complex stacking fault is created which can be considered both of chemical fault (due to the nearest neighbor Al-Al and Ni-Ni bond violations) and stacking fault since now the ABC stacking sequence is destroyed (Figure 5.9b). After the passage of the second partial dislocation, a 2-layer complex stacking fault is created. (Figure 5.9c) When the third partial displaces the crystal the atomic stacking sequence is that of a (Figure 5.9d) Due to the nearest neighbor bond violations the twinned structure that is created is not a true twin but rather a pseudotwin.

5.4.3 Theories of Twin Nucleation in FCC Crystals

The experimental evidence described in the results sections indicate that decorrelation of a/2<110> dislocations is a necessary precursor to microtwinning. Several possibilities are now briefly mentioned in light of the present understanding. A comprehensive review of deformation twinning has been provided by Christian and Mahajan [58]. Many authors have proposed a pole mechanism to describe twinning in fcc crystals [89, 90]. In these models, the common theme for the formation of a twin is the creation of a pole and twinning dislocation from a perfect a/2<110> dislocation. The pole dislocation essentially acts as an anchor whereas the twinning dislocations (a/6<112> Shockley partial
dislocation) can sweep out on a \{111\} glide plane, rotating about a pole. There are opposing views concerning how twins form via a pole mechanism.

In the model proposed by Mahajan and Chin \[91\] two co-planar a/2\langle 110\rangle dislocations with different Burgers vectors are envisioned to react and form three identical Shockley partial dislocations on adjacent \{111\} glide planes, which can then act as the nucleus of a three layer twin. From the limited experimental evidence of microtwinning in the polycrystalline \(\gamma^\prime\) strengthened superalloys investigated recently \[54, 60, 73\], all of the a/2\langle 110\rangle dislocations that are observed to dissociate into Shockley partial dislocations have the same Burgers vector. This observation suggests that the microtwinning mechanism in these alloys differs from that proposed by Mahajan and Chin.

In the double cross slip model originally proposed by Pirouz a single screw orientated a/2\langle 110\rangle dislocation dissociates into two Shockley partial dislocations \[92\]. Due to the larger shear stress acting on the leading Shockley partial dislocation, it will bow out in a manner reminiscent of a Frank-Read source and create an intrinsic stacking fault. When the leading Shockley partial re-combines with the static, trailing partial, the a/2\langle 110\rangle dislocation that is once again created can cross-slip (along its screw orientation) by a single \{111\} plane, then re-dissociate into Shockley partial dislocations in the same manner as the dissociation on the initial \{111\} plane. If this operation were to be replicated on multiple, adjacent \{111\} planes, then Shockley partial dislocations possessing the same Burgers vector would now be operative and thus a microtwin could potentially develop. Based on the present, limited TEM evidence, it is not possible to
definitively validate this mechanism; however, it is noted that this mechanism is consistent with the operation of single a/2<110> slip systems as the source of the twins.

5.4.4 Theories of Microtwin Nucleation in γ’ Strengthened Ni-base Superalloys

One of the earliest experimental evidence of microtwinning in a γ’ strengthened Ni-base Superalloy was reported by Guimier and Strudel in Waspaloy deformed above 500ºC [72]. Electron diffraction experiments showed the presence of twin reflections and it was reported that the ordered structure of the γ’ precipitates has been maintained after the twinning which led the authors to suggest that the microtwins form by the motion of a/3<112> type superlattice Shockley partials in a polar mechanisms. In the L12 structure of the γ’ precipitates, this would result in twin formation if the a/3<112> partial dislocations shears the precipitate on adjacent {111} glide planes. Other authors have proposed that the microtwins are formed by the passage of a/3<112> superlattice. Although this mechanism seems quite feasible since it accounts for the ordered L12 structure to be preserved, shearing the crystal by such a large displacement would require high stresses. Since then, little attention has been given until recently since it seems that this mechanism is highly dependent upon alloy chemistry, γ’ precipitate size scale/morphology, γ channel spacing, orientation, stress, and temperature.

The occurrence of microtwins as a prime deformation mechanisms in crept nickel based single crystal blade superalloys [51, 52, 56] and more recently in polycrystalline disk superalloys [54, 60, 73] have been reported; however, there are opposing views in the literature as to how microtwins initiate and propagate. Ardakani et al [51, 55] and Kakehi et al [56] have not provide an explanation as to how microtwins form in their
respective studies; however, they did mention that twin formation by a a/3<112> dislocation may be the root cause. In contrast, Knowles and Chen [52] have shown through TEM work in CSMX-4 that twins are almost always associated with SESF instead of SISF’s, which suggests that microtwins may be produced by an entirely different mechanism.

Kolbe on the other hand has proposed a hypothesis that microtwins can form by the pairwise passage of identical a/6<112> Shockley partial dislocations that shear through both γ and γ’ on an adjacent {111} glide planes [59]. Taking into account that shear of the γ’ precipitates would result in a high energy CSF for the passage of a single a/6<112> Shockley partials dislocation through γ’ and a two layer CSF would form from the pair, Kolbe proposed that diffusion mediated atomic reordering in the wake of the leading partials is required to revert the pseudotwin structure created in the γ’ into a true twin structure with reshuffling of atoms to establish the correct nearest Ni-Al neighbor bonds as that in the L12 order γ’ structure. Although direct TEM evidence was not given, it was later substantiated through direct TEM evidence that shown that microtwins do actually form when both the γ matrix and γ’ precipitates are sheared conservatively by identical, paired a/6<112> Shockley partial dislocations traveling on adjacent type octahedral glide planes [54, 59].

5.4.5 Proposed Mechanism for Microtwin Nucleation

Based on the results from the room temperature in-situ TEM straining experiments performed on the pre-crept material, it seems quite logical that microtwins can form by cross-slip and subsequent dislocation dissociation process where it is possible to create
leading Shockley partial pairs of the same Burgers vector that could then cooperatively shear the coarser secondary $\gamma'$ precipitates and create a microtwin in the manner as originally described by Kolbe [59]. In this model the perfect matrix dislocation that is emitted from the dislocation source has a $a/2[\overline{1}0]$ Burgers vector while the leading and trailing Shockley partial dislocations have a $a/6[\overline{2}11]$ and $a/6[\overline{1}2\overline{1}]$ and Burgers vector, respectively. To illustrate how this is possible a series of schematics is presented in Figure 5.10 and a step-by-step explanation of the process is given as follows:

Step 1: A $a/2[\overline{1}0]$ dislocation is emitted from a dislocation source such as a carbide, boride, or grain boundary. (Figure 5.10a)

Step 2: The $a/2[\overline{1}0]$ dissociates into a leading $a/6[\overline{2}1\overline{1}]$ and trailing $a/6[\overline{1}2\overline{1}]$ Shockley partial dislocations. The lead partial dislocation is pinned at $\gamma/\gamma'$ interface as it would create a CSF if it were to shear the $\gamma'$ precipitate. (Figure 5.10b)

Step 3: Since the leading Shockley partial dislocation does not shear the secondary $\gamma'$ precipitates it instead forms a Shockley loop around it. (Figure 5.10c)

Step 4: On a parallel $\{111\}$ glide plane another $a/2[\overline{1}0]$ is released from dislocation source. (Figure 5.10d)

Step 5: The $a/2[\overline{1}0]$ cross slips onto a $\{111\}$ plane adjacent to the already established stacking fault. (Figure 5.10e)

Step 6: The second $a/2[\overline{1}0]$ dislocation dissociates into a second set of Shockley partial dislocations with possessing the same Burgers as the leading and trailing Shockley partial dislocations. (Figure 5.10f)

Step 7: As the leading Shockley partial dislocation comes into contact with the looped $\gamma'$ precipitate, the now identical, paired $a/6[\overline{2}11]$ on adjacent $\{111\}$ glide planes can now cooperatively shear the $\gamma'$ creating a 2-layer CSF, which can then reorder into a 2-layer SESF and begin to form a microtwin following the Kolbe mechanism. (Figure 5.10g)

Step 8: This process begins to repeat itself when another $a/2[\overline{1}0]$ is emitted from the same dislocation source. The dislocation would then cross slip onto an adjacent $\{111\}$ glide plane and begin to thicken the microtwin. (Figure 5.10h)
5.5 Conclusions

Through a detailed TEM characterization study of the early stages of creep deformation, it was possible to identify discrete locations where microtwins may have nucleated. The following conclusions can be drawn from this study:

1) It was found that carbides and borides, which are either situated at the grain boundaries or within the grain interiors, acted as dislocation sources of $a/2<110>$ matrix dislocations.

2) Due to a narrow $\gamma$ matrix channel the perfect $a/2<110>$ cannot bow between the $\gamma'$ precipitates and so it dissociates into a leading and trailing $a/6<112>$ type Shockley partial dislocations. It is evident the leading Shockley partial dislocation decorrelates from the trailing Shockley partial since matrix intrinsic stacking faults are left in its wake.

3) The dislocation dissociation and decorrelation process maybe be due to a combined effect between the fine $\gamma$ channels, orientation effects, and Suzuki segregation.

4) From room temperature in-situ TEM straining experiments microtwins were found to develop in the $\gamma$ matrix by a cross slip and dissociation process.

5) The experimental observations were used to propose a new model for microtwin nucleation whereby identical paired Shockley partial dislocations form adjacent $\{111\}$ glide planes setting up a situation where it can shear the $\gamma'$ precipitates by the microtwinning mechanisms originally proposed by Kolbe [59].
Figure 5.1: BFSTEM image of the deformation substructure following creep to 0.05% strain at 677°C and 724MPa.
Figure 5.2: BF TEM micrographs depicting the locations grain boundary and grain boundary carbide/boride deformation sources in Ni-base superalloy Rene 104 that the was crept at 677°C and 724MPa to 0.05% plastic. $B=[112]$, $g=111$
Figure 5.3: TEM identification of an MC intragranular carbide that acts as a dislocation source. a) BF TEM image of the carbide and b) corresponding EDX spectrum.

Figure 5.4: TEM analysis of a grain boundary phase through spot EDX analysis of the chemical composition. A) BF TEM image highlighting the location of the spot EDX and b) corresponding EDX spectrum. Inset EDX spectrum at the lower keV showing the presence of both carbon and boron.
Figure 5.5: BF TEM micrographs of diffraction contrast analysis of the perfect and partial dislocations bounding a stacking fault in the $\gamma$ matrix with the following imaging conditions: a) $g=\overline{111}, B=[211]$ b) $g=1\overline{11}, B=[211]$ c) $g=\overline{2}20, B=[111]$, d) $g=2\overline{2}0, B=[111]$, e) $g=\overline{2}02, B=[111]$, f) $g=2\overline{0}2, B=[111]$, g) $g=0\overline{2}2, B=[111]$, h) $g=0\overline{2}2, B=[111]$, i) $g=\overline{1}11, B=[110]$, j) $g=\overline{1}1\overline{1}, B=[110]$. 

Continued
Figure 5.5 Continued
Figure 5.6: Sequence of room temperature in-situ TEM straining of the material pre-crept to 0.05% strain at 677°C and 724MPa.
Figure 5.7: Creation of a twin in an fcc crystal by the passage of identical $a/6<112>$ Shockley partial dislocations on adjacent $\{111\}$ glide planes. a) perfect fcc crystal b) intrinsic stacking fault c) extrinsic stacking fault d) twin.

Figure 5.8: Creation of a true twin structure by the passage of identical $a/3<112>$ super Shockley partial dislocations on adjacent 111 planes. a) perfect L1$_2$ crystal structure b) superlattice intrinsic stacking fault c) superlattice extrinsic stacking fault and 3) true twin with correct nearest neighbor bonds.
Figure 5.9: Creation of a pseudotwin structure by the passage of identical a/6<112> Shockley partial dislocations on adjacent 111 planes. a) perfect L1$_2$ crystal structure b) complex stacking fault c) 2-layer complex stacking fault and 3) 3-layer fault or pseudotwin with incorrect nearest neighbor bonds.
Figure 5.10: Proposed model of cross-slip dislocation mechanism for microtwin formation.
Step 4: An additional $a/2<110>$ dislocation is emitted from the dislocation source on a parallel {111} glide plane either adjacent to the faulted layer below or a few atomic layers above.

(d)

Step 5: The $a/2<110>$ dislocation cross-slips on to the adjacent and parallel {111} glide plane.

(e)

Step 6: The $a/2<110>$ dissociates into a leading and trailing $a/6<112>$ Shockley partial which are identical to the on the Shockley partial dislocations on the glide plane below. This creates a dislocation configuration that involves paired $a/6<112>$ Shockley partial dislocations that are operative on adjacent {111} glide planes that have identical Burgers vectors.

(f) Continued
Step 7: The paired Shockley partial dislocations with identical Burgers vectors that are on adjacent \{111\} glide planes shear through the \(\gamma'\) precipitate creating initially, a 2-layer CSF which is converted to a 2-layer SESF after atomic reordering.

(g)

Step 8: An additional a/2<110> dislocation is emitted from the dislocation source, which cross slips onto the \{111\} glide plane adjacent to the previously defined 2-layer stacking fault configuration. The a/2<110> further undergoes a dissociation event and creates and additional layer of Shockley partial dislocations which begin to creating a microtwin in the \(\gamma\) matrix and \(\gamma'\) precipitates.

(h)
CHAPTER 6

SUMMARY

An investigation into the creep deformation mechanisms of a newly developed Ni-base superalloy was conducted in this study. Ni-base disk superalloy Rene 104 was developed specifically for use as that material for a turbine disk in aircraft gas turbine engines where it will be subjected to elevated temperature creep deformation during actual service conditions. Therefore, it was important to conduct an in depth deformation characterization study in order to correlate microstructure with the underlying creep deformation mechanisms and overall macroscopic creep deformation behavior. Such a correlation would give aircraft engine manufacturers a fundamental understanding of what microstructural features can be tailored for improved creep resistance and the fundamental processes that control deformation in these alloys.

Creep specimens were extracted from turbine disk forgings that had been given a proprietary supersolvus heat treatment. This specific heat treatment resulted in the development of a bimodal $\gamma'$ precipitates size distribution. After creep deformation at different levels of stress and temperature, a detailed TEM characterization study was conducted in order to identify creep deformation mechanisms and correlate this with the macroscopic creep deformation behavior. It was found that a variety of deformation
mechanisms have become operative. Examples of the different deformation mechanisms that have been observed during creep deformation are: (1) Orowan looping and shearing of $\gamma'$ particles by coupled a/2<110> dislocations, (2) isolated faulting of the $\gamma'$ particles, (3) thermally-activated microtwinning, (4) extended, continuous faulting of precipitates and matrix, and (5) dislocation climb by-pass at the highest temperatures. The preference for these mechanisms are dependent on factors such as alloy chemistry, the initial microstructure, stress, and temperature.

The effect of microstructure on dictating creep rate controlling deformation mechanisms was revealed for specimens with a bimodal $\gamma'$ size distribution that possessed different secondary $\gamma'$ size, tertiary $\gamma'$ volume fraction, and $\gamma$ channel width spacing. It was found that the less creep resistant microstructure was the one with a greater secondary $\gamma'$ size, wider $\gamma$ channel width, and higher volume fraction of tertiary $\gamma'$. Deformation in this microstructure commences by way of a/2<110> dislocations that are concentrated in the $\gamma$ matrix at lower strains, which then transition to a SISF precipitate shearing mode at larger strains. The more creep resistant microstructure possessed a finer $\gamma$ channel width spacing, which promoted a/2<110> dislocation dissociation into a/6<112> Shockley partials at lower strains and microtwinning at higher strains.

A more in depth investigation on the microtwinning deformation mechanisms was conducted in order to identify how microtwins nucleate. Grain boundary and intragranular carbides were determined to be the sources of deformation when examined at lowest amount of creep strain. The dislocations that were emitted from these sources immediately underwent a dislocation dissociation process. The dislocation dissociation and decorrelation process maybe be due to a combined effect between the fine $\gamma$ channels,
orientation effects, and Suzuki segregation. The experimental observations were used to propose a new model for microtwin nucleation whereby identical paired Shockley partial dislocations form adjacent \{111\} glide planes setting up a situation where it can shear the $\gamma'$ precipitates by the microtwinning mechanisms.
CHAPTER 7

RECOMMENDATION FOR FUTURE RESEARCH

The research presented in this dissertation was focused on developing a fundamental understanding of the mechanisms that are responsible for creep deformation. Moreover, it was shown that the operative deformation mechanisms were highly influenced by the internal factors such as the size scale of microstructural features and external factors such as stress and temperature. Based on these results a recommendation of future research activities is presented below.

In Chapter 3 it was found that there are a variety of deformation mechanisms that have been observed and of which were affected by the stress and temperature at which specimens with approximately similar starting microstructure. A creep deformation mechanism map was created with only a limited samples that were thoroughly characterized. To develop the deformation map further, it would be better conduct creep experiments with an expansion of the experimental test matrix. Furthermore, the deformation mechanism map does not take into account the $\gamma'$ precipitate size scale, $\gamma'$ volume fraction, and $\gamma$ channel width spacing. The combinations of these microstructural features are known to have a large influence on the underlying creep deformation
mechanisms. The deformation mechanism map could then be constructed in a 3-d plot where the axis would be stress, temperature and microstructure.

The identification of deformation mechanisms presented in this study has only been characterized for specimens, which were crept. The effect of strain rate on deformation mechanisms therefore has not yet been explored in any great detail for this particular superalloy. There has been a significant amount of high temperature tensile testing that has already been performed as part of mechanical property measurements such as yield strength, ultimate tensile strength, etc. The deformation mechanisms may be significantly different for faster strain rates experience in common tensile tests as opposed to strain rates that are considerably lower as in creep tests.

An interesting deformation mechanism that was identified through TEM characterization was microtwinning. As previously mentioned, this mechanism is quite surprising to observed under low strain rate creep conditions. There have been some more recent experimental findings that show segregation of heavier elements to the microtwins interface. This was characterized using HRSTEM imaging. It is important to know precisely what the elements that are segregating to this interface are since this most likely will have an influence on that rate at which the twins thicken and also on the atomic reordering kinetics that occur during the microtwinning process. This can be accomplished with fine probe EELS analysis of the atomic columns that show this segregation and also through 3-d atom probe tomography.

In Chapter 4, the effect of microstructure on creep deformation mechanisms was experimentally explored where it was found that by altering the heat treatment conditions, one can alter the microstructure which will in turn have an influence on the
deformation mode and macroscopic creep behavior. It would be a good idea to further explore the effect of microstructure size scale by conducting creep experiments under much wider range of microstructures and test conditions. The heat treatment that is generally applied to the disk superalloys results in a bimodal $\gamma'$ size distribution. The heat treatment could be altered such that it will produce a monomodal, bimodal or multimodal $\gamma'$ size distribution. A systematic creep study could then be performed in order to determine the heat treatment and hence microstructure that would be tailored for optimal creep resistance.

In Chapter 5, the microtwinning mechanism was further explored by identifying microtwin nucleation sources and tracking their evolution into fully developed microtwins. In-situ TEM straining was conducted in order to experimentally observe the microtwinning process as it is occurring. Only room temperature straining results could be interpreted since in the high temperature straining experiments the material had developed cracks that propagated along grain boundaries and therefore results could not be attained. It is important that the high temperature in-situ deformation studies are continued since this could provide valuable information on how the material behaves at high temperature.
APPENDIX A

EFFECT OF THERMAL EXPOSURE ON MICROSTRUCTURAL EVOLUTION

Isothermal aging experiments were conducted in order to determine an approximate time in which the dissolution of the tertiary $\gamma'$ precipitates occurs since it was experimentally observed that dissolution of the tertiary $\gamma'$ precipitates has occurred during creep at 760ºC and 815ºC. Cube specimens that were approximately 125mm$^3$ were placed into an alumina crucible and subjected to an isothermal aging heat treatment in a horizontal tube furnace in air at 815ºC for 24, 48, 72, 96 and 120 hrs. When the aging heat treatments were completed, the samples were mounted in conductive bakelite and prepared using standard metallographic techniques. An etchant consisting of HNO$_3$, HF and Lactic acid was applied in order to selectively etch the $\gamma'$ precipitate phase. Using a swab technique, the etchant was swabbed on the surface for approximately 10 seconds. The microstructure was then imaged using an FEI Sirion SEM using a high resolution through lens detector in UHR mode.

During the microscopic evaluation of the $\gamma'$ precipitate microstructure, representative images were acquired in regions where the tertiary $\gamma'$ precipitates tend to be concentrated in such as in areas between the coarser secondary $\gamma'$ and/or along grain boundaries. Representative images following the aging heat treatments are presented in
Figure A1. Tertiary γ’ precipitates are observed at aging times approaching 72 hrs where only the coarsest tertiary γ’ precipitates were stable. After longer aging times all of the tertiary γ’ precipitates begin to dissolve as shown after 96 and 120hrs. This simple aging study demonstrates how quickly the microstructure can change during thermal exposure at elevated temperatures. Based on these results and its implications that they have on creep deformation behavior and long term alloy stability, a further study aimed at determine the coarsening and dissolution kinetics of the γ’ precipitate phase is warranted if the projected use of this material is to be used at increasing operating conditions.
Figure A1: Representative SEM images depicting the evolution of the $\gamma'$ precipitates during an isothermal aging heat treatment at 815°C for a) 0hrs b) 24hrs c) 48hrs d) 72hrs e) 96hrs and f) 120hrs.
APPENDIX B

ETMT TESTING TO DETERMINE YIELD STRENGTH vs. TEMPERATURE

The Electrothermal Mechanical Testing System (ETMT) was developed by the National Physical Laboratory and Instron to measure the mechanical properties of materials and components. It has the capability of performing elevated temperature tensile testing through the use of a resistive heating device and therefore was chosen to experimentally determine yield strength as a function of temperature for the Ni-base superalloy used in this study. The ETMT system has recently been used to determine the high temperature mechanical properties of In 718 and CMSX-4 by Roebuck et al [93]. In terms of tensile testing in Roebuck’s study, true strain was computed based on the measured change of resistance during the test. A more precise method of measuring strain using image based correlation techniques has been demonstrated by B. Peterson as part of his Ph.D. dissertation on the use of the ETMT for mechanical behavior testing of titanium alloys. For further details of the advantages of using this measuring technique refer to B. Peterson’s Ph.D. dissertation from the Department of Materials Science and Engineering at The Ohio State University 2008 [94].

Specimens were extracted from the same region of the turbine disk forging where the creep specimens tested in Chapter 4 were extracted. Specimens were EDM’ed into
rectangles measuring 40mm in length and a cross section of 1 x 2mm. The specimens were then sprayed with high temperature paint: a white base coat and a speckled black top-coat. A thermocouple was attached to the central portion of the test piece to control and monitor the temperature. A resistive heater was used to bring the specimen to the test temperature where they were all strained at a strain rate of $1.8 \times 10^{-3}$. Strain was determined through image correlation with a high resolution CCD camera and Vic-2D digital image correlation software using the 2 point linear extensometer function. An example of the images acquired during testing for later post processing and image correlation is shown in Figure B1.

Engineering stress vs strain curves were generated and the yield strength at 0.2% offset was determined for the rim and bore microstructure under a range of test temperatures between 649-760°C. The experimental stress vs. strain curves are presented in Figure B2 along with the 0.2% offset yield strength as a function of temperature data that is presented in Figure B3. As was to be expected the yield strength decreases with temperature for both the rim and bore microstructures. However, there appears to be an outlier for the rim microstructure tested at 649°C. The yield strength is much lower than the yield strength measured at 677 and 704°C. Since there does appear to be some scatter in the data several addition constant strain rate tensile tests should be replicated under the same test conditions in order to gain a better statistical representation.
Figure B1: Experimental images of an actual ETMT test acquired using a CCD camera during tensile testing of the rim microstructure tested at 760°C a) before heating and deformation, b) during straining and c) after final fracture. The speckled dots on the surface of the specimen are high temperature paint that was applied for image correlation processing using Vic-2D software and for strain measurements.
Figure B2: Plots of engineering stress vs. strain acquired from ETMT testing of the a) rim and b) bore microstructures for at different temperatures.
Figure B3: Yield strength (0.2% offset) as a function of temperature.
There has been little research on comparing the tension and compression creep behavior of polycrystalline γ′ strengthened Ni-base superalloys. Recently, Sondhi et al have investigated the tension-compression creep asymmetry in polycrystalline turbine disk superalloy IN100 and reported a consistent difference in tension vs. compression creep behavior for specimens crept at 704°C at stresses of 400 and 738MPa [95]. The creep specimens tested in tension exhibited an inverse primary transient where the initial creep rate is extremely low but does accelerate as creep deformation progresses. Conversely, a normal primary transient creep curve is observed in compression where the initial creep rate is rapid but slowly decelerates to a constant minimum strain rate. The authors attribute the creep asymmetry to the role of an internal compressive stress field which is presumed to be present within the matrix and that will favor the initial high creep rates in compression vs. tension. A major limitation of this work is that the authors did not report the fundamental physics behind deformation by characterizing the underlying deformation mechanisms, which govern the macroscopic creep response. It was the purpose of this study to explore the creep deformation mechanisms that results after tensile and compression creep experiments.
Specimens were extracted from the rim region of a fully heat treated turbine disk forging. A complete analysis of the microstructure has been completed and is presented in Chapter 4. For tensile creep experiments, strain was measured using a dual averaging extensometer assembly while a single averaging extensometer was used for compression creep experiments. Images of the specimen geometry and experimental setup can be seen in Figure C1 and Figure C2 or both the tensile and compression creep tests a stress of 724MPa and a test temperature of 677ºC was chosen. Creep tests were carried out to approximately 0.2% plastic strain. The experimental creep curves can be seen in Figure C3. As can be seen, both microstructures exhibit normal transient creep behavior where there is an initial period of rapid strain accumulation during the early stages of creep followed by a period of decreasing strain rate. Initially, the samples creep more rapidly in compression as compared to tension and eventually the compression creep specimen reaches 0.2% strain first in approximately 400hrs while the specimen crept in tension reaches 0.2% strain in approximately 750hrs.

To explore the differences in creep deformation behavior, the deformation substructure was characterized using diffraction contrast TEM characterization techniques. A thorough detailed analysis of the deformation microstructure after deformation in tension was previously reported in Section 4.3.3.1.2 where numerous stacking faults were observed predominately in the \( \gamma \) matrix phase of the material. These faults were identified to be intrinsic stacking faults and they have arisen due to the dissociation of \( a/2<110> \) type matrix dislocations into two \( a/6<112> \) Shockley partial dislocations. This clearly shows that it is the matrix phase where a majority of the plastic deformation is being concentrated.
In compression, several mechanisms were found to be simultaneously operative. The primary deformation mode is similar to the matrix intrinsic stacking faults that were observed during tensile creep. A representative BF and CDF image of the matrix stacking faults and identification of the nature of the faults is presented in Figure C4. In addition to the matrix intrinsic stacking faults, continuous stacking faults that shear through both the $\gamma$ matrix and $\gamma'$ precipitates were observed. (Refer to Figure C5) Through BF and CDF imaging pairs the continuous stacking faults were identified to be superlattice extrinsic stacking faults. The increase in the amount of creep deformation that occurs in compression, as compared to tension, may be attributed to the combination of $\gamma$ matrix deformation (via intrinsic stacking faults) and continuous shearing of $\gamma/\gamma'$ by an SESF related shearing mechanism. Additional work needs to be continued in order to compare grains with similar crystallographic orientations crept in tension and compression to determine if there are any orientation effects and differences in deformation mechanisms.
**Figure C1:** Specimen geometries used for tensile (round bars 2.25) and compression (parallelepiped 4x4x12mm) creep specimens.

**Figure C2:** Experimental creep testing setup for a) tensile creep with a dual averaging extensometer and b) compression creep with a single averaging extensometer.
Figure C3: Experimental creep curves comparing the creep behavior between tension and compression creep behavior for the rim microstructure at 677°C and 724MPa.
Figure C4: a) BF and b) CDF TEM micrographs of the rim microstructure crept in compression at 677°C and 724MPa depicting matrix stacking faults that shear through are deforming only the γ matrix. The faults were identified to be intrinsic in nature.

Figure C5: a) BF and b) CDF TEM micrographs of the rim microstructure crept in compression at 677°C and 724MPa depicting continuous stacking faults that shear through the γ matrix and γ' precipitates. The faults were identified to be superlattice extrinsic stacking faults.
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