Exploration of Local Strain Accumulation in Nickel-based Superalloys

Dissertation

Presented in Partial Fulfillment
of the Requirements for the Degree
Doctor of Philosophy
in the Graduate School
of The Ohio State University

By
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The Ohio State University
2012

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Abstract

Deformation in polycrystalline nickel-based superalloys is a complex process dependent on the interaction of dislocations with both the intra-granular γ′ particles and the grain boundaries. An extensive body of work exists on understanding the interaction between dislocations and the γ′ particles, but understanding the interaction between dislocations and grain boundaries has been historically hindered by the experimental techniques. In this work a full field strain mapping technique was developed and utilized to explore surface strain accumulation at grain boundaries of René 104 samples with different microstructures. The full field strain mapping technique utilized Correlated Solutions VIC-2D software for digital image correlation to measure strain accumulation from secondary electron images taken during constant load tests at elevated temperature. This technique indicated that the two different microstructures of René 104, one with microscopically flat grain boundaries and the other with serrated grain boundaries, accumulate strain by different methods. Analysis of discrete offsets in grid lines placed prior to deformation indicate that grain boundary sliding (GBS) is an active deformation mechanism at these temperature and strain rate regimes, and that the development of serrated high angle grain boundaries can decrease the activity of this mechanism by 30%. Slip transmission parameters, which mathematically assess the ease of slip transmission across a grain boundary, were calculated based on grain boundary misorientation and grain boundary trace. These
parameters proved unsuccessful at predicting strain localization sites in these materials, indicating that slip transmission is not the only factor dictating strain localization sites.

Full field strain maps were used to site-specifically extract grain boundaries of interest to study dislocation interaction and sub-surface grain boundary neighborhood. Representative from each of four types of boundaries was selected for scanning transmission electron microscopy (STEM) analysis: high angle grain boundaries that either did or did not experienced strain accumulation, high angle grain boundaries that experienced GBS, and special annealing twin boundary that experienced both strain accumulation and grain boundary sliding. The STEM analysis indicates that high angle grain boundaries that experienced strain accumulation showed increased dislocation content near the grain boundary, while a grain boundary that did not show accumulation and a grain boundary that experienced grain boundary sliding showed no indication of increased dislocation content near the grain boundaries. The STEM analysis also indicated that grain boundary surface roughness and the sub-surface grain boundary neighborhood was substantially more complex for the grain boundaries that experienced strain accumulation as compared to the boundary that experienced GBS.

Since STEM foils only provide a two dimensional representation of the grain boundary surface, serial sectioning data sets were reconstructed from stacks of 2D images acquired using the Focused Ion Beam (FIB). These reconstructions confirm that indeed the surface roughness of a boundary that experienced strain accumulation was an order of magnitude greater than a grain boundary that experienced GBS. The observations from the STEM and serial sectioning work indicate that grain boundary
neighborhood and grain boundary topography also need to be considered if models are to predict strain localization and GBS sites.
Dedication

Dedicated to my husband Robert Carter who is my rock.
Acknowledgments

This work was funded by the Air Force Research Laboratory and Air Force Office of Scientific Research under the STW-21 program FA9550-09-1-0014. René-104 material was provided by GE Aviation, and special thanks should go to Andrew Wessman for providing the necessary heat treatment parameters. OIM data was collected and in-situ experiments were conducted using equipment at WPAFB.

Special thanks should go out to each of my collaborators. Thank you for making this body of work more interesting and substantial to the Superalloy and Engineering communities. First, I would like to thank my advisor Dr. Mills, for dealing with me when I make mountains out of mole-hills, and being there when I really needed your advice. Next, the members of the dissertation committee, Dr. Fraser and Dr. Wang, should be commended for participating in this research; your insight and collaboration was indispensable. Dr. Uchic deserves recognition for being an active collaborator and mentor during this process. Thank you for listening to my concerns and working to put me in contact with the right people to fix each problem that arose during the process.

In no particular order, all of the other collaborators should also be thanked. Their participation in this body of work was no less valuable. Each in your own way have motivated me to be a better person. Adam Pilchak, thanks for sharing the
techniques for collecting better OIM data and many a useful conversation about things related to this project, research, and life in general. As the primary collaborator on the slip transmission parameters discussed in Chapter 9, without you the Matlab code would never have materialized in all its glory. To Robert Wheeler, thanks for spending the time to help me work through the little issues that plague the Fullam Tensile Stage.

I would like to thank Aimee Price for her friendship and help in the development of the e-beam lithography techniques. It is still amazing that it worked so relatively easily. John Sosa and Paul Shade need to be commended for dedicating hours of work related to the serial sectioning data collection and visualization. Without them, Chapter 10 would not exist. Special thanks to Michael Kuper, who took on the task of measuring thousands of grain boundary sliding instances, and characterizing the particle distributions in these materials. Ning Zhou was instrumental in the development of the Phase Field Modeling predictions, he supported this work even after he got a real job; your dedication is commendable. Patrick Phillips and Matt Brandes provided valuable insight into the operation of the FEI Tecnai for scanning transmission electron microscope, without you my images would probably still be badly stigmated, and it would have taken me much longer to understand the bigger picture of kikuchi patterns.

I would also like to take a moment to thank all the other graduate students who have been my friends during this process. Without you all graduate school would not have been the wonderful experience that it has been. Thank you for listening and providing support related to my PhD research, and probably more importantly for helping me maintain what little sanity I have left.
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Chapter 1: Introduction

Nickel based superalloys are an important class of materials specifically designed to withstand the operating environment of aircraft gas turbine jet engines. Their physical properties make them aptly suited for this extreme environment; with polycrystalline alloys operating upwards of 600°C under high stress states, and corrosive atmosphere. Creep deformation is of great concern for turbine jet engine applications of these alloys since this time dependent deformation mechanism occurs readily at elevated temperature. Understanding and predicting the behavior of full-scale components is imperative for creating substantial advances in the design of the next generation of superalloys.

Currently, the development of physics-based full-scale deformation models with predictive capabilities for these high-temperature alloys systems is hindered by the type of validation data that can be provided via traditional testing techniques. Traditional tensile and creep tests determine averaged bulk properties, but provide limited insight into local deformation behavior. Even with post-testing microstructural evaluation, only a limited understanding can be gleaned relative to how local strain accommodation is transmitted across grain boundaries. This lack of localized characterization data makes it difficult to directly apply this information to
computational models. Therefore, novel experimental techniques are required to characterize the local deformation fields produced during creep of these materials.

This dissertation focuses on the mechanisms of creep deformation at grain boundaries in these alloys. In particular, attention has been paid to how grain boundary morphology and misorientation relationships play a role in the localized creep deformation of these materials. The dissertation is divided into chapters and explores the following key points. Chapter 2 presents a literature review which discusses the physical metallurgy of nickel based superalloys, and how constitutive phases contribute to the deformation of these materials at a dislocation level. It will also discuss our current understanding of how grain boundaries contribute to deformation at elevated temperatures and explore novel experimental techniques that can be adapted to explore local strain accommodation at the grain size scale.

The research entailed the development and utilization of new in-situ experimental techniques that provided full-field strain information, which was coupled with microstructural characterization to quantify how grain-to-grain interactions affected local strain distributions at elevated temperatures. Chapter 3 expounds on the techniques needed for sample preparation for microstructural evaluation, and the new experimental set-up for the in-situ scanning electron microscope technique.

Chapter 4 describes the characteristics of the two material systems analyzed in this body of work. Two microstructures of René-104 were prepared through different super-solvus heat treatments to create statistically-equivalent grain size distributions with very different macroscopic grain boundary structures. The standard heat treatment
used for turbine disc application creates macroscopically planar grain boundaries and a bimodal distribution of $\gamma'$ particles, while a “serrated” heat treatment uses heterogeneous nucleation of $\gamma'$ particles on the grain boundaries to create macroscopically serrated grain boundaries and a quadmodal $\gamma'$ particle distribution.

Chapter 5 discusses imaging, and patterning optimization for digital image correlation using Correlated Solutions VIC-2D software. It can be seen from this analysis that several factors contribute to intensity and spatial strain resolution, including image integration techniques, speckle orientation and speckle pattern density.

In Chapter 6, the development of local strain distributions as a function of time at constant load, during creep, will be explored in the two microstructures of René-104. The full field strain maps indicate that these two materials accumulate strain differently. The standard microstructure shows accumulation along grain boundaries and triple points, while the serrated microstructure first localizes slip over large regions at approximately 45° angles to the tensile axis indiscriminate of the underlying microstructure. These two materials also experience differing amounts of grain boundary sliding (GBS). In this chapter we characterize this by conventional theories developed by the superplasticity community (expounded in Chapter 2), as well as develop a new empirical relationship.

Since most of the observations in Chapter 6 are statistical in nature, it was desired to be able to quantitatively characterize each grain boundary that did or did not show strain accumulation or grain boundary sliding. In Chapter 7, fracture prediction parameters developed for gamma-TiAl are altered to account for the crystal geometry
and the full and partial dislocation motion in these superalloys materials. Code was developed to characterize “hot” and “not” boundaries based on their crystallographic orientation, and grain boundary orientation to determine if any existing parameter could be used to statistically assess which boundaries should exhibit strain accumulation while others do not. This work indicated that only accounting for grain boundary misorientation was not enough to predict strain localization sites.

Chapter 8 compares predicted slip systems and deformation mechanisms from Schmid Law and Phase Field Modeling with experimental observations. Analysis from both slip trace analysis from post deformation scanning electron microscopy (SEM) images and scanning transmission electron microscopy (STEM) brightfield (BF) images concluded that in most cases Schmid Law could accurately predict the active slip plane. STEM BF images were also used to characterize both dominant intra-granular and intergranular dislocation mechanisms and compare these with Phase Field Predictions. Using the Schmid Laws it was shown that a Phase Field model could be used to predict the dominant deformation mechanism in the grain interiors. These predictions break down near grain boundaries that experienced strain accumulation. The STEM analysis indicates that annealing twin boundaries deform inherently different than random high angle grain boundaries and that high angle grain boundaries that experience strain accumulation have more complex sub-surface grain boundary structure when compared to grain boundaries that experience grain boundary sliding.

Since STEM analysis is inherently a two dimensional representation of the three dimensional nature of the grain boundary structure, Chapter 9 explores the three
dimensional structure of select grain boundaries through the creation and analysis of reconstructed volumes of microstructure.

Finally, Chapter 10 discusses the conclusions from the body of work, including general conclusions based on the experimental techniques and more focused conclusions of specific interest to the community of researchers discussing superalloy materials. In this chapter, future work will also be discussed.
Chapter 2: Literature Review

2.1 Introduction

The development of nickel-based superalloys began in the late 1930’s with the birth of gas turbine aircraft engines [1]. These alloys are extensively used in the hottest sections of these engines due to their superior strength, toughness and tolerance to environmental degradation at elevated temperatures [2]. The application of nickel-based superalloys in more demanding environments of next generation engines is dependent on our understanding of how alloy chemistry and processing affect the microstructure and in-turn mechanical and corrosive properties of these materials.

This chapter discusses the fundamental physical metallurgy of nickel-based superalloys and how their microstructure determines mechanical properties. An emphasis has been placed on exploring how microstructural features, such as: $\gamma'$ precipitate distribution, grain size, and alloying additions control creep deformation mechanisms. A brief discussion is also presented describing how these microstructural features dictate other mechanical properties such as tensile strength and fatigue resistance. A section is dedicated to discussing grain boundary sliding, since there is evidence that grain boundary sliding is likely to contribute to creep deformation at the higher operating temperatures required for next generation turbine engines. These
sections are intended to highlight to the reader the current lack of understanding of how grain boundaries contribute to local creep strain distribution. Finally, procedures for characterizing localized strain fields associated with grain boundary sliding and other inter and intra-granular mechanisms will be presented, including the conventional marker offset method and newer digital image correlation techniques. This chapter will illuminate the need for developing in-situ testing techniques to further explore the local deformation behavior of nickel-based superalloys.

2.2 Physical Metallurgy of Nickel Based Superalloys

The microstructure and in turn engineering properties of a superalloy rely heavily on the chemical composition and thermal mechanical processing of the alloy in question. Superalloys in their simplest form are a solid-solution strengthened nickel matrix, with a dispersion of coherent ordered phases: γ’ (Ni3Al,Ti), or γ” (Ni3Nb) [1]. For this discussion, only the γ’ phase will be considered. Also present are several carbide and boride phases that can contribute beneficial properties depending on morphology [2], and Topologically Close Packed phases (TCP) that are deleterious to properties and are thus avoided through alloying additions. Over decades of development, the complexity of the superalloy composition has greatly increased, producing industrial alloys that contain ten or more alloying elements, while over thirty other contaminant elements must be controlled [3, 4].
2.2.1 **Nickel Matrix**

The nickel matrix (γ) has a face centered cubic structure; this highly symmetric close packed structure possesses high ductility. Alloying elements with similar atomic radii to nickel (0.125nm [5]) will preferentially partition to the γ phase. These include: cobalt, iron, chromium, ruthenium, molybdenum, rhenium and tungsten. To some extent each of these elements provides solid-solution strengthening due to differences in structure, lattice parameter, and modulus as compared to nickel, however tungsten, molybdenum and chromium are the most potent strengtheners [6]. These γ partitioning elements also lower the stacking fault energy of the γ matrix, which dramatically effects the nature of dislocation motion [1, 6]. A list of most alloying effects is presented in Table 2.1. The effects listed do not include complications that arise due to interactions between alloying elements [7].

<table>
<thead>
<tr>
<th>Element</th>
<th>Partitioning</th>
<th>atomic radii (nm)</th>
<th>Effects</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>γ, (GB)</td>
<td>0.125</td>
<td>γ SSS, oxidation resistance</td>
</tr>
<tr>
<td>Co</td>
<td>γ</td>
<td>0.125</td>
<td>γ SSS, carbide formation</td>
</tr>
<tr>
<td>Mo</td>
<td>γ, (GB)</td>
<td>0.136</td>
<td>γ SSS, carbide formation</td>
</tr>
<tr>
<td>W</td>
<td>γ, (GB)</td>
<td>0.137</td>
<td>γ SSS, carbide formation</td>
</tr>
<tr>
<td>Nb</td>
<td>γ’, γ”</td>
<td>0.143</td>
<td>γ’ strengthener</td>
</tr>
<tr>
<td>Al</td>
<td>γ’</td>
<td>0.143</td>
<td>γ’ former</td>
</tr>
<tr>
<td>Ti</td>
<td>γ’</td>
<td>0.147</td>
<td>γ’ former, carbide former</td>
</tr>
<tr>
<td>Ta</td>
<td>γ’</td>
<td>0.147</td>
<td>γ’ strengthener, carbide former</td>
</tr>
<tr>
<td>C</td>
<td>GB</td>
<td>0.077</td>
<td>GB strengthener, carbide formation</td>
</tr>
<tr>
<td>B</td>
<td>GB</td>
<td>0.097</td>
<td>GB strengthener, boride formation</td>
</tr>
<tr>
<td>Hf</td>
<td>GB</td>
<td>0.159</td>
<td>GB strengthener, carbide former</td>
</tr>
<tr>
<td>Fe</td>
<td>γ</td>
<td>0.124</td>
<td>γ SSS</td>
</tr>
</tbody>
</table>

Table 2.1. The effects of common alloying elements on the properties of Ni-based superalloys (SSS-solid solution strengthening, GB – grain boundary segregation) [1].
Deformation in the $\gamma$ phase occurs on the \{111\} close packed planes, in the close packed directions $\langle 1\bar{1}0 \rangle$. The low stacking fault energy produced by alloying additions allows unit dislocations, $a/2 \langle 1\bar{1}0 \rangle$, to dissociate into pairs of partial dislocations of type $a/6 \langle 2\bar{1}\bar{1} \rangle + a/6 \langle \bar{2}1\bar{2} \rangle$ that are separated by a stacking fault, a local region in which the regular packing sequence has been interrupted [8]. Since each partial dislocation is only glissile on a single \{111\} slip plane the pair of partials must recombine before cross slip can occur, thus making cross slip difficult.

2.2.2 Gamma Prime ($\gamma'$) Phase

Stoichiometric $\gamma'$ is Ni$_3$Al with an L1$_2$ crystal structure, consisting of a primitive cubic cell with aluminum atoms located at the corners and nickel atoms located on the face centers of the cube. The lattice parameter of this structure is 0.357 nm, which is similar to the lattice parameter of nickel (0.3517 nm). Due to the low lattice misfit between $\gamma$ and $\gamma'$, $\gamma'$ precipitates coherently in the $\gamma$ matrix with a cube-cube orientation relationship $\{100\}_\gamma/\{100\}_\gamma'$ and $\langle 100 \rangle_\gamma/\langle 100 \rangle_\gamma'$ [1]. Alloying elements with atomic radii larger than nickel partition to this phase, including: titanium, tantalum, niobium, ruthenium and rhenium (see Table 2.1). These additions also affect lattice misfit, but the cube-cube orientation is still maintained, since the coherency strains remain relatively small [7].

Dislocations propagate in $\gamma'$ along the \{111\} close packed planes but their motion is substantially inhibited by the ordered nature of the structure. The $\gamma$ matrix
unit dislocation, \(a/2 < 1 \bar{1} 0\rangle\), is only half a unit dislocation in the \(\gamma'\) phase, Figure 2.1 (a). Therefore, when a single \(\gamma\) unit dislocation passes through \(\gamma'\) on a \{111\} plane an Anti-Phase Boundary (APB) is produced, Figure 2.1 (b). The creation of an APB is associated with significant energy due to the creation of “wrong” bonds (Ni-Ni and Al-Al). As such, \(\gamma\) unit dislocations most often travel through \(\gamma'\) in pairs so that the second dislocation removes the “wrong” bonds created by the first dislocation. Each \(\gamma\) unit dislocation can also dissociate to produce a pair of partials \(a/6 < 21 \bar{1} > + a/6 < \bar{1} 21 >\) that travel through the \(\gamma'\) as a pair to create a Complex Stacking Fault (CSF). This dissociation produces an APB that is preceded and followed by CSF's, Figure 2.1 (d). These two configurations are often seen during tensile testing [1, 9]:

\[
\text{APB:} \\
a/2 < 1 \bar{1} 0 > + \text{APB} + a/2 < 1 \bar{1} 0 >
\]

\[
\text{CSF-APB-CSF:} \\
a/6 < 2 \bar{1} \bar{1} > + \text{CSF} + a/6 < \bar{1} 21 > + \text{APB} + a/6 < 2 \bar{1} \bar{1} > + \text{CSF} + a/6 < \bar{1} 21 >
\]

At elevated temperatures, thermally activated climb becomes easier making other dislocation configurations possible. For instance, a pair of \(\gamma\) unit dislocations can create a pair of \(a/6 < 21 \bar{1} > + a/6 < \bar{1} 21 >\)-type dislocations which together produce Superlattice Extrinsic and Intrinsic Stacking Faults (SISF and SESF), Figure 2.1 (c).
Figure 2.1. Various fault configurations in L12 structure formed by shearing on the (111) plane (a) initial structure; large, medium and small circles represent atoms in the top, middle and bottom planes respectively. Open and closed circles are Al and Ni atoms respectively. Shearing the top layer by (b) \( \frac{a}{2} < 101 > \) creates an APB, by (c) \( \frac{a}{3} < 2\bar{1}2 > \) creates a SISF and by (d) \( \frac{a}{6} < 1\bar{1}2 > \) creates a CSF [1].

Also, successive passing of the same type of \( \frac{a}{6} < 2\bar{1}\bar{1} > \) partial dislocations on adjacent \{111\} planes can create a pseudo-twin, and by short range reordering cause micro-twin formation, Figure 2.2 [10-12]. The configurations mentioned above have lower energy than an APB configuration because no “wrong” bonds are produced on adjacent \{111\} planes, only on secondary \{111\}. Each of these mechanisms, SISF SESF and micro-twinning, require local reordering to create their configurations so that diffusion is the rate limiting step and thus are only active at elevated temperatures and
slow strain-rates [1, 9, 13, 14]. The exact nature of this reordering is still being researched in the superalloy community, and for this review it is only important to understand that its required [10]. This discussion alludes to the fact that under simple loading conditions the dislocation motion in γ’ can be very complex even when only a single slip system is activated.

Figure 2.2. Schematic of microtwin formation. The top insert shows model geometry and the bottom insert shows the transformation of a pseudotwin to microtwin. The block arrows indicate dislocation motion direction [11].

Dislocation interactions in the γ’ are responsible for the anomalous strength-temperature relationship observed in Ni₃Al and nickel-based superalloys. Most materials exhibit a decrease in strength with increasing temperature. However, Ni₃Al and γ+ γ′ alloys exhibit an increase in strength with increasing temperature up to a peak strength before strength drops with further increases in temperature. This anomalous strength relationship is attributed to cross-slip of the screw component of $a/2 <1 \bar{1} 0>$
type dislocations from the \{111\} to the \{100\} planes. Once cross-slip has occurred, the edge component of the dislocation can no longer glide and the dislocation becomes pinned. Since $a/2 < 1\overline{1}0 >$ type dislocations travel in pairs through the $\gamma'$, this also pins the sister dislocation. This pinning mechanism is termed a Kear-Wilsdorf lock [1, 9, 15]. Strength increases with temperature as cross-slip becomes common, and more Kear-Wilsdorf locks form; at sufficiently high temperature (600-800°C) thermal activation allows slip on the cube planes to take place, resulting in a subsequent loss of strength [9].

As the primary strengthening phase in nickel-based superalloys, the morphology and distribution of $\gamma'$ precipitates play an important role in determining mechanical properties [7, 9, 12-15]. The morphology of $\gamma'$ depends on particle size. It has been shown that the $\gamma'$ morphology always progresses in the following fashion with respect to size, independent of lattice misfit: spheres, cubes, arrays of cubes, and dendrites [7, 16]. The magnitude of the lattice misfit determines the size at which the morphology transitions; alloys with lower misfit are capable of sustaining larger spherical particles [7, 16].

The distribution of $\gamma'$ in the $\gamma$ matrix is heavily dependent on heat treatment parameters: solutionizing temperature and time, quench rate, and aging temperature and time. The solutionizing step entails holding the alloy, for a period of time, at a temperature either above or below the $\gamma'$ solvus temperature. The solvus temperature of $\gamma'$ in nickel-based superalloys is between 1050°C and 1200°C depending on chemical
composition [1]. Subsolvus (below solubility temperature) heat treatments produce a trimodal distribution of primary (several μm in size) secondary and tertiary γ’. Primary γ’, termed such because these precipitates were present before heat treatment, act to limit grain growth during subsolvus heat treatments to maintain a small grain size. Super-solvus (above solubility temperature) heat treatments dissolve the primary γ’ to create a bimodal distribution of secondary γ’ (hundreds of nm in size) and tertiary γ’ (tens of nm in size). The quench rate from solutionizing temperature dictates the formation of secondary and tertiary γ’ precipitates. This is particularly important for super-solvus heat treatments, as quench rate determine where these γ’ will precipitate. Under moderate cooling rates the γ’ precipitate homogeneously within the γ matrix. Under slow cooling rate the γ’ preferentially nucleate heterogeneously along grain boundaries of the γ matrix before significant undercooling allows γ’ to subsequently nucleate homogeneously in the grain interiors. This second form of nucleation can be used to engineer boundary morphology to optimize fatigue crack propagation resistance [12, 17]. Alloys are subsequently subsolvus aged to facilitate the growth of secondary and tertiary γ’ distributions.

2.2.3 Minor Phases

Carbon and boron can act as grain boundary strengtheners. In small quantities (0.03wt% B, 0.025wt%C) these elements segregate to grain boundaries leading to better boundary cohesion, the exact nature of this interaction is unknown but it has shown to limit grain boundary sliding [1, 18, 19]. Boron in solid solution also interacts
with hafnium to have beneficial effects on creep ductility and crack growth rates [19]. Higher quantities of carbon promote carbide nucleation during melt solidification. These carbides, MC, with M standing for Ti, Ta, or Hf elements, act as primary grain boundary pinning mechanisms during super-solvus heat treatment [19]. During heat treatment MC carbides can dissolve to produce more stable $M_23C_6$ and $M_6C$ carbides which are rich in Cr, Mo and W [1]. These secondary carbides preferentially form along grain boundaries either as globules, cubes, or needles. Excess amounts of boron promotes thermally induced porosity that can have negative effects on creep lives [19]. Control of the morphology and distribution of these grain boundary phases through thermal mechanical treatments imparts grain boundary strengthening [1, 2, 6, 22]. There are also several deleterious intermetallic phases rich in Cr, Mo, W, and Re. These phases ($\mu, \sigma, P, R$) are collectively termed topologically closed packed structures due to their high uniform packing, and complex crystal structure. These structures are avoided through composition selection and heat treatment parameters [3].

2.3 Elevated Temperature Creep Deformation Mechanisms

The previous section discussed how the primary dislocation mechanisms occur in both the nickel and $\gamma'$ phases. Also discussed was how the morphology of $\gamma'$ precipitates can be altered through alloy addition (misfit) and thermal treatments. The volume fraction and $\gamma'$ particle size allows for several competing mechanisms to occur at elevated temperature. In this section the various intra-granular mechanisms will be assessed according to $\gamma'$ distribution, and grain boundary character.
2.3.1 Intra-granular Mechanisms

The active intra-granular mechanisms in superalloys during elevated temperature loading vary with temperature, applied stress, and microstructural features particularly γ' size and spacing and γ-γ' dislocation interactions. Examples of various deformation mechanisms that have been observed during creep deformation of these alloys include: shearing of γ' by stacking faults that are either isolated in the γ' particle or are extended to both the matrix and the γ' particles, Figure 2.3 (a,b) [12, 14], shearing of γ' by thermally activated microtwinning, Figure 2.3 (c) [13, 20], and Orowan bypass looping of γ' particles, Figure 2.3 (d) [1, 20]. As previously discussed, stacking fault mechanisms require either interactions of γ unit dislocations to create \( a/3 <1 \bar{2} 1> + a/3 <2 \bar{1} \bar{1}> \) pairs, or can rely on the successive passing of partial dislocations \( a/6 <2 \bar{1} \bar{1}> \) on adjacent planes[13, 21]. With sufficient reordering extended faults can transform to microtwins. The activation of extended faulting and microtwinning appears to be interrelated, at lower total creep strains extended faults are observed, but at higher total creep strains microtwinning is observed [10, 13, 21]. Orowan looping occurs when γ dislocations loop around the γ' particles [14, 21].
It has been concluded that microstructures that exhibit microtwinning and extended stacking faults have lower creep rates than microstructures that promote isolated faults, and that γ’ particle looping imparts the lowest creep resistance [14, 22, 23]. Models of the different mechanisms indicate that microtwinning and extended stacking fault mechanisms can be promoted by controlling secondary γ’ size and volume fraction and tertiary γ’ volume fraction. The following trends have emerged in the literature on how each of these microstructural features promotes particular creep deformation mechanisms:
- **Secondary γ’ size**: If secondary γ’ particles are too large, Orowan looping occurs increasing the creep rate since the γ’ are no longer actively inhibiting deformation [14, 24]

- **Secondary γ’ volume fraction**: A low volume fraction of secondary γ’ combined with large particle sizes will promote looping, while low volume fraction with small particle sizes will promote isolated faulting [24]. High volume fractions promote small channel spacing between secondary γ’, this encourages extended faulting over isolated faulting or looping.

- **Tertiary γ’ volume fraction**: The absence of tertiary γ’ promotes larger channels between secondary γ’ particles and in-turn isolated faulting [13, 14].

Since microstructures that exhibit creep deformation by Orowan looping exhibit the least resistance to creep deformation, it would be advantageous to predict when a particular γ’ distribution promotes this mechanism. A simplified analysis of the strengthening behavior of a spherical particle distribution at ambient temperature can be used to confirm experimental evidence that γ’ size and spacing are the primary variables.
From classical dislocation analysis a particle produces a pinning force on a single dislocation such that the stress needed to by-pass or cut a single particle is [8]:

$$\tau_{\text{crit}} = \frac{2\Gamma \cos(\phi/2)}{b\lambda}$$  \hspace{1cm} (2-1)

Where $\Gamma$ is the binding force of the obstacle (particle), $b$ is the burger vector of the passing dislocation, $\lambda$ is the spacing between particles, and $\phi$ is the angle between the line directions of the bowing dislocation on opposite sides of the particle, Figure 2.4 (a). At ambient temperatures the binding force of a $\gamma'$ particle is a function of the amount of APB created, $\Gamma \propto 2\gamma_{\text{APB}} r$. Where $\gamma_{\text{APB}}$ is the surface free energy associated with creating an APB and $r$ is the radius of the particle. When the applied stress is greater than $\tau_{\text{crit}}$, the particle will be cut. For strong particles, large radii, the angle $\phi/2$ will be close to $0^\circ$ and the applied stress needed will to be close to $\Gamma/b\lambda$. For weak particles the angle $\phi/2$ will be closer to $0^\circ$, providing very limited pinning. When the binding force of the particle, $\Gamma \propto 2\gamma_{\text{APB}} r$, is greater than the force needed to create a semicircular shaped dislocation between pinning points $\lambda$ apart, $\Gamma_{\text{particle}} \geq Gb^2$ the dislocation will loop around the particle instead of cutting through the particle [8, 25]. This analysis assumes spherical particles, and that APB formation is the primary shearing mechanism and neglects the complication that in nickel-based superalloys the $\gamma$ unit dislocations travel in pairs to minimize the binding energy of the particle. As such the second dislocation will push the first dislocation through the particle so that it
can remove the APB created. This analysis is also complicated by the fact that creep mechanisms do not rely on APB formation, but the fact still remains that \( \gamma' \) particles will pin the dislocations by exerting a binding force on the dislocations associated with the difficulty of producing a stacking fault. When the particles are large enough that binding force is greater than that needed for looping, looping will be preferred over cutting.

Figure 2.4. (a) geometry for force balance between line tension and pinning force and \( \gamma' \) particles being sheared by pairs of (b) weakly and (c) strongly coupled unit dislocations [1].

Pairs of \( a/2<1 \bar{1}0> \) type dislocations travel in the \( \gamma \) matrix and through the \( \gamma' \) population as either weakly or strongly coupled pairs depending on volume fraction, particle size and spacing. Weakly coupled dislocations travel with significant space between them so that leading and trailing dislocations are not interacting on the same
particle, Figure 2.4 (b). In strongly coupled dislocations the fault associated between dislocations is isolated to single particles, Figure 2.4 (c).

The force analysis for particle pinning of a pair of dislocations becomes more complex because two additional forces have to be accounted for in the analysis. First, the second dislocation is slightly attracted to the first dislocation because the system wants to remove the fault in the particles. Second, the second dislocation has the same sign as the first dislocation, both positive edge dislocations for example, and as such each also experiences a repulsive force exerted by the other. (see Reed [1]). The coupling of the dislocations depends heavily on particle spacing. For under-aged materials, when the spacing between particles is much larger than the particle diameter and the volume fraction of particles is small, and dislocation pairs interact as though they are weakly coupled. In peak-aged or over-aged conditions, when the particle spacing is similar to the particle diameter and the volume fraction of particles is high, the dislocations are strongly coupled. There is an optimum particle size that provides the most strengthening when both weakly and strongly coupled dislocations cut through particles.

Several models exist for predicting the optimum γ’ distribution needed to promote one creep mechanism over another. These models demonstrate how the γ’ distribution interacts with dislocations to produce weakly or strongly coupled dislocation pairs, and how the pinning forces are exerted by the particles when isolated or extended faulting, or looping occurs [11, 13, 21, 26]. The merits of each of these models were not analyzed for this literature survey. An experimental study by
Neumeier [7] confirms the modeling efforts to show that there is an optimum $\gamma'$ size for producing a minimum creep rate, Figure 2.5. The optimum $\gamma'$ size is dependent on the alloy misfit determined by alloying additions. Alloy ReRu has a larger magnitude of misfit than Alloy Re. As such, $\gamma'$ particles transition from spherical to cubic morphology at a smaller size in Alloy ReRu as compared to Alloy Re. The cubic morphology decreases the channel spacing and promotes strongly coupled dislocation pairs. Neumeier hypothesized, but did not prove by TEM analysis, that the increase in creep rate beyond the optimum particle size is controlled by the propensity of the structure to promote bypass looping [7].

![Graph](image)

Figure 2.5. Creep rate dependence on initial particle size for an alloy modified by adding Re or Re and Ru [7].
2.3.2 Inter-granular Mechanisms

Grain boundaries are known to have dramatic effects on creep, fatigue and corrosion resistance of nickel-based superalloys. Grain boundaries provide unique sites for strain localization due to compatibility issues associated with grain misorientation and chemical segregation that occurs during solidification. Work in the 1960's and 1970's by Pratt and Whitney introduced directionally solidified and single crystal alloys for turbine airfoils. Research concluded that transverse grain boundaries act as crack initiations sites either due to grain boundary sliding or strain accumulation leading to reduced creep and fatigue life of randomly oriented polycrystalline superalloys as compared to directionally solidified (DS) columnar grain,

Figure 2.6. Further work on processing methods created single crystal (SC) airfoils which eliminated the longitudinal grains as inter-granular stress corrosion cracking sites [27, 28].
Figure 2.6 (a). Increased creep resistance of directionally solidified (DS) and single crystal (SC) MAR-M200 as compared to conventionally cast material, at 1255K and 200MPa. (b) DS and SC MAR-M200 also show increased fatigue lifetimes as compared to conventionally cast material [27].

The complex loading conditions imparted on turbine discs do not lend themselves to application of single crystal and directionally solidified alloys. This necessitates the use of polycrystalline superalloys. Research into grain boundary engineering has indicated that the susceptibility of a particular grain boundary to hot corrosion cracking, creep, and fatigue crack growth is a function of their misorientation. Special grain boundaries, known as coincident site lattice (CSL) boundaries are characterized by the inverse of the number of coincident structural sites between one grain and the other. A $\Sigma 5$ CSL, for example, indicates that every fifth
lattice site in each grain is coincident with a lattice site in the other grain. It has been shown that increasing the concentration of these special boundaries, $\Sigma 1 < \Sigma n \leq \Sigma 29$, can dramatically increase mechanical and corrosion properties [29, 30]. A thermo-mechanical process consisting of repetitive applications of deformation and recrystallization steps can be used to increase the number of $\Sigma n$ boundaries so they account for 30-70% of the total boundary length [30]. The improved resistance to intergranular corrosion attack, creep and fatigue deformation has been attributed to the unique low energy structure of the CSL boundaries. This structure leads to limited hot corrosion attacks due to limited chemical segregation (reducing particle denuded zones) and difficultly nucleating cracks induced by strain accumulation, grain boundary sliding. These theories were proposed, but the mechanism of grain boundary sliding activity was not adequately assessed.

More in-depth studies into the active deformation mechanisms in Ni-based superalloys, in creep regimes above 700°C, have indicated that grain boundary deformation/sliding might contribute to the accommodation of strain [14, 18, 31-34]. A historical search into grain boundary sliding in metal alloys indicates that two types of grain boundary sliding (GBS) are recognized by the community, Lifshitz sliding and Rachinger sliding. Lifshitz sliding arises as direct consequence of diffusional creep mechanisms (Nabarro-Herring, Coble, Harbor-Dorn) at very high temperatures and much lower stresses than those applied to nickel-based superalloys [35]. This mechanism does not contribute to strain accumulation but is geometrically necessary to accommodate grain elongation during diffusion mechanisms. Rachinger sliding occurs
when creep strain is accommodated by grains displacing relative to each other, this mechanism does not change the grain shape and must be accompanied by intra-granular dislocation motion [36, 37]. This second mechanism can contribute to creep strain and is possibly active in temperature and stress regime of interest for nickel-based superalloys applications. It has been theorized that this mechanism occurs when intra-granular dislocations enter a grain boundary and instead of the boundary acting as an impeding force (low temperature) the grain boundary absorbs the dislocations which are able to subsequently move along the boundary plane due to a combination of climb and glide [37, 38]. Studies have indicated that both hexagonal close packed [39] and face centered cubic metals [34] exhibit sliding primarily at random high angle random boundaries, which is consistent with the climb-glide model since the lattice matching associated with CSL boundaries and low angle boundaries would hinder dislocation movement along the boundary plane. These studies have also indicated that small grain sizes promote GBS, giving rise to the field of superplasticity [40, 41]. The climb-glide mechanism theory is consistent with findings that incoherent particles (requiring dislocation by-pass) and impurities located on the boundary can effectively suppress GBS, both of which are known to effect dislocation mobility [18, 34, 42].

Based on traditional literature previously discussed, the grain boundary strengthening elements and grain boundary carbides should limit grain boundary sliding in random high angle boundaries. γ’ particles along grain boundaries might inhibit GBS if they significantly alter the characteristics of the boundary. Bi-crystal experiments on a blade alloy, by Chen et al., has indicated that high angle boundaries
in these heavily strengthened, γ' volume fraction greater than 60%, still exhibit GBS. This indicates that the coherent γ' provides little resistance to GBS. When carbides are introduced into the system along the high angle grain boundary (18° misorientation) GBS was suppressed and a dramatic increase creep life was observed [18].

Other studies have indicated that γ' particles can be marginally used to hinder grain boundary sliding [12]. An alloy heat treated to produce serrated grain boundaries had substantially lower fatigue crack propagation rates then the same alloy heat treated to produce straight grain boundaries [12]. They observed that the fracture in the serrated materials occurred trans-granularly, not along grain boundaries, while the straight boundary materials fractured inter-granular. They hypothesized, but did not conclusively confirm that this trans-granular fracture was preferred since serrated boundaries would impede GBS [12]. Further work is needed to determine if an alloy originally susceptible to GBS is less susceptible when heat treated to produce serrated grain boundaries. This would conclusively determine the validity of the earlier hypothesis.

A recent study on polycrystalline disk alloys presented by Locq was exploring the behavior of the tertiary γ' distribution to optimize the creep properties of alloy NR3 at 700°C, they observed that $a/2 <1\bar{1}0>$ dislocations concentrating along grain boundaries [14]. This observation indicated that inter-granular deformation mechanisms might be active in these alloys at these temperatures. This led to work with alloy NR6 to quantify the contribution of GBS on total creep strain [31, 43].
Utilizing a 5µm hafnia grid pattern printed on the sample surface, digital image correlation (DIC) techniques were used to determine strain heterogeneities. More specifics of this technique are discussed in section 2.5 of this document. They showed, Figure 2.7, that strain becomes localized along grain boundaries, and intra-granular deformation mechanisms also leads to strain heterogeneities in the grain interiors. Based on manual measurements of discontinuities along grid lines across the boundaries, they hypothesize that the strain localization along the grain boundaries as seen by the DIC technique was indeed grain boundary sliding. A limitation to this particular technique is the relatively large spacing between grid nodes, this limits the DIC technique to only be able to discern strain localization between 5 micron node points.

![Figure 2.7. Creep test at 750°C and 525MPa to 3.5% total strain (a) SEM secondary image mode showing localized both intra-granular slip and inter-granular GBS; (b) overlay of strain map and EBSD data showing boundary characteristics (black=twin, blue=special and red=general boundaries) [43].](image-url)
Consistent with literature on single phase alloys, Locq observed that GBS was more probable at high angle grain boundaries versus special and low angle grain boundaries. They also conducted tests at various temperatures, Figure 2.8, which indicates that GBS might only be a contributing factor in a particular temperature, or stress, range. They hypothesized that the decrease in observable strain heterogeneities at elevated temperatures is related to the thermal stability of tertiary γ’ [31]. As the tertiary γ’ dissolve, the secondary γ’ grow promoting Orowan looping, looping promotes homogeneous strain distributions.

![Figure 2.8. Influence of temperature on the strain heterogeneity and GBS contribution [31].](image)

These studies and others [32, 33] have indicated that crack initiation is a function of susceptibility of grain boundary sliding. Decreasing the propensity of failure at grain boundaries is a function of:
- \( \gamma' \) distribution: create serrated grain boundaries
- Grain boundary strengthening elements: addition of carbide phases
- Grain boundary type: CSL boundaries are less susceptible to grain boundary sliding.

It is imperative that we determine exactly how these microstructural features affect GBS, but traditional testing and post mortem TEM analysis will be difficult without first developing an *in-situ* testing techniques that can determine which grains are exhibiting GBS.

2.4 More Elevated Temperature Deformation Relationships

Any or all of the mechanisms discussed could be active during elevated temperature deformation of these alloys. The above discussion on microstructural manipulation focused on optimizing creep properties. Specifically, it was noted that large grain size, increased number of CSL boundaries, and a fine distribution of \( \gamma' \) particles were beneficial for increasing creep life. The discussion disregarded how these microstructural features controlled other pertinent mechanical properties. This section will briefly discuss trends in the literature to give the reader an understanding of the difficulties involved in designing materials for gas turbine engine discs.

The bore of the disc requires high tensile strength and fatigue resistance, while the rim of the disc requires creep resistance and dwell crack growth resistance at a more elevated operating temperature than the bore [44, 45]. Experiments that have...
investigated all these properties have indicated that the best composition and microstructure for creep resistance is not always the best for strength and fatigue resistance [17, 22, 44, 46]. Boron and carbon are shown to have detrimental effects on creep rate while beneficially retard crack growth rates depending on the morphology of carbide and boride phases forming at the grain boundaries, though the amounts of carbon and boron used for this study were well beyond those seen in commercial alloys [19]. The same study concluded that additions of hafnium and tantalum have beneficial effects on both creep lives and crack growth rates, indicating that not all alloying additions will hurt one property while bolstering another [19].

Multiple studies have concluded that the tensile strength is not only dependent on γ' distribution, but also shows a Hall-Petch relationship with grain size (GS), \( \sigma \propto (GS)^{-1/2} \), indicating that this design property is optimized for smaller grain sizes with large γ' volume fraction and small γ' particle sizes [22, 47]. They determined statistical trends for yield strength, ultimate strength, creep life, and crack growth rate (da/at) listed in Table 2.2. For the particular heat treatment parameters used to develop Table 2.2, it was determined that during aging the secondary γ' coarsened at the expense of the tertiary γ', increasing the channel spacing and probability of dislocation looping [22]. Since grain size and particle size are not independently controllable, small grains are formed during subsolvus heat treatments and often lead to large γ' sizes, it is more advantageous to present data on how heat treatment parameters effect
mechanical properties. Table 2.2 shows that the creep life and fatigue crack growth rates show inverse relationships with respect to solutionizing temperature.

<table>
<thead>
<tr>
<th>Feature</th>
<th>YS</th>
<th>UTS</th>
<th>Life</th>
<th>da/dt</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain size</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0</td>
</tr>
<tr>
<td>Cooling γ’ size</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0</td>
</tr>
<tr>
<td>Aging γ’ size</td>
<td>0</td>
<td>0</td>
<td>-</td>
<td>+</td>
</tr>
</tbody>
</table>

Table 2.2. Effect of microstructural features and heat treatment parameters on yield strength (YS), ultimate strength (UTS), creep life (Life) and crack growth rate (da/dt). Negative effects (-), positive effects (+) no effect (0) [32].

The competing design requirements at the bore (high tensile strength, and fatigue resistance) and the rim (creep resistance and crack growth resistance) of turbine discs drives the desire to develop hybrid disc alloys [45]. Discs are often very large, leading to differences in cooling rate from solutionizing temperature and variations in γ’ distributions in bore and rim that are advantageous for balancing properties [44, 46, 48]. It was hypothesized that more substantial variations in γ’ and grain size could be designed into the structure by developing novel heat treatment and processing procedures. These novel processing techniques could be utilized to create discs with substantial gradients in microstructure to simultaneously optimize properties at the bore and rim [45, 49-51]. One processing method being explored includes the development of a dual-heat treatment procedure, where the bore of the disc is heat treated subsolvus while the rim is heat treated super-solvus by placing the disc in a fixture that impedes heating and cooling rate in the bore Figure 2.9.
It was discussed at the beginning of the paper that implementation of hybrid alloys is dependent on our ability to determine how local strain heterogeneities are affected by gradients in microstructure. We have also indicated that understanding the contribution of GBS to total strain accumulation also requires a method to determine local strain heterogeneities. This section will focus on traditional methods measuring local strain accumulated by GBS ($\varepsilon_{gbs}$) and follow with full-field digital image correlation (DIC) techniques for measuring $\varepsilon_{gbs}$ and other local deformations.

Empirical evidence of GBS in materials that exhibit steady state creep rate, show a power-law relationship between strain-rate and stress of $n=2$, as well as an inverse relationship with grain size (smaller grain size, more GBS) [52]. The ratio of $\varepsilon_{gbs}$ to total strain ($\varepsilon_t$) appears to be a strong function of stress, temperature, strain-rate, and microstructure [40, 53]. Since nickel-based superalloys do not exhibit steady-state
creep rate, direct measurements of strain accumulation from sliding must be conducted to prove if it is an operating mechanism. The most widely accepted ex-situ procedure for direct measurements of sliding strains entails placing marker lines on the sample surface prior to testing and measuring the offsets in the marker lines after deformation [54]. Relationships between sliding offsets, Figure 2.10, measured from marker lines either parallel or perpendicular to applied stress, and strain accumulation due to grain boundary sliding have been developed [36]:

$$\varepsilon_{GBS} = n_l \bar{u}_l = 2n_l (w / \tan \theta)_l$$

$$\varepsilon_{GBS} = n_l (u \tan \theta)_l = k^2 n_l v_i$$

Where \( n_l \) and \( n_t \) indicate the number of grains per unit length of marker line running in either the longitudinal or transverse, parallel and perpendicular, to stress axis respectively. \( k^2 \) is an experimental constant that has been shown to depend on strain assisted grain boundary migration in the interior of the sample and can range between 1 - 2 [36, 55].

![Figure 2.10](image-url) Sliding offsets \( u \), \( v \), and \( w \) when grain 2 slides with respect to grain 1 with applied stress [36].
Analysis of these measurements can be complicated by boundary migration, limited sampling size, and geometric approximations [36, 55]. A generalized method of indirectly measuring GBS in the interior of samples was proposed by Rachinger [56], but was subsequently shown by Ishiada [57] to grossly overestimate GBS contributions to total strain accumulation. This was because the Rachinger method assumed isotropic grain growth during deformation, but Ishiada proved that not to be the case [57]. Another limitation of these measurements is that intra-granular deformation is not directly measured. Therefore, techniques for measuring both $\varepsilon_{\text{gbs}}$ and intra-granular contributions to total strain accumulation at elevated temperatures would be advantageous for understanding local strain accommodation across gradients in microstructure.

Image correlation methods for determining full-field, heterogeneous, deformation have been utilized since the 1950’s [58]. With the advent of digital imaging these methods have become common-place for mapping 2-D and 3-D large scale deformation heterogeneities under both static and very high strain rate loading conditions [58]. Investigators are beginning to couple the high magnification capabilities of SEM with digital image correlation (DIC) techniques to investigate local strain heterogeneities at the grain scale in polycrystalline materials. DIC works by applying mathematical algorithms to match points in one image, before deformation, with points in a second image, taken during deformation, to determine local
displacement and strain fields [58]. Investigators have determined that there are specific requirements for patterning samples for ambient temperature [59] and at elevated temperature [31, 60], including vacuum capable and contrast. There is also emerging work showing that SEM images contain spatial and drift distortions not seen in optical images which need to be taken into consideration before quantitative assessment of small strains can be conducted [61, 62]. Since these distortions vary with time, they are difficult to remove for _ex-situ_ experiments since there is no set time between images.

Studies of full-field deformation behavior in nickel-based superalloys have employed the technique of _ex-situ_ loading, interrupting tests at a predetermined amount of strain and analyzing the changes in a pattern imaged using an SEM, Figure 2.7 [31, 32, 43]. The grid pattern used in this study (5μm pitch, 0.5μm line width) shows local strain accumulation at each node, and discontinuities in the grid lines were used to quantify GBS using traditional methods. The relatively large distances between node points of the pattern limit the resolution of this DIC method. DIC was not able discern localized deformation around boundaries and slip lines, but was able to show a trend that certain boundary configurations can lead to strain localization.

2.6 Conclusions

Deformation in nickel-based superalloys is a complicated heterogeneous process often involving multiple intra-granular mechanisms that evolve with ever changing microstructures. Understanding how these mechanisms operate as a function
microstructural size, temperature and stress is a requirement for designing and implementing more efficient alloys. Experimental and theoretical work has indicated that these intra-granular mechanisms are most strongly dependent on $\gamma'$ distribution. Chemical composition and processing history have dramatic effects on the $\gamma'$ distributions, and knowing how these dictate the $\gamma'$ distributions and $\gamma'$ thermal stability will allow further development of these alloys for higher temperature applications.

Crack nucleation at grain boundaries leads to creep and fatigue failure. Altering the boundary network can dramatically decrease crack nucleation rate. This has been done with two different techniques: increasing CSL boundaries, or increasing boundary tortuosity. Initial work has indicated that in the 700-800°C range inter-granular creep mechanisms are active in these alloys and might be dependent on $\gamma'$ distribution, grain morphology, and temperature. A mechanistic understanding of this type of deformation is only beginning to form. Future work is needed utilizing in-situ strain analysis and post-mortem TEM analysis to understand how these inter-granular mechanisms vary with grain size, grain morphology, grain boundary segregation, and $\gamma'$ distribution evolution with time during testing.

There is a drive toward creating hybrid nickel-based superalloys that can have microstructures optimized at the bore and rim of the disc to accommodate competing design requirements. These hybrid alloys are one reason for the necessity of developing an in-situ testing technique. It would be advantageous to understand how local strains are accumulating in the gradient between the bore and the rim of the disc to determine if this region has become the vulnerable to failure.
The few *ex-situ* experiments that have tried to quantify the contribution GBS to total strain accumulation are limited by the resolution of their grid pattern, and have not taken into consideration distortions that can arise in SEM images. These limitations are another reason for developing an *in-situ* straining technique that has the resolution to more readily assess GBS contributions through the use of a random speckle pattern which has higher spatial resolution than a regular grid patterns previously used.

The development of this *in-situ* technique is a technical challenge which will require over-coming several issues: specimen heating, speckle pattern development, imaging samples in SEM at elevated temperatures, running tests to limit creep deformation during image acquisition, and mitigating the distortions in the imaging system. Development of this testing system is not only useful for nickel-based superalloys, but for any investigation of strain heterogeneities in a material system of interest.
Chapter 3: Experimental Techniques

3.1 Introduction

The development of an in-situ experimental technique relies on the direct correlation of deformation fields with the microstructural features of interest. For this study, Correlated Solutions VIC-2D software was used to perform digital image correlation (DIC) analysis to determine local deformation fields on samples tested at elevated temperature under constant load conditions. This chapter will discuss the material system used in this study and the methods for conducting microstructural analysis prior to deformation testing. Additionally, the steps for conducting DIC experiments are addressed, including development of a suitable high temperature speckle pattern, and the capabilities and limitations of conducting in-situ creep tests in an SEM using the Fullam sub-stage. Optimization of the random speckle pattern and VIC-2D fitting parameters are discussed in Chapter 4. The specifics of scanning transmission electron microscopy analysis of deformation mechanisms and examination of grain boundary character through 3D reconstruction are discussed in Chapters 5 and 6.
3.2 Material Development

The material used for this study is the γ'-strengthened nickel-base superalloy René-104 (15wt% Cr, 18.2% Co, 3.8% Mo, 1.9% W, 1.4% Nb, 3.5% Al, 3.5% Ti, 2.7% Ta, 0.03% C, 0.03% B, 0.05% Zr, balance Ni) [1]. Samples were subjected to one of two different aging treatments by solutionizing above γ’ solvus temperature followed by aging to produce two distinct microstructures with similar grain sizes. One microstructure consisted of relatively planar grain boundaries and a bimodal γ’ distribution while the other had macroscopically serrated grain boundaries with a quad-modal γ’ distribution. The heat treatment to create planar grain boundaries was the standard disc alloy heat treatment, solutionizing at super-solvus temperature followed by a fast quench (1-3°C/sec) to room temperature to promote heterogeneous nucleation of γ’ in the grain interiors followed by an extended subsolvus age. The serrated microstructure was created by promoting homogeneous nucleation at the grain boundaries by slow quenching (1-5°C/min) from super-solvus temperature to an intermediate temperature hold prior followed by a fast quench to room temperature and subsequent aging [12, 63].

3.3 Microstructural Analysis

As previously discussed, the primary features of interest in nickel-based superalloys are γ’ and grain size distributions. Since these features exist on an order of magnitude difference in length scale, they require different analysis techniques.
3.3.1 Analysis of γ′ Precipitate Distribution

The volume fraction and size distribution of the γ′ precipitates were characterized on representative samples for each heat treatment examined, using the sample preparation, imaging techniques, and segmentation algorithms developed by E. Payton et al. [64]. Samples were first mechanically polished for SEM imaging, and a grid of hardness indents were created on the sample surface. EBSD analysis was conducted over the area containing the hardness indents to identify grains of particular orientation. Since there is a cube-cube relationship between the nickel matrix and the γ′ precipitates, and deformation in the structure occurs on {111} planes, it is important to conduct analysis of γ′ distributions in a grain oriented such that a {111} plane normal is parallel to the sample surface normal. After EBSD analysis, samples were then γ′ selectively etched in a 61:36.6:2.4 solution of 85% concentrated Lactic acid, 69% concentrated Nitric acid, and 49% Hydrofluoric acid [64]. The solution was swabbed onto the sample surface for a period of 5-15 seconds to selectively etch the γ′ precipitates from the sample. The hardness indents were used to locate previously determined grains of {111} orientation.

Several imaging techniques for automated characterization of γ′ distributions are discussed in E. Payton et.al [64]. Medium voltage (15kV) small spot size (50μm) backscatter imaging in ultra high resolution mode in a FEI Sirion XL-30 FEG-SEM provided the best contrast between γ′ precipitates and the γ matrix without producing loss of spatial resolution or “haloing” around precipitates as seen in secondary electron
imaging mode. Multiple images were acquired, with each image containing approximately 100 precipitates. The distribution of the secondary particle size was calculated by combining measurements over multiple images so that over 500 particles were analyzed.

Next, using Fovea Pro Plug-in for Adobe Photoshop (produced by Reindeer Graphics, Asheville, NC) a series of segmentation parameters were applied to transform the SEM images into fully segmented data sets for analysis by removing signal noise and channeling contrast. The specific parameters for each segmentation step are listed and discussed in great detail by E. Payton et.al [64]. An example of before and after segmentation images are presented in Figure 3.1. The semi-automated technique was applicable for analyzing the distribution secondary γ’ particles, but proved impossible for the larger particle distributions due to inconsistent contrast issues.

Figure 3.1. An example of segmentation done on an etched sample for γ’ analysis
The serrated microstructure also had a distribution of larger particles, both dendritic shaped particles in the grain interiors and globule shaped particles on the grain boundaries. Semi-automated segmentation was not possible due to contrast variations, so segmentation was conducted by hand on over 1000 particles. An example of a region segmented for large particle measurements is presented in Figure 3.2.

![Segmentation of particles on the grain boundaries, light grey, and dendritic particles in the grain interiors, dark grey.](image)

Figure 3.2. Segmentation of particles on the grain boundaries, light grey, and dendritic particles in the grain interiors, dark grey.

Fovea Pro Plug-in has several methods of analyzing the segmented data. The average precipitate size can be calculated as an equivalent circle with the same number of pixels: for small secondary and tertiary $\gamma'$ this is a valid assumption because they are spherical and cuboidal in nature. The software can also calculate particle aspect ratio...
which is useful to characterize the irregular and sometimes dendritic nature of the larger secondary \( \gamma' \) particles. Ning Zhou conducted analysis of the inter-particle spacing for the cuboidal secondary \( \gamma' \) distribution, his technique and results are presented in this document for completeness [65]. A Delaunay triangulation algorithm was used on segmented images to determine \( \gamma \) channel width between \( \gamma' \) particles by first determining the center of mass for each particle and connecting the center of masses. Thus, individual channel widths are defined as the segments between particles along the connecting lines, excluding the portions within the particles [66]. Model predictions are discussed in tandem with the TEM analysis in Chapter 6. The interaction between \( \gamma' \) size and spacing with dislocation mechanisms were discussed in the Chapter 2.

3.3.2 Analysis of Grain Size and Orientation Distributions

The primary goal of full-field strain mapping is to develop and understanding of how local microstructure influences strain distribution. The local grain structure and orientation were characterized using EDAX TSL electron backscatter diffraction (EBSD) equipment using an FEI SEM with a field emission source. An area that was 1 mm\(^2\) was characterized using a 1 \( \mu \)m step size. For an area that size there are several distortions that can occur due to sample alignment and beam rastering. To conduct EBSD analysis with reliable spatial and orientation measurements the first thing that must be considered is sample alignment. A leveling holder was used to remove sample tilt at zero stage tilt before validating stage tilt calibration with a silicon calibration
specimen. The orientation calibration was checked by comparing orientation of the silicon sample with the expected orientation for the sample tilted at 70°. Spatial calibration was checked by comparing images of a grid pattern at zero and 70° tilt. It was observed that depending on the microscope; zero scan rotation for a batch scan may not be exactly zero degrees, this was fixed by conducting scans at various scan rotations until no rotation was observable when compared with backscatter images. Distortions due to beam deflection during rastering were limited by conducting scans at high enough magnification so that spatial distortions were less than the scan step size. For most SEM's this magnification is between 500-1000 times magnification. Once EBSD patterns were collected over areas to insure measurement of more than 500 grains, in most cases over 2000 grains were analyzed to determine distributions. Grain size distributions were calculated for a linear intercept method in accordance to ASTM standards E112, E1382. Three dimensional equivalent diameters were calculated assuming a lognormal distribution of tetrakaidecahedron in accordance with ASTM standard E112

3.4 Speckle Pattern Technique

Electron beam (e-beam) lithography was used to apply the different speckle patterns used in this study. The technique used a focused scanning e-beam to locally expose photo-resist on the sample surface, which resulted in a highly repeatable point-by-point constructed pattern [67]. Once exposed, the resist is developed to remove the exposed resist and a metal or dielectric is deposited onto the sample surface. After
deposition the developed resist is removed to reveal the pattern. The following section provides details on how to deposit patterns using standard clean room procedures.

Patterns were developed using the L-Edit Layout Editor by Tanner EDA. This software was specifically designed for silicon wafer integrated circuit design and therefore does not readily produce random speckle patterns. The software works by creating “cells” of information that are nested (instances) in larger “cells” to create a complex pattern. To create a random pattern, first separate cells containing each type of speckle are created, for example diamonds with edge lengths between 200-500 nm. Then instances of these cells are placed by hand in a random array in a cell that is 25 μm for example. This process is repeated to create several 25 μm cells with a random array of speckles. These 25 μm cells are then tessellated to create 50 μm cells etcetera until a random pattern of the desired size was created, see Figure 3.3 for a graphical representation of this method.
Figure 3.3. The method used to create a large random pattern from smaller random patterns using the L-Edit software. Each box is a cell, and each larger cell consists of many instances of the smaller cells.

Since the larger random pattern is a tessellated version of smaller random patterns it is not completely random over the whole region. To make sure alignment with EBSD was accurate for site specific extraction of TEM foils, and serial section samples, a unique pattern of alpha numeric markers was added. A regular repeating pattern of grid lines was also added to the pattern in a similar fashion. These grid lines were used to measure grain boundary sliding. A subset of a larger 1 mm\(^2\) pattern made from hafnium oxide is presented in Figure 3.4.
Patterns were applied to the sample surface using standard clean room procedures at The Ohio State University Nano-Technology Cleanroom facility. First, 950k A4 PMMA resist was spin coated onto the sample surface. 950k A4 PMMA resist was deposited onto the sample surface (approximately 75% coverage) using a Luer lock syringe, care was taken to remove air bubbles before spinning the sample to 1500 rpm at 200 rpm/s for a total processing time of 90 seconds. The sample was baked at 200°C before spin coating to remove residual water and baked again after coating to cure the 950k A4 PMMA. Once coated, the sample was exposed at 50 keV with a 900 μC/cm² dose using a Vistec EBPG 5000 e-beam lithography system. The resist was developed in a 1:3 mixture of methyl isobutyl ketone and isopropyl alcohol. The location of the speckle pattern on the sample surface was determined by microhardness indents placed on the surface before microstructural analysis. A Denton 502A evaporator was utilized to deposit a 100 nm thick layer of hafnium oxide onto the surface, the density of hafnium oxide is 9.68 g/cm² and sapphire z-contrast is 24.45.
Once coated, the developed resist was lifted off the sample using heated (above 80°C) n-methyl 2-pyrrolidinone, leaving speckles on the sample surface.

3.5 Fullam Tensile Stage: Capabilities and Limitations

An Ernest Fullam (MTI Instruments Inc) SEM tensile sub-stage, belonging to The Air Force Research Laboratory, Materials & Manufacturing Directorate, at Wright Patterson AFB was used for these experiments. This screw driven machine can be controlled either in load or displacement control and has a maximum load capacity of 4448 N (1000lbf) and a maximum travel distance of 10 mm. The tensile sub-stage also has a 1200°C rated resistance heater that can be aligned in contact with the backside of the specimens using six alignment screws.

3.5.1 Specimen Geometry

Specimen geometry was dictated by two factors: the load capabilities of the Fullam tensile stage, and the e-beam lithography equipment. It was desired to have specimen yield near 50% of maximum of the load cell capacity. Assuming yield strength near 1 GPa, yield should occur at approximately 2520 N. Since the e-beam lithography system is originally purposed for silicon wafer patterning, the specimen thickness should be no more than 1.08 mm. Specimen geometry used is presented in Figure 3.5.
Prior to elevated temperature testing, samples were prepared for SEM imaging using conventional polishing techniques on an Allied Multiprep system, culminating with a final vibratory polish using 0.02 μm colloidal silica. After polishing, four microhardness indents were placed near the center of the gage section to define the four corners of a 1 mm² area. These hardness indents were used to define the location where the speckle pattern was subsequently deposited on the surface, and were also useful in coarse registration of microstructural images and electron backscatter diffraction (EBSD) data with the local displacement maps. Next, the samples were characterized via EBSD mapping and a speckle pattern was deposited using parameters described.
below. Once patterned, two type K thermocouples were spot welded onto the surface along the gage length on opposite sides of the patterned area, as shown in Figure 3.6.

![Schematic diagram](image)

Figure 3.6. Schematic diagram showing the locations of the speckled area, two type K thermocouples located on the sample surface, and the size and placement of the resistance specimen heater with respect to the sample. The loading direction (F) is along the horizontal axis.

3.5.2 Experimental Limitations

*High Temperature Imaging*

In order to determine the best imaging conditions for high temperature DIC testing a series of incremental heating experiments were performed at 100°C intervals up to a maximum temperature of 750°C. One of the findings from these experiments was that BSE imaging was only possible to about 300°C, above which thermal radiation significantly degraded the image; this observation is consistent with other studies [68, 69]. By comparison, secondary electron images showed only minimal degradation in quality up to 750°C. For prolonged exposure to temperatures at and above 700°C, white lines appeared in the SE images that degraded the quality of the
image. These imaging artifacts were removed by placing an aluminum foil heat shield between the SE detector and the specimen heater. This heat shield limited radiative heat transfer to the secondary electron detector, enabling the use of the secondary electron imaging for these elevated temperature experiments.

Images during creep testing were acquired at regular time intervals. The load was reduced to 80% of testing condition (880MPa for 1100MPa testing condition). These unloading intervals were dependent on expected creep rate as extrapolated from traditional testing, i.e. shorter intervals for faster creep rates and longer intervals for slower creep rates. Intervals were determined based on the desire for enough strain accumulation to occur during each interval to provide unique strain data. Time interval was determined by estimating the required length of time needed such that the average strain would increase by more than three times the noise limit. For these experiment, a 20% drop in applied load arrested creep deformation during imaging; for slow strain rate experiments this unloading might not be necessary and for higher strain rates the unloading during imaging may need to be larger and should be determined for each material type. Assuming uniform deformation, the maximum creep rate above which a stress drop would be required can be calculated as a function of imaging parameters and noise in the calculated strain data, discussed below. For this experimental set-up that would be approximately 4e-5 /sec, but since deformation is not uniform and can vary dramatically it would be expected that this is not a conservative estimate. Therefore, even though experimental strain rates were less than this maximum strain rate a 20% stress drop was still conducted for all experiments.
Specimen Thermal Stability

During the initial assessment of the test frame performance, loading and unloading of a sample with two welded thermocouples showed that displacement rates of 0.01 and 0.005 mm/sec produced substantial and repeatable fluctuations in the sample temperature. The source of these fluctuations was attributed to changes in the total contact surface area of the backside of the specimen and the resistance heater during testing. When the displacement rate was reduced to 0.003 mm/sec, these thermal fluctuations were eliminated, presumably because the response time for the thermal controller and substrate heater is commensurate with the variation in contact that may occur during the test at lower strain rate. Since a goal of the present investigation is to study time-dependent deformation processes, testing at slower strain rates is not a severe limitation.

The two thermocouples on the sample surface allowed for the measurement of a possible thermal gradient along the gage length due to improper alignment of the resistance heater and the specimen surface. The temperature acquisition equipment was calibrated to within ±2°C. Therefore, the two thermocouples on the sample surface should read the same value within ±4°C if the specimen heater is properly aligned relative to the sample surface. A sample was heated to 700°C prior to tensile testing to validate that the heater was evenly contacting the specimen surface. In an iterative fashion the specimen heater was tilted until the two thermocouples read within 7°C of each other.
Chapter 4: Material Characterization

4.1 Gamma Prime Distributions

4.1.1 Standard Microstructure

The γ' distribution of the standard microstructure had a bi-modal distribution of small spherical tertiary γ' particles and cuboidal secondary γ' particles. Since deformation occurs on {111} planes, particle sizes were analyzed on a {111} plane and cuboidal particles look spherical or triangular in cross-section. For simplistic reasons, the particle diameter was calculated from area measurements, assuming spherical shape. Figure 4.1a, shows the particle distribution on the {111} planes, and the mean and standard deviation of the distribution presented in Table 4.1.

The Delaunay analysis concluded that like the particle size distribution, the distribution of the inter-particle spacing was also right-skewed distribution, Figure 4.1b. The mode of this distribution is presented in Table 4.1. It is intuitively expected, and confirmed by Phase Field simulations, that the mode of the secondary γ' spacing dictates the ease of dislocation motion [66].
Figure 4.1. Secondary $\gamma'$ particle (a) size and (b) spacing distributions for the standard microstructure.

<table>
<thead>
<tr>
<th></th>
<th>Units</th>
<th>Standard</th>
<th>Serrated</th>
</tr>
</thead>
<tbody>
<tr>
<td>secondary $\gamma'$ size</td>
<td>nm</td>
<td>114 ± 82</td>
<td>69 ± 50</td>
</tr>
<tr>
<td>secondary $\gamma'$ spacing</td>
<td>nm</td>
<td>38 ± 49</td>
<td>35 ± 36</td>
</tr>
<tr>
<td>intergranular $\gamma'$ size</td>
<td>$\mu$m$^2$</td>
<td>~</td>
<td>3.2 ± 2</td>
</tr>
<tr>
<td>intra-granular dendritic $\gamma'$ size</td>
<td>$\mu$m$^2$</td>
<td>~</td>
<td>1.0 ± 1.2</td>
</tr>
</tbody>
</table>

Table 4.1. Mode and standard deviation in $\gamma'$ particle sizes and spacing for the standard and serrated microstructures.

4.1.2 Serrated Microstructure

The $\gamma'$ distribution in the serrated microstructure was quad-modal in nature. Presented in increasing size: spherical tertiary, cuboidal secondary, and dendritic...
secondary were all present in the grain interiors, while globular secondary $\gamma'$ were present on the grain boundaries. An image of this distribution is presented in

Figure 4.2. The large globular $\gamma'$ particles on the grain boundaries cause the formation of a denuded zone that on the smallest scale is free of cuboidal secondary $\gamma'$ and on a larger scale is free of dendritic secondary $\gamma'$ particles. The cuboidal $\gamma'$ particles were selected through automated selection methods, but the larger particles were selected by hand because of contrast issues. The cuboidal secondary $\gamma'$ size and spacing distributions are presented in Figure 4.3, and mode and standard deviations are presented in Table 4.1. It is interesting that even through the cuboidal secondary $\gamma'$ particles in the serrated microstructure are smaller, their spatial distribution is similar to the standard microstructure.

Figure 4.2. Example of the $\gamma'$ particle distribution in the serrated microstructure, the tertiary particles are not visible at this magnification.
Figure 4.3. Secondary $\gamma'$ particle (a) size and (b) spacing distributions for the serrated microstructure.

The distributions of enclosed area for the dendritic secondary $\gamma'$, and globular grain boundary secondary $\gamma'$ were analyzed over 857 and 133 particles respectively, and are presented in Figure 4.4a and Figure 4.5a. Each distribution showed significant deviations, mostly due to the fact that particles were analyzed from a variety of grain orientations. The mode of each distribution is presented in Table 4.1. The dendritic secondary $\gamma'$ particles observed were consistent in shape as expected during the growth of large $\gamma'$ particles as described by R. Ricks *et al.* [16]. The aspect ratio of the globular grain boundary $\gamma'$ was analyzed, the distribution is shown in Figure 4.4b. The mode of the distribution was an aspect ratio of about 1.6, indicating an average ellipse major
axis of 0.8 ± 0.63 μm. In this microstructure there were also two regions of denuded zones, a large region between the globular and dendritic γ’ and a smaller region free of spherical secondary γ’ around each globular or dendritic γ’, the average size and standard deviation of each of these denuded zone are also presented in Table 4.1.

Figure 4.4. Globular grain boundary γ’ particle (a) size and (b) aspect ratio distributions for the serrated microstructure.
4.2 Grain Size Distributions

Grain size distributions were analyzed on samples prepared for full-field strain mapping. Regions of 1 mm$^2$ were analyzed by EBSD analysis, and specifics are presented in section 3.3.2. Representative microstructures for the standard and serrated materials are presented in Figure 4.7. Texture analysis of these two materials, indicates that in both cases there is effectively random texture. Grain size distributions calculated in accordance with ASTM standards are presented in Figure 4.6, and average and standard deviation for the standard and serrated microstructures were 20 ± 12.5 μm and 18 ± 18.5 μm respectively. Both distributions were similar except for the apparent “bi-modal” component of the serrated grain boundary distribution. The “bi-modal” component was centered around 2.2μm, which is consistent with the distribution of the
globule shaped \(\gamma'\) located on the grain boundaries. Therefore, the “bi-modal” component of the grain size distribution as calculated from the EBSD analysis is an artifact associated with the fact that the scans could not discern \(\gamma'\) from the \(\gamma\) phase. Figure 4.8 is a magnified region of the serrated microstructure, and two “small grains” are pointed out along on the larger grain boundaries. It is hypothesized that these misinterpretations are responsible for the “bi-modal” distribution peak in Figure 4.6.

![Graph showing grain size distributions](image)

**Figure 4.6.** Grain size distributions for the standard and serrated microstructures as analyzed over more than 2000 grains for each material type.

- Standard: \(20 \pm 12.5 \mu m\)
- Serrated: \(18 \pm 18.5 \mu m\)
Figure 4.7. Inverse Pole Figure (IPF) maps showing representative microstructures for the standard (top) and the serrated (bottom) heat treatments. Twin boundaries are outlined in black.
It is also obvious from Figure 4.7 that both the standard and serrated microstructures contain a significant fraction of annealing twins, and Table 4.2 enumerates the percentage of grain boundaries that are special in nature (i.e. $\Sigma^3$ annealing twins and other $\Sigma n$ $3<n<31$ Coincident-Site Lattice boundaries).

<table>
<thead>
<tr>
<th>% of boundaries that are:</th>
<th>Standard</th>
<th>Serrated</th>
</tr>
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<tbody>
<tr>
<td>HAGB</td>
<td>53</td>
<td>48</td>
</tr>
<tr>
<td>$\Sigma^3$</td>
<td>45</td>
<td>44</td>
</tr>
<tr>
<td>$\Sigma n$ $3&lt;n&lt;31$</td>
<td>2</td>
<td>7</td>
</tr>
</tbody>
</table>

Table 4.2. Fraction of grain boundaries that are high angle grain boundaries (HAGB), $\Sigma^3$ annealing twins, or other special boundaries ($\Sigma n$ $3<n<31$).

Figure 4.8. Magnified region of the IPF map for the serrated microstructure showing "small grains" along a grain boundary that are likely misinterpretation of the large $\gamma'$ particles. Twin boundaries are highlighted in black.
Chapter 5: Optimization of strain resolution from VIC-2D

5.1 Introduction

Characterization of localized strain accommodation as a function of microstructural features is a budding field of research. Digital image correlation (DIC) techniques provide full-field deformation information, and have been used to examine localized deformation mechanism in various materials and loading conditions. Some examples include; monotonic loading of bulk and thin film materials [70, 71], fatigue damage accumulation and fatigue crack growth [72, 73], and stress induced transformations in shape memory alloys [74]. These few examples demonstrate the flexibility of the DIC for studying localized deformation behavior in heterogeneous materials.

Recently, scanning electron microscopy (SEM) DIC has allowed for higher resolution studies of strain localization on the grain scale of engineering materials with conventional grain sizes, 50-500 μm [61, 62, 72, 75-77]. In order to use SEM imaging systems for DIC it is necessary to establish a vacuum compatible, electronically conductive pattern on the sample surface. Several techniques are available for applying such patterns including chemical vapor deposition, sputter deposition through transmission electron sample grids, and electron beam lithography [59]. Electron beam
(e-beam) lithography provides the most control over the patterns created, and allows for optimization of pattern parameters to enhance strain resolution. E-beam lithography permits the experimentalist to apply both a random pattern of speckles and an organized set of linear grid markers for grain boundary sliding measurements [78]. Random speckles for DIC can be created with a variety of geometries; square, diamond, and round are some examples. For elevated temperature experiments it is advantageous to know if strain accumulation measured from DIC is a product of grain boundary sliding. Grain boundary sliding has been conventionally measured by discrete steps in linear markers [36]. To measure these discrete steps in the linear markers requires that there not be significant overlap between them and the random speckle pattern, thus providing an experimental design in which limiting the random speckle density for DIC would be preferred. Therefore, it is advantageous to understand how speckle shape and pattern density can have detrimental effects on the strain resolution of the DIC strain calculations.

This chapter will focus on how different parameters effect localized strain measurements from SEM DIC experiments. For these experiments Correlated Solutions VIC-2D software was used for strain measurements. The VIC-2D DIC algorithm partitions the image into subsets and correlates the motion of rigidly adhered speckles in images taken during deformation with their original position in an image taken prior to deformation. Local strain is measured by iteratively applying a deformation tensor, and minimizing the error associated with the unique grayscale, to subsets in the deformed image relative to a subset in the reference image. By stepping
the subsets across the imaged region by a particular overlap, the deformation field on the surface of the specimen can be assessed [58]. As a result of this algorithm, for particular imaging conditions, the stain resolution of DIC analysis is a function of the speckle pattern, subset size and step size used during the DIC analysis. The effects of each of these parameters on strain measurement resolution are addressed in this Chapter. Noise in the full field strain measurements has both intensity and spatial components, and decreasing the noise will increase the strain resolution of the measurement. As such, these two terms “signal noise” and “strain resolution” are used interchangeably through this chapter. Each component of the signal noise will be addressed; the intensity component dictates how much strain above/below its neighbors a data point need to be to be considered a localization site, and the spatial component dictates how localized a strain measurement can be (important for characterizing very localized deformation at slip traces and grain boundaries).

5.2 Experimental

Random speckle patterns were applied to samples of a wrought nickel-based superalloy using e-beam lithography techniques. Samples were polished for metallographic examination prior to patterning using standard polishing procedures on an Allied Multi-prep system, finishing with a 0.2 µm colloidal silica vibratory polish. For microstructural analysis methods used for metallographic examination please see Chapter 3.3 or J.L. Walley et al. [78].
Correlated Solutions Inc. VIC-2D digital image correlation software was used to analyze local deformation fields. Digital image correlation for deformation measurements using random speckles is best when the speckles are 3 pixels in diameter [58]. Lagrangian strains were calculated from the displacement measurements using a size 15 decay filter. For small strains, engineering strain can be calculated from Lagrangian strains by the following equation: 
\[ \varepsilon_{e_{ng}} = \sqrt{1 + 2E_{11}} - 1 \] [58]. Strain resolution is a function of initial calculation length scales, which are defined by the DIC parameters subset and the step size. Two sets of experiments were conducted to assess the how patterning and DIC parameters produced noise in the strain measurements and thus limited strain resolution. To assess optimal DIC parameters for the image acquisition settings, DIC parameters were systematically varied from 20 pixels to 100 pixels for subset size, and 1-10 pixels for step size. This analysis was conducted on a sample under zero load condition, and sets of five or more images taken over the same region. Since the strain accumulated from one image to the next should be zero, this technique allows us to assess the noise in the strain measurements due to image acquisition and how DIC parameters can be optimized to minimize this noise through averaging. When there is no strain applied it is also possible to determine how the speckle pattern density varies the DIC measurements. Non-optimized and optimized DIC parameter sets were applied to strain data collected on a sample deformed at 700°C at constant load and comparisons were made to show how parameter optimization effects data interpretation.
The material examined for this study has an average grain size of 32± 4 μm, and it was desired to make the subset sizes vary from one third to half the average linear grain size. Image resolution was set at 2048x1768 pixels and contrast and brightness of the images were adjusted to utilize the entire grayscale. For an image to incorporate 15 grains across the longest dimension the image frame, a 6.8 pixel/μm magnification is required. Based on these resolution requirements, and the minimum speckle size criteria listed in Chapter 3, the minimum speckle size is 0.2 μm. Random speckle patterns of variable densities were generated using square speckles with edge lengths between 0.2 to 0.7 μm. Table 5.1 enumerates the different pattern densities developed for this study. Strain resolution as a function of speckle orientation was also assessed by comparing patterns of the same speckle density with both square and diamond shaped speckles. In one set of experiments the specimen was aligned so that edges of square speckles were aligned parallel to the rastering directions of the SEM images, while in another set of experiments the specimen was aligned so that square edges were oriented 30° with respect to the rastering direction.

<table>
<thead>
<tr>
<th>Pattern</th>
<th>Area Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>12</td>
</tr>
<tr>
<td>1</td>
<td>23</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>35</td>
</tr>
<tr>
<td>4</td>
<td>37</td>
</tr>
<tr>
<td>5</td>
<td>58</td>
</tr>
</tbody>
</table>

Table 5.1. Area fraction of speckle coverage for each pattern examined
Previous work has indicated that DIC from SEM images created by integrating the image intensity over eight images creates lower intensity strain noise [62]. Therefore, all the images were acquired using 1 microsecond dwell time with eight images integration using the FEI integration option for a total image acquisition time of 28.8 seconds. The SEM used for this experiment was an FEI Quanta SEM with a field emission source, at 15kV. A secondary electron image of the speckle patterns, densities 1 through 4, is shown in Figure 5.1.

![Secondary electron image of four different speckle pattern densities](image.png)

Figure 5.1. Secondary electron image of four different speckle pattern densities, top-left corner to bottom-right corner are 35%, 37%, 23%, and 12% area fraction.
5.3 Results

5.3.1 Speckle Density

The area fraction of coverage for each of the speckle patterns developed is listed in Table 5.1. Independent of DIC parameters there was consistent substantial reduction of signal noise intensity between the pattern with 12% area fraction coverage and 23% area fraction coverage. Above this threshold area fraction there was only marginal reduction of signal noise with increased pattern density. Figure 5.2 shows these trends in noise intensity variability for 40 pixel subset size and a 3 pixel step size. For all speckle densities with square speckles the $e_{yy}$ signal noise is approximately double the $e_{xx}$ noise. Images acquired, at zero load (zero average strain) over the course of 30 minutes showed no systematic variation in the signal noise intensity. Since a traditional tensile experiment could be conducted over the course of 30 minutes, it would be expected that time dependent image distortions should not be a contributing factor to the noise seen in the strain data.
Previous work has indicated that the increased noise intensity in the y-direction is caused by the interaction between the discrete rastering characteristic in the y-direction of SEM system and the top and bottom edges of square speckles in a regular array [76]. Since a similar relationship was observed for random square pattern densities, a series of images were acquired by rotating the patterns relative to the rastering direction by 30 degrees. A comparison of the strain noise for the same pattern density with square and diamond speckles (pattern 2, 30% area fraction) is presented in Figure 5.3. It was observed that rotating square speckles so that the cube edge was not to align with the SEM beam rastering decreased the signal noise in the y-direction.
5.3.2 VIC-2D Parameters

The VIC-2D DIC parameters had dramatic effect on the intensity and spatial distribution of the noise in the strain data. Increasing the subset or step sizes effectively decreased the intensity of the strain noise, independent of speckle pattern density. This is a trade-off, as increasing the subset and step size also decreased spatial resolution. It has previously been shown that VIC-2D calculation of strain introduces a non-random spatial distribution of elliptical spots with the long axis perpendicular to the strain measurement [78]. Increasing the subset or step size parameters increases the size and decreases the periodicity of these elliptical spots, while also decreasing their relative intensity, and effectively smoothing out the data. The spatial noise as a function of
subset and step size for the 35% area fraction pattern is presented in Figure 5.4, with a table of noise intensities presented in Table 5.2. Figure 5.4 indicates that larger subsets and step sizes increase intensity noise near the outer edges of the correlated region.

Comparison of the spatial distributions and the intensity variations in the signal noise indicates that increasing the subset size to reduce signal noise intensity produces less spatial resolution loss than increasing the step size. Therefore, the optimum method for minimizing signal noise while maintaining the best spatial resolution would be to minimize step size while using a moderate subset size. This optimization method is slightly subjective depending on the requirements of the measurement, (measuring elastic strains requires low intensity noise, while plastic strain localization at grain boundaries requires high spatial resolution). For the patterns and image resolution used for these experiments, the optimum parameters for characterizing plastic strain accumulation at grain boundaries were subjectively determined, to minimize the signal noise while maintaining good spatial resolution, to be 60 pixel subset size and 3 pixel step size, this puts the strain resolution limit for this data set around 400 micro-strain (as seen in Table 5.2).

Speckle pattern density can limit the smallest possible subset size that could be applied to the data sets. When the subset size was on the same order of magnitude as the spacing between speckles the DIC software was unable to make correlation measurements between the reference image and subsequent images. This issue was observed with the 12% area fraction pattern, using subset sizes less than 30 pixels.
Figure 5.4. Signal noise decreases with step size, with a decrease in spatial resolution.

<table>
<thead>
<tr>
<th>subset (pixels)</th>
<th>step (pixels)</th>
<th>3σ noise (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>3</td>
<td>0.08742</td>
</tr>
<tr>
<td>40</td>
<td>3</td>
<td>0.05406</td>
</tr>
<tr>
<td>60</td>
<td>3</td>
<td>0.03597</td>
</tr>
<tr>
<td>80</td>
<td>3</td>
<td>0.0264</td>
</tr>
<tr>
<td>40</td>
<td>1</td>
<td>0.1257</td>
</tr>
<tr>
<td>40</td>
<td>3</td>
<td>0.05406</td>
</tr>
<tr>
<td>40</td>
<td>10</td>
<td>0.0114</td>
</tr>
</tbody>
</table>

Table 5.2. Noise in the strain data, as calculated as three times the standard deviation, as a function of subset and step size.
5.4 Discussion

This work determined that e-beam lithography provided a unique opportunity to probe how speckle patterns could be optimized for DIC measurements. Depending on the grain size of the material being studied the speckle size and density could be tailored accordingly. The Vistec EBPG 5000 e-beam lithography system repeatability produces a pattern with resolution of 25 nm. This makes it easy to produce a variety of repeatable patterns for DIC. Though, if much smaller or larger speckles are required, the exposure voltage and current densities presented here should be varied to provide accurate edge retention.

5.4.1 Intensity Resolution

The noise intensity of the strain data was calculated as a function of speckle pattern size. Representative comparison of strain noise were presented, in Figure 5.2, as three times the standard deviation as calculated using a subset size and step size of 40 and 3 pixels respectively. The data indicated that speckle pattern density less than 23% coverage would lead to substantial increase in signal noise. Above this threshold value, there appeared to be marginal but continual reduction of signal noise as the pattern density is increased. It was expected that pattern density 5, 58% area fraction, would have similar noise intensity to a pattern with 42% area fraction coverage, since above 50% the “random speckle” becomes the substrate and the hafnium oxide becomes the neutral background. The signal noise in the y-direction was always higher than the signal noise in the x-direction for patterns created with square speckles.
Diamond shaped speckles dramatically decreased the y-direction signal noise, with little effect on the x-direction signal noise. This noise variation is due to the nature in which images are acquired in an SEM. SEM images are acquired in a simple raster pattern where pixels in the x-direction are samples continuously along a row before stepping the beam down in the y-direction to sample the next row of intensities. The comparatively long time between sampling pixels in the y direction leads to higher probability of image drift that can affect strain calculations. This leads to noise when the speckle shape is aligned along horizontal speckle edge (parallel to the x-direction raster) as intensity could belong to one row of pixels in one image and another row of pixels in the next image creating virtual deformation, making it difficult to get sub-pixel resolution. When the speckle edge was not parallel to the raster direction, it will always belong to multiple rows of pixel, and thus increasing the strain intensity resolution. Though the diamond speckles for this study were merely rotated square speckles, further work with creating diamond speckles indicated that there was no dramatic increase in processing time as compared with square speckles.

Increasing the subset and step size DIC parameters also affects the intensity resolution of the measurement. Large subset and step sizes lowered the intensity noise, and in turn increased the intensity resolution. It was seen that increasing the subset and step size parameters had a detrimental effect on the spatial resolution.
5.4.2 **Spatial Resolution**

The density of the speckle pattern had a very small effect on the spatial resolution of the DIC measurements. The minimum subset size was limited by the spacing between speckles. The data indicates that a minimum speckle density of about 25% area fraction is needed so that the speckle pattern does not limit the spatial resolution of the DIC measurements. If additional spatial resolution was required, then higher resolution images should be collected, and a smaller speckle pattern incorporated.

Spatial resolution was maximized with small subset and step size DIC parameters. The smaller the subset size, the better the spatial resolution and the worse the intensity noise distribution. The minimum subset size is a function of the speckle density. For the least dense speckle pattern the minimum subset size was 30 pixels, while for the denser patterns the minimum subset size was limited by the DIC software not the speckle pattern. The best spatial resolution is obtained by minimizing subset and step sizes, while increasing the intensity noise. Therefore, optimum parameters are set to minimize intensity noise while maximizing spatial resolution. For the experiments conducted these parameters were 60 pixel subset and 2 pixel step size.

A comparison of actual data can be seen in Figure 5.5, which shows that increasing the subset size to 100 pixels broadens the strain data around the grain boundaries. When parameters are used to optimize spatial resolution (25 pixel subset and 3 pixel step), the strain becomes highly localized at the grain boundaries, though
this increases the noise intensity in grains exhibiting average strain accumulation. Optimal parameters for minimizing intensity noise are also shown, it can be seen that these optimized parameters maximize the spatial resolution but minimize the effects of intensity noise.

![Figure 5.5](image)

Figure 5.5. The affect of VIC-2D parameters on the visual interpretation of the data. (a) Optimal intensity noise, (b) optimal intensity/spatial resolution, and (c) maximizing spatial resolution. Variations in intensity noise are clearly visible in the grains showing little strain accumulation.

### 5.5 Conclusions

Different patterning parameters can be altered to optimize strain resolution of DIC from SEM images, with higher density speckle patterns enhancing both the intensity and spatial resolution of the measurements. A speckle pattern with 50% area fraction would provide optimum resolution, but patterns with at least 25% area fraction and optimum speckle size exhibit little loss in resolution. This indicates that decreasing
the speckle density to add secondary information, grid markers for grain boundary sliding measurements and alphanumeric markers for subsequent alignment for example, will not greatly reduce the effectiveness of the DIC measurements.

It was also concluded that square speckles were not the optimal speckle geometry. Alignment with the rastering direction of the SEM imaging system led to a pixilated edge. This was particularly apparent with this pattern because speckles were only 9-49 pixel\(^2\). A diamond pattern dramatically decreased the intensity noise in the \(\varepsilon_{yy}\) measurements, and by extension a round speckle would be the optimum speckle geometry.

Optimizing intensity and spatial resolution of the DIC measurement proved to be contradictory requirements. Decreasing intensity noise and thus increasing intensity resolution by increasing subset and step sizes simultaneously lowers spatial resolution. Therefore, parameters should be tailored depending on data required. Since optimal spatial resolution is often more important than the absolute intensity value, unless of course modulus measurements are required for example, the smallest subset and steps possible should be used when analyzing data sets.
Chapter 6: Application of in-situ technique to materials with different grain boundary morphologies

6.1 Introduction

Creep deformation in polycrystalline nickel-based superalloys is a heterogeneous process that is primarily a function of γ’ distribution [1-4], but grain size and orientation also play a role [5, 6]. The propensity of creep cracks to nucleate at grain boundaries was the primary motivation for creating directionally solidified and single crystal turbine blades [27, 28]. Unfortunately, the loading conditions and component geometry of most applications do not lend themselves to removing grain boundaries. Previous work with nickel-bases superalloys has explored the effects of grain boundary character on deformation mechanisms, and has concluded that increasing the fraction of special Σn boundaries with n values ranging between 3 and 29, as determined by Coincidence-Site Lattice (CSL) Theory [7], dramatically increases creep, fatigue and corrosion resistance due to a decreased propensity for crack nucleation at Σn boundaries [8-10]. This is because the highly ordered structure of Σn grain boundaries increases their resistance to crack nucleation and grain boundary sliding (GBS) as compared to random high angle grain boundaries (HAGB).
GBS occurs in these materials when creep strain is accommodated by grains displacing relative to each other, and must be accompanied by intra-granular dislocation motion [11, 12]. Preliminary findings on modern turbine disc alloys have also indicated that intentionally creating micro-scale serrated grain boundaries increases creep resistance by limiting GBS [14]. These conventional studies have concluded these relationships through comparisons of macroscopic creep curves of materials with variations in microstructure and inferring that variations in properties have to be attributed to changes in GBS behavior. Soula et al. used a full-field digital image correlation (DIC) technique with grid markers to conclude that GBS was active at conventional creep strain rates [13]. Soula et al. measurement of GBS was based on the techniques developed by the super-plasticity community. In which GBS is measured by placing a set of grid markers on the sample surface prior to deformation and measuring discrete offsets in these markers after deformation [36]. The contribution of GBS to the total plastic strain can be determined by measuring the average displacements of the grid lines after deformation.

\[ \varepsilon_t = \varepsilon_g + \varepsilon_{GBS} \]  
\[ \varepsilon_{GBS} = 2n_i \left( \frac{w}{\tan \theta} \right) = n_i \left( x \tan \theta \right) \]

The total plastic strain, \( \varepsilon_t \), is a function of the intra-granular deformation, \( \varepsilon_g \), and GBS deformation, \( \varepsilon_{GBS} \). The strain from GBS is calculated as a function of average of
discrete offsets on either longitudinal or transverse grid lines, w or x, the average number of grains per unit length of line, \( n_l \) or \( n_t \), and \( \theta \), the angle between the tensile axis and grain boundary trace.

Since the above equations only rely on an averaged displacement measurement they are not dependent on the number of active boundaries in a particular area. For super-plastic materials this is of no consequence because GBS is the primary mechanism of deformation, so it is assumed that all grain boundaries exhibit a degree of sliding. In superalloy materials GBS is a secondary deformation mechanism and does not occur at all grain boundaries; therefore an averaged approach may not be able to distinguish the difference in sliding behavior of two materials. Instead a new equation was developed in which the strain accommodated by GBS is calculated as a summation of displacements, instead of an average.

\[
\varepsilon_{GBS} = \frac{\bar{g}}{A} \sum s_i n_i
\]  

(6-3)

Where \( \bar{g} \) is the average grain boundary length, \( A \) is the area analyzed, \( s_i \) is the sliding displacement, and \( n_i \) is the grain boundary normal each respectively projected in the tensile direction. With this equation, the number of events per area comes into play allowing for a comparison of materials with similar sliding offsets but different numbers of active grain boundaries.
Beyond just understanding GBS mechanisms, it is also unclear how the complex nature of polycrystalline samples dictate why some grain boundaries do or do not accumulate strain, either because of intra-granular slip inactivity or easy slip transfer across boundaries. Full field strain maps measured from digital image correlation can provide insight into how the grain boundary structure is contributing to the localization of deformation.

The objective of this study was to utilize full-field deformation maps created through DIC to probe strain localization, and GBS in two materials processed to have very different grain boundary structures, one with macroscopically planar boundaries and one with macroscopically serrated grain boundaries. These two microstructures were created in alloy René-104 by following two different super-solvus heat treatments: the standard heat treatment for turbine disc applications which creates macroscopically straight grain boundaries, and a proprietary heat treatment similar to that described by Danflou et al. [63] to create macroscopically serrated grain boundaries.

6.2 Experimental Technique

6.2.1 Material

Samples of René-104 were heat treated under two different super-solvus conditions to produce microstructures with either macroscopically straight (standard) or serrated grain boundaries, similar to those described by Danflou et al. [16]. Heat treatments were tailored such that both microstructures had similar γ grain size.
distributions, 22 ± 8 μm and 23 ± 8 μm for the standard and serrated microstructures respectively. The standard heat treatment produced a bimodal \(\gamma'\) distribution of cuboidal secondary and tertiary particles. The serrated heat treatment produced a complex distribution with very large secondary \(\gamma'\) on the grain boundaries, and in the grain interiors: dendritic and cuboidal shaped secondary \(\gamma'\), and tertiary \(\gamma'\) are present. The \(\gamma'\) distributions of these materials are very different, the cuboidal secondary \(\gamma'\) have a size and spacing of 114 ± 82 nm and 38 ± 49 nm for the standard microstructure and 69 ± 50 nm and 35 ± 36 nm for the serrated microstructure. Since the secondary \(\gamma'\) size for both microstructures should promote particle cutting and not looping, and since the \(\gamma'\) particle spacing is very similar, it is expected that these materials should have similar active deformation mechanisms.

Rectangular dog-bone specimens for constant load testing were electrical discharge machined with a gage length of 1.78 cm, and a cross-section with nominal dimensions of 0.31 cm x 0.081 cm. Specimens were polished on a Allied MultiPrep system through 1200 grit SiC to maintain specimen planarity, before a final vibratory polish with 0.02 μm colloidal silica polish. After polishing, four hardness indents were placed in the center of the gage section 1 mm apart. These were used as reference markers so electron backscatter diffraction (EBSD) and DIC could be performed on the same area.
6.2.2 Characterization

EBSD analysis was conducted to determine grain orientation and shape prior to deformation, using the EDAX TSL OIM software on an FEI scanning electron microscope (SEM) with FEG source and probe current of 49.5 nÅ and the TSL Hikari camera, settings were optimized to collect patterns at 200 frames per second. The 1 mm$^2$ areas were analyzed using the EBSD BatchScan software to collect a batch of higher magnification images to minimize spatial distortions [79]. Once collected the smaller regions were compiled using AnyStich [80]. The advantage of using these two sets of code over the internal commands in TSL software include but are not limited to: decreased the acquisition time and less subjective and time consuming tile merging. Identification of $\Sigma n$ boundaries was conducted using Brandon's criterion: which specifies that the angular tolerance for a $\Sigma n$ boundary is equal to $15/\sqrt{n}$ [81, 82].

6.2.3 In-situ Experiments

After EBSD analysis a pattern of hafnium oxide was deposited onto the sample surface using electron beam lithography techniques. The pattern consisted of random diamond shaped speckles 200-700 nm in size for DIC, a repeating grid pattern with lines 10 $\mu$m apart for GBS measurements, and a series of alpha-numeric markers were placed 200 $\mu$m apart for locating and aligning regions of interest. Specifics of the technique can be found in Chapter 3 and Walley et al. [78].

Correlated Solutions VIC-2D software was used to conduct the full field strain measurements from secondary electron (SE) images created during in-situ elevated
temperature constant load tests. SE images were acquired at preload (approximately 200 MPa) and after set periods of time at the test load in a step-wise fashion where the peak load was dropped by 20% for image acquisition. Peak loads varied between 900 to 1100 MPa from test to test. Images were acquired at either 63 nm/pixel or 150 nm/pixel resolution with total dimensions of 258 x 223 μm. For a direct comparison of strain localization sites, DIC parameters were varied so that subset and steps sizes were the same size: 4.5 μm and 0.45 μm respectively. Additionally, analysis was repeated on the higher resolution images with subset size of 1.9 μm and step sizes of 190 nm to conclude if the DIC analysis could measure GBS events. During constant load testing specimens were heated by a resistance heating unit that was in direct contact with the underside of the specimen surface. Sample temperature was maintained at 700°C ± 15°C. Plastic strain versus time was calculated from the average of the full field strain measurements, and elastic strain was removed assuming an average Young's Modulus of 200 GPa.

6.2.4 Grain Boundary Sliding Measurements

Measurement of GBS events was conducted over areas approximately 0.3 mm² in size with image resolution at 25 nm/pixel, in an attempt to characterize a statistical number of sliding events. For each grain boundary investigated, the discrete offsets on either longitudinal or transverse lines were measured. Also measured was the angle that the boundary plane made with the tensile direction, and the EBSD data was used to characterize each boundary, i.e. random high angle grain boundary (HAGB), annealing
twin ($\Sigma 3$), or higher order special boundary ($\Sigma n$ with $3<n<31$). The analysis revealed that some boundaries exhibited multiple sliding events. When using the Langdon equations this is of no great importance because the displacement events are averaged to get a strain value. For use in equation 6-3 the average of multiple sliding events on a single boundary were averaged together before conducting the summation. An example of the types of offsets seen in these materials is shown in Figure 6.1.

![Figure 6.1. GBS events between grains 1 and 2. Discrete offsets in the grid markers are circled in white, and the white line indicates the grain boundary normal. The loading direction is along the horizontal direction.](image)

6.3 Results

6.3.1 Average Strain Behavior

Calculating the average strain from the full field strain mapping provides some interesting results. By comparing averaged measurements from multiple images acquired of the same region, this provides a technique to understand the precision of
any measurement and could indicate an error associated with the imaging process. Figure 6.2 shows how the averaged value depends on the image, indicating that the error associated with any averaged value is on the order of 0.006% plastic strain. In some cases it was observed that the standard deviation was significantly larger than this, which on inspection of the images confirmed that one or more images had significant distortions, often due to unexplained image drift. When comparing the averaged plastic strain measured from multiple regions of the sample, it is apparent from Figure 6.2 that the 258 x 223 μm image area is large enough to predict bulk behaviors, as the averaged plastic strain measured from different locations on the samples are similar within a standard deviation of less than 0.18%.

Figure 6.2. The variation in average creep strain measured as a function of time and image position for both the standard (a) and serrated (b) microstructures. The error bars indicate the standard deviation in average from four images taken at the same location. The inset in (b) shows that the error from imaging is very small.
Unfortunately, when comparing samples that were tested at the same applied stress, or even different stress levels, as seen in Figure 6.3, it becomes obvious that the inherent lack of testing temperature control makes it impossible to compare bulk strain rates from one sample to the next, let alone compare the creep strain rates of one material type with another. It is noticeable that for most tests the strain verses time curves have the correct shape, indicating that even if the tests cannot be used to compare bulk creep strain rates between different material types, the methods of local strain accumulation can be compared between different material types.

Figure 6.3. Creep strain as a function of time for a pair of (a) standard and (b) serrated microstructure samples, showing that the measured the creep lives are inconsistent because of the inability to reliably replicate specimen heating.
6.3.2 Full Field Strain Maps

Strain development as a function of time shows that the standard (Figure 6.4) and serrated (Figure 6.5) microstructures demonstrate different attributes. In the case of the standard microstructure, strain initially localizes at grain boundaries and triple points, as indicated by ellipses in Figure 6.4a, and then linkage between these initial sites occurs later in the deformation process. On the other hand, the serrated microstructure appears to produce less intense localizations that traverse multiple grain interiors and grain boundaries at macroscopically 45° angles, as indicated by ellipses in Figure 6.5a and b, before strain localizes at particular boundaries and slip traces within these initial strain bands with additional strain accumulation.
Figure 6.4. Strain development as a function of time for the standard sample, showing that strain localizes at grain boundaries and slip traces occurs early at (a) 0.92% average strain accumulation and continue to accumulate in strain at (b) 2.75% average strain, until at (c) 4.41% average strain, the localization sites consume the neighboring microstructure.
Figure 6.5. Strain accumulates in the serrated microstructure on a macroscopic scale, independent of the underlying microstructure at 45° angles to the loading direction (horizontal) showing no preferred localizations at (a) 0.85% and (b) 1.73% average strain accumulation. It isn’t until later that localization occurs on grain boundaries in the vicinity of earlier macroscopic “slip bands” as seen at (c) 2.75% total strain accumulation.
When comparing how strain localized in both microstructures, it was important to compare moments in time when both samples had similar amounts of average strain accumulation. Figure 6.6 shows a direct comparison between both microstructures with an average plastic strain of 2.75%. Next, each localization site with strain value above twice the average strain was categorized based on its location in the microstructure, either along a HAGB, $\Sigma 3$ or $\Sigma n$ boundaries, or along intragranular slip bands. This analysis was done as a function of strain accumulation as seen in Figure 6.7 and Figure 6.8 for the standard and serrated microstructures respectively. Figure 6.7 indicates that the percentage of strain localization sites that are associated with triple points, HAGB, $\Sigma 3$, $\Sigma n$, and intergranular locations varies with material processing. The standard microstructure initially localizes pretty evenly on all intergranular sites, but as a function of time the several of the HAGB sites do not continue to show strain accumulation above the average strain rate and therefore near the end of the test $\Sigma 3$ and triple point locations account for a majority of localization sites, as seen in Figure 6.7. Even though some intragranular locations accumulate strain early in the test these sites do not account for much of the strain accumulation beyond average in the standard microstructure. The serrated microstructure indiscriminately forms localization sites at all the intergranular locations and intragranular sites account for consistent percentage of localization sites, as seen in Figure 6.8. This difference in localization site phenomena is possibly due to the fact that the serrated microstructure indiscriminately forms strain bands across multiple grains, before forming more intense
localization sites while the standard microstructure forms localization sites before these sites grow to consume other areas of the sample.

Figure 6.6. A comparison of strain accumulation between the (a) standard and (b) serrated microstructures. The loading direction is along the horizontal direction, and the average plastic strain is both samples is 2.75%. 

93
Figure 6.7 Percentage of strain localization sites as a function of strain accumulation for the standard microstructure. The data indicate that the triple points and $\Sigma 3$ boundary sites continue to accumulate strain while some of the random HAGB locations do not continue to accumulate strain at a rate higher than average.

Figure 6.8 Percentage of strain localization sites as a function of strain accumulation for the standard microstructure. Data indicates that strain accumulation at $\Sigma 3$ initially occurs early in deformation but and more sites triple point and HAGB sites form as strain accumulation continues.
6.3.3 **Grain Boundary Sliding Measurements**

Grain boundary sliding measurements were conducted over very large areas of each microstructure type, such that a statistically viable number of sliding events could be measured. For the standard and serrated microstructures, which had image areas of 0.312 mm$^2$ and 0.298 mm$^2$ respectively, each microstructure had over 700 grain boundaries that exhibited GBS. The standard microstructure had 31% more active boundaries than the serrated microstructure with 3359 active boundaries per square mm versus 2416 active boundaries per square mm respectively, in both cases there are approximately 9300 total boundaries per square mm. This confirms that for these materials only 36% or fewer grain boundaries are deforming by GBS, indicating that equation 6-2 will provide a substantial overestimate of the strain accommodated by GBS. Table 6.1 compares the percentage of active boundaries that were random HAGB, $\Sigma 3$ twin boundaries and $\Sigma n$ special boundaries. The results confirm the previous conclusions that the creation of serrated grain boundaries reduces the propensity of those grain boundaries to deform by GBS. As the mechanism of forming GBS only effects the HAGB and the $\Sigma n$ special boundaries, it would be expected that the sliding behavior of the $\Sigma 3$ would be unaffected. Table 6.1 confirms that even though the total number of active grain boundaries per unit area decreased with the serrated microstructure this was not because of equal decreases in the activity of all boundaries types. When comparing the total number of active boundaries in each material type, there was a reduction in active HAGB and $\Sigma n$ by 35% while there was
only a 19% drop in the total number of active $\Sigma 3$, and thus the $\Sigma 3$ boundaries account for a higher percentage of active grain boundaries seen in Table 6.1.

<table>
<thead>
<tr>
<th></th>
<th>Standard</th>
<th>Serrated</th>
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</thead>
<tbody>
<tr>
<td>HAGB</td>
<td>69%</td>
<td>66%</td>
</tr>
<tr>
<td>$\Sigma 3$</td>
<td>23%</td>
<td>28%</td>
</tr>
<tr>
<td>$\Sigma n$</td>
<td>8%</td>
<td>6%</td>
</tr>
<tr>
<td>$\varepsilon_t$</td>
<td>3.11%</td>
<td>2.75%</td>
</tr>
<tr>
<td>$\varepsilon_{GBS/\varepsilon_t}$ (6-2)</td>
<td>52%</td>
<td>48%</td>
</tr>
<tr>
<td>$\varepsilon_{GBS/\varepsilon_t}$ (6-3)</td>
<td>13%</td>
<td>9%</td>
</tr>
</tbody>
</table>

Table 6.1. The percentage of GBS events that occurred at different grain boundary types. Also, the percentage of total strain accommodated by GBS as calculated from the two different equations.

The strain associated with GBS was calculated using both equations 6-2 and 6-3. Since the total plastic strain accumulated in both samples is slightly different, for comparison purposes the percentage of total plastic strain accommodated by GBS is presented in Table 6.1. In both cases the standard microstructure was more susceptible to grain boundary sliding. Since equation 6-2 assumes all grain boundaries are actively sliding, it over estimates the percentage of total strain accommodated by GBS. Instead, when the number of active grain boundaries is taken into consideration using equation 6-3 it is apparent that there is a substantial difference in the sliding behavior of these two material types.

A comparison of GBS events measured from discrete offsets and the strain localization sites measured from DIC indicates that there is no correlation between
strain localization sites and GBS events. This could indicate that a 190 nm step size and 1.9 μm subset size are not small enough to measure GBS.

6.4 Discussion

The average strain measurements confirmed that for the amount of total plastic strain accumulated in the samples, less than 5% average plastic strain, that an imaging region of 258 x 223 μm was large enough to measure bulk strain measurements. The data indicates that at higher amounts of strain accumulation this observation might not hold, since the standard deviation between average strain measurements in each area increased with increased strain accumulation.

A statistical representation of the localized deformation behavior could not be obtained for the original 258 x 223 μm image area. In order to understand how the microstructure was dictating strain localization, regions five times the original size were necessary. This large sampling area shows that the standard and serrated microstructures accumulate strain as a function of time in slightly different manners. The standard microstructure shows localization on grain boundaries early in the process, and strain accumulates beyond these initial regions to the surrounding microstructure. The serrated microstructure accumulates strain on macroscopic 45° bands before more intense localizations occur at grain boundaries in these larger regions. The result of these differences in strain accumulation mechanisms leads to variations on the percentage of boundary types that show accumulation in each microstructure. The standard microstructure surprisingly showed that strain
accumulation at $\Sigma 3$ twin boundaries was statistically more likely to occur than accumulation over other microstructural sites. This is interesting because in many cases the addition of $\Sigma 3$ twin boundaries is associated with better mechanical and environmental properties. This may indicate that even though strain accumulation readily occurs at these boundaries, they may not be the first boundaries to experience crack initiation and failure. The serrated microstructure showed no preferential placement of strain accumulation sites with regards to the microstructural features. This would be expected if strain is first localizing on a macroscopic scale, independent of microstructural features, before further straining leads to localizations at microstructural sites in these pre-existing deformation bands.

After performing a comparison of GBS behavior for the two material types, it becomes apparent that using equation 6-2 for an average measurement of GBS is not adequate. When it is assumed that all grain boundaries are active, the percentage of total strain accommodated by GBS is on the order of 50% of the total plastic strain, but in both cases the percentage of total active sliding boundaries is much smaller, 36% and 26% of boundaries were active in the standard and serrated microstructures respectively. It is an interesting coincidence that multiplying the percentage of strain calculated from equation 6-2 by the percentage of active grain boundaries gives a percentage of total plastic strain accommodated by GBS of 19% and 13% for the standard and serrated microstructures, respectively, values that are more in-line with the values calculated by equation 6-3. When the total number of grain boundaries actively sliding is explicitly taken into consideration by using equation 6-3, it can be
seen that creating a serrated microstructure limits the activations of sliding mechanisms, such that the proportion of plastic strain accommodated by grain boundary sliding is reduced by 31% as compared to the standard microstructure. Grain boundary sliding was limited but not eliminated because the mechanism that creates serrated grain boundaries does not affect all grain boundaries equally. Particularly, the \( \Sigma 3 \) twin boundaries are unaffected by this mechanism, and are therefore still susceptible to grain boundary sliding as seen by the fact that they contribute to a higher percentage of active grain boundaries in the serrated microstructure.

It was interesting that strain localization sites measured by DIC did not seem to correlate with GBS events. This result arises from the method by which the DIC technique measures displacements, and in turn calculates strain, which relies on applying an affine transformation over a given subset size. It is not possible to apply an affine transformation to an area that does not maintain continuity. This of course is the goal behind using very small subset sizes, as on either side of the sliding event there would be continuity. A 1.9 \( \mu \)m subset size and 190 nm step size were not small enough to capture GBS events in these materials. As the average displacement for each sliding event is on the order of 100 nm it would indicate that a much smaller subset size and step size would be needed to measure the discrete nature of GBS. To measure GBS it would be advantageous to have subset size on the same order of magnitude of the displacement event. This leads to significant restrictions to conducting the experiment: image resolution, speckle pattern generation, and statistical selection of grain boundaries would be of primary concern.
6.5 Conclusions

The limited repeatability of the specimen heating system for the *in-situ* experiments made it impossible to conclude if the differences between the two materials lead to different creep lives. Conventional experiments on dwell fatigue properties do indicate that the serrated microstructure has better creep-fatigue lives, but it is unclear if this comparison would be true for these testing conditions [12]. This leads to the desire to either develop an *ex-situ* approach with better temperature control, or conduct a multi-faceted testing approach to understand both bulk and localized properties.

Full field strain mapping provides a unique opportunity to assess how strain accumulates in different microstructures as a function of time at constant load. The small imaging regions proved to be large enough to measure averaged properties, but these were not large enough of a sampling area to gain an understanding of trends in the localization behavior. Larger sampling regions were needed to assess trends in both the strain localization behavior and the grain boundary sliding behavior of these two materials. A comparison of region with both full field strain maps with the boundaries identified as active sliding boundaries did not show a direct correlation between the strain localization sites and the grain boundaries that showed grain boundary sliding. The standard microstructure was more susceptible to strain localization near $\Sigma3$ twin boundaries, while the serrated microstructure was equally as likely to form localizations anywhere in the microstructure, independent on grain boundary type.
Chapter 7: Statistical Parameters for Predicting Strain Localization at Grain Boundaries

Nickel-based superalloys are used in the demanding conditions of the combustion chamber of gas turbine engines. Understanding how the mechanical properties of these systems vary as a function of microstructure is an extensive ongoing field of research. The primary mechanism controlling mechanical behavior of these systems is the size and distribution of the second phase particles, $\gamma'$, and optimizing the creep properties of polycrystalline superalloys requires a bi-modal distribution of $\gamma'$ particles [13, 65]. This distribution is developed through super-solvus heat treatment, which also creates large grains. Though larger grain sizes are advantageous for creep life, and limiting crack initiation at grain boundaries was the primary reason for the creation of single crystal turbine blades [27, 28], a large grain size is considered detrimental to the fatigue properties these materials [83]. With every generation of jet engine, increased engine efficiency is expected by increasing operating temperatures and load rates. These design requirements are pushing the development polycrystalline turbine disc alloys to be optimized for both creep and fatigue strength. Since these properties have contradictory requirements on grain size, it is imperative that an understanding be developed of how deformation accumulates and is transmitted across
grain boundaries so that a compromise between competing requirements can be devised.

Slip transmission across grain boundaries is dependent on the compatibility of the active slip systems at the grain boundary. Figure 1 shows a schematic representation of two slip systems meeting at a grain boundary. Several metrics have been proposed to describe the ease of slip transmission. One metric proposed by Luster and Morris is the product of the direction cosines between the slip plane normals and slip directions in each grain, and is termed $m'$. This basic concept has been modified by researchers in an attempt to predict fracture initiation parameters at grain boundaries of $\gamma$-TiAl by also accounting for the effect of Schmid Factor (SF) from either simulated local or experimental global stress states. In their work, the authors found that boundaries amenable to slip transmission were statistically more likely to form cracks than boundaries which slip could not transmit across. As the failure mechanisms in $\gamma$-TiAl, brittle fracture, and nickel-based superalloys, ductile fracture, are very different it may be very likely that easy slip transfer would not be a precursor to crack initiation. As such, in this work we only discuss difficult slip transfer (“hot”) verses easy slip transfer (“not”) at grain boundaries but not if these mechanisms dictate crack initiation.

Whether or not grain boundaries accumulate strain during loading is a function of many variables including the activity of slip in each grain, the ease of slip transfer from one grain to the next, the availability of secondary slip systems in each grain to relieve the strain field at the head of a dislocation pileup and the elastic mismatch
between the grains. In this work, various geometrical criteria based on crystallography have been used to interrogate these phenomena at grain boundaries exhibiting strain accumulation (“hot” boundaries) versus those that did not (“not hot” boundaries). The simplest slip transmission parameter (STP) can be calculated taking into account the likelihood of nucleating particular slip systems by using a summation over the $m'$ term, weighted preferentially for systems with high SF:

$$S_1 = \sum_{i \neq j} SF_i SF_j \hat{b}_i \cdot \hat{b}_j \|\hat{n}_i \cdot \hat{n}_j\|$$

(7-1)

Where $SF$ stands for Schmid Factor, and $\hat{b}$ and $\hat{n}$ are respectively the Burgers vector and slip plane normal for all slip system in each grain. This weights the $m'$ based on the probability that that some slip systems are more likely to be activated than others. Since this summation is occurring over all slip systems, this $S_1$ term is accounting for the probability of multiple slip system activation.
Figure 7.1. Schematic showing the compatibility of a slip system in grain 1 with a slip system in grain two can be described by the angles between the Burgers vectors (κ) and slip plane normal (ψ).

If S1, or any of the other STP, can be used to discern between those boundaries that either do or do not show strain, than the distributions of the STP values for hot and not hot boundaries should be centered around different values, this is shown schematically in Figure 7.2.

Figure 7.2. Schematic showing what would be expected if the STP S1 calculated a difference between the boundaries that experience strain accumulation and grain boundaries that do not experience strain accumulation.
The $S_1$ term can be modified to account for the interaction between the grain boundary plane and the slip planes in each grain. In this case it is interesting to know the trace of the each slip plane projected in the grain boundary plane, which is calculated by the cross-product between the grain boundary plane normal and the slip plane normal. This equation calculates the mismatch of the two slip planes with respect to the grain boundary plane, instead of calculating a three dimensional mismatch.

\[
S_2 = \sum S_i S_j \left[ \hat{b}_i \cdot \hat{b}_j \left( \hat{n}_{gb} \times \hat{n}_i \right) \left( \hat{n}_{gb} \times \hat{n}_j \right) \right]
\]

(7-2)

where $\hat{n}_{gb}$ is the normal to the grain boundary plane. It is not possible to calculate $\hat{n}_{gb}$ from a 2D section of the structure, so unless otherwise noted it is expected that $\hat{n}_{gb}$ is actually the trace of the grain boundary plane on the specimen plane. It is understood that this is a limitation to using the grain boundary normal in the slip transmission parameters because the out-of-plane component is unknown. Both the $S_1$ and $S_2$ terms are only concerned with the magnitude of the dot products, because $S_1$ and $S_2$ terms are analogous to a probability and therefore cannot be negative. Also, only the magnitude of the SF needs to be considered since the tensile loading is symmetric. Since grain boundaries can act as dislocation sources and sinks, transmission and emission of partial dislocations from full dislocations can be considered without requiring recombination before transmitting across the grain boundary.
Previous work on γ-TiAl has indicated that accounting for anisotropic modulus effects can increase the probability of predicting micro-crack initiation. Zener anisotropy coefficient, \( A = 2C_{44}/(C_{11} - C_{12}) \), for γ-TiAl is 1.924 [88]. Since slip transmission maybe a precursor to crack initiation, and superalloys have a higher Zener anisotropy coefficient, 2.85 based on stiffness coefficients from Pottebohm et al. [89], it is expected that incorporating variations of modulus into the \( S_1 \) and \( S_2 \) terms would also be effective for predicting slip transmission in nickel-based superalloys. Variations in moduli were considered through the ratio of the moduli along the tensile axis for the two grains, \( E_{\text{min}}/E_{\text{max}} \). The elastic modulus parallel to the tensile axis can be calculated using the crystal orientations measured through electron backscatter diffraction (EBSD) analysis.

\[
S_3 = E_{\text{min}}/E_{\text{max}} \sum SF_{i}SF_{j} \left| \hat{b}_i \cdot \hat{b}_j \right| \left| \hat{n}_i \cdot \hat{n}_j \right| \\
S_4 = E_{\text{min}}/E_{\text{max}} \sum SF_{i}SF_{j} \left| \hat{b}_i \cdot \hat{b}_j \right| \left( \hat{n}_{gb} \times \hat{n}_i \right) \left( \hat{n}_{gb} \times \hat{n}_j \right)
\]

(7-3)  \hspace{1cm} (7-4)

Of course, there are any number of additional parameters that can be tested based on components of each of the STP already described. These are useful for understanding if there is any preferential selection of localization sites based on, for example, slip direction, slip plane or modulus ratio.

\[
S_5 = \sum \left| \hat{n}_i \cdot \hat{n}_j \right| \\
S_6 = \sum \left| \hat{b}_i \cdot \hat{b}_j \right|
\]

(7-5)  \hspace{1cm} (7-6)
In an effort to understand deformation localization on the grain size scale, a full field digital image correlation (DIC) technique was developed [90]. This technique was used to measure local strain accumulation at grain boundaries in a nickel-based superalloy tested at elevated temperature and strain rates of interest. This work focuses on determining if any of the various STP’s can be used to predict grain boundaries that would experience strain localization or grain boundary sliding as measured by full field DIC measurements.

7.1 Experimental

The material examined in this study was a wrought nickel-based superalloy, René-104. Material was super-solvus heat treated and aged to produce a bi-modal $\gamma'$ particle distribution with an average grain size of $22 \pm 7.8 \mu m$. Rectangular creep specimens were prepared with a nominal initial cross-section of 0.31 cm x 1.78 cm, and mechanically polished for electron backscatter diffraction (EBSD) analysis of grain size and orientation. EBSD analysis was conducted prior to deformation on an FEI XL-30 scanning electron microscope (SEM), nominally at 20kV. The trace of each grain boundary normal was analyzed from the surface EBSD patterns through the
reconstructed grain boundary function of EDAX TSL OIM analysis software. Slip transmission parameters were calculated from the grain orientations analyzed by EBSD. For comparison STP’s were calculated by applying a filter to only account for slip systems with SF greater than 0.3. This filter was applied since STEM analysis of selected grain boundaries, shown in Chapter 7, has indicated that only the highest SF are activated in surface grains during deformation. Once STP’s for all boundaries were calculated, boundaries were separated into those that experienced strain accumulation by indexing all the strain localization sites determined by DIC using the unique grain identification integer assigned by the EBSD analysis software.

Constant load testing was conducted using an Ernest Fullam tensile stage with a resistance heater mounted in direct contact with the underside of the sample. Tests were conducted in a large chamber FEI Quanta SEM at constant stress, 1000 or 1100 MPa, at 700°C. SEM images of the sample surface were acquired at regular intervals at 2048x1886 dpi, and a magnification such that there were 6.8 pixels/micron. Correlated solutions VIC-2D was used to analyze the localized strain field from the in-situ images. This software requires a random speckle pattern to conduct DIC. A pattern of hafnium oxide was deposited on the sample surface using standard electron beam lithography techniques; the pattern consisted of a the random speckle pattern for DIC and 10 μm repeating grid for grain boundary sliding measurements [90]. Post deformation analysis of grains that exhibited grain boundary sliding was conducted by measuring discrete offsets in grid line at the grain boundaries of post deformation images taken at 25
nm/pixel resolution. For a more detailed description of the experimental techniques see Carter et al. [90].

7.2 Results

The full field strain maps show that strain localizes in this material primarily at grain boundaries and triple points, as seen previously in Figure 6.7 and Figure 6.4. The area of interest of this study is presented in tensile strain map in Figure 7.3. It can be seen that there are far more grain boundaries that don’t show any strain accumulation, termed not hot boundaries, than grain boundaries that show varying degrees of strain accumulation. Grain boundaries that exhibit strain accumulation values twice the average strain accumulation measured in $e_{xx}$, $e_{xy}$, and $e_{yy}$ measured strain maps were termed hot boundaries, of which there are 79 total boundaries. There are two methods of comparing the distribution of STP values for the hot and not hot boundaries, first by looking at a percentage of total boundaries that have a particular STP parameter value, and secondly by randomly selecting grain boundaries from the not hot array so that the same number of boundaries are compared to the hot array. This second method is similar to site-specifically characterizing a select number of grain boundaries, and would mostly provide similar comparisons to work conducted based on slip trace analysis or TEM analysis [85, 87]. Results using both comparison techniques will be presented in this work. When the not hot boundary array is filtered so that only the same number of grain boundaries as the hot array are considered that is little effect on the shape of the STP histograms. This is shown schematically for $S_1$ in Figure 7.4 in
which the histogram from all of the not hot boundaries (termed not in Figure 7.4) is compared with histograms created by filtering the number of not hot boundary by randomly selecting three different subsets of the not hot array (termed not.1, not.2 and not.3 in Figure 7.4).

Figure 7.3. Strain localizations measured by full field strain mapping. Loading direction is along the horizontal axis, black boundaries are random high angle grain boundaries, white boundaries are $\Sigma 3$ twins, and gray boundaries are $\Sigma n$ boundaries.
Figure 7.4. A comparison of the frequency histograms for all of the not hot boundaries (not) with three different subsets for the not hot array (not.1, not.2 and not.3) in which a random selection of boundaries equal in number to all the hot boundaries shows that filtering the data does not dramatically effect the STP histograms, shown specifically for S1. The total number of hot boundaries is 79.

7.2.1 Percentage of Boundary Comparisons

A comparison of the eleven STP histograms for all the hot boundaries and unfiltered not hot boundaries indicates that none of the STP parameters can distinguish between boundaries that show strain localization and those that do not show strain accumulation. Some parameters hint at a variation in properties as seen in Figure 7.5, STP’s S1, S2, S3, S9 and S10 indicate that there are a higher percentage of hot boundaries that exhibit a particular STP value, but the histograms overlap suggesting that these cannot be used to predict grain boundary deformation behavior. Histograms for all of the STP values are presented in Appendix A.
Figure 7.5. histograms for STP’s (a) S1  (b) S2 (c) S3 (d) S9 showing that the only variation between hot and not hot boundary histograms are the standard deviation of the peaks. The same comparisons hold true for all the other stp.

Previous work has compared hot boundaries to not hot boundaries with very particular orientations with respect to the tensile axis [84, 85, 87]. In an attempt to conduct a similar analysis the hot and not hot boundaries were further segmented based on the angle the grain boundary trace makes with respect to the tensile axis, using 10° angle increments. Table 7.1 shows that there are a higher percentage of boundaries
with orientations at 60-70° angles with respect to the tensile direction than any other orientation independent of grain boundary behavior. When the STP values for both hot and not hot boundaries are segmented by boundary orientation, there was no observed correlation between STP histogram peak values and the grain orientation, seen in Appendix A for the distributions for the hot arrays. These results would indicate that partitioning by grain boundary orientation show have limited effect on the calculated stp distributions. There were two possible distinctions between the STP distributions when separated by grain boundary orientation: in many cases (58% of the time) the magnitude of the hot histogram peak was greater than the not hot histogram peak by more than 10%, as indicated by the “+” in Table 7.2. There were no instances when the hot histogram peak was less than the not hot histogram peak. Independent of peak intensity there were several instances where the peak intensities of the two boundary conditions did not coincide with the same STP values. The other variation that was observed was that is some cases the distribution peaks for the hot and not hot boundaries sets did not coincide with the same STP value. Table 7.2 indicates difference in the number of bins between peak locations, (example: “1” indicates that the not hot distribution is centered one bin above the hot distribution peak bin). Table 7.2 shows that S4 had most consistent variations in STP distributions with grain boundary type in almost all cases the not hot boundaries had a distribution peak centered at a larger value than the hot boundaries, the distributions of which are shown in Appendix A. S7 had a consistent variation to S4, but a comparison of the distributions, one of which is shown in Figure 7.6, indicates the relative scatter in the
distributions is substantial. Analysis of all the distributions indicates that in all cases the two distributions overlap significantly, indicating that none of the parameters can statistically distinguish between hot and not hot grain boundaries. Figure 7.6 shows a selection of some STP distributions showing the possible correlations between STP value and grain boundary property.

<table>
<thead>
<tr>
<th></th>
<th>10-20°</th>
<th>20-30°</th>
<th>30-40°</th>
<th>40-50°</th>
<th>50-60°</th>
<th>60-70°</th>
<th>70-80°</th>
</tr>
</thead>
<tbody>
<tr>
<td>hot (%)</td>
<td>16</td>
<td>10</td>
<td>15</td>
<td>14</td>
<td>13</td>
<td>24</td>
<td>8</td>
</tr>
<tr>
<td>not hot (%)</td>
<td>13</td>
<td>16</td>
<td>14</td>
<td>14</td>
<td>5</td>
<td>26</td>
<td>12</td>
</tr>
</tbody>
</table>

Table 7.1. Table of the percentage of grain boundaries with particular orientations with respect to the tensile axis that contribute to either the hot or not hot arrays.
Table 7.2. A comparison of histogram peak location and histogram peak intensities for each of the STP. The numbers indicate by the number of bins the not hot boundary distribution was located above the hot boundary distribution peak. The “+” indicate when the intensity of the hot peak exceeded the not hot peak by more than 10%.
Figure 7.6. Histograms for STP’s (a) $S_1$ and (b) $S_{11}$ for boundaries 10-20° (c) $S_7$ for boundaries 40-50° (d) $S_4$ for boundaries 30-40° (e) $S_6$ for boundaries 70-80°.
7.2.2 Comparison of Equal Numbers

When STP histograms for all the hot boundaries are compared with an equal sized subset of not hot boundaries, the same conclusions are gleamed when comparing the percentage of grain boundaries, these histograms are shown in Appendix A. Next histograms for hot boundaries segmented as a function of grain boundary orientation with respect to the tensile axis were compared with histograms created from the same number of not hot boundaries of similar orientations. Three not hot arrays were created from a random selection of not boundaries of similar orientations, labeled not.1 not.2 and not.3. When this analysis is conducted artificial correlations between STP values and hot or not hot properties become apparent; artificial in the sense that these correlations were not consistent from one grain boundary orientation sub-set to another, and were not consistent from one subset of not boundaries to another (i.e. not.1 and not.3). A comparison of STP value for random high angle grain boundaries and ∑3 twin boundaries also indicates that there is no perceivable difference between grain boundaries of different types of misorientation.
Figure 7.7. Histograms for STP’s (a) $S_1$ and (b) $S_{11}$ for boundaries 10-20° (c) $S_1$ for boundaries 30-40° (d) $S_1$ for boundaries 50-60° (e) $S_5$ for boundaries 60-70°.
7.3 Discussion

It was interesting that no particular correlation between STP values and the probability of strain accumulation was determined. This could be due to several different reasons, first the STP were originally developed to predict fracture initiation in γ-TiAl at room temperature [87]. The conclusions from this previous work indicated that slip transmission is a precursor to micro-crack initiation at grain boundaries, with the idea that slip transmission causes increased defect content in the grain boundary. The fact that there was no correlation between STP values and strain localization sites could indicate that ease or difficulty of slip transmission is not the reason behind the increased deformation at the grain boundaries. When the hot boundaries were segmented by the angle between the grain boundary trace and the tensile axis, it indicated the STP $S_4$ might actually indicate a difference between the hot and not hot boundary set. For boundaries that made angles with the tensile axis above 20° the distribution peaks were centered on different values, but there was significant overlap between the distributions. This may indicate that an STP value might be developed but at this point $S_4$ is not a sensitive enough parameter.

When the hot and not hot arrays were segmented into smaller subsets artificial correlations between STP values occurred. These artificial correlations occurred because of the limited sampling number when creating a subset of the not boundary array by the number of hot boundaries with a particular orientation with respect to the tensile axis. This indicates that there is a threshold in the number of observations
needed to really make a comparison of STP histograms between hot and not hot grain boundaries. Since there was not substantial deviation in histograms for the not array when sub-sets based on the total number of hot grain boundaries was used as the subset size, it would indicate that if comparisons of STP distributions, as function of grain boundary trace orientation with respect to the tensile axis, would require the number of observations related to a particular orientation be equal or greater than 80 observed grain boundaries.

### 7.4 Conclusions

Slip transmission parameters were calculated between neighboring surface grains in an attempt to correlate observed strain accumulation sites with parameters associated with misorientation of slip systems across the grain boundary. These STP present a probability that easy slip transmission could occur at a particular grain boundary based on both physical and geometrical factors. The results concluded that there was no direct link between any of the eleven STP values for grain boundaries that exhibited strain accumulation and grain boundaries that did not exhibit strain accumulation. Though, when the results were filtered so that only a subset of not hot boundaries was considered, apparent correlations begin to emerge; apparent in the sense that these correlations were not consistent from one grain boundary orientation subset to another (10-20° versus 40-50°), or consistent from one subset of not boundaries to another (i.e. not.1 and not.3). This could indicate that there is a lower limit on the number of grain boundaries needed to conduct a comparison. The lack of
correlation between STP values and strain localization sites is not surprising, and alludes to the likelihood that strain accumulation at a particular grain boundary is not only a function of the misorientation of the two grains but also dependent on grain neighborhood both from proximity of surface and sub-surface grains.
Chapter 8: Correlation Between Observed and Predicted
Deformation Mechanisms in Nickel-Based Superalloys

The full field strain maps have provided insight into how strain accumulates at grain boundaries in these superalloy materials. If physics based models are to predict deformation in these heterogeneous structures it is important to understand and predict the deformation mechanisms occurring in the grain interiors. Previous work has shown that, in the strain rate and temperature regime of interest for this study, the active deformation mechanisms are dependent on grain orientation and $\gamma'$ particle distribution these materials [10, 21, 65, 91]. These primarily include: Orowan looping, $\gamma'$ shearing by coupled $a/2<110>$ dislocations, isolated faulting of the $\gamma'$ particles through the motion of dissociated partials, or continuous faulting of both the $\gamma'$ particles and the matrix through the motion of decorrelated partials.

This chapter will focus on experimental work conducted to validate the model predictions of active slip systems and subsequent deformation mechanisms on those slip systems. First, slip trace analysis was conducted on surface grains after deformation and results were compared with predicted slip systems from Schmid Factor (SF) analysis. The SF analysis was conducted assuming an idealized situation where the local stress state was that in which the critical resolved shear stress was
determined using the maximum Schmid Factor (SF) value calculated from pure tensile loading. Additionally, full field strain maps were used to measure the development of surface strain distributions and particular grain boundaries of interest were extracted for scanning transmission electron microscopy (STEM) analysis. SF analysis was conducted and compared with observed active slip and Phase Field simulations were used to predict if the slip system should deform by full dislocation motion or by either dissociated or decorrelated partial dislocation motion.

8.1 Experimental

8.1.1 Material

The materials used in this study was René-104 super-solvus heat treated to create a microstructure was average grain size of 22 ± 7.8 µm and a bimodal γ′ distribution of 114 ± 82 nm cuboidal secondary γ′ and smaller spherical tertiary γ′. Since the secondary γ′ size and distribution is the primary strengthening mechanism, the tertiary particles were not characterized. The critical interparticle spacing between secondary γ′ particles was determined to be 38 nm from Delaunay calculations, explained in section 3.3.1. Specifics of microstructural characterization procedures and results are found in Chapter 3 and Chapter 5 respectively.

8.1.2 Phase Field Simulations

Phase Field simulations were conducted by Ning Zhou. The Phase Field model was used to create a Dislocation Activity Diagram (DAD) of the active dislocation
mechanisms as a function of the angle tensile axis and the active slip system. All simulations assumed an intrinsic stacking fault energy of 25 mJ/m$^2$, shear modulus of 67 GPa, and a Burgers vector 2.53 Å. Specifics of the simulation can be found in Carter et al. and Unocic et al.[65, 66].

The DAD shows the influence of the magnitude and orientation of the stress on the dissociation behavior of $\frac{1}{2}\langle 110 \rangle$ type dislocations into pairs of $\frac{1}{6}\langle 112 \rangle$ partial dislocations. The DAD for the standard microstructure is presented in Figure 8.1. The red curve indicates the critical stress needed for the leading partial to pass the $\gamma$ channel, while the blue line is the critical stress needed for the trailing partial to pass the $\gamma$ channel. In the region between the two lines, decorrelated motion of partials occurs and leads to the formation of extended stacking faults and nano-twins [65], as the stress magnitude increases the motion of decorrelated partial dislocations leads to the motion of dissociated ones. Along the vertical axis, which is in units of resolved shear stress, the angle between the Burgers vector and loading direction is 0° and this angle can vary from ±90°, for angles greater than ±90° the nature of the leading and trailing partials flips. Since the nature of the leading and trailing partials flips for angles greater than ±90°, the DAD is mirrored along the horizontal axis.
8.1.3 Characterization

Prior to deformation, grain orientations were determined through electron backscatter diffraction (EBSD) [66, 79, 80]. After EBSD analysis, electron beam lithography was used to deposit a speckle pattern on surface that was needed to measure the strain fields. Constant load tests were conducted on an Ernest Fullam tensile stage mounted in a scanning electron microscope (SEM) at elevated temperature $700^\circ C \pm 15^\circ C$. Images were acquired during mechanical testing, and subsequently analyzed with Correlated Solutions VIC-2D software to measure local strain fields through digital image correlation (DIC). More in-depth specifics about sample preparation and data analysis are presented in Chapter 3. After deformation experiments, a Bruker Veeco optical profilometer was used to measure out-of-plane displacements, a 50x magnifier optic was used for a vertical displacement resolution of 50 nm. High resolution SEM images were also taken after deformation, and the angle that the slip traces made with the tensile axis were measured for each grain with visible
slip steps. The full field strain maps indicated that there were three general grain boundary behaviors:

- boundaries that showed strain accumulation
- those that exhibited grain boundary sliding
- boundaries that showed no distinct behavior relative to the adjacent grain interiors

Boundaries that showed each type of behavior were extracted for scanning transmission electron microscopy (STEM) analysis. Electron transparent specimens less than ~200nm in thickness were prepared for STEM analysis using an FEI Helios 600 dual-beam focused ion beam (FIB) system via a “lift-out” technique with an Omniprobe micro-manipulator. A platinum cap was deposited onto the sample surface before the membranes, approximately 40 x 15 x 1 μm in size, were milled out of the bulk samples with Ga\(^+\) ions accelerated to 30 kV. Membranes were then plucked and attached to an Omniprobe grid before thinning to less than 300 nm at 30 kV at an incidence angle of \(\leq 2^\circ\), and final thinning to less than 200 nm at 5 kV at an incidence angle of \(\leq 5^\circ\). STEM analysis was conducted on a Phillips Tecnai STEM at 200kV accelerating voltage. Bright field (BF) on zone and two beam conditions were conducted to characterize dominant slip systems.
8.1.4 **Schmid Factor Analysis**

Schmid Factor (SF) analysis was conducted using code developed in MATLAB. First Bunge Euler orientation angles were converted to a rotation matrix, using equation 8-1. This rotation matrix can be used to convert the slip plane and directions orientations from the crystal reference frame to the sample reference frame using equations 8-2. The Schmid Factors for each slip system can be calculated by a series of dot products because to loading direction is along the sample [0 1 0] direction.

\[
g = \begin{bmatrix}
\cos \varphi_1 \cos \varphi_2 & \sin \varphi_1 \cos \varphi_2 \\
-\sin \varphi_1 \sin \varphi_2 \cos \Phi & + \cos \varphi_1 \sin \varphi_2 \cos \Phi & \sin \varphi_2 \sin \Phi \\
-\cos \varphi_1 \sin \varphi_2 & -\sin \varphi_1 \sin \varphi_2 \\
-\sin \varphi_1 \cos \varphi_2 \cos \Phi & + \cos \varphi_1 \cos \varphi_2 \cos \Phi & \cos \varphi_2 \sin \Phi \\
\sin \varphi_1 \sin \Phi & -\cos \varphi_1 \sin \Phi & \cos \Phi
\end{bmatrix}
\]

\[
(8-1)
\]

\[
\mathbf{n}_{\text{sample}} = g^t \mathbf{n}_{\text{xfi}}
\]

\[
SF = (\mathbf{\sigma} \cdot \mathbf{n}_{\text{sample}}) (\mathbf{\sigma} \cdot \mathbf{n}_{\text{direction}})
\]

Where the subscript indicates the appropriate reference frame, and the superscript indicates the type of vector. Beyond just calculating the Schmid Factors it was also of interest to calculate the angle that the slip planes/directions made with either the sample surface or the STEM foil edge. In the case of slip trace analysis with the sample reference frame, this was easy because the EBSD sample reference frame is
the same reference frame so it was easy to calculate angles between planes and the EBSD reference frame. For the STEM foils, the reference frame was now aligned with the two foil edges, one of which was perpendicular to the EBSD sample frame, thus necessitating another set of coordinate rotations to describe the STEM foil reference frame in terms of the EBSD sample frame. Once this was conducted the trace of the slip plane in the foil plane can be calculated by the cross-product of the foil normal and the slip plane normal, equation 8-4, and then equation 8-5 can be used to calculate the angle between the slip plane trace and the other two foil edges so that positive and negative rotations could be assessed.

\[
\begin{align*}
    n_{STEM \ plane} &= \text{FoilNormal}_{sample} \times n_{sample} \\
    \theta &= \cos^{-1}(n_{STEM \ plane} \cdot \text{FoilEdge}_{sample})
\end{align*}
\]

(8-4) (8-5)

8.2 Results

8.2.1 Slip Trace Analysis

Grains of interest were selected based on visible slip traces on the sample surface observed after deformation. In the region analyzed for full field strain mapping this included 14 grains, shown in Figure 8.2.
Figure 8.2. Pre-deformation EBSD scan fit over post deformation SE image for assessing the angle between slip planes and the loading (horizontal) direction.

The angle that the slip plane made with the horizontal axis, assuming positive rotation was counterclockwise (noon is the top of the image), was compared with the predicted active slip systems assuming Schmid Factor analysis. Table 8.1 shows the comparison for all grains analyzed. For each \{111\} slip plane, there can be three possible $\frac{1}{2}<110>$ slip systems. Table 8.1 indicates that the slip plane observed is generally consistent with the first or second mostly highly stressed slip system (slip direction and plane). Also shown are the predicted mechanisms from consideration of the DAD (section 8.1.2), For instance, dissociation into partials will tend to occur when the Schmid Factor for the leading partial is higher than that for the trailing partial.
<table>
<thead>
<tr>
<th>Grain #</th>
<th>Slip Trace Angle</th>
<th>Predicted Slip Trace Angle</th>
<th>Predicted Mechanism</th>
<th>Schmid Factor Rank</th>
</tr>
</thead>
<tbody>
<tr>
<td>33</td>
<td>-56</td>
<td>-54</td>
<td>dissociated</td>
<td>1,2</td>
</tr>
<tr>
<td>47</td>
<td>-75</td>
<td>-77</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>55</td>
<td>67</td>
<td>69</td>
<td>non-dissociated</td>
<td>2</td>
</tr>
<tr>
<td>63</td>
<td>70</td>
<td>70</td>
<td>dissociated</td>
<td>1,2</td>
</tr>
<tr>
<td>109</td>
<td>-57</td>
<td>-56</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>109</td>
<td>65</td>
<td>66</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>110</td>
<td>-76</td>
<td>-77</td>
<td>dissociated</td>
<td>3,4</td>
</tr>
<tr>
<td>151</td>
<td>78</td>
<td>80</td>
<td>non-dissociated</td>
<td>2</td>
</tr>
<tr>
<td>160</td>
<td>-77</td>
<td>-76</td>
<td>dissociated</td>
<td>1,2</td>
</tr>
<tr>
<td>187</td>
<td>74</td>
<td>74</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>210</td>
<td>80</td>
<td>82</td>
<td>dissociated</td>
<td>2</td>
</tr>
<tr>
<td>250</td>
<td>55</td>
<td>56.5</td>
<td>dissociated</td>
<td>1,2</td>
</tr>
<tr>
<td>326</td>
<td>-42</td>
<td>-40</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>352</td>
<td>42</td>
<td>43</td>
<td>dissociated</td>
<td>1</td>
</tr>
<tr>
<td>369</td>
<td>-73</td>
<td>-72</td>
<td>dissociated</td>
<td>1,2</td>
</tr>
</tbody>
</table>

Table 8.1. Comparison of slip trace angle measurements and predicted active slip systems from Schmid Factor analysis.

8.2.2 STEM Analysis

Grains of interest were selected based on full field strain measurements, the full field strain maps for the same area analyzed for slip trace analysis is shown in Figure 8.3. The grains that exhibited visible slip traces are also labeled in Figure 8.3, and in many cases the slip traces produced visible strain accumulation as determined by the full field method. In most cases the strain accumulated at the grain boundaries exceeds the strain localization due to the intra-granular slip observed at slip traces. Grain boundaries of interest were extracted from four locations indicated by black lines with white inward-pointing arrows on each end overlaid the grain boundaries: tensile strain accumulation (left of grain #369), grain boundary sliding (lower left corner of SEM image), shear strain accumulation (left of grain #250), and no strain accumulation, (left...
of grain #352). A fifth grain boundary was extracted from a separate sample, this $\Sigma 3$ twin boundary exhibited both strain accumulation and grain boundary sliding as measured from the out-of-plane displacement measurements conducted on the optical profilometer, Figure 8.4. The optical profilometry measurements indicate that this type of out-of-plane sliding is not unique for this particular $\Sigma 3$ twin boundary, but readily occurs at other $\Sigma 3$ twin boundaries. This boundary is unique because strain accumulation was also measured in conjunction with the out-of-plane sliding.
Figure 8.3. Tensile and shear strain accumulation maps for a standard sample tested at 1100MPa to an average strain of 1.48% using DIC parameters 30 pixel subset and 3 pixel step size.
Figure 8.4. (a) tensile and (b) shear strain accumulation at a $\Sigma 3$ twin boundary as measured by DIC. (c) Vertical displacement measurements from optical profilometry indicate that the parent grains can exhibit sliding out of the imaging surface with respect to the twin. Loading is along the horizontal axis, black boundaries are $\Sigma 3$, gray are random HAGB, and red boundaries are higher order $\Sigma n$ boundaries. The top surface of the STEM foil is indicated by the gray arrow.

The summary of results of the STEM analysis are listed in Table 8.2. In over 80% of the cases, the first or second highest SF system was observed in each extracted grain independent of the observed deformation behavior from full field strain mapping. There was good agreement between the Phase Field predictions and the observed
deformation mechanism, though there was some ambiguity of predicting decorrelated and dissociated partial dislocation motion. Since the Phase Field predictions assumed that the local resolved shear state was only dependent on the macroscopic stress state the more subtle distinction between decorrelation versus dissociation requires more precise knowledge of the magnitude and direction of the applied shear stress, which is beyond the scope of the present Schmid factor analysis in which grain interaction is ignored. Therefore, the distinction between these two deformation modes is not presented in Table 8.2, but it is assumed that if dissociated motion was predicted than decorrelated motion might actually be observed. The in-depth analysis for three grain boundaries, the shear strain accumulated, random HAGB that showed grain boundary sliding, and the twin boundary that exhibited both strain accumulation and grain boundary sliding will be discussed further.

The STEM analysis also provided insight into the 3D structure of the grain boundaries that experienced different deformation behavior. In the case of the boundary that experienced GBS, this boundary was fairly planar in nature and extended almost perpendicular to the sample surface, more than 10 μm below the sample surface. The boundaries that experienced strain accumulation did extend practically perpendicular to the sample surface, but were not very planar, and extended below the sample surface to shallower depths (2-6 μm) before meeting with a sub-surface twin boundary or other high angle grain boundary. In contrast to all the other observations the grain boundary that did not experience GBS or strain accumulation extended below the sample surface at a very shallow angle.
<table>
<thead>
<tr>
<th>Grain #</th>
<th>Slip Trace Angle</th>
<th>Predicted Slip Trace Angle</th>
<th>Predicted Mechanism</th>
<th>Observed Mechanism</th>
<th>Schmid Factor Rank</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strain Accumulation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>32</td>
<td>33</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>1, 2</td>
</tr>
<tr>
<td>2</td>
<td>112</td>
<td>113</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>1</td>
</tr>
<tr>
<td>t2</td>
<td>152</td>
<td>non-dissociated</td>
<td>non-dissociated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shear Strain Accumulation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>35</td>
<td>36</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>1</td>
</tr>
<tr>
<td>1</td>
<td>131</td>
<td>132</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>2</td>
</tr>
<tr>
<td>2</td>
<td>135</td>
<td>139</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>1, 2</td>
</tr>
<tr>
<td>t2</td>
<td>~</td>
<td>43</td>
<td>non-dissociated</td>
<td>non-dissociated</td>
<td>1</td>
</tr>
<tr>
<td>HAGB Grain Boundary Sliding</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>129</td>
<td>132</td>
<td>non-dissociated</td>
<td>non-dissociated</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>126</td>
<td>129</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>1, 2</td>
</tr>
<tr>
<td>2</td>
<td>18</td>
<td>20</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>4</td>
</tr>
<tr>
<td>(\sum_3), Strain Accumulation + Grain Boundary Sliding</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>1</td>
<td>77</td>
<td>76</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>1</td>
</tr>
<tr>
<td>1</td>
<td>52</td>
<td>57</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>2</td>
</tr>
<tr>
<td>2</td>
<td>24</td>
<td>27</td>
<td>decorrelated</td>
<td>decorrelated</td>
<td>2</td>
</tr>
<tr>
<td>No Strain Accumulation</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>60</td>
<td>65</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
<td>40</td>
<td>dissociated</td>
<td>decorrelated</td>
<td>4</td>
</tr>
</tbody>
</table>

Table 8.2. Comparison experimentally observed mechanisms by STEM analysis and predicted active slip systems from SF analysis. Angles are measured from the top foil edge, and very between zero and one hundred and eighty.

In addition to the overall results, the specifics of a few boundaries will be assessed in more detail. First, the analysis of the grain boundary that that exhibited significant shear strain accumulation is presented in Figure 8.5. It can be seen in the full field strain map that strain accumulates at the quad-point between the twins of each grain and the grains themselves. The grain boundary between grain 1 (G1) and grain 2 (G2) is a random high angle grain boundary (HAGB) with a misorientation, described by an axis-angle pair in G1 reference frame, of \([0.64\ 0.1\ 0.76]38.6^\circ\). The STEM
analysis indicated that both G1 and G2 exhibited extended stacking faults as the dominant deformation mechanism, while the twin of G2 exhibited non-dissociated dislocation motion as the primary mechanism. The comparison of the experimental measurements from STEM and DAD model predictions for G1 and G2 are shown in Table 8.2. The discrepancy between the slip plane angles arises from the fact that angles were measured on images of the STEM foils, which could be tilted slightly from the idealized case, and the model predictions were based on EBSD orientation data assuming no rotation of the sample occurred during preparation or when placing the sample in the STEM. G1 had two active slip systems with very similar Schmid factors (SF) of 0.47 and 0.45. A comparison of both systems with the DAD model confirmed that in both cases the observed extended stacking fault mechanism was predicted as dissociated partials by DAD analysis, analysis is shown in Table 8.2. G2 also had two possible active slip systems, both on the same slip plane (111), with SF of 0.41 and 0.34 for \( \frac{1}{2} [-110] \) and \( \frac{1}{2} [-101] \) respectively. The DAD model predictions indicate that \( \frac{1}{2} [-110] \) should decorrelate into partials while the lower SF system \( \frac{1}{2} [-101] \) should only dissociate into partials. The DAD model predicted that the twin of G2 should exhibit non-dissociated dislocation activity (not shown in Figure 8.5c), which was experimentally observed. The STEM analysis indicated that there was increased dislocation content and activation of a second dislocation mechanism in G2 near the grain boundary, as seen in Figure 8.5b. Activation of a secondary mechanism was also observed at another extracted boundary that DIC indicated to have exhibited above average strain accumulation.
Figure 8.5. a) Shear strain accumulation near a grain boundary after 1.46% tensile strain accumulation at 1100 MPa. The top surface of the STEM foil is indicated by the gray arrow. Σ3 are white, Σn are gray, and HAGB’s are black. b) STEM BF image on grain 2 (112) zone indicating the angle between the dominant slip planes and the foil edge. Activation of a second mechanism (full dislocation activity) is seen in grain 2 near the grain boundaries. c) DAD predicted dislocation mechanisms for grain 1 (black) and grain 2 (white).
Similar analysis was conducted on a HAGB that exhibited grain boundary sliding, the misorientation of this boundary as described by an axis-angle pair in grain 1 reference frame is [0.6 0.5 0.62]50.2°. From the sample surface in Figure 8.6a, it can be seen that the boundary trace appears to be mostly planar, with limited serrations. Experimental analysis confirmed DAD model predictions that grain 2 should exhibit decorrelated, extended stacking faults on the slip systems with the first and second highest SF, both of which are on the same slip plane that makes a predicted 129° with the foil edge, Figure 8.6. The fourth highest SF system was also observed in STEM, with predicted angle with the foil edge of 20°. The DAD model predicted that this system should also deform by decorrelated motion, which was observed in STEM images. Grain 1 on the other hand was predicted, and was confirmed, to deform by non-dissociated full dislocation behavior. STEM analysis indicated that near the serrations of the grain boundary some extended stacking faults formed in grain 1. In contrast to the boundaries that exhibited strain accumulation there was very limited activation of this secondary mechanism, leading to a lack of apparent gradient in dislocation content on either side of the grain boundary. The analysis of the slip systems in grain 1 was complicated by the curtaining effect perpendicular to the foil edge due to FIB damage during milling, Figure 8.6.
Figure 8.6 a) Grain boundary exhibiting grain boundary sliding, as seen by the circled discrete offsets in grid markers. The top surface of the STEM foil is indicated by the white arrow. b) STEM BF image on grain 2 (112) zone showing that the dominant dislocation mechanism in grain 2, extended faults, shows minimal breakdown near the grain boundary. c) DAD predicted dislocation mechanisms for grain 1 (black) and grain 2 (white).
The twin boundary in Figure 8.4 was of interest because of the coupled measurement of both strain accumulation and grain boundary sliding. Previous work on high cycle fatigue in René88DT indicates that slip deformation parallel to the coherent interface of the annealing twin is associated with fatigue crack initiation in very large grains[92]. So even though the testing condition of this study is very different, creep verses fatigue, it is interesting to note that the annealing twins are not immune to localized deformation and much be considered if a complete model is developed. Schmid Factor analysis confirms that the highest Schmid Factor systems in both the grain (grain 1) and the twin (grain 2) is on a {111} plane parallel to the coherent twin boundary. The DAD model predicts that deformation on these highest Schmid Factor systems in grain 1 and grain 2 should be dissociated and non-dissociated motion respectively. The STEM analysis confirms that these predicted mechanisms are active. The STEM analysis also indicates that both grains deforms by extended stacking fault mechanisms on the slip systems with the second highest Schmid Factors, as seen in Figure 8.7. The DAD model also predicts that these are the expected deformation mechanisms for these slip systems. The deformation on the slip systems parallel to the coherent twin can be seen nucleating from a portion of the incoherent boundary, and it is conceivable that this mechanism could be the cause of the out-of-plane grain sliding seen in the optical profilometer measurements in Figure 8.4 c. A more magnified section of the deformation close to coherent annealing twin boundary indicates that deformation on the {111} boundary parallel to the coherent boundary appear to have constrained additional dislocation content on a different slip
plane, such that there is increased deformation in very close proximity to the grain boundary. Burger’s vector analysis from tilting experiments confirms that the dislocation content between the coherent twin boundary and the deformation twin boundaries is indeed on another slip plane, as seen in Figure 8.8.

Figure 8.7 (a) STEM brightfield image taken on 112 zone for both grains, showing the activation of dominate dislocation mechanisms in the grain interiors, and the activation of a second mechanism in grain 1 parallel to the coherent twin boundary. (b) the Phase Field model confirms that the observed extended stacking fault mechanism is the predicted mechanism.
Figure 8.8. Bright Field STEM images taken under various two beam conditions off the 101 zone (three on left) and the 2-11 zone (three on right).
8.3 Discussion

Two evaluations were of interest for this work, and they will be assessed below. First, do the Schmid laws apply to predicting the active slip systems during the deformation of surface grains at elevated temperature. Second, do the Phase Field (DAD) predictions of deformation mechanisms agree with the observed dislocation mechanisms.

In all cases, except two, analyzed either by slip trace analysis or STEM analysis, the Schmid Factor (SF) analysis, assuming the local stress state was equivalent to the macroscopic stress state, could be used to predict the dominant active slip system for the surface grains analyzed. In the cases indicated in Table 8.2, it was often the case that the first and second highest SF systems existed on the same slip plane. In the case of grain #110, the slip plane with the third/fourth highest SF was observed in slip trace analysis; because these occur on the same slip plane it was not possible to assess which system was active without conducting more detailed STEM analysis. The 1st and 2nd highest SF systems of grain #110 were on the same slip plane. When the slip systems for the grains adjacent to grain #110, are assessed, it can be seen in Figure 8.9, that the slip planes in grain #110 with the 3rd and 4th highest SF are most aligned with likely slip systems active in both the adjacent grains. This indicates that grain neighborhood can play a role in the selection of a particular slip plane in a grain. It would be expected that if additional grains were analyzed that there would be more
than one instance in which the highest order SF system was not initiated due to preferential alignment with slip systems in adjacent grains.

Figure 8.9. Slip trace lines overlaid for grains near grain #110. The black lines indicate measured slip traces. The gray lines indicate most probably slip systems calculated from SF analysis. It can be seen that the observed slip trace in grain #110, is most aligned with its neighbors, both observed and predicted.

The sliding twin boundary provides interesting insight into the possible nature of the grain boundary sliding mechanism observed in these materials. It was observed that the second highest SF systems were the dominant intragranular deformation mechanisms in both grains. The grain boundary sliding mechanism that is mostly likely active in this temperature range relies on the activation of intragranular deformation to accommodate deformation along the sliding boundary. Therefore, slip transfer across
the grain boundary should account for the dislocation content needed for grain boundary sliding. There are a couple of possible vector combinations that can be considered to see if the slip transfer mechanism might be active: (1) slip transfer from grain 2 to grain 1 \((b_2 + \mathbf{b}_{\text{gb}} = b_1)\) which is the reverse sense if (2) slip transfer from grain 1 to grain 2 without lose of generality or if (3) slip is accumulating/emitting on both sides of the grain boundary \((b_2 + b_1 = b_{\text{gb}})\). The vector math suggest that if case three is active, such that the second highest Schmied Factor systems are accumulating/emitting from the grain boundary, the additional dislocation content \(b_{\text{gb}}\) is not only contained in the grain boundary plane, but is also parallel to the grain boundary sliding direction. This would indicate that the additional dislocation content needed to relieve the slip accumulation, or cause slip to emit from the grain boundary, would reside in the grain boundary plane and could cause grain boundary sliding along the coherent twin plane. Analysis of the dislocation content in the grain boundary, through tilting experiments, concluded that there is indeed dislocation content in the grain boundary with Burger’s vectors parallel to the direction of sliding.

**8.4 Conclusion**

For the prediction of dislocation mechanisms for grains on the surface of the sample, SF analysis appears to correlate with those observed in most cases. It was not always observed that the highest Schmied Factor system was activated but in all cases the observed slip plane was that of slip systems with a Schmied Factor above 0.35. This indicates that instituting a SF cut-off for any prediction analysis might be appropriate.
The STEM analysis confirmed that in most cases the predicted mechanisms from Phase Field simulations were the observed dislocation mechanisms in STEM analysis. In the contrary cases, the predicted mechanism was dissociated dislocation motion but the observed mechanism was decorrelated dislocation motion. This discrepancy can be accounted for if the actual stress state in the grain interior was less than the resolved shear stress calculated from the macroscopic stress state, or if the orientation of the stress state was rotated in the positive sense a few degrees.

The STEM analysis of the sliding twin boundary provided some interesting results. Though the primary intragranular mechanisms in each grain were on slip planes that were not parallel to the coherent interface, a secondary mechanism near the grain boundary was observed. The emission of faults parallel to the coherent interface, at steps in the incoherent interface, can trap additional dislocation content near the grain boundary. How this mechanism contributes to grain boundary sliding is still unknown, but it does reiterate that $\Sigma 3$ boundaries are not inert during the deformation process. Studies into the fatigue properties of these materials by Miao et al. and unpublished work by P.J. Phillips has concluded that similar localized deformation at $\Sigma 3$ boundaries are important active deformation mechanisms [92]. This work indicates that $\Sigma 3$ boundaries contribute an important deformation mechanism during creep loading conditions, indicating that any useful physics based model will have to account for the deformation mechanisms near $\Sigma 3$ boundaries in addition to the random high angle grain boundaries.
An examination of the grain boundary structure in the extracted grain boundaries for STEM analysis suggest that differences in grain boundary structure and local sub-surface neighborhood might play a pivotal role in dictating which grain boundaries experience different deformation phenomena. This conclusion needs to be further addressed and confirmed through additional boundary extractions or the creation of the 3D microstructures – examples of which are described in Chapter 9.
Chapter 9: Site specific 3-D microstructural analysis of select grain boundaries

The deformation behavior of grain boundaries is a function of the misorientation, the orientation of the grain boundary with respect to the loading direction, and the surface structure of the grain boundary. These last two parameters are difficult to characterize with scanning electron microscopy (SEM) or transmission electron microscopy (TEM) because these techniques only provide a single projection of the grain boundary structure. Therefore, assessing the 3-D structure of grain boundaries necessitates the creation of 3-D microstructures through serial sectioning techniques. Full field strain mapping and grain boundary sliding (GBS) measurements from discrete offsets in grid markers indicate that some grain boundaries experience strain accumulation while other grain boundaries experience GBS (Chapter 6). No conclusive evidence could be found that either of these types of behaviors could be predicted based on grain boundary misorientation or the orientation of boundary projections in SEM images with respect to the tensile axis (Chapter 7). This leads to the hypothesis that grain boundary structure or sub-surface grain neighborhood plays a pivotal role in dictating grain boundary properties. In this study, two different high
angle grain boundaries were characterized by focused ion beam: one exhibiting grain boundary sliding, and the other exhibiting strain accumulation.

9.1 Experimental

The 3-D microstructure was investigated through serial sectioning using an FEI Nova 600 SEM-FIB. Volumes of material were extracted and each placed on an molybdenum OmniProbe grid using an OmniProbe in-situ micromanipulation system in a manner similar to thin foil preparation, Figure 9.1 shows a volume of material prepared for lift-out of the bulk sample. First a platinum cap was deposited over the area of interest, and then trenches were milled along three faces of the volume. Once trenches were cut, the sample was under-cut on two sides to create suspended beam of material that was only connected along once side. The volume was attached to the OmniProbe in-situ micromanipulation needle using platinum and before severing the samples concenction to the bulk material. Next, an molybdenum OmniProbe Grid, seen in the top right insert in Figure 9.2 was silver painted to an SEM stub and inserted flat into the SEM-FIB chamber such that when imaged with the electron source, the grid looked like what is presented in insert in Figure 9.2. Molybdenum grid was used because it mills more slowly when subjected to the FIB than conventional copper grids. The sample was than welded to the tip of the “B” finger of the grid using platinum.
A custom script utilizing FEI Runscript software was used to automate the serial sectioning process, and consisted of cross-section milling, collection of ion-induced secondary electron (ISE) images, and repositioning of the stage between the two conditions [93]. The beam settings for each step were 30kV and 2.8 nanoAmps and 0.47 picoAmps respectively. The sample, with OmniProbe grid still silver painted to the SEM stub was mounted on a 45° tilted specimen holder, so that the sample could be tilted to collect ISE images of both the milling and cross-section planes, as illustrated in Figure 9.2. A set of fiducial marks, FIB milled circles, in both the milling and imaging planes were used for alignment purposes. ISE images were used for this study because the thinning process induced substantial SE contrast between the γ and γ′
phases while not providing adequate contrast between grains, making impossible to conduct automated segmentation of the grain boundary.

Figure 9.2. Schematic showing sample rotation during image collection, first the sample is oriented to cut the free edge, and then the sample is rotated 180° to image the cut plane. Also shown is the placement of the OmniProbe grid, as viewed from the electron source, used for welding the extracted sample.

Two grain boundary volumes were extracted for analysis, in both cases the section thickness was approximately 50 nm. The 406 μm³ volume containing a boundary that exhibited grain boundary sliding was reconstructed from 116 slices with
each image having in-plane resolution of 223 pixels/μm. The volume containing the
grain boundary that exhibited strain accumulation was approximately 2080 μm$^3$ in size
and was reconstructed from 110 slices with each image having in-plane resolution of
24 pixels/μm.

The larger volume contained several grain boundaries of interest for
reconstruction. In this case when the cut plane was perpendicular to the ion-beam,
images did not provide adequate contrast between adjacent grains. Therefore, images
instead were acquired at an angle of -16° from perpendicular to the ion. These images
were pre-processed to remove the tilt distortion, due to the exceptional depth of focus
there is no distortion in the horizontal dimension, so only a magnification correction in
the vertical direction was applied 1/cos(θ).

Each volume required slightly different processing for automated boundary
identification. The GBS sliding volume was processed using the following algorithms.
Since the alignment feature in the FEI run script is only requiring alignment within 3
pixels, an additional alignment using digital image correlation was conducted before
applying a levels adjustment for contrast enhancement, and a standard deviation filter
was applied where every pixel was re-labeled with the standard deviation of its 11x11
pixel neighborhood. This served to highlight pixels near the boundary, as their standard
deviation (i.e. new pixel-value) would be considerably higher than pixels in the grain
interiors, Figure 9.3b. Next, binary image segmentation was done by applying a global
threshold to highlight the grain boundaries, Figure 9.3c. In some slices the boundary
was artificially discontinuous, and watershed segmentation was employed to fill
most of these discontinuities. Pixel clusters not belonging to the boundary were removed by rejecting any objects below a size of 100 pixels. Any remaining pixel clusters were manually removed and remaining discontinuous boundary segments were manually connected, Figure 9.3d. This algorithm was applied to each slice and resulting binary images were stacked to produce a 3-D volume of non-cubic voxels.

At this point, the grain boundary network was represented as thin lines (1-2 pixel thick) separating the grain interiors. Although the boundary was fully continuous within each slice (i.e. the imaging plane), the boundary was not continuous in the planes perpendicular to the imaging plane and therefore, the grains could not be interpreted as separate objects. To create a continuous 3-D boundary that fully separated the grains, a 3-D watershed algorithm was applied. A closed surface was constructed from the voxelized boundaries and this surface was smoothed to facilitate visualization.

In addition to the sliding grain boundary, carbides and deformation twins were also segmented and visualized using algorithms similar to those employed on the grain boundary network. The different segmentations were merged and visualized to create a composite dataset where all microstructural features and their interactions could be analyzed.
Figure 9.3. Segmentation process from the (a) as received image by (b) applying a standard deviation filter (c) boundary identification with a global threshold and (d) final grain boundary trace after water-shed and removal of spurious data points.

The strain accumulation boundary volume proved to be more difficult to process for automated boundary identification. The standard deviation filter was not effective for this data set because of the inherent contrast variation between the $\gamma'$ particles and the matrix phase as seen in Figure 9.4a. Instead after images were further aligned using digital image correlation two different techniques were employed; either a global threshold method or a region grow feature. The region grow feature was used for grains of similar intensity contrast as the global threshold method would not work. For the darkest and lightest intensity grains in Figure 9.4a, a global intensity threshold filter was applied, Figure 9.4b. Next a series of dilation and erosion steps were applied to fill in holes in the grain interior and feature size threshold was applied to remove spurious data points in other grains, Figure 9.4c. Then the boundary was manually inspected and altered to confirm the an accurate boundary trace, Figure 9.4d. For the
grains of similar contrast, a grow region approach was used. In this technique a pixel in
the grain interior was selected and the area surrounding that pixel was iteratively
expanded to encompass all contacting pixels of similar intensity within a
predetermined threshold. The area stops expanding around the selected point when
99% of the pixels selected have intensity values beyond the threshold of the original
pixel. Once an area was selected, a series of dilation and erosion steps were applied to
fill in holes in the grain interior caused by contrast variations of the γ′ particles, and the
boundary was manually inspected to confirm the boundary trace. A voxelized surface
was fit and smoothed to facilitate visualization.

Figure 9.4 (a) as received image (b) after applying a global intensity threshold (c)
removing spurious data points with a size threshold and (d) inspected final grain area.

The individual grains and their boundaries were then visualized using the
commercial software Avizo developed by Visualization Sciences Group. In addition to
visualizing the data in Avizo, Matlab codes were used to analyze macroscopic surface
curvature, and the nano-scale grain boundary serrations due to the presence of $\gamma'$ and carbide particles located on the grain boundary surface.

9.2 Results

9.2.1 Grain Boundary Sliding Volume

The orientations of each grain in the volume were measured with EBSD analysis. The grain boundary misorientation was calculated with respect to grain 1 coordinate system as $54.6^\circ[17,9,23]$. Brandon’s criteria for acceptable deviations in misorientation axis and angular deviations in rotation assuming $K=15$ and $n=\frac{1}{2}$ indicate that is boundary is a $\Sigma 11$ coincidence site lattice grain boundary which has a angle-axis pair of $50.47^\circ[1,0,1]$[82].

Figure 9.5 is a rendering of the surface of the reconstructed grain boundary that experienced GBS. Also shown are deformation twins which emanate from intragranular carbides. Since these structures emanate from the carbides, and are very narrow, the hypothesis is that these are deformation twins and not annealing twins. The very large twin in the lower right corner of Figure 9.5 is not consistent with the other twins in the structure, it is significantly larger than the other deformation twins in all dimensions, and it terminates not only at intragranular carbides but also at a subsurface grain boundary, that is not visualized in any of the figures. The dimensions and the termination points of this twin may indicate that this structure is not a deformation twin but may indeed be an annealing twin.
Figure 9.5. Grain boundary that exhibited GBS, grain 1 is presented as purple and grain 2 is transparent. The deformation twins (yellow) emanate from intragranular carbides (white) and terminate at the grain boundary. The slicing/sliding direction is presented as a white arrow, and the total dimensions of the bounded volumes are 6.7 μm along the slicing direction and by 7.8 and 5.6 μm in the image plane.

Figure 9.5 shows that this grain boundary plane contains both macro-scale curvature, which is dictated by the grain boundary triple lines, and micro-scale tortuosity due to the γ′ structure. To analyze the micro-scale tortuosity of the grain boundary, a plane was fit to the macro-scale curvature of the grain boundary with an $R^2$ value of 0.98. A comparison of the fit plane, in the same coordinate system as the EBSD analysis indicates that the axis of rotation for this $\Sigma 11$ grain boundary creates a
53° angle with the grain boundary normal, so that plane of coincidence \( \overline{\mathbf{T}} \ 0 \ \overline{T} \) is not the grain boundary plane. This indicates that the atomic structure of this grain boundary should not be atomically flat like the coherent \( \Sigma 3 \) twin boundaries, which is consistent with the observations from Figure 9.5. The micro-scale topography was calculated as the difference between each data point and the surface fit. In Figure 9.6a, the topological map is overlaid on the original grain boundary surface, and shows the degree to which the local boundary varies from an ideal surface. Most regions vary between \( \pm 150 \text{nm} \), with maximum and minimum values of \( +250 \text{ nm} \) and \(-450 \text{ nm}\). A comparison of Figure 9.6a with Figure 9.5 shows that the deepest trenches are due to the intergranular carbides and the raised region in the lower right corner of the boundary occurs because the boundary is being pinned by a third grain (not visualized in any of the figures). For comparison, the fit plane can be overlaid with the micro-scale topography, as seen in Figure 9.6b. From this figure it can be seen that the topography varies greatly in the direction perpendicular to the slicing (sliding) direction, while the topography is fairly consistent along channels parallel to the grain boundary sliding direction. Also consistent with the observations from STEM foils, these volumes was free of any additional sub-surface grain boundary structure, assuming of course that the observed twins are caused by deformation and were not present prior to deformation.
Figure 9.6 (a) Topographical color scheme showing variations in surface height based off a fit plane, and the (b) topographical color scheme overlaid with the fit plane showing connectivity of ridges along the sliding/slicing direction (presented as a white arrow). The total dimensions of the volume are 6.7 μm along the slicing direction and 7.8 by 5.6 μm in the image plane.

9.2.2 Strain Accumulation (Non-Slapping) Boundary Volume

The volume containing the grain boundary that exhibited strain accumulation was extracted from the location shown in the strain mapped region of Figure 9.7a, with the volume location indicated by a white translucent box. The grain boundary network in this region was reconstructed and is seen in Figure 9.7b. Also presented in Figure 9.7b are the grayscale images of the image plane, along the back of the reconstruction,
and the composite grayscale image created from the slicing plane. It can be seen from the top surface that there is excellent alignment between the slicing images as the twin boundary is very planar.

Figure 9.7 (a). Region of a grain boundary that experienced strain accumulation shown as the box overlaid the full field strain map. Random HAGB are black and $\Sigma$3 twin boundaries are red. (b) Serial sectioning data visualized to show the network of grain boundaries in the volume. The slicing direction is presented as a white arrow in both images, the volume analyzed was 5.5 $\mu$m along the slicing direction and 14.8 by 14.7 $\mu$m in the image plane.
EBSD analysis prior to deformation demonstrates that, except for the $\Sigma 3$ boundaries between grain 1 (G1) and its twin (TG1) and grain 2 (G2) and its twin (TG2), all of the grain boundaries were random high angle grain boundaries with misorientation as follows: between G1-G2 is $45^\circ\left\langle 22 \ 7 \ 2 \right\rangle$, TG1-G2 is $43.5^\circ\left\langle 4 \ 29 \ 0 \right\rangle$, and G1-TG2 is $28^\circ\left\langle 2 \ 7 \ 21 \right\rangle$ where in all cases the axis of rotation is described in the reference to the orientation of the grain listed first in the sequence. It can be seen in the reconstruction that even though the EBSD analysis prior to deformation, shown in Figure 9.7a, indicates that G1 and G2 are in contact with each other, this is not actually the case since G1 and TG2 are in direction contact. Since the step size in the EBSD analysis was 1 $\mu$m, and the twin boundary between G2 and TG2 is very shallow, the analysis could very easily have mis-identified the boundary trace on the sample surface between TG2 and G2 in the region of interest.

The micro-scale topography of the grain boundary between G1 and TG2 was analyzed by fitting a $2^{nd}$ order polynomial to the grain boundary surface. Though visually this was a good fit the $R^2$ value was only 0.69, indicating that the micro-scale tortuosity leads to substantial differences between the estimated plane and the actual data points. Figure 9.8 shows the measured micro-scale grain boundary topography, it can be seen that the peaks and valleys in this grain are on the order of $\pm 2 \mu$m.
Figure 9.8. Grain boundary topography overlaid the original grain boundary surface between TG2 and G1. (a) at the same specimen orientation presented in Figure 9.7, and (b) with the boundary was rotated for a better view of the surface topography.

9.3 Discussion

Automated grain boundary segmentation was a trial and error process, in many cases the steps and settings that worked for segmentation of one grain boundary would not work for another grain boundary. The most difficult grains to segment were those for which the $\gamma'$ particles had slightly different intensity than the grain interiors. Even though automated segmentation was conducted, every slice was manually inspected to insure proper grain boundary structure was identified.
The data from the volume containing the grain boundary that experienced grain boundary sliding showed that along the general direction of GBS there is connectivity of ridges and valleys. It is unclear if this effect allowed for easy sliding or if this structure was created by the process of grain boundary sliding. This connectivity occurs even in the lower right corner of the data set, where this grain boundary becomes pinned by a third grain so it is hypothesized that this structure existed prior to deformation. Since the deliberate creation of grain boundary serrations on the order of tens of μm limits GBS in these materials, as seen in Chapter 5, it is expected that pre-existing nm scale serrations might also affect the propensity of GBS. Additional work is necessary on grain boundaries both prior to and post deformation to conclusively determine if this structure is important to facilitating GBS. If it is assumed that the serrations in both grain boundaries were present prior to deformation, it can be seen that there are substantial differences in grain boundary topography. When presented using the same topography legend, Figure 9.9, it can be seen that the inherent grain boundary serrations in the grain boundary that experienced strain accumulation is an order of magnitude greater than the boundary that experienced grain boundary sliding. These results are consistent with measurements of grain boundary tortuosity measured from STEM foils.
The other striking difference between the two grain boundary structures was the relative simplicity of the grain boundary that experienced GBS versus the complexity of the grain boundary neighborhood of the grain boundary that experienced strain accumulation. The grain boundary that experienced strain accumulation had several other sub-surface grain boundaries in close proximity (≤ 6 μm), while the boundary that experienced GBS was free of sub-surface grain boundaries (≥ 8.5 μm). These results are consistent with observations from STEM foils and areas FIB cross-sectioned for boundary observations, and with observations from the full field strain maps that indicate that strain localization sites readily occur at grain boundary triple points.
9.4 Conclusions

The use of a dual-beam FIB for 3-D serial sectioning proved to be a valuable tool for exploring grain boundary sub-surface structure. A technique was developed to characterize both macro-scale grain boundary curvature and micro-scale grain boundary tortuosity. The analysis indicated that a grain boundary exhibiting grain boundary sliding was planar on the macro-scale and had topography on the order of the secondary γ’ particle size. The grain boundary that experience strain accumulation was a component of a very complex local network of sub-surface grain boundaries. The toruosity of this grain boundary was an order of magnitude greater than that measured for the boundary that experienced GBS.

Strain accumulation occurs readily at \( \Sigma 3 \) annealing twins and random high angle grain boundaries early in the deformation of René 104. The serial sectioning reconstructions indicate that localization of strain at these random high angle grain boundaries might be associated with the proximity of the sub-surface grain boundaries particularly the presence of \( \Sigma 3 \) annealing twins. The reconstructed data also seems to confirm that grain boundary tortuosity plays a role in the likelihood of grain boundaries to experience grain boundary sliding. More observations of both types of grain boundaries are needed to provide further support of these hypotheses.
Chapter 10: Conclusions and Future Work

10.1 Mechanistic Insights

The full field deformation maps confirm that purposefully manipulating the grain boundary structure indeed changes the mechanisms by which strain localizes at grain boundaries in these materials. Introducing micron-scale serrations into the grain boundary structure of the high angle grain boundaries (HAGB) decreases the propensity of all grain boundaries to experience grain boundary sliding (GBS), though it appears to be preferentially more effective to limiting GBS at the HAGB. The limitation of sliding along the $\Sigma 3$ coherent twin boundaries is presumably a secondary effect due to the creation of micron-scale serrations on adjacent boundaries, since the serration mechanism does not occur on the $\Sigma 3$ twin boundaries. Since the network of HAGB’s are less likely to experience GBS the $\Sigma 3$ twin boundaries are likely to be more constricted to slide because the surrounding structure can not accommodate the mechanism.

The full field strain maps provide an opportunity to assess how strain develops as a function of creep strain. The maps indicate that these two microstructures accumulate deformation differently. The serrated microstructure more readily deforms
on localized slip regions at approximately $45^\circ$ angles to the tensile direction independent of the underlying microstructure. For the standard microstructure, deformation localizes initially at grain boundaries roughly orientated at $45^\circ$ angles to the tensile axis, and these localization sites act to nucleate deformation throughout the microstructure. The use of the slip transmission parameters (STP’s) indicates the need for larger sampling sets, but provides little insight into why some grain boundaries show strain localization while others do not. This could indicate that the correct STP has not been determined for this material system, or it could indicate that there is an inherent limitation to the full field strain maps because the 3D microstructure is not being considered. Scanning transmission electron microscopy (STEM) and slip trace analysis indicate that there is some validity to using Schmid Factors for predicting intra-granular deformation in surface grains, but additional work is needed on sub-surface grains to conclude if this a universal agreement. The STEM analysis also indicates that the prediction of deformation mechanisms (i.e. predict full dislocation motion or partial dislocation motion in these materials) can be made based on Schmid factor analysis. Considering the results of both the STEM analysis, and the serial sectioning analysis, there appears to be an inherent difference in the complexity of the sub-surface grain boundary structure of grain boundaries that exhibit GBS and strain localization.
10.2 Experimental Techniques

Understanding deformation at grain boundaries requires a multi-level experimental approach due to the statistical likelihood of observing an occurrence and the inherent localized nature of the events. Observations indicate that full field strain mapping can be used to gauge the statistical nature of strain localization events, and probably even failure initiation events, but has limitations in measuring the very discrete nature of grain boundary sliding events. To measure grain boundary sliding, it appears measurement of offsets in grid lines is still the only viable method of measurement, though optical profilometry measurements show promise for measuring out of plane displacements above 100 nm.

Digital image correlation (DIC) from scanning electron microscope images can be used for measurements of local strain, but care must be taken to insure that no image distortions are causing erroneous conclusions. A limitation to the DIC approach is that only surface strains and microstructures are measured. Advancement on this approach would be to conduct DIC on coupled x-ray scattering in-situ experiments, so that 3D strain fields and microstructures can be assessed. Another option would be to conduct DIC on micro-scale samples that could be serial sectioned, post deformation, so that 3D grain structure and final orientations could be collected. Without information concerning the 3D microstructure, only a statistical understanding of mechanisms can be determined from the DIC techniques.
The *in-situ* testing technique has proven to provide repeatable results for room temperature tensile tests, but issues with obtaining reliable temperature control make it difficult to obtain consistent strain rate measurement from elevated temperature tests. Additional work is needed to either improve specimen heating, or create an *ex-situ* approach to correlate bulk strain rates with localized deformation mechanisms. An *ex-situ* approach would necessitate the development of tensile grips for pre-existing inert atmosphere or vacuum creep frames so that flat dog-bone specimens could be used for creep testing. The flat specimens are necessary for using pre-existing electron beam lithography equipment which was developed for patterning of silicon wafers. The primary difficulty of developing an *ex-situ* approach would be the development of a method for aligning the specimen in the SEM for imaging prior and post deformation. To solve this problem some sort of alignment fixture would be needed and a reference region that does not experience deformation (somewhere in the grip region for example) would be needed to remove rigid body motions between images.
Bibliography


Appendix A: Slip transmission parameters

Slip transmission parameters calculated using all of the hot and not hot boundaries.
Slip transmission parameters calculated using all of the hot boundaries segmented by the angle the grain boundary trace made with the tensile axis. These histograms show that there was no dependency of calculated STP values on the grain boundary orientation with respect to the tensile axis, indicating that this might not be a useful segmentation technique.
Slip transmission parameters for all hot boundaries compared with the three sub-sets of the not hot boundary. Three not hot sub-sets were created by randomly selecting a number of the not boundaries, equivalent to the number of analyzed hot boundaries.
Distributions for the $S_4$ parameter as a function of grain boundary orientation with respect to the tensile axis.