Microstructural Deformation and Failure of an Al-Mg Alloy with a Bimodal Grain Size Distribution

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Microstructural Deformation and Failure of an Al-Mg Alloy with a
Bimodal Grain Size Distribution

Andrew C. Magee, Ph.D.
University of Connecticut, 2014

Grain size reduction has been known as a strengthening mechanism in most metals. The improvements in strength are at the cost of ductility in Al 5083, so a microstructure with a bimodal grain size distribution consisting of coarse grained (CG) and ultrafine grained (UFG) phases was developed. This creates a complex, inhomogeneous microstructure that can be difficult to predict and analyze. In this work this material is studied through a combination of experimental work and finite element simulations.

A full-factorial experimental design is developed for tensile tests under a variety of experimental conditions using a custom developed small scale specimen design. These tests followed by microstructural analysis examine the effects of temperature, anisotropy, strain rate, and CG ratio on the elastic-plastic constitutive behavior and failure of the material. Temperature is found to modulate many of the observed phenomena. A major finding is that while the UFG material exhibits significantly improved strength at room temperature, its strength quickly degrades with increasing temperature. Eventually, around 493 K, its refined grain size becomes detrimental to its strength. A proposed explanation for this is the activation of grain boundary mediated plasticity effects such as grain boundary sliding. Additionally, changes in fracture texture are noted at different temperatures and between loading orientations.
To further investigate some of these findings, a multiscale simulation approach is developed. These simulations study the deformation and failure of the material at a microstructural level, incorporating crystal plasticity and grain boundary modeling techniques in procedurally generated finite element models to represent emergent effects at the grain level. The models are used to extract from the experimental data the appropriate crystal plasticity material constants for both the UFG and CG phases at two temperatures. These models showed crack initiation at the CG/UFG interface with lateral crack propagation through the matrix. At higher temperatures, these sites moved into the UFG matrix. Grain boundary activity can be quantified through these techniques and the simulations show that grain boundary sliding becomes more active at higher temperatures, while grain rotation is predominant at lower temperatures.
Microstructural Deformation and Failure of an Al-Mg Alloy with a Bimodal Grain Size Distribution

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M.S., The University of Alabama, 2012
B.S., The University of Alabama, 2011

A Dissertation

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Doctor of Philosophy Dissertation

Microstructural Deformation and Failure of an Al-Mg Alloy with a Bimodal Grain Size Distribution

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University of Connecticut
2014
## Abbreviations and Symbols

### Abbreviations:

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>CG</td>
<td>Coarse grained</td>
</tr>
<tr>
<td>CIP</td>
<td>Cold isostatic pressing</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron backscatter diffraction</td>
</tr>
<tr>
<td>ECAP</td>
<td>Equal channel angular pressing</td>
</tr>
<tr>
<td>EDM</td>
<td>Electric discharge machining</td>
</tr>
<tr>
<td>FEA</td>
<td>Finite element analysis</td>
</tr>
<tr>
<td>GB</td>
<td>Grain boundary</td>
</tr>
<tr>
<td>GBS</td>
<td>Grain boundary sliding</td>
</tr>
<tr>
<td>GS</td>
<td>Grain scale</td>
</tr>
<tr>
<td>HIP</td>
<td>Hot isostatic pressing</td>
</tr>
<tr>
<td>LS</td>
<td>Large scale</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron microscope</td>
</tr>
<tr>
<td>UFG</td>
<td>Ultrafine grained</td>
</tr>
</tbody>
</table>
Symbols:

Scalar values are represented in italics such as $E$. First order tensors or vectors are represented as bold lowercase letters such as $\mathbf{m}$. Second order tensors are written as uppercase bold letters such as $\mathbf{F}$. Fourth order tensors are set in an open face font such as $\mathbb{C}$.

- $a$: Interfacial strength coefficient
- $b$: Burgers vector
- $C$: Elasticity tensor
- $c$: Ratio of dislocation segment length to grain size
- $d$: Grain size
- $E$: Young’s modulus
- $F$: Deformation gradient
- $\mathbf{F}^e$: Elastic component of deformation gradient
- $\mathbf{F}^p$: Plastic component of deformation gradient
- $\mathbf{F}^{\dot{p}}$: Time increment of plastic deformation gradient
- $G$: Shear modulus
- $g$: Grain boundary thickness
- $g_c^\alpha$: Total slip resistance of system $\alpha$
- $\dot{g}_c^\alpha$: Time increment of total slip resistance of system $\alpha$
- $g_{c,0}$: Initial slip system strength
- $g_c^\alpha$: Saturation strength of slip system $\alpha$
- $g_{c,s_0}$: Material parameter used in calculation of $g_c^\alpha$
- $H$: Hardening matrix
- $h_0$: Initial slip system hardening rate
- $I$: Second order identity tensor
- $K$: Interfacial elastic stiffness matrix
- $k$: Boltzmann constant, Hall-Petch material constant
- $K_N$: Interfacial normal elastic stiffness
- $K_T$: Interfacial tangential elastic stiffness
Dislocation segment length $l$

Plastic slip velocity gradient $\mathbf{L}_p$

Direction of slip plane $\alpha$ $\mathbf{m}^\alpha$

Total number of slip systems $N$

Interfacial strain hardening exponent $n$

Number of grains per unit length $n_l$

Normal of slip plane $\alpha$ $\mathbf{n}^\alpha$

Shape constant for glide resistance profile $p$

Shape constant for glide resistance profile $q$

Second Piola-Kirchoff stress tensor $\mathbf{S}$

Grain boundary yield strength $s_0$

Grain boundary normal yield strength $s_N$

Grain boundary tangential yield strength $s_T$

Interfacial traction $\mathbf{t}$

Melting temperature $T_m$

Interfacial normal traction $\mathbf{t}_N$

Interfacial tangential traction $\mathbf{t}_T$

Current slip system $\alpha$

Tangential interface displacement coupling parameter $\beta$

Slip rate on system $\alpha$ $\dot{\gamma}^\alpha$

Reference strain rate $\dot{\gamma}_0$

Material parameter used in calculation of $\gamma^\alpha$ $\dot{\gamma}_{so}$

Activation free energy $\Delta F$

Total interfacial displacement $\delta$

Elastic interfacial displacement $\delta_e$

Plastic interfacial displacement $\delta_p$

Total plastic interface displacement $\delta_p$

Plastic displacement at interface failure $\delta_{p,\text{fail}}$
$\varepsilon$  Strain  
$\varepsilon_c$  Characteristic strain  
$\varepsilon_{gbs}$  Strain attributable to GBS  
$\varepsilon_p$  Plastic strain  
$\varepsilon_t$  Total strain  
$\theta$  Temperature  
$\mu$  Interfacial friction coefficient  
$\zeta$  Contribution of GBS to total strain  
$\sigma_o$  Hall-Petch material constant  
$\sigma_f$  Flow stress  
$\sigma_s$  Saturation stress  
$\sigma_y$  Yield stress  
$\bar{\tau}$  Magnitude of interfacial traction  
$\tau^\alpha$  Resolved shear stress on slip system $\alpha$  
$\Phi_N$  Interfacial yield surface in normal direction  
$\Phi_T$  Interfacial yield surface in tangential direction
ACKNOWLEDGMENTS

I would like to thank everyone who has helped me along the way, including my lab mates, friends, and teachers who are too numerous to name here. I would especially like to thank my advisor Dr. Ladani and my committee, my family, and Jess for their support and encouragement. This work has been supported by funding from CMMI-1416682 and CMMI-1053434 as well as by the NSF under grant number 1053434.
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CHAPTER 1
INTRODUCTION

1.1 Background

Aluminum and its alloys play a very important role in the modern world. It is the most abundant metallic element in the earth’s crust, but it has not been commonly used in its metallic form until relatively recently. Due to its high reactivity it is very rarely found in nature as a pure metal. Until the advent of improved smelting processes in the late 19th century that allowed for large scale production, the metal was considered rare and valuable. Since then, it has become ubiquitous in the modern world, used in a wide variety of applications ranging from aerospace and marine to food and drink packaging. It is favored for its light weight, workability, resistance to oxidation and corrosion, recyclability, and relatively low cost.

Traditionally, a major limitation of aluminum is its low strength when compared to other structural materials. This drawback has commonly been addressed through the development of aluminum alloys. Furthermore, grain size reduction has long been known to enhance the strength of metals. With recent advances in material fabrication and synthesis processes, it is now possible to make many different metals with grain sizes ranging from hundreds to tens of
nanometers. Unlike the development of new alloys, in this technique the elemental makeup of the alloy is left unchanged and the microstructure is modified through a variety of processes to produce a stronger material.

This is of particular significance to aluminum alloys because of their wide use in applications where weight is a critical factor. The promise of these high strength aluminum alloys has been recognized as being uniquely suited to aerospace, marine, armor, and automotive applications. In these situations, high strength aluminum alloys can contribute to weight reduction through the substitution of aluminum for heavier materials in the design, as well as allowing for the use of less material than for a conventional aluminum alloy while maintaining the same factor of safety. In turn, these weight reductions are manifested as, for example, improved gas mileage of an automobile or as reduced launch cost of a rocket.

High strength metals can be produced through reduction of their grain size due to dislocation pile up at grain boundaries, known as the Hall-Petch effect. This relationship, shown in equation (1), states that as the grain size, $d$, of a material decreases, its yield strength, $\sigma_y$, increases with $\sigma_\circ$ and $k$ being material constants [1].

$$\sigma_y = \sigma_\circ + \frac{k}{\sqrt{d}}$$

(1)

Procedures to achieve this grain size reduction are very well documented, one of the most studied being ball milling at cryogenic temperatures, or cryomilling, of metal powders. This ultrafine grained (UFG) powder can be consolidated through processes such as hot or cold isostatic pressing (HIP or CIP) and is usually subsequently subjected to some secondary working process, such as high strain rate extrusion [2–5]. Materials produced in this manner have shown substantial improvements in strength, but at the cost of greatly limited ductility.
Various solutions to this drawback have been proposed, but one of the ones that has received the most attention is the addition of coarse grains (CGs), to the UFG powder before consolidation, which results in a bimodal grain size distribution [6,7]. The addition of the CGs returns some ductility to the material, at the cost of a small reduction in strength as shown in Figure 1. One system that has been studied extensively in relation to this process is Al-Mg, specifically Al 5083 (about 5% Mg).

Figure 1. Tensile curves of bimodal Al-Mg alloys with different CG ratios [6].

Even though the overall behavior of this material has been investigated, the underlying governing mechanisms that result in its bulk scale mechanical behavior are still not well investigated or understood. The anisotropic and nonhomogeneous behavior that is caused by the complex microstructure can only be investigated through extensive experimental work and the implementation of multi-scale modeling techniques capable of simulating the crystalline elastic-plastic behavior, grain boundary deformation, and microscale damage and failure. In this work, Al 5083 with a bimodal grain size distribution will be studied through mechanical
testing and computer simulations of the microstructure’s response to loading. This work will seek to characterize the unique behaviors of this material in order to fully utilize its properties in engineering designs. It is hoped that the methods developed and applied in this way are able to be extended and adapted to similar problems and materials.

1.2 Motivation and Objectives

This research began with the motivation to understand the anisotropic, temperature and strain rate dependent mechanical behavior of this bimodal Al 5083 alloy, which had not been investigated before. Although the initial objectives were only to understand the bulk scale mechanical behavior and failure of this material, interesting discoveries pertaining to the material’s behavior at higher temperatures, including its reduced strength compared to the traditional CG material, motivated further understanding of the complex interactions of the microstructure’s phases, grain boundaries, phase interfaces, dispersoids, and solute atoms. Experiments have shown effects including strain rate sensitivity, dynamic strain aging, and dynamic recovery in addition to the segmented stress and strain fields arising from the non-homogeneous microstructure. Examination of fracture surfaces has indicated the influences of the CG/UFG interfaces as well as grain boundaries in the UFG region.

Studying these effects experimentally are very challenging and often inconclusive and non-generalizable, as they are specific to local phenomena observed in specific experiments or not confirmed statistically. One solution to these difficulties is the implementation of multi-scale modeling and simulation. However, the presence of the effects that have been discussed and their underlying causes make the simulation of this material system a complex task. Attempts
to do so in this field have mainly focused on simplistic representations of the phases, not accounting for the crystalline nature of the grains or their interfaces.

This work will approach these phenomena from two angles. Extensive testing is performed on samples of the material under a variety of conditions to examine the effects of temperature, strain rate, anisotropy, and more. These experiments are supplemented by the application of multi-scale finite element methods to study the grain scale nonlinear and plastic deformation effects that occur during mechanical loading through crystal plasticity modeling and simulation. Due to the inhomogeneous nature of this material, the stress-strain distributions are complex and varied. Additionally, the experimental work shows some phenomena that are believed to be a result of effects that become pronounced at the small scale of the UFGs. To provide insights into the material’s behavior that are difficult or impossible to obtain experimentally, these models will represent the relevant microstructural features of the metal at the grain scale. They will incorporate methods to describe crystalline plasticity and the anisotropy of individual grains, which will be linked by grain boundaries with distinct loading behaviors and properties. The effect of grain boundary deformation is included through nonlinear, elastic-plastic behavior models.

Together, it is expected that these two methods of inquiry will allow for a deeper understanding of the microscale processes at work in the deformation behaviors of bimodal alloys. Specifically, the objectives for this work are:

- To understand mechanical behavior of a bimodal grain size Al alloy under different conditions using a full factorial experimental design. This will allow for accurate comparisons of the material’s behavior in different circumstances that are currently
unavailable due to the wide variety of manufacturing and experimental techniques employed in the literature.

- To examine the microstructure, deformation, and failure of this material through microscopic analysis of the fracture surface and grain structure using electron and light microscopy. Additionally, the composition of the material will be evaluated through spectroscopic techniques.

- To create realistic, procedurally generated microstructural models for use with finite element analysis techniques.

- To adapt and apply crystal plasticity and cohesive interface models to this problem. These models will allow for the more accurate representation of grain effects such as crystalline anisotropy, crystalline plasticity, and grain boundary influences.

- To use these models to show the interactions and interrelations of the different components of the microstructure and help to illustrate the microscale deformation effects that occur when the material is loaded.

Figure 2 shows how the experimental and simulation approaches will be utilized to meet these objectives. Tensile tests will be conducted to examine the material’s behavior under a variety of conditions. The custom specimens used for these tests are validated through finite element simulations. The material behaviors and properties determined in the tensile tests are used to create microstructural models that examine the interplay of the CG and UFG regions at two scales, accounting for grain-level effects in the smaller one. The results of these models are used to explore the experimental results obtained through the tensile tests as well as fractography and other microscopic studies. At each of the interfaces between the simulations and experiments, each is used to supplement the other and provide a richer understanding of
not just experimentally observed phenomena but also techniques for representing bimodal microstructures at these scales.

Figure 2. Data flow path in this project between experimental work and simulations at multiple scales.

In Chapter 2, the nuances of ultrafine grained and bimodal materials will be examined. Manufacturing techniques, elements of the microstructure, mechanical properties, and some effects that become relevant at the microscale will be discussed. Chapter 3 contains the experimental segment of this work. The methods and results of these efforts will be explained and used to set the stage for a discussion of simulation techniques in Chapter 4. This chapter lays the foundation for crystal plasticity and cohesive interface models and explains the procedure for the generation of the microstructural models. Finally, in Chapter 5 these models are used to examine the grain scale behavior of the material under different conditions,
including temperature and loading direction. The observations in this chapter are used to supplement and explore concepts developed in the experimental work.
2.1 Fabrication Techniques

The general process of creating a bimodal alloy through powder metallurgy techniques is fairly well understood. First the grain size of some parent material must be reduced, usually through a process known as cryomilling, to create a powder with a UFG grain size. Then the powder is mixed with unmilled powder to create a bimodal microstructure. The mixture is degassed to remove impurities then consolidated. Finally, the material is subjected to some sort of working process to break up prior particle boundaries and improve the properties of the final material. In this section, each of these steps will be examined with respect to how they affect the mechanical properties and behavior of the final product. The process used for the material used in this study is also given.

As discussed in Chapter 1, the strength of this material lies in its small grain size. One technique used to reduce the grain size of a material is known as cryomilling, which is the method employed to produce the material used in this study. In the cryomilling process, a barrel is loaded with the metal powder and the milling medium, typically stainless steel balls.
A process control agent such as methanol, stearic acid, or paraffin is usually added to the mix to prevent the milled powder from becoming welded to the balls or recombining into larger particles [2]. As the name suggests, cryomilling takes place at very low temperature, which also helps prevent recombination of the particles. To achieve these temperatures, liquid nitrogen is circulated through the mixture and replenished as it evaporates. As the material is agitated, the milling medium and powder collide and the powder is broken up into smaller particles.

In addition to the Hall-Petch grain size strengthening, the material has also been observed to be strengthened through Orowan mechanisms resulting from the presence of dispersoids in the material [8]. As may be expected, many of these are compounds of elements Al, Mg, and O [9]. However, it is interesting to note the presence of some N-Al compounds contributing to the strengthening. The N in these compounds was introduced from the cryomilling process, illustrating another and somewhat unintentional pathway for the process to contribute to the material’s strengthening [10].

After the cryomilling run is completed, the remaining liquid nitrogen is allowed to evaporate and the milled powder is mixed with the appropriate amount of unmilled powder to create the desired CG volume ratio. The mixed powder is then hot vacuum degassed to remove the process control agent and other contaminants resulting from the cryomilling process. The powder is placed under a vacuum and heated to a prescribed temperature and held there for several hours. Naturally, the elevated temperature results in some undesired grain growth. However, this step is necessary in order to maximize the density of the billet after consolidation [11].
The choice of consolidation method can have large impact on the properties of the final material. The two most common methods are CIP and HIP (cold and hot isostatic pressing), although other methods such as quasi-isostatic forging or spark plasma sintering can be implemented [12–14]. In CIP and HIP, the powder is subjected to high pressure and, in the case of HIP, temperature to consolidate the powder into a cohesive unit. While higher densities can be obtained through HIP, it does cause more undesired grain growth [11]. Therefore, CIP, which is done at room temperature but requires a higher pressure, is sometimes preferred for the consolidation procedure. Additionally, CIP is more cost and time-effective than HIP when producing the material [15].

Regardless of whether CIP or HIP was chosen, the material now contains prior particle boundaries which adversely affect its properties. To remove them, some method of plastic deformation such as rolling, forging, or extrusion must be utilized. This step also serves to remove some of the remaining porosities in the material and bring it to its final density [16].

The material is now ready to be shaped into its final form. As shown in Figure 3, it now consists of CG bands embedded in a UFG matrix. The material shown in Figure 3 has been consolidated by CIP and extruded. Note the directionality of the microstructure imparted by the extrusion process. As may be expected, this property of the material’s microstructure has a large impact on its properties and will be explored in-depth below.
Figure 3. EBSD images of the finished material. Arrows indicate extrusion direction.

The bimodal microstructure of this material allows both the CG and UFG regions to share a load applied to the material and enables each region to exhibit its strong points. Initially, the CGs bear the load but their deformation is constrained by the UFGs. As loading continues, the CGs transfer the load to the UFGs through the activation of slip systems \[17\]. Each region also has its own dominant deformation mechanisms. In the CGs deformation was found to occur through dislocation slip, while twinning was observed in the UFGs \[18\].

To create the material for this study, UFG Al powder was synthesized by cryomilling Al-5083 powder in liquid nitrogen (~77 K) for 8 hours. The UFG powder was V-blended with the unmilled powder to create 10, 20, and 30% CG mixtures. The mixtures were then hot vacuum degassed at 723 K for 8 hours in order to reduce contaminants such as H, C, and O resulting from cryomilling. The powder was then consolidated by CIP at room temperature at a pressure of approximately 300 MPa for 5 minutes. To break up prior particle boundaries, the material was then extruded. The billet was placed in a furnace at 797 K for 30 minutes prior to extrusion. The extrusion process, with a ratio of about 6:1, was performed in a high-strain rate Dynapak extrusion press which utilizes gas pressure (rather than hydraulics) to force the billet through the die in a matter of milliseconds, resulting in a rod about 2 cm in diameter.
2.2 Mechanical Properties

It has been well established by a variety of research groups that UFG and bimodal Al alloys exhibit improved strength compared to conventional Al 508. The penalty of this strengthening, reduction in ductility, as well as how to combat this problem is also understood. The uncertainty in using this material in design applications lies in its less well known responses to the specific conditions of an application.

There has been some study on the different effects acting on bimodal Al 5083, but it is by no means complete and, in areas such as the effects of strain rate discussed below, there is some disagreement. In addition, differences in the conditions of the experiments described below leave room for further examination of the effects. For example, there has been much work on the compressive properties of this material. However, compression-tension asymmetry has been observed in this material [2,19] as well as in similar materials such as a cryomilled Al-10Ti-2Cu alloy [20]. Therefore, compression tests may not be sufficient to adequately describe the material’s behavior when loaded in tension. Also, as described in Section 2.1, the process parameters used during the creation of the material, notably CIP versus HIP, also affect the material’s properties [4]. Thus, in order to provide fundamental insight into the behavior of these bimodal materials, additional mechanical behavior studies are required.

At the most basic level, the CG ratio used to produce the material affects its properties; this is indeed the reason why the CGs are included. As may be expected, as the CG ratio rises the material behaves more like a conventional Al alloy (low strength, high ductility) and less like a UFG alloy (high strength, low ductility). Thus, for a given application and its attendant strength and ductility requirements, there exists an optimal CG ratio. In their work, Han et al.
attribute the enhanced ductility to the crack bridging effect of adding CG powder to the material, while high strength is retained from the UFG regions [21].

Another effect, perhaps the most extensively examined one acting on the material, is that of strain rate. However, conclusions on this effect seem to have been drawn exclusively from compression tests of the material. Two studies, conducted on bimodal Al 5083, have examined the strain rate effect in compression tests between $10^{-4}$ and $10^{-1}$ s$^{-1}$ [19,22]. In one (Fan et al., 2006), tensile tests were also conducted, but the results of these tests in relation to the strain rate effect are not presented. These studies show that this material is strain rate sensitive and that as strain rate is increased, its strength decreases and ductility increases.

Others have studied the strain rate effect in UFG-only Al. Han et al. varied the tensile strain rate between 4E-4 and 4E-2 s$^{-1}$, and did not observe a significant strain rate effect [23]. A different study by Han et al. again found a small increase in ultimate strength of the material at lower strain rates but, contrary to the studies mentioned above, noted a decrease in ductility as strain rate was increased [24]. Strain rate jump experiments conducted by Hayes et al. on nanocrystalline pure Al also showed little strain rate sensitivity [25].

Another property of bimodal Al 5083 that has had some study devoted to it is the anisotropy derived from the extrusion process. Han et al. studied the differences in longitudinal and transverse samples in compression tests of the material [21]. They observed a significant decrease in strength and ductility in the transverse specimens when compared to the longitudinal specimens. This sort of anisotropic effect is common in extruded materials.

Size is another factor that has been shown to produce significant effects on mechanical behavior [26–29]. There are several different manifestations of the size effect that may serve to either strengthen or weaken a material. The most obvious size effect in this material is its
increased strength due to the reduction in grain size. Other size effects occur as the dimensions of a specimen become comparable to the dimensions of a single grain and thus the specimen may have only a few grains across its cross-section. A consequence of this is that the properties and orientations of individual grains become more significant in the behavior of the material [26]. This effect can work to decrease the strength of specimens as they become smaller, or improve their strength as defect density decreases and theoretical strength values can be approached [26–28]. EBSD (electron backscatter diffraction) analysis of the material used in this study has shown that the UFGs are about 100 nm (Figure 3) and that even the large grains of this material are much smaller than the size of the specimen. Therefore, this effect is not expected to contribute much to the material’s behavior unless there is an analogous effect due to the coarse grained bands which can be up to 20 µm wide and 240 µm long, depending on the initial CG ratio of the powder [7].

Another type of size effect occurs due to the unavoidable surface damage resulting from machining. In large specimens, the properties of the damaged region are not significant because this region is vanishingly small compared to the total volume of the specimen. However, the damaged areas make up an increasingly significant portion of the total material volume as the size of the specimen is reduced. A study on laser sectioned pure Al showed that narrower specimens were stronger due to the laser cutting process, which resulted in a hardened area near the cut surfaces [29]. Similarly, an affected area has been noted in materials sectioned by electric discharge machining (EDM), which is how the specimens used in this study are produced [30,31]. This affected area, which consists of a heat affected zone (HAZ) and a recast layer, is typically less than 30 µm thick. Some reports indicate that HAZ’s may not even occur in Al sectioned by EDM [32].
While the general effects of increased temperature (e.g., reduced strength and increased ductility) are straightforward, these effects can be complicated in a variety of ways making the ultimate effect of temperature not entirely predictable. For example, the failure strain of some nanostructured Al-Mg alloys has been noted to have a non-monotonic dependence on temperature, meaning that not even the general maxim of increased ductility with increased temperature can be taken as absolute [33]. Furthermore, temperature may affect not only the material itself, but also interact with the other effects acting on the material, making the problem of predicting the material’s properties in a given environment more complicated.

As mentioned previously, this aluminum alloy has been observed to exhibit slight negative strain rate sensitivity (a decrease in strength at higher strain rates) at room temperature. The effects of temperature on strain rate sensitivity are difficult to predict. The room temperature sensitivity has been explained by dynamic strain aging (DSA), where solute atoms diffuse to block the movement of dislocations. At higher strain rates the atoms cannot move fast enough to effectively block the dislocations, leading to negative strain rate sensitivity.

The effect of temperature on the strain rate sensitivity exponent of the pure UFG form of this material has been examined through compression tests [34]. The exponent was small and negative at room temperature and increased to 0.15-0.28 with increasing temperature. The increase in the exponent is attributed to diminished work hardening at elevated temperatures. Additionally, the effects of loading at dynamic rates have been studied and suggest a change in dominance of thermal softening mechanisms with strain rate [35]. At higher rates, the activation of effects such as DSA and creep are limited making thermally activated dislocation motion the primary method of thermal softening.
At least when consolidated by high temperature methods such as HIP or quasi-isostatic forging, the UFG microstructure of this material is very stable [35,36]. No significant grain growth has been noted after annealing times of as long as 996 hours at 573 K [37]. It is possible that the heat added during the HIP process allows the material to recover from cryomilling. If the powder is consolidated at relatively low temperatures such as by CIP, it may be that this recovery is not possible and that the method of consolidation affects the material’s response to temperature.

With these effects in mind, a full factorial experiment was designed to test the effects of strain rate, CG ratio, temperature, specimen thickness, and anisotropy on a cryomilled bimodal Al 5083 alloy consolidated by CIP and extruded. This experiment will test these effects on the material’s mechanical properties though uniaxial tensile tests.

2.3 Microscale Effects

In many ways, the properties of ultrafine grained and bimodal materials discussed in the last section are attributable to microscale effects influenced by their grain size. The most obvious of these is the Hall-Petch effect, an inverse relationship between strength and the square root of grain size [1]. As indicated before, this is the primary source for the improved strength properties (and reduced ductility) associated with bimodal and UFG alloys. However, effects attributable to other phenomena may become significant as the scale changes. For example, some sources have reported an “inverse Hall-Petch effect” as grain sizes decrease further, beyond about 10 nm [38]. Below this size, strength may begin to decrease due to the increased significance of grain boundary effects. Other processes, such as mechanical twinning or grain
boundary diffusion, can create appreciable effects as the scale changes but are ultimately outside the scope of this work.

In this work, the main scale effect that will be considered in conjunction with the Hall-Petch effect is that of grain boundary sliding which is considered as a possible explanation of some experimentally observed features discussed in the next chapter. In grain boundary sliding (GBS), two grains move past each other along their interface as a result of an external stress [39]. This mechanism was postulated to exist in the early 20th century and demonstrated in the 1930’s [40–43]. Since then, two distinct types of GBS have been identified: Rachinger sliding and Lifshitz sliding. The main difference between these two is that the former is accommodated by intragranular slip whereas the latter is due to stress-directed vacancy diffusion [39]. The end result of both modes is similar, but only Rachinger-type sliding is considered in this work as the other is more of a diffusion creep mechanism. Additionally, grain rotation has been identified as another possible deformation mechanism in nanostructured materials, which could have similar effects as the activation of GBS [44,45]. In this process, the crystallographic orientations of the grains rotate in response to applied stresses [46]. These two processes, referred to as grain boundary mediated plasticity, can become the primary deformation mechanism under the right conditions [47].

GBS mechanisms can be promoted by high temperatures, but there is evidence that the small grain sizes of nanostructured materials does not confine these effects to high temperature regimes. There have been several reports of room temperature GBS in UFG materials. In one such study, UFG pure aluminum produced through equal channel angular pressing (ECAP) was investigated through nanoindentation [48,49]. Using atomic force microscopy to investigate the topography of the material around the indentation sites, unambiguous evidence
of grain boundary sliding was observed though the deformation had occurred at room temperature. This is also supported though study of the orientation of surface scratches on samples that had undergone tensile loading, which also saw evidence of room temperature GBS [50]. Another study found room temperature GBS in UFG pure copper and nickel, again produced through ECAP [51]. These cases are similar in the fact that they deal with pure metals than have been subjected to severe plastic deformation. Both of these characteristics promote GBS, so this may account for it occurring at low temperatures in these cases [48]. The grain sizes of these materials are substantially smaller than that of the material used in this study but as it happens, the high stacking fault energy of aluminum implies that for even the relatively large sizes of the grains in this material, it may be on the edge of the transition from typical dislocation motion mediated deformation mechanisms to ones dominated by GBS effects [52]. This feature, along with the severe plastic deformation incurred in the fabrication process and added energy from high temperatures may result in significant GBS effects in this material.

The measurement of GBS has been a topic of interest for nearly as long as the phenomenon has been recognized. Obtaining the contribution of grain boundary sliding to the total strain of a material, symbolized by $\xi$, is conceptually simple:

$$\xi = \frac{\varepsilon_{gbs}}{\varepsilon_t}$$

where $\varepsilon_{gbs}$ is the strain attributable to GBS and $\varepsilon_t$ is the total strain [39]. The main difficulty is in the actual measurement of $\varepsilon_{gbs}$. Direct measurement of the separation of grains is possible but would be quite tedious and error-prone. In these cases, if marker lines parallel to the direction of tension (the longitudinal direction) are present on the specimen’s surface prior to
testing, the average grain boundary displacement in the longitudinal direction, \( \bar{u}_l \), can be measured directly and the GBS contribution calculated as:

\[
\varepsilon_{gbs} = n_l \bar{u}_l
\]  

(3)

where \( n_l \) is the number of grains per length. In another method, the vertical (perpendicular to the surface) offset between grains can be easily measured through interferometry [39]. In these cases, an equation such as

\[
\varepsilon_{gbs} = kn_v \bar{v}_r
\]  

(4)

can be used, where \( \bar{v} \) is the average value of the vertical movement, \( n \) is the number of grains per length, and \( k \) is a constant. The subscript \( r \) indicates that the measurements are taken at randomly selected boundaries. The value of \( k \) can be determined experimentally and depends on features of the material such as surface finish [53].

This discussion highlights another advantage of using simulations to study microscale deformation. In the simulations in this work, the separation of grain boundaries can be directly computed, eliminating the need for any experimental approximations as described above. However, in order to be useful all simulations must have a physical basis rooted in experimental work. This is the subject of the next chapter.
3.1 Overview

One of the oldest methods of materials characterization is the tensile test, through which large amounts of data can be gathered despite the relative simplicity of the experiment. This procedure was employed to explore the behavior of bimodal Al 5083 in response to different test and environmental conditions. To begin to understand the differences between this material and its conventionally-grained counterpart many experiments were performed at different temperatures, strain rates, CG ratios, and more. The goal of these efforts is to gain an understanding of the unique properties of this material, so that its strengths can be better utilized in design applications. In this chapter, these experiments are described and their results are examined, with emphasis on the findings that are most relevant to the simulation aspects of the project described later. For a full report of the experimental findings, the reader is referred to [54,55].
3.2 Methods

The material was fabricated as described in Section 2.1. The resulting extruded billet, which was about 2 cm in diameter, was sectioned via electric discharge machining (EDM) into custom-designed dog bone tensile specimens, shown in Figure 4. These specimens were specially designed for this experiment due to the constraints of the diameter of the bulk material. Since it was desired to test the anisotropy imparted from the extrusion process, it was necessary to cut specimens across the face of the extruded rod (transverse specimens) and it was of course preferable that these specimens be the same as the ones used in longitudinal tests (where the directions of extrusion and tension are parallel). Since no standards are available for this size specimen, a useful shape had to be designed through a combination of engineering analysis and trial and error.

![Figure 4. Test specimen. Dimensions in mm.](image)

The original specimen design was more like a traditional dog bone sample, with a relatively small radius of curvature between the gauge length and the grip section. This stress concentration caused fractures to occur at these corners, which was not useful. The logical extension of increasing the radius of curvature is the design in Figure 4. This design features a variable cross-section which moves the site of fracture into the gauge length at a predictable location. It has relatively wide grip areas to maximize the surface area in contact with the grips while keeping the total length within constraints. This specimen design was analyzed through
FEA, shown in Figure 5, to evaluate its performance in the accurate determination of mechanical constants. This analysis showed a highly uneven stress distribution with strain localized at the point of minimum cross sectional area, as would be expected from the variable cross section. Therefore, measurements taken over the gauge length, such as by an extensometer, can significantly affect the measured properties. However, localized strain measurement techniques such as strain gauges can accurately predict these properties. This was verified by averaging the strain at all points in a “grid area,” much like a strain gauge applied to this area would. This produced minimal deviation from theoretical results.

![Stress and strain contours in specimen’s gauge length.](image)

Tests were conducted on a Test Resources 800LE load frame (Figure 6) under displacement control with a 2000 lb (~9 kN) load cell. Designed for use with small specimens such as these, this frame/load cell combination provides a displacement resolution of 0.5 μm and a force accuracy of about ±0.2%. A method of strain measurement that was compatible with the size of these specimens as well as the high temperatures that would be encountered during the experiment was also required. Measuring the strain within the gauge length avoids
errors attributable to crosshead and load cell compliance and fixture tolerances. Therefore, tiny strain gauges (Vishay Micro- Measurements EP-08-105DJ-120 and EP-08-031DE-120) were attached to the specimens as shown in Figure 7. These gauges featured a grid area of only about 0.25 mm², the ability to measure high strains, and could withstand temperatures up to 473 K. A Vishay P3 strain recorder was used to measure the output from the strain gauges. From the factory, these units can only measure up to about 3% strain. To enable their use with the strain gauges, the range of the devices was extended using precision resistors on the strain gauge leads, attenuating the signal and increasing the range.

Figure 6. Test Resources 800LE load frame and thermal chamber. P3 recorder is visible in the lower left.
A full-factorial experiment was performed to examine the effects of the parameters included in the study. These parameters are shown in Table 1. For each combination in Table 1 at least three replicants were run, resulting in over 200 individual tests. Statistical analysis of the results of these tests was conducted to determine the relevant effects on the material’s behavior.

Table 1
Experiment Plan
Repeated for 10%, 20%, and 30% CG ratios, 1 mm and 0.5 mm specimen thickness

<table>
<thead>
<tr>
<th>Run</th>
<th>Temperature K (°C)</th>
<th>Orientation</th>
<th>Strain Rate s⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>293 (20)</td>
<td>Long.</td>
<td>1E-04</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>Trans.</td>
<td>1E-04</td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
<tr>
<td>5</td>
<td>383 (110)</td>
<td>Long.</td>
<td>1E-04</td>
</tr>
<tr>
<td>6</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
<tr>
<td>7</td>
<td></td>
<td>Trans.</td>
<td>1E-04</td>
</tr>
<tr>
<td>8</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
<tr>
<td>9</td>
<td>473 (200)</td>
<td>Long.</td>
<td>1E-04</td>
</tr>
<tr>
<td>10</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
<tr>
<td>11</td>
<td></td>
<td>Trans.</td>
<td>1E-04</td>
</tr>
<tr>
<td>12</td>
<td></td>
<td></td>
<td>1E-05</td>
</tr>
</tbody>
</table>
In addition to the tensile tests, the specimens were subjected to microscopic and chemical analysis. Optical and scanning electron microscopes (with EBSD capabilities) were used to examine the specimens’ grain structure and fracture surfaces. The specimens were prepared for EBSD by mechanical polishing up to 800 grit followed by 4 hours of vibratory polishing in colloidal silica. The elemental composition of the final product was determined through ICP (inductively coupled plasma) spectroscopy for metallic elements. For non-metallic elements, inert gas fusion was used to detect nitrogen, oxygen, and hydrogen; carbon content was analyzed through the combustion method. These results are shown in Table 2 and are compared to the manufacturer’s grade certification report for the batch of as-received Al 5083 powder. Non-metallic elements were not analyzed in this report, so the change in composition of these elements due to the manufacturing process is unknown.

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>Mg</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>H</th>
<th>C</th>
<th>N</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before</td>
<td>Bal.</td>
<td>4.28</td>
<td>0.08</td>
<td>0.13</td>
<td>0.72</td>
<td>0.09</td>
<td>0.06</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>After</td>
<td>Bal.</td>
<td>4.96</td>
<td>0.12</td>
<td>0.18</td>
<td>0.94</td>
<td>0.13</td>
<td>0.02</td>
<td>0.0086</td>
<td>0.18</td>
<td>0.14</td>
<td>0.48</td>
</tr>
</tbody>
</table>

<0.02% Ni, Ti, Zn

### 3.3 Results

The experiments found significant effects attributable to strain rate, material orientation, CG ratio, and temperature. It was suspected that that specimen thickness may affect the material properties due to the correlation in scale between the thinner specimens and the length of the CG bands, especially in transverse specimens. Another suspected source of thickness effect was the heat affected zone (HAZ) from the EDM cutting, which could make up a significant
portion of these small specimens, since HAZ depths up to 500 μm have been reported [32]. However, the experiments ultimately showed no statistically significant differences in strength between the two specimen thicknesses.

Increasing CG ratio of the material had the expected effects of decreasing strength and increasing ductility, which is of course why the CGs are included. In previous works, this decrease had been approximated as linear [56]. This is a useful approximation and allows for the examination of the material by treating it as a composite material with a UFG matrix and CG inclusions. This imagery is often employed in this work as a means for understanding the interactions between the phases, however, the experiments conducted in this work showed a saturation effect at higher CG ratios. This is thought to be related to the closer spacing of CG bands as their ratio increases, resulting in easier interaction between the bands and reducing their individual effects.

Figure 8 illustrates the roles played by both the CG and UFG regions in the deformation and failure of this material. It can be seen that the CG experienced much more plastic deformation than the surrounding CG region, as evidenced by its pronounced necking. Additionally, voids can be seen inside of the CG band which nucleated as the region began to fail. These indicators of ductility are of course much less evident in the surrounding UFG region. Also of note is the fact that there is minimal delamination between the two regions. That is, the final failure path cut across the CG band at the same level as it had been propagating through the matrix, with no pullout.
Additionally, negative strain rate sensitivity was observed in strain rate tests, along with improved ductility at lower strain rates. Evidence of dynamic strain aging was observed in serrated stress-strain curves. DSA occurs when dislocations are temporarily blocked by obstacles in the material, such as solute atoms. This impediment to dislocation motion causes the material to become harder. The dislocations are more easily arrested at low strain rates, thus producing the observed negative strain rate sensitivity [57]. The ductility effect was attributed to diffusion mediated mechanisms acting to relieve local stress concentration sites [24].

Anisotropy was observed in a manner consistent with extruded materials, with reduced strength and ductility in the transverse direction. It was found that increasing CG ratio in the transverse direction served to increase the material’s overall strength as shown in Figure 9. This may indicate that, at least in the transverse direction, the material is strain limited and is capable of withstanding higher stresses (although still not as high as in longitudinal loading). Support for this theory is shown in Figure 10, which shows the strength of the material in the

Figure 8. SEM micrograph showing CG region embedded in UFGs in the longitudinal fracture surface.
longitudinal and transverse directions as a function of temperature. As temperature (and thus ductility) increases, the differences in strength between the two directions, which was quite drastic at room temperature, becomes negligible at higher temperature.

Figure 9. Effect of anisotropy and CG ratio on strength.

Figure 10. Effect of temperature and anisotropy on ultimate strength.
Fractographic analysis, shown in Figure 11, showed the prevalence of intergranular failure mechanisms in this failure mode when compared to the dimpled intragranular texture of longitudinal specimens. The grooved texture of the transverse specimens, which is oriented in the extrusion direction, is believed to be an artifact of the material’s failure along grain and particle boundaries. Although correlation does not imply causation, it may be that the material’s strain limited behavior is attributable to intergranular mechanisms. Even at the highest test temperatures, these characteristics of the fracture surfaces were maintained, although the mechanical strength was unaffected by orientation as shown in Figure 10. It is unknown how the ultimate strain was affected, but qualitative analysis suggested that the transverse orientation continued to exhibit lower ductility. This is indicative of the continued contributions of the intergranular failure mechanisms at higher temperatures. Intergranular fracture modes can also be accentuated by the presence of hydrogen, which is known to cause grain boundary embrittlement [58]. Table 2 indicates that this element is indeed present in the material but no direct measurements of the material’s final strain were available to indicate embrittlement, which would anyway be difficult to extract from the grain size induced decrease in ductility.
Figure 11. Specimen fracture surfaces in (a) longitudinal and (b) transverse material orientations.

There were two distinct textures found in the fracture surface of specimens tested in the longitudinal orientation, shown in Figure 12. The inner dimpled region is attributable to the nucleation and growth of voids at the initiation of failure. This central region would have grown outward until sudden final failure was reached, creating the smoother outer region shown in the micrographs. As shown in Figure 12, the size of these two regions is affected by temperature. At increasing temperature, the dimples of the dimpled region become more pronounced and the size of the fast fracture region shrinks. Both of these features are qualitative indicators of increased ductility after the onset of failure.
Experimental observation of temperature effects were somewhat hindered due to the inability to measure ductility past approximately 9% due to the limitations of the strain gauges and their adhesive and the range of the strain gauge recorder. Although this was not a problem for the relatively small strains encountered at room temperature, at even the intermediate test temperature, the final ductility was off scale. This limited total ductility observations at these temperatures to qualitative measures. Grain size analysis through pre- and post-test EBSD of
the samples showed no significant changes in the grain size distribution attributable to either
temperature or mechanical stress. Grain size stability with respect to temperature is a known
quality of this material and has been observed after annealing times of as long as 996 hours at
573 K [33–35]. Pole figures taken from these maps, Figure 13, showed no evidence of a
preferred crystallographic orientation in the microstructure.

![Figure 13. EBSD pole figures.](image)

Some of the most interesting experiment results were observed in relation to the high
temperature tests. Dynamic recovery was observed as evidenced by the reduction in stress as
the test progresses. As would be expected, this effect is more pronounced at higher
temperature, with these tests showing a clearly defined stress peak near the yield point. As seen
in Figure 14, the rate of recovery also appears to be affected by the CG ratio. The materials
with lower CG ratio show greater recovery over the range studied. This was interpreted in
terms of the dislocation density of the UFG region. Since recovery is driven by the dislocations
in thermodynamically unstable configurations, a material with higher dislocation density has a
higher driving force for recovery [59]. TEM foils prepared at the University of California,
Davis, showed higher dislocation densities in the UFG region. Therefore, the increased
concentration of UFGs that accompanies a deceasing CG ration increases the overall dislocation density and thus the driving force for dynamic recovery.

![Figure 14. Yield region of stress-strain curves of material with different CG ratios at 473 K showing various rates of dynamic recovery.](image)

Naturally, the strength of the material decreased with increasing temperature. However, significant differences were observed in the temperature response of this material when compared to the response of conventional Al 5083 (Figure 15). While the conventional material is much weaker at room temperature, it maintains its strength better at higher temperatures so that it is stronger than the bimodal alloy at 473 K (200 °C). Higher temperature tests of 0% CG material have shown a similar trend when compared to a conventional alloy [36]. In these tests, the material’s losses in strength begin to level out soon after about 473 K (200 °C), though it remains weaker than the conventional material at the same temperature.

The same pattern of greatly increased strength at room temperature coupled with no improvement compared to the conventional material at increased temperatures was also
observed in a similar nanostructured Al$_{5083}$-Al$_{85}$Ni$_{10}$La$_{5}$ composite [60]. It appears that the strengthening effect of grain size plays a diminishing role as temperature increases. A possible explanation is presented in the study conducted by Mallick et al., who observed the effect at temperatures up to 523 K [33]. They suggest that at higher temperatures, grain boundary sliding (GBS) becomes activated and leads to thermally assisted sliding, accounting for the losses in strength. They also found dislocation pileups to be reduced at higher temperatures, resulting in a loss of strain hardening and strength. Combined, these observations could account for the reductions in strength observed in this and other similar nanostructured materials.

Figure 15. Ultimate strength as a function of temperature for bimodal Al 5083 with different CG ratios compared to conventional values for Al 5083 O and H tempers.

To aid in the statistical analysis of these experimental results, they were fit to a Voce-type plastic hardening law:

$$\sigma_f = \sigma_s - (\sigma_s - \sigma_y)e^{-\frac{\varepsilon_f}{\varepsilon_c}} \quad (5)$$
where $\sigma_f$ is the flow stress, $\sigma_s$ is the saturation stress, $\sigma_y$ is the stress at the start of the plasticity model, $\varepsilon_p$ is the plastic strain, and $\varepsilon_c$ is the characteristic strain. The saturation stress and characteristic strain were determined for each set of data by performing a least-squares regression. The yield stress in this model is the elastic limit, the point at which the stress-strain curve becomes nonlinear. In these experiments, this value is significantly different from the more commonly reported 0.2%-offset yield stress and this must be considered when comparing these results to other sources. The saturation stress is the value that the model approaches asymptotically at large plastic strains. Finally, the characteristic strain is a value that describes the shape of the stress-strain curve. A smaller characteristic strain indicates that the model approaches the saturation stress more quickly. This model has been used in other studies to describe the plastic behavior of this material with good agreement [56].

3.4 Need for Simulation

These experimental results illustrate some of the complexities in a bimodal material’s deformation, which is influenced by the properties, concentrations, and interactions of the phases and compounded by effects of grain boundaries, inhomogeneous dislocation densities, and other microstructural features. These complexities provide the motivation for the modeling and simulation that is the focus of the rest of this work. In this section, the experimental findings will be evaluated in the context of modelling, to provide the direction for this work and to determine which effects are necessary or pertinent for inclusion in the simulations.

Many of the experimentally observed effects had to be disregarded in the simulations just to make these efforts feasible. Any strain rate sensitivity was not modeled and simulations were all run at the same strain rate. Additionally, effects such as dynamic strain aging and
recovery were not modeled since they are attributable to very small scale features that are below the scale of the present model. Other effects such as CG ratio and anisotropy are not explicitly included, but may arise from the models themselves.

With these considerations, the realm of the model to be created becomes clearer. The model will incorporate the crystallographic behavior of individual grains and their boundaries. Material properties can be applied to both the CG and UFG phases in the model to mimic their different mechanical properties. This model can thus be used to study the behavior of the bimodal microstructure at different temperatures, in different loading configurations, and to make predictions about failure modes and paths. The development of this model, from the constitutive equations to the actual microstructural representation, is the subject of the next chapter.
CHAPTER 4
SIMULATING MICROSTRUCTURAL DEFORMATION

4.1 Overview

Simulations have long been employed in the study of bimodal materials due to the complex effects arising from their non-homogeneous microstructures as well as their ability to circumvent the time, cost, and difficulties associated with traditional experimental observations. The results from these simulations are useful in supplementing experimental findings and theories as well as providing new directions for inquiry. A goal of this work is to implement advanced microstructural simulation techniques to creating some of the most sophisticated models used for the study of this material.

Previously, models used to study bimodal materials have been idealized representations of the microstructure, used to provide high level information on the material’s behavior. Models depicting a bimodal “unit cell,” a quarter-symmetric circular CG embedded in a homogeneous UFG region, have predicted crack initiation at CG/UFG interfaces, due to the large differences in the phases’ properties, followed by propagation normal to the loading direction [61]. Other models have captured the overall extruded structure of the material, showing failure initiation
at an interface followed by propagation through UFG and CG regions [62]. These models have captured large scale mechanisms active in the material, and although they differentiate between the CG and UFG phases, inside of these regions they are treated as isotropic and homogeneous.

In order to capture the grain scale behavior of materials, special techniques must be employed. At these scales, the crystalline nature of individual grains becomes significant and the material can no longer be treated as isotropic. The crystallographic slip systems of each grain must be considered and their activity and behavior represented. Additionally, the properties of the grain boundaries can become significant at this scale. While these features are perhaps best represented through atomic scale models, they can be incorporated at this scale by representing them as interfaces between discrete grains.

Though often complex, these approaches are classical and well documented in the literature. However, one challenge presented by their use is tailoring them for use in a specific problem, such as the one considered in this work. In this chapter, techniques to calculate the constitutive response of individual grains as well as their grain boundaries are examined. It is desired that these techniques are able to account for the changes in behavior that occur at elevated temperatures, in order to investigate the contributions of the microstructural effects to the experimentally observed phenomena. The implementation of these models in commercial finite element packages, specifically Abaqus, is considered and the methods used to procedurally generate the actual finite element models are examined. Finally, the techniques used to obtain the material constants for these models are discussed.
4.2 Crystal Plasticity Methods

Previous investigations into the microscale deformation behavior of bimodal metals have usually utilized isotropic material definitions which are homogeneous within the phases. At the grain scale, these assumptions may no longer be appropriate due to the anisotropy attributable to the crystalline structure of the individual grains as well as the increased influence of grain boundaries at smaller scales. In the literature, constitutive equations have been developed to address these features and are implemented in this study. Crystal plasticity methods are used to capture the orientation dependent elastic and plastic response of grains modeled as single crystals separated by grain boundaries represented as cohesive interfaces.

Crystals exhibit anisotropic plasticity due to deformation being confined to specific slip planes. When considering a polycrystalline aggregate containing a large number of grains, the individual contributions of crystalline anisotropy of each grain negate each other because of the random nature of grain orientations and bulk scale constitutive material models can safely be used. However, when the grains are considered as discrete entities and in small numbers, capturing their crystalline behavior becomes important to preserving the accuracy of the model. Crystalline behavior includes elasticity, which only causes anisotropy of the grains, and the plastic deformation which is the cause of slip on different crystalline slip systems. Implementation of elastic anisotropy caused by this crystalline nature is fairly simple and straightforward with current computational capabilities. However, implementation of the plastic deformation of each individual grain is far more complicated. It is this issue that is addressed through crystal plasticity finite element theory. These crystal plasticity methods analyze the resolved shear stresses on the crystallographic planes of a grain to determine which slip systems are active and how quickly they are deforming. There is much information on the
formulation of these approaches available in the literature [63–69] and they have been used successfully on a wide range of problems, including microstructure modeling. These models are all similar in that they multiplicatively divide the deformation gradient into elastic and plastic components. The treatment of the elastic component of deformation is fairly standard, usually described by cubic elastic constants, but the models vary in their treatment of plasticity and determination of active slip systems. Some formulations consider the discrete effects of solute strengthening and grain size. In these cases solute strength can be considered a linear addition to the strength of the base metal [58], while grain size strengthening has been represented through strain gradient methods [70–72].

The individual grains of the polycrystal model are represented as single crystals through crystal plasticity methods. In this work, crystal plasticity methods are used to evaluate the non-isotropic mechanical response of the grains at the microstructural level due to their crystalline nature. The fundamental argument of this technique is a multiplicative decomposition of the deformation gradient $F$ into elastic and plastic parts $F^e$ and $F^p$ such that

$$F = F^e F^p.$$  

(6)

The time rate of change of the plastic component of the deformation gradient is given by

$$\dot{F}^p = L^p F^p,$$  

(7)

where $L^p$ is the plastic slip velocity gradient and can be obtained through the summation of slip rates $\dot{\gamma}^\alpha$ for each slip system $\alpha$ over the total number of slip systems $N$ as

$$L^p = \sum_{\alpha=1}^{N} \dot{\gamma}^\alpha m^\alpha \otimes n^\alpha,$$  

(8)

where vectors $m^\alpha$ and $n^\alpha$ are the slip plane directions and normals, respectively, and the slip rate on a system is a function of the resolved shear stress on that system. With an updated
plastic deformation gradient calculated in this manner, and the total deformation gradient at the end of a time step known, the elastic part can be extracted via equation (6). All that remains is to calculate the second Piola-Kirchoff stress tensor $\mathbf{S}$

$$
\mathbf{S} = \frac{\mathbf{C}}{2} (\mathbf{F}^e \mathbf{F}^e - \mathbf{I}),
$$

where $\mathbf{I}$ is the second order identity tensor and $\mathbf{C}$ is the fourth order elasticity tensor.

The above process is the general calculation cycle for many crystal plasticity studies, though the order can be changed depending on the known values for a scheme [65]. Much of the variation between approaches stems from treatment of the function to obtain the crystallographic slip rate from the resolved shear stresses. These other approaches vary in complexity from simple power laws directing the relationship between the critical and current shear stresses to much more complex representations accounting for more crystal parameters or thermodynamic effects [73–75].

In this work, the general slip rate equation given by Marin in [64] is used:

$$
\dot{\gamma}^\alpha = \dot{\gamma}_o \exp \left\{ -\frac{\Delta F}{k \theta} \left[ 1 - \left( \frac{\tau^\alpha}{g_c^{\alpha}} \right)^p \right]^q \right\} \text{sgn}(\tau^\alpha),
$$

where $\dot{\gamma}_o$ is a reference strain rate, $\Delta F$ is the activation free energy required to overcome slip obstacles without applied stresses, $k$ is the Boltzmann constant, $\theta$ is temperature, $\tau^\alpha$ is the resolved shear stress on slip system $\alpha$, $g_c^{\alpha}$ is the total resistance to slip, and $p$ and $q$ are constants describing the shape of the glide resistance profile.

To describe the strain hardening of the crystal, the slip resistances must also be updated for each iteration. This is done, again following Marin, through the equation
\[
\dot{g}_e^\alpha = h_0 \left[ \frac{g_{c,S}^\alpha - g_{c,0}^\alpha}{g_{c,S}^\alpha - g_{c,0}^\alpha} \sum_{\alpha=1}^{N} |\dot{\gamma}_\alpha^\alpha| \right],
\]

(11)

where \( h_0 \) is the initial hardening rate, \( g_{c,0} \) is the initial strength of a slip system, and \( g_{c,S}^\alpha \) is a saturation strength given by

\[
g_{c,S}^\alpha = g_{c,S0} \left[ \frac{\sum |\dot{\gamma}_\alpha^\alpha|}{\dot{\gamma}_{S0}^\alpha} \right]^{k_{S0}^\alpha} \Delta F
\]

(12)

with material parameters \( \dot{\gamma}_{S0} \) and \( g_{c,S0} \).

Through this formulation, the crystals behave as elastic materials with cubic symmetry until the resolved shear stress on a crystallographic plane exceeds a critical value necessary for plastic slip. This critical stress increases with the total amount of plastic deformation, creating a plastic hardening effect. In this study, the hardening effect is implemented by updating the slip resistance through equation (11). With the constitutive behavior of the crystals themselves defined in this manner, it remains to determine how the crystals interact in a polycrystalline aggregate. This is the role of the cohesive interface model discussed in the next section.

### 4.3 Cohesive Interface Modeling

The second component of an accurate microstructural model is a grain boundary description. Grain boundary models attempt to capture the roles that grain boundaries play in the intricacies of a material’s overall deformation behavior, in addition to its failure mechanisms. Recognition of the boundaries’ importance has led to recent study and development of techniques which incorporate a variety of features. Some of these approaches to grain boundary modeling are considered for use with the proposed model. These include defining continuously varying
mechanical properties as a function of the distance from the grain edge or through crystal plasticity or other descriptions of a boundary region’s properties [76–78]. Alternatively, cohesive zone models can be used to describe the traction-displacement relationship of grain separation [79–81]. This method has been used in studies of grain boundary plasticity and failure in UFG and nanocrystalline materials [80–83].

Finite element models based on unit cells or idealized microstructures have shown many experimentally observed fracture features. Simple models of a “bimodal” unit cell have predicted damage initiation at CG/UFG interfaces, followed by crack propagation perpendicular to the loading direction. These models postulate that CG plasticity and CG/UFG interface delamination are major energy sinks that promote overall material ductility [61]. Fracture path studies in idealized bimodal Cu showed dependence on the actual geometry of the CG regions. It is noted that interfacial delamination is promoted when the interfaces are oriented continuously and parallel to the direction of crack propagation [84]. Models incorporating the extruded morphology of the grains in bimodal Al 5083 have also shown crack initiation at CG/UFG interfaces, with the inhomogeneity of the microstructure influencing the crack propagation path [62,85]. However, each phase is modeled as homogeneous and isotropic. At that scale, it is a good approximation, but this view can be refined to account for the presence of a regime where deformation is controlled by grain boundary effects, as suggested by recent simulations [86].

The constitutive model describing the grain boundary behavior in this work is based on the cohesive interface model presented by Wei and Anand [80] which depicts elastic-plastic interface deformation with plastic hardening. This model, shown schematically in Figure 16, can also include an interface failure criterion for crack propagation studies. The assumption of
this model is that two grains are joined together by an interface with negligible thickness. In addition to the deformation of the grains themselves, the separation between the grains can increase based on the applied loads.

![Interfacial traction-displacement relationship.](image)

Figure 16. Interfacial traction-displacement relationship.

The model is formulated in a coordinate system based on the local geometry of the interface. At a point on the interface, the system is defined such that \( \mathbf{e}_1 \) is the unit vector normal to the interface at this point and \( \mathbf{e}_2 \) and \( \mathbf{e}_3 \) are orthogonal vectors in the plane tangent to the interface as shown in Figure 17.

![Interfacial coordinate system for grain boundary model.](image)

Figure 17. Interfacial coordinate system for grain boundary model.
It is assumed that the total displacement $\delta$ between the interfaces can be additively decomposed into elastic and plastic parts, that is,

$$\delta = \delta_e + \delta_p,$$

(13)

where $\delta_e$ and $\delta_p$ are its elastic and plastic components, respectively. It can be shown [87,88] that the traction-displacement relationship is of the form

$$t = K\delta_e,$$

(14)

where $t$ is the traction vector and $K$ is the interface stiffness matrix, which is assumed to be isotropic in the tangential directions. Thus its component $K_{ij}$ has the value $K_N$, the normal elastic stiffness. All other $K_{ij}$ are the tangential elastic stiffness $K_T$. The traction vector can be decomposed into normal, $t_N$, and tangential, $t_T$, components such that

$$t = t_N + t_T$$

(15)

and

$$t_N = t_N e_1,$$

(16)

$$t_T = t - t_N.$$  

(17)

Since the tangential directions were assumed to be isotropic, the yield surface can be defined by the functions $\Phi_N$ and $\Phi_T$. In this model, these functions take the form

$$\Phi_N = t_N - s_N \leq 0$$

$$\Phi_T = \bar{\tau} + \mu t_N - s_T \leq 0$$

(18)

where $s_N$ and $s_T$ are the critical values for the onset of plasticity, $\bar{\tau}$ is the magnitude of $t_T$ ($\bar{\tau} = \sqrt{t_T \cdot t_T}$), and $\mu$ is a friction coefficient representing the dependence of the tangential interfacial resistance on the normal stress at the interface. Using, for instance, a radial return
algorithm the elastic-plastic response can be determined and the yield surface expanded to account for plastic hardening [89,90].

In this work, the interfaces plastically harden according to a power law. Inside the interface model, the law is discretized into linear segments whose slope is a function of the accumulated plastic displacement from both deformation modes, $\overline{\delta}_p$, such that

$$\frac{\partial t}{\partial \overline{\delta}_p} = an\overline{\delta}_p^{n-1},$$

(19)

where $a$ is the strength coefficient and $n$ is the strain hardening exponent. The total plastic displacement is given by

$$\overline{\delta}_p = \sqrt{\delta_{p,N}^2 + \beta\delta_{p,T}^2},$$

(20)

where $\beta$ is a constant coupling parameter taken as 0.25 in this work [80].

Equation (20) can also be used to enforce a plastic displacement-based failure criterion, $\overline{\delta}_{p,\text{fail}}$. After this criterion has been met, the strength of the interface degrades at a rate governed by $C_{\text{fail}}$, which is a constant between 0 and 1 in the equation

$$t^{i+1} = C_{\text{fail}} t^i,$$

(21)

where $t^i$ is the traction at the beginning of the time increment and $t^{i+1}$ is the traction at the end of the increment. It was observed that a value of 0 for this parameter (that is, instantaneous interface failure) resulted in reduced stability of the model. Trials found that a value of 0.85 produced a good balance of computational stability and stress concentration at the crack tip.

In this and the previous sections, the techniques that will be used in the microstructural models have been examined. The remainder of this chapter will be concerned with their implementation. This will involve the coding of these constitutive equations into a form usable
by commercial finite element software, generating suitable models of the microstructure, and finally determining appropriate values for the material constants introduced in these sections which are not available in the literature.

### 4.4 User Subroutines in Abaqus

Commercial finite element packages such as Abaqus and ANSYS currently do not natively support the models discussed in Sections 4.2 and 4.3. They do, however, provide the framework for advanced users to create custom subroutines that handle the aforementioned models. Abaqus was used for this work, due to its ease of use, extensive and accessible documentation, and powerful and intuitive scripting features. To support the microstructural models, the user subroutines UMAT and UINTER were developed using the Fortran programming language. These auxiliary files are compiled at the start of an analysis and provide the procedure for the determination of the material’s or interface’s response, respectively. Additionally, these routines must also update information pertinent to Abaqus’s solution procedure and the state variables used in the subroutine itself.

The UMAT subroutine is used to define a material’s constitutive behavior. The subroutine is called at all of the calculation points in elements assigned to the user defined material and updates the stresses at that point given the strain increment for the time step. Additionally, the subroutine must calculate the Jacobian matrix, $\partial \Delta \sigma / \partial \Delta \varepsilon$, which is used by Abaqus when solving each time step. The UMAT subroutine used in this work is a heavily modified version of the one published by Huang [91]. This existing framework was useful to work from because it already had working methods for matrix inversion and rotation (adapted from [92]), calculation of Schmid factors, as well as a method to determine the Jacobian matrix, the
determination of which can be fairly complex. Also included with this approach were bookkeeping procedures to accomplish tasks like parsing the material property array, determining all the slip systems in a family, and updating the state variables. The code also has the ability to solve the constitutive equations iteratively through the Newton-Raphson method to improve stability. This portion of the code was deactivated to avoid having to compute the derivatives associated with Newton-Raphson iteration, at the cost of increased solution time.

The code was designed to be extensible for use with other constitutive equations, so the changes were fairly localized, requiring rewriting of several functions but little modification to the process flow of the subroutine. The function to calculate the strain rate was modified to incorporate equation (10) into the subroutine. The existing code is formulated in terms of a variable \( x \), the ratio of the current shear stress on a slip system its current critical stress. The subroutine also calculates the derivative of the strain rate with respect to \( x \) for later use in the solution procedure. This variable substitution was done and the derivative was calculated:

\[
\frac{\partial \dot{\gamma}^\alpha}{\partial x} = \dot{\gamma}_0 \frac{-\Delta F}{k\theta} \frac{pq \exp \left\{ \left. \frac{-\Delta F}{k\theta} \left[ 1 - \left( \frac{|x|}{\theta} \right)^\rho \right] \right\} \right\} \left[ 1 - \left( \frac{|x|}{\theta} \right)^\rho \right]}{(1 - |x|)^{\rho-1}}. \tag{22}
\]

The second major modification was to function which calculates the increment in the critical shear stress based on the plastic hardening law. The expected output of this subroutine is a hardening matrix \( H \) such that

\[
\dot{g}_c^\alpha = \sum_{\beta=1}^{N} H_{\alpha\beta} \dot{\gamma}^\alpha. \tag{23}
\]

In this implementation, the differences between latent and self hardening are disregarded, making \( H_{\alpha\beta} \) constant for a given slip system and thus corresponds to equation (11) for this model. Therefore, the function was rewritten to include this calculation in its determination of
the hardening. Other modifications to the code included tracking of the additional state variables associated with the new constitutive equations and code to track the rotational activity of the grains.

The UNITER subroutine is in many ways similar to UMAT. The interfacial traction per unit area is calculated given a displacement increment. An analog to the Jacobian matrix, the interface stiffness matrix, must also be calculated. This subroutine was written from scratch, based on the procedure outlined by Wei and Anand [80]. Trial stresses are calculated assuming completely elastic interfacial deformation then corrected if necessary to include plasticity and remain on the yield surface defined by equation (18). Interface failure is also checked for and the stress updated accordingly. Determination of the interface stiffness matrix was one of the major challenges in the creation of the UINTER. The returned interface stiffness matrix, like the Jacobian matrix, is not required by Abaqus to be exact, though this does improve stability. If these matrices are “close enough,” the correct solution will be achieved (if any solution at all is reached), although possibly more slowly [93]. This was utilized to create a much simpler, although approximate, interface stiffness matrix. Useful references in the development of this matrix were [94,95].

Although not simple, the creation of these subroutines is a powerful tool and is well documented in the software manuals as well as the literature. A thorough understanding of the problem and of the relevant theory is required, but through the implementation of these subroutines Abaqus was able to be used to solve grain scale finite element models incorporating crystal plasticity and grain boundary deformation.
4.5 Model Generation

The user subroutines described in the previous section are of limited usefulness without a finite element model to run them on. This model must be representative of the relevant features of the microstructure and must also be computationally tractable. To address the first requirement, methods to digitize micrographs such as those obtained through EBSD were considered. While these methods are quite fascinating, they were ultimately abandoned for this work in favor of procedurally generated models. These models have the advantages of providing increased control of microstructural features, allowing the user to examine features and effects in isolations and explore ideas in ways unachievable through experimental methods, which is after all the purpose of simulations. Due to the large difference in scale between the CG and UFG phases of the material studied in this work, the second requirement of computational tractability proved to be challenging. A model developed on a scale that includes CGs has so many UFGs that the model becomes unmanageable. Conversely, a model with a practicable number of UFGs cannot capture their larger scale interactions with the CGs. Therefore, two types of models were developed as shown in Figure 18, the large scale (LS) and grain scale (GS) models.
The LS model replicates the bimodal microstructure of the material studied in this work. It represents a 50 μm square 2D section of the material, with the extruded CGs represented as procedurally generated ellipses. These shapes are generated according to a Gaussian distribution around uniformly randomly selected center points. The number of CG regions can be varied to achieve different CG ratios. Due to the large number of UFGs at this scale, it is assumed that the individual contributions of grain scale anisotropy cancel out and thus CG and UFG phases are homogeneous and isotropic.

The GS model is somewhat more complicated. This model, representing a 1 μm square, takes into account the behavior of individual grains and their boundaries through the previously discussed crystal plasticity and cohesive interface methods. At the heart of this model’s generation lies the Voronoi diagram. This map divides an area into cells based around a number of seed points, as seen in Figure 19a. Each cell is the region of the map that is closer to its seed point than any of the others [96]. This diagram, which can be traced all the way back to the 19th century, has been used in the description of widely different problems in various fields such as epidemiology, computer science, graphics, and computer aided design. Another
common application of the diagram (and one of the earliest, circa 1930’s) is in the representation of polycrystalline grain structures. The patterns of nucleation and growth of grains during crystal growth is similar to the Voronoi diagram, which can be thought of as the radial and exclusive growth of circles around the seeds [96]. Thus, the figures can be used to produce more realistic grain structures than the patterns of regular polygons, such as hexagons, that are sometimes used instead. Additionally, the figures have the advantage of being easily generated, an analytical definition, and containing triple junctions and straight grain boundaries [97]. Due to the venerability of this description, some complex techniques have been developed to improve the suitability of these diagrams for microstructural models [98], but a basic approach was deemed to be sufficient for this work.

The procedure for the generation of the GS models used in this work are shown in Figure 19. The base of the model is the generation of a Voronoi map in MATLAB around a number of quasi-randomly generated seed points. The points are not truly random (aside from the pseudorandomness of the generator itself) due to the imposition of a minimum separation condition between seeds, to ensure more uniformity in the resulting grain sizes and shapes. The relative size of the grains is controlled by the number of points generated. A square boundary is fit to the diagram and the vertices are output to a file. An Abaqus script was written using the programming language Python to read this file and create the finite element model. This script reads the vertex file and recreates the Voronoi diagram. The diagram is then used to partition a square area into grains, each of which is stored as a part. Each grain, and thus the model, is “quasi-2D.” That is, the plane area depicted in the model has a thickness of a few elements, but deformation in the out of plane direction is restricted and the model is treated as if it were 2D. The grains are assembled into a model and the matching faces of each pair of
grains are found and assigned interaction properties to define their cohesive interfaces. Each grain is uniformly randomly assigned an orientation, in accordance with the observed lack of preferred orientation seen in the EBSD pole figures in Section 3.3. The model is meshed with C3D8 8-node linear brick elements. Models contained approximately 12,000 elements, a number determined through mesh sensitivity analyses.

Figure 19. Model creation process. (a) Generation of Voronoi map around randomly selected seed points. (b) Creation of individual grains. (c) Assembly of grains and assignment of orientations. (d) Meshing.

This procedure is also used when creating models depicting the CG/UFG interfaces at the grain level. After the Voronoi diagram is generated, the script creates one or more ellipses
whose size and position are user-defined, as shown in Figure 20. Vertices of the diagram that fall within these ellipses are deleted from the figure, merging the Voronoi cells together. This process sometimes requires manual post-processing of the figure, as it can result in edges that “hang” into the interior of the CG and are nonphysical artifacts. Some effort was spent in an attempt to improve this process through the creation of a boundary representation scheme, described in Appendix A, which was applied to the figure to improve the geometrical manipulations in the procedure, such as merging cells. This scheme, which developed an algorithm to trace the edges of the Voronoi cells during the creation of the boundary representation, proved to be more complex than relatively simple manual post-processing warranted. This could, however, be an avenue for further study into improving procedural methods for generating microstructures.

Figure 20. Generation of CGs. (a) Voronoi diagram with elliptical CG regions defined. (b) Diagram with CGs added. Note the hanging edges in the lower CG.

In addition to the code written to generate these microstructures and turn them into finite element models, auxiliary programs were written to support and extend this functionality. For postprocessing of the models, another MATLAB program (Appendix B) was written to take output data from Abaqus analyses and plot the state variables at the grain boundaries, showing
the results as an animation or frame-by-frame. The figures generated by this program feature prominently in the next chapter.

4.6 Estimation of Constants

In order for the models discussed up to this point to be useful in any way, they must be used with realistic material constants. Unfortunately, many of the values associated with these models are not directly available for the specific conditions considered in this work or at all. A list of the parameters necessary for the models and their values is given in Table 3, which also indicates the constants that are not readily available in the literature and must be estimated through the techniques discussed in this section. Some of the techniques involve fitting models to experimental data while others are theoretical estimates available in the literature. Every effort has been made to use the most suitable values for the material constants in this work, but even where the approximation is rough it should be noted that these values are still suitable for qualitative comparisons and examination of important effects, which are the main focus of this work.
Many constants for the crystal plasticity model were available in the literature, such as the cubic elastic matrix, Burgers vector, Boltzmann constant, etc. The ones that were unavailable were obtained through curve fitting to experimental data. Referring back to Section 4.2, the constants determined in this manner were the initial slip system hardening rate $h_0$, the slip system initial strength $g_{c,0}$, and $g_{c,s0}$, a parameter used in the determination of the saturation stress. These constants were determined by matching the stress-strain curve obtained for the GS model to experimental stress-strain curves for conventional Al 5083 found in the literature. These curves were available for a variety of different temperatures, which enabled the high temperature simulations discussed in the next chapter.

To fit the experimental curves, these trial values were changed singly and used in the GS model without CGs until the stress-strain response of the model matched the experimental data, shown in Figure 21. In all such model fitting work, the question of uniqueness invariably arises. In response to this, it is noted that the constants varied in this study have distinct effects on the resultant stress-strain curve, which can be adjusted to match the different features of the
experimental data. In other words, a stress-strain curve represents many degrees of freedom, allowing for each of the relevant features to the model to be matched to the experimental curve. Therefore it is expected that the values for constants obtained through model fitting are suitable in the conditions modeled.

![Stress-strain curve](image)

Figure 21. GS models (dashed lines) fit to published stress strain curves for conventional CG Al 5083 at 293 K and 473 K [100,101].

A similar approach was used in the case of the LS models. In these models, the CG regions could be easily modeled with handbook values, but that the correct properties for the UFG region were unknown. However, the experimental data described in Chapter 3 could be used to extract this data, with the slight complication that it represents the composite behavior of both regions. Therefore, to extract the properties of the UFG region, a LS model with 9% CG ratio was compared to experimental results. The UFG regions were described with the Voce plasticity law given in equation (5) so the three parameters of this model ($\sigma_y$, $\sigma_s$, $\varepsilon_c$) were varied to match the stress-strain curves. The results of this procedure are shown in Figure 22. Again,
this could be also be done for higher temperatures where the experimental stress-strain curves were available.

![Stress-strain curves for 293 K and 473 K](image)

Figure 22. LS models (dashed lines) matched to experimental data at 293 K and 473 K.

<table>
<thead>
<tr>
<th>Property</th>
<th>293 K</th>
<th>473 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$ [GPa]</td>
<td>70</td>
<td>59</td>
</tr>
<tr>
<td>$\sigma_y$ [MPa]</td>
<td>275</td>
<td>70</td>
</tr>
<tr>
<td>$\sigma_s$ [MPa]</td>
<td>470</td>
<td>108</td>
</tr>
<tr>
<td>$\varepsilon_c$</td>
<td>0.0025</td>
<td>0.002</td>
</tr>
</tbody>
</table>

*CG curves from references [100,101].

Table 4
Material properties for UFG* Al 5083 in large scale model.

For the interface model, the unknown values are the elastic stiffness of the boundaries, $K$, their yield strength, $s_0$, and the components of the power hardening law, $a$ and $n$. The first two values are estimated in the manner described by Wei and Anand in their work, and the power law constants are estimated from experimental data. The estimates arrived at through these methods are analyzed and their accuracy is considered through examination of their effect on the model’s behavior.
Recall that the elastic stiffness matrix $K$ is composed of two components, $K_{N}$ and $K_{T}$. These values are estimated using the elastic constants of the bulk material as

$$
K_{N} = \frac{E}{g} \\
K_{T} = \frac{G}{g}
$$

where $g$ is the thickness of the grain boundary and $E$ and $G$ are Young’s and shear moduli [80]. Alternatively, this value can be thought of as the interfacial stress corresponding to one length unit of interfacial displacement, making it somewhat independent of the actual grain boundary thickness. In these simulations, $g$ is taken to be 1 nm and Young’s and shear moduli as 70 and 26 GPa respectively.

The elastic properties of the grain boundary region have been studied through molecular dynamics simulation and have shown reduced elastic constants in the vicinity of the grain boundary [102,103]. Other studies have shown increased elastic constants or some combination of increased and decreased constants in certain directions relative to the boundary, depending in part on the misorientation angle [104]. To determine realistic values for this property, the effect of changing this parameter was evaluated by adding a constant factor to equation (24) to vary the boundary stiffness between 70% and 150% of the bulk elastic constants, corresponding to the range of predictions in the literature. It was found that changing the interface stiffness in this range had an almost indiscernible effect due to the much greater influence of the grain interiors. Plastic displacement in the boundaries was almost identical for all values studied, except for some locations near the CG/UFG interface, one of which is depicted in Figure 23. Away from the boundary, it can be seen that the curves are the
same, with the only difference being the maximum value and rate of decay near the boundary. However, since these effects are quite small, equation (24) was used without modification.

Figure 23. Plastic displacement as a function of distance from CG/UFG interface along the highlighted boundary in the indicated direction.

The boundary yield strength was estimated by assuming that grain boundary plasticity is accommodated by the injection of dislocations from the boundary into the grain. The stress necessary for this to occur can be estimated by

$$s_0 = \frac{Gb}{l} = \frac{Gb}{cd}$$  \hspace{1cm} (25)

where $G$ is the shear modulus, $b$ is the Burgers vector, and $l$ is the dislocation segment length which is assumed to scale with the grain size. If the dislocation is confined within the grain, $l$ can be at most the grain size $d$. However, it is more generally some fraction of the grain size, that is, $l = cd$, where $c$ is the scaling factor between dislocation length and grain size [80]. For nano-sized grains, $c$ may be close to 1, but for the size of the grains studied in this work, it is likely to have a smaller value. Using the relationship between material constants, grain size, $c$,
and $\sigma_y$ given by Cheng, Spencer, and Milligan [105], $c$ was estimated to be between 0.1 and 0.2 for this grain size and yield strength which is in good agreement with values used in that work’s discussions. Another way to estimate this value is that it is proportional to the inverse root of the forest dislocation density (density of dislocations not on the primary slip plane) [59]. This implies that higher dislocation densities can increase $s_0$ although this effect is not considered here in the determination of this value.

Finally, the values of the constants of the grain boundary’s power hardening law ($a$ and $n$) are based on their values for the grain interiors. As a first approximation, the plasticity of the interface was taken to be described by the same constants as used for the grain interiors. The interfacial strength coefficient $a$ was extracted from the bulk stress-strain curve. The strain hardening exponent was also estimated from the experimental data, though values taken from more direct measurements were also investigated. Nanoindentation experiments showed a value of 0.4 for $n$ [106]. That this value was arrived at through nanoindentation is relevant to these experiments because the small size of the indenter tip compared to the grains means that this value is representative of only the grain interiors, separating out any contributions from the grain boundaries that would be present in bulk experiments. However, in this case a value is needed for the grain boundaries themselves, which would be much harder to extract through nanoindentation. It is expected that the increased dislocation density in the boundaries would result in increased hardening behavior in these regions. Therefore, it was rationalized that the value for $n$ in the grain boundary model must be higher than that of the grain interiors. It was found that models with a higher value of the strain hardening exponent, that is, a more linear plastic hardening effect, had increased plastic displacement in their interfaces attributable to an early increase in their displacement rate due to the low-strain compliance of higher $n$ models.
With these results in mind, a value of 0.05 was chosen since it is around the maximum value typically found in aluminum alloys [107].

![Graph](image)

**Figure 24.** Plastic displacement (a) and displacement rate (b) at the node with the highest displacement with variable \( n \).

These values were estimated in the same method for the high temperature simulations. Since this requires, for example, that the entire change in \( s_0 \) with temperature be encapsulated in the change in \( G \) for that temperature, which is probably not an accurate assumption. However, this is a starting point for further investigation and refinement in the area. Additionally, other values were not adjusted for the increased temperature such as \( \dot{\gamma}_0 \) or \( p \) or \( q \), simply because no good estimates could be obtained for their relationship with temperature. While these do need to be refined, these values are acceptable for the granularity of the comparisons made in this work since their effects are relatively small compared to the elastic constants, yield strength, etc. that were updated to the higher temperature.
CHAPTER 5
MICROSTRUCTURAL SIMULATIONS

5.1 Overview
In the previous chapter, models were developed to characterize the microscale deformation of a bimodal alloy. Due to the relative magnitudes of the features of interest in this material, models at two scales were developed. The LS models were useful for a general overview of the material’s behavior, without accounting for grain scale effects. These models are considered representative of the material and can be used in comparisons to experimental data. The smaller models, GS models, are useful for examining the unique effects that become relevant at small length scales. These models account for crystalline anisotropy of individual grains and joins the polycrystal together with grain boundaries, allowing for examinations of the interplay between the CG and UFG phases. Due to the small scale of these models and the fact that they are localized, without all the characteristics of bulk models, their comparison with experiments at larger scale is difficult. The multiple length scale approach is adapted to bridge the gap between the grain scale models and bulk scale mechanical behavior. In this chapter, the insights provided by these models will be explored.
5.2 Large Scale Models

The details of the approach in generating and assigning properties for this model was explained in Sections 4.5 and 4.6. These models were displacement loaded in plane strain longitudinal tension. The stress and strain contours in the large scale bimodal model are shown in Figure 25. It can be seen that stresses are concentrated in the UFG region, with relaxation regions occurring at the tips of the CGs. Strain is mostly concentrated in bands between CG regions, with the highest values occurring in the UFG matrix between closely spaced CGs. For a strain-limited failure, as is expected for this material, this would indicate damage initiation at these sites. These results show that the GS model, with its closely spaced CG regions, is a good tool for the study of damage initiation effects, even if it is not representative of the entire microstructure.
The features of the simulation are consistent with experimental observations of the fracture surface in tension, which showed a ductile type failure texture and load segregation between the two phases [54,108]. Recall the micrograph shown in Figure 8 which also showed failure occurring through CG regions, as opposed to interfacial delamination with the UFG matrix [54]. This is consistent with the model results, with failure originating in the high strain sites and transferring load to the CGs and the UFG matrix loses its load bearing capacity and the crack begins to propagate.

At high temperatures, the roles of the two regions appear to switch. This is attributable to the phenomenon shown in Figure 15 where conventional Al 5083 exhibits improved high
temperature strength compared to the bimodal alloys, so that matching the stress-strain curves requires a weaker UFG region. This is an admissible assumption since other observations of this effect have been on completely UFG material [33], thus reducing the likelihood of this phenomenon being the results of an interaction effect of the CG and UFG regions. The stress contours shown in Figure 26 illustrate this. This reversal in the stress-bearing regions of the microstructure is accompanied by a change in the previously described strain concentrations between closely spaced CG regions. At the higher temperature, these concentrations disappear and are replaced by a strain band that avoids the CG regions. Instead of occurring at CG/UFG interfaces, these high strain points now occur well within the UFG matrix. Based on this evidence, the path of failure at high temperatures is expected to progress mainly through the matrix, avoiding CGs entirely.

One may recall from Chapter 3 that fractographic evidence of a change in failure mechanism with temperature was not observed. However, the nature of this change may not present itself in the fractographic data. In both cases, the failure path progresses through the UFG matrix, with the difference being the role of the CGs. Since the fracture is mainly in the UFG region in both cases, the textures should look mostly the same. The fractography would not necessarily show the fracture path avoiding the CGs at the higher temperature. It should be noted that the CG region observed in Figure 8 was in a specimen tested at room temperature and was an uncommon feature anyway. This suggests that while failure may initiate at these CG/UFG interfaces and initially progress through CGs, the final failure may also propagate through the matrix alone.
Figure 26. Stress (MPa) and strain contours of LS model with 9% CGs at two temperatures.

The LS models were also used to investigate CG ratio effects. For this, models with 9%, 18% and 26% ratios were created, shown in Figure 27. As expected, increasing the CG ratio of the models reduced their strength and increased their compliance (Figure 28). Common between all of these models is the low stresses in the CGs compared to the UFGs. However, as the CG ratio increases the stress gradients in the material appear to become steeper as evidenced by the increased variation in the contours in the matrix. In these models it appears that there are more low stress regions in the UFG matrix of the model, segmented by the CGs. This function of the CGs, breaking up the stress bands in the matrix, is expected to be one of the mechanisms by which the CGs improve the material’s ductility. However, the strain contours shown in Figure 27 illustrate a possibly conflicting mechanism. In these contours, it
can be seen that increasing CG ratio can create points of high strain resulting from the UFG matrix being required to accommodate increased deformation.

Figure 27. Stress (MPa) and strain contours in LS models with various CG ratios.

Figure 28. Stress strain curves for LS models with different CG ratios.
The competition between mechanisms such as this could support the experimental findings of a saturation effect occurring with increasing CG ratio. However, the maximum stresses of the models shown in Figure 29 depict a very linear relationship. Similarly, examination of the stress in each model at a given strain produces a linear relation. Therefore, whatever is responsible for the experimental findings is not captured by this model. Thus, these simulations support works such as [56] which assume a linear relationship between the CG ratio and material properties. These linear relations have the added benefits of easy extrapolation to intermediate points and an ideological conformity with the view of bimodal materials as a type of composite. Further investigation of the saturation effect observed in this work is required.

![Figure 29. Maximum stress in LS models as a function of CG ratio.](image)

5.3 Grain Scale Models

The large scale models have shown the general behavior of the microstructure and have highlighted areas of interest that can be investigated in greater detail with the GS models. In
this section, these GS models will be examined. Additionally, the role of the crystal plasticity model will be analyzed in comparison with more simple descriptions to evaluate its effectiveness and to justify its added complexity.

In this section, the bimodal GS models developed in Section 4.5 are used to study the interactions of the CG and UFG phases. These models are loaded in longitudinal tension as shown in Figure 30. The left edge of the model is fixed and displacement is applied to the right face. All of the nodes in the model are fixed in the out of plane direction. Stress-strain curves were generated from these simulations from the displacement of the loaded edge and the sum of the reaction forces on the fixed edge.

![Figure 30. Longitudinal tension loading used for models in this section.](image)

5.3.1 Microstructural Effects

To examine elements of the GS model independently, the simulation was run using the crystal plasticity model and an isotropic model (with material properties from the large scale model) for the grain interiors, with and without the grain boundary model. Models without a grain boundary model have the grains rigidly attached, allowing for no interfacial deformation. The stress-strain curves from these trials are shown in Figure 31. It can be seen that inclusion
of the grain boundary model resulted in reduced stresses, owing to the added compliance of the grain boundary region when compared to rigidly attached grains. It is also apparent that the isotropic model consistently predicts a higher stress-strain response than the crystal plasticity description, due to the low strength exhibited by some crystallographic orientations that the isotropic model does not account for.

![Stress-strain curves for crystal plasticity (CP) and isotropic (iso) models, with and without grain boundaries (GB).](image)

Figure 31. Stress-strain curves for crystal plasticity (CP) and isotropic (iso) models, with and without grain boundaries (GB).

The stress and strain contours for the four test cases are shown in Figure 32 and Figure 33. The isotropic models of course show a more homogeneous stress distribution within the phases, the crystal plasticity model shows that the load is concentrated in a few favorably oriented grains in the UFG matrix. Inclusion of the grain boundaries has a small but noticeable effect on the contours. The strain contour without grain boundaries shows a largely unbroken high strain band between the two phases, indicating that they are more cohesive and deform as one unit. The inclusion of the boundaries in the model seems to distribute strain more evenly throughout the phases, especially the CG region.
Figure 32. Mises stress (GPa) for models (a) with crystal plasticity and grain boundary models, (b) crystal plasticity without grain boundaries, (c) isotropic grains with grain boundaries, and (d) isotropic grains without grain boundaries.
Figure 33. Maximum principal strain for models (a) with crystal plasticity and grain boundary models, (b) crystal plasticity without grain boundaries, (c) isotropic grains with grain boundaries, and (d) isotropic grains without grain boundaries.

The plots of grain boundary displacement in Figure 34 show that the sites of high interfacial displacements were generally the same in both the crystal plasticity and isotropic models. These sites lay on the CG/UFG interface, with their maxima on the interface and decreasing with distance into the UFG matrix. The two models did not show the same magnitude of displacements, however. That is, while boundary motion tended to be concentrated in the same areas in both models, the actual amount of deformation in these areas was not the same. The crystal plasticity model tended to distribute lower amounts of deformation across more sites, while the isotropic one produced more concentrated distributions.
Figure 34. Total plastic displacement at grain boundaries (nm) in (a) crystal plasticity model and (b) isotropic model. Some displacement concentration sites are highlighted with red circles for comparison.

These results show that in the UFG region, several favorably oriented grains experience high strains. To accommodate this, the surrounding grains, which are oriented in less compliant configurations, experience high stresses. For a strain-based failure criterion, it is expected that the intergranular type fracture observed in experiments would nucleate in these high-strain grains. An intergranular-type failure mechanism could initially operate independently of a grain-boundary based failure path, with the two modes combining for final failure behavior. The competition and interaction between inter- and intragranular failure cases would of course depend on the specific failure criteria for each case. Comparison of the two plots in Figure 34 shows that the initiation of grain boundary failure is not dependent on the crystal orientation, since the sites of high deformation are similar in both plots. This suggests that grain boundary failure mechanisms may not be a direct result of grain deformation, but rather an independent event that is influenced by the configuration of the microstructure at the grain level.
This model also highlights the role that the grain interfaces play in the material’s deformation. Their inclusion serves to distribute strain more evenly across the microstructure, reducing concentrations and delaying the onset of failure, in addition to serving as failure initiation sites under some possible crack propagation cases. This suggests that engineering efforts directed at the properties of the grain boundaries could be utilized to improve the mechanical response of this and similar systems. Finally, it emphasizes the interactions between the grain interiors and boundaries over the course of loading and shows that both effects must be considered for an accurate microstructural representation. The main drawback of this description of the grain boundaries is that they are treated as distinct entities from the grain interiors, which is of course not the actual case where a more gradual transition in properties might be expected. However, at the scale of the grains in this model which are two orders of magnitude larger than the grain boundary thickness, depicting the boundaries as sharp, well defined interfaces is expected to be reasonable.

5.3.2 Temperature Effects

The models were extended to the high temperature regime using the properties extracted from the LS models as described in Section 4.6. These results are shown in Figure 33. These models show a much more even stress distribution than at room temperature, but with the highest stresses occurring in the CGs as opposed to the UFGs. However, the regions of highest stress continue to be at the CG/UFG interface, but now on the CG side. Conversely, the highest strains are located mainly in the UFG region. While some of these high strain areas are located at the phase interface, others are located between UFGs. This correlates with the observations
of the LS models at high temperatures and may indicate a change in the location of failure initiation at elevated temperatures, even if the final failure path is largely unaffected.

Figure 35. Stress (GPa) and strain contours in high temperature bimodal model.

By matching the material properties of the UFG region to the experimental data, the observed weakening of the UFG region is included in these material properties. While this should empirically represent the state of the microstructure, a small experiment can shed light on this weakening mechanism. By assuming that at the sub-grain level the CG and UFG regions are identical, we can determine whether the weakening effect occurs at this scale. To investigate this, the bimodal model was run using only the crystal plasticity properties of the CG region in both phases, the results of which are shown in Figure 36.
These models show very nearly the same strain distribution as the model with different properties in the two phases. The strain concentration points are the same in the two models, with a reduction in magnitude for the model with only the CG properties. However, the stress contour shows a significant increase in stress borne by the UFG region, in addition to the stress concentration points at the interface. The stress strain curve for the two models is shown in Figure 37. It can be seen here that the CG only model shows significantly higher strength than the model that has been matched to actual experimental data. Included for comparison are the experimental results from the 30% CG material tested at the same temperature (the bimodal GS model is about 40% CG by area). This comparison shows that inclusion of distinct CG and UFG properties brings the overall behavior of the model closer to the experimental results than treating both phases equally does. This implies that the effects considered in this model alone are not enough for the weakening behavior to arise without the use of ad hoc adjustments. In this simulation the two phases were assumed to be identical at the crystal level, with the differences arising in number of grains and grain boundaries arising in each region. Since this did not produce the results observed through other means, it is concluded that, in addition to
any grain-level differences, CG and UFG regions must have distinct crystalline properties to account for the observed experimental phenomenon.

![Stress strain curves](image)

**Figure 37.** Stress strain curves for models with CG and UFG properties for their respective regions and with on CG properties for both. Experimental results from 30% CG tests at 473 K are included for comparison.

### 5.4 Grain Boundary Effects

Experimental measurement of grain boundary sliding can be done, though it relies on the mathematical relationships of the geometry of the boundaries and components of the deformation vector (see Section 2.3). That is, while the component of deformation parallel to the direction of tension is desired, the measurements of deformation in other directions are often more easily obtained and are then related through equations such as equation (4) to obtain the result. However, measurement of grain boundary deformation in the finite element models is more straightforward. In this case, the UINTER subroutine knows the components of deformation in the grain boundary coordinate system (Figure 17) and also the rotation of this
system relative to the global coordinate system. Using this information, it is a straightforward process to obtain the deformation in the direction of tension, essentially measuring $u$ in equation (3) directly. In this way, equation (2) can be used to calculate the contribution of GBS to total strain, $\xi$.

Using this type of analysis, the role of the grain boundaries in the material’s deformation can be evaluated by examining the effects of the properties of the boundaries on the contribution of GBS, as well as the overall stress-strain response. This was done using the polycrystalline model (Figure 18b) with high temperature (473 K) properties as developed in Section 5.3 with several different sets of parameters for the grain boundary model. The nominal values for grain boundary model were estimated using the methods described in Section 4.3 but, as shown in Figure 38, this resulted in a grain boundary strength $s_0$ too high to produce an appreciable contribution from the grain boundaries. Therefore, it is believed that equation (18) significantly overestimates the grain boundary yield strength, at least at elevated temperatures.
Figure 38. Stress strain curves for models with a variety of grain boundary properties. UFG curve as extracted from LS model included for comparison.

The simulation was then run with weaker grain boundaries, both with a lower yield and a softer plastic region and with only the lower yield and the plastic constants as originally estimated. Lowering the yield strength of the boundaries did increase their involvement in the simulation, with the effect being much more pronounced when the plastic region of the boundaries’ traction-displacement relationship was reduced. This can be seen as an overall weakening of the stress-strain response in Figure 38. Comparison of the two curves corresponding to models with softer grain boundaries indicates that softening of the plastic region has a much more pronounced effect on the material’s overall behavior than just a lowering of its yield point. This suggests that a sufficiently low yield point is necessary to involve the boundaries in the deformation process, but that the greatest effect on the overall strength of the material comes from a softer plastic response after yield has been reached. However, there remains a large discrepancy between the model with the weakened boundaries and the UFG properties according to the LS model. The grain boundaries were not able to be
weakened enough to match this curve, so this requires that other effects be at play in the
determination of the UFG region’s high temperature strength reduction.

These results also show that, in order for the grain boundaries to have any significant
contribution to the material’s behavior, they must be softer than the grain interiors and also
softer than they were initially estimated to be. The original estimate of the boundaries’ yield
strength arises from the assumption that grain boundary plasticity occurs when injection of
dislocations into the grain interiors is possible. However, the only thermal dependence of this
model is from the change of the shear modulus with temperature which is apparently not
sufficient. This estimate does not account for additional diffusion controlled processes which
can become active at temperatures greater than \( \sim 0.5 \ T_m \) and can result in plastic deformation at
lower stresses [59]. These effects should be considered in order to develop more realistic
descriptions of the grain boundaries’ behavior.

A lower yield strength and consequent increase in grain boundary activity also had a drastic
effect computationally. Models with more grain boundary recruitment became much harder to
solve, requiring hundreds of load increments over the course of the simulation (compared to 31
increments for the model without boundaries and 36 for the one with nominal properties). This
had the effect of greatly increasing solution time and also usually caused the simulation to
abort prematurely when the time step became too small. This is why some of the curves in
Figure 38 and the other figures in this section are shorter than the curves with minimal grain
boundary recruitment.

Figure 39 shows the contribution of GBS to the total strain for different grain boundary
behaviors. For the model with the original estimate of grain boundary parameters, this value
was very small. It peaked at 0.3% around a strain of \( 10^{-3} \) and then decreased to near zero as the
total strain increased. It can be seen that the grain boundaries are quickly recruited early on in the deformation. The contribution levels off and begins to slowly decrease as intragranular strain begins accounting for more of the total. When the plastic region of the boundary is softened, the GBS contribution increases substantially, as would be expected from their increased compliance. Experimental measurements of GBS contributions at a total strain of 12% have shown that about 25% of the strain is attributable to the boundaries [109]. The curve for the model with the softer plastic region roughly corresponds to this value, though at a much lower strain. However, this indicates that, compared with the grains themselves, the grain boundaries must have a significantly reduced yield point as well as a softer plastic response. In all trials, the contribution of GBS to the total strain peaked at a relatively small overall strain and then began to decrease slowly. This suggests that, for boundaries that are softer than the grains themselves, their recruitment occurs early on based on their comparatively lower yield strength. The boundaries can accommodate low total strains, but they become saturated quickly and the continued deformation must occur inside of the grains and the effect of the boundaries on the overall curve becomes negligible at higher strains.
Figure 39. GBS contribution to total strain for models with softened grain boundaries.

While it is apparent that under the right conditions the grain boundaries can play a large role in the material’s behavior, it does not appear that this effect alone can account for the UFG’s experimentally observed decrease in strength. Figure 40 compares the models without grain boundaries and with softened grain boundaries to the UFG properties extracted from the large scale model. In the elastic region, the stress reduction accounted for by the grain boundaries quickly increases and peaks around 40% of the total stress difference shortly after the UFG model enters the plastic region. Once plasticity is achieved in all the models, the grain boundaries account for less and less of the stress difference. The rate of decrease is slower than its increase and apparently linear with a slope of approximately -25 %/%. This rate is partly affected by isotropic Voce plasticity model of the UFG curve, which shows almost perfect plasticity, while the crystal plasticity model of the other two curves continues to harden. However, the main factor is that the model which includes grain boundaries gradually approaches the curve of the model without them as the boundaries become saturated in their
ability to accommodate strain. Assuming the linear trend continues (which it probably will not), the contribution could approach zero in as little as 2% total strain, a rather small amount for a ductile material at an elevated temperature.

![Stress-strain curves showing effect of inclusion of grain boundaries compared to UFGs. X’s indicate the sample locations for plot (b), the percent of stress reduction attributable to grain boundaries. The values corresponding to the elastic and plastic regions of the stress-strain curves are demarcated.](image)

Figure 40. (a) Stress-strain curves showing effect of inclusion of grain boundaries compared to UFGs. X’s indicate the sample locations for plot (b), the percent of stress reduction attributable to grain boundaries. The values corresponding to the elastic and plastic regions of the stress-strain curves are demarcated.

The prevalence of grain rotation under these circumstances can also be investigated with these models. The crystal plasticity subroutine tracks the rotation of the slip systems through increments to the systems’ normal and direction vectors. Since several slip systems might share the same normal, it was generally more useful to consider the systems’ directions. In Figure 41, the magnitude of the increment of the slip direction vector summed over all slip systems is plotted. Through this analysis for models with and without a grain boundary description, the interaction of the two deformation modes can be analyzed. It can be seen that the sites of high activity are similar in both cases, but that the model without grain boundaries shows much higher values. This indicates that the freedom of movement afforded by the grain
boundaries does not translate into greater rotational activity, and in fact produces the opposite effect. The two motions, grain boundary sliding and grain rotation, seem to compete and an increase in one necessitates a decrease in the other.

![Figure 41. Magnitude of grain rotation summed over all slip systems in models (a) with and (b) without grain boundary descriptions.](image)

To this point, the discussion has only been on models at high temperature. Now, the effect of temperature on grain boundary sliding and rotation will be considered. Much more grain rotation was noted at the lower temperature, as shown in Figure 42 (note the increased range of the scale compared to Figure 41). However, this figure also shows that the distributions are much the same and that mainly the magnitude changes. This is similar to the observations of grain rotation with and without grain boundaries. As the model becomes more constrained, in this case because of less compliant grains, more rotation occurs. Additionally, the grain boundary sliding contribution decreased to almost negligible amounts, a maximum of about 1% early in the elastic region and a steady decrease from there. Therefore, this is similar to the “no grain boundary” scenario examined above, promoting grain rotation. This shows a very large increase in GBS activity at higher temperatures, which could account for the UFG material’s decrease in strength with temperature.
5.5 Failure and Fracture

For the models examined up to this point, the interfacial failure criterion has been deactivated in order to ensure comparable results across all models. In this section, this feature will be activated and used to study the crack propagation path. To do this, a value for the failure criterion must be assigned. This could be selected based on experimental ductility measurements, but this is not the purpose of this section. Making a selection based on experimental data would be necessary for a predictive type model, but here the goal was to study the failure mechanisms qualitatively. For this it is much more effective to decide on a failure criterion with effective computational properties. Here, a value of 0.5 nm of plastic interfacial was used for interfacial failure. This value served to focus on the failure behavior without extraneous load steps while still providing a good representation of the pre-failure elastic-plastic behavior.

For these studies the bimodal GS model was used, shown loaded in longitudinal tension is shown in Figure 43. In this figure, several of the typical features of these results can be
observed. The stronger UFG region experiences higher stresses than the more ductile CGs. As the load is applied, the CGs begin to yield first, as expected. The discrepancy between the yield points of the CG and UFG regions causes plastic displacement to initiate at the interfaces between the two regions. These regions of high interfacial displacement then begin to radiate outwards, into the matrix. Thus, for this description, it appears that intergranular cracks have preference for propagation through the matrix as opposed to running along the CG/UFG interface. These sites also create regions of high strain in the CG as shown in Figure 43, so there is another possibility of crack propagation into the CG. It appears that grain boundary deformation is initially the preferred mechanism, with high levels of strain in the CG occurring later in the simulation. Thus, these results indicate crack propagation initially around UFG boundaries in the matrix with movement into the CGs as part of the final failure of the material.

Figure 43. Evolution of deformation in the microstructure. (a) Stress contour (GPa) at 5% total strain. (b) Total plastic interfacial displacement (in nm) initiating at CG/UFG interface.
between 2 and 5% total strain. (c) Plastic strain ultimately transferred to CG region between 2 and 5% total strain. One grain hidden to show interfaces.

Figure 44 shows the progression of the crack path through the matrix. Again, it can be seen that failure initiates at the CG/UFG interface and begins to propagate laterally into the UFG matrix. It joins with another crack propagating from the other CG, eventually bridging the matrix between the two CGs. At this point, the CG regions begin to bear large amounts of strain, concentrated at the crack tip at the CG/UFG interface.

![Crack evolution](image)

Figure 44. Crack evolution in longitudinal tension at 293 K at simulation time steps $t$.

When these tests were repeated using the high temperatures properties for the model, the crack initiation site was observed to move from the CG/UFG interface to the matrix. In Figure 45 it can be seen that two of these crack initiations appear in the matrix in quick succession and
begin to grow laterally through the matrix. This supports the conclusions drawn from the LS models of changes only in the crack initiation site but not the propagation path. This shows a change in the role of the CG/UFG interfaces with increasing temperature as cease to be crack initiation sites and could serve more to blunt the propagation of cracks moving towards the CG from the matrix.

Figure 45. Crack evolution in longitudinal tension at 473 K at simulation time steps $t$.

These failure models of course only consider intergranular failure, which is a limitation when considering the prevalence of intragranular fracture mechanisms in the longitudinal direction that was observed in the fractographic study. These models have been able to be interpreted in a way consistent with the experimental observations, suggesting that the fracture
follows roughly the same path but is partially or entirely in the grains’ interiors. These models could be improved in a significant way through the inclusion of intragranular failure mechanisms, but this was ultimately outside of the scope of this project. The other improvement that could be made to this failure model is based around the somewhat arbitrary failure criterion. In future work, it is hoped that this value will be obtained experimentally or otherwise. With these considerations in mind, it is believed that this model represents a qualitative step in the study of the grain scale failure of bimodal metals.

5.6 Effects of Loading

One advantage of the modeling techniques used in this work is that they can easily be extended to other loading conditions. In this section four conditions will be investigated using the bimodal GS model. They are shown in Figure 46, along with the longitudinal tension which has been studied up to this point. Initial investigations had the interface failure disabled so that two models could be compared consistently at any level of loading. Later analyses were re-run with $\bar{\gamma}_{\text{fail}} = 0.5 \text{ nm}$ to look at crack initiation and propagation paths. In this section, the results of these simulations will be considered in the context of some of the anisotropic effects observed experimentally.
Figure 46. Loading conditions investigated. In biaxial tension, faces were constrained only in the direction of the applied displacement on the opposite face. Shears were constrained to remain parallel.

5.6.1 Transverse Tension

When loaded in the transverse direction, the upper CG in the model bears much of the strain in the model, effectively shielding the lower layers from deformation. Examination of the interfacial displacements (Figure 48) showed that in both cases, the boundaries showing the greatest deformations are oriented perpendicularly to the direction of tension, indicating that the interfacial deformation occurs mainly through the normal mechanism. Total deformation was greater in the longitudinal direction and it can be seen in Figure 48 that the sites of the greatest values correspond to CG/UFG interfaces. Comparison of Figure 47 and Figure 48 shows much higher strains in the interfaces than in the grain interiors.
Figure 47. Stress (GPa) and strain contour plots for transverse tensile loading at 5% total strain.

Figure 48. Plastic interfacial displacement in (a) longitudinal tension and (b) transverse tension. Interfacial deformation occurs mainly on faces oriented perpendicular to the loading direction.

Recall that in longitudinal tension, cracks were predicted to initiate at the CG/UFG interface and propagate laterally into the UFG matrix. While cracks in transverse tension were still observed to propagate roughly perpendicular to the loading direction, they initiated in the UFG matrix close to the CGs as shown in Figure 49. Later, initiation also occurred at the CG/UFG interface and continued along the interface to join with the crack in the matrix. These cracks initiated at appreciably higher strains than in the longitudinal direction. In experiments
the transverse direction exhibited significantly less ductility, seemingly in conflict with these results. However, these models do not account for intragrain failure mechanisms, which were observed to be significant in longitudinal failure whereas more grain-boundary type failures were observed in the transverse failure surface [54,108]. It is expected that the speed of crack propagation along grain boundaries is higher than in the CG interiors, so that while the analysis in the longitudinal direction stops just as the crack would begin to enter the CG (which will have to fail before the material can fail completely), final failure is rapidly occurring in the matrix of the material loaded in the transverse direction. These observations agree well with failure mechanism predictions made by fractographic analysis.
In experimental tensile tests, the ultimate strength of the material was reduced by about 25\% [54]. A small decrease in strength was observed in the models’ stress-strain curves (Figure 50) in the initial plasticity region, but not enough to account for the entire experimentally observed effect. The difference becomes negligible at higher strains, implying that the difference in strength in the simulation is attributable to the increased load on the upper CG initially, before the UFGs begin to bear more load.
Figure 50. Stress strain curves for models tested in longitudinal and transverse tension.

5.6.2 Shear

The stress and strain contours of models tested in longitudinal and transverse shear are shown in Figure 51. In both cases, the UFG region experiences the highest stress but in longitudinal shear, the stress is spread throughout the UFG region and is concentrated in a narrow band in the middle of the region in transverse shear. Therefore, in shear loading, it appears that the CGs serve to blunt high stress bands. A similar effect can be seen for the strain contours in Figure 51, in the transverse loading, the strain is confined to the CG regions. However, in longitudinal shear the bands have begun to join through the UFG region. This suggests that the material inhomogeneity introduced by the bimodal microstructure allows for the segregation of both stresses and strains depending on the loading configuration.
Figure 51. Stress (GPa) and strain contour plots for (a) longitudinal and (b) transverse shear loading at 5% total shear strain. Contours for models without interfaces considered were very similar.

There was very limited interfacial deformation in both shear cases, so no interfacial failure was observed over the course of the simulation. In these loading cases, it was observed that many more interfaces were experiencing compression than in the tensile cases. Since the interfacial model used here treats all compressive loads as elastic (that is, \( s_0 \) in compression is infinite), this description of grain boundaries could not play much of a role in these cases. This resulted in very low plastic displacements in both shear loading cases, though what very low amounts did occur initiated at a CG/UFG interface. A better description of compressive behavior is necessary if the role of grain boundaries in these configurations.
5.6.3 Biaxial Tension

In biaxial tension, both stress and strain contours (Figure 52) are largely affected by the inclusion of a grain boundary model. Without them, the contours are quite homogeneous with a uniform strain across both regions and clearly showing the outlines of the CG and UFG regions when plotting stress. However, when grain boundaries are considered, both contours become much more disjointed. Slightly higher strains are observed in the CG regions, with concentrations at the CG/UFG interfaces. The stress contour shows that the stress fields initiate at the grain boundaries and move towards the center of the grain.

Figure 52. Stress (GPa) and strain contours in biaxial tension at 1.3% strain in each direction, with consideration of grain boundaries (a) and without (b).

These observations indicate that in this loading configuration, the grain boundaries play a large role in the deformation of the model. They are recruited early in the load step and serve...
to absorb a large portion of the deformation induced on the model, indicated by the larger strains in the model without interfaces. As shown in Figure 53, this results in very large pressures and displacements at the interface compared to the grain interiors. In these models it appears that damage would initiate in the UFG region and possibly on some of the CG/UFG interfaces. The largest values for both interfacial stress and strain are mainly located on boundaries, both in the matrix and between UFGs and CGs, that are parallel to the CGs.

Figure 53. Interfacial plastic displacement (a) and stress (b) in biaxial tension at 1.3% strain in each direction.

Failure occurs very rapidly in this case, illustrated by Figure 54. Several cracks initiate almost simultaneously at different points in the matrix. The interfaces rapidly fail, and complete failure of the region is achieved rapidly after the cracks' first appearance. Failure mainly occurs on interfaces in the matrix parallel to the CGs, but some perpendicular branches are also observed, creating a crack path that is angled across the UFG region. This load case involves the GBs to a much greater extent than the others studied because the interfaces are beings simultaneously loaded through both their normal and transverse mechanisms simultaneously.
Figure 54. Crack evolution in biaxial tension at simulation time steps $t$. Interface failure first occurred at 0.51% strain. The crack spanned the model by 0.63% strain ($t = 0.177$).

This section has provided a quick overview of some of the capabilities of this model. Using these tools, alternative loading conditions were able to be investigated relatively quickly and conclusions about their deformation and failure could be drawn. Of course, experimental validation of these results are needed, but the models provided some insight into the anisotropic effects influencing the material’s deformation.
CHAPTER 6
CONCLUSION

6.1 Summary
Throughout this work, the mechanical behavior of bimodal materials, specifically Al 5083 has been considered. Fabricated through cryogenic milling techniques, this material exhibits greatly improved strength compared to conventional Al 5083 while maintaining some of the benefits of aluminum alloys such as light weight. In order for this material to be used to its fullest extent in design applications, a thorough understanding of these strengths, as well as its shortcomings, must be achieved. To this end, this work has investigated the behavior of this material under a variety of conditions using mechanical testing techniques, microscopic observations, and finite element methods.

A full-factorial experiment was designed and implemented to determine the effects of strain rate, specimen size, anisotropy, CG ratio, and temperature on a bimodal Al alloy in uniaxial tension. To accomplish these tests, custom small scale tensile specimens were designed and validated using FEA. Through stress-strain data collected during the tests as well
as post-test data gathered though fractography and microscopy, the following conclusions were drawn:

- Increasing the CG ratio of the material was found to increase its ductility and slightly lower its strength. The effect of adding CGs appears to become saturated at some point as there was little difference in the strength and ductility of the 20% and 30% CG materials.

- The material is anisotropic and exhibits drastically reduced strength and ductility when loaded in the transverse direction. The fracture surface between these two directions is also noticeably different.

- In the transverse direction, increasing CG ratio actually serves to increase the material’s strength. This is believed to be due to the material’s failure when loaded in the transverse direction being limited by ductility rather than strength.

- The material’s strength decreased and ductility increased as temperature was increased from 293 to 473 K. At 473 K, the strength of this material is less than that of Al 5083 at the same temperature.

- EBSD analysis failed to find a significant amount of grain growth in tests conducted at high temperature compared to tests at room temperature.

The results of these tests highlighted some of the complexities associated with the material’s microstructure, providing the motivation for the simulation aspect of this work. To address these issues, procedurally generated finite element models were employed. Two models were developed: a large scale one for a holistic overview of the bimodal microstructure and a grain scale one for a detailed study of the interaction between the CG and UFG phases. In order to represent the grain level effects in the smaller scale model, crystal plasticity and
grain boundary modeling methods were applied and fit to experimental data. This has produced, for the first time, material properties useful for crystal plasticity and grain boundary simulations for both CG and UFG Al 5083 at room and elevated temperatures. Simulations using these models showed:

- At room temperature, sites of high strain occur between closely spaced CGs. The change in mechanical properties with increasing temperature moves these sites into the UFG matrix.

- Unlike the experimental data, the simulations showed a linear decrease in ultimate strength with increasing CG ratio.

- In longitudinal tension, sites of high interfacial deformation occurs at the CG/UFG interface due to the boundary accommodating the mismatch in mechanical properties between the two phases. Cracks are expected to initiate at these locations and propagate laterally through the matrix. At higher temperatures, the initiation sites move into the UFG matrix but continue to propagate laterally.

- Simulations showed evidence of increased grain boundary activity with temperature. The fraction of the total strain attributable to GBS could be extracted and showed that as the model was loaded, the fraction increased rapidly and then began to decrease.

- Inclusion of the grain boundaries in the model can account for some, but not all, of the stress difference between CG and UFG materials at high temperature. The role of the grain boundaries appears to decrease at higher strain levels.

- The models were used to investigate microstructural loading and failure patterns in loading conditions other than longitudinal tension.
These simulations were used to investigate and examine some of the experimental observations. These models showed the distribution of roles between the two phases according to their properties and how that distribution changes with temperature. The fracture process was examined and correlated with fractographic observations. While the models were useful for these somewhat qualitative descriptions and comparisons, they need to be improved for more quantitative work. The models used need to be extended to account for more effects influencing the material’s overall behavior.

6.2 Contributions

The experimental results of this work provide a consistent baseline, which was lacking in the literature, for the comparison of the studied effects in this material. This work has added to the understanding of how the mechanical properties of bimodal materials, this alloy in particular, respond to changes in their testing conditions, allowing for their strengths to be more fully recognized in design applications. In the collection of this data, a method for small scale tensile tests was illustrated, from analysis of a custom specimen design to considerations associated with the actual data collection.

A method for the procedural generation of bimodal microstructures is presented and a boundary representation scheme is outlined. These two features provide a starting point to further computational investigations into bimodal and similar microstructures. Additionally, the constitutive equations used here have been adapted to this problem and should be easily extensible further to other similar investigations. In this way, this work has demonstrated in some detail a method for computational modeling and analysis of the grain level effects in bimodal microstructures. This work has also produced appropriate crystal plasticity and grain
boundary material properties for both CG and UFG Al 5083 at two temperatures. This is expected to be useful in further simulation studies on this material system, as well as provide a basis for the development of these properties for similar materials.

Together, these results provide insight into the behavior of bimodal materials and other inhomogeneous microstructures. The experiments have shown the presence of complex and interacting effects that may sometimes result in a UFG material behaving in ways unexpected of its CG counterpart. The simulations have provided insight into some of these occurrences, as well as providing information that is difficult or impossible to obtain experimentally such as crack initiation and propagation paths or the specific roles of grain boundaries in the deformation process. It is expected that these findings will contribute to the understanding necessary to properly exploit the strengths of this material so that its full potential can be realized.

6.3 Future Work

This study provided a basis for the exploration of the effects present in this material. Larger and different parameter ranges than those used in this work should be investigated in order to have a broader understanding of this material’s behavior under all conditions. Other possible effects which were not considered in these experiments, such as creep, should be examined.

In parallel with an extended experimental study, the constitutive models used for the simulations should likewise be expanded to capture a more complete depiction of the material’s behavior. Some of the shortcomings of the current crystal plasticity and interface descriptions have been noted in the text. The determination of the constants for these models can also be improved upon from the methods presented in this work. Additionally, the
procedurally generated models themselves can be improved. Appendix A provides some information on a possible starting point for this undertaking.

Both the experimental and simulations techniques developed in this work should be able to be extended to other materials with bimodal or otherwise inhomogeneous microstructures. The small scale tensile specimens could be useful in other areas where specimen size is limited by other experimental considerations. The models developed for this problem could also be easily extended to other testing conditions or material systems. Therefore, it is hoped that the work presented here will provide a useful building block for future research in other areas.
APPENDIX A

BOUNDARY REPRESENTATION FOR MICROSTRUCTURE MODELS

A.1 Overview

The method used to produce the procedurally generated models used in this work is described in section 4.5. The main advantage to this approach is its relative simplicity. However, it has some disadvantages, especially when it comes to the generation of the CGs. This process can produce unwanted features, such as grain boundaries that are disconnected and “hang” into the CG region, which must be cleaned up manually before the model is imported into the finite element software. This method of model generation also affords relatively little fine control over the resulting CGs.

To address these issues, a boundary representation method was developed. The representation stores the data necessary to construct the microstructural model hierarchically, from the vertices making up each grain’s polygon to the grains themselves. This allows for much better control over the model generation process. This representation scheme will be explored here. It was developed in MATLAB since that was the software used to generate the original Voronoi diagrams, but the methods discussed are general and should be able to be implemented in other environments if necessary. These methods were developed as a side project during the work that makes up the main text. As such, it was completed relatively late in the development of the project and is not implemented in the models in the main text. However, it is expected that this information will be useful for future forays into this area.
A.2 Representation

The only geometric information resulting from the model generation process in Section 4.5 is a list of edges in the model defined the coordinates of each of their endpoints. This is sufficient for creating the model in Abaqus, but it was quickly realized that a different approach was necessary in order to provide the control over the model’s features that was desired. The previous approach of obtaining the seed points, creating the map, and fitting it to a square was preserved. After this point, the available data were reorganized into the boundary representation outlined in Figure 55. This proved to be challenging, since this representation had to be built from the bottom up to fit the existing geometry.

![Diagram of Ustruct, Grains, Seed, Edges, Vertices]

Figure 55. Boundary representation of a microstructure model.

Vertex objects were created for each of the vertices in the original model. Then, using data from the model generation process, edge objects were created between corresponding vertices. Next, these edges were organized into grains with each grain corresponding to one cell of the Voronoi map. Each grain also contains the coordinates of its seed point. It turned out that organizing these edges into grains was difficult, although the correct solution is quite obvious from inspection. The procedure to do this (the “NASCAR algorithm”) is described in the next section.
Since each feature of the microstructure now contains information about its constituents, operations are greatly simplified. The length of edges and their angle with other edges can easily be queried. Merging grains to create CGs is also easier, since it can be quickly determined what geometry is shared by two grains.

A.3 Constructing Grains

Although the edges corresponding to a grain’s seed point are obvious from inspection, as seen in Figure 56, it was not so trivial to find these edges automatically when building the boundary representation. Various methods, such as selecting the maximum number \( n \) of closest vertices such that the convex hull of these points does not contain any other points, were tried with limited success. This led to the development of the so-called NASCAR algorithm (since the algorithm makes turns in the same direction until it ends up back where it started, like race cars going around a track).

![Grains and corresponding seed points.](image)

This algorithm relies on relative direction of a vector with respect to another being given by the sign of their cross products. Thus, when presented with the choice of which edge to take
when making a circuit of the grain’s boundary, the circuit will eventually be correctly completed if the edge which gives the same sign for the cross product is consistently chosen (i.e., only left or right turns are made). The first edge and direction is chosen as shown in Figure 57. A vector is drawn from the grain’s seed to the nearest vertex. Because of the Voronoi map, this point must belong to this seed. From the figure, it can be seen that taking the cross product of the green vector with the blue vectors corresponding to the edges leaving this point produces two results which have the same sign and one with a different one. This must always be the case if the green vector is inside the grain. A special case is encountered if the vector corresponding to the edge that does not belong to the grain is parallel to the green vector, resulting in a cross product of zero. In this case, the sign of either of the non-zero cross products can be chosen arbitrarily.

![Figure 57. Choosing the first edge and direction. The sign of the cross product of the green vector with one of the blue vectors is different from the other two.](image)

The direction (sign) of the cross product chosen in this manner is stored. Now, the algorithm creates a vector corresponding to the edge it is traveling along. At the other end, it can either go left or right (blue arrows). It chooses the edge which gives the same sign of the cross product as before. In this manner, the algorithm continues to make turns in the same
direction, storing the edges that are chosen, until it reaches the first point. The list of edges that bound the grain corresponding to this seed is now known and the boundary representation for this grain can be constructed.

![Diagram](image)

Figure 58. The next edge is chosen, based on which blue vector gives the same cross product sign as the previous step.

One requirement of this algorithm is that all of the grains be closed. However, when initially generating the square-bounded Voronoi map, grains that intersect the square do not have an edge on the boundary. This is required for model generation in Abaqus, where the square boundary is generated first and then partitioned into individual grains. Therefore, a function to close all of the grains had to be constructed. This function travels around the vertices on the boundaries of the model, creating edges to close the grains. The identifiers for these edges are stored separately so that they can be excluded from being written to the Abaqus input file, maintaining compatibility with the existing script.

A.4 Creating CGs

Instead of deleting vertices as in the previous implementation, UFGs are merged to create CGs. A function was written that removes any common edges between two grains from the model.
new grain is created with all of the other edges from both grains. The seed point for this new grain is just the average of the two individual grains’ seeds. This could be improved by implementing some kind of area-weighted average to keep the seed closer to the center of the larger grain.

To create a CG, an ellipse is again generated. All UFGs whose seed falls within this ellipse are merged, as in Figure 59. This solves the problem of hanging edges, but small edges and sharp angles still exist. However, the new description of the model makes it much easier to obtain statistics about a particular grain’s boundaries. For a particular grain, with its list of associated edges, it is very straightforward to obtain information about lengths and angles. This data can be used to develop some kind of metric for the “goodness” of the grain boundary and then algorithmically optimized.

![Figure 59. Examples of CGs generated by merging UFGs. Hanging edges are not present, but sharp angles and small edges remain.](image)
APPENDIX B

GRAIN BOUNDARY VIEWER

Abaqus does not natively facilitate viewing the simulation results for many interfaces simultaneously. One can view the interfaces in the model directly by hiding one of the contacting parts, but this was not a good solution for the models used in this work. To aid in the understanding of the behavior of the grain boundaries, a script in Abaqus was written to output the desired state variables of the interfaces to a CSV (comma separated values) file. The value of each node on the interface as well as its coordinates in the model are stored. A user interface was written in MATLAB to view this data and features were added to it over the course of the project. It is presented here for use in similar situations.

After running the script, the main user window appears (Figure 60). From this window, the user can navigate to the folder where the CSV file is stored. If multiple frames have been output from Abaqus, the viewport will show the animation once through the first time the file is opened and then can be viewed frame-by-frame as necessary. On the user interface the following features are available:

1. Viewport.
2. Abaqus frame number and file name.
3. Legend.
4. File browser.
5. Output variable selection.
6. Color scheme for plots.
7. Reverse palette. If selected, reverses the color gradient of the legend.
8. Max value. Filters out points greater than this number. Useful when the color scale is compressed due to large outliers.
9. Range hold. When toggled, retains the current limits of the color map. Useful for plotting several figures using the same legend.
10. Save multiple frames as an animated GIF image.
11. Plot the figure using the selected options.
12. Frame-by-frame navigation controls.
13. Copy figure to clipboard.
14. Select data in figure. A cursor will appear allowing the user to select points in the viewport. The data corresponding to these points will be saved to the MATLAB session. This feature relies on the open source `selectdata` function developed by John D’Errico [110].

Figure 60. Grain boundary viewer user interface.
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