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A Framework for Uncertainty Quantification in Microstructural Characterization with Application to Additive Manufacturing of Ti-6Al-4V

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A FRAMEWORK FOR UNCERTAINTY QUANTIFICATION
IN MICROSTRUCTURAL CHARACTERIZATION
WITH APPLICATION TO ADDITIVE MANUFACTURING OF Ti-6Al-4V

A dissertation submitted in partial fulfillment of the
requirements for the degree of
Doctor of Philosophy

By

GREGORY THOMAS LOUGHANE
B.S.M.E., Wright State University, 2011

2015
Wright State University
I HEREBY RECOMMEND THAT THE DISSERTATION PREPARED UNDER MY SUPERVISION BY Gregory Thomas Loughnane ENTITLED A Framework for Uncertainty Quantification in Microstructural Characterization with Application to Additive Manufacturing of Ti-6Al-4V BE ACCEPTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF Doctor of Philosophy.

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Abstract

Loughnane, Gregory T., Ph.D. in Engineering Program, Wright State University, 2015.  
*A Framework for Uncertainty Quantification in Microstructural Characterization with Application to Additive Manufacturing of Ti-6Al-4V*

The sampling of three dimensional (3D) mesoscale microstructural data is typically prescribed using simple rules, likely resulting in data under- or oversampling depending on the measurement(s) of interest. The first part of this work investigates one approach for determining a minimally sufficient sampling scheme for 3D microstructural data, using computer-generated phantoms of polycrystalline grain microstructures. Sources of error that are observed experimentally are modeled using phantoms, in order to determine the effect that errors have on the microstructural statistic(s) of interest. Minimally-sufficient sampling schemes are then established based on a required accuracy in the microstructural statistic(s). The characterization error modeling framework is subsequently demonstrated on experimentally-derived statistics from high resolution 3D serial sectioning data, in order to inform future experiments on the same material. The second part of this work lends the aforementioned approach to the additive manufacturing (AM) of Ti-6Al-4V. Statistical analysis and virtual modeling tools developed herein are used to analyze α and β phase microstructures in two thin-walled Ti-6Al-4V samples. Ultimately, this research aims to provide a virtual modeling framework for analyzing uncertainty in microstructural characterization, and to produce an offering of novel solutions for addressing current issues associated with rapid qualification methods for AM of Ti-6Al-4V components.
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<td>Lognormal mean for grain size distribution</td>
<td>-</td>
</tr>
<tr>
<td>( \sigma )</td>
<td>Lognormal standard deviation for grain size distribution</td>
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<tr>
<td>( \text{LN}(\mu, \sigma) )</td>
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<td>Squared Euclidean distance between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>( d_{IP} )</td>
<td>Inner Product between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>( d_{Fid} )</td>
<td>Bhattacharyya Coefficient, Fidelity, or Hellinger Affinity between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
<td>Units</td>
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<tr>
<td>------------</td>
<td>-----------------------------------------------------------------------------</td>
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<tr>
<td>(d_{MBC})</td>
<td>Modified Bhattacharyya Coefficient between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>(d_{BD})</td>
<td>Bhattacharyya Distance between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>(d_{HD})</td>
<td>Hellinger Distance between two discrete distributions</td>
<td>-</td>
</tr>
<tr>
<td>(MBC)</td>
<td>Calculated Modified Bhattacharyya Coefficient between phantom and reference microstructures</td>
<td>-</td>
</tr>
<tr>
<td>(\overline{MBC})</td>
<td>Observed average of MBC values</td>
<td>-</td>
</tr>
<tr>
<td>(n)</td>
<td>Number of samples (i.e. instantiations of phantom microstructures)</td>
<td>-</td>
</tr>
<tr>
<td>(s)</td>
<td>Sample standard deviation</td>
<td>-</td>
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Dedication

To my daughter, Evelyn Marie Loughnane

and

To the memory of my dear friend and colleague, Mitch McCabe
1 Introduction

This research provides statistical analysis and virtual modeling toolsets for the characterization of grain and sub-grain level microstructures in two and three dimensions. This section provides an introduction to the two key areas of focus: virtual modeling of three dimensional (3D) microstructures, and solidification microstructure process mapping approaches for rapid qualification of additive manufactured components.

Section 1.2 describes recent developments within materials science and engineering related to the investigation of material microstructures in three dimensions (3D). Details on select experimental and modeling tools for analyzing microstructures are described, followed by an explanation of how this research provides a basis to inform (a priori) state-of-the-art experimental tools via computational models.

Additive Manufacturing (AM) processes are discussed in section 1.3, with a narrowed focus to the development of solidification microstructure process mapping approaches for direct metal AM processes. Additional considerations are given to developments related to real-time sensing and adaptive control of direct metal AM, as microstructure quantified in this work is also compared to real-time thermal imaging data collected during component builds. This is followed by an explanation of how experimental microstructural characterization, combined with statistical analysis tools and/or virtual microstructural modeling approaches, can be used to enhance process mapping for direct metal AM, and more generally be applied to the characterization of AM microstructures required for rapid qualification of AM components.

1.1 Motivation

The automation of experimental tools and the development of new computational modeling approaches available for microstructural characterization require more efficient, cost-effective ways for collecting 3D microstructural data. At the same time, investigating the best methods
for constructing and qualifying direct metal AM components in order to achieve the most
desirable properties requires analysis of the resulting solidification microstructures. Moreover,
the complexity of alloy systems used in engineering applications including AM often
necessitates 3D characterization at the microscale in order to accurately predict component-level
properties. Thus, this research aims to inform not only microstructural characterization
processes in general, but also those required for qualification and a more complete understanding
of additive manufactured metal components.

1.2 Three-Dimensional Microstructure

The importance of characterizing microstructure in three dimensions (3D) to accurately
quantify the true size, true shape, local neighborhood, and connectivity of microstructural
features has been well documented [1,2]. Recent technological advances have contributed to the
development of automated and semi-automated microstructure characterization systems that can
collect large 3D data sets of material microstructure [3-8]. Furthermore, the wide availability of
advanced computing power has led to the development of software that is capable of creating
virtual (phantom) 3D microstructure data that closely mimics real microstructure [9,10]. The
maturation of both experimental and computational 3D microstructure data generation methods
has provided the materials community with an unprecedented ability to digitally represent the
morphology of microstructural ensembles with high fidelity. These digital representations of
structure—especially microstructure in 3D—are likely to play a key role in the success of
recently instituted Integrated Computational Materials Engineering (ICME) initiatives [11-13],
as depicted visually in Figure 1. The four defining disciplines of practice for materials science
and engineering are all inherently linked to one another, and in a holistic ICME approach they all
depend either directly or indirectly on representations of structure [14].
In 2D quantitative microstructural analysis, it is well-known that low resolution images (corresponding to large pixels relative to the size of the feature-of-interest) can result in a loss of microstructural information. Conversely, excessively high resolution images result in a needlessly large memory size and may not provide any significant improvement in the accuracy of the desired analysis [15]. Naturally, the same problem exists for 3D microstructural data, and thus it is necessary and prudent for efficient quantitative microstructural analysis to determine an optimal data sampling scheme, including speed of data collection, as well as in- and out-of-plane sampling resolutions. This issue is especially important for data collection methods like serial sectioning, as simply collecting data at higher and higher resolutions may not be practical, due to the potential cubic growth in both collection time and computational resources for data post-processing and analysis.

1.2.1 Collecting Experimental 3D Microstructural Data

Experimental techniques for investigating microstructures in 3D include both destructive and nondestructive methods. Destructive methods remove material in a layer-by-layer approach,
collecting morphological, crystallographic, and/or chemical data on the surface of each layer via optical or scanning electron microscopy. Nondestructive techniques provide a non-contact approach, and for metallic materials rely on high-power synchrotron X-ray machines that detect bulk material diffraction of X-rays [16]. More thorough review books on the vast suite of current and historical microstructural characterization techniques are available for the interested reader (e.g., [17]). This work will focus on one destructive method in particular, known as serial sectioning, however the general approach outlined can be extended to all types of 3D microstructural characterization techniques.

1.2.1.1 Serial Sectioning

Serial sectioning is simply a technique that consists of collecting data on many 2D sections of material, typically through use of an optical or scanning electron microscope (SEM), as sections of material are polished away from the top down. Examples of data produced by serial sectioning are shown in Figure 2.

Figure 2: Serial Section Data Examples: Image Credits: Dr. Anthony Rollett, Carnegie Mellon University (left) and Dr. David Rowenhorst, Naval Research Laboratory (right)
Serial sectioning can be accomplished with a few different experimental apparatus, and can also be accomplished, very tediously, by hand. For very small scale microstructural feature investigations, dual beam focused ion beam-scanning electron microscopy (DB FIB-SEM) [18-28] is a very popular technique. However, the DB FIB-SEM does not work well with samples which deal with features tens or hundreds of microns in diameter. In these cases, it requires mechanical polishing to remove a greater amount of material per section to obtain a statistically significant number of grains. Fully-automated mechanical polishing or micromilling devices that are capable of collecting data at this scale include the Alkemper-Voorhees Micromiller [3], RoboMet.3D [4], Genus_3D [5], and the LEROY system at AFRL, which leverages a 6-axis sample transport robot arm [6] (see Figure 3) to move samples between polishing and imaging stations. In 2015, RoboMet.3D, which uses a single optical mode of data collection, is a commercially available system. Further, the development of multi-modal commercial systems leveraging SEMs in tandem with automated mechanical polishers are currently an area of high interest for manufacturers.

Figure 3: Fully Automated Serial Sectioning Characterization Systems: From left to right, the Alkemper-Voorhees micromiller (Northwestern University), Genus_3D (National Institute of Materials Science, Japan), the LEROY multimodal characterization system (Air Force Research Laboratory), and RoboMet.3D (UES, Inc.). Image credits: Dr. Michael Uchic, AFRL.
1.2.1.2 Multi-Modal 3D Data Collection

State-of-the-art microstructural characterization systems are often multi-modal, or consisting of multiple types of microstructural data inputs. This characteristic provides additional levels of detail and complexity to representations of microstructure. For example, most state-of-the-art SEM systems are outfitted with both Electron Back-Scatter Diffraction (EBSD) and Energy-Dispersive X-ray Spectroscopy (EDS/EDX) detectors. These provide, respectively, crystallographic and chemical data. Crystallographic data via EBSD is especially powerful, in that it can alone generate 3D volumetric reconstructions of grain ensembles [28], and has also seen application as a complement to SEM imaging when reconstructing microscale morphologies [26].

1.2.2 Modeling & Simulation with 3D Phantom Microstructures

Phantom microstructures are widely used for a variety of materials science investigations. These digital representations, which are based either directly or implicitly on experimental data, can be translated to 3D mesh structures and used as input to finite element simulations, including both processing and property-prediction models [30-36]. The type of mesh structures used for input are also an active area of research, both the geometric nature (i.e., tessellations [37] and parametric representations [38]) and the implicit properties contained within the mesh structures themselves (i.e., Representative Volume Elements (RVEs) [39]). However, the estimation of the uncertainty associated with digital representations of microstructure has been underdeveloped [40,41].
1.2.3 Review of Microstructural Uncertainty Quantification using Phantoms

The use of phantoms, i.e., simulated objects that mimic the expected characteristics of experimental data, has recently been employed to study the effect of image resolution on the accuracy of measurements derived from image data. For example, the accuracy of selected size and shape parameters relative to the spatial resolution of tomographic X-ray data was examined using cylindrical-shaped phantoms [42]. Re-sampling of the cylinder data to lower resolutions in this study resulted in approximately 100 and 1000 voxels per cylinder being required to keep cylinder surface area and 3D Feret shape, respectively, below ~10% error. Another recent work investigated the effect of spatial resolution on very small (< 50 grains) phantom grain ensembles, for select regional and topological grain properties [43]. Error analysis was performed for the volume, surface area, and mean width of each grain, as well as the length of each triple line and the location of each quad point. The accuracy of quantitative measurements from digital 2D images was also investigated by Tiwari and Tewari, although this study only considered the effect of sampling resolution using simple objects such as lines and circles [44].

1.2.4 Using Phantom Microstructures to Quantify Uncertainty in Serial Sectioning Experiments

Many known sources of error can be observed in serial sectioning experiments, and in turn these sources of error can be modeled using phantom microstructures in order to determine their effect on the microstructural statistic(s) of interest. This work focuses on two sources of error associated with multi-modal serial sectioning experiments thought to have a potentially large effect on accuracy: voxel discretization of data (i.e., resolution), and non-indexing of pixels within EBSD maps (i.e., noise).
1.2.4.1  **Modeling the Effect of Voxel Resolution**

In contrast to prior efforts that examine the effect of spatial resolution on the measurements of individual features, the study provided in section 3.1 examines the effect of spatial resolution on discrete probability density functions (PDFs) derived from analysis of 3D phantom grain ensemble microstructures consisting of thousands of grains. Phantom voxel-based microstructures are generated with very high spatial resolution relative to the typical microstructural feature (e.g., grain), providing a common reference from which to quantitatively assess the effect of sampling resolution. The minimum voxel resolution required to accurately represent the statistics of the phantom reference volumes is determined by down-sampling the volumes to coarser resolutions and comparing the resulting microstructural statistic(s) of interest.

1.2.4.2  **Modeling the Effect of EBSD Noise**

Noise present in data collected during serial sectioning experiments is also a topic of study in this research, in particular the noise resulting from EBSD analysis. During EBSD analysis, also known as Orientation Imaging Microscopy (OIM), electrons impinge on the surface of the sample, and then diffract onto a detector that identifies characteristic Kikuchi bands within the diffraction patterns corresponding to each of the diffracting lattice planes in the material. However, the Kikuchi bands collected are sometimes unable to distinguish between orientations, especially when the pixel under investigation lies across a grain boundary or is contained mostly by an inclusion, void, precipitate, or damage in the material. In these cases, software is unable to index the pattern with confidence, and thus a “noise” effect occurs where uncertain or missing data is recorded, as depicted in Figure 4. EBSD noise is classified into two categories for this study as portrayed in Figure 4: noise that occurs along the boundaries of grains (i.e., boundary noise) and noise that occurs randomly throughout the structure, including within grains (i.e., random noise). The study provided in section 3.2 examines these effects to determine minimum
voxel resolutions and maximum allowable noise levels for accurate representation of microstructural statistics. Experimental clean-up algorithms are applied to virtual noise before calculation of microstructural statistics in an effort to closely mimic experimental conditions.

![Image of EBSD pattern with labels for random and boundary noise]

**Figure 4: Electron Back-Scatter Diffraction (EBSD) Pattern**: Raw EBSD scan showing the two types of noise observed in the diffraction patterns. Random noise appears throughout the structure; for example, in the center of grains, while boundary noise appears only along grain boundaries.

### 1.2.4.3 Modeling the Effect of Additional Sources of Error

Although this work focuses on only two sources of error, one could posit that complete uncertainty quantification might include effects associated not only with resolution and noise, but with layer thickness tolerances, section planarity, and alignment of 2D scans and images. Further, in multi-modal experiments, each material system must be investigated to optimize not only the appropriate amount, but also the type of data being extracted from each section. If multiple types of data exist, then the best way to combine morphological, crystallographic, and chemical data must also be investigated for all microstructural parameters that could be targeted as output from an experiment. The framework provided by this dissertation is capable of handling any errors that might be present during experimental characterization, as long as a suitable technique for virtual modeling of the error is developed.
1.3 Additive Manufacturing

The process of Additive Manufacturing (AM) has come to be known by many names, including Layer-based Manufacturing, 3D printing, Stereolithography, Rapid Prototyping, Freeform Fabrication, Solid Freeform Fabrication, and Automated Fabrication [45]. AM encompasses all of these terminologies and represents any automated technique that directly converts 3D Computer-Aided Design (CAD) data into 2D slice data. The 2D slice data is formatted and subsequently transferred to an AM machine that builds the 3D physical object defined by the CAD model using a layer-by-layer approach. Many commercial AM machines currently exist, from those in the consumer market (e.g. Makerbot®, Cube®, or Form 1+® by Form Labs) to industrial-scale polymer systems (e.g. FDM by Stratasys) to high-dollar direct metal systems (e.g. Arcam EBM® or EOS Laser Sintering).

Originally, plastics were widely used in AM, but with the advent of direct metal processes, AM can be used for final production parts in the aerospace, automotive, and electronics industries, as well as for medical, textile, furniture, and many other applications [46]. By comparison to conventional methods of subtractive manufacturing (i.e., machining), AM provides an increase in precision relative to the CAD model, as well as a means to more efficiently leverage material and energy resources [47,48]. However, in moving from prototyping with plastics to near net shape component design with direct metal AM, the strength, reliability, and overall quality of the metal parts must be assessed and qualified using microstructural characterization techniques.

1.3.1 Direct Metal AM Processes

Direct metal additive manufacturing (AM) refers to any process that manufactures near-net shape components via direct deposition of metallic material into a pool of molten metal. These processes differ primarily in the power source (laser- or electron-beam) and feedstock delivery
system (powder stream, wire feed, powder bed). For each type of process, microstructural characterization techniques are used to assess and qualify the quality of metallic components. Further, while AM has advantages over traditional machining processes, such as lead time improvements and lower per-unit costs for relatively small batches, the inherent temperature variability in all cyclic direct metal AM processes often results in inconsistent and undesirable solidification microstructures.

1.3.1.1 Laser Beam Powder Stream Process

Laser Engineering Net Shaping (LENS™) uses a constant stream of powder particles that is injected into the weld pool created by the laser. In this process, the stage (substrate) moves in x- and y- directions beneath the powder delivery nozzle, and the nozzle is incrementally moved upwards in the z-direction for the addition of each layer [70]. The process was developed by a team at Sandia National Laboratories and was branded by Optomec Design Company the following year in 1997. Currently the process is used on an array of commercially available systems from Optomec that have found widespread use in both industrial and academic settings.

The LENS™ system used to build components investigated in this work represents a collaborative effort between Penn State University’s Center for Innovative Metal Processing through Direct Digital Deposition (CIMP-3D), Stratonics, and Optomec. The integrated multi-sensor/process model-based control system leverages an Optomec MR-7 LENS™ system, outfitted with a Stratonics ThermaViz® thermal imaging camera. Additional discussion about the AM components used for microstructural characterization and analysis is provided in section 4.1.1.

1.3.1.2 Electron Beam Wire Fed Process

Rather than using a stream of powder, the wire-feed process uses a solid wire of material that is melted directly with an electron beam. Wire feedstock is used in this process due to the
difficulties in feeding powder into the vacuum environment required for electron beam use [47]. Operation of wire-feed systems is much like the operation of LENS™ systems, in that the stage moves beneath the electron beam to create the part geometry, and the wire feed is subsequently incremented upwards in the z-direction for each new layer. The Electron Beam Freeform Fabrication (EBF³) system was the first wire feed system, and was developed by a team at the National Aeronautics and Space Administration (NASA) at Langley Research Center.

1.3.1.3 **Laser and Electron Beam Powder Bed Processes**

Powder bed processes operate in a fundamentally different way than the powder stream and wire feed processes. In these cases, a moving heat source selectively melts powder particles that lay on a large pre-heated bed of powder. In order to move to the next layer, the stage is incremented downward in the z-direction. The Arcam EBM® process is the most popular electron beam powder bed system, while many different commercial companies manufacture laser beam powder bed systems.

1.3.2 **Review of Solidification Microstructure Process Mapping**

One approach for understanding temperature- and solidification microstructure-variability in direct metal AM is a method termed “process mapping” developed by Beuth *et al.* and summarized in [49]. This methodology maps process outcomes in terms of process variables, where variables represent experimentally-controlled factors such as beam power and velocity and outcomes refer to experimental results such as stress or microstructural characteristics. The idea behind the process mapping approach is to allow all direct metal AM processes, which act in different regions of processing space, to be characterized and analyzed in a cohesive way. Early work focused on residual stress in metal and polymer AM processes [50-54]. Subsequent work investigated control of melt pool dimensions under steady state conditions for the full range
of the Laser Engineering Net Shaping (LENS®) process through investigation of nondimensional thermal metrics [55-59]. The work was then extended to provide direct predictions of solidification microstructure [60-65]. Additionally, many process mapping approaches have leveraged analytical and thermal finite element models to predict cooling rates and thermal gradients, parameters that are used to predict trends in solidification microstructures [62-67]. Further, within recent years, detailed microstructural observations have shown correspondence to theoretically calculated melt pool geometries for given sets of process variables [68,69].

1.3.3 Review of Real-Time Sensing and Adaptive Control of AM Process Parameters

Real-time sensing and adaptive control utilizes process mapping techniques by monitoring deposition and altering process variables in order to maintain constant melt-pool geometry or alternatively to control component build parameters. Over the years, significant research regarding adaptive control of laser processes has been conducted in which monitoring methods are based on the physical phenomena that occur due to laser-materials interaction (see Ref. [71] for a recent thorough review). Many of the techniques and sensors used for investigation of AM processes have historically been used for laser cladding and welding processes (i.e., optical and acoustic techniques). For laser AM processes, researchers have leveraged optical imaging techniques to monitor temperature during laser-based manufacturing of various materials [72]. Griffith et al. studied melt-pool temperatures and gradients collected during deposition of H13 tool steel [73], and Hofmeister et al. achieved significant stabilization of melt-pool size via control of laser power in 316 stainless steel through use of an optical thermal imaging system [74]. Correlation of thermal data to solidification microstructure has also been a topic of interest for various materials, including 316 stainless steel [75] and Ti-6Al-4V [76-80]. However, most
microscale observation of AM components is qualitative in nature and does not lend itself to the
process mapping approach.

1.3.4 Solidification Microstructure of Ti-6Al-4V

In alpha-beta titanium alloys such as Ti-6Al-4V, the body-centered cubic (BCC) β (beta)
phase transforms to a hexagonally close packed (HCP) α (alpha) phase upon cooling through the
β transus, which occurs at approximately 1000° C. The smaller α features of Ti-6Al-4V begin
growing within the metastable β phase from grain boundaries to produce α lamellae/laths within
a β matrix. This transformation follows a classic Burgers relationship [82], such that directions
of the parent β and product α phases are parallel to one another [83]. The α phase that forms
assumes a needle, lath, lamellar, or plate morphology often referred to as acicular α having a
high aspect ratio (10:1) [81]. The overall α+β morphology that results is called the
Widmanstätten morphology, which can take two forms: colony α or basketweave α. It is also of
interest for this work to note that the tendency to form basketweave α increases with cooling rate
[84]. These types of structure are illustrated in the phase diagram shown in Figure 5. In AM
processes, depending on part geometry and AM process parameters, cooling rates can vary
significantly across a single part, especially in between added layers and on the edges of parts.
As a result, multiple α structures are often observed in single β grains. For the interested reader,
much more detailed information on the solidification microstructure of Ti-6Al-4V can be found
in [81].
Figure 5: **Phase Diagram for Ti-6Al-4V**: Showing Widmanstätten $\alpha$ morphologies that form below the beta transus, including colony $\alpha$ and basketweave $\alpha$ [81]. See Figure 43 for actual experimental data showing both colony $\alpha$ and basketweave $\alpha$ microstructures.
1.3.5 Using Computational and Modeling Tools to Enhance Process Mapping and Rapid Qualification Methods

Despite significant research into solidification microstructure process mapping, the quantification of uncertainty in microstructural measurements has been underdeveloped. Investigations of Ti-6Al-4V generally categorize β grain morphologies as simply equiaxed, columnar, or mixed based on a qualitative visual analysis, and often report a single one-dimensional β grain width to describe grain morphology. Further, geometries investigated are typically primitive, constructed of single- or multiple-bead passes with a limited number of material layers. This provides only a handful of grains for analysis, while microstructural statistic(s) of interest and their distributions often require hundreds or thousands of grains for accurate representations.

In the spirit of ICME, this work provides new techniques for analysis of solidification microstructures in AM Ti-6Al-4V by leveraging existing computational tools yet to be applied to AM, as well as the computational tools and virtual modeling procedures developed in section 2. Namely, a methodology for quantitative analysis of α-lath microstructures in Ti-6Al-4V that can be used to enhance direct metal AM process mapping is demonstrated. This methodology has been applied to α-lath thicknesses in forged billets of Ti-6Al-4V [85], but is applied here to AM components with the latest materials image-processing optimization tools. Additionally, a previously unpublished technique for comparing distributions of α-lath thicknesses obtained from experimental data in thin-walled components of Ti-6Al-4V is presented. Finally, a procedure for estimating total serial sectioning time required for the investigation of β grain size distributions in AM Ti-6Al-4V is presented, which leverages state-of-the-art experimental hardware and software tools, in addition to virtual modeling. As the components constructed via
direct metal AM processes become larger, and the sub-grain level details of microstructure associated with desired properties become a broad topic of interest, toolsets like the ones developed in this dissertation will be leveraged for more rapid qualification of AM components.

1.4 Overview and Contributions

The primary objective of this research was to enhance the ability of fully automated microstructural characterization systems, in particular the Air Force Research Laboratory’s (AFRL) LEROY system [6], to collect 3D microstructure data more quickly and efficiently. A secondary objective was to facilitate process mapping approaches for direct metal AM processes using a combination of statistical analysis tools and virtual modeling. The research presented herein provides new contributions to the fields of 3D microstructural characterization and additive manufacturing that include:

1. A novel quantitative analysis framework that performs virtual microstructural characterization to determine optimal data sampling schemes for real experiments
2. Quantitative conclusions regarding the effect of resolution on the accuracy of select grain ensemble statistics for a single-phase lognormal microstructure
3. Quantitative conclusions regarding the effect of noise from EBSD at various spatial resolutions on the accuracy of select grain ensemble statistics for a single-phase lognormal microstructure
4. A virtual validation of the accuracy of a previously published 3D microstructural characterization experiment with novel insights into optimizing future data collection
5. A previously unpublished method for determining and comparing alpha lath width distributions obtained from SEM and EBSD imaging of additive manufactured Ti-6Al-4V components
A 3D serial sectioning investigation into the microstructure of additive manufactured Ti-6Al-4V, leveraging virtual characterization to optimize data sampling parameters, with ultimate application to more rapid qualification of AM components
2 Microstructural Characterization Error Modeling Framework

This section details the framework used for analyzing uncertainty in microstructural characterization processes, and represents the foremost contribution of this research. The framework, when viewed as a generic error modeling tool, can be customized to any microstructural characterization process, include any and all identifiable sources of error associated with the process, be used for investigation of any material system, and target any combination of microstructural statistic(s) of interest. The framework represents the vision of a fully-automated microstructural characterization system that can collect a minimally sufficient amount of microstructural data based only on output requirements defined by the end user, so as to provide the most efficient, cost-effective means of data collection and allow characterization time to be shared and managed in a more productive way than state-of-the-art experimental characterization practices currently allow.

In this section, the framework is demonstrated for virtual modeling and prediction of resolution sampling schemes that are minimally sufficient to represent various morphological microstructural parameters-of-interest in a single-phase lognormal microstructure. This is followed by a demonstration of an additional source of error, noise from EBSD, combined with sampling resolution analysis and experimental cleanup algorithms in order to determine the effect that noise can have on various morphological parameters at different resolutions.

2.1 Digital Representation Environment for Analyzing Microstructures in 3D (DREAM.3D)

The creation of the reference volumes and subsequent down-sampling was performed using the 3D materials analysis software DREAM.3D, which stands for Digital Representation
Environment for Analyzing Microstructure in 3D [10] (dream3d.bluequartz.net). The procedure used to create and resample these volumes is described in the following sections.

2.2 Phantom Voxel-Based Microstructure Generation Procedure

First, volumes of virtual microstructure that represent a real sample prior to any experimentation are generated. These are referred to as reference volumes, and it is these reference structures that are used as the basis for the virtual modeling of characterization errors. For the interested reader, a more detailed review of synthetic microstructure generation methods has been reported previously [9], and a basic outline of the microstructure generation procedure is as follows. First, the dimensions of the phantom microstructural volume are defined based on the desired number of grains and their associated size distribution. Next, a geometric packing algorithm is used to fill the space with sampled grains, where the grain sizes are randomly selected from the prescribed lognormal distribution. During grain placement, the grains are allowed to be inserted, removed, or translated within the volume, and final grain placement is based on matching a number of governing criteria until no additional grains can be incorporated into the reference volume. Lastly, an isotropic grain growth algorithm is used to fill any empty space within the reference volume, by assigning any unassigned voxels to one of the existing grains.

2.3 Microstructural Parameter Probability Distribution Functions (PDFs)

In the current section the microstructural parameters that are of interest are described: grain size, number of nearest neighbors, aspect ratios \(b/a\) and \(c/a\), and moment invariant \(\Omega_3\).

Although this work considers only morphological parameters, the framework is applicable to
examine resolution sensitivity for any microstructural statistics that can be computed from phantom grain ensembles.

### 2.3.1 Grain Size: Equivalent Sphere Diameter

The grain size descriptor chosen for this work is the equivalent sphere diameter (ESD), a derivative of grain volume. It is computed by using Eq. (1):

\[
ESD = 2 \cdot \left( \frac{3}{4\pi} N_v V_v \right)^\frac{1}{3}
\]

where \(N_v\) is the number of voxels that comprise the grain and \(V_v\) is the volume of a single voxel [36].

### 2.3.2 Grain Shape: Aspect Ratios and Curvature

The shape of microstructural features can be characterized using numerous descriptors, which typically fall into one of four categories. Descriptors can be based on measurements of either the interior volume or the surface, and they can be complete or reductive [86]. This work considers a classical measure of shape, aspect ratio, for which the calculation procedure is outlined below. A more detailed description of the method applied to voxel-based microstructural representations is available in the literature [87, 88].

For each grain, the centroid coordinates of a best-fit ellipsoid are computed using zeroth and first-order area moments, and the solution of the eigenvalue problem for the moment of inertia tensor yields the directions of the principal axes. Axis lengths are then solved for in closed-form (see [87]) and ratios of the secondary to primary axis \((b/a)\), and tertiary to primary axis \((c/a)\) are computed.

However, aspect ratios cannot distinguish between objects with different surface curvatures (e.g., an ellipsoid and a parallelepiped with the same dimensions have identical aspect ratios), and
thus a more descriptive metric based on higher order moments is considered. In particular the 3\textsuperscript{rd} invariant quantity of the moment of inertia (MOI) tensor for each grain, when normalized by grain volume, is referred to as the moment invariant $\Omega_3$ [90]. This quantity is similarity invariant, which refers to the property of being invariant to translation, rotation, and isotropic scaling. The quantity $\Omega_3$ also possesses the special characteristic of affine invariance – meaning that any transformation that preserves collinearity and ratios of distances does not change the quantity. In short, any combination of translations, rotations, dilations, and shears has no effect on the value of $\Omega_3$ for a given grain geometry. The quantity is calculated using Eqs. (2) and (3) as

$$\Omega_3 = \frac{\nu^g}{\sigma_3}$$  \hspace{1cm} (2)

$$\sigma_3 = \mu_{200}\mu_{020}\mu_{002} + 2\mu_{110}\mu_{101}\mu_{011} - \mu_{200}\mu_{011}^2 - \mu_{020}\mu_{101}^2 - \mu_{002}\mu_{110}^2$$  \hspace{1cm} (3)

and is simply a normalized combination of constituents from each grain’s MOI tensor. Note that the $\mu_{pqr}$ terms represent second order moments, where the moment order is equal to the sum of $p$, $q$ and $r$. Shapes become qualitatively smoother and less complex with increasing values of $\Omega_3$, up to the maximum value corresponding to that of spheres and ellipsoids [90].

### 2.3.3 Number of Nearest Neighbor Grains

The number of nearest neighbors is also tabulated for each grain. Since the microstructural data is represented on a voxelized grid, a neighbor grain is defined as any grain sharing at least one voxel face with the grain under consideration [87,88]. Therefore, voxels that only share a common edge or corner are not considered as neighbor grains in this analysis.
2.4 Virtual Resolution Down-Sampling

There is little information in the materials characterization literature to guide the selection of sampling resolution for data collection in 3D. Prior guidance is particularly important for destructive experiments such as serial sectioning, where the sample volume is incrementally and irreversibly consumed during the experiment. In the serial sectioning literature, it is generally espoused that one would like a minimum of ten sections through a microstructural feature to accurately describe its size and shape [8], but this guidance is simply a rule-of-thumb and is wholly insufficient for efficient quantitative microstructural analysis in 3D.

As in digital signal processing, virtual resolution down-sampling is a decimation process [90] which reduces sampling rate. Here, the “rate” is spatial rather than temporal in nature, so the down-sampling is executed along each coordinate direction in a Cartesian coordinate system, rather than in time.
The process for performing isotropic down-sampling begins by creating a second volume that is essentially superposed onto the reference volume. Each voxel in the second volume is assigned the grain identification number of the reference-volume voxel that corresponds to its centroid location, and this procedure is graphically depicted in Figure 7b. Note that the voxels of the reference volume are not being averaged in the creation of the down-sampled volume, but rather the down-sampled volume is a discrete sampling of the reference volume, as represented in this figure by voxels I-IV being assigned to the down-sampled volume. The anisotropic down-sampling procedure is also depicted in Figure 7. In this example, the down-sampled volume shown in Figure 7c is comprised of voxels twice as large along only one axis. The down-sampling process itself is identical to that described previously, only the superposed second volume no longer has uniform voxel dimensions.
2.4.1 Isotropic and Anisotropic Down-Sampling

![Diagram of Isotropic and Anisotropic Down-Sampling](image)

**Figure 7: Isotropic & Anisotropic Down-Sampling**: Schematic depiction of the down-sampling procedure used in this study: A) Representative reference volume, B) Isotropic down-sampling by a factor of 2, C) Anisotropic down-sampling by a factor of 2 only along one axis.

2.5 Virtual Noise Down-Sampling

Virtual noise down-sampling provides a means for modeling EBSD noise as it would appear in experiments, and the introduction of noise is also fundamentally a decimation process. To induce noise, first the down-sampling of grain identification information is performed, then individual voxels have their internal memory cleared, representing a state that occurs when EBSD analysis is unable to discern crystallographic orientation via Kukuchi patterns. Examples of inducing both boundary noise and random noise into virtual models are shown in Figure 8.
Figure 8: **Modeling Boundary and Random Noise in Down-Sampled Phantom Microstructures:** The process of modeling boundary and random noise following isotropic down-sampling: A) High resolution reference volume (0.1 µm resolution), B) Down-sampled volume (1.0 µm resolution), C) Down-sampled volume with 25% boundary noise, and D) Down-sampled volume with 25% random noise.

### 2.5.1 Noise Cleanup

After noise is introduced, it is subsequently cleaned using the experimental cleanup algorithm that is currently used by the Materials Characterization Facility at AFRL to clean up experimentally collected data. The cleanup routine leverages the use of grain IDs assigned
during initial reference volume construction. The algorithm is designed to check all 6 face-sharing neighbor voxels of the voxel under consideration, and assign the grain ID to the noisy voxel based on the highest number of grain ID counts out of the 6 neighboring voxels. Following a sufficient number of iterations to clean each noisy voxel in the microstructural volumes, a new representation of structure exists from which to calculate microstructural statistics (see Figure 9).

When modeling EBSD noise, it must be done after a resolution down-sampling corresponding to the spot size planned for 2D EBSD analysis. During experimentation, noise can only occur at a scale equal to or greater than the spot size of the data collection instrument, which is typically represented in virtual experiments by a down-sampled resolution, rather than by a reference resolution. The biggest benefit of virtual modeling of microstructure as a whole is the ability to build reference volumes, which represent a known material microstructure with zero uncertainty, at resolutions that cannot feasibly be collected experimentally. Thus, if both effects are being modeled, resolution down-sampling is performed first to mimic 3D serial sectioning processes in the most realistic way possible.
Figure 9: **Modeling Noise + Cleanup in Down-Sampled Phantom Microstructures:** The process of modeling noise + cleanup following isotropic down-sampling: A) High resolution reference volume (0.1 µm resolution), B) Down-sampled volume (0.5 µm resolution), C) Down-sampled volume with noise (75% boundary noise, 10% random noise), and D) Final volume following noise + cleanup.

The cleanup routine used to handle noise in this work operates under the specific assumption that the grain to which each voxel belongs is already known. In practice, this is never the case, as experimental data provides either orientation (EBSD) or grayscale information (SEM, optical imaging) for pixels (voxels) rather than grain IDs. Therefore if pixels that share the same orientation, but only share a voxel edge or corner after a 3D reconstruction, they are incorrectly
grouped into different grains using this and similar cleanup algorithms. This is an inherent drawback of geometric cleanup routines, as they do not include any information about underlying microstructural physics. Although this particular point is not expanded upon further and quantified in this research, it is noteworthy that the framework presented does provide a quantitative computational means for analyzing the validity and accuracy of new or under-development cleanup algorithms using virtual (phantom) microstructures. In this way cleanup algorithms can be tested virtually before they ever get used on experimentally-collected 3D data.

2.6 Statistical Analysis and Uncertainty Quantification of Microstructural PDFs

This work introduces two types of down-sampling, for modeling the effects of both resolution and EBSD noise, which can be executed independently or in tandem to model relevant effects. In the following discussion about statistical analysis, error volumes containing either down-sampled representations of structure, or down-sampled representations of structure with the addition of noise, will be referred to as down-sampled volumes. This will facilitate the discussion of statistical comparisons.

The PDFs for grain size, grain shape, and number of nearest neighbors from the down-sampled volumes were compared to the corresponding reference volume PDFs via two different measures. In addition to individual statistics, such as the mean and standard deviation of PDFs, the similarity between full and partial PDFs is also investigated. These approaches are outlined in the section below.
2.6.1 Means and Standard Deviations of Microstructural PDFs

First, the mean value for each microstructural parameter is computed and these values are subsequently compared via a percentage error between true (reference) and estimated (down-sampled) values, calculated as

$$\text{Percent Error} = \left| \frac{\text{mean}_{\text{reference}} - \text{mean}_{\text{down-sampled}}}{\text{mean}_{\text{reference}}} \right|$$  \hspace{1cm} (4)

Percentage error in the mean is investigated as an absolute value in section 3.1 and with directionality (i.e. positive/negative) in section 3.2. This is based on the manner in which this research was conducted and published, and then further investigated.

2.6.2 Similarity Analysis of Microstructural PDFs

The PDFs constructed from down-sampled structures are compared to PDFs constructed from reference volumes. Full PDF comparisons were made initially by surveying seven distance measures that describe similarity between two discrete PDFs: the inner product, Euclidean distance, squared Euclidean distance, Bhattacharyya Coefficient (BC), a Modified Bhattacharyya Coefficient (MBC) that gives the quantity metric properties [94], the Bhattacharyya Distance, and the Hellinger Distance. This list includes a direct multiplication of histogram bin populations (dot product), as well as two classical (Euclidean) distances derived from Pythagorean’s theorem [91], each of which has seen widespread application. Additionally, the BC and its variants—derived using the sum of geometric means—were also included, as they have seen significant application in signals analysis [92-94], as well as limited and recent application in the aerospace [95] and microstructural image analysis [96] communities.
2.6.2.1 Metrics for Comparing the Geometric Similarity of Two Discrete PDFs

Comparisons are made initially using seven different distance measures. The Euclidean distance, or Euclidean L2 Norm, and the squared Euclidean distance, both derived from Pythagorean’s Theorem [91], are investigated. The inner product between two PDFs was also investigated, as well as several distances derived using the sum of geometric means, including the Bhattacharyya Coefficient (BC) (a.k.a. Fidelity or Hellinger Affinity), a modified version of the BC that gives the quantity metric properties [94], the Bhattacharyya Distance (BD), and the Hellinger Distance (HD).

Eqs. (5) - (11) that follow use \( R_i \) and \( S_i \) to denote the fractions of microstructure data contained in bin \( i \) for the discrete distributions \( R \) and \( S \). Distributions \( R \) and \( S \) can be thought to correspond to reference volume PDFs and down-sampled PDFs, respectively.

\[
d_{\text{Euc}} = \sqrt{\sum_{i=1}^{n} (R_i - S_i)^2} \quad (5)
\]

\[
d_{\text{SqEuc}} = \sum_{i=1}^{n} (R_i - S_i)^2 \quad (6)
\]

\[
d_{\text{IP}} = \sum_{i=1}^{n} R_i S_i \quad (7)
\]

\[
d_{\text{Fid}} = \sum_{i=1}^{n} \sqrt{R_i S_i} \quad (8)
\]

\[
d_{\text{MBC}} = \sqrt{1 - \sum_{i=1}^{n} \sqrt{R_i S_i}} \quad (9)
\]

\[
d_{\text{BD}} = -\ln(\sum_{i=1}^{n} \sqrt{R_i S_i}) \quad (10)
\]

\[
d_{\text{HD}} = 2\sqrt{1 - \sum_{i=1}^{n} \sqrt{R_i S_i}} \quad (11)
\]

Other various distance/similarity measures were investigated for this study as well, including those from the inner product family (see Ref [91]). In the case of these divergence-type (i.e.
non-metric) measures, although conclusions could be drawn from numerical results relative to one another, the interpretation of these quantities was found to be cumbersome, and additionally such divergence-type measures do not possess any qualities superior to those of the metrics chosen.

A sample set of results for a virtual microstructure, down-sampled to various resolutions, is shown in Table 1. It can be seen that the Modified Bhattacharyya Coefficient is superior metric of the three possible metrics shown. Not only does it scale linearly with increasing or decreasing resolution, but it also resolves small differences in distributions better than the other metrics, especially at relatively high resolutions (10-30 sections through the average feature). In this way, each of the metrics listed above were investigated.
Table 1: Comparison of Similarity Metrics: A sample set of down-sampling results on a virtual microstructure is shown. As can be clearly seen, the MBC is a superior metric, as it not only scales linearly with resolution (unlike the Inner Product), but is also excellent at resolving differences between distributions at high resolutions (unlike the Euclidean L2 Norm).

<table>
<thead>
<tr>
<th>Resolution</th>
<th>ESD</th>
<th>b/a</th>
<th>c/a</th>
<th>Ω3</th>
<th>NNN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modified Bhattacharrya Coefficient</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 section/avg. feat.</td>
<td>0.7393</td>
<td>0.7076</td>
<td>0.7165</td>
<td>0.9375</td>
<td>0.3102</td>
</tr>
<tr>
<td>2 section/avg. feat.</td>
<td>0.3665</td>
<td>0.5746</td>
<td>0.6220</td>
<td>0.8864</td>
<td>0.1792</td>
</tr>
<tr>
<td>3 section/avg. feat.</td>
<td>0.2367</td>
<td>0.4428</td>
<td>0.4982</td>
<td>0.8125</td>
<td>0.1292</td>
</tr>
<tr>
<td>5 section/avg. feat.</td>
<td>0.0657</td>
<td>0.2920</td>
<td>0.3209</td>
<td>0.7106</td>
<td>0.1391</td>
</tr>
<tr>
<td>6 section/avg. feat.</td>
<td>0.0654</td>
<td>0.2625</td>
<td>0.2815</td>
<td>0.6593</td>
<td>0.1260</td>
</tr>
<tr>
<td>10 section/avg. feat.</td>
<td>0.0133</td>
<td>0.1275</td>
<td>0.1365</td>
<td>0.4927</td>
<td>0.1150</td>
</tr>
<tr>
<td>15 section/avg. feat.</td>
<td>0.0063</td>
<td>0.0634</td>
<td>0.0737</td>
<td>0.3447</td>
<td>0.0933</td>
</tr>
<tr>
<td>30 section/avg. feat.</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>Euclidean L2 Norm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 section/avg. feat.</td>
<td>0.3286</td>
<td>0.1155</td>
<td>0.1112</td>
<td>0.6912</td>
<td>0.0468</td>
</tr>
<tr>
<td>2 section/avg. feat.</td>
<td>0.0421</td>
<td>0.0734</td>
<td>0.0836</td>
<td>0.4320</td>
<td>0.0042</td>
</tr>
<tr>
<td>3 section/avg. feat.</td>
<td>0.0149</td>
<td>0.0381</td>
<td>0.0486</td>
<td>0.3202</td>
<td>0.0011</td>
</tr>
<tr>
<td>5 section/avg. feat.</td>
<td>0.0003</td>
<td>0.0134</td>
<td>0.0163</td>
<td>0.2307</td>
<td>0.0006</td>
</tr>
<tr>
<td>6 section/avg. feat.</td>
<td>0.0002</td>
<td>0.0100</td>
<td>0.0124</td>
<td>0.2000</td>
<td>0.0008</td>
</tr>
<tr>
<td>10 section/avg. feat.</td>
<td>0.0000</td>
<td>0.0026</td>
<td>0.0024</td>
<td>0.1158</td>
<td>0.0004</td>
</tr>
<tr>
<td>15 section/avg. feat.</td>
<td>0.0000</td>
<td>0.0007</td>
<td>0.0007</td>
<td>0.0596</td>
<td>0.0003</td>
</tr>
<tr>
<td>30 section/avg. feat.</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>Inner Product</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1 section/avg. feat.</td>
<td>0.0466</td>
<td>0.0249</td>
<td>0.0151</td>
<td>0.0019</td>
<td>0.0787</td>
</tr>
<tr>
<td>2 section/avg. feat.</td>
<td>0.0954</td>
<td>0.0466</td>
<td>0.0261</td>
<td>0.0057</td>
<td>0.0684</td>
</tr>
<tr>
<td>3 section/avg. feat.</td>
<td>0.1014</td>
<td>0.0718</td>
<td>0.0418</td>
<td>0.0151</td>
<td>0.0655</td>
</tr>
<tr>
<td>5 section/avg. feat.</td>
<td>0.0988</td>
<td>0.0986</td>
<td>0.0654</td>
<td>0.0351</td>
<td>0.0644</td>
</tr>
<tr>
<td>6 section/avg. feat.</td>
<td>0.0991</td>
<td>0.1045</td>
<td>0.0700</td>
<td>0.0498</td>
<td>0.0646</td>
</tr>
<tr>
<td>10 section/avg. feat.</td>
<td>0.0988</td>
<td>0.1223</td>
<td>0.0860</td>
<td>0.0995</td>
<td>0.0642</td>
</tr>
<tr>
<td>15 section/avg. feat.</td>
<td>0.0987</td>
<td>0.1281</td>
<td>0.0907</td>
<td>0.1496</td>
<td>0.0640</td>
</tr>
<tr>
<td>30 section/avg. feat.</td>
<td>0.0988</td>
<td>0.1311</td>
<td>0.0917</td>
<td>0.2317</td>
<td>0.0000</td>
</tr>
</tbody>
</table>

For investigating microstructural distributions, the Modified Bhattacharrya Coefficient (MBC) was selected, as it produced comparisons between reference and down-sampled distributions with relatively high sensitivity to subtle variations in the PDFs. The MBC is bounded between 0 and 1, where a value of 0 implies that two models are identically distributed, and higher values imply greater variance between distributions. The MBC, which was used for
analysis of both isotropically- and anisotropically down-sampled structures, is applied to discrete PDFs as shown in Eq. (12).

\[
MBC = \sqrt{1 - BC} = \sqrt{1 - \sum_{i=1}^{n} \sqrt{R_i S_i}}
\]  

(12)

In Eq. (12) \(R_i\) and \(S_i\) correspond to the fraction of microstructure data contained in bin \(i\) for the discrete probability distributions (i.e., histograms) \(R\) and \(S\), and correspond to reference volume distributions and down-sampled distributions, respectively. This can also been seen visually in Figure 10.

**Figure 10: Similarity Analysis of Microstructural Parameter PDFs:** Schematic depicting similarity analysis of full discrete PDFs.

### 2.6.2.2 Similarity Analysis of Microstructural PDF Tails

In the interest of extending statistical analysis tools for ultimate application to the needs of microstructural characterization laboratories, the relationship between reference and down-sampled volumes has been quantified not only by comparing mean values and the geometric similarity of full PDFs as before, but by using the modified Bhattacharyya Coefficient to compare also the geometric similarity of the tail(s) of the PDFs. Figure 11 demonstrates the utility of this additional analysis, showing that for grain size distributions, analysis of either tail corresponds directly to the analysis of small (left-tail) or large (right-tail) grain sizes. This allows for specific parts of the grain size distributions to be analyzed separately, which corresponds to applications where either large or small features could be of interest. For
example, investigations into the effects of grain size on component-level properties such as yield and fracture often employ “weakest link” approaches that look only at extremes present in grain size distributions [97]. Moreover, if only large features relative to the mean are of interest, then intuitively less data can be collected than would be required for smaller features.

**Figure 11: Similarity Analysis of PDF Tails:** In the case of reference and down-sampled grain size distributions, left and right tails of the size distributions correspond to small and large grains, respectively.

**2.6.2.3 Confidence Intervals on Similarity**

To quantify variability in the phantom microstructure generation and down-sampling procedure, multiple instantiations of structure are generated in DREAM.3D from the same inputs for each of the following two studies. In section 3.1, five independent reference volumes are created and down-sampled, and in section 3.2, twenty instantiations of noise at each noise level are generated from a single phantom reference volume. In each case, computed MBC values were assumed to be normally distributed, and thus the general form of confidence intervals can be expressed as

\[
MBC \pm t_{1-\alpha} \frac{s}{\sqrt{n}}
\]  

(13)
The observed values of the average and standard deviation of MBCs at each down-sampled resolution are denoted by $\overline{MBC}$ and $s$, respectively. Student’s t-distribution on (n-1) degrees of freedom was used as a benchmark with a significance level $\alpha = 0.05$ to provide two-sided 95% confidence intervals for each computed MBC. Note that a t-distribution was used here as a conservative estimate to the standard normal distribution, due to the limited sample sizes of $n = 5$ and $n = 20$. 
3 Framework Applications using Phantom Microstructures

This section presents applications of the previously described microstructure modeling framework. Section 3.1 details work published by the author et al. [98,99] that deals with modeling the effect of resolution on the accuracy of grain ensemble statistics for a single-phase lognormal microstructure. Section 3.2 similarly details an investigation into the effect of both resolution and noise on the accuracy of grain ensemble statistics for a single-phase lognormal microstructure. Section 3.3 details a case study where a virtual model was created and down-sampled based on actual experimental data for the nickel-based superalloy Inconel 100 [87], and conclusions are drawn regarding the replication of the experimental data in efficient ways for future experiments, depending on the microstructural statistic(s) of interest.

3.1 Resolution Down-Sampling of Single-Phase Lognormally-Distributed Phantom Microstructures

3.1.1 Phantom Generation

A lognormal grain size distribution was used to instantiate phantom grain ensembles for this study, with a mean and standard deviation of $\mu = 0.85$ and $\sigma = 0.705$. The mean of the distribution corresponds to an equivalent sphere diameter of 3.0, and the standard deviation selected results in a heavy tail for the lognormal distribution, providing a variety of large and small grains for all of the reference volumes. Note that a hard limit for the lognormal size distribution was selected, where the maximum equivalent sphere diameter is equal to 14.0. The shapes of the grains were constrained to be nearly equiaxed with no preferential spatial orientation. The reference volumes all contained over 3000 grains, including approximately 1500 grains that did not contact the surface of the reference volumes. Surface grains are considered to have biased microstructural statistics and therefore were not considered in this analysis. The spatial resolution for each reference volume was selected to have 30 voxels
spanning the diameter of a grain that corresponds to the mean of the lognormal grain size
distribution. Hereafter, spatial resolutions of both the reference and down-sampled volumes are
defined relative to the mean grain diameter, and referred to as VRAD (Voxels Relative to the
Average feature Diameter).

3.1.2 Down-Sampling

As mentioned previously, the reference volumes were generated at relatively high
resolutions, 30 VRAD. Figure 12A shows one of the high-resolution reference volumes, which
visually appears to have smooth, curved boundaries. This is compared to the isotropically and
anisotropically down-sampled volumes shown in Figure 12B and Figure 12C, which have voxel
dimensions at least ten times larger than the reference volume.

3.1.3 Isotropic Down-Sampling Results and Discussion

Isotropic down-sampling was performed on reference volumes, resulting in the following
resolution values that are reported in VRAD: 1, 2, 3, 5, 6, 10, and 15. Using this nomenclature,
the reference volume was constructed with a VRAD of 30, and an isotropically down-sampled
volume with a VRAD of 10 refers to voxels with edge lengths that are 3 times larger than voxels

Figure 12: High Resolution Reference Volume Down-Sampling: An example of isotropic and
anisotropic down-sampling of a polycrystalline phantom microstructure. A) High resolution
reference volume constructed at 30 VRAD. B) Isotropic down-sampling to 3 VRAD, C)
Anisotropic down-sampling to 3 VRAD “in-plane” and less than 1 VRAD “out-of-plane.”
in the reference volume. The down-sampling resolution values were selected so that the voxel centroids of the down-sampled volume directly corresponded to selected voxel centroids in the reference volume. This was done to eliminate aliasing artifacts in the discrete voxel sampling process.

The data from all five phantom grain ensemble microstructures was used to generate the discrete PDFs shown in Figs. 13-17. Each data point in the discrete PDF plots (part A in each figure) represents the average value for that particular bin. The associated error bars, derived using Student’s t distribution and the process described previously, represent the variation observed between PDFs derived from the five individual phantom microstructures. Further, part B of each figure plots the error in individual PDF bins between the approximate (down-sampled) and exact (reference) values. These error plots highlight the region(s) of each microstructural PDF that are particularly affected by voxel down-sampling.

3.1.3.1 Grain Size Distributions

Grain size PDFs as a function of isotropic down-sampling are shown in Figure 13A. A 28-bin scheme ranging from 0 to 14.0 was used to create the grain size PDFs, effectively providing each individual bin with a size range of a 0.5. The solid black line in this figure corresponds to the reference PDF. One can observe from this figure that the grain size distributions are well-represented even at relatively coarse resolutions, where only a few voxels span the average feature diameter. In fact, representing the grain microstructure with a resolution of more than 5 VRAD does not markedly change the prediction of the underlying PDF, as evidenced by the overlapping confidence intervals at all PDF bins. Only when the resolution decreases from 30 down to 3 VRAD is there a significant change in the estimation of the underlying grain size distribution.
The error-in-bin-population plot in Figure 13B shows quantitatively which bins of the underlying grain size PDF are most affected by voxel down-sampling. Structures down-sampled to 10 VRAD approximate the underlying size distribution exceptionally well, and are shown to have nearly zero bin population error across the entire range of the PDF. Even resolutions as low as 5 VRAD sustain individual bin population errors of 0.01 or lower. Also, the PDF bins most affected by increasing voxel dimensions correspond to the grains with the smallest equivalent sphere diameters. This is intuitive because as voxel dimensions become larger, smaller grains are represented by fewer and fewer voxels. As coarsening continues, voxels grow large enough to result in the elimination of entire grains as a byproduct of the discrete down-sampling process.

Any alteration or loss of microstructural feature information is undesirable, and should be avoided if possible in real experiments. This begs the question of how much information loss is too much. Analysis of Figure 13A provides insight into this issue through the comparison of down-sampled phantom PDFs, at 3 and 5 VRAD, respectively. In both of the down-sampled phantom distributions, the bin corresponding to the smallest grain volumes no longer contains any grains. However, the histogram for the 3 VRAD down-sampling also grossly overestimates the bin that contains the highest fraction of grains, which is not observed in the 5 VRAD volumes. Even though both phantom distributions experienced information loss, only when the phantom microstructures were down-sampled to a 3 VRAD did the PDF no longer appear visually to be a good fit. In order to provide a more quantitative analysis of the effect of spatial resolution on the similarity of the feature histograms, use of the Modified Bhattacharyya Coefficient (MBC) is employed, and these results are presented and discussed in section 3.1.3.4.
Figure 13: **Grain Size Distributions**: Reference and down-sampled grain size PDFs from the 5 phantom ensemble microstructures investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 13B shows the population error contained within each bin.
3.1.3.2 **Grain Shape Distributions**

The shape distributions examined in this study are those having aspect ratios \(b/a\) and \(c/a\) and the moment invariant \(\Omega_3\). Each of these parameters is always bounded between 0 and 1, and in this study PDFs were constructed using 50 equally-spaced bins for each case. Figures 14 and 15 show the reference and down-sampled aspect ratio PDFs and the corresponding individual bin population errors for the PDFs. In comparison to the grain size distributions discussed in the previous section, the PDFs of aspect ratio values are more sensitive to changes in the spatial resolution of the data. For example, one can observe that coarsening the voxel size by a factor of 2 (change in spatial resolution from 30 to 15 VRAD) results in at least one histogram bin for the \(b/a\) ratio having a bin population error value greater than 0.01. Decreasing spatial resolution further to a VRAD of 6 results in more significant bin population errors, with a moderate number of bins displaying errors greater than 0.04. While the bin population errors associated with the grain size PDFs were concentrated in the left tail—corresponding to the smallest grains—the bin population errors in the PDFs for aspect ratio tend to be more evenly distributed. Note that the curves shown in the bin population error plots (Figures 14B and 15B) appear essentially offset from one another, indicating that all of the grain aspect ratio bins are being affected via changes in spatial resolution. As mentioned in section 3.1.1, all of the grains were ascribed roughly spherical shapes—corresponding to aspect ratios close to 1.0, and as a result there was no significant bias in the aspect ratio PDFs based on grain sizes. Therefore, it is not surprising that all bins for the aspect ratio PDFs are affected during down-sampling for these phantom microstructures.
Figure 14: Aspect Ratio b/a Distributions: Reference and down-sampled aspect ratio b/a PDFs from the 5 phantom ensemble microstructures investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 14B shows the population error contained within each bin.
Figure 15: Aspect Ratio c/a Distributions: Reference and down-sampled aspect ratio c/a PDFs from the 5 phantom ensemble microstructures investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 15B shows the population error contained within each bin.
The PDF for the higher order shape descriptor $\Omega_3$ is even more affected by spatial resolution, and none of the down-sampled data provide even a qualitatively accurate representation of the underlying reference PDF. As shown in Figure 16A, the two most-populated bins contain population errors in excess of 0.05, even at the minimum down-sampling from 30 to 15 VRAD. Note that the large error bars for the most populated $\Omega_3$ bins signify that the 5 reference volumes do not consistently reproduce the same $\Omega_3$ distribution. This result indicates that even higher resolution reference volumes are necessary. Consequently, this study was repeated with extremely high resolution reference volumes (that contained fewer grains due to computational memory limits), and the results of this high-resolution study are discussed in detail in section 3.1.3.4.
Figure 16: Moment Invariant $\Omega_3$ Distributions: Reference and down-sampled moment invariant $\Omega_3$ PDFs from the 5 phantom ensemble microstructures investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 16B shows the population error contained within each bin.
3.1.3.3 **Number-of-Nearest-Neighbor Distributions**

The number of nearest neighbors for any single grain in the 5 reference volumes fluctuated from 1 to 121 neighbors. Upon examination of the nearest neighbor distributions, it was observed that for bins greater than 25 neighbors, the down-sampled volumes produced nearly identical PDFs. Therefore, the number-of-nearest-neighbor distributions in Figure 17A present only bins ranging from 1 to 25 neighbors.

Figure 17A and 17B show that the number-of-nearest-neighbor PDFs are relatively insensitive to changes in spatial resolution down to 3 VRAD. One can observe that the error bars in Figure 17A, which signify 95% confidence in each PDF bin population estimate, vary with the number of nearest neighbors being considered. For coarse down-samplings, the error bars corresponding to grains with the largest number of neighbors remain consistently small, while the smaller grains associated with fewer neighbors have a high degree of potential error in their bin estimates. The bin population error plot in Figure 17B clearly supports this conclusion by highlighting the region of nearest neighbor PDFs that is most affected by voxel size, the left tail. As was also the case with grain size distributions, structures down-sampled to 10 VRAD approximate the underlying PDF exceptionally well, and are shown to have nearly zero bin population error across the entire range of the distribution. Note that the high accuracy of this descriptor at a relatively low resolution is to be expected, as the grains’ shapes must be significantly distorted and/or grains must be removed from the volume via the down-sampling process in order to affect this quantity.
Figure 17: **Number-of-Nearest-Neighbor Distributions**: Reference and down-sampled number-of-nearest-neighbor PDFs from the 5 phantom ensemble microstructures investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 17B shows the population error contained within each bin.
3.1.3.4 Similarity Analysis of Isotropically Down-Sampled PDFs

A statistical analysis of the isotropically down-sampled data is shown in Figure 17, where part A compares average values of each distribution via the true percentage error—defined as the absolute value of the difference between reference and down-sampled means, divided by the reference mean and reported as a percent—and part B presents the results of the Modified Bhattacharyya Coefficient (MBC) analysis. The general trends described in previous sections are supported by Figures 18A and 18B. For example, in Figure 18B one can observe that the MBCs for both the grain size and number-of-nearest-neighbor distributions are relatively insensitive to changes in resolution. Additionally, this figure also clearly shows that the shape descriptors of aspect ratio and $\Omega_3$ are much more sensitive to changes in resolution, as discussed previously.

It is interesting to note that all of the number-of-nearest-neighbor PDFs exhibit some modest dissimilarity to the initial reference volume (MBC values > 0.1), but the relative change in the MBC appears to be very small between subsequent down-samplings to values as low as 3 VRAD. Meanwhile, the other microstructural PDFs investigated appear to maintain much more consistent and monotonically increasing rates of change in the MBC with increasing VRAD. Although the source of this anomalous down-sampling response of the number-of-nearest neighbor distributions has not clearly been identified, one possible explanation is that there is fine local morphological detail within the initial phantom reference microstructure that is quickly eliminated during down-sampling, even for the first down-sampling by a factor of 2.

One application for utilizing the results shown in Figure 18 with regards to experimental data collection is to define a criterion for the maximum allowable variation in the measured distribution(s) relative to the reference volume, perhaps considering multiple microstructural
descriptors, which can be used to define a minimally-sufficient sampling resolution (VRAD). A simple example would be to define a threshold value for the estimated error of any single bin of the PDFs. For instance, if a threshold value of 0.02 was selected, inspection of Figures 13 and 18 reveal that values of 5 and 3 VRAD would be acceptable for the grain size and the number-of-nearest-neighbor PDFs, respectively. Aspect ratio PDFs require much higher resolutions, as the 10 VRAD structures from the \( \frac{b}{a} \) and \( \frac{c}{a} \) PDFs produced max bin errors of 0.032, and 0.020, respectively, while the 15 VRAD structures resulted in max bin errors of 0.016 and 0.008. One can see from this simple analysis that if the experimental data is to be used to calculate PDFs for all three descriptors with the same threshold for allowable error, the aspect ratio measurements define the isotropic VRAD for data collection. Note that using these values of 5, 3, and 15 VRAD for the grain size, number-of-nearest-neighbor, and aspect ratio (\( \frac{b}{a} \) and \( \frac{c}{a} \)) PDFs, corresponding MBC values can be found in Figure 18B, which are 0.07, 0.13, 0.09, and 0.06, respectively. Therefore, in this particular example a maximum allowable bin error of 0.02 is equivalent to selecting a MBC \( \leq 0.13 \), recognizing that this equivalency is only demonstrated for both the microstructure metrics and for the lognormally-distributed, single-phase phantom microstructure under consideration. Extending this analysis to consider additional metrics and alternate microstructures is the subject of a future study.
Figure 18: Isotropic Down-Sampling Mean and Modified Bhattacharyya Coefficient (MBC) Results

Reference volumes constructed at 30 VRAD were subsequently down-sampled to 1, 2, 3, 5, 6, 10, and 15 VRAD. Figure A) The percentage error in estimating the average value of the PDF for equivalent sphere diameter (ESD), aspect ratios (b/a, c/a), the moment invariant $\Omega_3$, and number of nearest neighbors (NNN) compared to the reference volume. Figure B) The value of the MBCs calculated between the reference and down-sampled PDFs.
3.1.3.5 **High Resolution Shape Study**

As mentioned earlier in section 3.1.3.2, in order to improve the quantitative analysis of the effect of spatial resolution on the PDF of the moment invariant $\Omega_3$, a second higher resolution study was performed. Reference volumes were constructed at much higher voxel resolution compared to the initial reference volumes, at VRAD of 30, 40, 50, 60, 75, and 100. The MBC analysis results for the down-sampling of these very-high resolution microstructures are presented in Figure 19. Due to computational memory limits, the number of grains generated for these high resolution phantom microstructures decreased with increasing resolution; likewise, the total volume also decreased with increasing VRAD. For comparison, while the original reference volumes at 30 VRAD contained approximately 1500 unbiased grains, the 100 VRAD reference volume contained only 131. This is a direct result of the limitations of computational systems used in this study, as all microstructures were created on a 64-bit machine with 16 gigabytes (GB) of random access memory (RAM). Despite the limited number of grains that are contained in the higher resolution phantoms, one can observe from Figure 18 that for threshold criteria of MBC ≤ 0.13, the sampling resolution to achieve at least this amount of similarity with the reference distribution is no less than a value of approximately 20 VRAD. Additionally, increasing spatial resolution of the reference volume beyond 30 VRAD resulted in MBC-vs-resolution curves that appear to converge on one another at lower VRAD values, despite the differences in individual phantom grain ensemble sizes. This observation indicates that going to higher voxel resolution is a potential remedy for examining the resolution sensitivity of higher order shape descriptors, although these shape studies should be repeated on polycrystalline ensembles with smoothed grain boundaries to eliminate artifacts caused by a voxel-grid representation.
Figure 19: High Resolution Shape Study: Reference volumes constructed at 30, 40, 50, 60, 75, and 100 VRAD were each down-sampled, and these high resolution structures suggest that a threshold MBC of 0.13 can be achieved for $\Omega_3$ distributions between 20 and 50 VRAD. Note that smaller phantom grain ensembles were examined due to limitations of the available computational systems used for this study.

3.1.4 Anisotropic Down-Sampling Results and Discussion

In addition to examining the effect of spatial resolution using isotropic down-sampling procedures, this study also considered an anisotropic down-sampling procedure. Anisotropic voxel data is often associated with experimental 3D data collection, for both destructive and nondestructive microscopy experiments. Here, down-sampling has been explored similar to what would be associated with serial sectioning experiments, where the in- and out-of-plane resolutions are governed by independent systems. In serial sectioning, the in-plane resolution is determined by the choice of microscopy method and the user-defined sampling frequency, while the fidelity of the sectioning process and the user-defined sampling frequency often determines the out-of-plane resolution.
In this study, down-sampling was performed on reference volumes in such a way as to allow the in- and out-of-plane resolutions to vary as multiples of one another. As with the isotropic down-sampling procedure, care had to be taken to select combinations of in- and out-of-plane resolutions to minimize aliasing artifacts. Anisotropy was first considered by allowing the out-of-plane resolution to be reduced by factors of 2 and 3 from the in-plane resolution. In a similar way, anisotropy was also considered by allowing the in-plane resolution to be reduced by factors of 2 and 3 from the out-of-plane resolution. Reducing the out-of-plane resolution mimics using larger sectioning thicknesses, and reducing the in-plane resolution allows the isotropic case to represent the use of a higher detector resolution.

The results from the anisotropic down-sampling study are shown in Figure 20, which plots the MBC as a function of VRAD for both the ESD and primary aspect ratio (b/a). The legend in Figure 20 is labeled using x-, y-, and z-direction multipliers, and these multipliers are used to discern the x-, y-, and z- resolutions in VRAD for a particular data point. For instance, label 1:1: \( \frac{1}{2} \) corresponds to an out-of-plane sampling resolution that is twice as large compared to the in-plane resolution. Therefore, for this sampling resolution (1:1: \( \frac{1}{2} \)), a data point located at a resolution of 10 VRAD corresponds to a phantom microstructure with resolutions in the x-, y-, and z-directions of 10, 10, and 5 VRAD, respectively.

Figure 20 represents one way to analyze the effect of various experimental settings as a precursor to 3D experimental data collection. As an exercise, one can consider maintaining a constant value of similarity to the reference distribution (i.e., keeping a constant MBC value) and varying resolution either in- or out-of-plane depending on experimental conditions to optimize the data collection process. If one considers the example discussed in the previous section of a threshold MBC value of 0.13, this value can be achieved using a variety of different anisotropic sampling schemes. If one is interested in only the grain size distribution, Figure 20A shows that
data collected at 5-6 VRAD can withstand anisotropic decreases in the out-of-plane resolution with minimal losses in accuracy, as the 1:1: $\frac{1}{2}$ and 1:1: $\frac{1}{3}$ anisotropic curves satisfy this MBC threshold. Therefore, rather than the original number of sections, every other section could have been eliminated (or even every two-out-of-three sections) while still achieving a sufficient level of similarity to the reference PDF. The aspect ratio $b/a$ results presented in Figure 20B again show that shape measurements are more sensitive to changes in spatial resolution.
Figure 20: **Anisotropic Down-Sampling of Grain Size and Aspect Ratio b/a Distributions:**
Reference volumes constructed at 30 VRAD were down-sampled to demonstrate potential tradeoffs between PDF accuracy and experimental efficiency in 3D microstructural characterization. Results for grain size and aspect ratio b/a PDFs are shown in (A) and (B), respectively.
3.1.5 Conclusions

This study presents a general computational framework to analyze the accuracy of microstructural distribution measurements derived from 3D voxel data, using phantom grain ensembles and discrete down-sampling procedures. The methodology was applied to microstructural descriptors for grain size, grain shape, and the number of nearest neighbor grains on a single-phase microstructure. The similarity of down-sampled probability distributions to the reference distribution has been investigated by examining the relative bin error as well as using the Modified Bhattacharyya Coefficient (MBC) similarity metric. It is impossible to draw universal conclusions regarding the appropriate sampling resolution for all of the possible microstructural descriptors and provide an improved general rule of thumb of “10 sections through the average feature.” Rather, it has been shown for a phantom-generated lognormally-distributed grain structure (μ = 0.85 and σ = 0.705) that the following isotropic sampling resolutions result in microstructural distributions that are very similar to the reference distributions, as defined by a Modified Bhattacharyya Coefficient threshold value of 0.13:

- 3 VRAD for the number-of-nearest neighbor PDF
- 5 VRAD for the equivalent sphere diameter PDF
- 10 VRAD for aspect ratio b/a and c/a PDFs
- 20-50 VRAD for the moment invariant Ω₃ PDF

3.2 Resolution and Noise Down-Sampling of a Single-Phase Lognormally-Distributed Phantom Microstructure

3.2.1 Phantom Generation

The same lognormal grain size distribution (LN(0.85, 0.705)) used in the previous study was used to instantiate the phantom reference volume used for this study. The shapes of the grains
were again constrained to be nearly equiaxed with no preferential spatial orientation, and the reference volume contained over 3000 grains including approximately 1400 grains that did not contact the surface. The spatial resolution was also similarly selected to be 30 VRAD ($V_{\text{O}} \times \text{R}_{\text{el}}$ $V_{\text{oxels}}$ $R_{\text{el}}$ $A_{\text{verage feature D}_{\text{iameter}}}$). In this study, only one reference volume was generated, however for each noise level investigated, twenty instantiations were used. This resulted in particularly small error bars for nearly all analyzed data, with slight exception to only the highest noise levels.

### 3.2.2 Down-Sampling

The process of inducing errors into the reference microstructure begins by isotropic down-sampling. Following down-sampling, virtual noise is introduced into the microstructure. The noise is introduced in one of two ways, either as noise located along grain boundaries or as noise randomly distributed throughout the microstructure. These classifications were chosen in an effort to mimic data obtained via experimental EBSD scans, as referenced in Figure 4.

Selected noise models from this work are depicted prior to cleanup in Figs. 21-22. Figure 21A and 22A both show the high resolution reference volume, which can be compared to the corresponding down-sampled (5VRAD) volumes that each contain 75% noise, where 75% boundary noise is depicted in Figure 21B and 75% random noise (i.e. 75% of total voxels) is shown in Figure 22B.
A key point that should be highlighted is that random noise percentages correspond to the percentage of the total voxels contained in the reference or down-sampled volume, while boundary noise percentages are applied to only those voxels that lie on grain boundaries. Moreover, the percentage of the total number of voxels located on grain boundaries increases for
lower resolutions. A comparison of this effect relative to the total number of voxels at various resolutions is shown in Table 2.

**Table 2: Boundary Voxels as a Percentage of Total Voxels**: Comparison of the number of boundary voxels found at different resolutions and the percentage of total voxels corresponding to 75% boundary noise.

<table>
<thead>
<tr>
<th>Resolution (microns)</th>
<th>Resolution (VRAD)</th>
<th>% Boundary Voxels (of Total Voxels)</th>
<th>75% Boundary Noise = % Total Noise</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>1 VRAD</td>
<td>95.63</td>
<td>71.72</td>
</tr>
<tr>
<td>1</td>
<td>3 VRAD</td>
<td>59.75</td>
<td>44.81</td>
</tr>
<tr>
<td><strong>0.6</strong></td>
<td>5 VRAD</td>
<td>42.10</td>
<td>31.58</td>
</tr>
<tr>
<td>0.3</td>
<td>10 VRAD</td>
<td>23.78</td>
<td>17.84</td>
</tr>
<tr>
<td><strong>0.2</strong></td>
<td>15 VRAD</td>
<td>16.49</td>
<td>12.37</td>
</tr>
<tr>
<td><strong>0.1</strong></td>
<td>30 VRAD</td>
<td>8.61</td>
<td>6.46</td>
</tr>
</tbody>
</table>

Examination of Table 2 reveals that that only in the limiting case where every voxel lies on a boundary (i.e. each voxel is a single grain) are the percentages of boundary and random noise equivalent to one another in a number-of-voxels sense. However, since data collection would not likely be performed at resolutions 1 VRAD or below, one must take care in drawing conclusions regarding the type of noise that has a larger effect on the overall accuracy of microstructural statistic(s) of interest.

### 3.2.3 Isotropic Down-Sampling with Noise Results and Discussion

Isotropic down-sampling was performed on the single reference volume constructed at 30 VRAD, resulting in the following resolution values that are reported in VRAD: 3, 5, 10, and 15. Random and boundary noise are investigated at levels of 0, 10, 25, 50, and 75%. The data from all twenty phantom grain ensemble microstructures was used to generate the discrete PDFs shown in Figs. 23-28. Each data point in the discrete PDF plots represents the average value for that particular bin from the twenty instantiations of noisy structures created. The associated error bars, derived using Student’s t distribution and the process described previously, represent the
variation observed between PDFs from different instantiations. The error in individual PDF bins is displayed for the 0% noise case (i.e. resolution only) in part B of Figs. 23, 25, and 27 relative to bin values observed in the reference volume; however, these figures contain no error bars due to the investigation of noise being conducted on a single phantom reference volume. Figs. 24, 26, and 28 focus on the highest noise case investigated (75%) for both random and boundary noise, and provide the associated distributions collected from the set of microstructures following cleanup.

Similarity analysis via percent error in mean values as well as the MBC is addressed in Figs. 29-32. Figure 29 provides an analysis of percent error in mean values observed for all noise levels at different resolutions, for both grain size and the number-of-nearest neighbors. Figs. 30-31 show a similarity analysis using the MBC for grain size at each noise level for both boundary and random noise, where Figure 30 plots the MBC vs. resolution in VRAD with lines of constant noise levels, and Figure 31 plots the MBC vs. percent noise with lines of constant resolution. Figure 32 analyzes the left- and right-tail of grain size distributions observed for different levels of random noise.

3.2.3.1 Grain Size Distributions

Grain size PDFs as a function of isotropic down-sampling and noise + cleanup are shown in Figs. 23 and 24. As in the previous study, a 28-bin scheme ranging from 0 to 14.0 was used to create the size PDFs. The solid black line in these figures corresponds to the reference PDF with 0% noise.

One can observe from Figure 23 that the grain size distribution is well-represented down to 5 VRAD, a result consistent with the previous study. Interestingly, Figure 24 provides insight into the similar effects that both boundary and random noise have on size distributions. The effect of noise is observed to be greatest at the lowest resolution investigated (3 VRAD). The surprising
outcome from Figure 24 is that the application of even an extremely high level of noise (75%) produces size distributions that are qualitatively similar to those observed in the 0% noise case. This means that noise, even when it is applied to eliminate ¾ of the overall data contained in the volume, can actually have little-to-no-effect on the resulting size distributions. This rather counterintuitive result is observed throughout this study, and is due to the combination of two effects: 1) equiaxed microstructures are used for analysis, and 2) the geometric cleanup routine used to eliminate noisy data operates in a very similar way to the packing algorithm used to create the virtual data in the first place. Thus, even though the cleanup algorithm was not designed to be based on microstructural physics, it happens to capture the physics of equiaxed structures quite well. Therefore, a high degree of noise can be tolerated during the down-sampling process.
Figure 23: **Grain Size Distributions, 0% Noise**: Reference and down-sampled grain size PDFs from the phantom ensemble investigated. Data points shown correspond to the left edges of bins used for PDF construction. Figure 23B shows the population error contained within each bin.
Figure 24: Grain Size Distributions, 75% Boundary and Random Noise: Reference and down-sampled grain size PDFs from the 20 phantom ensembles investigated with 75% boundary noise (A) and 75% random noise (B). Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures.
Classic electron microscopy suggests poor signal-to-noise ratios resulting from reduction in electron beam currents can be “serious problems” [17] from a microstructural characterization standpoint, and that the “best” data results from data collection procedures that produce as little noise as possible. However, for equiaxed structures using the cleanup algorithm described here, it appears that the most efficient and cost-effective data collection may not be the one that reduces noise, but rather be one that uses lower beam currents, larger spot sizes, and much faster data collection times, albeit with much higher uncertainty in the experimentally-collected data prior to cleanup.

The implications of these results are far-reaching in that even a simple visual analysis of these size distributions supports collecting significantly less data than is actually required to represent equiaxed structures, then letting the cleanup algorithm take care of filling in the blanks. For example, comparing Figures 23A and 24B it can be observed that collecting data at 10 VRAD, but only collecting \( \frac{1}{4} \) of data overall, produces a much better result than collecting data at 5 VRAD, where each data point is obtained with confidence. Since 5 VRAD corresponds to 0.6 \( \mu \)m step sizes for EBSD analysis, and 10 VRAD corresponds to 0.3 \( \mu \)m step sizes, on a single section of data that is 50 x 50 \( \mu \)m\(^2\) (the 2D field of view of the virtual microstructures generated in this study), the 5 VRAD case corresponds to \((50/0.6)^2 = 6944\) total pixels and the 10 VRAD case, collected at every 4th pixel also corresponds to \((50/0.3)^2 \times 0.25 = 6944\) total pixels. Thus, collecting the same amount of data in approximately the same time, but doing so in a more intelligent and counter-intuitive way, can provide significant benefit and increase the overall quality and of data following cleanup.

### 3.2.3.2 Grain Aspect Ratio b/a Distributions

The shape distributions examined in this study are those of the aspect ratio \( b/a \). As in the previous work, \( b/a \) and \( c/a \) distributions produced similar results, so here only distributions from
the aspect ratio \( b/a \) used to draw conclusions. This study used 25 equally spaced bins for inspection of aspect ratios, rather than 50 as in the previous study. By comparison of Figs. 14 and 25, it can be observed that there is not a significant difference in the resulting conclusions about data sampling required; namely, that roughly 10 VRAD is an appropriate resolution to accurately describe aspect ratio distributions.

Although aspect ratios are more sensitive to resolution in general, a trend similar to that identified for grain size distributions can be observed. Rather than the minimum sampling resolution that is required for accuracy being between 3 and 5 VRAD, as in the case of grain sizes, Figure 25 shows that minimally-sufficient sampling for aspect ratios lies somewhere between 5 and 10 VRAD. However, inspection of Figure 26 reveals similar conclusions to Figure 24; namely, that random noise has a more significant effect, especially at lower resolutions, and that a very high amount of noise can be tolerated based on the cleanup algorithm that is employed. In this case, if one were to collect at 15 VRAD with 75% noisy voxels, the underlying distribution could still be well represented. For a volume of 50 µm\(^3\) as investigated for this study, this would result in total data collection volume (assuming isotropic sampling) of \((50/0.3)^3 = 4,629,629\) voxels for the 10 VRAD case, and \((50/0.2)^2*0.25*(50/0.2) = 3,906,250\) voxels for the 15 VRAD, 75% random noise case. It should be noted that the 15 VRAD, 75% random noise case would also require more sections of data, and thus more polishing time. However, the total amount of data still decreases when using a sampling scheme with such high levels of allowable noise.
Figure 25: Grain Aspect Ratio b/a Distributions, 0% Noise: Reference and down-sampled aspect ratio b/a PDFs from the phantom ensemble investigated. Data points shown correspond to the left edges of bins used for PDF construction. Figure 25B shows the population error contained within each bin.
Figure 26: Grain Aspect Ratio $b/a$ Distributions, 75% Boundary and Random Noise:
Reference and down-sampled aspect ratio $b/a$ PDFs from the 20 phantom ensembles investigated with 75% boundary noise (A) and 75% random noise (B). Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures.
3.2.3.3 Number-of-Nearest Neighbor Distributions

For this study, the number of nearest neighbors for any single grain in the reference volume fluctuated from 1 to 83 neighbors for this study. As in the previous study, for bins greater than 25 neighbors, the down-sampled volumes produced nearly identical PDFs. Therefore, the number-of-nearest-neighbor distributions in Figure 27A present only bins ranging from 1 to 25 neighbors.

The effect of noise + cleanup on equiaxed grains is perhaps more apparent for the number-of-nearest neighbors distributions than for the grain size and aspect ratio $b/a$ distributions investigated in the previous sections. These distributions are relatively insensitive to noise overall, up to the point of minimally-sufficient sampling (this case between 3 and 5 VRAD, as observed for grain size). By inspection of Figure 28, it is clear that increased noise levels tend to increase the number of grains with a small number of neighbors, likely due to the elimination of entire grains associated with the process of inducing noise. When entire grains are eliminated, there are fewer small grains left to serve as neighbors throughout the microstructure.
Figure 27: **Number-of-Neighbor Distributions, 0% Noise**: Reference and down-sampled Number-of-Neighbor PDFs from the phantom ensemble investigated. Data points shown correspond to the left edges of bins used for PDF construction. Figure 27B shows the population error contained within each bin.
Figure 28: Number-of-Neighbor Distributions, 75% Boundary and Random Noise:
Reference and down-sampled Number-of-Neighbor PDFs from the 20 phantom ensembles investigated with 75% boundary noise (A) and 75% random noise (B). Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures.
3.2.3.4 Similarity Analysis of Isotropically Down-Sampled PDFs

Formulas in materials science and engineering are often focused on the mean value of distributions, especially for grain size (e.g., Hall-Petch Relationship [100]). A statistical analysis of the mean values in isotropically down-sampled data for grain size and number-of-nearest-neighbor distributions is shown in Figure 29, where part A shows the results from the boundary noise study and part B represents results from the random noise study. Average values of each distribution are compared via the percentage error relative to the reference mean, and in this investigation no absolute value simplification is used. As a result, the directionality associated with increasing or decreasing mean values as errors are introduced can also be observed.

Grain size and number-of-nearest neighbor mean values can be seen to trend in opposite directions as shown in Figure 29. The mean grain size predictions increase with resolution down-sampling, while predictions for the mean number-of-neighbors decrease. This is intuitive, because as smaller grains are eliminated from the volume via down-sampling, there are fewer neighbors left to be had. Similarly, as the volume of smaller grains is absorbed by larger ones, the average grain size increases.

As shown for distributions above, Figure 29 shows clearly that for mean values, noise has a much greater effect at lower resolutions. In comparing Figures 29A and 29B, it is also clear that the mean value of the number of neighbors is predicted significantly better with higher and higher levels of noise, while the prediction of mean grain size becomes worse and worse for increasing noise levels. This is again a result of the cleanup algorithm used, because as grains with unknown data between them grow isotropically outward, there is an inclination for converging grains to become neighbors even if they did not share any voxel surface area in the original reference volume.
For the 75% noise case, the mean value of both grain size and the number-of-nearest neighbors is surprisingly well approximated (≤ 5% error) at resolutions as low as 5 VRAD. This means that if only mean values are of interest, data can not only be collected using extremely coarse resolution, but also be collected in a very sparse manner with a significant amount of unknown data. The results from the 10, 25, and 50% noise cases all fell within the bands exhibited by the solid and dashed lines for grain size (blue) and number-of-nearest neighbors (green).
Figure 29: Isotropic Down-Sampling with Noise, Mean Value Results: Reference volumes constructed at 30 VRAD were subsequently down-sampled to 3, 5, 10, and 15 VRAD. Figs A and B show the percentage error in estimating the average value of the PDF for equivalent sphere diameter (ESD), and number of nearest neighbors (NNN) in boundary noise and random noise, respectively.
Similarity analysis via the MBC for grain size is shown in Figure 30 and 31 for all values of resolution and noise investigated. Figure 30A shows results for the application of boundary noise + cleanup, and Figure 30B shows results for the application of random noise + cleanup. Figure 31 is an alternative representation of Figure 30 that plots the MBC vs. noise while holding resolution constant.

Grain size distributions were considerably affected only at extremely high levels of random noise for low resolutions (3 VRAD, 5 VRAD), while MBC values remained almost unaffected by even the highest levels of boundary noise until the lowest resolution (3 VRAD) structures were investigated. This can be seen clearly in Figs. 31A and 31B, where the 3 VRAD line corresponds an MBC value that increases rapidly from 50 to 75% for both types of noise. In Figure 31B, there is also a slight increase from 50 to 75% random noise for the 5 VRAD case. This is consistent with data displayed in Table 2, where 75% boundary noise at 3 VRAD represents 44.81% of total voxels. Since 50% of total voxels at 5 VRAD is where the random noise curves start to deviate, it may be that approximately 50% noise is the limiting amount that can be tolerated before significant errors occur for this structure and cleanup algorithm combination.
Figure 30: Similarity Analysis of Grain Size Distributions between Boundary Noise Levels:
Similarities computed for full microstructural parameter distributions, ESD, b/a, c/a, and NNN relative to the reference volume.
Figure 31: Similarity Analysis of Grain Size Distributions between Boundary Noise Levels @ 5 VRAD: Similarities computed for full microstructural parameter distributions, ESD, b/a, c/a, and NNN relative to the reference volume at a resolution of 5 VRAD.
Using a threshold of $MBC \leq 0.13$, it appears that grain size distributions can be well-represented down to 5 VRAD, and tolerate up to 75% noisy data. However, when one applies the additional threshold of percent difference in each bin $\leq 2\%$, this is not the case. Figure 32 shows that in fact 10 VRAD is mandatory at 75% noise in to satisfy this requirement in addition to the $MBC \leq 0.13$. Thus, it is not sufficient to draw conclusions when doing virtual modeling studies based solely on the similarity value computed between reference and down-sampled distributions. Care must be taken to observe the actual distributions in addition to computing quantitative similarity metrics. This type of analysis could be enhanced by adding additional thresholds for other statistics such as mean values, as is done in the case study presented in following section.
Figure 32: Grain Size Distributions, 75% Noise: Reference and down-sampled grain size PDFs from the 20 phantom ensembles investigated. Data points shown correspond to the left edges of bins used for PDF construction. Note that 95% confidence intervals indicate the variation observed between PDFs derived from individual phantom microstructures. Figure 32B shows the population error contained within each bin.
A good example of how the effects of resolution and noise can be partitioned into various parts of the distributions can be seen by inspection of Figure 33 with comparison to Figure 30B. Figure 30B shows the analysis of full distributions from down-sampling with random noise + cleanup experiments. Figure 33A correspondingly shows the effects that resolution and random noise + cleanup have on only the smallest 25% of grains, where Figure 33B displays results for only the largest 25% grains. It is clear that the errors present in the full distribution of grain sizes show up mostly on the smallest 25% of grains, as indicated by the scale of the MBC in Figure 33B. The biggest grains go largely unaffected by resolution down-sampling or the addition of noise at any resolution + noise combination investigated. This means that if one were only interested in the extremely large values of the distribution, data could be collected at a significantly coarser and noisier level, resulting in an even faster characterization experiment.

Similar effects were observed in this study for the number-of-nearest neighbors, since grains with a low number of neighbors typically correspond to grains that are small in size. Application of left- and right-tail MBC analysis is perhaps less useful for aspect ratio distributions that range between 0 and 1, where essentially all grains in the reference volume have aspect ratios of at least 0.5 (i.e. relatively equiaxed structure).
Figure 33: Similarity Analysis of Left- and Right-Tails of Grain Size Distributions between Boundary Noise Levels: Similarities are computed for microstructural parameter distributions left-tails (A) and right-tails (B) for ESD, b/a, c/a, and NNN relative to the reference volume, and all points are shown at a resolution of 5 VRAD.
3.2.4 Conclusions

This study extended the general computational framework to include the effects of noise, both boundary and random, in addition to sampling resolution to determine resolution sufficiency. The methodology was again applied to microstructural descriptors for grain size, grain shape, and the number-of-nearest neighbor grains on a single-phase microstructure. The phantom-generated lognormally-distributed grain structure ($\mu = 0.85$ and $\sigma = 0.705$) exhibits low sensitivities to boundary and random noise based on the equiaxed nature of the structure and the geometric cleanup algorithm that minimizes shared surface area between grains. This is true only until resolution becomes low enough that grain elimination within volumes reaches a critical level, and at this point noise has a much more significant effect.

The results from this study support the idea of collecting less data than is required for an accurate structural representation if the appropriate physics-based cleanup tools can be leveraged. In other words, this study supports using compressed (compressive) sensing techniques [101-103] for microstructural characterization. This has been a very popular area of research in recent years in various scientific communities, and it seems that characterization of material microstructures in 2D and 3D provides yet another application.
3.3 Inconel 100 Case Study: Analysis of a 3D Microstructural Characterization Experiment via Virtual Modeling

This case study performs virtual experiments via the modeling tools developed in this thesis to an experimentally-derived set of microstructural statistics in order to gain insights into optimal data collection schemes for specific microstructural statistics of interest. More generally, it shows a methodology that can be used a priori on microstructural data derived from pilot-run small-scale 3D experiments or even from a single 2D section. However, one must be cautious in using statistics derived from limited data, as they may not necessarily be representative of the overall volume of material being investigated. Thus, these virtual experiments can only be as accurate as the microstructural data used to inform them.

The details of the 3D serial sectioning experiment are as follows: the 96\(\mu\)m x 36\(\mu\)m x 46 \(\mu\)m volume of microstructure collected and was made up of 384 total sections, containing a total of 4373 grains of the nickel-based superalloy Inconel 100 (IN100) [87] and was performed in a DB FIB-SEM.

3.3.1 Phantom Generation

A lognormal grain size distribution was created based on experimental data collected for IN100 as is shown in Figure 34 [87], and provided the best fit to measured grain sizes of the types of distributions investigated. A mean \(\mu = 0.942\) and standard deviation \(\sigma = 0.4209\) were used to instantiate the phantom reference volume for this study, corresponding to an average grain size of 2.8 microns. The average grain size as measured by experiment was reported as approximately 3 microns, so the lognormal fit provides a slightly lower average size. A limit for the size distribution was based on what was observed during experimentation, which as computed from Figure 34 provides no grains having a diameter larger than 10 \(\mu\)m.
**Figure 34: Grain Size Distribution from Experiment for Virtual Model Input:** IN100 experimental grain size data, presented in units of normalized equivalent sphere radius (ESR), where $<\text{ESR}>$ was reported as 1.76 $\mu$m [87].

The shapes of the grains in the virtual model were constrained to be nearly equiaxed with no preferential spatial orientation. The reference phantom was constructed of the same volume ($96\mu$m x $36\mu$m x $46\mu$m) as the experimental data, and was built at with an isotropic resolution of 0.25 $\mu$m in all directions, also corresponding to the experimental data collected. The comparison of the experimental data and the virtual model used for analysis is shown in Figure 35.
Figure 35: Experimental vs. Virtual 3D Representations of Inconel 100: The 3D reconstruction from the actual experiment is shown on the top, and a 3D model of the virtual volume constructed from grain size statistics collected during the experiment is shown on the bottom.
At first glance, these volumes appear visually similar, but not exact replicas of one another. This is due to the fact that the virtual model is assumed to be a single-phase, equiaxed structure. While approximating IN100 as a single-phase, equiaxed structure is common in state-of-the-art microstructural characterization, there are in reality additional complexities associated with IN100 microstructure. For example, the structure contains features known as twins, which are the plate-like features that can be seen within grains in the experimental reconstruction in Figure 35. When performing analysis on IN100 microstructures, these features are often removed by merging them together so that the set of twins is considered to be a single grain. This was done in a follow-up study on the same experimental data by Tucker et al. [104], which reduced the total number of grains contained in the bulk (i.e. unbiased and not touching the surface) from 2265 to 1818 total grains. Additionally, the largest grain in the IN100 data set was nearly 23 μm in diameter, greater than double the largest value in the virtual model. Moreover, there was an observed skewness to the experimental data, with approximately 70% of grains having a grain size less than the average [87].

The experimental volume contained 4373 grains, with 2265 grains considered unbiased, whereas the virtual volume contained 9741 grains, with 5564 considered unbiased. Despite these differences, however, both sets of microstructures produced the same grain size distribution statistics. If it is of interest to the experimentalist to derive information specifically about twins or to get information on individual grains, sampling schemes would need to be tailored toward this task. However, this study assumes that only underlying distributions for grain size, aspect ratio $b/a$, or number-of-nearest neighbors represent the sole desired experimental output to be used for future analytical or computational modeling. It should also be noted that this virtual modeling tool can similarly be applied to down-sample existing 3D representations of structure.
using file formats compatible with DREAM.3D; however, it is of interest here to assume that an experiment has not yet been completed when the virtual modeling tool is applied.

3.3.2 Isotropic and Anisotropic Resolution Down-Sampling

Grain size, grain aspect ratio $b/a$, and the number-of-nearest neighbor grains are analyzed isotropically and anisotropically to determine minimally-sufficient down-sampling schemes required to collect data at least as accurate as the data obtained by the high resolution 3D serial sectioning experiment. For each case shown in Figs. 36-38, “minimally-sufficient” corresponds to an $MBC \leq 0.13$ between reference and down-sampled distributions, less than 2% error in any individual bin relative to the reference phantom, and less than 2% error in the mean value predicted by the virtual data relative to the mean value of 2.8 µm in the reference phantom.

The reference phantom was constructed using an isotropic resolution of 0.25 µm with an average grain size of 2.8 µm, which corresponds to a resolution in VRAD of $2.8/0.25 = 11.2$. Reference volume distributions are shown by the black curves in Figs. 36-38, while other curves depicted in the figures correspond to resolution sampling schemes that meet the accuracy criteria described above. Isotropic down-sampling was performed at resolutions of 0.5, 1.0, and 2.0 µm corresponding to resolutions in VRAD of 5.6, 2.8, and 1.4. This was following a pilot virtual study of isotropic down-sampling performed at all 0.25 µm increments from 0.25 to 2.5 µm, however many of these resolutions correspond to very similar VRAD values and thus produced similar results. Additionally, anisotropic out-of-plane multipliers of 1-10 were studied relative to in-plane resolutions at each isotropic resolution listed above.

This study makes the following assumptions about in- and out-of-plane data collection time to perform calculations related to time improvements: 1) the same beam current would be used for milling, thus twice as much material removal would take roughly twice the time, and 2) the
same experimental settings (other than step size, i.e. resolution) for EBSD data collection would be used, so that an in-plane resolution cut in half amounts to \( \frac{1}{4} \) of the overall number of pixels collected, thus resulting in a \( \frac{3}{4} \) time savings. These are reasonable and conservative assumptions to make, because while the experimentalist could likely improve upon these speeds by optimizing parameters on experimental apparatus, the estimates still provide a basis on which to plan an experiment. Note that this technique to examine experimental potential \textit{a priori} via virtual modeling is not one that any 3D microstructural characterization facility currently employs.

Experimental settings from a smaller 3D serial sectioning experiment collected on the same type of material, using the same experimental equipment, and performed by the same personnel at AFRL is used in this study to conservatively estimate data collection times (see Ref [18]). Although settings from the early 2006 experiment [18] had been tweaked and modified to improve the data collection processes for the 2008 experiment [87], previous experimental settings were recorded in greater detail and AFRL personnel believe that they serve quite well as a baseline for this study.

As a result, it is assumed herein that each 2D section of EBSD data collected with a point-to-point spacing of 0.25 μm takes approximately 25 minutes to collect, where 11 minutes are spent collecting EBSD data, 9 minutes are spent milling material away with the FIB, and 5 minutes are spent aligning the sample while moving between imaging and milling positions [18]. This results in a total experiment time of approximately (25/60) hours * 384 sections = \( \sim \)7 days for the 2008 experiment.

### 3.3.2.1 Grain Size Distribution

For the grain size distribution, the resolution sampling scheme that satisfied all of the accuracy criteria (i.e. MBC \( \leq 0.13 \), bin error \( \leq 2\% \) mean error \( \leq 2\% \)) was 5.6 VRAD in plane
with a 3x anisotropic multiplier out-of-plane, as shown in Figure 36. This corresponds to an in-plane resolution of 0.5 µm and an out-of-plane resolution of 1.5 µm. In order to estimate a time savings that could be realized when collecting grain size distributions, the breakdown of time required to collect each section must be considered. During the experiment, the milling of each section required 5 minutes for movement to and from the milling station in the DB FIB-SEM regardless of the amount of material milled. However, based on down-sampling results, EBSD scan time and the time required for milling do not remain constant.

This particular down-sampling scheme for grain size distributions produces an experiment with the following parameters relative the actual 3D characterization: 64 total sections vs. 384 sections, and a 0.5 µm step size vs. a 0.25 µm step size. Thus, using the conservative estimate of the same beam current, the time it takes to mill 1.5 µm steps away is 9 min \(\times (1.5/0.25) = 54\) minutes and the time it takes for a single EBSD scan is 11 min \(\times (0.25/0.5)^2 = 2.75\) minutes. Thus the total time per section is \(2.75 + 5 + 54 = 61.75\) minutes. With an experiment of only 64 sections, however, this sampling scheme decreases the total experiment time from \(~7\) days to \(~2.75\) days. Even with the extremely conservative FIB milling time estimate, the total time is still less than half of the original experiment.
Figure 36: Grain Size Distribution Obtained from Down-Sampling: Results from the virtual microstructure model for grain size, showing the minimally sufficient anisotropic down-sampling case of 5.6 VRAD (1:1:1/3) required to accurately describe the virtual model’s size distribution.
3.3.2.2 Number-of-Nearest Neighbor Distribution

There were two distinct resolution down-sampling schemes that satisfied the all of the accuracy criteria for the number-of-nearest neighbor distributions. The first used the same resolution in-plane that was collected experimentally (11.2 VRAD), but was able to withstand a 1.4 VRAD resolution out-of-plane (see Figure 37). Since the in-plane resolution is the same as that used during the actual experiment, time savings for this sampling scheme come primarily from the smaller number of sections that must be collected. However, even scaling the milling time to polish off 8x more data at each step (so that each section would require 88 minutes total rather than 25) the experiment time is still cut in less than half (~ 3 days) since only 48 slices must be collected.

The second case that also satisfied the criteria could be argued to be a more intuitive way to collect data more efficiently. In this case, sampling resolution is cut in half in all directions. It was computed previously that at 5.6 VRAD in-plane data collection would require 2.75 minutes. Here, FIB milling would require 18 minutes per section, thus a total sectioning time of $2.75 + 5 + 18 = 25.75$ min. However, the 5.6 VRAD isotropic down-sampling case requires 4x as many sections as the 11.2 VRAD (1:1:1/8) case. As such, to collect the 192 sections would require approximately 3 ½ days. This result highlights the importance of searching many different sampling schemes in order to optimize experiment time based on the output of interest, because in some cases collecting a higher in-plane resolution and letting the out-of-plane resolution grow can be more cost-effective with the same accuracy. For reference, the two phantom down-sampled volumes that satisfy the criteria for the number-of-nearest neighbors are shown in Figure 38.
Figure 37: Number-of-Nearest Neighbor Distributions Obtained from Down-Sampling:
Results from the virtual microstructure model for number-of-nearest neighbors, showing the minimally sufficient isotropic and anisotropic down-sampling schemes required to accurately describe the virtual model’s number-of-nearest neighbor distribution.
Figure 38: Virtual Volumes Providing Accurate Representations for the Number-of-Nearest Neighbor Distributions: Both volumes shown satisfy the accuracy criteria for describing the number-of-nearest neighbor distributions. An isotropically down-sampled 5.6 VRAD volume is shown at the top, and an anisotropically down-sampled 11.2 (1:1:1/8) VRAD volume is shown at the bottom.
3.3.2.3 Aspect Ratio $b/a$ distribution

As shown in Figure 39, there were also two distinct resolution down-sampling schemes that satisfied the accuracy criteria for the distributions of aspect ratio $b/a$. The first was an isotropically down-sampled volume at 5.6 VRAD, resulting in an approximately 3 ½ day experiment by previous result. However, this result is surprising relative to the results obtained in the resolution and resolution with noise + cleanup studies. In each of the previous two sets of work, aspect ratios required approximately 10 VRAD to estimate accurately. However, in this study reference volumes were built at resolutions that were nearly 10 VRAD to begin with (i.e. 11.2 VRAD) rather than 30 VRAD, and additionally the number of grains used for analysis was much greater. This provides insight into potential future directions for this work that might include investigating the effects of performing virtual modeling on reference volumes that are not created at a high enough resolution to begin with, or perhaps an investigation aimed at determining the number of grains that must be present in a phantom grain ensemble microstructure to allow for lower resolution microstructural data than would otherwise be required.

The second resolution that satisfied the aspect ratio $b/a$ accuracy criteria was the 11.2 (1:1:1/3) anisotropic down-sampling case, corresponding to a resolution of 3.7 VRAD out-of-plane. This sampling scheme provides an estimate of 3.8 days overall for the experiment, which is a longer total experiment time than estimated from the 5.6 isotropic down-sampling scheme. Note that this conclusion is the inverse of the one drawn for the number-of-nearest neighbors. In particular, collecting data at a higher resolution throughout the structure is better for determining aspect ratios, whereas collecting at higher in-plane and lower out-of-plane resolution is better for determining neighbors (at least for this instantiation of virtual microstructure).
Figure 39: Aspect Ratio $b/a$ Distributions Obtained from Down-Sampling: Results from the virtual microstructure model for aspect ratio $b/a$, showing the minimally sufficient down-sampling schemes required to accurately describe the virtual model’s $b/a$ distribution.
3.3.3 Additional Considerations

The phantom reference microstructure used in this study was constrained to have nearly equiaxed grains, and the results of the noise + cleanup analysis showed that these types of microstructure are extremely insensitive to the amount of noise in the data. This means that there is a significant amount of time that could potentially be saved by choosing in-plane data collection speeds.

Although it was not applied here, an interesting way to apply noise modeling tools in addition to down-sampling for analysis of experimentally-derived 3D characterization data is to use a look-up table related to EBSD data collection speed at various levels of noise. This can be accomplished by collecting a few EBSD scans on the material to be investigated at different speeds, and computing the number of pixels on each scan with unknown or low-confidence crystallography indices (i.e. noise). Scans collected on a nickel-based superalloy at AFRL were analyzed in this way, and the results are shown in Figure 40, which plots % zero solutions vs data collection speed for a typical nickel-based superalloy. Using this type of information can help the experimentalist decide on appropriate data collection speeds. It can also be incorporated into a virtual modeling study similar to the one presented here that leverages resolution down-sampling as well as noise + cleanup with the data healing algorithm applied to the experimental data. This is just one suggestion to further enhance this virtual modeling framework, so that a more informed optimization of data sampling schemes can be developed prior to an experiment.
**Figure 40: EBSD Analysis % Zero Solutions vs. Data Collection Time per Pixel for a Nickel-Based Superalloy:** This data, based on EBSD scans collected by Dr. Michael Uchic (AFRL), is representative of typical nickel-based superalloys and is used to relate % zero solutions (i.e. % noise) to data collection time per pixel.

This 3D serial sectioning experiment performed on IN100 represents one of only a handful of high resolution, fully 3D serial sectioning experiments that have been completed to-date. It should be noted that the actual IN100 experiment was performed with many goals in mind, the scope of which is greatly over-simplified by this study. The experiment was in-part a demonstration of newly developed state-of-the-art experimental data collection tools and methodologies, and also provided a look at the supporting cleanup, 3D reconstruction, and statistical analysis software capabilities at AFRL. The collection of sets of statistics about different microstructural parameters, while they were desired as outcome from the work, were not viewed as the primary objective. This study is not meant to draw conclusions about inefficiencies related to the 3D characterization experiment. Additionally, in order to collect higher order shape data or precise grain boundary data, which was also of interest to AFRL.
during the experiment, the experimentalist would want to collect at the highest resolution possible (as evidenced by the high resolution shape study discussed earlier).

Further, discretized, stair stepped voxel representations are inherently imperfect depictions of the material microstructures found in nature. Thus, when any-and-all information that can be gathered is of interest, characterization experiments which ultimately yield voxelized reconstructions of 3D microstructure should be performed so as to achieve the highest resolution possible. Additional research is required related to the modeling of 3D microstructures as they are in nature (i.e., with smooth boundaries); however, such representations are not addressed in detail in this dissertation and are left for future work.

3.3.4 Conclusions

This case study leveraged the grain size distribution obtained from an actual high resolution 3D serial sectioning experiment to build a virtual phantom microstructure, which was then down-sampled both isotropically and anisotropically to determine minimally sufficient sampling schemes for the following distributions of microstructural statistics: grain size in ESD, aspect ratio b/a, and number-of-nearest neighbors. Results obtained from this case study decisively support previous conclusions that experiments should be carried out with specific goals in mind, and additionally that anisotropic down-sampling can be a very powerful tool when it comes to optimizing data sampling schemes for specific microstructural statistic(s) of interests. Using the set of defined accuracy thresholds (MBC ≤ 0.13, bin error ≤ 2%, mean value ≤ 2%), the following conclusions were drawn related to total experimental time required for down-sampled schemes:

- **Grain size**: a fully 3D experiment seeking to capture the size distribution only could be executed on the same sized volume in ~ 2.75 days using an anisotropic sampling scheme of
5.6 VRAD (1:1:1/3), corresponding to a 0.5 µm step size in-plane, a 1.5 µm step size out-of-plane, and a time savings of ~4.25 days relative to the actual experiment conducted.

- **Number-of-Nearest Neighbors**: a fully 3D experiment seeking to capture the number-of-nearest neighbor distribution only could be executed on the same sized volume in ~3 days using an anisotropic sampling scheme of 11.2 VRAD (1:1:1/8), corresponding to a 0.25 µm step size in-plane, a 2.0 µm step size out-of-plane, and a time savings of ~4 days relative to the actual experiment conducted.

- **Grain Aspect Ratio b/a**: a fully 3D experiment seeking to capture the aspect ratio *b/a* distribution only could be executed on the same sized volume in ~3.5 days using an isotropic sampling scheme of 5.6, corresponding to a 0.5 µm step size in-plane, a 0.5 µm step size out-of-plane, and a time savings of ~3.5 days relative to the actual experiment conducted.
4 Applications to Additive Manufacturing of Ti-6Al-4V

This chapter of the dissertation focuses on characterization of AM components built using Penn State University CIMP-3D’s LENS™ system.

4.1 Statistical Analysis of Widmanstätten α-laths in additive manufactured Ti-6Al-4V

In contrast to the previous research described in section 3, this work seeks to provide a methodology for the quantitative analysis of AM Ti-6Al-4V sub-grain level microstructures, using two thin-walled L-shaped components built via the LENS® process, and leveraging techniques similar to those utilized for quantifying the average size of Widmanstätten (basketweave) α-laths in forged billets of Ti-6Al-4V [85]. The Materials Image Processing and Automated Reconstruction (MIPAR™) [105] software was used to optimize recipes for conversion of back-scattered electron (BSE) and electron back-scatter diffraction (EBSD) images into binary data suitable for mean linear intercept (MLI) [106] analysis and thus α-lath thickness calculations. In addition, a quantitative comparison technique for α-laths observed in different regions of the two components is presented, where discrete PDFs of MLIs are compared for geometric similarity via a Modified version of the Bhattacharyya Coefficient (MBC) [91]. Lastly, quantitative α-lath data is analyzed for trends relative to the real-time thermal data collected during the 3D directed energy deposition experiment used to create the two thin walls. Kriczky et al. published an overview of the experiment [107] and this work compares thermal gradients and melt-pool areas to α-lath thicknesses in the resulting solidification microstructure for both thin-walled L-shaped components.
4.1.1 Additive Manufacturing and Thermal Imaging of Ti-6Al-4V Components

The LENS™ system used to build components investigated in this work represents a collaborative effort between Penn State University’s Center for Innovative Metal Processing through Direct Digital Deposition (CIMP-3D), Stratonics, and Optomec. The integrated multisensor/process model-based control system leverages an Optomec MR-7 LENS™ system, outfitted with a Stratonics ThermaViz® thermal imaging camera. Using the integrated system, CIMP-3D manufactured the two relatively-large (~50 mm tall), L-shaped, thin-wall samples of AM Ti-6Al-4V, shown in Figure 41. Each sample is composed of two legs: the first leg is constructed from a single bead, while the second has three beads per layer. The key difference between these samples is the dwell time used between added material layers. One sample was built with zero dwell time between layers, while the other sample had a dwell time of four seconds. These two samples are shown in Figure 41A and Figure 41B, respectively.

![Figure 41: AM Ti-6Al-4V Components Constructed Using the LENS™ Process](image)

The complete thermal history of each component was captured in real time during the experiment. Detailed information regarding the step-by-step procedures to transform point-by-point thermal data into thermal gradients, melt pool areas and the like is available in [107]. Here,
only the authors’ designations for the lower 10%, upper 90%, and center 33% are of interest for both the 1- and 3-bead legs of each sample; these designations are illustrated using the zero second dwell specimen shown in Figure 42. For this work, α-lath data was collected for both legs of each sample (0s dwell, 4s dwell) within the center 33% region and along the entire height of the thin walls (z-direction as pictured below).

![Figure 42: Thermal Gradient Reconstruction Showing Spatial Designations for AM Ti-6Al-4V Components](image)

**Figure 42: Thermal Gradient Reconstruction Showing Spatial Designations for AM Ti-6Al-4V Components:** Isometric 3D reconstruction of calculated thermal gradients for the zero second dwell sample [107].

### 4.1.2 Microstructural Characterization of Ti-6Al-4V α-laths

Depending on geometry and AM process parameters, cooling rates in AM processes can vary significantly across a single part, an effect most prevalent on the edges of parts and between added material layers. As a result, multiple α structures are typically observed in a single β grain. This is exemplified in Figure 43, which shows two BSE images depicting colony- and Widmanstätten-α in the same field of view. While microstructural variation is characteristic of the LENS™ samples under investigation, such inconsistent microstructure does not lend itself to
rapid qualification methods for AM nor does it make quantifying microstructural features using existing toolsets a straightforward task.

![Image of colonies and basketweave microstructures](image)

**Figure 43: Colony α and Basketweave α Microstructures Observed in LENS™ Ti-6Al-4V**: BSE image showing colony α microstructures present amidst a majority of basketweave α, as observed in the 1-bead leg of the sample with zero dwell time between layers.

Inspection of both samples and their solidification microstructures resulted in a designation of five zones along the height of each sample, as pictured in Figure 44 below. Additionally, this inspection yielded the best way to image α-laths of various sizes. For all sizes, ultra-fine polishing (down to 0.05 μm) and thorough cleaning was required; however, the mode of data collection changed for different lath sizes. For thicker laths like those pictured in Figure 43, back-scattered electron (BSE) imaging at a magnification of 2000x (working distance 5 mm, accelerating voltage 20 kV, spot size 5) proved to be a repeatable methodology. However, the ability to distinguish smaller laths required crystallographic analysis via electron back-scatter diffraction (EBSD) (step size 0.1 μm).
Zones 2, 3, and 4, those not located at the extreme ends of the sample (top, bottom), appeared to have steady-state microstructures. More specifically, the central zones along the height of the thin walls appeared to have qualitatively similar lath sizes, which can be seen in Figure 45. However, zones 1 and 5 were very difficult to image using BSE techniques, and produced BSE images unsuitable for image processing. As a result, EBSD data was required, as shown in Figure 46. The laths requiring EBSD appear smaller based on the more acicular nature of the observed α-microstructure.

**Figure 44: Zones 1-5 Used for α-lath Width Analysis:** Spatial locations along the sample height for zone designations used in α-lath width analysis.

**Figure 45: Steady State Zones of AM Ti-6Al-4V Components:** BSE images of zones 2, 3, and 4 showing easily quantifiable basketweave α structures; taken from the 1-bead leg of the sample with zero dwell time between layers.
4.1.3 Image Processing via MIPAR™

Lath thicknesses and distributions are determined using the image analysis software MIPAR™ (Materials Image Processing and Automated Reconstruction) [105], developed by Sosa, et al. at The Ohio State University. For each imaging technique, a series of image processing steps was completed within MIPAR™ to convert the data into binary images for quantification.

For BSE imaging in steady state zones, a 6-step series of image filtering techniques was implemented to convert grayscale images to binary, as shown in Figure 47. In particular, the following series of filters was used to segment features: Wiener filter, Fast Fourier Transform filter, adaptive threshold, dilation, erosion, and feature rejection. Although filter choices were based on a combination of experience and trial-and-error, the optimum parameters for each filter
were objectively determined based on segmentation-to-image mutual information for each image-processing algorithm used in the sequence (see Ref [105] for details).

![Zone 2 Original](image1.png) ![Zone 2 Processed](image2.png)

**Figure 47: BSE Image Filtering for Steady State Zones**: Example of a BSE image converted to binary for α-lath width analysis using MIPAR™.

Colored EBSD images were converted to grayscale from their default RGB format by adding (R+G+B) content for each pixel. Similarly to the images in the steady state zones, a unique multi-step image-processing sequence was developed. Specifically, a median filter and edge find sequence was applied for three rounds, followed by a final erosion and feature rejection, producing binary data as shown in Figure 48. Optimum filter parameters were again determined based on segmentation-to-image mutual information.
Figure 48: **EBSD Image Filtering for Top and Bottom Zones:** Example of an EBSD image converted to binary for α-lath width analysis using MIPAR™.

### 4.1.4 Determining α-lath Thicknesses and Quantifying Uncertainty in Microstructure

Having obtained binary images from the microstructural data, Widmanstätten α-lath thicknesses were calculated via the mean inverse of the linear intercept [106], a procedure also performed within MIPAR™. This method superimposes a grid of parallel lines on the image, subsequently rotates the grid by 10° increments to collect measurements in multiple directions, and records linear intercepts (λ) and inverse intercepts (1/λ) computed for each lath (see [85, 106]) so that the true 3D thickness of α-laths can be estimated as

\[
\text{thickness} = \frac{1}{1.5(1/\lambda)_{\text{mean}}}. \tag{1}
\]

The application of this method approximates laths as layered structures and estimates the true thickness of a series of “infinite” plates of finite thicknesses (i.e., finite only in 1 dimension). Further, it works best for more randomly oriented (i.e., 100% Widmanstätten) structures; thus the
α colonies visible in Figure 43 provide an additional source of uncertainty in these measurements.

Average lath thicknesses, computed for each image and averaged based on the number of images taken for each section, are shown in Table 3 below. The confidence intervals were computed using Student’s t-distribution on (n-1) degrees of freedom as a benchmark, with a significance level of 0.05 to produce two-sided 95% confidence bounds. In all cases, the calculated α-lath thicknesses are larger in the steady-state region than in the top and bottom regions; further, the bottom regions produce a smaller average thickness than the top regions.
Table 3: Average α-Lath Thickness Comparison: Comparison of average α-lath thicknesses for all zones across each leg of both samples, along with 95% confidence intervals based on the number of images collected.

<table>
<thead>
<tr>
<th>Zone</th>
<th>α-Lath Width (microns)</th>
<th>95% Confidence Interval</th>
<th># Images</th>
</tr>
</thead>
<tbody>
<tr>
<td>0s Dwell</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Bead Leg</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0s Dwell</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Bead Leg</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0s Dwell</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3-Bead Leg</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4s Dwell</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1-Bead Leg</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4s Dwell</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3-Bead Leg</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

In addition to quantifying mean α-lath thickness, consideration is given to the variability between entire distributions of inverse intercept length, which are taken to represent the lath microstructure observed in each binary image. Inverse intercept distributions are constructed using all inverse intercepts calculated for the set of images collected in each zone (see Table 3 above for the number of images collected in each set). Discrete distributions are binned using a 30-bin scheme between 0 and 5 that was based on the observed data, which typically ranged between 0 and 5. The larger-valued outliers (corresponding to extremely small alpha lath
widths) inherently result from this analysis due to complicated three-dimensional basketweave microstructure being observed in only two dimensions, although imperfect binary representations likely play a role as well. Once constructed, the distributions within the zones of each sample leg are compared for geometric similarity the Modified Bhattacharyya Coefficient, as has been presented in previous studies. For this work, distributions are compared within sample-leg steady state zones (i.e. zone 2 to 3, zone 2 to 4, and zone 3 to 4) as well as between sample-leg top and bottom zones (i.e. zone 1 to 5). Comparing BSE and EBSD results separately ensures that no aliasing effects occur based solely on variations in data collection procedures and image processing techniques.

For each leg, MBC calculations between zones are shown in Table 4. MBC values above the threshold of 0.13 are highlighted in red, and in each of these cases a clear qualitative difference in microstructure can be observed in BSE or EBSD images. The highest MBC in Table 4, corresponding to a comparison of zone 1 vs. 5 in the 1-bead leg of the 4s dwell sample, was determined to be attributable to poor data collection quality of the EBSD scan used for analysis of zone 1.

Table 4: MBC Comparisons of Inverse Intercept Distributions: MBC calculations for each sample leg, comparing top and bottom regions as well as steady-state zones.

<table>
<thead>
<tr>
<th></th>
<th>1-Bead Leg, 0s Dwell</th>
<th>1-Bead Leg, 4s Dwell</th>
<th>3-Bead Leg, 0s Dwell</th>
<th>3-Bead Leg, 4s Dwell</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zone 1 - Zone 5</td>
<td>0.045</td>
<td>0.328</td>
<td>0.097</td>
<td>0.092</td>
</tr>
<tr>
<td>Zone 2 - Zone 3</td>
<td><strong>0.195</strong></td>
<td>0.072</td>
<td>0.045</td>
<td>0.041</td>
</tr>
<tr>
<td>Zone 2 - Zone 4</td>
<td><strong>0.189</strong></td>
<td>0.092</td>
<td>0.043</td>
<td>0.074</td>
</tr>
<tr>
<td>Zone 3 - Zone 4</td>
<td>0.029</td>
<td><strong>0.152</strong></td>
<td>0.075</td>
<td>0.103</td>
</tr>
</tbody>
</table>

Distributions of inverse intercept length can also be analyzed graphically for each leg. The 1-bead leg zero second dwell case is shown as one example of the analyzed data in Figs. 49-50. Zones 3 and 4 (Figure 49), as well as zones 1 and 5 (Figure 50) appear to have significantly similarities, respectively. These conclusions are supported by the data in Table 4 above, where
the comparison of zones 3 and 4 produces an MBC = 0.029 and zone 1 to zone 5 produces an MBC = 0.045, both providing values well below the threshold of MBC≤0.13. Notably, these zones had extremely similar lath thicknesses, 0.69 vs. 0.67 µm for zone 3 and 4 and 0.55 vs. 0.59 µm for zone 1 and 5. Further parallels can be drawn with the lath thickness data in Table 3. In particular, the distributions with higher fractions at smaller inverse intercepts are those with larger lath thicknesses.

Interestingly, the inverse intercept lengths observed in particular bins of zones 1 and 5 for each leg produced distributions that were not smooth in certain ranges (see Figure 50). This occurred for all zone 1 and 5 data, and is perhaps due to the image processing routines used for analysis; however this is a bias left for investigation in future work.

![Inverse Intercept Distributions, 0s Dwell, 1 Bead Leg, Zones 2-4](image)

**Figure 49:** Inverse Intercept Distributions, Zones 2-4, 0s Dwell, 1-Bead Leg: $1/\lambda$ distributions plotted between 0 and 4.
4.1.5 Comparison of Widmanstätten $\alpha$-lath Widths to Thermal Imaging Data

Lastly, a comparison of the quantitative $\alpha$-lath thicknesses to the thermal imaging data provided in Ref [107] is in order. For these comparisons, zones 2-5 are grouped to correspond to the upper 90% region of the thermal imaging data, leaving zone 1 to represent the lower 10%.

All microstructural analysis was completed within in the center 33% of each leg of the samples (see Figure 42 for reference). Thus, Region 1 is designated as the lower 10% and center 33% area of the samples (i.e., zone 1), while Region 2 is the upper 90% and center 33% of the samples (i.e., zones 2-5).

Table 5 shows a comparison across build regions, including averages for thermal gradients, melt pool areas and $\alpha$-lath thicknesses. Table 6 shows a comparison between dwell times, while Table 7 compares each of these metrics using the number of beads per layer.
Table 5: **Comparison of Thermal Gradient, Melt Pool Area, and α-Lath Thickness Across Build Regions**: This table compares the lath thicknesses in zone 1 to the lower 10% thermal data and the average of lath thicknesses observed in zones 2-5 to the upper 90% thermal data.

<table>
<thead>
<tr>
<th></th>
<th>Region 1</th>
<th>Region 2</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>0 sec</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermal Gradient [K/cm]</td>
<td>1 Bead 6047</td>
<td>3 Bead 6177</td>
<td>Higher for Region 1</td>
</tr>
<tr>
<td>Melt Pool Area [mm²]</td>
<td>1 Bead 2.53</td>
<td>3 Bead 1.27</td>
<td>Larger for Region 2</td>
</tr>
<tr>
<td>α-lath Width [µm]</td>
<td>1 Bead 0.56</td>
<td>3 Bead 0.54</td>
<td>Thicker for Region 2</td>
</tr>
<tr>
<td><strong>4 sec</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermal Gradient [K/cm]</td>
<td>1 Bead 7061</td>
<td>3 Bead 6925</td>
<td>Higher for Region 1</td>
</tr>
<tr>
<td>Melt Pool Area [mm²]</td>
<td>1 Bead 2.53</td>
<td>3 Bead 1.10</td>
<td>Larger for Region 2</td>
</tr>
<tr>
<td>α-lath Width [µm]</td>
<td>1 Bead 0.33</td>
<td>3 Bead 0.41</td>
<td>Thicker for Region 2</td>
</tr>
</tbody>
</table>

Table 6: **Comparison of Thermal Gradient, Melt Pool Area, and α-Lath Thickness Across Dwell Times**: This table compares the averages of lath thicknesses observed in zones 1-5 for both 1- and 3-bead legs to average thermal data for each dwell time used in the samples.

<table>
<thead>
<tr>
<th></th>
<th>0 sec</th>
<th>4 sec</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Gradient [K/cm]</td>
<td>5823</td>
<td>6795</td>
<td>Thermal gradient increases with pause length</td>
</tr>
<tr>
<td>Melt Pool Area [mm²]</td>
<td>2.39</td>
<td>2.19</td>
<td>Melt pool area decreases with pause length</td>
</tr>
<tr>
<td>α-lath Width [µm]</td>
<td>0.71</td>
<td>0.51</td>
<td>α-lath thickness decreases with pause length</td>
</tr>
</tbody>
</table>

Table 7: **Comparison of Thermal Gradient, Melt Pool Area, and α-Lath Thickness Across Number of Beads**: This table compares the averages of lath thicknesses observed in zones 1-5 for both 0s and 4s dwell times to average thermal data for 1- and 3-bead legs of the samples.

<table>
<thead>
<tr>
<th></th>
<th>1 Bead</th>
<th>3 Bead</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Gradient [K/cm]</td>
<td>5951</td>
<td>6667</td>
<td>Thermal gradient increases with bead count</td>
</tr>
<tr>
<td>Melt Pool Area [mm²]</td>
<td>2.99</td>
<td>1.59</td>
<td>Melt pool area decreases with bead count</td>
</tr>
<tr>
<td>α-lath Width [µm]</td>
<td>0.50</td>
<td>0.72</td>
<td>α-lath thickness increases with bead count</td>
</tr>
</tbody>
</table>
Based on Table 5 above, α-lath thicknesses are generally larger when thermal gradients are lower, and α-lath thicknesses are also larger for increased melt pool areas. Consideration of the dimensionless thermal gradient and dimensionless cooling rate used by Bontha et al. [64] reveals that the thermal gradient in the horizontal (layer) direction scales with cooling rate. Recent unpublished theoretical work has also shown that the trailing edge thermal gradient at the solidus-liquidus interface occurs strictly in the horizontal (layer) direction at the top surface of the melt pool. Thus, this particular thermal gradient data, which was collected from a downward-facing thermal imaging camera and computed at the top of the melt pool, can be interpreted in the same way as a cooling rate for trend identification purposes since the velocity of the beam used in this experiment was constant. Therefore, α-lath thicknesses appear to be larger when cooling rates are lower and smaller when cooling rates are higher, a very intuitive result.

In each of the tables above, thermal gradient increases as the melt pool area decreases. Noting Gockel and Beuth showed that lines of constant melt pool area correspond to lines of constant cooling rate, and hence β grain size for wire-feed AM Ti-6Al-4V [49,108], it appears that larger melt pool areas correspond not only to enlarged β grain sizes but also to thicker α-laths within those grains. Table 6 and Table 7 reveal that α-lath thicknesses increase with the number of beads used per layer and decrease with pause length. Both an increase in number of beads and a decrease in pause length correspond to an increase in the available heat (and hence lower thermal gradients and cooling rates) in the system, providing a mechanism for continued α-lath growth.

It should be noted, however, that though these trends exist it can also be observed from inspection of the data that neither thermal gradient or melt pool area are sufficient to predict lath thicknesses. For example, the measured thermal gradients from the three-bead leg of the zero
second dwell sample increased from region 1 to region 2; a result that is inconsistent with the other three sample legs. Another anomalous comparison can be observed from the three-bead leg of the four second dwell sample, where thermal gradients of 6925 and 6893 K/cm were observed in region 1 and region 2, respectively, while alpha lath widths were computed to be 0.41 and 0.67 μm. This suggests that more work is necessary to properly correlate in situ thermal measurements to α microstructures in AM processes, perhaps by considering thermal history below the β transus when lath growth is expected to be active.

4.1.6 Conclusions

This study presents a general framework for quantitative analysis of sub-beta-grain level Widmanstätten α microstructures in AM Ti-6Al-4V. The methodology includes techniques for analyzing α-laths of various sizes, as well as an uncertainty quantification tool for comparing inverse linear intercept distributions in various regions of AM components. The quantitative results agree with qualitative visual analysis, and favorably correspond to published theoretical and experimental results. In confirming intuitive trends between thermal gradient, melt pool size, and solidification microstructure, this work provides a quantitative means for verifying intuition about α-level structure in AM Ti-6Al-4V. However, although trends were identified, it was also observed that the neither thermal gradient or melt pool size alone is sufficient to predict α-lath thicknesses, and thus additional research is required to further understand the complex thermal cycles present in AM-fabricated structures.
4.2 Estimating 3D Serial Sectioning Time for AM Ti-6Al-4V Based on Experimentally-Derived β Grain Size Data

This study is performed on the sample analyzed in the previous section, in particular on the 1-bead leg of the four second dwell sample. This study provides a look into the feasibility of collecting 3D microstructural data on β grains in AM Ti-6Al-4V, and also provides insights into the difficulties associated with collecting a significant amount of microstructural data from a relatively large component. An estimate of total serial sectioning time required for the 1-bead leg is developed, based a set of 2D EBSD batch scans that span the height of the leg (2” = 50,800 µm).

Following EBSD data collection, a 2D reconstruction of β grains based on EBSD α-lath data is obtained through use of state-of-the-art software analysis tools developed specifically for Ti-6Al-4V. Then, once a β grain size distribution is determined, virtual modeling and subsequent down-sampling is performed. A methodology for estimating both EBSD data collection time and serial sectioning time is presented, and calculations to estimate total serial sectioning time for accurate 3D representation of grain sizes are made based on sample geometry.

4.2.1 Experimental EBSD Data Collection and β Grain Reconstruction

The first step in the process of determining β grain sizes was to determine the resolution at which to collect EBSD data. A section of each set of data collected is compared visually in Figure 51. The first set of automated EBSD data collection was performed using a step size of 5 µm. At this resolution, one face of the sample that included the entire height (corresponding to an area of (50.8+3*1.24e+3) µm² = 62,992,000 µm²) could be collected in a total time of approximately 8 hours. However, this data did not appear to be of high enough quality for the analysis. The next set of data was performed at half the resolution, corresponding to a 2.5 µm
step size, and at this resolution it took approximately 18 hours to collect data for roughly half of the sample height. A third set of data was collected, this time using a 1.0 μm step size. Although only about 1/20 of the sample could be collected in 16 hours, this scan produced the highest quality of data of any resolution investigated. For reference, these scans were taken at the very bottom of the 1 bead leg, as depicted in Figure 52.

Figure 51: EBSD Batch Scans of AM Ti-6Al-4V at Various Resolutions: The series of batch scans from left to right used for this study were collected with 5 μm, 2.5 μm, and 1.0 μm step sizes, respectively.
**Figure 52: Sample Geometry of LENS™ Ti-6Al-4V Samples:** Sample geometry of the LENS™ Ti-6Al-4V samples investigated, including dimensions.

Using the 1.0 µm step size EBSD batch scan, a 2D reconstruction of the underlying β grain orientations and β grain boundaries was developed based on the Widmanstätten α-lath orientations measured from the crystallographic analysis. This was executed using a version of the commercially-available software MRL-TiBor (Materials Resources LLC, Dayton, OH). The MRL Ti-Bor software uses a method similar to that developed by Humbert *et al.* [109,110] and Glavicic *et al.* [111,112] to extract β grain information based on measured α-lath variants that can be observed via EBSD analysis.

Based on the nature of the β grains observed in the AM Ti-6Al-4V (namely that they were extremely columnar with widths increasing with vertical position in the sample) only a subset of β grains more suitable to perform virtual modeling with were analyzed. This subset is depicted visually in Figure 53A. Following the selection of an appropriate subset, TiBor analysis was
used to determine β grain boundaries from the α-lath data as shown in Figure 53B. It should be noted that the TiBor analysis to go from part A to part B in Figure 53 took approximately 30 minutes. Performing an additional analysis on slightly larger section, but still much less than half of the sample in the field of view on the left below, took close to 3 hours.

Following determination of β grain boundaries, a cleanup routine for the data was employed and executed via the following steps: 1) a grain dilation using a minimum grain size of 100 pixels, followed by 2) a neighbor phase correlation filter with a maximum allowable misorientation of 5° between grains. This was based on trial-and-error, as well as suggestions from AFRL personnel. The data following cleanup is shown in Figure 53C, which depicts a mix of columnar and equiaxed grains. Modeling of this type of structure can be accomplished in DREAM.3D using a combination of rolled and equiaxed distribution inputs to create a multi-phase microstructure. Once a microstructure is generated, it can be analyzed using virtual down-sampling as performed previously on more equiaxed structures.
Figure 53: Determination of Grain Size Distribution for Input to Virtual Model: Grain size distribution developed using a subset of the 1.0 µm step size scan: A) The subset of grains chosen for analysis, B) Determination of β grain boundaries from α lath orientations, C) β grains following cleanup, D) Further subset of roughly equiaxed β grains ultimately used for virtual modeling.

Rather than building a virtual model of these grains, it was decided to use an even smaller subset of the β grain data for virtual modeling, as shown in Figure 53D. This was done for one key reason, and it is not motivated by the difficulty of modeling the more columnar + equiaxed structure in a virtual environment. Rather, the motivation for choosing the more equiaxed, fine-grained, relatively well-behaved structure to model is that in AM research the development of this type of microstructure is seen as a top priority. Even though it is not yet a microstructure that can be produced consistently in AM Ti-6Al-4V components, there are significant research efforts to this end.
Therefore, the estimate of serial sectioning data collection times calculated in this study provides a “what if” analysis of experimental requirements for the ideal output from AM Ti-6Al-4V component builds. In so doing, it is aimed towards providing insight into microstructures to which the significant time and effort required for 3D microstructural characterization might more feasibly be applied. Additionally, this study is meant to investigate that if a component of this size is built, how long it would take to get an accurate 3D representation of the entire grain structure throughout the component. Of course, this would constitute a serial sectioning experiment that is orders of magnitude larger than has ever been attempted previously. However, with virtual modeling tools and very limited experimental data collected in 2D, studies like this one can provide realistic “ballpark” numbers for 3D microstructural characterization experiments that will likely be pursued in the not-to-distant future as experimental tools find more optimized ways of collecting data.

### 4.2.2 Phantom Generation

A grain size distribution was developed by first using Orientation Imaging Microscopy (OIM™) Analysis software to compute grain sizes in 2D. The set of grains used to construct the distribution is the 31-grain set shown in Figure 54. The corresponding 2D diameter of each grain computed from OIM™ Analysis was modified via a stereological correction factor of $4/\pi$ [2] to convert these 2D diameters to 3D equivalent sphere diameters that could be used as input to DREAM.3D. The resulting distribution is shown as a histogram along with the associated lognormal fit and 95% confidence bounds in Figure 55. The mean values for the fit were used as input to create the phantom reference volume.
Figure 54: Set of Equiaxed $\beta$ Grains Used for Virtual Modeling: The set of $\beta$ grains used to construct a lognormal grain size distribution for input to the virtual model.

Figure 55: Grain Size Distribution Used as Input to Virtual Model: Histogram and corresponding lognormal fit created from 2D experimental data and used as input to DREAM.3D.

Using the distribution generated from the 2D grain sizes, a virtual phantom reference volume was created using a resolution of 1.0 microns in all directions. This was chosen based on the experimental EBSD data that was also collected using a 1.0 $\mu$m step size. The distribution

```
Lognormal Distribution For Reference Volume

LN(mu,sigma)
mu = 3.3815
sigma = 0.520616
Confidence Intervals(mu,sigma)
mu = +/- 0.190963
sigma = +/- 0.175277
```

```
Histogram
Lognormal Fit
```
shown in Figure 55 has an average grain size of 26.45 μm, thus the associated resolution of the phantom reference volume is 26.45 VRAD. The dimensions of the phantom reference volume were selected to be 508 x 508 x 1240 μm³, and were chosen based firstly on the dimensions of the 1-bead leg of the thin wall component as shown in Figure 56, and secondly by computational power limitations. The single reference volume structure file is > 6.5 GB, and currently the virtual structures that can be built currently require at least the amount of RAM as the size of the microstructure data file being created.

**Figure 56: Dimensions of Virtual Model Chosen Based on Thin-Wall LENS™ Sample Dimensions:** The dimensions of the phantom reference volume were selected to be 508 x 508 x 1240 μm³ based on the dimensions of the 1-bead leg in the LENS™ samples.

Based on the dimensions shown in Figure 56, one hundred reference volumes stacked upon one another are equal to the total height of 2” (50.8 mm) height, and 50 reference volumes aligned along the base of the sample are equal to the total length of 1” (25.4 mm). Therefore, the total volume of the 1-bead leg is the same as 5000 of the reference volumes used in this study.
These comparisons back to the sample geometry are required for estimating total serial sectioning times.

A comparison of a 2D section the same size as the experimentally-collected data is shown in Figure 57. The experimental and virtual data can be seen as very similar to one another, having approximately the same number of grains in the field of view. It is also clear from inspection of grain boundaries that the reference volume was constructed at the same resolution used to collect the EBSD data.

Figure 57: Comparison of 2D Experimentally-Collected Section with a 2D Portion of the Virtual Phantom Reference Microstructure: The experimentally-derived microstructure compared to the virtual model, showing a qualitatively representative result.
4.2.3 Grain Size Distribution Similarity Analysis

The fundamental assumption in these virtual modeling studies is that the grain size PDF generated from experimental data is representative of the 3D microstructure to be investigated. In this case, rather than providing a representative sample of the chaotic columnar $\beta$ grain distribution, a representative distribution of a microstructure more desirable to the AM community is used as a reference.

Following down-sampling and analysis of the grain size distribution, three resolution sampling schemes were able to satisfy all of the accuracy criteria that has previously been discussed (i.e. MBC $\leq$ 0.13, bin error $\leq$ 2% mean error $\leq$ 2%). The three sampling schemes that produced an appropriately representative size distribution are shown in Figure 58. These can be seen to be the 3.31 VRAD isotropic down-sampling case, the 4.41 (1:1:1/3) VRAD anisotropic down-sampling, and the 5.29 (1:1:1/4) VRAD anisotropic scheme. These correspond, respectively, to in- and out-of-plane resolutions of 8 $\mu$m all around, 6 $\mu$m in-plane with 18 $\mu$m out-of-plane, and 5 $\mu$m in-plane with 20 $\mu$m out-of-plane. It should also be noted that anisotropic sampling was oriented along the length of the sample rather than the height, so that the 2D EBSD data collected along the height could be used to provide estimates data collection time per slice. In order to compare serial sectioning times for each sampling scheme, a breakdown of time required to collect each data slice is required. This breakdown is considered in the following section.
Figure 58: Grain Size Distributions from Virtual Down-Sampling of AM Ti-6Al-4V β Grain Microstructure: Results from the virtual microstructure model for grain size, showing the minimally sufficient down-sampling cases of 3.31 VRAD isotropic down-sampling, 4.41 (1:1:1/3) VRAD anisotropic down-sampling, and 5.26 (1:1:1/4) VRAD anisotropic down-sampling required to accurately describe the virtual model’s size distribution.
4.2.4 Estimating Serial Sectioning Time

In the actual experiment, only the time required for EBSD data collection at 5, 2.5, and 1.0 µm step sizes was investigated. Correspondingly, these scan times resulted in approximate full-height scan times of 8 hours, 36 hours, and 311 hours, respectively. This data is shown along with a line of best fit that can be used to predict the time required per section based on EBSD scan step size (i.e. in-plane resolution) for resolutions that were not investigated experimentally. Note that the coefficient of determination ($R^2$) is also shown in Figure 59, showing that the power fit provided an excellent match for the data being analyzed. Additionally, the power fit was chosen because it appeared to predict scan times done at lower resolutions (higher step sizes) much better than exponential or higher-order polynomial fits.

![EBSD Scan Time Per Section, LENS Ti-6Al-4V Sample](Image)

**Figure 59: EBSD Scan Time per Section vs. EBSD Scan Step Size**: Relationship between resolution and time in hours for EBSD scans of AM Ti-6Al-4V, used for prediction of total serial sectioning time.
Similarly to the study performed on IN100, this study computes total serial sectioning times by computing the time it takes to collect a single slice of data. Correspondingly, the time to collect a single data slice is made up of the time required for the EBSD scan plus the time needed for material removal and sample alignment. The time required for material removal and alignment is set to 30 ± 15 minutes as a conservative estimate. This is approximately the amount of time it would require the LEROY system at AFRL [6] to polish any reasonable amount of material off (0 to ~100 μm), prep the surface for data collection, and transport the sample to the SEM while ensuring its consistent alignment from the previous data collection step.

For reference, the down-sampling sets that resulted in the best matches for grain size distributions are given in VRAD with their corresponding resolution in μm below:

- 3.31 VRAD isotropic sampling: 8 μm in-plane, 8 μm out-of-plane
- 4.41 (1:1:1/3) VRAD anisotropic down-sampling: 6 μm in-plane, 18 μm out-of-plane
- 5.29 (1:1:1/4) VRAD anisotropic down-sampling: 5 μm in-plane, 20 μm out-of-plane

Taking the first case of 3.31 VRAD isotropic down-sampling, the time per EBSD scan is computed from the equation in Figure 59, resulting in 2.67 hour scan time per 2D section. Using 6.0 μm polishing steps, it would require 3,175 sections to get through the entire 1” length of the 1-bead leg. Assuming a material removal rate of 30±15 minutes results in a total experiment time of between 1.06 and 1.24 years.

The second case provides a 6 μm in-plane resolution, corresponding to 5.14 hours per scan, and an 18 μm polishing step, requiring a total of ~1,411 sections through the sample’s length. Using this data collection scheme, the experiment time is computed to be between 0.87 and 0.95 years, corresponding to a minimum data collection time of roughly 10 ½ months. Although this
results in experimental data collection time that is still absolutely infeasible, it can be seen to be a
marked improvement to the time required from the previous sampling scheme.

The last case, which uses a 5 µm in-plane resolution (8 hours/EBSD scan as collected), only
requires 1,270 sections and results in total data collection time very similar to the first case, with
a maximum of 1.27 years. It is also interesting to note, that if one were to collect at the
resolution used to build the reference volume (1.0 µm in all directions), the same experiment
would require between 902 ½ and 904 years! However, this is assuming that the current state of
computational power could analyze and process the set of 1.0 µm scans, which seems unlikely.

It is much more likely that if a 3D characterization experiment were to be planned, it would
be planned for the collection of a representative volume of microstructure. Assuming that the
phantom reference volume could serve as a representative microstructure that captures all of the
grain size statistic(s) present throughout the 1-bead leg of the thin wall sample, these estimates of
total time required for 3D characterization decrease drastically.

In order to perform these calculations, EBSD scan times calculated as before using the
equation in Figure 59, however here this result can be divided by the number of phantom
reference volumes required to represent the entire height of the sample (i.e., 100 volumes). Thus, the time per EBSD scan decreases by two orders of magnitude. Similarly, if only the
phantom reference volume is considered, the number of sections required to get through the
length of the sample decreases by a factor of 50. Using an analogous approach to computing
total experiment times as above, the 3.31 VRAD, 4.41 VRAD anisotropic, 5.29 VRAD
anisotropic, and 26.45 VRAD (1.0 µm) resolutions would require maximum total experiment
times of 2 days, 22.6 hours, 21 hours, and ~ 82 days, respectively, to collect a volume the size of
the phantom microstructure. Thus, collecting only representative volumes of microstructure
provides a feasible alternative to collecting entire sets of grains contained within component-
level geometries. The final results for each down-sampling scheme investigated are summarized in below.

**Table 8: Comparison of Total Serial Sectioning Time between Entire 1-Bead Leg and a Representative Volume:** This table compares the total serial sectioning times computed for 3D characterization of equiaxed β grains at minimally-sufficient sampling schemes as well as extremely high resolution sampling for both the 1-bead leg in its entirety and a representative volume of microstructure.

<table>
<thead>
<tr>
<th>Resolution Sampling Scheme (VRAD)</th>
<th>Maximum Data Collection Time Required for Entire 1-Bead Leg (Years)</th>
<th>Maximum Data Collection Time Required for Representative Volume (Days)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.31 Isotropic</td>
<td>1.24</td>
<td>2.06</td>
</tr>
<tr>
<td>4.41 (1:1:1/3) Anisotropic</td>
<td>0.95</td>
<td>0.94</td>
</tr>
<tr>
<td>5.29 (1:1:1/4) Anisotropic</td>
<td>1.27</td>
<td>0.88</td>
</tr>
<tr>
<td>26.45 Isotropic</td>
<td>903.93</td>
<td>81.70</td>
</tr>
</tbody>
</table>

**4.2.5 Experimental and Computational Considerations**

There are many details intrinsic to microstructural data collection that must be considered when performing analyses like the one presented in the previous section. A number of implicit assumptions were made in order to estimate total 3D characterization experiment time in addition to the ones that have already been explicitly stated. Moreover, it is likely that some of these assumptions work to discredit the numbers reported in the previous section for total experiment times. However, the study was performed in part to highlight these difficulties, and only serves as demonstration of the capabilities of the microstructural characterization error modeling framework developed in this dissertation. As more computational power becomes available, and experimental tools used to characterize microstructure in 3D become more robust, the previous study will represent more-and-more a reasonable estimation of total experimental time at various sampling resolutions. The list of additional assumptions is discussed below:

1. The physical sample could be mounted in a 1 ½” diameter mounting puck, standing the full 1” tall (the distance along the length), and be mounted perfectly straight up-and-down. The
2” height would have to be cut into at least 3 sections to fit on a single 1 ½” puck, and the experimental apparatus (polishing system, robot transfer arm, SEM) would need to be capable of supporting a 1” tall sample. Note: sawing of the sample to mount it on a standard sized puck would remove material and decrease the total height that could be analyzed.

2. An EBSD scanning routine would have to be programmed into the SEM so that a mechanical stage within the microscope would maintain alignment with the exact geometric positions of each of the sample sections on the puck at each layer. Correspondingly, the various sections would require post processing to align them in-plane so that they would represent the entire sample height.

3. EBSD analysis would be used to analyze the entire sample. In practice, if one were to set out and perform such an experiment it is highly likely that at least part of the data would be collected using grayscale optical or SEM imaging (i.e. SE, BSE), and EBSD scans (if used) would serve as complements to the images and be collected much more infrequently. The polishing and sample preparation for each of these imaging modes is also different for investigation of Ti-6Al-4V microstructures.

4. EBSD batch scans would be reconstructed and processed via TiBor as single layers. Computational feasibility of this is suspect, as nowhere near the 1/20 of the sample height investigated with 1.0 μm step sizes could be analyzed to determine β grain boundaries in a reasonable amount of time. Options for parallel processing while the experiment is ongoing could be a potential workaround for this issue, provided that the appropriate computational power is available.

5. TiBor analysis and cleanup routines would be as accurate with lower in-plane resolutions. This uncertainty has not been investigated, although from inspection of Figure 51 seems highly unlikely.
From an experimentalist’s viewpoint, there are likely many additional sources of uncertainty associated with this study; however, the above list is meant to provide the reader with an idea of some of the difficulties associated with collecting fully 3D serial sectioning data using state-of-the-art tools in their current form.
5 Conclusions and Future Work

5.1 Conclusions

The investigations presented in this dissertation represent important extensions in two key areas of engineering research: 1) 3D microstructural characterization and 2) rapid qualification of components in direct metal additive manufacturing processes.

The novel framework developed for uncertainty quantification in 3D microstructural characterization is the primary contribution of this dissertation. When viewed as a generic tool, the framework can be applied across characterization processes, material systems, and microstructural statistics of interest. The framework was used here to perform virtual serial sectioning experiments that quantified the effect of known experimental errors on measurements of interest, so that future serial sectioning on single-phase materials could be more informed a priori. In fact, throughout the duration of this work, results and conclusions drawn via virtual characterization have helped to make an impact at AFRL by not only providing insights into data collection schemes for roughly equiaxed materials, but also by giving experts a way to quantitatively evaluate a required accuracy for data collection in single-phase materials without actually performing an experiment. The results presented continue to help AFRL researchers collect experimental microstructural characterization data more efficiently and in more cost-effective ways.

The methods developed related to rapid qualification of AM components are timely, because additive manufacturing continues to pick up steam as a major area of interest for academic institutions, government organizations, and industrial companies alike. A novel method for comparing distributions of Ti-6Al-4V Widmanstätten α-lath statistics, using the same analysis techniques leveraged by the characterization error modeling framework, has been demonstrated.
Additionally, the feasibility of collecting 3D microstructural data on β grains in Ti-6Al-4V has been explored.

Overall, the research presented herein provides new contributions to the fields of 3D microstructural characterization and additive manufacturing that include:

1. A novel quantitative analysis framework that performs virtual microstructural characterization to determine optimal data sampling schemes for real experiments
2. Quantitative conclusions regarding the effect of resolution on the accuracy of select grain ensemble statistics for a single-phase lognormal microstructure
3. Quantitative conclusions regarding the effect of noise from EBSD at various spatial resolutions on the accuracy of select grain ensemble statistics for a single-phase lognormal microstructure
4. A virtual validation of the accuracy of a previously published 3D microstructural characterization experiment with novel insights into optimizing future data collection
5. A previously unpublished method for determining and comparing alpha lath width distributions obtained from SEM and EBSD imaging of additive manufactured Ti-6Al-4V components
6. A 3D serial sectioning investigation into the microstructure of additive manufactured Ti-6Al-4V, leveraging virtual characterization to optimize data sampling parameters, with ultimate application to more rapid qualification of AM components

5.2 Future Work

There are numerous ways that the microstructural characterization error modeling framework developed herein can be leveraged for future research. The topics currently of highest interest related to 3D microstructural characterization include investigations into additional classes of
microstructures; including but not limited to, elongated microstructures, bi-modal microstructures consisting of multiple average grain sizes, and multi-phase microstructures. Additionally, although this work focuses on only two sources of error, more complete uncertainty quantification would include effects associated with layer thickness tolerances, errors related to the planarity and alignment of sections, and others. The best way to analyze uncertainty in multi-modal data is also an area that has been underdeveloped. Furthermore, data collection procedures and settings should be analyzed so as to optimize estimates of sectioning time and the like for specific experimental apparatus. To collect even a single EBSD scan, there are many layers of uncertainty associated with each individual pixel that are often ignored once data is collected. Yet another key area of interest for 3D characterization is how to model grain structures with shapes that provide more realistic representations of the complexity of natural materials, rather than using simplistic voxelized representations. In other words, how can smoothing routines and unique structural representations be used to more accurately analyze (or even collect) data in different ways? How can a microstructural characterization error modeling framework for smooth structures perform the equivalent of voxel down-sampling?

In terms of the analysis of microstructures in AM Ti-6Al-4V, there is a plethora of ways to begin analyzing uncertainty associated with both experimental and image processing tools, based on specific measurement(s) of interest. Considering only the \( \alpha \)-lath width analysis example presented in this dissertation, the effects of uncertainty as a function of experimental SEM or EBSD settings is one area that is primed for research contributions. For example, what are the effects associated with changing accelerating voltages or spot sizes during data collection on measurements of mean linear intercepts and corresponding predictions of \( \alpha \)-lath thicknesses? How can the bias induced by a specific image processing routine be quantified for the uncertainty that it adds to \( \alpha \)-lath thickness measurements? How does the variability of
experimentally-controlled factors (i.e., powers, velocities, etc.) used to build AM Ti-6Al-4V components affect the distributions of lath sizes?

Within both general 3D microstructural characterization as well as the analysis of solidification microstructures found in AM components, there are certainly more questions than answers. However, these future directions provide a small glimpse into the wide-open frontier of microstructural characterization research, which at this point appears to have limitless potential.
6 References


11 Materials Genome Initiative for Global Competitiveness, June 2011.


96 Callahan, P.G. (2012) Quantitative characterization and comparison of precipitate and grain shape in Ni-base superalloys using moment invariants (Electronic Dissertation). (Publication No. 3532718)


7 Appendix

This appendix provides raw code and a step-by-step procedure for performing virtual microstructural analysis.

7.1 Appendix A: How to Perform Resolution & Noise Analysis on Virtual Microstructures using DREAM.3D

All of the virtual microstructural analysis performed throughout this dissertation used various versions of DREAM.3D [10] (dream3d.bluequartz.net), which has been under constant development for the duration of the research. The link above can be used to download any current or historical version of the software. Note that the analysis for the journal publication associated with section 3.1 [99] was performed using version 2, while the more recent case studies on Inconel 100 and Ti-6Al-4V β grains were completed with version 5. Version 6, the most current version set for release this year, was used for the resolution and noise + cleanup analysis, and it is this most recent version employed herein.

Outlined below is a process by which anyone can perform similar analyses to those presented in this dissertation using DREAM.3D and MATLAB™, including investigating the effects of both resolution and noise (boundary and/or random) by comparing geometric similarity of PDFs and the percentage error in mean values of PDFs for the following microstructural distributions: grain size (equivalent sphere diameter), grain shape (aspect ratio $b/a$ and $c/a$), and the number-of-nearest neighbor grains. Although the code provided operates within the parameters listed above, it can be easily modified to include additional types of microstructures, sources of error, microstructural parameters, statistical analyses, etc. Much of this modification can be completed through use of these files and/or the DREAM.3D graphical user interface (GUI). Screenshots included in this procedure were taken on a machine using a Windows 7 64-bit operating system, but DREAM.3D is compatible with Windows, Apple,
Linux platforms. Lastly, please note that some code modification will be required, as hard-coded file paths are based on my file structures, and some of the hard-coded character counters in the MATLAB™ files PipelineCreator.m and PipelineRunner.m will need to be modified, as will the file path hard-coded in ResAndNoise.m. The file paths in Phantom_Build_Stats.json and DownSample.json can be easily updated via the DREAM.3D GUI.

Feel free to contact the author if you have any questions or comments regarding the use of this analysis code. ~ *Corresponding author. Email Address: greg.loughnane@gmail.com

1. **Software Download and File Construction**: Download Version 6 of DREAM.3D and create the files listed below in the appropriate format based on the raw code provided in Appendix B.

   1. Phantom_Build_Stats.json
   2. Downsample.json
   3. PipelineCreator.m
   4. ScanPrintLine.m
   5. PipelineRunner.m
   6. GetCSVs.m
   7. ComputeGrainStatistics.m
   8. ResAndNoise.m

2. **Create Statistical Input Size Distribution**: After downloading DREAM.3D and copying the MATLAB files provided in Appendix B, navigate to the new folder and open StatsGenerator.exe.
Choose the microstructure you wish to model by defining an input distribution for grain size. The user can choose to define additional distributions for shape, neighbors, and grain orientation, but based on the grain size input and the Preset Statistical Model, the other distributions will be assigned automatically when “Create Data” is clicked. These other distributions are generated based on microstructural correlations known to exist for the type of Preset Statistic Model (see Ref [87] for an example of these correlations).

After creating the input distributions, save the file in the main DREAM.3D folder.

3. **Modify Phantom_Build_Stats.json & Downsample.json via DREAM.3D GUI**: Open DREAM3D.exe and then open Phantom_Build_Stats.json. You will likely be prompted to replace the input statistics file with the one that you just generated. Go through each filter and make sure that there are no remaining red filters like the one shown below. Red denotes
an error that will prohibit DREAM.3D from running the pipeline. Update all file names and file paths so that they are associated with your file structures.

Go through the same procedure with the DownSample.json file. Note that you can also run a single experiment using just these input files and the DREAM.3D GUI. If this is your first time using DREAM.3D this is a good idea, because you can simply click “Go” in the GUI, perform a single phantom build and down-sampling, and then visualize your new microstructures.

4. **Download the Latest Version of Paraview to View the Microstructures You Just Created:**

Navigate your web browser to Paraview.org and download the latest version. This software is used to visualize the microstructures that are created with DREAM.3D.

Once downloaded, open the software, navigate to the folder where you are saving your created files (this should be the same DREAM.3D folder that StatsGenerator.exe and DREAM3D.exe are in), and open the phantom reference volume generated from
Phantom_Build_Stats.json in DREAM.3D (.xdmf file type). You should see something like this once the file loads:

From the drop down menu that reads “Outline,” select “Surface”. Then, from the drop down menu that reads “Solid Color” select “FeatureIds”. You should now be able to see your microstructure and use the mouse to move it as you would with any typical 3D modeling software.
To compare the two visually, feel free to go through these steps with the down-sampled volume as well.

5. **Modify PipelineCreator.m for Your File Structure**: Count the number of characters in the file path string associated with the .dream3d and .csv output files that you created from DownSample.json. Update PipelineCreator.m to reflect these changes in lines 38 and 46, respectively.

6. **Modify ResAndNoise.m for Your Experiment**: Open ResAndNoise.m and modify the file directory, the resolutions desired for analysis, the noise levels to be investigated, the average grains size, and the instantiation number (which represents the number of times you wish to perform each experiment). Assuming that you will only analyze grain size, aspect ratios, and number-of-nearest neighbors as a first cut, there are just a few modifications that you still must make to the ComputeGrainStatistics.m file, which are noted at the top of the code in Appendix B.
7. **Run ResAndNoise.m:** Try to run the ResAndNoise.m file. If it is successful, you should see
the command window in MATLAB™ doing something like this:

![MATLAB Command Window](image)

If so, congratulations! If not, then it’s time to debug the code. Check that all of your files are
located in the DREAM.3D folder that you originally downloaded, check file paths, etc.

8. **Compute Statistics from Down-Sampled Volumes:** Once you have all of your statistical data
following the simulations, run ComputeGrainStatistics.m from the command window using,
for example, the following input parameters:

```
>> ComputeGrainStatistics(25,10,0.25,0.75)
```

Select the reference volume statistics file for comparison first:
Then choose the down-sampled volume statistics file(s):
You will be prompted to name the output file, and when you do results there will be some select results written to the command window. You can ignore these, as they are saved along with other data automatically in .csv file format.
9. **View Final Statistics Computed between Reference and Down-Sampled Volumes**: Navigate to the files you just created and open “YourFileName_MBCandPercErrMean.csv”. It will appear without descriptive text, however the results correspond to the data shown below, where the numbers in column C correspond to the 95% confidence interval on the answers reported in column B.

Next open “YourFileName_DistData.csv”. These are the collective distributions, with corresponding error bars for each bin and the percent difference in down-sampled
distribution bins relative to the reference volume. ESD is shown first, followed by number-of-nearest neighbors, aspect ratio $b/a$, and aspect ratio $c/a$.

You have officially completed this tutorial. Have fun plotting the data and identifying trends to inform future microstructural characterization!

Cheers,

[Signature]

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7.2 Appendix B: Raw Code to Facilitate Virtual Microstructural Analysis

7.2.1 Phantom_Build_Stats.json

```json
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    "Filter_Human_Label": "Read DREAM.3D Data File",
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                  ],
                  "Version": 2
                },
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                    1
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                  ],
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                }
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            }
          ]
        }
      ]
    }
  }
}
```
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  "Version": 2
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"Version": 2
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"Component Dimensions": [3],
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"Path": "/DataContainers/SyntheticVolume/CellFeatureData",
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  "Flag": 0,
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  "Version": 2
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"Version": 2},
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  "Flag": 0,
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  "Version": 2
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  "Flag": 0,
  "Name": "ShapeVolumes",
  "Object Type": "DataArray<float>",
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  "Version": 2
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  "Flag": 0,
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  "Object Type": "DataArray<float>",
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  "Tuple Dimensions": [

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    "Data Array Name": "",
    "Data Container Name": "StatsGeneratorDataContainer"
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    "Data Array Name": "FeatureIds",
    "Data Container Name": "SyntheticVolume"
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    "y": 0.3,
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        "Data Array Name": "Centroids",
        "Data Container Name": "SyntheticVolume"
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    },
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        "Data Container Name": "SyntheticVolume"
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    "Filter_Human_Label": "Find Biased Features (Bounding Box)",
    "Filter_Name": "FindBoundingBoxFeatures",
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        "Data Container Name": ""
    },
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        "Data Array Name": "SurfaceFeatures",
        "Data Container Name": "SyntheticVolume"
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"Filter_Name": "DataContainerWriter",
"OutputFile": "C:\\Users\\Greg\\Documents\\__rx114data\\2015_MatChar_Noise_Random\\Clean.dream3d",
"WriteXdmfFile": 1
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  "FeatureDataFile": "C:\\Users\\Greg\\Documents\\__rx114data\\2015_MatChar_Noise_Random\\Clean.csv",
  "FilterVersion": "6.0.0",
  "Filter_Human_Label": "Write Feature Data as CSV File",
  "Filter_Name": "FeatureDataCSVWriter",
  "WriteNeighborListData": 0
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    "Data Array Name": "FeatureIds",
    "Data Container Name": "SyntheticVolume"
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  "Filter_Name": "FindEuclideanDistMap",
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  "NearestNeighborsArrayName": "NearestNeighbors",
  "QPEuclideanDistancesArrayName": "QPEuclideanDistances",
  "SaveNearestNeighbors": 0,
  "TJEuclideanDistancesArrayName": "TJEuclideanDistances"
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  "Filter_Name": "ConvertData",
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  "Filter_Name": "AddBadData", 
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  "PoissonVolFraction": 0.25 
},
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    "Data Container Name": "SyntheticVolume" 
  },
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  "ZDirOn": 1 
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    "Data Array Name": "FeatureIds", 
    "Data Container Name": "SyntheticVolume" 
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  "Filter_Name": "FindFeatureCentroids" 
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"FeatureIdsArrayPath": { 
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  "Data Array Name": "FeatureIds",
  "Data Container Name": "SyntheticVolume"
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"Filter_Name": "FindSizes",
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  "AxisLengthsArrayName": "AxisLengths",
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    "Data Array Name": "",
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    "Data Array Name": "Centroids",
    "Data Container Name": "SyntheticVolume"
  },
  "FeatureIdsArrayPath": { 
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    "Data Array Name": "FeatureIds",
    "Data Container Name": "SyntheticVolume"
  },
  "FilterVersion": "6.0.0",
  "Filter_Human_Label": "Find Feature Shapes",
  "Filter_Name": "FindShapes",
  "Omega3sArrayName": "Omega3s",
  "VolumesArrayName": "ShapeVolumes"
},
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  "BoundaryCellsArrayName": "BoundaryCells",
  "CellFeatureAttributeMatrixPath": { 
    "Attribute Matrix Name": "CellFeatureData",
    "Data Array Name": "",
    "Data Container Name": "SyntheticVolume"
  },
7.2.3 PipelineCreator.m

function [] = PipelineCreator(PipelineIn,PipelineOut,D3DFileName,CSVFileName,newres,newpnoise,newbnoise)

    fid = fopen(PipelineIn,'r'); %open data file
    newfile = fopen(PipelineOut,'w'); %Need to name new file here

    end_of_file = 0; %end of file identifier
    ender = 0;
    linecount = 0;

    while end_of_file == 0 && ender == 0
        lines = fgetl(fid);
        linecount = linecount+1; % Initiate counter
        if linecount < 453
            ScanPrintLine(lines,newfile);
        end
    end

end

ScanPrintLine(lines,newfile)
 elseif linecount == 453 %%% XRES
    xres = sscanf(lines,'%c');
    nxres = strcat(xres(1,1:17),newres); fprintf(newfile,'%s,n',nxres);
elseif linecount == 454 %%% YRES
    yres = sscanf(lines,'%c');
    nyres = strcat(yres(1,1:17),newres); fprintf(newfile,'%s,n',nyres);
elseif linecount == 455 %%% ZRES
    zres = sscanf(lines,'%c');
    nzres = strcat(zres(1,1:17),newres); fprintf(newfile,'%s
',nzres);
elseif linecount > 455 && linecount < 485
    ScanPrintLine(lines,newfile);
elseif linecount == 485
    %%% Output
    d3d = sscanf(lines,'%c')
    nd3d = strcat(d3d(1,1:96),D3DFileName);
    fprintf(newfile,'%s"
',nd3d);
elseif linecount > 485 && linecount < 494
    ScanPrintLine(lines,newfile);
elseif linecount == 494
    %%% Output
    csv = sscanf(lines,'%c');
    ncsv = strcat(csv(1,1:101),CSVFileName);
    fprintf(newfile,'%s"\n',ncsv);
elseif linecount > 494 && linecount < 533
    ScanPrintLine(lines,newfile);
elseif linecount == 533 %%% Boundary Noise
    bn = sscanf(lines,'%c');
    nbn = strcat(bn(1,1:31),newbnoise); fprintf(newfile,'%s\n',nbn);
elseif linecount > 533 && linecount < 543
    ScanPrintLine(lines,newfile);
elseif linecount == 543 %%% Poisson Noise
    pn = sscanf(lines,'%c');
    npn = strcat(pn(1,1:30),newpnoise); fprintf(newfile,'%s \n',npn);
elseif linecount > 543 && linecount < 647
    ScanPrintLine(lines,newfile);
end

end_of_file = feof(fid);

end
fclose(fid);
fclose(newfile);
7.2.4  **ScanPrintLine.m**

```matlab
function[] = ScanPrintLine(lines,newfile)
    A = sscanf(lines,'%c');
    fprintf(newfile,'%s\n', A);
end
```

7.2.5  **PipelineRunner.m**

```matlab
function [] = PipelineRunner(dir,file)
    % This function acts as PipelineRunner.exe in DREAM.3D via MATLAB
    %\% Call PipelineRunner
    runner = sprintf('%s', 'PipelineRunner.exe -p ');
    runfile = char(strcat(runner,{' '},file))
    system(dir);
    system(runfile);
end
```

7.2.6  **GetCSVs.m**

```matlab
function [ RefFile, DSFiles ] = GetCSVs()
    % This function retrieves reference and error output information from
    % .csv files generated by DREAM.3D
    [filename,pathname]=uigetfile('*.csv','Choose Original Microstructure Stats File');
    RefFile = char(strcat(pathname,filename));
    [filename,pathname]=uigetfile('*.csv','Choose Corresponding Down-Sampled Files','MultiSelect','on');
    DSFiles=char(strcat(pathname,filename));
end
```

7.2.7  **ComputeGrainStatistics.m**

```matlab
function [mbcfull, mbclow, mbchigh, ds_meanerrors] = ComputeGrainStatistics(no_bins_shape, no_bins_quantiles, qlow, qhigh)
    % This function computes the modified bhattacharyya coefficient for grain
    % size distributions from output .csv files created using PipelineRunner
    % no_bins_shape is the number of bins for full distribution histograms of b/a, c/a
    % no_bins_quantiles is the number of bins for partial distribution histograms
    % qlow is the low quantile of interest for MBC computation
    % qhigh is the high "quantile" of interest for MBC computation
    % Notes:
```
format compact

[RefFile, DSFiles] = GetCSVs();

%% Get Data From ORIGINAL Structure
orig_data = csvread(RefFile,3,0);
[numrows, numcols] = size(orig_data);
cnt=0;
for j = 1:numrows
% Note (cnt,:) makes the resulting vectors to be column vectors
    if orig_data(j,14) == 0; % sort using bounding box algorithm
        cnt = cnt+1;
        orig_grainids(cnt,:) = orig_data(j,1);
        orig_esd(cnt,:) = orig_data(j,18);
        orig_ba(cnt,:) = orig_data(j,2);
        orig_ca(cnt,:) = orig_data(j,3);
        orig_nnn(cnt,:) = orig_data(j,24);
    end
end
orig_esd_sorted = orig_esd(orig_esd~=0); % Remove any zero volume grains
orig_ba_sorted = orig_ba(orig_ba~=0);
orig_ca_sorted = orig_ca(orig_ca~=0);
orig_nnn_sorted = orig_nnn(orig_nnn~=0);

%% Define binning for histograms
mid_bins_esd = [0.25:0.5:13.75]; % Define bins based on grain sizes
bins_esd = [mid_bins_esd inf];
bins_shape = [0:1/no_bins_shape:1]; % b/a, c/a
bins_nnn = [0:max(orig_nnn_sorted)]; % Number of Nearest Neighbors

%% Compute Mean & Standard Deviation for each reference data set
orig_mean_esd = mean(orig_esd_sorted); orig_std_esd = std(orig_esd_sorted);
orig_mean_ba = mean(orig_ba_sorted); orig_std_ba = std(orig_ba_sorted);
orig_mean_ca = mean(orig_ca_sorted); orig_std_ca = std(orig_ca_sorted);
orig_mean_nnn = mean(orig_nnn_sorted); orig_std_nnn = std(orig_nnn_sorted);

%% Determine quantiles and bins for quantile analysis
tp_orig_low_esd = quantile(orig_esd_sorted, qlow);
tp_orig_high_esd = quantile(orig_esd_sorted, qhigh);
tp_orig_low_ba = quantile(orig_ba_sorted, qlow);
tp_orig_high_ba = quantile(orig_ba_sorted, qhigh);
tp_orig_low_ca = quantile(orig_ca_sorted, qlow);
tp_orig_high_ca = quantile(orig_ca_sorted, qhigh);
tp_orig_low_nnn = quantile(orig_nnn_sorted, qlow);
\[ tp_{\text{orig\_high\_nnn}} = \text{quantile}(\text{orig\_nnn\_sorted}, q\text{high}); \]

\[ \text{LowBins}\_\text{esd} = \text{linspace}(0, tp_{\text{orig\_low\_esd}}, \text{no\_bins\_quantiles}); \]
\[ \text{LowBins}\_\text{ba} = \text{linspace}(0, tp_{\text{orig\_low\_ba}}, \text{no\_bins\_quantiles}); \]
\[ \text{LowBins}\_\text{ca} = \text{linspace}(0, tp_{\text{orig\_low\_ca}}, \text{no\_bins\_quantiles}); \]
\[ \text{LowBins}\_\text{nnn} = \text{linspace}(0, tp_{\text{orig\_low\_nnn}}, \text{no\_bins\_quantiles}); \]

\[ \text{HighBins}\_\text{esd} = \text{linspace}(tp_{\text{orig\_high\_esd}}, \text{max(mid\_bins\_esd)}, \text{no\_bins\_quantiles}); \]
\[ \text{HighBins}\_\text{ba} = \text{linspace}(tp_{\text{orig\_high\_ba}}, 1, \text{no\_bins\_quantiles}); \]
\[ \text{HighBins}\_\text{ca} = \text{linspace}(tp_{\text{orig\_high\_ca}}, 1, \text{no\_bins\_quantiles}); \]
\[ \text{HighBins}\_\text{nnn} = \text{linspace}(tp_{\text{orig\_high\_nnn}}, \text{max(\text{orig\_nnn\_sorted})}, \text{no\_bins\_quantiles}); \]

%% Partition original data set into low and high quantile data sets
for \( w = 1: \text{length(\text{orig\_esd\_sorted})} \)
    if \( \text{orig\_esd\_sorted}(w) < tp_{\text{orig\_low\_esd}}; \)
        \( \text{orig\_esd\_low}(w) = \text{orig\_esd\_sorted}(w); \) % Partition for low
    end
    if \( \text{orig\_esd\_sorted}(w) > tp_{\text{orig\_high\_esd}}; \)
        \( \text{orig\_esd\_high}(w) = \text{orig\_esd\_sorted}(w); \) % Partition for high
    end
end for \( w = 1: \text{length(\text{orig\_ba\_sorted})} \)
    if \( \text{orig\_ba\_sorted}(w) < tp_{\text{orig\_low\_ba}}; \)
        \( \text{orig\_ba\_low}(w) = \text{orig\_ba\_sorted}(w); \) % Partition for low
    end
    if \( \text{orig\_ba\_sorted}(w) > tp_{\text{orig\_high\_ba}}; \)
        \( \text{orig\_ba\_high}(w) = \text{orig\_ba\_sorted}(w); \) % Partition for high
    end
end for \( w = 1: \text{length(\text{orig\_ca\_sorted})} \)
    if \( \text{orig\_ca\_sorted}(w) < tp_{\text{orig\_low\_ca}}; \)
        \( \text{orig\_ca\_low}(w) = \text{orig\_ca\_sorted}(w); \) % Partition for low
    end
    if \( \text{orig\_ca\_sorted}(w) > tp_{\text{orig\_high\_ca}}; \)
        \( \text{orig\_ca\_high}(w) = \text{orig\_ca\_sorted}(w); \) % Partition for high
    end
end for \( w = 1: \text{length(\text{orig\_nnn\_sorted})} \)
    if \( \text{orig\_nnn\_sorted}(w) < tp_{\text{orig\_low\_nnn}}; \)
        \( \text{orig\_nnn\_low}(w) = \text{orig\_nnn\_sorted}(w); \); % Partition for low
    end
    if \( \text{orig\_nnn\_sorted}(w) > tp_{\text{orig\_high\_nnn}}; \)
        \( \text{orig\_nnn\_high}(w) = \text{orig\_nnn\_sorted}(w); \) % Partition for high
    end
end
\( \text{orig\_esd\_low\_sorted\_no\_zeros} = \text{orig\_esd\_low}\text{(orig\_esd\_low\_sorted\_no\_zeros\_~0);} % Remove any zeros
\( \text{orig\_esd\_high\_sorted\_no\_zeros} = \text{orig\_esd\_high}\text{(orig\_esd\_high\_sorted\_no\_zeros\_~0);} % Remove any zeros
\( \text{orig\_ba\_low\_sorted\_no\_zeros} = \text{orig\_ba\_low}\text{(orig\_ba\_low\_sorted\_no\_zeros\_~0);} % Remove any zeros
\( \text{orig\_ca\_low\_sorted\_no\_zeros} = \text{orig\_ca\_low}\text{(orig\_ca\_low\_sorted\_no\_zeros\_~0);} % Remove any zeros
\( \text{orig\_nnn\_low\_sorted\_no\_zeros} = \text{orig\_nnn\_low}\text{(orig\_nnn\_low\_sorted\_no\_zeros\_~0);} % Remove any zeros
\( \text{orig\_nnn\_high\_sorted\_no\_zeros} = \text{orig\_nnn\_high}\text{(orig\_nnn\_high\_sorted\_no\_zeros\_~0);} % Remove any zeros

orig_ba_high_sorted = orig_ba_high(orig_ba_high~=0); % Remove any zeros
orig_ca_low_sorted = orig_ca_low(orig_ca_low~=0); % Remove any zeros
orig_ca_high_sorted = orig_ca_high(orig_ca_high~=0); % Remove any zeros
orig_nnn_low_sorted = orig_nnn_low(orig_nnn_low~=0); % Remove any zeros
orig_nnn_high_sorted = orig_nnn_high(orig_nnn_high~=0); % Remove any zeros

%%% Construct original structure histograms
%%% FULL
esd_o = histc(orig_esd_sorted,bins_esd);
norm_esd_o = esd_o./(length(orig_esd_sorted)); % normalize
ba_o = histc(orig_ba_sorted,bins_shape);
norm_ba_o = ba_o./(length(orig_ba_sorted)); % normalize
da_o = histc(orig_ca_sorted,bins_shape);
norm_ca_o = ca_o./(length(orig_ca_sorted)); % normalize
nnn_o = histc(orig_nnn_sorted,bins_nnn);
norm_nnn_o = nnn_o./(length(orig_nnn_sorted)); % normalize

%%% LOW
esd_o_low = histc(orig_esd_low_sorted,LowBins_esd);
norm_esd_o_low = esd_o_low./(length(orig_esd_low_sorted)); % normalize
ba_o_low = histc(orig_ba_low_sorted,LowBins_ba);
norm_ba_o_low = ba_o_low./(length(orig_ba_low_sorted)); % normalize
da_o_low = histc(orig_ca_low_sorted,LowBins_ca);
norm_ca_o_low = ca_o_low./(length(orig_ca_low_sorted)); % normalize
nnn_o_low = histc(orig_nnn_low_sorted,LowBins_nnn);
norm_nnn_o_low = nnn_o_low./(length(orig_nnn_low_sorted)); % normalize

%%% HIGH
esd_o_high = histc(orig_esd_high_sorted,HighBins_esd);
norm_esd_o_high = esd_o_high./(length(orig_esd_high_sorted)); % normalize
ba_o_high = histc(orig_ba_high_sorted,HighBins_ba);
norm_ba_o_high = ba_o_high./(length(orig_ba_high_sorted)); % normalize
da_o_high = histc(orig_ca_high_sorted,HighBins_ca);
norm_ca_o_high = ca_o_high./(length(orig_ca_high_sorted)); % normalize
nnn_o_high = histc(orig_nnn_high_sorted,HighBins_nnn);
norm_nnn_o_high = nnn_o_high./(length(orig_nnn_high_sorted)); % normalize

[numrows,numcols] = size(DSFiles);
for k = 1:numrows;
    %% Get Data From Structures with Errors
    ds_file = DSFiles(k,:);
    ds_data = csvread(ds_file,3,0);
    [numrows,numcols] = size(ds_data);
    cnt=0;
    for j = 1:numrows;
        % Note (cnt,:) makes the resulting vectors to be column vectors
        if ds_data(j,10) == 0; %sort using bounding box algorithm
            cnt = cnt+1;
            ds_grainids(cnt,:) = ds_data(j,1);
            ds_esd(cnt,:) = ds_data(j,14);
        end
    end
end
ds_ba(cnt,:) = ds_data(j,2);
ds_ca(cnt,:) = ds_data(j,3);
ds_nnn(cnt,:) = ds_data(j,16);
end
end
ds_esd_sorted = ds_esd(ds_esd~=0); % Remove any zeros
ds_ba_sorted = ds_ba(ds_ba~=0);
ds_ca_sorted = ds_ca(ds_ca~=0);
ds_nnn_sorted = ds_nnn(ds_nnn~=0);

%%% Construct down-sampled structure histograms
%%% Construct down-sampled structure histogram (FULL)
esd_ds = histc(ds_esd_sorted,bins_esd);
norm_esd_ds = esd_ds./(length(ds_esd_sorted)); % normalize
ba_ds = histc(ds_ba_sorted,bins_shape);
norm_ba_ds = ba_ds./(length(ds_ba_sorted)); % normalize
ca_ds = histc(ds_ca_sorted,bins_shape);
norm_ca_ds = ca_ds./(length(ds_ca_sorted)); % normalize
nnn_ds = histc(ds_nnn_sorted,bins_nnn);
norm_nnn_ds = nnn_ds./(length(ds_nnn_sorted)); % normalize

%%% Partition original data set into low and high quantile data sets
for w = 1:length(ds_esd_sorted);
    if ds_esd_sorted(w) < tp_orig_low_esd;
        ds_esd_low_sorted(w) = ds_esd_sorted(w); % Partition for low
    end
    if ds_esd_sorted(w) > tp_orig_high_esd;
        ds_esd_high_sorted(w) = ds_esd_sorted(w); % Partition for high
    end
end
for w = 1:length(ds_ba_sorted);
    if ds_ba_sorted(w) < tp_orig_low_ba;
        ds_ba_low_sorted(w) = ds_ba_sorted(w);
    end
    if ds_ba_sorted(w) > tp_orig_high_ba;
        ds_ba_high_sorted(w) = ds_ba_sorted(w);
    end
end
for w = 1:length(ds_ca_sorted);
    if ds_ca_sorted(w) < tp_orig_low_ca;
        ds_ca_low_sorted(w) = ds_ca_sorted(w);
    end
    if ds_ca_sorted(w) > tp_orig_high_ca;
        ds_ca_high_sorted(w) = ds_ca_sorted(w);
    end
end
for w = 1:length(ds_nnn_sorted);
    if ds_nnn_sorted(w) < tp_orig_low_nnn;

ds_nnn_low_sorted(w) = ds_nnn_sorted(w);
end
if ds_nnn_sorted(w) > tp_orig_high_nnn;
    ds_nnn_high_sorted(w) = ds_nnn_sorted(w);
end
end
ds_esd_low_sorted = ds_esd_low_sorted(ds_esd_low_sorted~=0); % Remove any zeros
ds_esd_high_sorted = ds_esd_high_sorted(ds_esd_high_sorted~=0); % Remove any zeros
ds_ba_low_sorted = ds_ba_low_sorted(ds_ba_low_sorted~=0); % Remove any zeros
ds_ba_high_sorted = ds_ba_high_sorted(ds_ba_high_sorted~=0); % Remove any zeros
ds_ca_low_sorted = ds_ca_low_sorted(ds_ca_low_sorted~=0); % Remove any zeros
ds_ca_high_sorted = ds_ca_high_sorted(ds_ca_high_sorted~=0); % Remove any zeros
ds_nnn_low_sorted = ds_nnn_low_sorted(ds_nnn_low_sorted~=0); % Remove any zeros
ds_nnn_high_sorted = ds_nnn_high_sorted(ds_nnn_high_sorted~=0); % Remove any zeros
%%% LOW
esd_ds_low = histc(ds_esd_low_sorted,LowBins_esd);
norm_esd_ds_low = esd_ds_low./(length(ds_esd_low_sorted)); % normalize
ba_ds_low = histc(ds_ba_low_sorted,LowBins_ba);
norm_ba_ds_low = ba_ds_low./(length(ds_ba_low_sorted)); % normalize
ca_ds_low = histc(ds_ca_low_sorted,LowBins_ca);
norm_ca_ds_low = ca_ds_low./(length(ds_ca_low_sorted)); % normalize
nnn_ds_low = histc(ds_nnn_low_sorted,LowBins_nnn);
norm_nnn_ds_low = nnn_ds_low./(length(ds_nnn_low_sorted)); % normalize
%%% HIGH
esd_ds_high = histc(ds_esd_high_sorted,HighBins_esd);
norm_esd_ds_high = esd_ds_high./(length(ds_esd_high_sorted)); % normalize
ba_ds_high = histc(ds_ba_high_sorted,HighBins_ba);
norm_ba_ds_high = ba_ds_high./(length(ds_ba_high_sorted)); % normalize
ca_ds_high = histc(ds_ca_high_sorted,HighBins_ca);
norm_ca_ds_high = ca_ds_high./(length(ds_ca_high_sorted)); % normalize
nnn_ds_high = histc(ds_nnn_high_sorted,HighBins_nnn);
norm_nnn_ds_high = nnn_ds_high./(length(ds_nnn_high_sorted)); % normalize
%%% Compute MBCs
mbc_esd(k,:) = real(sqrt(1-sum(sqrt(norm_esd_o.*norm_esd_ds))));
mbc_esd_low(k,:) = real(sqrt(1-sum(sqrt(norm_esd_o_low.*norm_esd_ds_low))));
mbc_ESD_high(k,:) = real(sqrt(1-sum(sqrt(norm_ESD_o_high.*norm_ESD_ds_high))));
mbc_ba(k,:) = real(sqrt(1-sum(sqrt(norm_ba_o.*norm_ba_ds))));
mbc_ba_low(k,:) = real(sqrt(1-sum(sqrt(norm_ba_o_low.*norm_ba_ds_low))));
mbc_ba_high(k,:) = real(sqrt(1-sum(sqrt(norm_ba_o_high.*norm_ba_ds_high))));
mbc_ca(k,:) = real(sqrt(1-sum(sqrt(norm_ca_o.*norm_ca_ds))));
mbc_ca_low(k,:) = real(sqrt(1-sum(sqrt(norm_ca_o_low.*norm_ca_ds_low))));
mbc_ca_high(k,:) = real(sqrt(1-sum(sqrt(norm_ca_o_high.*norm_ca_ds_high))));
mbc_nnn(k,:) = real(sqrt(1-sum(sqrt(norm_nnn_o.*norm_nnn_ds))));
mbc_nnn_low(k,:) = real(sqrt(1-sum(sqrt(norm_nnn_o_low.*norm_nnn_ds_low))));
mbc_nnn_high(k,:) = real(sqrt(1-sum(sqrt(norm_nnn_o_high.*norm_nnn_ds_high))));
%%% Compute Mean & error relative to reference for each down-sampled data set
ds_mean_esd(k,:) = mean(ds_esd_sorted); ds_std_esd(k,:) = std(ds_esd_sorted);
ds_mean_error_esd(k,:) = 100*(ds_mean_esd(k,:) - orig_mean_esd)/orig_mean_esd;
ds_mean_ba(k,:) = mean(ds_ba_sorted); ds_std_ba(k,:) = std(ds_ba_sorted);
ds_mean_error_ba(k,:) = 100*(ds_mean_ba(k,:) - orig_mean_ba)/orig_mean_ba;
ds_mean_ca(k,:) = mean(ds_ca_sorted); ds_std_ca(k,:) = std(ds_ca_sorted);
ds_mean_error_ca(k,:) = 100*(ds_mean_ca(k,:) - orig_mean_ca)/orig_mean_ca;
ds_mean_nnn(k,:) = mean(ds_nnn_sorted); ds_std_nnn(k,:) = std(ds_nnn_sorted);
ds_mean_error_nnn(k,:) = 100*(ds_mean_nnn(k,:) - orig_mean_nnn)/orig_mean_nnn;

%% Store dist data for each down-sampled set
esd_ds_stack(:,k) = norm_esd_ds;
ba_ds_stack(:,k) = norm_ba_ds;
ca_ds_stack(:,k) = norm_ca_ds;
nnn_ds_stack(:,k) = norm_nnn_ds;
clear ds_esd ds_esd_low ds_esd_high ds_ba ds_ba_low ds_ba_high ds_ca ds_ca_low
ds_ca_high
end

%% Output
% Avg & CI for each full MBC
mbc_esd_avg = mean(mbc_esd);
mbc_esd_moe = std(mbc_esd)/sqrt(length(mbc_esd)) * tinv(0.975,length(mbc_esd)-1);
mbc_ba_avg = mean(mbc_ba);
mbc_ba_moe = std(mbc_ba)/sqrt(length(mbc_ba)) * tinv(0.975,length(mbc_ba)-1);
mbc_ca_avg = mean(mbc_ca);
mbc_ca_moe = std(mbc_ca)/sqrt(length(mbc_ca)) * tinv(0.975,length(mbc_ca)-1);
mbc_nnn_avg = mean(mbc_nnn);
mbc_nnn_moe = std(mbc_nnn)/sqrt(length(mbc_nnn)) * tinv(0.975,length(mbc_nnn)-1);
mbcfull = [mbc_esd_avg mbc_esd_moe
mc_ba_avg mbc_ba_moe
mbc_ca_avg mbc_ca_moe
mbc_nnn_avg mbc_nnn_moe];

% Avg & CI for each low MBC
mbc_esd_avg_low = mean(mbc_esd_low);
mbc_esd_moe_low = std(mbc_esd_low)/sqrt(length(mbc_esd_low)) * tinv(0.975,length(mbc_esd_low)-1);
mbc_ba_avg_low = mean(mbc_ba_low);
mbc_ba_moe_low = std(mbc_ba_low)/sqrt(length(mbc_ba_low)) * tinv(0.975,length(mbc_ba_low)-1);
mbc_ca_avg_low = mean(mbc_ca_low);
mbc_ca_moe_low = std(mbc_ca_low)/sqrt(length(mbc_ca_low)) * tinv(0.975,length(mbc_ca_low)-1);
mbc_nnn_avg_low = mean(mbc_nnn_low);
mbc_nnn_moe_low = std(mbc_nnn_low)/sqrt(length(mbc_nnn_low)) * tinv(0.975,length(mbc_nnn_low)-1);
mbclow = [mbc_esd_avg_low mbc_esd_moe_low
mc_ba_avg_low mbc_ba_moe_low
mbc_ca_avg_low mbc_ca_moe_low
m
mbc_nnn_avg_low  mbc_nnn_moe_low];

% Avg & CI for each high MBC
mbc_esd_avg_high = mean(mbc_esd_high);
mbc_esd_moe_high = std(mbc_esd_high)/sqrt(length(mbc_esd_high)) *
tinv(0.975,length(mbc_esd_high)-1);
mbc_ba_avg_high = mean(mbc_ba_high);
mbc_ba_moe_high = std(mbc_ba_high)/sqrt(length(mbc_ba_high)) *
tinv(0.975,length(mbc_ba_high)-1);
mbc_ca_avg_high = mean(mbc_ca_high);
mbc_ca_moe_high = std(mbc_ca_high)/sqrt(length(mbc_ca_high)) *
tinv(0.975,length(mbc_ca_high)-1);
mbc_nnn_avg_high = mean(mbc_nnn_high);
mbc_nnn_moe_high = std(mbc_nnn_high)/sqrt(length(mbc_nnn_high)) *
tinv(0.975,length(mbc_nnn_high)-1);
mbchigh = [mbc_esd_avg_high mbc_esd_moe_high
mbc_ba_avg_high  mbc_ba_moe_high
mbc_ca_avg_high  mbc_ca_moe_high
mbc_nnn_avg_high  mbc_nnn_moe_high];

% Avg & CI for each percentage error in the mean
ds_mean_error_esd_avg = mean(ds_mean_error_esd);
ds_mean_error_esd_moe = std(ds_mean_error_esd)/sqrt(length(ds_mean_error_esd)) *
tinv(0.975,length(ds_mean_error_esd)-1);
ds_mean_error_ba_avg = mean(ds_mean_error_ba);
ds_mean_error_ba_moe = std(ds_mean_error_ba)/sqrt(length(ds_mean_error_ba)) *
tinv(0.975,length(ds_mean_error_ba)-1);
ds_mean_error_ca_avg = mean(ds_mean_error_ca);
ds_mean_error_ca_moe = std(ds_mean_error_ca)/sqrt(length(ds_mean_error_ca)) *
tinv(0.975,length(ds_mean_error_ca)-1);
ds_mean_error_nnn_avg = mean(ds_mean_error_nnn);
ds_mean_error_nnn_moe = std(ds_mean_error_nnn)/sqrt(length(ds_mean_error_nnn)) *
tinv(0.975,length(ds_mean_error_nnn)-1);
ds_meanerrors = [ds_mean_error_esd_avg ds_mean_error_esd_moe
                ds_mean_error_ba_avg  ds_mean_error_ba_moe
                ds_mean_error_ca_avg  ds_mean_error_ca_moe
                ds_mean_error_nnn_avg ds_mean_error_nnn_moe];

% Avg & CI for each distribution  
ds_esd_dist_avg = mean(esd_ds_stack,2);
ds_esd_dist_moe = std(esd_ds_stack,0,2)/sqrt(min(size(esd_ds_stack))) *
tinv(0.975,min(size(esd_ds_stack))-1);
ds_ba_dist_avg = mean(ba_ds_stack,2);
ds_ba_dist_moe = std(ba_ds_stack,0,2)/sqrt(min(size(ba_ds_stack))) *
tinv(0.975,min(size(ba_ds_stack))-1);
ds_ca_dist_avg = mean(ca_ds_stack,2);
ds_ca_dist_moe = std(ca_ds_stack,0,2)/sqrt(min(size(ca_ds_stack))) *
tinv(0.975,min(size(ca_ds_stack))-1);
ds_nnn_dist_avg = mean(nnn_ds_stack,2);
ds_nnn_dist_moe = std(nnn_ds_stack,0,2)/sqrt(min(size(nnn_ds_stack))) *
tinv(0.975,min(size(nnn_ds_stack))-1);
% Compute percent difference for each bin relative to reference

ds_esd_dist_bin_error = 100*abs((norm_esd_o - ds_esd_dist_avg));
ds_ba_dist_bin_error = 100*abs((norm_ba_o - ds_ba_dist_avg));
ds_ca_dist_bin_error = 100*abs((norm_ca_o - ds_ca_dist_avg));
ds_nnn_dist_bin_error = 100*abs((norm_nnn_o - ds_nnn_dist_avg));

ResultsToWrite = [mbcfull
    1e+100*ones(1,2)
    mbelow
    1e+100*ones(1,2)
    mbchigh
    1e+100*ones(1,2)
    ds_meanerrors];

DistDataToWrite = [bins_esd' norm_esd_o ds_esd_dist_avg ds_esd_dist_moe
ds_esd_dist_bin_error
    1e+100*ones(1,5)
    bins_shape' norm_ba_o ds_ba_dist_avg ds_ba_dist_moe ds_ba_dist_bin_error
    1e+100*ones(1,5)
    bins_shape' norm_ca_o ds_ca_dist_avg ds_ca_dist_moe ds_ca_dist_bin_error
    1e+100*ones(1,5)
    bins_nnn' norm_nnn_o ds_nnn_dist_avg ds_nnn_dist_moe ds_nnn_dist_bin_error];

DescribeResults = input('What do you want to call these results? : ','s')

%Write MBC and Percent Error Results to .csv
filename = strcat(sprintf('%s',DescribeResults),sprintf('%s', '_MBCandPercErrMeanData'),sprintf('%s', '.csv'));
csvwrite(filename,ResultsToWrite,1,1);

%Write distribution data to .csv
filename = strcat(sprintf('%s',DescribeResults),sprintf('%s', '_DistData'),sprintf('%s', '.csv'));
csvwrite(filename,DistDataToWrite,1,1);

% dir ~ Directory
dir = sprintf('%s', 'cd C:\Users\Greg\Documents\__rx114data\2015_MarChar_Noise_Random');
%% Build Phantom & Compute Stats
rp = sprintf('%s', 'Phantom_Build_Stats.json');
PipelineRunner(dir,rp)

%% Create and Run Pipelines for Error Analysis
res3 = linspace(1.0,1.0,4); % 3 VRAD Results
res5 = linspace(0.6,0.6,4); % 5 VRAD Results
res10 = linspace(0.3,0.3,4); % 10 VRAD Results
res15 = linspace(0.2,0.2,4); % 15 VRAD Results
res = [res3 res5 res10 res15];

%NOISE
pn_repeat = 0.01*[10 25 50 75]; % Random Noise Levels to be investigated
bn_repeat = linspace(0.0,0.0,4); % Boundary Noise Levels to be investigated
pn = [pn_repeat pn_repeat pn_repeat pn_repeat]; % Repeat for each res
bn = [bn_repeat bn_repeat bn_repeat bn_repeat]; % Repeat for each res

k = 20; % Instantiation Number (# structures at each noise level)
for i = 1:k
    instant_num = num2str(i);
    for j = 1:length(res)
        AvgGrainSize = 3;
        A = sprintf('%i', (AvgGrainSize/res(j)));
        B = sprintf('%i', (pn(j)*100));
        C = sprintf('%i', (bn(j)*100));

        % New Pipeline .txt File Names
dsp{j,1} = strcat(sprintf('%s', '_'), num2str(A), sprintf('%s', 'VRAD'), ...        sprintf('%s', '_'), num2str(B), sprintf('%s', 'pn'), ...        sprintf('%s', '_'), num2str(C), sprintf('%s', 'bn'), ...        sprintf('%s', '_'), instant_num, sprintf('%s', '.json'));

        % DREAM.3D File Names
d3d{j,1} = strcat(sprintf('%s', '_'), num2str(A), sprintf('%s', 'VRAD'), ...        sprintf('%s', '_'), num2str(B), sprintf('%s', 'pn'), ...        sprintf('%s', '_'), num2str(C), sprintf('%s', 'bn'), ...        sprintf('%s', '_'), instant_num, sprintf('%s', '.dream3d'));

        % CSV File Names
csv{j,1} = strcat(sprintf('%s', '_'), num2str(A), sprintf('%s', 'VRAD'), ...        sprintf('%s', '_'), num2str(B), sprintf('%s', 'pn'), ...        sprintf('%s', '_'), num2str(C), sprintf('%s', 'bn'), ...        sprintf('%s', '_'), instant_num, sprintf('%s', '.csv'));

    end
end

% Run New Pipeline
NewRes = sprintf('%2.2f', (res(j)));
NewPN = sprintf('% 2.2f', (pn(j)));
NewBN = sprintf('% 2.2f', (bn(j)));
PipelineCreator('DownSample.json', dsp{j}, d3d{j}, csv{j}, NewRes, NewPN, NewBN);
PipelineRunner(dir, dsp{j});

end

end