Qualification and Characterization of Metal Additive Manufacturing

by

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B.S. Chemical Engineering, University of Maine, 2009

Submitted to the MIT Sloan School of Management and the Department of Aeronautics and Astronautics in partial fulfillment of the requirements for the degrees of

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Abstract

Additive manufacturing (AM) has emerged as an effective and efficient way to digitally manufacture complicated structures. Raytheon Missile Systems seeks to gain limited production capability with metals AM, which can only be achieved with qualified, predictable processes that reduce variation. The project documented in this thesis produced two results needed to qualify AM for use on flight-critical parts: i) creation of a standard qualification process building upon Raytheon's product development knowledge, and ii) selection and identification of key metals AM process factors and their corresponding experimental responses.

The project has delivered a qualification test plan and process that will be used next year to drive adoption and integration of Raytheon's metals AM technology. The first phase of the designed experiment on AM process factors was completed by experimenting with coupon orientation, position on the build platform, coupon shape and hot isostatic pressing (HIP) post-treatment for an Al alloy (AlSi10Mg) produced via laser powder bed fusion using 400-watt laser equipment. Only coupon orientation had a statistically significant effect on dimensional accuracy, increasing the variance of y-axis (within the build plane) error by ~50%, although this is considered a small increase. HIP decreased yield and ultimate stresses by ~60% while increasing ultimate strain by ~250%. Vertical orientation of coupons decreased yield and ultimate stresses by ~25% and increased ultimate strain by ~30%. Small coupon area on the build platform, associated with thin rectangle coupons, decreased yield stress and ultimate strain by ~5%. The processes and case study from this thesis represent a general advance in the adoption of metals AM in aerospace manufacturing.

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Contents

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List of Figures

List of Tables

Acronyms

AM — Additive Manufacturing

AMF — Additive Manufacturing File Format

ANOVA — Analysis of Variance

ASTM — formerly American Society for Testing and Materials

AWS — American Welding Society

CAD — Computer-Aided Design

CI — Confidence Interval

CMM — Coordinate-Measuring Machine

CT — Computed Tomography

DFAM — Design For Additive Manufacturing

DED — Directed Energy Deposition

DoD — Department of Defense (United States of America)

DoE — Design of Experiments

EB — Electron Beam

EDM — Electrical Discharge Machining

ESD — ElectroStatic Discharge

FDM — Fused Deposition Modeling

FEA — Finite Element Analysis

HIP — Hot Isostatic Pressing

IV&V — Integration, Verification & Validation

IR — InfraRed

ksi — thousands of pounds per square inch (psi)

LEAP — Leading Edge Aviation Propulsion

LS — Laser Sintering

μm — micron (micrometer)

MMPDS — Metallic Materials Properties Development and Standardization

MPa - Megapascals

M&P — Materials & Practices

MRA — Manufacturing Readiness Assessment

MRL — Manufacturing Readiness Level

NASA — National Aeronautics and Space Administration

NDE — Non-Destructive Evaluation

NIST — National Institute of Standards and Technology

OLS — Ordinary Least Squares

PBF — Powder Bed Fusion

PDR — Product Design Review

PSD — Particle Size Distribution

PM — Preventative Maintenance

R_a — arithmetic average of absolute values of roughness

RMS — Raytheon Missile Systems

RMSE — Root Mean Square Error

RPM — Raytheon Precision Machining

SAS — (Raytheon) Space and Airborne Systems

SEM — Scanning Electron Microscope

SLA — StereoLithography Additive (manufacturing)

SLS — Selective Laser Sintering

SME — Subject Matter Expert

SPC — Statistical Process Control

STL — STereoLithography (file format)

TRL — Technology Readiness Level

ULA — United Launch Alliance

UTS — Ultimate Tensile Strength

UV — UltraViolet

Chapter 1

Introduction

Raytheon is a global technology company with four major divisions that support a wide range of defense-oriented systems and products. One of those divisions, Raytheon Missile Systems, seeks to incorporate additive manufacturing of metals into new programs and develop the body of knowledge around applying additive manufacturing to aerospace challenges.

This section is an introduction to the motivations of the project, what current business opportunity prompted the research topic, and an overview of the contents of the thesis.

1.1 Purpose of Project

Additive manufacturing (AM), sometimes known as "3-D printing", is a manufacturing process by which computer-aided designs (CAD) are converted into a physical item through the successive combination of multiple layers of material(s) and optional post-processing steps. ASTM Standard F2792 defines additive manufacturing as "a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies" (ASTM Standard F2792, 2012). While AM has existed for decades and has already revolutionized rapid prototyping and production of plastic components, new developments in AM manufacturing technology have provided aerospace companies with a growing opportunity to build flight-ready structural components out of a variety of metal alloys, ceramics and composites. There are three major drivers of interest in AM: the ability to produce components that cannot be made with machining or casting, a reduction in waste material and a reduction in time required between CAD and finished component. Raytheon seeks to apply the advantages of AM to manufacturing metal components on vehicles with flight capability in their precision guided weaponry business and in other areas throughout the company.

Raytheon Missile Systems, out of Tucson, AZ, has recently acquired a laser Powder Bed Fusion (PBF) machine and seeks to understand the process required to qualify and characterize the performance of the equipment and the raw materials currently available on the marketplace for aerospace applications. In the defense industry, customer requirements dictate that all new manufacturing technologies must pass certain levels of capability and reliability before use in products that are available to purchase. The U.S. Department of Defense (DoD) uses a system of manufacturing readiness assessment (MRA) and levels (MRL) to benchmark manufacturing technologies as they are developed from initial concept to full-scale production. As a DoD vendor, Raytheon has implemented the MRA system and needs to assess Raytheon's current capabilities in metal AM, as well as understand the schedule and cost associated with increasing the MRL of metals AM for specific applications. This thesis project is designed to provide Raytheon Missile Systems an improved awareness of its metal AM capabilities and provide a roadmap for driving increased metals AM readiness levels throughout its product lines.

1.2 Problem Statement

Metals AM provides many unique opportunities for aerospace production environments, including significantly reducing component lead times and enabling innovative designs that are impossible to create with machining, casting or forging. However, metals additive manufacturing is a term that encompasses many different methods of manufacturing, almost all of which create non-isotropic (sometimes called anisotropic) properties in their final products and introduce new kinds of defects. Qualification standards and procedures for metals AM have not been fully defined in industry, with many different standards organizations like ASTM International and the American Welding Society (AWS) creating parallel but distinct standards for metal alloys produced with AM.

As a result of incomplete standards and limited public research, many large aerospace corporations have been investing substantial resources into metal AM to use in their next generation of products. While this behavior drives demand for and development of AM metal alloys and production equipment, it has created fragmentation of information within the industry. Organizations like America Makes are working to create a more collaborative environment, but most companies interested in remaining competitive in advanced manufacturing techniques are required to invest capital or time into qualifying metals AM capability.

Raytheon has invested over the past few years in various forms of additive manufacturing, mostly in the rapid prototyping or tooling support domains. With the addition of a laser PBF machine to their factories, AM will now have the potential to support program requirements for airframe components. However, without the support of this thesis project, the required qualification and characterization steps are unclear: the framework of AM qualification within Raytheon Missile Systems had yet to be defined.

1.3 Problem Approach and Hypothesis

To solve some of the issues apparent in the problem statement above, the proposed approach is to create a broad "roadmap" for AM qualification (specifically as it pertains to metals) and develop more detail in a specific area of qualification testing. A deep dive into the early stages of qualification work will lay the foundation for successful testing in later stages. The more rigorous examination also provides the opportunity to pose several hypotheses and test them using experimental data. In this thesis, it is hypothesized that there are several part configuration choices that will have an impact on the output quality and mechanical properties of components constructed using metal AM.

To develop Raytheon's AM qualification process, the first task is to collect information about current qualification processes used within Raytheon's production. Data gathered from previous test plans can be combined with interviews to create an internal body of knowledge that represents the current state of qualification. The project will also incorporate current industry findings and conclusions to create a comprehensive and instructive framework for metals AM qualification. In order to refine and prove the value of this framework to stakeholders within the organization, the framework will be applied in parts to a suitable pilot project that seeks to use AM in flight-ready programs.

After the qualification process has been fully developed and tested, the opportunity arises to design and analyze some of the preliminary experiments within the first phase of the qualification framework. This thesis project proposes applying the principles of experimental design, usually referred to as Design of Experiments (DoE), to the initial parameter testing for the operation of metals AM equipment. DoE is designed to maximize information collection given schedule and budget constraints, allowing the Operations group within Raytheon to begin to understand the capabilities of their equipment and material quickly and less expensively. Experimenting can provide any future users of the technology a baseline understanding of the mechanical properties of the AM equipment/material system, while also screening many of the factors that could impact system performance. Conclusions generated from the completed screening test can be used to avoid problem areas in the future or identify areas that require additional research and experimentation.

1.4 Thesis Overview

The thesis begins with an introduction to the content matter and challenges facing the implementation of metals AM at Raytheon. The next chapter summarizes the available research and scientific consensus on the state of metals AM (with a focus on laser PBF) and provides insight into future developments. Chapter three covers a general overview of metal manufacturing at Raytheon and the current progress in incorporating metal additive manufacturing. The methodology used in the research and analysis of the thesis project is covered in chapter four, including the development process of the qualification plan and DoE. Chapter five describes in detail the proposed metals AM qualification process and identifies opportunities for future applications. Chapter six explains the experimental design used to screen some of the laser PBF operating parameters and presents the analysis of experimental results. The final chapter presents the overall conclusions of the thesis and recommendations for further development in metals AM.

Chapter 2

Literature Review

This chapter's purpose is to review current and recent studies on the field of metals additive manufacturing to better understand the current state of the art and any existing areas that need further research. In this way it is possible to integrate the contents and purpose of this thesis into the already-existing body of scientific work. In addition, this chapter will begin by providing needed background information on metals AM and the concept of MRLs mentioned earlier.

2.1 Manufacturing Readiness Levels and Applications

The Manufacturing Readiness Level (MRL), as defined by the Department of Defense (DoD), is an extension of the well-known Technology Readiness Level (TRL) already used to systematically describe the technological maturity of research and development efforts. The formal definition of MRL comes from the DoD MRL Deskbook and "in conjunction with TRLs, are key measures that define risk when a technology or process is matured and transitioned to a system" (OSD Manufacturing Technology Program, 2015). Table 2.1 provides the description and alignment of the nine TRLs and ten MRLs.

TRLs have been adopted by many US defense contractors and have been incorporated into Raytheon's gated product development process. However, a Technology Readiness Assessment is incapable of answering the question, "is this level of performance reproducible in items 2-1000?" (Morgan, 2006). Only a Manufacturing Readiness Assessment (MRA) can

Table 2.1: TRL/MRL Levels (OSD Manufacturing Technology Program, 2015)

	TRL	**MRL**
1	Basic principles observed and reported	Basic manufacturing implications identified
2	Technology concept or application formulated	Manufacturing concepts identified
3	Experimental and analytical critical function and characteristic proof of concept	Manufacturing proof of concept developed
4	Component or breadboard validation in a laboratory environment	Capability to produce the technology in a laboratory environment
5	Component or breadboard validation in a relevant environment	Capability to produce prototype components in a production relevant environment
6	System or subsystem model or prototype demonstrated in a relevant environment	Capability to produce a prototype system or subsystem in a production relevant environment
7	System prototype demonstration in an operational environment	Capability to produce systems, subsystems or components in a production representative environment
8	Actual system completed and "flight qualified" through test and demonstration	Pilot line capability demonstrated; ready to begin low rate initial production
9	Actual system "flight proven" through successful mission operations	Low rate production demonstrated; capability in place to begin full rate production
10	N/A	Full rate production demonstrated and lean production practices in place

consider the development of the technology in a manufacturing environment, and the MRL is used to describe the ability of a technology to go into full production.

In addition to providing guidelines used to qualify a technology according to its MRL, the DoD resources also describe what should be contained in the MRA. An MRA should consist of a briefing or written report that identifies and describes both the current and target MRL of the discussed technology. Based on observations made on a walkthrough of the shop floor or laboratory areas and information from the developing organizations, the report should identify areas where documented manufacturing readiness does not meet the stated target MRL. A useful report will also provide suggestions on what programs and plans will help a technology reach its target MRL, as well as assessing risk and cost associated with meeting that target (Morgan, 2008).

Adoption of MRLs has proceeded in stages at many DoD defense contractors. According to Grillon, MRAs have been required in some programs funded by the DoD in the format of an audit before approving milestone completion (Grillon, 2012). To better drive incorporation of the MRA into the standard technical review process at companies like Raytheon, the

DoD has provided its mapping of TRLs and MRLs onto the system development process, as seen in Figure 2-1. What should be noted in the figure is that MRLs of a single technology significantly lag the TRLs in time, as it takes substantial research and engineering effort to develop the technology for manufacturing. The diagram is intended to show that both TRLs and MRLs develop along similar gated pathways.

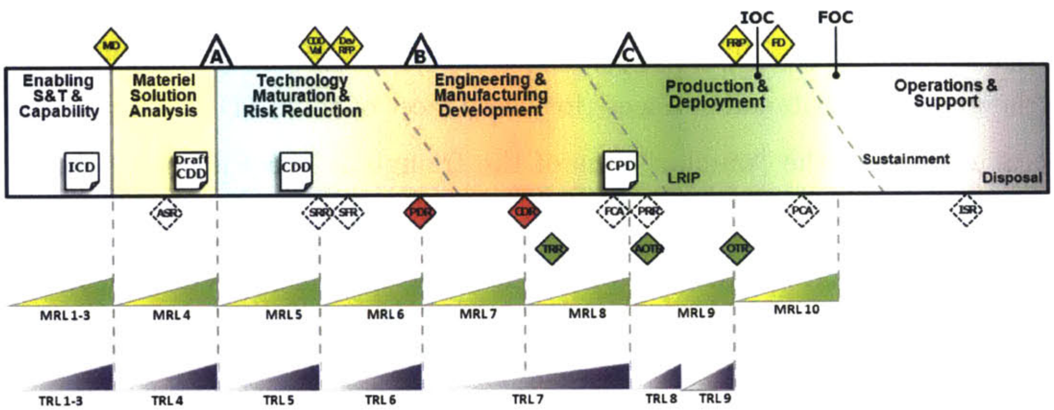

Figure 2-1: Relationship of MRLs to Decision Points, Milestones, Technical Reviews and TRLs (OSD Manufacturing Technology Program, 2015, p. 20)

In Section 5.5, MRLs will be assigned to different uses of metals AM for a selection of defense applications. Using the principles laid out in this section, a preliminary MRA will be completed for each of the new applications.

2.2 State of Metal AM Processes in Industry

This section provides an overview of metal additive manufacturing processes, both from the perspective of process steps inherent in all AM techniques and also the methods used specifically for processing metals. Later, some of the economics of metals AM are addressed in current research, supplemented with analysis of competitor progress (as reported in public record).

2.2.1 Overview of Metal AM

Additive manufacturing has existed for several decades and has been defined many times since its early roots in rapid prototyping. Gibson et. al. provide a summary of the seven major steps involved in most additive manufacturing technologies (Gibson et al., 2015):

1. CAD: AM parts start with a completely described 3D solid defined in any professional Computer-Aided Design (CAD) modeling software. All external geometry and internal surfaces must be fully defined.

2. Conversion to STL: This is the current standard for delivering design data to an AM machine. All surfaces must be fully enclosed because stereolithography (STL) files use triangles to describe all surfaces (for every facet of the CAD file, three points and a vector normal to the "outside" face of the triangle). There is an ongoing drive in the industry to adopt ISO/ASTM 52915 Specification for AM File Format (AMF) Version 1.1 (ISO/ASTM Standard 52915, 2013), as it includes all STL data as well as supporting full color definition and multi-material gradients (Frazier, 2014, p. 1918)

3. Transfer to AM Machine and STL File Manipulation: The STL file for the build must be transferred to the AM equipment. This can be done by portable storage or networked drives. Designs specified by STL files may be resized, manipulated or combined with other designs in order to modify part build orientation or increase utilization of the build process by creating multiple parts simultaneously.

4. Machine Setup: AM machines require initial setup before starting the build process. This usually involves modifying build parameters like layer thickness, energy source power, timing, etc. (discussed in Section 5.2.2). This step can be completed by selecting manufacturer-provided presets. Upon loading the STL into the AM equipment, internal software cuts the prescribed build into equal-thickness layers in a process called "slicing".

5. Build: Building the part is mostly an unattended and automated process that only requires intervention in the case of an error. Some equipment may require additional material to be loaded.

6. Removal: Parts completed by AM process must be extracted from the equipment, either manually or with automation. This can include waiting for safety interlocks to power down or removing considerable leftover raw material.

7. Post-processing: In addition to any more required cleaning of the material, parts may need several different type of post-processing treatment. These can involve heat treatment, machining work, surface finishing, plating or painting. This step is heavily dependent on AM process and final application.

The two AM techniques with the most relevance to metals manufacturing are powder bed fusion (PBF) and directed energy deposition (DED). PBF is a "process in which thermal energy selectively fuses regions of a powder bed", while DED is one where "focused thermal energy is used to fuse materials by melting as they are being deposited" (ASTM Standard F2792, 2012). PBF is an AM process that completely melts the powder exposed to thermal energy, as opposed to processes that leave powder only partially melted. Metal PBF can be further separated into two different types of processes, based on the energy source used for melting. Laser PBF, a form of laser sintering (LS), uses high-power fiber-optic lasers and optics to melt sections of the powder bed, and Electron Beam (EB) PBF does the same under vacuum with a magnetically-focused electron beam. NIST has provided a rough diagram of laser PBF in Figure 2-2.

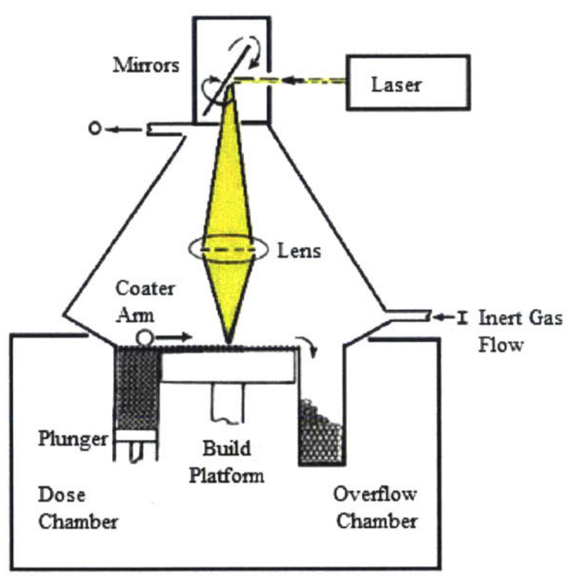

Figure 2-2: Optical Powder Bed Fusion Operational Diagram, modified from (NIST, 2013)

At the bottom of the operating chamber, there are three components: from left to right, the dose chamber, the build platform and the overflow chamber. For each layer indicated in the slicing plan dictated by the AM equipment, the dose chamber plunger elevates and pushes up a layer of powder material. The build platform descends a height equal to the desired layer thickness; this is referred to as coater blade height (relative to the build platform). A coater arm with a flexible blade moves across the dose chamber and drags powder across the build platform, leaving a smooth layer. Most equipment has a dose factor setting that can supply more material than required from the dose chamber; 100% corresponds to a powder thickness equal to layer thickness. Dose factor can be increased to improve spreading of powder on the build plate, at the expense of powder waste. Excess powder dragged across the surface falls into the overflow chamber and the coater arm returns to its home position.

In the upper right of Figure 2-2, an infrared (IR) laser emitter directs pulses of light toward a set of projection mirrors that aim the light through a focusing lens (seen in the center of the diagram). Laser light is aimed onto any part of the build platform's powder layer that should be solid. The laser energy melts the powder, and the melt pool immediately fuses when the laser is turned off. Once the laser has fused the cross-section of an entire layer, the series of powder coating and lasing steps repeat until all layers in the build file have been completed. EB PBF is very similar to laser PBF, with a trade of electron beam for laser as the energy source and the requirement of vacuum in the process chamber (laser PBF only requires a gas that is inert with the materials being processed).

2.2.2 Economics

As of 2011, aerospace usage of AM consisted of 12% of total AM production in the US, which represented 0.02% of all aerospace component shipments in dollars. However, this percentage is higher than any of the motor vehicle, industrial, architectural or consumer product industries (Thomas and Gilbert, 2014, p. 2). One of the largest drivers for adoption of metals AM in aerospace applications are the potential cost savings in materials. Many of the more desirable materials for aerospace materials, like titanium, Inconel and advanced aluminum alloys, have high bulk material costs relative to steels and other standard structural materials. To make the issue worse, subtractive manufacturing methods can drive significant losses of material during the part production process. The aerospace industry uses a ratio called "buy-to-fly", or the ratio of material purchased for a given part compared to the amount of material on the finished part after machining or molding is complete. It is usually calculated as the volume of the billet divided by the volume of the component. The critical buy-to-fly ratio has been found to be 12:1, where any aerospace components that exceed that value should be considered candidates for AM on materials cost alone (Thomas and Gilbert, 2014).

The cost of material usage in metals AM is also influenced by intrinsic requirements of the new process mechanics. When considering cost and manufacturability, Vayre et. al. noted that there is a large cost associated with powderizing any metal for use in AM. If an aerospace component producer can find an economical use case for AM technology, they are much more likely to be able to use titanium or aluminum alloys as needed. The powderizing cost substantially reduces the difference per mass unit between "expensive" metals like titanium and "cheap" metals like aluminum and encourages performance-based material selection (Vayre et al., 2012). Innovative uses of existing aerospace materials are only one aspect of the economic draw for AM users.

Beyond material cost concerns, there are other operational costs of AM that are being researched and analyzed. US Navy research has hypothesized that an application of AM to its maintenance programs, combined with an overhaul of its lifecycle management technology, would save $1.49 billion annually on staffing and organizational cost alone (Kenney, 2013). This is due to the reduction in supply chain, manufacturing, and deployment resources needed if the Navy were to replace some failed components with AM-produced components made on demand. This is sometimes known as digital inventory management, where AM is used to supply small-rate production of components and existing traditional production lines can be shut down or allocated to other work.

AM is known currently for being low-volume and low-rate, but developments on improving build rate and machine utilization could change the comparison between AM and traditional manufacturing. By utilizing more powerful laser sources now available on the market (up to 1000W, compared to 100 – 400W in most rapid protoyping equipment), Buchbinder et. al. were able to demonstrate a four-to-five-times increase in laser PBF build rates without having detectable effects on the hardness, density and tensile strength of the aluminum alloy tested (Buchbinder et al., 2011). Another experiment demonstrated the ability to use the entire build volume in a production environment, using five parts to fill 93% of the available area on the AM build platform (Baumers et al., 2015). Using the vertical space of the build volume is still challenging due to difficulties in stacking non-contiguous parts. There is an opportunity to develop software that could aggregate queued components into optimal configurations that would minimize wasted machine time while meeting production deadlines.

Enabling many of these economic comparisons with traditional manufacturing requires additional development work on the cost models of AM processes. The total cost, build time and energy consumption characteristics of PBF have been characterized by many different studies, but barely anything is understood about the cost and schedule models of DED (Thomas and Gilbert, 2014). Unfortunately, the cost modeling is heavily dependent on material type, machine manufacturer and application-specific post-processing requirements. While there is considerable research being done to support adoption of AM as an affordable manufacturing technology, there are many challenges to increasing the overall adoption rate in the aerospace industry.

2.2.3 Competitor Research

When developing a strategy for qualifying and characterizing metals AM, it is crucial to look outside of what Raytheon is doing and understand what other companies in the aerospace field are accomplishing with the technology. NASA has been examining potential applications of 3D printing, not just in Earth's gravity but also in microgravity, for aerospace missions. Researchers believe there is some opportunity to bring AM equipment to space and assemble spacecraft components as needed, but they are hesitant to adopt AM completely amid "a substantial degree of exaggeration, even hype, about its capabilities in the short term." (*3D Printing in Space*, 2014) There are definite applications of AM within NASA's mission, but it appears that the agency would prefer to have private companies lead the way publicly for AM.

Airbus and the United Launch Alliance (ULA) have been working with Ultem plastic made with fused deposition modeling (FDM) on reducing complexity of assemblies with AM. Ultem is a high-strength plastic intended for a flexible trade with some aluminum alloys (see chapter 3 for a more detailed comparison of Ultem with aluminum). Neither company is considering serial production at the moment and are mostly focused on rapid prototyping. ULA was able to demonstrate an assembly redesign for an Atlas V component that started as 140 aluminum components and ended as 16 plastic components (Warwick, 2015).

Aerojet Rocketdyne, a partner of Raytheon, is also developing components for its rocket motors using AM. Their director of advanced launch programs provides a stern warning to any company working in this segment: "If rigorous material characterization and design system work is not done up front, it [has] the potential to give the overall technology a bad name." (Butler, 2015). This quote highlights the safety and reputational high stakes intrinsic in creating new qualification procedures that are sufficient to demonstrate flight-quality parts.

GE has demonstrated a significant commitment to metals AM, so far contributing over $190 million to its own AM centers and pledging a total of $3.5 billion in investment for AM over the next five years (Zaleski, 2015b). Its first AM-produced part on a flying engine will be a sensor assembly redesigned for AM to reduce icing issues. The part was approved by the FAA in February 2015 (Zaleski, 2015a). GE's long-term goals are to support the designed-for-AM Leading Edge Aviation Propulsion (LEAP) engine nozzle made of a cobalt-chromium alloy. The parts selected for initial AM research echo the choices made by Airbus and ULA to focus on the benefit AM can provide in reducing assembly complexity.

In all of these cases, the data obtained in qualification studies has remained proprietary and, while focused here on metal AM, is typical for the current environment of general AM research outside academia. Investment costs are so high to develop a new manufacturing method that no company is independently willing to share information and give up a competitive advantage. To combat this tendency, America Makes, formerly known as the National Additive Manufacturing Innovation Institute, has opened membership to companies of all sizes in the US and provided funding for universities and businesses to collaborate on solving AM characterization and qualification problems. To reduce fragmentation within the industry, America Makes requires that the results from all research funded by their organization be released back to the other members of America Makes. Raytheon is a corporate member of America Makes and has garnered some of the research awards. This thesis, released publically through university channels, will also represent a direction in AM collaboration that is needed to advance US additive manufacturing capability.

2.3 Ongoing Research in Metal AM Qualification

The following section is designed to provide context for some of the choices made in the experimental sections of this thesis. By providing summaries of existing relevant research in metal AM qualification, this section will identify areas that are missing experimental results and procedures that can be recognized as best practice and should be adopted in future experiments. Included in this section are three subsections: development of experimental designs throughout the range of parameters that impact AM, additional characterization of material or process behavior and advances in in-situ process monitoring to support full-scale production efforts. For the purposes of this literature review, most of the research covered is limited to laser PBF.

2.3.1 Experimental Design

Parameters important to experimental design in this thesis are informed by prior work. One set of critical parameters for laser PBF is the control scheme for the laser emitter. Yadroitsev has estimated that there are over 130 parameters that can affect the quality of a part produced by laser PBF, but not all are equally important (Yadroitsev, 2009). Critical parameters would include the laser power, scan speed, and scan-line spacing all controlled by the AM equipment (Kamath et al., 2014). Kamath et. al. focused on the effects of laser power and scan speed on the melt pool behavior of 316 stainless steel powder. The experiment found that laser scan rates had an optimum value for a given laser power, due to bracketing by two different phenomena. When laser power was too low for a given scan speed, incomplete melting was observed. When laser power was too high for a scan speed, experimenters observed development of keyhole voids in multiple layers of metal. This was caused by metal evaporation and plasma formation in the path of the laser, causing vapor cavities throughout the material (Kamath et al., 2014, p. 66). Both of these outcomes caused porosity in the finished metal that was higher than desired. The authors emphasized that they completed physics-based modeling before undertaking experiments; this increased the robustness of their experimental design.

The research on power and scan speed pointed to restrictions based on a concept addressed in a different paper, the formula for applied energy density. Applied energy density, usually represented as E_A, is the energy applied by the laser power source per unit area of powder bed and can be described by Equation 2.1.

$$E_A = \frac{100P}{US} \tag{2.1}$$

E_A is the applied energy density in kJ/cm^2, P is the nominal laser power in watts (W), U is the laser beam scan speed in mm/s and S is the spacing between scan lines in microns (μm)—100 is a scaling factor for the units in the equation. For pure and alloy aluminum powders, Olakanmi demonstrated that there are specific melting properties associated with different ranges of E_A values (Olakanmi, 2013). This research indicated that it was not possible to select laser power, scan spacing and scan speeds that were totally independent: there is tight coupling between those parameters that ranges between effects of no melting at all, to overmelting and void formation. This frees future experiments that desire high-density parts from struggling to assign values to all three parameters and screen for independent effects. Selecting laser parameters that yield different E_A values reduces the unknown set of parameters and captures the physical melting phenomenon.

Some researchers were not content with using commonly available equipment to test the ranges and impact of different laser parameter settings. As mentioned earlier, Buchbinder et. al. obtained lasers for PBF that would provide up to 1000W of power and were able to increase build speeds (volume per time) up to four or five times higher than existing baseline equipment without impacting mechanical properties of the samples (Buchbinder et al., 2011). Some AM equipment manufacturers, like Concept Laser, have observed this same principle in their own testing and are shipping their highest-end equipment with single and dual kilowatt lasers.

Another group of researchers has developed an innovative laser pulsing technique to control surface finish issues on Inconel 625 laser PBF. The report observed that low laser scan rates improved surface finish of final products by allowing the melt pool to stabilize before solidifying. However, some metals like Inconel have metal "balling" issues when the metal is allowed to remain melted for longer duration. Instead of spreading out radially from the melt point, the melt pool forms into a sphere that then solidifies on top of the previous layer. The researchers proposed shaping the laser pulses applied to the powder bed in order to suppress the initial overshoot of laser power that tended to spatter the melt pool and worsen surface finish (Mumtaz and Hopkinson, 2010). The proposed method can be applied with only laser power control logic and may even be retrofitted to existing equipment.

The final two examples that inform experimental design in this section were completed in plastic material and provided insight on the impact of build orientation and spacing on final build quality. The first experiment with a stereolithographic process demonstrated that the orientation of layer-to-layer bonding had an impact on tensile strength of test specimens. When the test specimens were rotated so that their individual layers had a smaller cross-sectional area, there was a significant decrease in the ultimate tensile strength when tested along the plane axis of the layers (Quintana et al., 2010). The second experiment used a 3D printing method for plastics known as polyjet printing, which is dependent on ultraviolet (UV) exposure to harden and reach usable levels of material strength. The researchers demonstrated that orientation of different test specimens and their spacing on the build platform had an impact on UV light scattering during exposure that significantly affected final mechanical properties (Barclift and Williams, 2012). While laser optics do not suffer all of the same interference and scattering effects present in the mentioned experiment, both experiments demonstrate a need to understand the impact of orientation and spacing on the quality of metals AM.

2.3.2 Material and Post-Processing

In 2013, NIST held a conference to develop a measurement science roadmap for metals AM. Two of the high-priority challenges the group identified were a limited understanding of metal powder properties and a similar lack of information about post-processing steps. To help mitigate the issues with metal powder knowledge, NIST researchers began characterizing

Table 2.2: List of Powder Metrology Methods and Associated Physical Characteristics (Slotwinski et al., 2014)

Measurand	**Metrology Method**
Density of particles	Helium Pycnometry
Particle Size Distribution	Laser Diffraction
Particle Size and Morphology	X-Ray Computed Tomography
Particle Crystalline Phases	X-Ray Diffraction
Particle Morphology	Scanning Electron Microscopy
Particle Elemental Composition	Energy Dispersive Elemental Analysis
Particle Surface Molecular/Chemical Composition	X-Ray Photospectroscopy

several AM alloy powders with a variety of analysis methods. They were able to conclude that powders provided by their suppliers were consistent within a production lot, but more importantly they demonstrated and explained a large list of measurement techniques that were able to characterize aspects of the powder that would be critical for final product quality (Slotwinski et al., 2014). See Table 2.2 for a list of the metrology practices with the examined physical characteristic.

As mentioned earlier, a major economic advantage of AM lies in its conservation of materials. While scrap from a conventional machining process has to undergo a lengthy reclamation process, the leftover raw material from an AM process can often be reused on-site immediately after a minor quality control process. For PBF AM techniques, this poses a characterization challenge. The powder that remains on the build platform but is not affected by laser light can be reused, but it is no longer "virgin" powder and must be handled differently. In order to use this powder for multiple builds, the effect of reuse on the powder's final mechanical properties should be understood. Multiple studies have been completed on a variety of metal powders for use in the aerospace industry, including Inconel 718 (Ardila et al., 2014), Ti-6Al-4V (Tang et al., 2015) and AlSi10Mg (Rosenthal et al., 2014).The experiments on Inconel 718 demonstrated that there were very few physical effects on the powder morphology and particle size distribution (PSD) after multiple reuses. As long as a robust procedure for recycling is adopted that includes sieving powder to remove partially-melted aggregates and drying to remove moisture, the results demonstrated no changes in metallurgical or mechanical properties and only 6% powder loss up to 14 recycle iterations.

The titanium alloy did not perform as well, but was also subject to a different AM technique (EB PBF) that requires powder bed preheating, which can cause sub-melt particle shape changes. With 21 recycle iterations, the experiment observed the powder particles becoming less spherical, but mechanical properties of parts produced actually improved: ultimate tensile strength increased 12% and the results were statistically significant. The major concern of the results was the compositional change of the powder: element loss under vacuum and oxidation were changing the elemental composition of the powder, something that can shift the alloy content out of the acceptable range for certain customers. The final experiment on aluminum demonstrated very little change to the PSD and other powder characteristics under three recycle iterations, demonstrating that for low recycle counts, recycled powder is nominally equivalent to virgin powder. Further testing would be required to ensure that mechanical properties are not changed due to recycling.

One of the most promising post-processing techniques for metals AM is adapted from existing powder metallurgy processing. Hot isostatic pressing (HIP) is a heat treatment of over 500 °C combined with a high ambient pressure generated by gas at near 100 MPa. While HIP is used on its own to die-cast some metal parts, there are initial experiments that show it can have beneficial effects on the porosity of completed metal AM parts. A study on titanium showed that HIP decreased porosity in AM titanium alloys as well as improving the fatigue strength of the material to approaching wrought quality (Frazier, 2014). HIP reduced the tensile strength of Inconel 625 produced with EB PBF by 25% but increased the ductility by 57%, providing an opportunity to trade one mechanical property for another depending on desired application. HIP's promise of modifying mechanical characteristics is one reason it has been included in the experimental design of this thesis. Recycling powder has also been included in the experimental design to continue to develop the research around material properties and post-processing techniques, but recycling powder tests are outside the scope of this thesis.

2.3.3 Process Monitoring

The other key challenge identified at the NIST measurement science roadmap session was a near-complete lack of process monitoring technology and equipment available for metals

AM. At its current stage of manufacturing readiness, AM is considered a "special process" by Raytheon's Quality organization. Special processes are manufacturing techniques that have few acceptable methods of closed-loop process control, and instead quality assurance depends on comprehensive nondestructive evaluation (NDE) and destructive sample evaluation. This agrees with the two primary methods of manufacturing metrology accepted in the field described by Moylan et. al.: quality data collection can either monitor "a series of direct measurements of system components [and] characteristics" or execute tests *ex post facto* on artifacts specifically designed for analysis (Moylan et al., 2014). The requirement to test in two ways is similarly seen in aerospace carbon fiber applications, where both the process conditions and mechanical properties of samples must be qualified.

The designation of AM as a special process is due in part to the difficulty of integrating sensors into the AM equipment workspaces, which usually involve mechanized components moving in 3D space, constant exposure to radiation or material spray and residence in vacuum or a controlled gas environment. The fact that many available commercial AM machines have closed-source, restricted controllers also inhibits incorporating experimental improvements into the AM equipment and hinders development of *in-situ* process control (Slotwinski, 2014). Unfortunately, traditional NDE methods that would be used to address the issues with process monitoring have also failed to meet expectations for metals AM use. Waller et. al. have referred to a current "valley of death" for AM based on available NDE methods, where metals AM desperately requires better characterization methods to grow but the needed metrology technology is still in early stages of development, caught between TRLs 3 and 6 (Waller et al., 2014). See their report for additional details on the problems discovered with computed tomography (CT) scanning, dye penetrant testing and eddy current testing on as-built metal AM components.

One of the most promising developments in process monitoring for AM is a CT method that uses an IR camera to observe each layer as built in a laser PBF machine. Each camera image is combined later with software to provide a 3D representation of the completed part with areas of abnormally low or high heat highlighted (Zenzinger et al., 2015). One completed image can be seen in Figure 2-3 and shows features with a resolution of 0.1 mm/pixel.

While experimenters are still trying to solve challenges like how to maintain optical

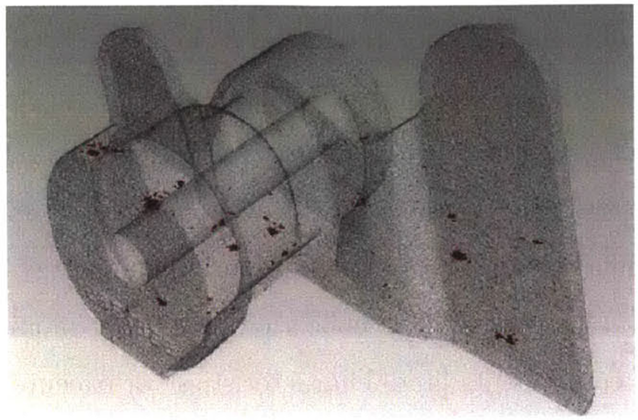

Figure 2-3: Completed 3-D Tomography Model with Temperature Variations Highlighted (Zenzinger et al., 2015, p. 170)

clarity for the viewing window in a metal spray environment, this technique is already being piloted on a collaboration with EOS, one of the laser PBF equipment manufacturers. Similar testing has been completed on an ultrasonic testing method for laser PBF of Inconel 718 (Rieder et al., 2015). Researchers affixed an ultrasonic transducer to the bottom of the build platform on an existing piece of AM equipment and were able to detect the surface appearance of the most recently melted layer of metal. While this method is restricted to non-complex geometries, it can be applied to building reference parts that accompany each AM component. While there are some developing opportunities for in-situ process monitoring, metals AM development is currently slowed by a lack of usable NDE techniques or parameters to enable closed-loop process control.

Chapter 3

Advanced Metal Manufacturing at Raytheon

At Raytheon, qualification of metals AM is being developed within an environment that has been at the leading edge of aerospace design and manufacturing for the better part of a century. This chapter discusses some of that background and the company's current progress in implementing metals AM, as well as describes the role that AM will provide in future operations.

3.1 Background on Company

Raytheon Corporation is a large multinational technology company that primarily provides defense-oriented solutions to the US federal government and the governments of allied countries around the world. The company employs about 70,000 people worldwide and is headquartered in Waltham, MA. The home site for the thesis execution is the headquarters of Raytheon Missile Systems, a wholly-owned subsidiary in Tucson, AZ that develops, produces and supports precision missile and combat systems. Raytheon Missile Systems (RMS) booked $6.3 billion in sales in 2014 on the success of its 25 weapons systems in active use by US forces and approximately 40 foreign countries (Raytheon, 2014). Raytheon Missile Systems began life in 1951 as a facility of Hughes Aircraft Company, based on eccentric founder Howard Hughes' fears that his West Coast facilities on the Pacific Ocean were vulnerable to enemy attack. The Tucson site changed ownership several times after Hughes' death but was eventually acquired by Raytheon Company in 1997 and has been the central site for much of the defense industry consolidation of precision guided weaponry driven by Raytheon acquisitions (Tucson Regional Economic Opportunities, 2013). Raytheon Missile Systems has continued to expand, with facilities in Mississippi, Texas and New Mexico.

3.2 Overview on Advanced Metal Manufacturing

Integration of components for a product whose customers demand 100% success requires advanced manufacturing capabilities for aerospace-grade metal alloys. The majority of RMS's workforce is within the Engineering directorate, but there is still substantial machining expertise in both the Tucson and Texas locations. While most casting and forging production of near-net shape components is completed by suppliers, RMS has several centers of excellence in high-precision finish machining designed to deliver components ready for paint and final assembly. Some products' key components are supplied solely by Raytheon's internal machining centers by production from raw metal stock, which significantly shortens the supply chain and reduces reliance on outside vendors to provide critical-path items. Machining operations are supported by well-developed Materials & Practices (M&P) labs that are capable of a variety of destructive and nondestructive analytical techniques. Raytheon has also invested in coordinate-measuring machines (CMM) to support verification of its machining quality on an ongoing basis.

Make-buy decisions are made for every metal component as program development completes its cost and schedule analyses. When program managers choose to source in-house, they must consider the cost of tooling and support equipment and the business opportunity cost of dedicating limited Raytheon labor and equipment to the current effort. In addition, suggestions from the end customer and past experience may limit adoption of new metal manufacturing technologies: cost-effective use of new manufacturing techniques requires adequate, non-"siloed" communication between internal development efforts and program engineers. When considering purchasing components for a new program, managers have to factor in long lead times based on industry demand as well as potentially high costs for individual components when purchased in low quantities. The sensitive nature of many of RMS' projects also preclude selection of many suppliers for critical components. While Raytheon has developed a robust Industrial Security program approved by the Defense Security Service, few suppliers can afford to reconfigure work areas and infrastructure to support work on classified projects.

3.3 Metal Additive Manufacturing Progress

A key component of improving a process is understanding and synthesizing its state today, before any changes are made. In this section, the current state of metals AM at Raytheon is discussed in terms of strengths and challenges.

While metals AM at Raytheon is relatively new, additive manufacturing in other materials has been adopted and developed within Raytheon for many years. There are several AM development centers throughout the company that have focused on fused deposition modeling (FDM) and stereolithography (SLA), two plastics AM manufacturing methods. FDM equipment is operated almost continuously to support tooling and manufacturing aids destined for the company's factories, while SLA machines build early prototypes and create patterns for investment casting. Raytheon funds AM equipment housed near the factory as well as in the company's own discreet development laboratory known as the 'Bike Shop'. The Bike Shop is well-known in the industry for bringing ideas and technology to working prototypes very quickly (Karp, 2004). Qualification efforts are limited due to the rapid turnover of projects and specifications in a prototype laboratory.

Some initial metals AM qualification efforts have started and primarily focus on thermal performance for housing flight components with high heat loads. The structural properties of metals produced with AM must meet certain minimum requirements for these designs, but do not require the stringent level of testing and certification expected out of components whose failure will result in total loss of mission. These initial efforts have been supported within the company by past efforts in qualifying composite materials for use on flight hardware, due to the similarity in concerns about material anisotropy and the challenges in completing effective NDE.

One of the major limitations in adopting AM throughout different programs at RMS has been missing or incomplete standards for design for additive manufacturing, or DFAM. Raytheon has standardized many of its manufacturing design guidelines in order to encourage standard work throughout the engineering design organization and reduce the difficulty of adapting certain designs to high-volume production. In April 2015, subject matter experts on AM within Raytheon Corporation as a whole completed their collaboration to create a similar standard set of DFAM guidelines that provides decision-making support on using AM for a given application. The guidelines also provide categories of material and AM technique performance to allow for preliminary trade considerations. Once a user has selected a material and manufacturing technique, he or she can then review the suggestions for modifying design principles that have been provided by AM experts in that area. For example, designers of components for powder- or liquid-based AM methods are instructed to ensure that any intentional voids in the component volume also connect to exit channels to permit drainage of excess material. While the DFAM guidelines have been successful in providing a needed milestone in the AM technology development process, the institutional lag time in adopting this new information still requires a significant amount of design support from the AM center operators. Unfamiliarity with the design principles causes design rework once a program's planning files are delivered to the AM center.

Another challenge faced in adopting metals AM has been in assessing the cost of using AM as compared to traditional manufacturing methods. Program managers typically use software like DesignProfit to study the cost of manufacturing each individual unit of production at the quantities desired by the customer (and in some cases, the long-term strategic view of the program's success in the market). DesignProfit allows for defining multiple scenarios where components are produced with different manufacturing techniques and costs. Scenarios can be compared on a total project cost or on cost/unit. This method of cost estimation is well understood within the company, but metals AM has been difficult to incorporate into the process for several reasons:

- The relationship between an AM component's volume and the required build time has not been clearly defined and varies between AM techniques, equipment within the same supplier family and the parameters defined in setting up the AM build. It also varies significantly based on the component's geometry and orientation during the AM build. With all of these sources of variation affecting predictions, the best estimate for build time can only be provided just before builds execute and are estimated by the AM equipment itself, which limits availability.

- Similarly, correlation between build volume and material usage (to obtain raw material costs) has not been fully defined for each AM technique. Best practices suggest using a percentage of waste associated with the finished component build, but this would vary with different policies of material reuse.

- Labor rates for AM equipment setup and support in existing factories have not been fully defined based on operational costs. Their colocation with traditional machining equipment but vastly differing operational requirements have confounded initial efforts to assign a per-hour rate that matches the lower attendance time required by AM equipment.

- Outside suppliers may have more experience with the above issues through AM builds for many different customers, but the customizability of design that AM enables prevents simple 'as-like' comparisons and limits a supplier's ability to provide reliable cost and time estimates.

With time, each of these areas will become better defined as experimentation and initial production runs generate data that can be used to predict time and cost, regardless of any actual understanding or modeling of the build process. In order to support further implementation of metals AM components, cost estimation requires more rigorous data based on the experiences of using AM equipment that Raytheon plans to use in the future.

3.4 Need for Metal Additive Manufacturing

Additive manufacturing is a technology that is transformational in its effect on any industry that implements it. In the long term, aerospace design and manufacturability will radically change as more people explore the technique's capability. However, even in the short term and given all of the challenges mentioned previously, there are distinct opportunities for RMS to pursue using metals AM. This section will cover three distinct use cases: new programs that utilize advantages unique to AM, rapid production of high-quality manufacturing supports and replacement of components in end-of-life programs.

One of the major difficulties with introducing metals AM has been identifying a 'killer' application, one for which additive manufacturing is perfectly suited as a nascent technology. New developments in software capability and processing power have created the ability to complete topology optimization on CAD projects. In summary, topology optimization combines finite-element analysis (FEA) with new drafting algorithms that are capable of redefining a given functional design and isolating only the material needed to support the projected loads. The optimization aspect of the software iterates on each of these FEA calculations and redesigns until reaching a point where the component has nearly constant strain energy throughout its volume (Gibson et al., 2015). At this point, the component's volume and mass (for homogeneous materials) has been minimized. More advanced software packages may then adapt the optimized design into one that is more manufacturable, either with AM or with traditional manufacturing techniques. The design parameters for AM are often more accommodating to 'organic' designs, but current costs per unit of AM would likely encourage using machined or cast parts for large-scale production. For certain applications, AM will be the only way to manufacture an optimized design because traditional machining is unable to create some features (e.g., internal voids). Figure 3-1 gives a visual representation of the topology optimization process.

RMS has programs that can benefit from this design and manufacturing process today. New programs currently in development require components made of Inconel 625, a high-temperature and high-pressure nickel alloy that is considerably more dense than aluminum used in aerospace applications. In order to meet the design requirements of those components and the overall package weight, each component would need its mass reduced by 50% of the mass of a fully dense part. With traditional manufacturing techniques, this could require substantial alteration of the external surface or require castings with extremely complicated internal structures. Metals AM enables this component to be manufactured as a single piece with its internal geometry optimized to meet the program requirements. While trade

Figure 3-1: Design Steps in Topology Optimization (Siemens PLM Software, 2011). From upper left, clockwise: (1) functional structure definition (2) finite-element analysis of loads (3) component redesign to minimize volume (4) normalization of design for manufacturability.

studies have not been completed for these critical components yet, there is strong evidence that these components cannot be produced without some contribution from AM. Internal structures and topology optimization are a 'killer' application for AM that has only started being investigated.

As mentioned previously, a common application of AM in Raytheon facilities is supporting manufacturing with tooling and equipment carriers. These are well-tested uses of the rapid deployment capabilities of AM and a demonstration of the utility of a production method that can be heavily customized for every part produced. While plastic tooling and carriers are suitable for many uses inside the factory, plastics do not meet strength and rigidity requirements for certain applications that require very close tolerances or high tool loads. Research within Raytheon has shown that plastic tools made with FDM can break or wear with surprising frequency. While this is a low concern for users with an FDM machine at the ready to make a new copy of the same tool, FDM machine utilization has been

Table 3.1: Comparison of Mechanical Properties of Common FDM Resins (Stratasys, 2015) and Metal Powders (Concept Laser, 2015)

Material	Nylon 12		Polycarbonate		Ultem 1010		ABS-ESD7	ABS-M30		CL 31AL	CL 101NB
Axis of Build	XZ	ZX	XZ	ZX	XZ	ZX	unknown	XZ	ZX	unknown	unknown
Tensile Strength, Yield (psi)	4,600	4,100	5,800	4,300	9,300	6,100	–	4,550	3,750	24,600	92,800
Tensile Strength, Ultimate (psi)	6,650	5,800	8,300	6,100	11,700	5,400	5,200	4,650	4,050	44,900	133,400
Tensile Modulus (ksi)	186	165	282	284	402	322	350	320	320	10,800	29,000
Elongation at Break	30%	5.4%	4.8%	2.5%	3.3%	2.0%	3%	7%	7%	3%	32%
Elongation at Yield	2.4%	2.7%	2.2%	2%	2.2%	1.5%	–	2%	2%	–	–

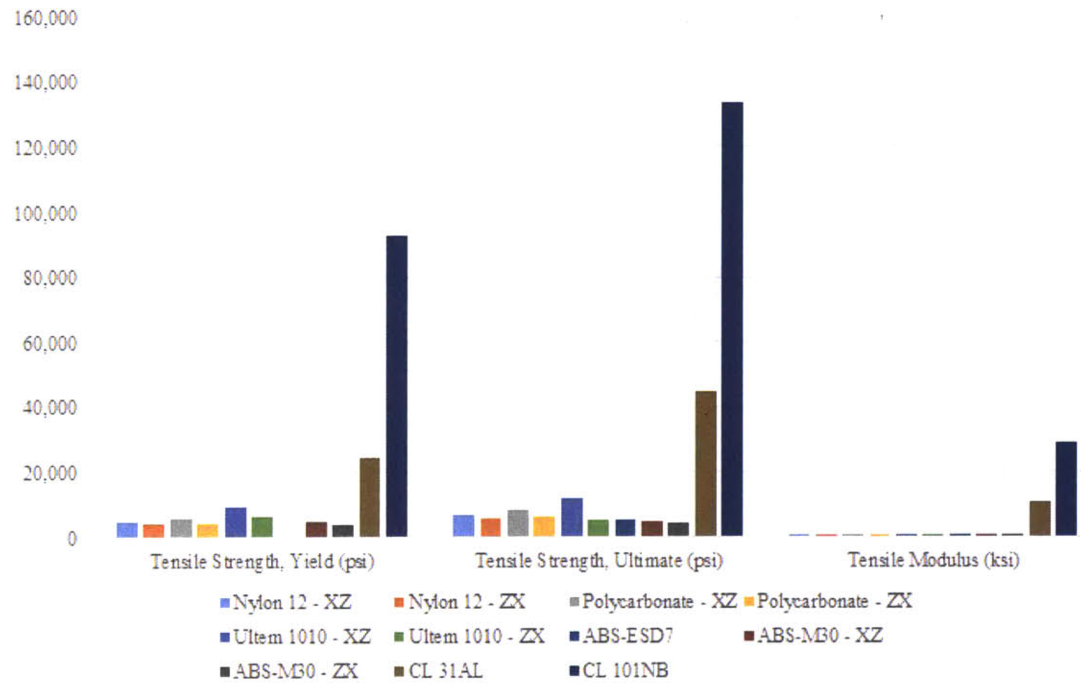

Figure 3-2: Comparison of Mechanical Properties of Common FDM Resins (Stratasys, 2015) and Metal Powders (Concept Laser, 2015)

steadily increasing. Tools made with metals AM have the opportunity to combine the rapid development advantages with the known strength of metal materials. Table 3.1 and Figure 3-2 show the differences between baseline mechanical properties of some common FDM resins available for Stratasys equipment, compared to the same properties of aluminum and Inconel metal components built with laser PBF on Concept Laser equipment.

While the strength of Ultem resin is on the same order of magnitude as the CL 31AL alloy (an AlSi10Mg composition), many tools and carriers used in Raytheon factories must be rated as electrostatic discharge (ESD) safe. This requires some level of conductivity much higher than most plastics, and so many tools are produced using carbon-filled ABS-ESD7, which has significantly lower strength properties than an AM aluminum alloy. In addition, suppliers for electroless nickel plating are more numerous for aluminum alloys than most plastics. Electroless nickel plating is a chemical plating process that deposits a very hard and smooth layer of nickel alloy onto the substrate of choice. This is a preferred surface treatment for tooling destined for work on space-related programs, as the cleanroom environment demands lower particle release behavior than either raw AM aluminum or plastic can provide. There are many advantages to developing and testing metals AM for improving factory support materials.

The final use case analyzed in the thesis is using metals AM for simple component replacement in aging or end-of-life programs. Native English speakers will be familiar with a proverb, abbreviated below, that warns against underestimating logistical issues with minor parts (Baldwin, 1912):

For want of a nail the shoe was lost;
For want of a shoe the horse was lost;
For want of a horse the battle was lost;
For the failure of battle the kingdom was lost—
All for the want of a horse-shoe nail.

For a company that produces precision weaponry, this platitude is slightly more literal than normal. When programs approach end-of-life and receive new orders, they may face extreme difficulty obtaining components like screws and brackets that are conceptually easy to produce but often problematic to acquire from suppliers who have gone out of business or simply discontinued the component. Program managers are faced with backing out of the agreement to produce additional rounds of weapons (usually not an option), or spending a significant amount of time and money sourcing a replacement component either through external suppliers or internal machining capability. Combined with 3-D scanning technology and digital inventory, metals AM provides the opportunity for programs lacking replacement parts to produce exact duplicates of the original parts, in only the quantities needed and with a short lead time (no tooling time required). The main obstacle to adoption of this replacement process today is the 'delta' qualification required after any changes to a component in a product. Some programs may lack the budget to do delta qualifications and may possibly lack funds to qualify metals AM processes for their single application. This roadblock to further metals AM development, as well as the path forward for the other applications discussed in this section, will be further analyzed in Section 5.5 in the context of the larger metals AM qualification process.

Chapter 4

Methodology

Developing a robust and rigorous manufacturing quality plan requires scientific rigor and should be based on past best practices. This chapter describes the methods used to collect information from industry and within Raytheon itself, the development stages of the AM characterization and qualification process and the genesis of the AM experimental design used as a pilot for the testing recommended by the characterization plan. This chapter also contains a summary of the key measures of success within the project to align the content of this thesis with the desired outcomes of the AM qualification projects at Raytheon.

4.1 Data Collection Methods

Most data for this project has been collected from Raytheon sources. Interviews of staff members from many different departments allowed for an overall view of the company and helped build an understanding of the current state of AM at RMS. This was followed by visits to manufacturing sites involved with AM work to build knowledge of the infrastructure available to support AM. Throughout the project, resources from past qualification efforts were used to provide frameworks onto which new information was attached. Finally, best-practice statistical analysis and experimental design was incorporated from academia and existing literature to supplement the subject matter expertise available at Raytheon.

4.2 Development of Qualification Plan

Starting a new qualification process for metals AM required the input of many people and integration of substantial work completed in the past at Raytheon. The first step in the process was meet with all stakeholders involved and collect requirements for the qualification steps. Engineering teams, quality personnel and materials & practices analysts were all consulted to build a cohesive view of what a qualification plan must contain to satisfy those groups and Raytheon's customers. After requirements were laid out, the initial framework for the qualification process was adapted from a similar effort completed at a different division of Raytheon, Space and Airborne Systems (SAS). The original process was focused on qualifying a component for thermal performance and needed to be adapted for use with components that would encounter aerodynamic loads. The adapted framework continued to develop with input from experts throughout RMS in additive manufacturing and other special processes like composites manufacturing. When a process flow chart was fully defined, it was submitted for approval and adoption to the Systems Engineering lead for a potential pilot application of metals AM. Upon approval of the overall qualification and characterization strategy, engineers and analysts continued to develop the framework with more detail from industry best practices. This involved replicating testing required to gain approval for Metallic Materials Properties Development and Standardization (MMPDS) and prove that a material's mechanical properties fall within design allowables for Raytheon's customers.

4.3 Design of Experiment for AM Process Parameters

After most of the metals AM qualification process was identified and described (see chapter 5), one element of that structured plan was selected for further study: the materials and equipment characterization phase. Raytheon had recently purchased metals AM equipment for laser PBF and needed to rapidly characterize its behavior for AlSi10Mg component production in order to meet demand for prototypes and small production runs. As covered in the literature review, significant effort was spent initially to understand the major drivers in the laser PBF manufacturing flow and identify possible areas for experimentation and opportunities for new discovery. The initial set of selected parameters included laser control parameters (like those that constitute E_A) as well as powder handling characteristics. Some portions of the experimental design were directly influenced by existing research. As the design was developed using internal statistical experts and academic resources, a visit to the facility that held the AM equipment was arranged. The factory visit provided the opportunity to develop procedures and practices that would be repeatable for the experiment as well as build the relationships with facility staff needed to work together long-distance.

Upon initial proposal of the experimental design, it was quickly discovered that many of the most desirable input factors in laser PBF were unavailable for modification due to manufacturer limitations on the equipment. Instead, a set of design parameters was adopted that still provides significant value for the group operating the AM equipment. That set formed the basis for a large screening experiment to remove certain factors from consideration in future experiments. As facility staff build confidence in their operations using this introductory experiment, they will be able to use the processes developed to run additional experiments and improve their equipment performance.

4.4 Key Performance Metrics

While there may be academic and educational gains from studies such as this thesis, the project has an intent of solving standing business problems for the host company, Raytheon. The success of this project will be measured against the following metrics:

- Improvement of MRL for specific metals AM processes and materials;
- Development of an AM characterization/qualification process that can be adapted to other materials and businesses within Raytheon or outside of Raytheon;
- Implementation of a pilot component of that process, a designed experiment for material and equipment qualification and
- Demonstrating the effectiveness of experimental design and other data-driven methods of qualification.

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Chapter 5

Additive Manufacturing Qualification Process

Leadership at Raytheon Missile Systems expects the manufacturing and engineering groups to take an active role in qualifying metal AM for use in flight-ready parts. As mentioned earlier, a robust qualification plan is required as a guide to help teams decide on the best actions and policies as a specific metal AM material and technology is selected. This section will describe the proposed qualification process, separated out into its three constituent phases. This section will also contain a summary of the initial qualification process implementation in a pilot program at Raytheon as part of this thesis, one with a component intended to be flight-ready in only a few years and few other manufacturing techniques capable of delivering the same cost-performance ratio as additive manufacturing.

5.1 Overview of Qualification Process Map

While Raytheon has an urgent need to develop a metal AM qualification process, the process proposed in this chapter is not the first. ASTM, NIST and NASA are all in various stages of work on different qualification process documents for metal AM as used in aerospace applications and other industries. The most complete plan published so far is ASTM's standard for using powder bed fusion (PBF) to produce components of the Ti6Al4V alloy (ASTM Standard F2924, 2014). However, this process design should not be applied directly to qualification of Inconel formulations and AlSi10Mg, two of the most critical metal alloys for aerospace applications. AWS has given the AM community notice that drafts of a standard are in progress, but no official document has been published (AWS D20, 2016). In addition, these plans do not address many of the operational issues faced by specific companies when trying to implement an AM qualification process.

While the qualification component of this thesis project borrows some of the structure of plans like F2924, it was heavily influenced by incorporating existing work completed within Raytheon SAS, as mentioned in the earlier Methodology section. Integrating a qualification process with existing program management goals was critical to both acceptance of the process and adequate funding to complete the qualification work. To address some of the needs for project planning that are left out of the standard organization documents, the completed qualification process also includes provisions for cost and scheduling that, while more specific to an individual project, should be easily adapted to other applications. The following sections will cover the major phases of this process and explain the key tasks required to complete each phase.

The major components of a proposed qualification process for metals AM, or other materials if adapted, in an aerospace flight-ready application can be separated into three major phases: process capability, design feasibility and integration, verification and validation (IV&V). These phases each have additional subdivisions that can be seen Figure 5-1 and will be described in greater detail.

Figure 5-1 indicates that each phase should be completed top to bottom, and while this is a best-case recommendation, there will likely be feedback loops that modify previous conclusions as the process evolves. There is some required sequencing for a few of the steps in each phase (which will be mentioned later), but on the whole, activities in each phase can be completed roughly in parallel; the first is process capability.

5.2 Process Capability

This phase of qualifying a metal AM process for aerospace applications ensures that the material and manufacturing equipment itself meets a set of basic requirements that any

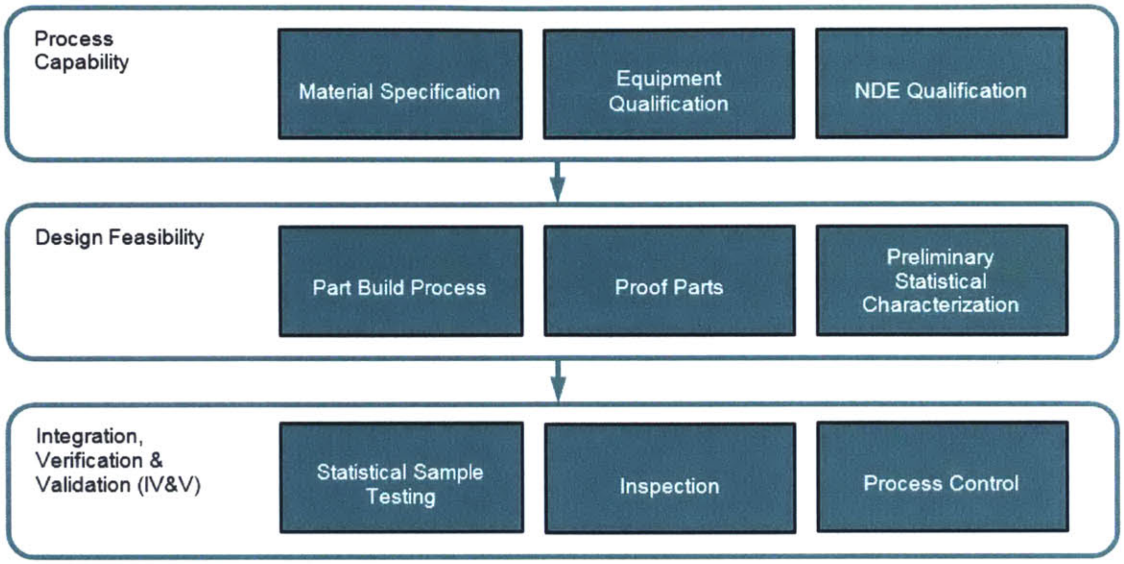

Figure 5-1: Outline of Metal AM Qualification Business Process

technology supplying flight-ready parts must meet. This includes assessing the quality of the materials to be used in the AM equipment and also understanding the technical capabilities of the AM equipment itself. This phase also requires testing the readiness of measurement techniques used to assess whether or not the material and manufacturing technology are ready for the desired application.

5.2.1 Material Characterization

Understanding the nature of the raw material used for metal AM is fundamental to qualifying the overall manufacturing process as well as generating uniform products. To achieve sufficient knowledge about how the raw materials behave, three aspects will be highly specified: the specifications and composition of the material used, the procedures for recovering unused material and the procedures for using metal AM equipment with multiple types of material.

Material Specification Raw material for AM processes is expected to go through two stages of characterization. Raw material suppliers will be required to provide some level of certification of their quality control process. Testing of materials after receipt will also be required to ensure that materials match the performance of batches used in the past. The

Table 5.1: Standard Guide for Characterizing Properties of Metal Powders Used for Additive Manufacturing Processes (ASTM Standard F3049, 2014)

Property	**Test Standard**	**Quantity**
Particle Size and Distribution	ASTM B761 or ASTM B822	25g
Particle Shape Analysis	ASTM F1877 Section 11.1 and Appendix X2	1g
Chemical Composition Analysis	OES: Green Compact or ICP	50g
Flow Rate	ASTM B964 or ASTM B213	150g
Tap Density	ASTM B527	50g
Apparent Density	ASTM B212	250g

following three sections cover these stages.

Testing Parameters Powder characterization and testing are required to evaluate the chemical and physical properties of metal powders, both as individual particles and in bulk form. This is important to ensure adequate control of powder production processes, required properties for parts manufacturing are met and produce consistent components with predictable properties. Powder used for the manufacture of hardware must be either certified to meet the requirements of industry standards or if a standard does not exist, an internal standard must be developed.

Powder characterization should follow the guidelines specified in ASTM F3049, Standard Guide for Characterization of Metal Powders Used for Additive Manufacturing Processes (Table 5.1). For metal powder used in additive manufacturing processes, the guide provides purchasers, vendors, or producers with a reference for existing standards that may be used to characterize properties of various types of metal powders.

Testing requirements will apply to new, unused powder and to each reuse of existing powder after any physical separation methods are complete. A robust testing apparatus with a good quality control process or an active relationship with a nearby testing contractor will be required to maintain desired testing frequency.

Physical and Chemical Composition Requirements Adequate raw material characterization should include a statement of certification from the material manufacturer indicating the tests completed on delivered product. A report shall be provided for each material batch received from the manufacturer, with the Standard Certificate of Analysis or

Table 5.2: Certificate of Analysis Minimum Requirements, Industry Derived (AP&C, 2016)

Raw Material Property
Raw Material Manufacturer
Raw Material Manufacturing Location
C of A Date of Issue
Material Trade Name
Manufacturers Material Designation
Date of Material Manufacture
Nominal Grain Size
10% Diameter
50% Diameter
90% Diameter
Chemical Composition
Signature Authority and Date

Certificate of Conformance report retained for not less than 10 years. Table 5.2 indicates the minimum required information that should be included in the report. If possible, manufactured test coupons and parts should be comprised of a single batch of material, or at least be traceable to the original material documentation for the batch(es) used.

Material Recycle Guidelines One of the advantages of AM techniques is that a minimum of material in excess of the built component is used, as compared to subtractive techniques that require substantial rework before reusing scrapped material. In order to accomplish this goal with a powder fusion process, unused material powder that has been in the build chamber along with fused powder must be recycled. Current procedures within Raytheon reuse the powder for a functionally infinite number of cycles, with an estimated loss of 5% mass in each recycle due to exclusion of agglomerated particles through sieving.

Sieving is required to remove agglomerated particles generated by the powder exposure to process conditions; therefore, experimentation should be executed to understand the effect of multiple reuse cycles on the performance characteristics. Current material standards do not provide a limit on reuse cycles, as it varies by material and process, but testing can be completed on both the resultant bulk material properties listed in Table 5.1 and on basic mechanical properties of AM test builds completed with powder with varying numbers of reuse cycles. From those experiments, process engineers should set guidelines on the number of times that a quantity of powder may be reused and possibly what fraction of reused powder is allowable when combined with virgin powder. Testing must demonstrate that material properties remain consistent through a certain number of powder reuses and provide a confidence interval estimate on the material properties throughout the reuse cycles. While Section 6.2 describes a method for future experimentation on recycled powder testing, analyzing the differences between new and recycled powder is outside the scope of this thesis. Please review Section 2.3.2 for current research on reuse cycles for a variety of materials applicable to aerospace designs.

Material Changeover Requirements A value proposition of AM equipment is the ability of that equipment to change from one build material to another, which increases flexibility and capability without incurring additional capital cost. However, there are substantial recurring costs associated with material changeover in PBF equipment. Process engineers within Raytheon currently estimate that a material changeover requires one week of equipment downtime, as well as 80 hours of touch labor that consists of nearly-continuous exposure to hazardous and reactive metal dusts. Thorough cleaning is often still incomplete even with best effort and a certain amount of built material is discarded after changeover to remove residual contaminants from the build chamber. In addition, nondestructive and destructive analytical equipment has limited capability to distinguish between the compositions of similar alloys. Any residual contamination of dissimilar alloy groups can compromise the quality of the AM build. Based on this information, most process planners should assume that all operations will be completed on AM equipment that is dedicated to a single material.

If capital availability or other factors dictate that an AM process must be qualified in an environment where material changeover occurs, process engineers must develop a thorough procedure for removing as much residual material as possible from the equipment. They must also suggest a testing procedure or sacrificial production schedule that allows for any residual build contamination to be mitigated or discarded before AM qualification activities continue. The testing in this thesis was completed on a single material, and therefore material changeover is outside the scope of this document.

5.2.2 Equipment Qualification

The next step of qualification required after characterizing raw material feedstock for the selected AM process is defining the operational characteristics of the specific manufacturing equipment. At this point in the technology lifecycle, manufacturing procedures, performance and results are typically unique between manufacturers and models of equipment. The following process outlined below[1] should provide sufficient information to ensure consistency of part production and provide a preliminary basis for equipment capability.

Operating Procedures Complete documentation of all equipment operating procedures is critical to repeatability of AM production. The process engineers responsible for the qualification project should work with experienced operators, manufacturer representatives and business SMEs to develop step-by-step procedures that cover the following stages of AM production:

Preventative Maintenance The qualification team will work with the equipment manufacturer and any available maintenance documentation to develop a plan for preventative maintenance (PM), with lists of consumable or repairable equipment marked with their maintenance intervals. PM plans should be in the form of a checklist with a requirement to mark dates of completion.

Initial Equipment Inspection Before any raw material is added to the manufacturing equipment, all components that directly interact with raw or finished materials will be inspected for damage and functionality. Any additional maintenance required as a result of observations will be completed. Engineers or operators will confirm that all process utilities are available and supplied to the manufacturing equipment. Any applicable ventilation or inert gas delivery systems will be tested for functionality. Any used material recovery/recycle equipment should also be inspected. Workspaces inside and around the AM equipment should be clean and clear of all extraneous equipment or parts, excluding organized sets of tools required to run the AM process.

[1]There are elements of this process that are specific to PBF AM. Changes will be required to create general procedures or procedures for other types of AM equipment.

CAD File Preparation and Support Structure Development Qualification tasks will be completed by building specific designs of AM parts and test coupons to use in a variety of mechanical and physical tests. Each design will be specified by a CAD file with clear version information. When the CAD file is received, it must be stored in a secure file system with version control to insure the integrity of the file. The CAD file will be transferred to a slicing program where engineers will create the slicing plan for each build layer and add any needed support structures. As more experience is developed around building support structures, operators should begin mapping that experience into a decision tree or set of general rules and best practices to maintain process knowledge. Support structure creation may not follow a rigid algorithm but should be documented similarly to any existing DFAM guidelines.

Raw Material Loading After raw material has been processed according to Section 5.2.1 above, the machine operator should select a batch of raw material. That raw material must be traceable to original material test results or the supplier's certificate of compliance certifying that such tests had been completed. If the material has been recycled, there must be a log that includes the number of reuse cycles experienced so far as well as the date of first use. The operator should follow safe procedures for moving the materials to the AM equipment with a minimum of lifting. If the material is not already in a container/vessel that can be directly connected to the AM equipment, it should be moved to a safe working space (with inert atmosphere, if required) to transfer to the process equipment. If materials are powder or liquid, the operator should follow a repeatable procedure for agitating the mixture to ensure consistency throughout the material volume.

Build Initialization and Monitoring AM builds are initiated through a computer process control system. Operators should load the version-controlled files defined by the slicing procedure onto encrypted removable media and transfer to the process control computer attached to the AM equipment. Network connections with adequate security can also be used to complete this transfer. Operators will select the file for the specific qualification build and initialize the AM equipment.

Build initialization is a critical portion of the AM build process and operators should develop a schedule for monitoring the early build, allowing them to intervene or cancel the build before a substantial amount of process time has passed. After initialization, operators should return on a periodic basis to monitor build progress or interruptions, or use a visual Andon system to monitor the health of the AM equipment. Most process equipment that has a human operator can be equipped with a green-yellow-red light stack to indicate operating, paused and fault status, respectively.

Build Completion and Finished Part Removal When a qualification build has been completed (successfully or not), it must be safely removed from the process chamber. After any required cooldown period, the finished build along with any excess working material must be moved to a safe working environment. Any excess raw material should be removed from the completed build without damaging the appearance or integrity of the completed build. When the build and build platform are nearly clean, they should be removed from the build area and transported to begin a process to fully remove any unwanted material in an inert environment. The build and build platform should then be transferred to machining equipment that is capable of separating the two without damage to the build, and with minimal damage to the reusable build platform. This equipment should be selected based on speed, dimensional accuracy, and material used for the AM build.

Post-Build Maintenance and Material Recycle Before another AM build can be initiated, operators and engineers must assess the current state of the AM equipment and its build chamber. A cleaning procedure should be implemented to return the AM equipment to the previous state for each run to ensure repeatability. As part of this procedure, all excess used raw material should be removed from the build chamber to be reused according to the recycle plan previously developed. This will likely require use of auxiliary equipment like sieves or filters that also require documented operating procedures.

Post-Process Thermal and Machining Work After separation from the build platform, the AM build may require additional thermal and mechanical post-processing work. The procedures for these operations will likely be well-defined by use with existing qualified manufacturing processes, but the documentation should still be associated with the AM process for later analysis and traceability. These procedures must be defined and standardized at this stage of testing so that they can be included as part of the part build process.

Exception Handling There are two broad classes of possible exceptions during the AM build process: interruptions that allow work to resume and interruptions that cause total failure of the build to complete. In both cases, operators should document the time, progress of the build and cause of the failure (if known) to allow inspection at a later time. If the exception is of a class where the build can be restarted, there should be specific restart procedures that indicate computer commands and physical steps required to safely restart production on a part while ensuring quality. If the build cannot be restarted, a similar procedure should exist to make the AM equipment safe to work with and to break down the aborted build in order to start a new build from the beginning.

Calibration Procedures There are many different factors that affect the quality of AM builds, some of which must remain under tight control. Again, ensuring repeatability requires calibration procedures to ensure that the equipment's baseline performance remains the same over time. The process engineers responsible for the qualification project should base calibration procedures and schedules on manufacturer documentation and recommendations, but should continue to add calibration procedures throughout the qualification process as needed. At a minimum, calibration procedures should contain certification activities for the following parameters, their sensors and controls (applicable mostly to laser PBF):

- Laser power output: average thermal energy applied to surface at a given rate
- Laser beam profile: how power is applied in each pulse (square wave, Gaussian, *etc.*)
- Laser focal length: distance of focus from the laser focusing lens
- Laser pattern scan rate: laser beam rate of motion through the X-Y plane
- Laser spot size, shape and aiming point (tested at center, edges, corners)

- Laser power on rise times and off fall times
- Build platform positioning accuracy (X, Y, Z)

Auxiliary equipment to maintain inert atmospheres or generate laser light should also have calibration procedures. Calibration intervals should be approved, documented and followed throughout all stages of the qualification process.

Baseline Mechanical Property Characterization The core validator of AM equipment qualification is the test of several mechanical properties of components produced with the AM equipment under a fixed set of process conditions. The tests will be used to select the best possible parameter settings for continued production and to understand the effect of various process choices on the performance of manufactured components. In the process capability phase, the properties should be tested on at least two batches of raw material and two different AM build efforts, with multiple samples per build.

Appendix A provides a summary of the tests that are required to sufficiently characterize performance for basic aerospace applications while minimizing the costs of testing at this phase.

Dimensional Precision Assessment As the procedures and parameters for optimal AM mechanical properties are developed, it remains necessary to ensure at an early stage that any selected build recipes will produce parts with the desired dimensions in critical areas. While this stage in qualification is too early to have any identified critical dimensions based on future applications of the AM process, a demonstration part can be used to test the AM equipment's capability to resolve small detailed features of a variety of shapes and sizes. This is referred to at Raytheon as an "obstacle" part due to its complex nature and the innate difficulty in finding build parameters that will generate all features equally well. The recommended form of the obstacle part is a test part and design proposed by NIST. Figure 5-2 is a rendering of the current state-of-the-art in test artifacts (sometimes referred to as obstacle parts) as suggested by NIST researchers.

As mechanical testing coupons are created with certain AM build recipes, the obstacle part should also be built at the same time. Specific features of the obstacle part can be

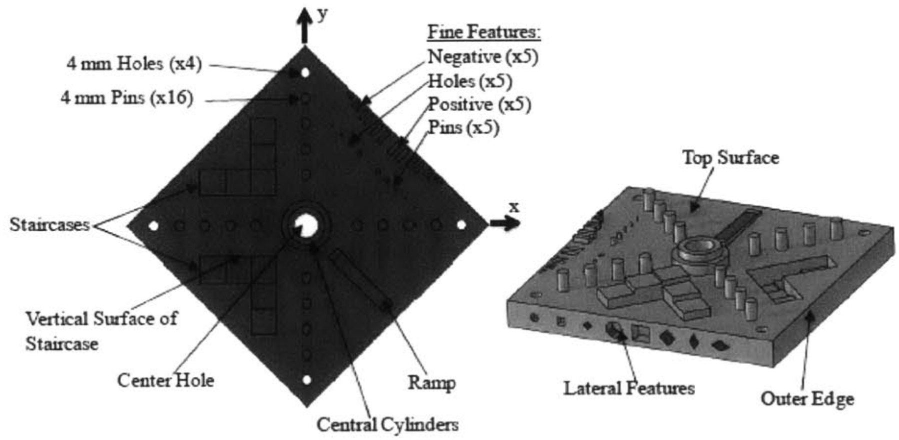

Figure 5-2: Test part suggested for dimensional precision testing, in top view and offset view (Moylan et al., 2014). Artifact is 100 mm square and 10 mm thick, with protrusions extending 7mm.

selected for further analysis with accurate measuring tools. An initial recommendation is to select features that are similar to those needed by upcoming projects. The obstacle part also provides an opportunity to observe surface roughness on multiple orientations of the material, including the lateral features in the right side of Figure 5-2. In addition, computed tomography (CT) scans can be used to analyze internal dimensions if qualification requires intricate internal structures.

Process Recipe Optimization As a qualified process is developed, there is a high probability that the original process parameters specified by process engineers or equipment manufacturers will not be optimal for producing the results desired by the sponsoring program. In order to accommodate the changes needed to approach optimal process behavior, the qualification process schedule can include several cycles of AM builds and subsequent testing. The parameters tested in this phase of the qualification plan are listed below and are also in Appendix A.

NDE will be used to assess the overall quality of the build and attempt to detect any macroscopic defects caused by the currently developed build process. The processes recommended for use are in the list below:

- Visual inspection including precision measurement of critical dimensions

- X-Ray – two-dimensional planar imaging using X-ray EM radiation (ASTM E1742)
- Dye Penetrant – application of a penetrating dye to the surface of a test specimen in order to demonstrate defects in the surface of the specimen (ASTM E1417)

The mechanical properties below are simple to test and critical to room-temperature performance of the material. The ability of the current iteration of a process recipe to meet basic mechanical requirements of the program will be considered gating to move to the next phase of the qualification process.

- Ultimate Tensile Strength
- Tensile Yield Strength
- Young's Modulus
- Ultimate Strain
- Reduction in Area %

Physical properties of the broken tensile bars, listed below, will be analyzed to determine if microstructure is similar to other material production techniques. Samples can also be used to verify composition of the AM-produced materials does not change from pre- to post-build.

- Density
- Composition
- Tensile Fracture Scanning Electron Microscopy (SEM) Documentation
- Microstructure

If the current process recipe does not meet some or all of the specified parameters above, several process settings on the AM equipment have been identified for modification to meet specifications. This thesis proposes using screening experimental designs to develop understanding of the properties above. Chapter 6 is dedicated to an explanation of experimental method as well as preliminary results completed on testing at Raytheon. Other studies using response-curve experimental design could provide initial information on what recipe parameters affect process output. If process performance dictates changing multiple variables at the same time, engineers should design another experiment to reduce the number of trials needed and further understand the relationship between the process recipe and output. Potential recipe parameters that can be modified in common metal AM equipment have been identified below:

- Laser Power: average thermal energy applied to surface at a given rate
- Scan Speed: laser beam rate of motion through the X-Y plane
- Scan Spacing: distance between successive scan patterns in X-Y plane
- Beam Diameter: width of laser beam focal point
- Dose Factor: amount of powder applied to surface of build platform in excess of desired layer thickness
- Coater Blade Height: setting for layer thickness

Post-Processing Optimization Several options are available to improve or modify material performance characteristics after AM builds are complete. One current recommendation for metals AM production is to complete a cycle of hot isostatic pressing (HIP) for all built components and test samples before assembly or test. HIP can be used in critical-path components in the aerospace industry to reduce microporosity and homogenize crystal structure (Carter et al., 2014).

Additional post-processing techniques include techniques that:

- improve surface finish (*e.g.*, sandblasting or polishing).
- modify surface mechanical properties (*e.g.*, peening).
- add additional materials (*e.g.*, thin film deposition or plating).
- complete the manufacture of net-shape parts (*e.g.*, machining).

If the program team desires, trade studies can be done between different post-processing steps to understand the effect of those techniques on many of the mechanical and physical characteristics for an AM build. Otherwise, program leadership can accept a recommended post-processing procedure developed by subject matter experts (SMEs) in the areas of machining and surface finish.

Material Analysis In addition to basic mechanical property tests, equipment qualification should include tests on the microstructure of samples and fracture surfaces of tensile test specimens. While these tests do not have specific associated requirements, the AM build process must demonstrate that it is capable of producing microstructures with similar behavior as qualified alternative production methods. In addition, fracture analysis should show that the AM process does not introduce new failure modes that are less predictable, or failure modes that cannot be mitigated by known post-processing technology or engineering design practices.

Equipment Process Control During initial process qualification, process engineers should take the opportunity to develop an understanding of the available process measurements available on the AM equipment and any ancillary equipment, like material handing or post-processing equipment. For laser melting AM techniques, common measurable parameters include melt pool diameter and build chamber temperature (if equipment is designed to heat chamber above ambient temperature). Analyzing the data of these parameters while working on initial testing is critical to setting up effective statistical process control later based on process variability. In addition, engineers need to verify that variability is low or controllable in the early phases of qualification to reduce the probability that process variability can invalidate testing results.

5.2.3 NDE Qualification

A key component of efficient AM production in small quantities is a reliance on nondestructive testing for most flaw detection. Production rates are low enough that destructive testing based on a statistical sample is insufficient to establish with any certainty that parts are produced with no defects—all parts will need NDE. In addition, AM's unique manufacturing method of fusing many small layers provides particular challenges for existing NDE methods.

Capability Assessment The first step of qualifying NDE techniques with AM is completing a basic capability test with candidate testing technologies. The preferred technologies for defect identification in manufactured metal products are X-ray and CT. The size of defects that occur in AM can be very small: defects generated by experimenting with laser parameters on titanium alloy ranged from 30-100 microns (Gong et al., 2014). CT equipment was capable of reaching resolutions of $1 - 15\ \mu m^3$ per voxel (the three-dimensional analog of a pixel) on 80 x 80 mm samples of carbon fiber reinforced plastic (Bull et al., 2013). Engineers should create sample builds that would represent defects that might be introduced unintentionally during normal production:

- Voids can be simulated by creating a layer or set of layers that is nearly solid except for a small area. The small area of missing material can be varied to attempt detection of different size defects.

- Layer separation can be controlled to a specific location in the part by purposefully pausing the machine and allowing the partially constructed specimen to cool. On restarting the fusing process, the layers of different temperatures should fail to fuse together normally.

- Incomplete layer fusing can be tested by either decreasing laser power for a series of layers or increasing the powder dose rate for several layers. In either case the laser pattern should be unable to completely fuse all of the powder deposited on those layers.

These builds can be tested with existing X-ray and CT techniques to verify that some detection of defects occurs. Through initial testing with metals AM, engineers within Raytheon have found that cracks in AM parts can often be aligned in a way that prevents detection with X-ray. It is likely that NDE will rely upon CT alone for visualizing defects inside the volume of AM parts, as CT avoids issues of aligning planar X-rays with layer defects.

Threshold Flaw Size When capable NDE methods are identified, engineers should develop sample builds that contain a range of different defects aligned with different axes of the build. This includes voids, layer separation, and incompletely fused layers. The selected NDE method should be used on a "perfect" build as well as a flawed build in order to ascertain what size and structure of defects can be reliably detected. Information from this testing must be compared against program requirements to provide the estimated "knock-down" from undetectable defects. Testing may require several iterations to refine a method that is capable of certifying a component to a small enough defect size threshold.

Probability of Detection Once an approximate defect size threshold is set for a specific NDE method, engineers should develop a sample build with defects of that average size distributed throughout the build volume. Defect size should be distributed normally about the mean defect size threshold. Based on the number of defects built into the sample and the number detected by NDE technicians, this test should provide an estimated probability of detection for the selected NDE method.

5.3 Design Feasibility

The next phase of AM process qualification should introduce the part design in an effort to combine design for additive manufacturing (DFAM) reviews with the qualification efforts. This portion of the qualification plan is intended to test the producibility of the desired part, develop a fixed plan for manufacturing the component and complete the characterization of all material performance in order to meet the design characteristics for the sponsoring program. This early "production" stage is intended to mitigate institutional risk for funding new manufacturing technologies by providing early stakeholder access to the potential benefits of (and issues with) the new manufacturing technology.

5.3.1 Part Classification

Part classification is used to assign appropriate levels of process control and qualification to AM parts. Parts are categorized alphabetically, A through C, by consequence of failure,

Table 5.3: Classification criteria of welded parts (AWS D17.1, 2010)

Classification	Consequence of Failure	Failure Description
Class A	Critical	Failure could cause loss of life or loss of a vital capability
Class B	Semi-Critical	Failure would reduce operating efficiency affecting product performance
Class C	Non-Critical	Failure would not affect safety, nor vital performance capability

with Class A being the highest critical, Class B being semi-critical, and Class C classified as non-critical. All AM parts shall be classified according to Table 5.3. The classification is based on the similarity of metal AM processes to welding material behavior.

5.3.2 Part Build Process

In order to replicate process conditions over multiple days and changes in operators, each part to be produced should have a documented build process. The entries in this section detail each of the process components that are needed to replicate identical parts over multiple attempts and an extended period of time.

Build Plan The build plan of a qualified AM part consists of the following information critical to repeating the process conditions needed for reliable production:

- Material to be used for the build, along with documentation of the qualification procedures used for the material
- Material handling properties, such as number of reuses
- All files needed for an accurate build, marked with dimensions and provided with multiple model views to describe the part.
  - versioned CAD file
  - versioned STL file including support structures
  - versioned AM equipment configuration file

- Part classification (A, B, non-structural) with justification
- List of witness tests required for each part
- List of inspections needed for each part and associated critical criteria
- Critical dimensions and measurements needed to verify conformance
- Build platform material and dimensions with tolerances
- Witness test coupons and arrangements to accompany the component

The build plan should include all procedures required from the operating procedures portion of Section 5.2.2 as well as the following procedures:

- Support material removal
- Method(s) of thermal post-treatment
- Method(s) of mechanical post-treatment
- Packaging and handling instructions

Build Recipe The build recipe for an AM part should consist of all of the configurable process parameters available within the AM equipment. If recipes are handled within the AM equipment as a single settings file, the file should be accurately versioned and hashed to allow verification that settings are the same from run to run.

Configuration Management A configuration management system must be employed to track all of the information in the sections above and correlate those settings to an individually identifiable and serialized AM part.

5.3.3 Proof Parts

Managing program stakeholder buy-in during the AM qualification process is critical. The equipment undergoing trial is high-cost and there are few examples of production components made with the technology. A way to mitigate concern and risk during the qualification process is to start creating examples of the final part early on in the process. In this way, the team responsible for qualifying the AM technology can demonstrate their current capability while providing some immediate utility for the stakeholders funding the qualification. Program managers can run meetings and demos with a prototype part, and some preliminary testing results allow them to adjust contingency plans, run another cycle of design or switch to an alternate manufacturing method if, at this stage, AM is shown to be unsuited for production of the final component.

Dimensional Accuracy Assessment All parts should undergo physical measurements and be compared to the technical drawings (the as-designed dimensions). Standard methods such as measuring with a coordinate-measuring machine (CMM) can be used to execute this testing rapidly and with high precision. Any internal structures that cannot be revealed without destructive means may be able to be measured using CT scanning. When one produced part is identified as sufficiently compliant with as-designed dimensions, this part should be maintained as a standard to speed tool calibration for later testing.

Nondestructive Material Analysis Any part that has a structural role in the overall design of the flight vehicle (Class A or Class B) should have a comprehensive suite of non-destructive evaluation designed to discover surface and volumetric defects. These techniques could include, but are not limited to:

- X-Ray – two-dimensional planar imaging using X-ray EM radiation (ASTM E1742)
- CT – computed tomography, three-dimensional imaging using the combined result of many individual planar images (ASTM E1441)
- Dye Penetrant – application of a penetrating dye to the surface of a test specimen in order to demonstrate defects in the surface of the specimen (ASTM E1417)
- Eddy current – an electromagnetic test that uses field induction in metals to detect defects below the surface of the test specimen (ASTM E571)
- Ultrasonic – a test that uses the reflection of high-frequency sound waves to detect the presence of internal defects (ASTM E2375)

Standards for all of the techniques above are currently being developed in ASTM WK47031 for adaptation to metal AM. Any discovered flaws should be compared to the critical flaw size selected in the procedures from Section 5.2.3. Any detected defects greater in scale than the desired threshold will require mitigation actions for the AM equipment operation and/or modifications to the component design to handle the new defect characteristics.

Destructive Material Analysis Most of the samples created in the Proof Parts phase should be evaluated only with non-destructive methods in order to make prototypes available for any functional or integration testing that the program team might desire. However, the unmonitored nature of metal AM production at this stage requires at least one representative sample be destroyed to obtain test coupons for tensile/compression strength, fatigue and fracture testing. A "proof test" apparatus can be designed to test the component with similar loads that it would experience in its use scenario. This may require combined load states of pressure, applied external forces, extreme temperature ranges, moisture and salinity. If the proof test fails to destroy the component, it should be analyzed for any plastic deformation.

After proof testing is complete, excised test coupons from the proof part and the witness coupons built with that part should be tested for a standard set of mechanical properties, repeated again here for convenience:

- Ultimate Tensile Strength
- Tensile Yield Strength
- Young's Modulus
- Ultimate Strain
- Reduction in Area %
- Hardness

Material composition analysis should be completed on the test remnants to verify that the AM build process has not altered the composition of the component. Researchers have determined that powder reuse can cause small but cumulative changes in the composition of the metal powder (Tang et al., 2015).

5.3.4 Preliminary Statistical Characterization

This section covers the wide extent of testing needed to assure that the mechanical, thermal, physical and structural properties of metals made with AM will meet standards set by bodies like the ASTM and cataloged in Metallic Materials Properties Development and Standardization (MMPDS). Parts for flight-ready hardware are expected to meet A-Basis requirements and so will require at least 100 individual samples for most critical mechanical properties[2]. Some examples of groups of testing required are in the list below:

- Physical Properties: density, surface emissivity, composition, microstructure and corrosion resistance
- Mechanical Properties: tension and compression tests, fatigue, fracture toughness, crack growth, bearing, bend, hardness, Poisson's ratio and weld tests (if needed)
- Thermal Properties: thermal conductivity, thermal diffusivity, specific heat

All the tests included above would be considered static property tests except for dynamic elements of existing ASTM procedures. As program requirements develop further and flight test requirements become known, this list of tests may also include dynamic testing, such as strength and strain testing under different temperature ramping rates. These requirements will flow down from program leadership.

The simplicity of this section is deceptive. Some of the tests listed above are extremely time-consuming and costly, and the amount of material required to supply test coupons for all of the above tests at 100 samples per test is nontrivial. Indeed, cost estimates for completing this section of the qualification range from the high $100,000s to low $millions. It is imperative that the qualifications steps that lead up to this point have ensured that the metal AM process can meet the desired performance and do so over many different repetitions.

[2]To meet A-basis level of characterization, properties shall be maintained at or above the 99% probability of no failure, with 95% confidence.

5.4 Integration, Verification & Validation (IV&V)

The final stage of AM process qualification comes just before an expected release to production in the main manufacturing facilities. This phase is dedicated to development of procedures and tests that ensure and verify a high quality product throughout the end of the development process. In addition, any effort completed in this phase can later be transferred to production resources, hopefully saving time and resources during the critical new product introduction phase of the flight vehicle.

5.4.1 Statistical Sample Testing

The production phase will have periodic sample testing and evaluation to ensure process controls and manufacturing conditions are being kept constant. Each type of production part is required to go through destructive evaluation on a specified interval of part production. The sacrificial part shall be excised to produce test specimens for the test standards shown in Appendix A. Tests results should be compared to test results obtained from the destructive evaluation completed as instructed in Section 5.3.3. Similar testing should be conducted when the manufacturing process is interrupted, *e.g.*, for calibration or maintenance procedures.

5.4.2 Inspection

During initial runs of parts on AM equipment still in the qualification stage, all parts produced should undergo detailed inspection with the four methods listed in this section.

Dimensional Analysis All critical dimensions dictated by the build plan in Section 5.3.2 should be tested to verify that they conform. CT should be used to confirm internal dimensions.

Visual Inspection All parts and witness specimens shall be visually inspected for defects such as missing material (voids), notches or chips caused by removal of support structure, dimensional defects or warping, etc. Articles shall be inspected for the defect criteria listed in Appendix B.

NDE All parts should again be evaluated on a surface and internal volume basis for defects that could potentially affect integration and use of the component, using the procedures listed in Section 5.3.3.

Proof Testing Testing here is also similar to the structural proof testing described in Section 5.3.3, with the changed explicit goal that all components are undamaged and usable after the proof test cycle. At this stage in qualification, the proof test should mimic all nominal load conditions that the component could exhibit and also send the component through multiple cycles of the test to check for any low-cycle fatigue issues.

5.4.3 Process Control

Production statistical process control (SPC) is based on the methodology of predicting and mitigating process variability by collecting measurements of critical process data and analyzing them with statistical methods. SPC shall be based on the following data sets at a minimum:

- Shape and statistical distribution of powder particle size
- Tensile and fatigue properties
- Any optical measurement data (melt pool, powder spreading, etc.)

Engineers should monitor the data collected for adherence to normality assumptions and then set up control charts that can alert maintainers when a process is out of specification. This stage of AM qualification does not represent full-scale production, but work done at this time can be easily transferred to the SPC required when a process goes into normal factory production.

5.5 Assessment of MRL

As mentioned in Section 3.4, one of the major motivations for this project has been to identify uses for metals AM within Raytheon businesses and characterize the manufacturing readiness of the technology to produce new components. To reiterate, three major use cases that are proposed in this thesis are metal airframe components with internal structures and topology optimization, robust tooling equipment and resupply/replacement of low-volume, low-criticality maintenance items.

Addressing the first use case requires summarizing some of Raytheon's most recent efforts in the area of metal AM qualification for flight-ready components. Most of this chapter, Chapter 5, was developed in partnership with a new undisclosed program at Raytheon that seeks to use metal AM for both ease of manufacturing and previously-impossible topology optimization. The qualification process previously described in detail is already in a "pilot program status". A manufacturing site for the initial prototypes has been identified, and the program requested and accepted a set of time and schedule estimates to carry the project throughout the 3 phases shown in Figure 5-1 on page 53. In addition, this manufacturing facility has delivered several representative samples of the material that will be used on this project, and initial testing on those samples has demonstrated mechanical properties that meet or exceed the estimates of the material manufacturer. This is a vast improvement over the readiness of the metal AM production process when the project started in June, when the AM equipment had only just been installed in May.

Based on the guidelines in Table 2.1, the original MRL of the metal AM production process for the flight-ready component application was approximately 3, or possibly 4: new equipment had been installed but generally ran untested or proven, and no concept of a production-relevant environment existed. At the conclusion of this project, the combination of several months of focused testing (including a statistically-designed experiment) and the development of an approved AM qualification process can qualify Raytheon's metal AM MRL as 5, for the purposes of building flight-ready prototypes with topology optimization as a primary feature. Given the need, the AM facility staff could begin following the qualification process at the time of this publication and produce prototypes ready for testing in several months hence.

It could also be argued that based on the Department of Defense (DoD) system development process presented in Figure 2-1 that the MRL for this specific application is actually at level 6. The aforementioned program, as of December 2015, has proceeded through the product design review (PDR) phase with metal AM components as a key technology in order to meet desired cost and schedule targets. PDR aligns with MRL 6 based on the mapping between system design and raw MRL value. While the AM qualification still has a lot of work yet to complete before fielding any AM-built prototypes, the confidence that Raytheon program leadership puts in the state of metal AM is due to the progress that the Manufacturing and Test Engineering group has accomplished in only a few months.

The second application, tooling equipment, has not seen much direct development over the course of the project. With the limited staff and equipment resources for metal AM directed mostly at the first application, no tooling engineers have requested or received time on the AM equipment to create any prototype tools. Several of the tooling applications require additional research into coating and finishing technologies to be able to use AM-produced tools. Based on the MRA criteria, the MRL for the tooling application is no greater than 3 at this stage. Without an identified design and time spent attempting to create a new tooling prototype, the MRL cannot be any higher. However, the work that has been completed for the first application is directly transferable. One of the advantages of AM is the short wait time between design concept and first prototype as a result of AM's "digital" nature. This has the effect of accelerating the transition between MRLs 2/3 and 4, or beyond. The barrier between successive levels is considerably lower for this manufacturing technology than for other legacy methods.

The final application proposed, replacement components made with AM, has a considerable organizational battle to fight for acceptance. The quality expectations in DoD aerospace applications are extremely high, and qualification of a new manufacturing technology is taken seriously. By the time a component or system makes it to full-scale production year-over-year, all major qualification activities have been completed. Any time a significant change is made to the design of an established system, a "delta" qualification is undertaken in order to assess the scale of the change's impact on the performance of the system (to compare to known system requirements). This occurs every time a bolt's supplier goes out of business or a bracket supplier consolidates their inventory and no longer produces the part that Raytheon originally procured.

At first glance, this seems like a perfect opportunity to replace a component that has gone out of service with one created using AM. However, the issue arises in the scope of the delta qualification required. When comparing a replacement component made with the same manufacturing technology, it can be fairly straightforward to compare material and manufacturing specifications provided by the supplier to understand the baseline changes. This would be followed by completion of functional testing to ascertain that the new component is still suitable for its intended application. For AM-produced components, the delta qualification would be much more substantial. Without a significant body of past experience or products that have used AM, it is difficult to be sure that the AM component will behave the same way in the desired application. As a result, program managers and quality engineers will demand a level of testing that looks more like a full component qualification (one represented in this thesis) rather than the delta qualification.

The disparity between a standard delta qualification and one completed for AM will stay in place until sufficient understanding of AM materials is gained. This will enable engineers to do one-for-one substitutions where they know that material properties are similar. As a result, metal AM for replacement components in DoD aerospace products has an MRL of 2, at best. No proof-of-concepts have been identified and no programs have volunteered to test the technology. While other industries can easily adopt AM for replacements due to the lower level of reliability required, aerospace applications will likely avoid using AM for replacement components in the near future. AM will be adopted where it is cost-effective to trade AM for another manufacturing technology, *i.e.*, during initial product design, before the costly qualification phase.

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Chapter 6

Experimentation on AM Parameters

Creating a business process to support the development of metal AM techniques within Raytheon is critical to the overall adoption success of those new manufacturing technologies, but Raytheon has also demonstrated an immediate and practical interest in developing their technical manufacturing capabilities in metal AM. As this project began, there was an opportunity to execute qualification and characterization testing on equipment newly installed within Raytheon facilities. This opportunity was coupled with an urgent need to understand the capability of the AM equipment to begin providing test components to different groups within Raytheon.

The constraints mentioned above led naturally to the mathematical area of experimental design in an effort to conserve time, effort and resources while providing characterization information. This section intends to explain the development process for experiments executed on Raytheon's new AM equipment, as well as present the results of said experiment as related to Raytheon's overall mission to incorporate metal AM into aerospace component production. The experiment described in the following section addresses only the first use case from Section 5.5.

6.1 Experimental Approach

The first components visible in the AM qualification process in Figure 5-1 are Material Characterization and Equipment Qualification. To develop understanding in either of these

Table 6.1: Goal Outcomes of Metal AM Experimentation on Nickel and Aluminum Alloys

Desirable Qualities	**Units**
Ultimate tensile strength	MPa
Yield tensile strength	MPa
Young's Modulus	MPa
Ultimate strain	%
Minimal metallographic defects	n/a
High apparent density	g/cm^3
High dimensional accuracy	μm
Smooth surface finish	μm
Uniform layer thickness	μm
Uniform melt pool radius	μm

areas, this project had to identify first the desirable qualities of components produced with metal AM and how to make a significant impact on those qualities.

6.1.1 Identification of Potential Responses

Based on the intended use of AM produced components as flight-ready elements of Raytheon products for sale, a specific set of important product qualities for nickel and aluminum alloys was developed with metallurgy subject matter experts (SMEs) and major stakeholders. This list of qualities can be found in Table 6.1.

Characterizing tensile strength, strain and metallography are considered basic requirements for component designers at Raytheon to incorporate a manufacturing technology and material in initial design reviews. While the manufacturer and supplier of the metal AM equipment and material provides estimates of key mechanical properties, internal Raytheon users of the equipment are verifying these properties to be able to confidently present their capabilities to customers. Metallographic analysis must show limited defect formation in order to meet most basic fatigue performance requirements. As Raytheon uses specifications from Metallic Materials Properties Development and Standardization (MMPDS) to justify to its Department of Defense (DoD) customers that its designs have sufficient mechanical strength, the stakeholders in this project sought to compare the bulk mechanical properties of AM-produced metals to wrought alloys of the same composition.

Dimensional accuracy, surface finish and layer consistency are key manufacturability outcomes that will dictate the amount of post-processing machine work that is required to ready a component for final integration. Initial testing with metal AM equipment had shown a limited capability to hold the as-designed dimensions and generally required machining to specifications. In addition, surface roughness was high enough to require abrasive polishing or surface machining techniques in order to meet customer expectations for appearance. Surface roughness can also be a source for fracture failure modes in components. The AM process needs to demonstrate a high layer of adhesion between individual layers of material to reduce the risk of separation under tensile loads and improve overall appearance of the final product. The final listed outcome, consistent melt pool behavior, is one of the few process variables that can be measured *in-situ* with optical equipment (Concept Laser, 2016) and is a key component of developing SPC around metal AM in the future.

6.1.2 Identification of Potential Factors

With the above expected outcomes, a set of process inputs had to be selected based on their intended effect. Information used to make this selection came from multiple sources: existing research, experience from metallurgical SMEs within Raytheon and internal experts in the operation of the new AM equipment. An initial proposal was made to focus on the process parameters seen in Table 6.2.

The first two factors listed fall under the category of configuration because they are set almost entirely by the operator of the AM equipment. Components can be placed in the AM equipment such that their longest axes or axes of expected mechanical load can be parallel, perpendicular or at some intermediate angle to the direction in which the individual layers are built up. The importance of these factors was demonstrated empirically in the initial trials leading up to this experiment, where noticeably different quality parts were produced depending on their orientation in the equipment build chamber. Surface finish was affected and preliminary tests showed a reduction in strength when layers were built along the test stress axis.

Evidence for the importance of the listed equipment factors can be found in the research described in Section 2.3.1, particularly the experiments that focused on varying laser power and scan velocity in order to create different metallographic qualities. Each of the

Table 6.2: Desirable Factors for Inclusion in AM Experimental Design

Configuration Factors
Part Orientation
Part Aspect Ratio
Equipment Factors
Laser Power
Scan Velocity
Scan Spacing
Beam Diameter
Feedstock Factors
Flowability
Dose Factor
Reuse/Recycle Cycles
Particle Size (sieving)
Coater Blade Height
Post-Process Factors
Hot Isostatic Pressing (HIP)

four equipment factors has a direct impact on the applied energy density (E_A) calculation demonstrated by Equation 2.1 on page 31. Unlike many of the parameters that are expected to impact metal AM, most AM equipment allows these factors to be adjusted within a range. In addition, there is limited research available on the specific alloys of interest to Raytheon, which further suggested that understanding those equipment factors is critical to the overall success of AM qualification for Raytheon's programs.

The feedstock factors focus mostly on the material handling aspects that are critical to the performance of metal AM equipment. Flowability of a metal powder impacts the ability of the AM equipment to deliver new material, whether through jetting mechanisms or spreading layers of powder. For powder bed fusion (PBF) equipment in particular, the dose factor and coater blade height will have an impact on material consumption and layer thickness, respectively. Sieving procedures to create uniform particle size distributions and limits on reuse cycles for unused material will both have impacts on the flowability of the powdered material as well as potentially affect the melting behavior of the powder as it transitions between solid and liquid. The final suggested factor, HIP, likely has the most impact on final cosmetic and mechanical characteristics of any other factor in the post-processing category. The method by which an AM specimen is removed and polished is

Table 6.3: Factors for Multiple Phases of AM Characterization DoE

Main Factors	**Phase**
Test specimen configuration	1A
Orientation of as-built coupon	1A
Location on build platform	1A
Hot Isostatic Pressing	1A
Powder Reuse/Recycle Cycles	1B
Laser power	2
Laser scan speed	2
Laser scan spacing	2
Laser beam diameter	2
Scanning plan	2

Table 6.4: Responses for AM Characterization DoE

Responses	**Units**
Dimensional Accuracy	% or μm (39 microinches)
Surface Finish	μm
Density (vs. wrought)	%
Ultimate tensile strength	MPa (0.145 ksi)
Yield tensile strength	MPa (0.145 ksi)
Young's Modulus	MPa (0.145 ksi)
Ultimate strain	m/m (%)

often dictated by available equipment and the specific needs of the customer, but variations in heat treatment procedures have significant impacts on crystallography and as-treated mechanical properties.

6.1.3 Selection of Experimental Factors and Responses

With the desirable outcomes and influencing variables identified by a multi-disciplinary team, it was then necessary to down-select the process settings and desired outcomes into a smaller set of "factors" and "responses", respectively. Despite the ability of Design of Experiment (DoE) techniques to produce what Montgomery says are the most efficient experiments given available resources and time (Montgomery, 2001), there was still a need to reduce the total number of factors. The final factors selected for the experiment can be found in Table 6.3, accompanied by the list of responses to be measured in Table 6.4.

In order to expedite the test execution and eventual collection of results, a decision was made to split the experimental factors into multiple phases, not all of which would be completed within the purview of the thesis project. The Phase 1A factors were selected for the scope of this thesis. Coupon orientation was an empirically obvious selection for experimentation because of AM's distinct difference in component buildup from other manufacturing techniques like machining or casting, as well as the documented variation caused by orientation mentioned in Section 2.3.1.

Location on build platform was a necessary addition as a test factor due to space and time limitations: testing only samples built in the center of the AM build platform would have wasted material and increased the test duration by a factor of 4. While AM equipment manufacturers assert that an entire build volume can be used, this must be confirmed before building aerospace components that use most of that space. Test specimen configuration was selected at the recommendation of metallurgical SMEs to understand the emergent behavior observed in some early testing where warping of individual layers varied between different shape test specimens, despite similar support structures used to anchor the specimen and transmit heat to the build platform.

The significance of the other two selected factors, HIP and powder recycling, was explained in Section 6.1.2. Due to the difficulty of changing powder types in the middle of the experimental trials, powder recycling will be tested as a separate test run in a split-plot experiment ("Phase 1B") and is out of the scope of this thesis. This experimental design will be discussed in greater detail in Section 6.2.

Phase 2 will be completed after additional characterization and training on the AM equipment is completed, and will be informed by the results of Phase 1. While the laser configuration parameters are a high priority to characterize the behavior of the AM equipment, inexperience with the AM equipment has pushed laser configuration testing to a later phase of testing. In addition, those parameters are usually guarded and/or fixed by manufacturers, adding to the qualification procedural difficulty mentioned in Section 2.3.3 (Slotwinski, 2014). It is this writer's hope that the Raytheon team's experience with DoE techniques will be instructive in later implementing this phase of the experiment.

The responses selected for this experiment were similar to the most important goal outcomes from Table 6.1, with the unfortunate removal of the melt pool monitoring. This equipment was not operational on Raytheon's AM equipment for the duration of the experiment. The remaining responses are measured in well-known units. In this report, dimensional accuracy will be analyzed in terms of absolute deviation from design (μm) and in relative deviation compared to the desired dimension. Surface finish will be described in the quantity mean roughness R_a (μm).

Ultimate and yield tensile strengths are standard mechanical properties and will be reported in MPa, as well as thousands of pounds per square inch (ksi). Young's Modulus is a property that defines the relationship between stress and strain within the elastic deformation regime and will be reported in MPa as well. Ultimate strain is a property that indicates the extent of strain at failure of the material, and is reported as dimensionless like with most other strain values. Each of the listed responses will be analyzed with the proposed factors to understand the impact of each factor, individually and in conjunction with each other.

6.2 Experimental Design Development

One of the most time-consuming activities in running experiments is setting up the structure of the tests to be completed. This is likely why many experiments are executed by changing only a single factor at a time, which is a considerable waste of time and resources. This section hypothesizes the effects of the selected factors on the desired outcomes, and then describes and evaluates an efficient experimental design to understand those effects.

6.2.1 Hypotheses

Each experimental factor selected for experimentation should have some theorized relationship between it and a response variable, stated as an alternate hypothesis. Statistical testing may then reject or fail to reject the opposite hypotheses, known as the null hypotheses. See (Barnett, 2015, p. 240) for further explanation of hypothesis testing. The resulting conclusion allows users of the experimental results to decide on the best course of action relative to the response's impact. All testing will use a p-value of below 0.05 to justify rejection of the null hypothesis.

The statements below are the hypotheses that will be tested in the proposed experiment:

- Vertical build orientation will have a negative impact on tensile strength and fracture characteristics. Structures with planes built parallel to the powder bed will have lowest tensile strength. Coupons built in the Z-axis orientation are also expected to have a higher tendency of undesirable failure characteristics.
- Distance from the center of the build platform will be negatively correlated with surface finish quality/roughness and dimensional accuracy.
- HIP post-processing will cause lower tensile strength properties but reduce porosity and improve toughness.
- Unlimited use of recycled powder will degrade mechanical properties (out of scope of this thesis)
- The test specimen configuration will have an impact on some of the measured response variables.

6.2.2 Experiment Type Selection

DoE is an extremely large mathematical and analytical field that contains a wide range of experimental techniques that are optimized for many kinds of experiments. Selecting an experimental design must be based on experience with the factors in the experiment, as well as authorities in the area of experimental design. Two sources in particular were consulted on experimental design: Montgomery's *Design and Analysis of Experiments* (Montgomery, 2001) and a Raytheon DoE expert who reviewed the proposed design and suggested improvements.

Given that so little was known about any of the experimental factor effects at the beginning of this experiment, it was most logical to consider the proposed experiment a screening experiment, or one "in which many factors are considered and the objective is to identify those factors (if any) that have large effects." (Montgomery, 2001, p. 303). Even if several experimental factors are found to have no effect on the responses of interest, that information allows future production to discount any potential risk from modifying those factors (within the ranges tested in the experiment).

Initially, the most likely candidate for this experiment's design was a standard factorial design to understand the effects and interactions of all included factors, up to a two-factor interaction. There was no information available in literature or experience at Raytheon that suggested any of the selected factors would have non-linear interactions with the responses. This ruled out the structure of most response surface designs that are intended to understand the curvature of a response over a range of factor settings. Based on that knowledge and a general tendency towards experiment simplicity, there was no need to run an experiment with greater than two levels on each factor.

This prompted the selection of a 2^k full factorial design as suitable for this class of experiment, where k is the number of factors under test and equal to five as listed in Table 6.3. In order to simplify later analysis of the design, each factor is assigned a high and low level that is then coded in the statistical analysis software as +1 and -1, respectively. 2^k or $2^5 = 32$, in this case, refers to the number of individual trials required to cover all possible combinations of the five factors. An example of this coding is shown in Table C.1 of Appendix C, with the sequencing of the coding randomized as required for an actual experiment. The uncoded values of the factor levels are described in the next section, Section 6.2.3.

The proposed design can be used at this point for many applications of DoE. But as mentioned earlier, there are differences between some of the factors that prevent their inclusion in a truly random set of trials. Powder type is a factor that requires a significant amount of physical labor and equipment downtime in order to change. Its inclusion in the experimental design as a normal, "easy-to-change" factor would be an inappropriate use of time and resources. This is easily handled in experimental design theory through the adaptation of a split-plot design. Split-plots are used when experimenters are unable to completely randomize the sequence of runs (Montgomery, 2001, p. 573) and allow the separation of one more factors into an entirely separate set of runs. The data from the separate runs for the "hard-to-change" factor like powder type can then be combined and analyzed, with an expected loss of power in understanding the relationship between powder type and the experimental responses. The loss of discrimination power between confounded effects is a necessary byproduct of running nonrandom trials, but analysis of the experimental design showed that power decreased only slightly (~ 0.7 *vs.* 0.98 for the other factors). The changed

Table 6.5: Four-Level Factor A Expressed as 2 Two-Level Factors (Montgomery, 2001, p. 385)

	Two-Level Factors		**Four-Level Factor**
Run	**P**	**Q**	**A**
1	−	−	a_1
2	+	−	a_2
3	−	+	a_3
4	+	+	a_4

run coding based on the split-plot design can be seen in Table C.2 of Appendix C.

After completing the coded design, each experimental factor was assigned two levels to match 2^k design. Most of the factors were easily separated into desirable high and low values, but when attempting to assign values for distance from the center of the build platform, it was discovered that two levels were insufficient to efficiently utilize the available space. In short, large bars and cylinders that are nearly the same size as the build platform can only be placed in two locations on the build platform. Even with the assumption of horizontal symmetry (that items built on the left will be similar to those built on the right), effective use of an experimental run required four level values. The traditional way of accommodating a four-level factor is to use two two-level factors to represent it. Table 6.5, reproduced from Montgomery, represents the coding for those two factors.

This design was implemented by setting one factor as a distance from the center of the build platform and the other factor as a distance offset, positive or negative from the distance of the first factor. Although this increases the number of factors for the experiment to six, the decision was made to decrease the fraction of the experiment to half factorial to keep the same number of trials required at 32 (experimental structure is 2^{k-p} or 2^{6-1} for a half fractional factorial). Fractional factorials continue to be useful in screening experiments because with an increasing number of variables, the process is more likely to be driven or influenced by only a few factors or interactions. In addition, the fractional factorial experiment's power can be increased by additional experiments afterward that complete the full fraction (Montgomery, 2001, p. 303).

Now that the number of possible test coupons per build was known to be a maximum of eight (four factor levels multiplied by two for symmetry in the X-Y plane of the build chamber), it was still necessary to identify how to separate 32 samples in a replicate in a way that accounted for variation between build runs. The solution was to consider each build run as a block that would be confounded with some of the interactions between factors. The experiment was already split into two plots of 16 each for the powder factor, so confounding the experiment required a break up into two blocks of eight experiments each. Based on a request from the group that would be doing much of the material testing, the entire experiment will also have two replicates. While providing alternate samples in case there are any test failures, it also increases the number of blocks and provides more understanding of the effect that the blocking, or the individual AM equipment runs, has on the experimental responses. The end result was the creation of four individual blocks of eight runs each as the final structure of the experiment.

6.2.3 Factor Level Descriptions

The proposed AM qualification experiment consists of 6 factors from the selected factors above:

- Powder Type
- Z-Orientation
- Distance from Center
- Distance Offset
- HIP
- Coupon Type

These will be the standard names of these factors throughout the rest of the analysis. In order to analyze a 2-level screening experiment based on $2^{k\text{-}p}$ dimensionality, each factor has a high and low level that will be coded in the statistical analysis software as +1 and -1, respectively. Powder type has the categorical levels "Virgin" and "Recycled". Virgin powder is defined as AlSi10Mg powder that has not been used for any AM processes since it was received from the powder supplier. Depending on lot quantities, the powder may need to come from separate containers from the same supplier. Blending can be used to create run uniformity, or runs with different powder lots can be further blocked to indicate a possible random factor in the design. The source and composition of recycled powder will be better defined by the time the second part of the split-plot experiment begins, but its current definition is any powder that has been accumulated from any number of AM builds completed at Raytheon Precision Machining (RPM), and then sieved to remove particles with diameter 63 μm and above. A 63 μm sieve was provided by the AM equipment manufacturer as ideal equipment to refine and recycle used AM powders. The history of this powder and ability to reproduce its characteristics is in question, so a procedure for creating a uniform quality of recycled powder will need to be created. Empirical observations suggested a link between undesirable powder characteristics (ease of spreading, agglomeration, *etc.*) and use of recycled powder greater than 15 times.

Z-orientation refers to the angle between the long axis of test coupons and the AM equipment build platform. The two numeric factor levels are 0° and 90°, where 0 degrees indicates that a coupon is built with its longest axis parallel to the build platform and 90 degrees indicates that the long axis is perpendicular. This is a simplification of possible orientations, as the AM build software is capable of orienting CAD files in any orientation. The extremes captured here reflect the greatest and least possible alignment of layer build direction with testing force direction.

Distance from center is the distance in mm between two points: the projection of the geometric center of the coupon onto the X-Y plane of the build platform, and the center of the build platform. The size of the build platform dictates that the center of a test coupon can be no more than 110 mm from center in any direction. This factor is combined with the second distance factor, distance offset (also in mm). Distance offset is the number of mm to adjust the distance from center value in order to get the true distance from center. For example, a distance from center of 30 mm and distance offset of -15 mm would equal an actual coupon distance of 15 mm from the center of build platform. The combination of these two factors creates a pseudo-factor with four levels referred to as "Actual Distance".

HIP, or hot isostatic pressing, is a factor with two categorical levels. HIP will be applied to some samples before completing response measurements. HIP is also an analog process with many different levels, but a process recipe recommended by the outside HIP processor for this material will be used for all samples that require HIP. Experimenting with different HIP parameters is outside the scope of this experiment.

The last factor is categorical on the shape of the test coupon, designated as coupon type. There could possibly be some impact on response performance based on the aspect ratio of the coupon. The round cylinder has a lower aspect ratio compared to the thin rectangle—the surface area of the round cylinder's footprint on the build plate is considerably larger than that of the thin rectangle. The effects of this factor may be correlated with the effects of Z-orientation because the surface area of a coupon in contact with the build plate increases drastically in the 0 degree orientation. The coupon types to be used are specified below and were selected based on their ability to be machined later into standard dogbone ASTM test specimens.

- Cylinder: 20.32 mm (0.8 in) diameter, 203.2 mm (8 in) length (152.4 mm standard length + 50.8 mm to be machined off to use for hardness testing)
- Thin Rectangle: 25.4 mm (1 in) wide, 203.2 mm (8 in) length, 6.35 mm (0.25 in) thick

A summary of the complete experimental design with uncoded factor levels can be found in Table D.1 of Appendix D. The first half of this table represents the replicated split-plot design of Phase 1A mentioned earlier and is the extent of what was executed at Raytheon facilities by the time of publication of this thesis.

6.2.4 Experiment Design Evaluation

The complexity of this experimental design made it difficult to holistically assess the statistical power of the experiment and the aliasing of factors and interactions that occurred as a result of the confounded design. Much of the initial experimental design work described previously was done with the assistance of the statistical software package SAS JMP, as requested and provided by Raytheon. JMP has the capability of evaluating custom experimental designs. One of the tools in the Evaluate Design platform is a color map of

Table 6.6: Main Effect Correlations in Proposed Experimental Design

	Powder Type	**Z-Orientation**	**Distance from Center**	**Distance Offset**	**HIP**	**Coupon Type**
Powder Type	1	0	0	0	0	0
Z-Orientation	0	1	0.125	0.125	0.125	0
Distance from Center	0	0.125	1	0	0	0.125
Distance Offset	0	0.125	0	1	0.125	0
HIP	0	0.125	0	0.125	1	0
Coupon Type	0	0	0.125	0	0	1

correlations between selected experimental factors, also referred to as main effects, and interactions. While the coloration is not as helpful to illustrate any undesirable confounding, the result of this color map has been transferred to Table 6.6, with each intersection of row and column representing the degree[1] from 0 to 1 which those two main effects correlate due to insufficient randomization of trials.

No main effect is unconfounded with all other main effects, but all correlation levels are relatively low (significantly less than 0.5) and only one factor, distance from center, is confounded with two other factors. This is unsurprising given the geometric constraint on the distance factor—the different levels of the factor have to be in the exact same sequence in every experimental block. Running this correlation analysis while including all interactions between two-factor shows a similar level of low correlation (not shown here). Some two-factor interactions reach a correlation of 0.5 but the likelihood of many or all of those two-factor interactions having a significant impact on the model is low and therefore being able to contrast their effects is less important. However, the level of correlation that is demonstrated in Table 6.6 shows the value in retaining a large number of experimental runs and blocks. Decreasing the size of the experiment to quarter-fraction would have shown more significant correlation as the limited number of degrees of freedom would have prevented contrasting

[1]Values of 0 are not absolute but are estimates of values <0.0001.

Table 6.7: Main Effect Correlations in Executed Experimental Design (Phase 1A)

	Z-Orientation	**Distance from Center**	**Distance Offset**	**HIP**	**Coupon Type**
Z-Orientation	1	0.5	0.25	0.25	0
Distance from Center	0.5	1	0	0.25	0.25
Distance Offset	0.25	0	1	0.5	0.25
HIP	0.25	0.25	0.5	1	0
Coupon Type	0	0.25	0.25	0	1

certain main effects.

A similar analysis should be completed on Phase 1A of the proposed experiment alone, considering that results for this project will consist of only the first half of the experiment. The adjusted correlations can be seen in Table 6.7.

As expected, correlation gets worse as more runs from the full set get removed, but there are still no correlations over 0.5. The increasing correlation indicates the importance of getting all available data, but does not prevent separating most of the main effects, as long as correlation is considered in conclusions taken from experimental results.

6.3 Experimental Implementation

Once the experimental design had been finalized and evaluated for capability, the experiment could be implemented and data collected. The proposed experiment was executed on equipment installed in one of Raytheon's machining excellence centers. The manufacturer of the equipment is one of several supplying powder bed fusion AM technology to a variety of industries, and the model used was a type designed for use with multiple types of metal powder, including the nickel and aluminum alloys of interest to Raytheon.

In this experiment, an AlSi10Mg alloy was used for all runs. In order to test with all unused powder, approximately 160 kg of powder was required to execute all 32 experimen-

Table 6.8: List of Builds and Contained Runs during Experiment

Build	Block Description
I	Block 1 (Runs 1 – 8)
II	Block 2 (Runs 9 – 16)
III	Block 3 (Runs 17 – 24)
IV	Block 4 (Runs 25 – 32)

tal runs in Phase 1A. These runs were partitioned into 4 separate blocks or individual AM equipment build cycles (partitioning shown in Table 6.8, see additional numbering in Appendix D). Builds have been stylized with Roman numerals to distinguish them from blocks. Builds III and IV are exact replicate designs of I and II, respectively; this can be confirmed by comparing blocks 1 and 3 or 2 and 4 in Table D.1.

Only 15 containers of 10 kg each were available, and sieved recycle material from build I had to be included to make up the difference for build IV. This may have no effect with only a single reuse of the powder, but it can be considered in analysis of data from build IV. All metal powder came from the same manufacturer lot of material.

Builds I and III were required to use the same design and therefore had the same CAD file driving the AM equipment (with the exception of different identification markings designed into the ends of the coupons). This CAD file was developed using the test coupon sizes and distance from center measurements listed in Section 6.2.3. Figure 6-1 and Figure 6-2 are two-dimensional representations of the CAD file; although they were generated with different software, they were used as templates for the CAD files actually used. A similar CAD file was created for use in builds II and IV.

When the files for all builds were checked for interference and any design issues, an issue was flagged with the orientation of the test coupons. In past experiments, any built surfaces that were parallel to the coater arm tended to cause powder to stick to those surfaces and upset the smooth flow of powder onto a new surface. This effect was cumulative to the point of creating a build failure in the past. To mitigate this risk, all build files were rotated 18 degrees counterclockwise in the XY plane (around the Z-axis) of the build platform. Figure 6-1 and Figure 6-2 show the build layout before rotation, and the XY plane is indicated in Figure 6-2. The preferred angle would be 45 degrees but at any higher rotation, the length

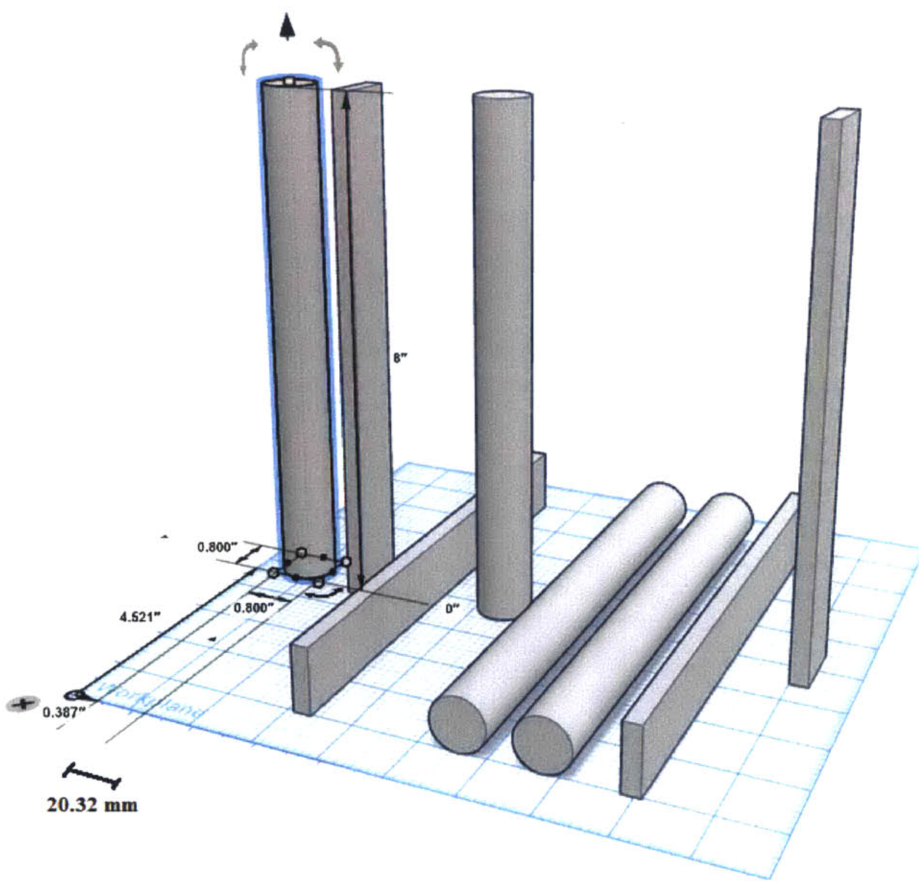

Figure 6-1: Isometric View of Layout for Build I and III

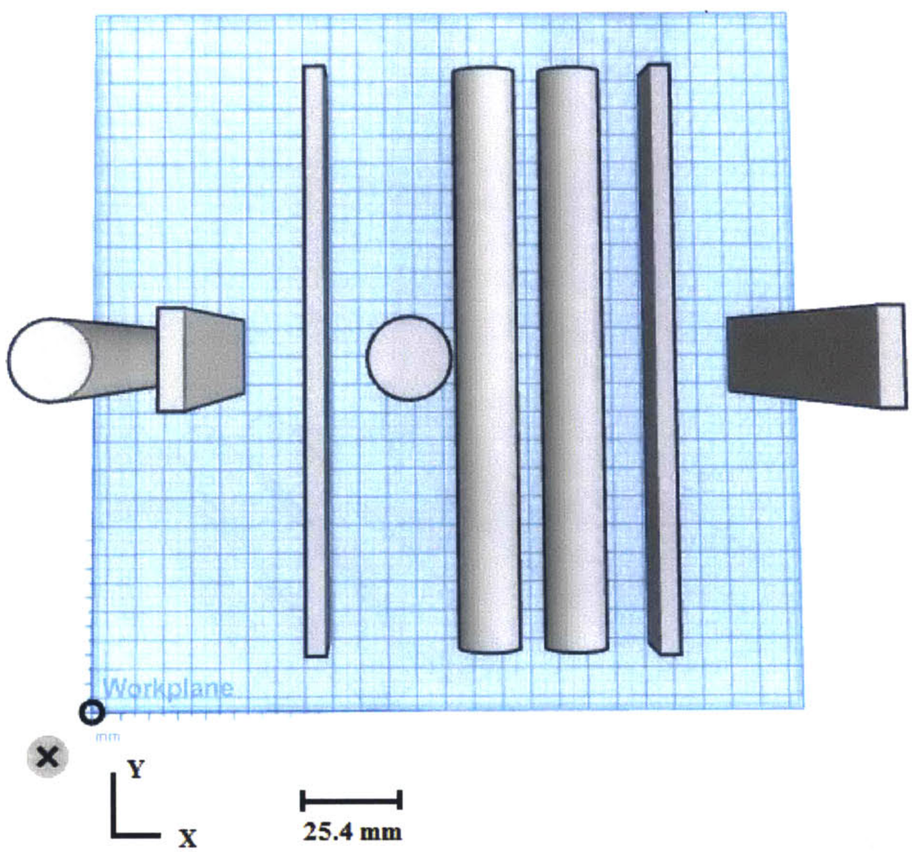

Figure 6-2: Top View of Layout for Build I and III

of the coupons would interfere with the bolt holes used to secure the build platform to the AM equipment. For the same reason, the sequence of two coupons was reversed from the original design, but this has been updated in the tables and statistical analysis.

The procedure used to execute each of the builds was consistent, and very similar to the procedure listed for PBF in Section 2.2.1. The laser power parameters set for each build were the same: the "performance" parameters specified by the AM equipment manufacturer for the manufacturer-supplied alloy. Each build required approximately 60 hours to complete. After completion of the build, excess powder was removed from the build platform and then the build was taken to a metrology lab. Measurements of each sample were made in the X, Y and Z directions, where the XY plane is parallel to the build platform and Z direction is parallel to the build-up of individual layers. For all dimensions of samples, the X direction is from left to right and Y direction is from bottom to top on Figure 6-2. Measurements were completed with a combinations of calipers and a MicroHeight gauge on a granite surface. Data as recorded are the arithmetic means of three repeated measurements for each data point.

After initial measurements were completed, all coupons were subjected to a thermal stress relief cycle recommended by the AM equipment manufacturer. This consisted of the following process:

1. Heat up within 1 hour to 240°C
2. Keep at temperature for 6 hours
3. Allow items to cool in oven to 100°C
4. Remove from oven and let cool to ambient temperature

After the coupons had cooled to ambient temperature, dimensional measurements were repeated. The coupons were then removed from the build platform using electrical discharge machining (EDM) and all dimensions were measured again. All coupons were then sent to the metrology lab for surface roughness testing. Surface roughness testing was completed with a Taylor-Hobson Surtronic 3 profilometer using a diamond stylus over a 3.175 mm (1/8 in) test distance. The rectangular coupons were tested on one side of the 2.54 mm (1 in) surface at one end of the longest dimension, then at the other end and finally in the center. Cylindrical coupon tests took place on the side of the cylinder at one end of the longest dimension, at the other end rotated 180 degrees and finally on the original end in a random rotation. All three measurements for each coupon were recorded and were only averaged in analysis.

After surface roughness measurements were completed, the coupons were separated by whether they were intended for HIP, based on Appendix D. Those coupons that were destined for HIP were sent to a local supplier capable of completing HIP. The other coupons were sent to a machining center to be milled into standard ASTM specimens for tensile testing. The rectangular coupons were machined into ASTM B557 Figure 6 Standard Flat Specimens with thickness 5.08 mm (0.2 in), and the cylindrical coupons were machined into ASTM B557 Figure 9 Standard Round Specimens (ASTM Standard B557, 2015). Tensile testing was completed with MTS Insight 50 and 300 uniaxial tensile testers.

The cylindrical samples had the bottom 50.8 mm (2 in) removed and saved for Rockwell hardness testing. After completion of machining, specimens were subject to tensile testing under ASTM B557. Rockwell hardness testing was completed on the remnants of the rectangular tensile test and the precut portions of the cylinders. (ASTM Standard E18, 2015). Density was also measured on the test remnants (ASTM Standard B311, 2013). The final test was a metallographic qualitative analysis of the test remnants. The final materials tests will be completed after publication of this thesis and are therefore out of scope.

6.4 Discussion of Results

This section will cover analysis of the data collected from the experiment designed and executed as described above. Each analysis will begin with an overview of the data set, followed by an effort to match a linear regression model using the experimental factors that applied to that data set. In most cases, the data used is an obfuscated and transformed data set to protect proprietary Raytheon information; unless otherwise specified, the data has been transformed by subtracting the mean and dividing by standard deviation. This is the same process that can be used to convert any normal distribution into the standard normal distribution.

The distributions of data before and after transformation are the same, allowing for continued analysis of the data's normality. This is supported by the identicalness of the untransformed and transformed data's boxplots. In addition, any linear regression models built on untransformed data had the same R^2 values and parameter significance values as the transformed data regression models.

6.4.1 Dimensional Accuracy

Dimensional accuracy was measured and recorded in three different directions—X, Y and Z relative to the build platform—as well as collected at three different times in the build process.

Before Heat Treat and Removal The first set of measurements was before removal from the build plate and before heat treatment: the as-built dimensions of the test coupons. X and Y dimensions can be interpreted as measured, but the measured thickness of the build plate for each build had to be subtracted from the recorded data. While the recorded data could be analyzed as is, the design dimensions of each of the samples was based on its role in the experimental design. Examining only the absolute measurement would cause clustering in the data around the nominal dimensions. Instead, an error value was calculated for each measurement by subtracting the nominal design dimension size (based on Section 6.2.3) from the recorded measurement.

When the error data were first analyzed, two anomalous points were visible in the boxplot (Figure 6-3) of Z-direction error data before heat treat and removal, known hereafter as Error Z-BR. The outliers are shown as gray dots, while the boxplot in the figure is so condensed, it appears only as a series of vertical lines.

After a short analysis, these data points were concluded to be data entry errors (based on the repeated measurement taken after the heat treatment). With this correction made, the transformed data set distributions for Error X-BR, Error Y-BR and Error Z-BR can be seen in Appendix E, Figure E-1. This analysis demonstrates that of the transformed data, only the Z-BR distribution is likely to be normal based on a goodness-of-fit test. This

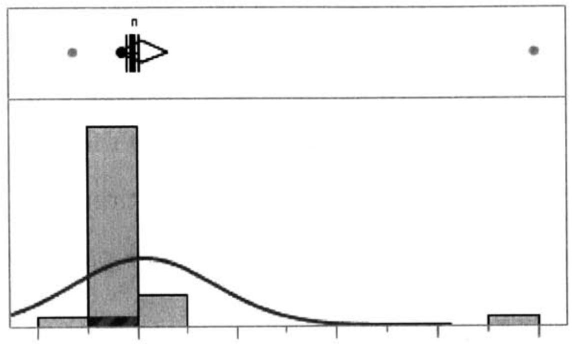

Figure 6-3: Obfuscated Distribution of Data for Error Z-BR, showing two data entry outliers

poses a problem for regression analysis, considering most satisfactory models are built on an assumption of normality.

Ordinary least squares (OLS) multiple regression models were built in the Fit Model platform of JMP for each of the transformed error data sets. The only regression model with a reasonable fit was the one built for transformed Error Y-BR. All interactions of main effects were included along with main effects in the initial model, then successive insignificant factors were removed to reduce overfitting of the data. The JMP Fit Model platform summary can be found in Figure E-2 and the distribution analysis of the model's residuals can be found in Figure E-3. The high F ratio of the model and low p-values of individual parameters in the model indicate, along with a high adjusted-R^2 value, that the selected parameters have a significant impact on errors in the Y-direction. The residuals of the model also show normality.

The Z-orientation and both distance from center factors were significant in the proposed multiple regression model. Coupon type was not significant on its own, but was in one of the interactions, but its interactions with the distance factor are likely due to correlation demonstrated from Table 6.7. The most interesting model effects noted are:

- The tendency of Y-Error to increase with Z-orientation rotation upward (*i.e.* smaller dimensions are oversized, larger dimensions are undersized)
- As distance from the center increases, the Z-orientation effect decreases substantially

This model points to some interesting speculative effects. The magnitude of the designed dimension has a significant effect on the amount of error in the final product. It's possible that some intermediate magnitude between the two extreme dimensions could reach a balance point and approach an average of zero error. There appears to be at least one mechanism of distortion in action here. In addition, if a goal is to minimize total variability between dimensional error on pieces with high aspect ratios (like these test coupons), building the part near the edge of the build platform can help reduce the extreme variation between samples and orientations.

After Heat Treat The second set of measurements was taken before removal from the build plate but after heat treatment. This represents an examination of any dimensional error caused by or normalized by the heat treat process. A single data entry error was found and corrected (impossible based on expected dimensions of specimen). Again, an error value for each dimension was calculated and transformed to obscure the true values of the data. These are represented as Error X-AHT, Error Y-AHT and Error Z-AHT, with a "T" prepended on the name to indicate a transformation. The distribution of TError X-AHT fails to reject the hypothesis of normality, but TError Y-AHT has an outlier that throws suspicion on the assumption of normality (see Figure E-4). After examining this outlier relative to the other dimensions of this same sample, it appears to not be a data entry error and is left in the data set.

Linear regression models for each dimension were built again. The models can be seen for TError X-AHT and TError Y-AHT in Figure E-5 and Figure E-6, and are followed by their residual analyses (Figures E-7 and E-8). The distortion caused by the outliers in TError Y-AHT are visible in the residuals normal probability plot in Figure E-8. TError Z-AHT was also analyzed, but there did not appear to be any strong modeled effects, and fit as measured by adjusted R^2 was very low (<0.2). This may be due to no effects on Z-dimension accuracy or additional noise that is introduced by the thickness of the build plate that must be tared off. The model effects of interest here are:

- The heat treat process appears to have reversed the trend seen in the previous analysis, where Y-Error increased as the specimen was rotated upward (Z-orientation). This is more in line with expectations of larger dimensions having a larger error (*i.e.*

dimensional error is a fractional property, rather than absolute).

- For Y-error and X-error, the coupon type has an interaction effect with Z-orientation. There is much less variation between Z-orientations for a thin rectangle coupon than a cylinder coupon.

- Location on the build plate again has an effect on X-error, but the effect is very small relative to the effects of changing the specimen orientation. It appears that as the specimen is placed closer to the edge of the build plate, X-error increases slightly. This is contrasted with the pre-heat treat observation that variation is decreased at the edge of the build plate. It appears that that advantage disappears after the heat treat process.

Here again, there is a factor that will reduce variation across the range of design values. While overall error was lower for a cylinder at 90°, a designer may prefer to create cross-sectional shapes that better match the thin rectangle, as these will not vary as significantly when rotated on the build platform. This provides the designer more flexibility when trying to plan builds and create support structures.

After Plate Removal While data was available for dimensions after the build plate removal, there was very little difference between the X-error and Y-error of "after heat treat", which could be due solely to variations in measurement from trial to trial. For Z-error, the variation induced by removing the build plate effectively prohibits analysis of any further Z-error data once the cut has been made. The behavior demonstrated in the "after heat treat" is sufficient to draw some conclusions.

Summary of Dimensional Accuracy Coupon type, distance from build platform center and coupon orientation had no significant effects on dimensional accuracy in any direction. The previously asserted hypothesis, distance from the center of the build platform will be negatively correlated with dimensional accuracy, had very little data to support rejecting the null hypothesis, that distance from the center has no effect on dimensional accuracy. The parallel null hypotheses for the remaining main effects failed to be rejected, except for orientation of the specimen. However, orientation is also coupled with the size of the dimension in the cases of Y- and Z-axes. For specimens that have been heat-treated, there appears to be a positive correlation between the length of the dimension and the amount of error, indicating a possible proportionality relationship. This is critical to understanding the final as-built dimensions for any components made using this technology.

6.4.2 Surface Roughness

To characterize surface roughness, three values of roughness were taken from each sample. In order to aggregate these results, the arithmetic mean and standard deviation of each sample set was calculated. The means were then transformed in the standard way to obfuscate the data. An OLS multiple regression model was then created from the means for each sample, and can be seen in Figure E-9. This model had a fairly high adjustedR^2 value (~0.5) and a normal distribution of residuals, as seen in Figure E-10. The main effects with significant impact on the roughness results were:

- Distance from center, which increases all roughness means as the distance from center increases
- An interaction between Z-orientation and distance offset. This is interpreted as a larger increase in surface roughness variation as distance offset (and therefore distance from center) increases, for specimens at a 90° orientation.

As a practical interpretation, this may confirm the knowledge that a calibrated PBF AM system has been setup so that its laser power source is focused on the surface plane of the build platform. For both of the main effects above with significant impacts or interactions, the changes in those factors would have increased the distance from the focal plane of the laser—distance from center creates longer beam length via Pythagorean Theorem, and an upright specimen shortens the distance between a specimen and the laser source. On the other hand, these issues may already be handled by the AM equipment's proprietary control algorithms, and the variation may have a different source.

A regression model of the surface roughness sample standard deviation was also completed, but its adjusted R^2 value was very low and there were no clear or significant effects from main factors. All standard deviation variation, or variation comparison between samples, appeared to be due to random fluctuations, indicating there is innately a high level of variability in the surface roughness of finished specimens.

6.4.3 Mechanical Properties

The tensile testing of all specimens produced in the experiment generated results for four different mechanical properties, repeated from Section 6.1.2 and listed in Table 6.9.

Table 6.9: Mechanical Properties Tested in Experiment

Responses	**Units**
Yield Tensile Stress	MPa (0.145 ksi)
Ultimate Tensile Stress	MPa (0.145 ksi)
Young's Modulus (E)	MPa (0.145 ksi)
Ultimate Strain	m/m (%)

The following paragraphs will address the distributions of all four mechanical response variables, then cover the experimental results for a single property at a time. This section will conclude with an overall summary of the effects of the experimental factors.

Mechanical Property Distributions As described at the beginning of Section 6.4, a distribution of results for each of the variables in Table 6.9 was generated and then compared to a transformed data set. Table 6.10 lists the name of the transformed data set for each property. Each transformed data set is unitless, with the values representing the data point's distance in standard deviations from the original sample data mean. In analyzing each data set as a whole, it was clear that the distribution of the parameter values was bimodal, with large standard deviations as a result. Figure 6-4 shows the distributions of all transformed data in Table 6.10. The histograms show bimodality in TYStress, TUTStress and TStrain, while the boxplot shows a large number of outliers for TE.

Examining the experimental design shows the HIP main effect as a common factor in the split distribution and is the most likely reason for the bimodality. Separating the transformed data into sets of specimens that have and have not undergone HIP creates more normal distributions, as seen in Figure F-1 and Figure F-2 in Appendix F. Except for TE, it can

Table 6.10: Mapping of Transformed Data Sets

Mechanical Property	Transformed Data
Yield Tensile Stress	TYStress
Ultimate Tensile Stress	TUTStress
Young's Modulus	TE
Ultimate Strain	TStrain

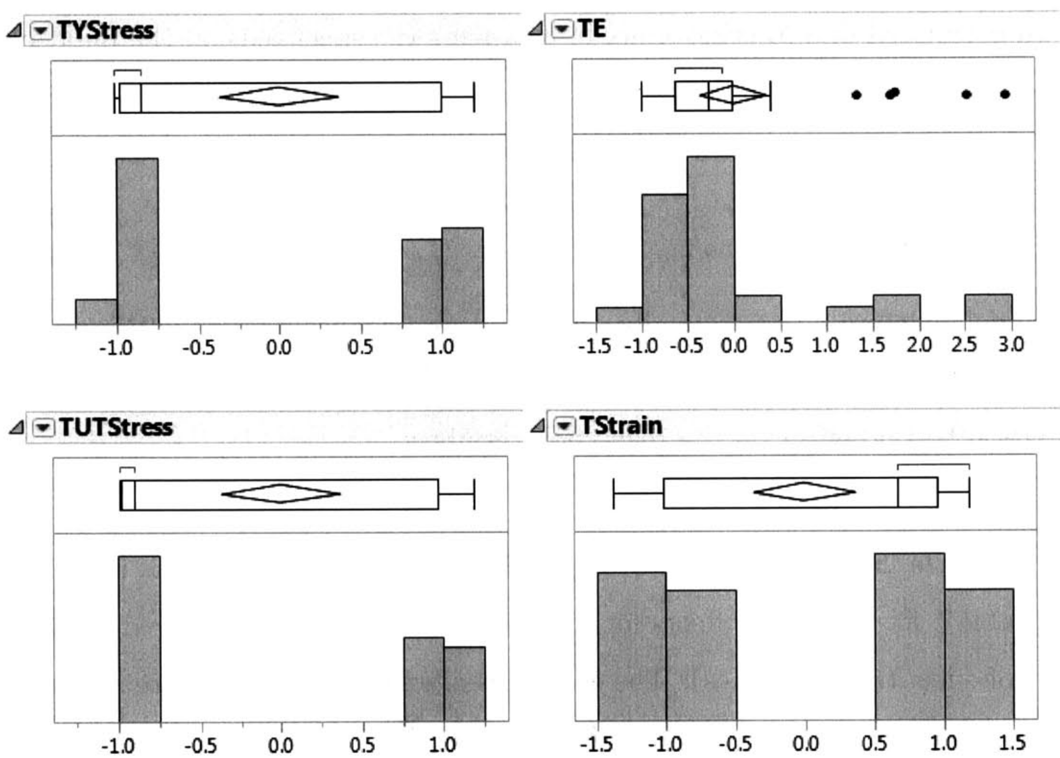

Figure 6-4: Distribution Boxplot and Histograms for Transformed Mechanical Properties

be seen that the peaks of the original bimodal distributions were each almost two standard deviations apart [1 - (-1)]. The outliers can again be seen on the boxplots as black dots. The outliers were examined for any data entry anomalies, but all data was recorded with an automated system. The outlier points have been left in the data set in an absence of any reason to remove them.

Mechanical Property Multiple Regression Models For each transformed mechanical property, an OLS multiple regression model was built using the Fit Model platform in SAS JMP in order to understand the impact of each main effect on that property. This multiple regression model began as a model that used all main effects and second-order interactions, *i.e.* a cross of each of the main effects (HIP * distance from center). On running each regression model, the correlations described in Table 6.7 aliased many of the results together and effects were unable to be separated. Distance from center effects were the most highly aliased and correlated due to constraints in arranging the specimens on the build platform. To reduce this effect, all second-order effects involving the two distance variables were removed. The resulting regression model was then analyzed and additional effects with high p-values were removed until the adjusted R^2 was maximized. In all cases, the completed models had Analysis of Variance (ANOVA) results with low F-test p-values, which indicates that the model's factors are statistically significant as a whole (Barnett, 2015, p. 573). The final regression models were matched against the untransformed data to ensure that the transformation had no effect on the regression results.

Yield Tensile Stress The TYStress multiple regression model was made up of the factors in Table 6.11. All main effects for distance from center were removed because of low probability of effect (high p-values). The estimates of effects in the table are measures of the impact in the model and represent the coefficients each factor would have in a linear equation describing yield tensile stress. Negative values indicate a reduction in yield tensile strength, which indicates that the metal will yield (undergo elastic deformation) sooner under an increasing load. Any p-value below 0.05 indicates a statistically significant contribution to the multiple regression model.

Based on the values in Table 6.11, no HIP had a substantial positive effect on yield tensile stress (indicating HIP had a negative effect). The upright 90° orientation also had a negative effect, while the cylinder coupons had slightly higher yield stresses. The interactions between HIP and the other factors indicate that HIP reduces the individual effects of Z-orientation and cylinder type.

Table 6.11: OLS Multiple Regression Model Parameters for Transformed Yield Tensile Stress

Model Parameter	**Estimate of Effect**	**p-value**
No HIP	0.961	<0.0001
90° Orientation	-0.071	<0.0001
Cylinder Coupon	0.047	<0.0001
90° Orientation * No HIP	-0.041	0.0003
No HIP * Cylinder Coupon	0.025	0.0191
90° Orientation * Cylinder Coupon	-0.020	0.0547

The model's fit statistics can be found in Table 6.12. The model shows very high adjusted R^2 values, indicating a high level of fit between the model and the data. Part of this can come from autocorrelation of two variables — referring back to Table 6.7 shows that the Z-orientation and HIP factors are correlated at 25%. However, this is the only set of correlated factors. Refer to Figure F-3 in Appendix F to see the full results from the multiple regression model.

Table 6.12: Summary of Model Fit for Transformed Yield Tensile Stress

R^2	0.9982
Adjusted R^2	0.9977
RMSE	0.0471
F Ratio	2250.3
F p-value	<0.0001

Ultimate Tensile Stress The TUTStress multiple regression model was made up of the factors in Table 6.13. The distance offset and coupon type main effects were removed because of low probability of effect (high p-values). Negative values indicate a reduction in ultimate tensile strength, which indicates that the metal will reach its maximum stress sooner under an increasing load, and then proceed into a failure mode.

Based on the values in Table 6.13, HIP again had a negative impact on ultimate tensile stress. The upright 90° orientation had a positive effect on ultimate stress, while the cylinder coupons had slightly higher yield stresses. The interaction between HIP and Z-orientation again reduces variation in results for Z-orientation.

Table 6.13: OLS Multiple Regression Model Parameters for Transformed Ultimate Tensile Stress

Model Parameter	**Estimate of Effect**	**p-value**
No HIP	0.986	<0.0001
90° Orientation * No HIP	0.053	<0.0001
90° Orientation	0.035	0.0026
90 mm Distance from Center	-0.026	0.0243

The model's fit statistics can be found in Table 6.14. The model again shows very high adjusted R^2 values, indicating a high level of fit between the model and the data. Correlation of Z-orientation and HIP factors may still be a factor in the overall fit. Refer to Figure F-4 to see the full results from the multiple regression model.

Table 6.14: Summary of Model Fit for Transformed Ultimate Tensile Stress

R^2	0.9977
Adjusted R^2	0.9973
RMSE	0.0512
F Ratio	2847.7
F p-value	<0.0001

Young's Modulus The TE multiple regression model was made up of the factors in Table 6.15. Only the coupon type was considered significant in the model. The other factors were left in because they contributed to the overall fit of the model, but were not significant on their own, indicating a high degree of autocorrelation in those factors. Most of the main effects remained in the model, but the only interaction effect that was close to significant was Z-orientation with HIP (likely due to correlation of Z-orientation and HIP factors). The main factor HIP was also not considered a significant effect. Negative values indicate a reduction in Young's modulus, the relationship between stress and strain demonstrated in Equation 6.1, where E is Young's modulus in MPa, σ is the applied stress in MPa and ε is the strain measured in elongation over total length (%).

$$\sigma = E\varepsilon \tag{6.1}$$

Lower values of E indicate lower stiffness. Within the elastic regime of metal deformation, metals with lower E values will elongate more for a given applied stress. Based on the values in Table 6.15, the cylinder coupon had a higher value of Young's modulus. It is not clear, however, that these are conclusive results given the low fit of the model. Specimens of different coupon type had to be tested on different uniaxial tensile testers, which could account for some of the variation.

Table 6.15: OLS Multiple Regression Model Parameters for Transformed Young's Modulus

Model Parameter	**Estimate of Effect**	**p-value**
Cylinder Coupon	0.431	0.0104
90° Orientation * No HIP	0.340	0.0553
90 mm Distance from Center	-0.289	0.0966
15 mm Distance Offset	0.266	0.1376
90° Orientation	-0.191	0.2809

The model's fit statistics can be found in Table 6.16. This multiple regression shows low R^2 values, indicating a poor fit. Refer to Figure F-5 to see the full results from the multiple regression model.

Table 6.16: Summary of Model Fit for Transformed Young's Modulus

R^2	0.5164
Adjusted R^2	0.4196
RMSE	0.7618
F Ratio	5.3
F p-value	0.0018

Ultimate Strain The TStrain multiple regression model was made up of the factors in Table 6.17. All distance main effects were removed because of low probability of effect (high p-values). Coupon type interactions were also removed from the model. Negative values indicate lower ductility and higher stiffness.

Based on the values in Table 6.17, HIP had a positive impact on ultimate strain. HIP creates a more ductile metal. The upright 90° orientation had a negative effect on ultimate strain, while the cylinder coupons had slightly higher ultimate strain values. The interaction between HIP and Z-orientation again reduces variation in results for Z-orientation.

Table 6.17: OLS Multiple Regression Model Parameters for Transformed Ultimate Strain

Model Parameter	**Estimate of Effect**	**p-value**
No HIP	-0.994	<0.0001
Cylinder Coupon	0.099	0.0004
90° Orientation * No HIP	-0.068	0.0127
90° Orientation	-0.058	0.0237

The model's fit statistics can be found in Table 6.18. This model, like two of the others, shows very high adjusted R^2 values, indicating a high level of fit. Correlation of Z-orientation and HIP factors may still be a factor in the overall fit. Refer to Figure F-6 to see the full results from the multiple regression model.

Table 6.18: Summary of Model Fit for Transformed Ultimate Strain

R^2	0.9859
Adjusted R^2	0.9837
RMSE	0.1277
F Ratio	453.7
F p-value	<0.0001

Summary of Mechanical Behavior Several of the factors in this experiment have a significant effect on the mechanical properties of the final product. Aerospace applications are often driven by high yield stress, ultimate stress and Young's modulus, combined with lower ultimate strain. These requirements seek out stiffer, higher-strength metals, as long as fracture characteristics are still acceptable. For example, a metal with high stress tolerance that fractured under small values of strain would be unsuitable for many aerospace (and other industry) applications.

The previous paragraphs described the effect of multiple factors on the tested mechanical properties, but the results should also be quantitatively compared to a standard. Coupon type, HIP and Z-orientation all have statistically significant effects on at least one mechanical property. Tables 6.19, 6.20 and 6.21 contain the mean values for each of the four mechanical properties, separated by the low and high values for HIP, Z-orientation and coupon type, respectively. The mean values are compared against the manufacturer's estimated capability of components built with the metal powder used in this experiment, with the manufacturer's specifications having a value of 1. The experimental mean values are reported as ranges of the 95% confidence interval (CI)[2] around the mean, μ.

Table 6.19: Mechanical Properties of Experimental Samples Separated on Factor HIP, Compared to Manufacturer Specification = 1

	95% CI of Mean	
Mechanical Property	**No HIP**	**HIP**
Yield Tensile Stress	$1.02 \le \mu \le 1.06$	$0.38 \le \mu \le 0.40$
Ultimate Tensile Stress	$1.08 \le \mu \le 1.12$	$0.42 \le \mu \le 0.43$
Young's Modulus	$0.92 \le \mu \le 1.09$	$0.88 \le \mu \le 1.10$
Ultimate Strain	$2.11 \le \mu \le 2.68$	$8.18 \le \mu \le 8.74$

Coupon type, HIP and Z-orientation all affect the values of yield stress and ultimate stress. The thin rectangle coupon type, with its lower cross-sectional area per layer, decreased yield and ultimate stress values by approximately 5%, while building coupons at the 90° Z-orientation decreased yield and ultimate stresses by ~25%. HIP had an extremely negative impact on both stress values for all samples, decreasing yield and ultimate stresses by approximately 60%. HIP also had the ability to reduce variation induced from other sources, but its penalty on performance for doing so is very high.

Table 6.20: Mechanical Properties of Experimental Samples Separated on Factor Z-Orientation, Compared to Manufacturer Specification = 1

	95% CI of Mean	
Mechanical Property	**0° Z-Orientation**	**90° Z-Orientation**
Yield Tensile Stress	$0.64 \le \mu \le 0.99$	$0.42 \le \mu \le 0.76$
Ultimate Tensile Stress	$0.67 \le \mu \le 1.02$	$0.46 \le \mu \le 0.85$
Young's Modulus	$0.94 \le \mu \le 1.14$	$0.87 \le \mu \le 1.03$
Ultimate Strain	$3.20 \le \mu \le 6.41$	$4.55 \le \mu \le 8.04$

Young's modulus values did not appear to have any significant modeled effects, but it is a derived value from the strain and stress. This likely accounts for why no significant effects

[2]Several of the confidence intervals between factor levels do overlap in the tables. This is likely due to comparing factors one at a time. The ANOVA completed earlier showed that each of the factors discussed in this section had significant effects on specific mechanical properties once compensation for variation from other factors was complete.

were found, except for the coupon type; the thin rectangle coupon type decreased Young's modulus by ~15%. It is possible that the different machines used to test each coupon type induced this difference.

Table 6.21: Mechanical Properties of Experimental Samples Separated on Factor Coupon Type, Compared to Manufacturer Specification = 1

	95% CI of Mean	
Mechanical Property	**Cylinder Coupon**	**Thin Rectangle Coupon**
Yield Tensile Stress	$0.55 \leq \mu \leq 0.91$	$0.50 \leq \mu \leq 0.87$
Ultimate Tensile Stress	$0.58 \leq \mu \leq 0.96$	$0.54 \leq \mu \leq 0.92$
Young's Modulus	$0.97 \leq \mu \leq 1.14$	$0.87 \leq \mu \leq 0.93$
Ultimate Strain	$3.96 \leq \mu \leq 7.36$	$3.64 \leq \mu \leq 7.13$

Ultimate strain results demonstrated mostly opposite effects to stress results. HIP increased the ultimate strain by approximately 250%, which prevents fracture failure at lower loads but also increases dimensional noncompliance as loads increase. Ultimate strain decreased by ~5% with the thin rectangle coupon and increased by ~30% in the 90° orientation, but this may be due to lower ultimate stress — they broke before reaching higher strain.

Chapter 7

Conclusions & Recommendations

Metal additive manufacturing (AM) is a manufacturing technology that is still very much in the early stages of development. An aerospace company that desires to incorporate metal AM components onto flight hardware in the near future has many challenges to solve. This project and resulting thesis present information on the path forward for Raytheon's leadership in manufacturing and provide additional insight on the capabilities of their current equipment using well-known analysis methods. This section is intended to deliver a review of the thesis as a whole, including the overall conclusions from a review of AM qualification efforts and the results from the experiments executed on aluminum AM equipment. In addition, this section is intended to suggest recommendations for near-term improvements and long-term strategic changes that Raytheon can use to support metal AM for more of its programs and products.

7.1 Qualification Process

The qualification process discussed in this thesis has provided genuine value for Raytheon, as demonstrated by the successful inclusion of metal AM through the PDR stage of a current program at RMS. The three-phase approach—process capability, design feasibility and IV&V—engages many different aspects of Raytheon's manufacturing engineering capability. This process provides the company, and others who may follow in its footsteps, the opportunity to merge a well-defined business process with best practices provided from existing research. This process and the experimental practice that Raytheon Missile Systems has gained during the duration of the project have demonstrably moved forward the manufacturing readiness of metal AM for some applications.

7.2 Experimental Results

The experimental design explained in this thesis has demonstrated that there are many possible ways to handle even complex experimental designs within the body of statistics knowledge. The experiment as presented here provides a complete template for future work to be completed on metal AM qualification, both as a follow-on to this experiment and for other experiments on different critical factors. With the results thus, some information has been revealed about the factors chosen in the experiment. Dimensional accuracy is still a challenge for this generation of metal AM equipment, with variation from design dimensions (on the order of several hundred microns) that exceeds most easily-achievable machining tolerances. Not many of the factors tested appear to have an effect on the accuracy of the AM production process, but the ones that do provide an area of focus for future experiments as well as indicate improvements for current best practices. Orientation of the component in the Z-direction may be the only factor that has a noticeable effect. Surface roughness values still exceed values that machining can reach, with the experiment showing no significant effects from the tested factors.

However, three of the tested factors – hot isostatic pressing (HIP), Z-orientation and coupon type – have significant effects on the mechanical property results, as hypothesized. HIP reduces the strength of the AM-produced metals by nearly 60% while increasing its elongation under stress by 250%. This is a process that will not be valid for use as a final metallurgical state in most of Raytheon's processes for aerospace parts. However, HIP can also be used as a precursor to other hardening and heat treatment procedures that would result in different mechanical properties than seen here. HIP is a versatile process that was constrained in its application for this experiment, and other opportunities may exist in the future to test the possibility of incorporating HIP in the overall heat treatment process. Designers will also want to pay attention to ensuring that high-stress axes are not aligned in the direction of layer deposition, and that AM-built components are oriented to maximize the surface area of each layer.

7.3 Suggestions for Immediate Implementation

Beyond developing a qualification process and demonstrating its capability to improve understanding of metal AM, this thesis seeks to provide an executable action plan for leadership at many levels within Raytheon Missile Systems. As demonstrated in Section 6.4, the field of experimental design is capable of revealing relationships between manufacturing parameters that are not easily separated. Screening experiments form only the first layer within the field of Design of Experiments (DoE), and there is considerable opportunity to apply these techniques to many other problems within the manufacturing technology development areas at Raytheon. AM development in particular may be augmented by using the experiments completed here to focus on some experimental factors and ignore others altogether, improving the overall efficiency of testing to understand the capability of the AM equipment. Elements of the experiment documented here could be reused in a response surface methodology (RSM) design. RSM designs have a higher resolution for a smaller number of factors to be able to extract nonlinear relationships of factors and responses. Using this project as a case study to educate other engineers on the promise of experimental design will be instrumental in adding to this project's legacy over time.

As the amount of testing increases within the AM development groups, they should ensure that they are testing the right things, or at least that which the academic and scientific communities believe to be most critical at this point in time. To that end, further metal AM testing should include incorporation of the NIST standard AM test artifact into future testing. A review of qualification for rapid prototyping tools suggested that test artifacts are essentially the only way to qualify AM equipment with a standard—current AM equipment lacks the functional degrees of freedom to be able to do individual tests, like those used to assess and calibrate a CNC machine (Scaravetti et al., 2008). As shown earlier, the NIST artifact is a freely available design that meets most of the feature requirements for testing that are needed now. The artifact developed by Moylan et. al already has a complete STL file ready for AM production and also includes a checklist of areas to examine for issues after the AM build is complete (Moylan et al., 2014). The artifact can almost be "read" like a report for issues that occur during AM processes, and is widely applicable to a number of AM processes. Adopting this free piece of technology from NIST is an easy and low-risk method of seriously improving the capability to assess the performance of existing AM systems.

The final suggestion for immediate implementation is to begin studying the organizational implications of incorporating metal AM and other digital manufacturing technologies into the business' capabilities. Metal AM is caught at the moment between a technology push and a market pull: customers are demanding more capabilities than ever from metals while AM technology producers are trying to shift the reputation of AM as a prototyping technology only (Mellor et al., 2014). Raytheon is already completing multiple AM evaluation projects at different sites located in time zones across the country, and the logistical complexity will only increase as the investment in AM equipment and qualification activities reaches higher levels. It's important to decide early on whether a strategy of early, costly investment will pay dividends on winning customer business later, or whether AM should wait until the customer demands the technology above all else. In either case, leadership around AM technology should focus on getting early "wins" in order to rally support within the company and begin developing the engineering support structure needed to adopt AM manufacturing techniques.

7.4 Long-Term Goals

In order to support metals AM in Raytheon long-term, regardless of the immediate tactical decision made, there are some specific strategies that require investment from prime aerospace contractors and their suppliers. The first (and likely most critical) is support for physics-based modeling of AM processes. Many of the tools used by engineers to model the design and construction process of complex mechanical systems are unable to adequately predict the mechanical properties of materials produced with AM. Considering there are decades, if not hundreds, of years of experience with the behavior of wrought, cast and machined metals, industry will require a substantial amount of time to build up the same experience with AM. In addition, a significant upgrade in computing capability will be required to simulate the effective strengths and life expectancy of components made with 1000s of combined layers. Engineering leadership at Raytheon has identified this as a major blocking point to incorporating metal AM into future designs. Investment into software design capabilities that put AM on parity with other forms of manufacturing are the first step to driving adoption within the aerospace industry.

A leap forward in nondestructive testing and defect analysis must also accompany the development of new simulation techniques. While many of the defect modes inherent in metal AM are well defined in research and academia, there are few analysis methods ready for market and use in a manufacturing line. Infrared computed tomography and ultrasonic probing provide the most hope for developing new techniques soon, but these methods are often difficult to implement for manufacturability or ease of testing on products that are made consistently. As new technology is developed for metal AM, companies must also develop ways to assure customers that products made with this new technology will work or operate to the required and intended specifications.

The final suggestion for long-term research is to invest more effort in creating process monitoring for AM processes as they are built. Process capability only exists by engineers' ability to understand a process as it exists today and assess its variations for hidden problems. Process control techniques and metrics are key to being able to produce components made with AM reliably and in a repeatable way.

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Appendix A

Material Properties Assessment

This appendix describes the tests defined by Raytheon Materials & Practices (M&P) as necessary to assess the material properties of a new metal AM process. Each test is either defined qualitatively by name or defined by the expected quantity and test directions required. All tests are accompanied by their respective specifications.

Table A.1: Tests Required for Process Capability Material Properties Assessment. Courtesy of Raytheon M&P division.

Material Properties			**Test Direction**	**Test Specification**
Non-Destructive Evaluation	X-Ray		n/a	ASTM E1742
	Dye Penetrant		n/a	ASTM E1417
Physical Properties	Density	g/cm^3	n/a	ASTM B311
	Composition		n/a	OES
	Tensile Fracture SEM Documentation		X, Y, Z	
	Microstructure		X, Y, Z	ASTM E3
Mechanical Properties	Ultimate Tensile Strength	MPa	X, Y, Z	ASTM E8
	Tensile Yield Strength	MPa		
	Modulus of Elasticity	MPa		
	Elongation	%		
	Reduction in Area	%		
	Hardness			ASTM E18

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Appendix B

Defect Criteria

This appendix describes the defects expected to result from metal AM manufacturing processes. Each entry has a definition that assists in developing test standards to detect the defect. Most of the listed defects are similar to those found in legacy manufacturing processes.

Table B.1: List of Defect Criteria, Courtesy of Raytheon M&P division

Defect	**Definition**
Void	Air entrapment within the material, usually spherical in shape.
Blister	Rounded elevation of the surface of the material, with boundaries that may be more or less sharply defined.
Chip	A small piece of parent material broken off an edge or surface.
Crack	An actual separation of the material, visible on opposite surfaces, and extending through the thickness.
Foreign Object Inclusion	Any object in the material foreign to its composition.
Porosity	Presence of numerous visible pits (pinholes).
Scratches or Gouges	Shallow mark, groove, furrow, or channel.
Burns	Large regions characterized by discoloration and warping.
Unconsolidated Powder	Any inclusions or regions of the material where the raw material powder has not fused to itself or the material bulk.

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Appendix C

Factorial Experiment Examples

This appendix provides examples for multiple formats of representing 2^n factorial experiments. The order of experiments has already been randomized in the tables below; the run column indicates the order in which to run each trial. -1 and 1 indicate low and high values, respectively for each of the factors in the column headers. There is an example of a standard factorial, as well as one that has been designed as a split-plot experiment.

Table C.1: Example of coded runs for a 2^5 full factorial experiment

Run	Pattern	Configuration	Orientation	Location	HIP	Powder
1	++−+−	1	1	-1	1	-1
2	− − − −+	-1	-1	-1	-1	1
3	++− −+	1	1	-1	-1	1
4	+++++	1	1	1	1	1
5	+++−+	1	1	1	-1	1
6	−+−++	-1	1	-1	1	1
7	−++− −	-1	1	1	-1	-1
8	+−++−	1	-1	1	1	-1
9	++−++	1	1	-1	1	1
10	− −++−	-1	-1	1	1	-1
11	−++−+	-1	1	1	-1	1
12	+− − − −	1	-1	-1	-1	-1
13	++− − −	1	1	-1	-1	-1
14	+—+−	1	-1	-1	1	-1
15	−+−+−	-1	1	-1	1	-1
16	+−+++	1	-1	1	1	1

Run	Pattern	Configuration	Orientation	Location	HIP	Powder
17	− − − − −	-1	-1	-1	-1	-1
18	++++−	1	1	1	1	-1
19	−++++	-1	1	1	1	1
20	−+− − −	-1	1	-1	-1	-1
21	− −+− −	-1	-1	1	-1	-1
22	− −+++	-1	-1	1	1	1
23	− − −++	-1	-1	-1	1	1
24	+−+−+	1	-1	1	-1	1
25	+−+− −	1	-1	1	-1	-1
26	− −+−+	-1	-1	1	-1	1
27	+− − −+	1	-1	-1	-1	1
28	+++− −	1	1	1	-1	-1
29	−+− −+	-1	1	-1	-1	1
30	− − −+−	-1	-1	-1	1	-1
31	+− −++	1	-1	-1	1	1
32	−+++−	-1	1	1	1	-1

Table C.2: Example of coded runs for a 2^5 split-plot experiment, split on powder type

Run	Configuration	Orientation	Location	HIP	Powder
1	-1	1	1	1	1
2	-1	-1	1	1	1
3	1	1	1	1	1
4	1	1	1	-1	1
5	-1	1	-1	1	1
6	1	-1	1	-1	1
7	1	-1	1	1	1
8	1	1	-1	1	1
9	-1	1	-1	-1	1
10	-1	-1	-1	1	1
11	1	-1	-1	-1	1
12	1	1	-1	1	1
13	-1	-1	1	-1	1
14	1	-1	-1	-1	1
15	-1	-1	-1	-1	1
16	-1	1	1	-1	1
17	1	1	-1	-1	-1
18	1	1	1	1	-1
19	1	-1	-1	-1	-1
20	-1	-1	1	-1	-1
21	-1	-1	-1	1	-1
22	1	1	-1	-1	-1
23	1	-1	1	1	-1
24	-1	-1	-1	1	-1
25	-1	1	-1	-1	-1
26	1	-1	1	1	-1
27	1	-1	1	-1	-1
28	-1	-1	-1	1	-1
29	1	1	-1	1	-1
30	-1	1	1	-1	-1
31	-1	1	1	-1	-1
32	-1	1	1	1	-1

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Appendix D

Test Matrix

This appendix is the record of the experimental design for the experiment proposed in this thesis project. Each of the factor levels, previously coded as -1 and 1, have been replaced with their true values. Block and Replicate columns indicate which experimental build contained each trial. Only the first half of the split-plot experiment (the first 32 rows) were within the execution scope of this project.

Table D.1: Complete Experimental Design for Phase 1 with Non-Coded Factor Levels

Run	Block	Replicate	Powder Type	Z-Orientation (°)	Distance from Center (mm)	Distance Offset (mm)	Actual Distance (mm)	HIP	Coupon Type
1	1	1	Virgin	90	90	15	105	Y	Cylinder
2	1	1	Virgin	90	90	-15	75	N	Thin Rectangle
3	1	1	Virgin	0	30	15	45	N	Thin Rectangle
4	1	1	Virgin	90	30	-15	15	N	Cylinder
5	1	1	Virgin	0	30	-15	15	N	Cylinder
6	1	1	Virgin	0	30	15	45	Y	Cylinder
7	1	1	Virgin	0	90	-15	75	Y	Thin Rectangle
8	1	1	Virgin	90	90	15	105	Y	Thin Rectangle
9	2	1	Virgin	90	90	15	105	Y	Thin Rectangle
10	2	1	Virgin	0	90	-15	75	N	Thin Rectangle
11	2	1	Virgin	0	30	15	45	N	Thin Rectangle
12	2	1	Virgin	0	30	-15	15	N	Cylinder
13	2	1	Virgin	0	30	-15	15	Y	Cylinder
14	2	1	Virgin	90	30	15	45	Y	Thin Rectangle
15	2	1	Virgin	90	90	-15	75	N	Cylinder
16	2	1	Virgin	90	90	15	105	Y	Cylinder
17	3	2	Virgin	90	90	15	105	Y	Cylinder
18	3	2	Virgin	90	90	-15	75	N	Thin Rectangle
19	3	2	Virgin	0	30	15	45	N	Thin Rectangle
20	3	2	Virgin	90	30	-15	15	N	Cylinder
21	3	2	Virgin	0	30	-15	15	N	Cylinder
22	3	2	Virgin	0	30	15	45	Y	Cylinder
23	3	2	Virgin	0	90	-15	75	Y	Thin Rectangle
24	3	2	Virgin	90	90	15	105	Y	Thin Rectangle
25	4	2	Virgin	90	90	15	105	Y	Thin Rectangle
26	4	2	Virgin	0	90	-15	75	N	Thin Rectangle
27	4	2	Virgin	0	30	15	45	N	Thin Rectangle
28	4	2	Virgin	0	30	-15	15	N	Cylinder
29	4	2	Virgin	0	30	-15	15	Y	Cylinder
30	4	2	Virgin	90	30	15	45	Y	Thin Rectangle
31	4	2	Virgin	90	90	-15	75	N	Cylinder
32	4	2	Virgin	90	90	15	105	Y	Cylinder

Run	Block	Replicate	Powder Type	Z-Orientation (°)	Distance from Center (mm)	Distance Offset (mm)	Actual Distance (mm)	HIP	Coupon Type
33	5	1	Recycled	0	90	15	105	N	Cylinder
34	5	1	Recycled	0	90	-15	75	Y	Thin Rectangle
35	5	1	Recycled	90	30	15	45	N	Thin Rectangle
36	5	1	Recycled	90	30	-15	15	Y	Cylinder
37	5	1	Recycled	0	30	-15	15	Y	Thin Rectangle
38	5	1	Recycled	0	30	15	45	N	Cylinder
39	5	1	Recycled	90	90	-15	75	Y	Cylinder
40	5	1	Recycled	90	90	15	105	N	Thin Rectangle
41	6	1	Recycled	90	90	15	105	N	Cylinder
42	6	1	Recycled	0	90	-15	75	N	Thin Rectangle
43	6	1	Recycled	90	30	15	45	Y	Thin Rectangle
44	6	1	Recycled	90	30	-15	15	Y	Thin Rectangle
45	6	1	Recycled	90	30	-15	15	N	Cylinder
46	6	1	Recycled	0	30	15	45	Y	Cylinder
47	6	1	Recycled	0	90	-15	75	N	Thin Rectangle
48	6	1	Recycled	0	90	15	105	Y	Cylinder
49	7	2	Recycled	0	90	15	105	N	Cylinder
50	7	2	Recycled	0	90	-15	75	Y	Thin Rectangle
51	7	2	Recycled	90	30	15	45	N	Thin Rectangle
52	7	2	Recycled	90	30	-15	15	Y	Cylinder
53	7	2	Recycled	0	30	-15	15	Y	Thin Rectangle
54	7	2	Recycled	0	30	15	45	N	Cylinder
55	7	2	Recycled	90	90	-15	75	Y	Cylinder
56	7	2	Recycled	90	90	15	105	N	Thin Rectangle
57	8	2	Recycled	90	90	15	105	N	Cylinder
58	8	2	Recycled	0	90	-15	75	N	Thin Rectangle
59	8	2	Recycled	90	30	15	45	Y	Thin Rectangle
60	8	2	Recycled	90	30	-15	15	Y	Thin Rectangle
61	8	2	Recycled	90	30	-15	15	N	Cylinder
62	8	2	Recycled	0	30	15	45	Y	Cylinder
63	8	2	Recycled	0	90	-15	75	N	Thin Rectangle
64	8	2	Recycled	0	90	15	105	Y	Cylinder

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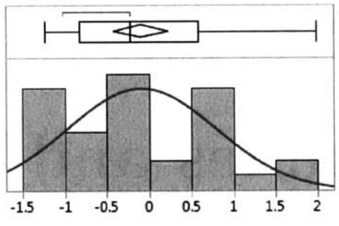
Appendix E

Dimensional Accuracy and Surface Roughness Data Analysis Tables and Figures

This appendix contains the output from Distribution and Fit Model platforms from SAS JMP for each of the experimental values recorded for dimensional accuracy and surface roughness. All data has been transformed through normalizing; therefore, each graph is unitless and values represent the number of standard deviations away from the untransformed mean of the data set. This transformation retains the same distribution and multiple regression results as the untransformed data.

TError X-BR



Quantiles

100.0%	maximum	1.9794748399
99.5%		1.9794748399
97.5%		1.9794748399
90.0%		1.1375716168
75.0%	quartile	0.5763028015
50.0%	median	-0.225509792
25.0%	quartile	-0.826869237
10.0%		-1.027322385
2.5%		-1.227775534
0.5%		-1.227775534
0.0%	minimum	-1.227775534

Summary Statistics

Mean	-0.089719
Std Dev	0.8758787
Std Err Mean	0.1573125
Upper 95% Mean	0.231556
Lower 95% Mean	-0.410994
N	31

Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	-0.089719	-0.410994	0.231556
Dispersion	σ	0.8758787	0.6999252	1.1707638

-2log(Likelihood) = 78.757475145835

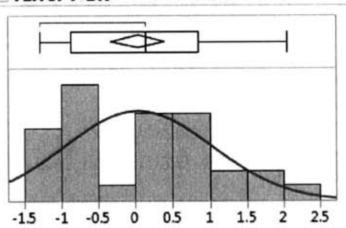
Goodness-of-Fit Test

Shapiro-Wilk W Test

W	Prob<W
0.917503	0.0203*

Note: Ho = The data is from the Normal distribution. Small p-values reject Ho.

TError Y-BR



Quantiles

100.0%	maximum	2.0569992379
99.5%		2.0569992379
97.5%		2.0569992379
90.0%		1.5911680259
75.0%	quartile	0.8417873804
50.0%	median	0.1329137969
25.0%	quartile	-0.879762751
10.0%		-1.163312184
2.5%		-1.28483337
0.5%		-1.28483337
0.0%	minimum	-1.28483337

Summary Statistics

Mean	0.0349128
Std Dev	0.996508
Std Err Mean	0.1789781
Upper 95% Mean	0.4004349
Lower 95% Mean	-0.330609
N	31

Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	0.0349128	-0.330609	0.4004349
Dispersion	σ	0.996508	0.7963214	1.3320057

-2log(Likelihood) = 86.7573051056503

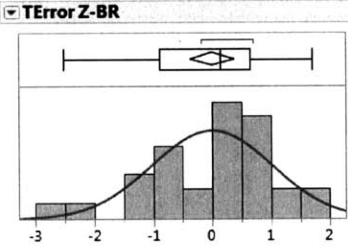
Goodness-of-Fit Test

Shapiro-Wilk W Test

W	Prob<W
0.925056	0.0322*

Note: Ho = The data is from the Normal distribution. Small p-values reject Ho.

TError Z-BR



Quantiles

100.0%	maximum	1.699969911
99.5%		1.699969911
97.5%		1.699969911
90.0%		1.4229377773
75.0%	quartile	0.6296184855
50.0%	median	0.1259236971
25.0%	quartile	-0.88146588
10.0%		-1.30960645
2.5%		-2.518473942
0.5%		-2.518473942
0.0%	minimum	-2.518473942

Summary Statistics

Mean	-0.006093
Std Dev	1.015926
Std Err Mean	0.1824657
Upper 95% Mean	0.3665516
Lower 95% Mean	-0.378738
N	31

Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	-0.006093	-0.378738	0.3665516
Dispersion	σ	1.015926	0.8118386	1.3579613

-2log(Likelihood) = 87.9538230277615

Goodness-of-Fit Test

Shapiro-Wilk W Test

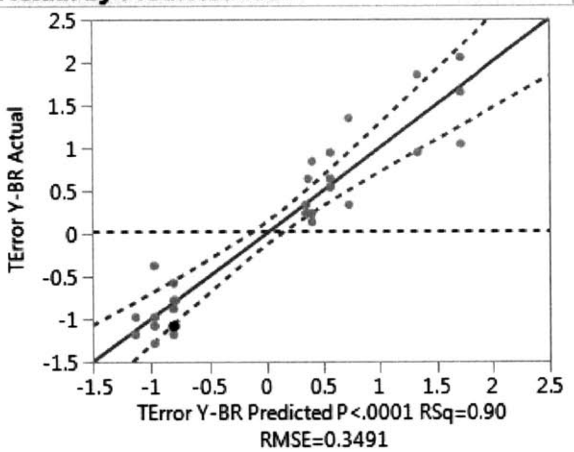
W	Prob<W
0.956275	0.2319

Note: Ho = The data is from the Normal distribution. Small p-values reject Ho.

Figure E-1: Distribution Analysis for the Transformed Error (TError) of X-, Y- and Z-BR

Response TError Y-BR

Actual by Predicted Plot



Summary of Fit

RSquare	0.901811
RSquare Adj	0.877264
Root Mean Square Error	0.349114
Mean of Response	0.034913
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	26.865713	4.47762	36.7378
Error	24	2.925132	0.12188	Prob > F
C. Total	30	29.790845		<.0001*

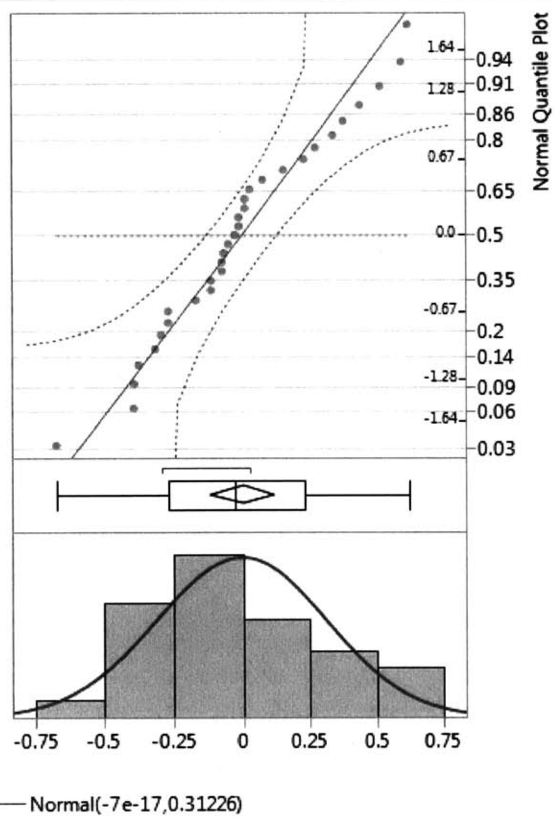
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0353598	0.075145	0.47	0.6422
Z-Orientation (deg from horiz)(0,90)	0.6264911	0.074989	8.35	<.0001*
Distance from Center (mm)(30,90)	0.4289539	0.087937	4.88	<.0001*
Distance Offset (mm)(-15,15)	0.2067755	0.065392	3.16	0.0042*
Z-Orientation (deg from horiz)*Distance from Center (mm)	-0.238652	0.076696	-3.11	0.0048*
Z-Orientation (deg from horiz)*Coupon Type[Cylinder]	-0.287976	0.069548	-4.14	0.0004*
Distance Offset (mm)*Coupon Type[Cylinder]	-0.370239	0.075872	-4.88	<.0001*

Figure E-2: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Error Y-BR

Distributions

Residual TError Y-BR



Quantiles

100.0%	maximum	0.6204849378
99.5%		0.6204849378
97.5%		0.6204849378
90.0%		0.5025547156
75.0%	quartile	0.2313375702
50.0%	median	-0.025130532
25.0%	quartile	-0.267800139
10.0%		-0.38900696
2.5%		-0.672821056
0.5%		-0.672821056
0.0%	minimum	-0.672821056

Summary Statistics

Mean	-6.54e-17
Std Dev	0.312257
Std Err Mean	0.056083
Upper 95% Mean	0.1145368
Lower 95% Mean	-0.114537
N	31

Figure E-3: Distribution Analysis for the Residuals of the Transformed Error Y-BR Multiple Regression Model

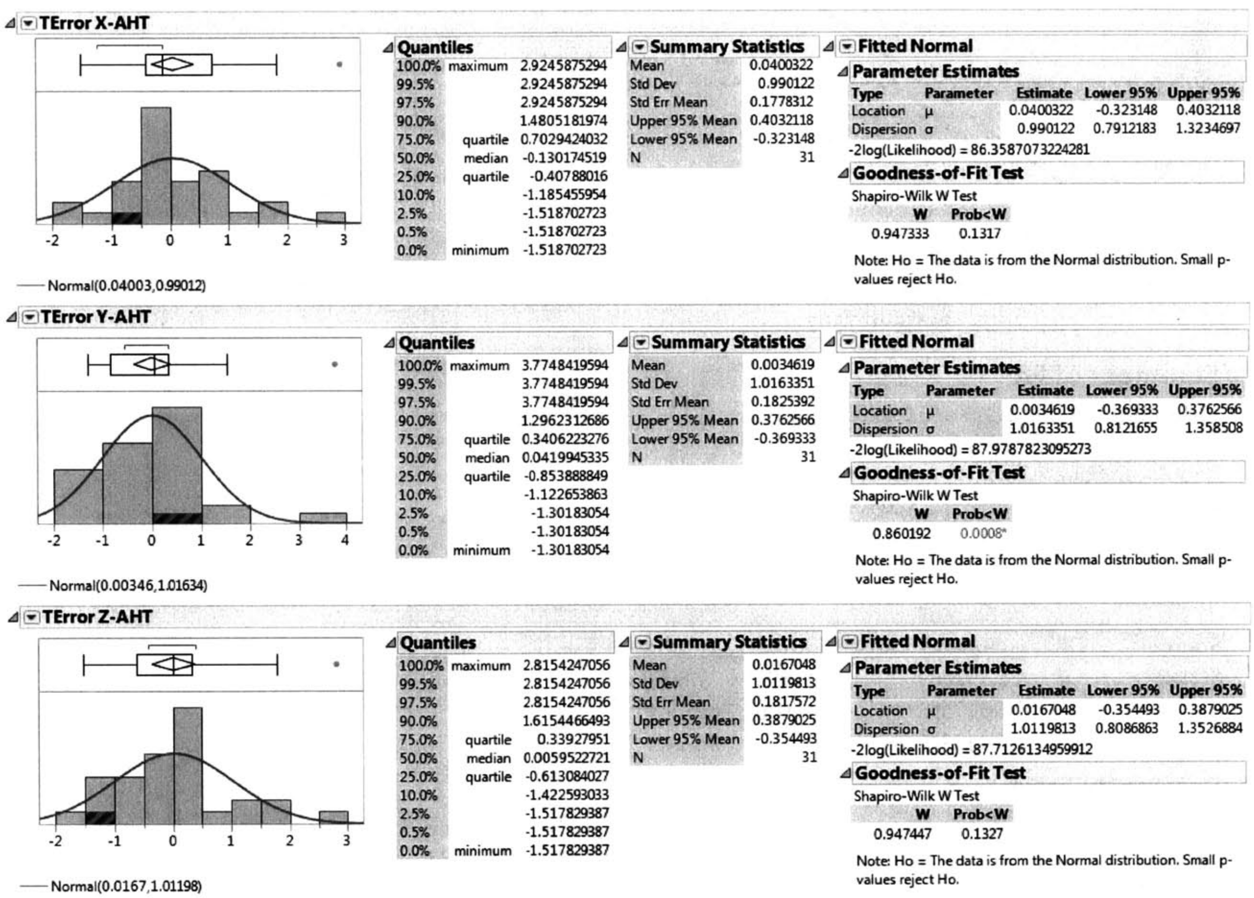
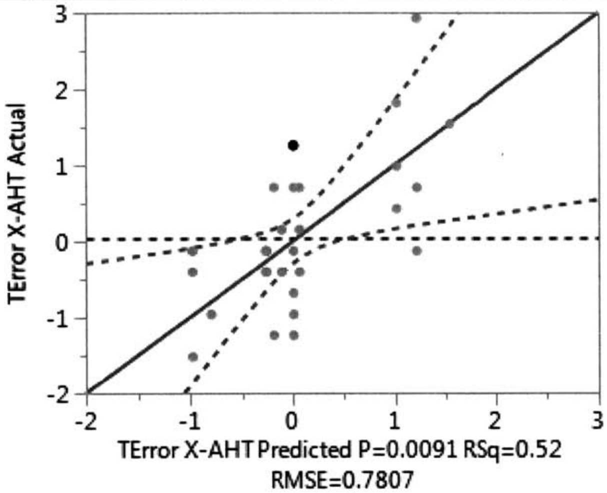


Figure E-4: Distribution Analysis for the Transformed Error (TError) of X-, Y- and Z-AHT

Response TError X-AHT

Actual by Predicted Plot



Summary of Fit

RSquare	0.523378
RSquare Adj	0.37832
Root Mean Square Error	0.780679
Mean of Response	0.040032
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	15.392689	2.19896	3.6080
Error	23	14.017558	0.60946	Prob > F
C. Total	30	29.410246		0.0091*

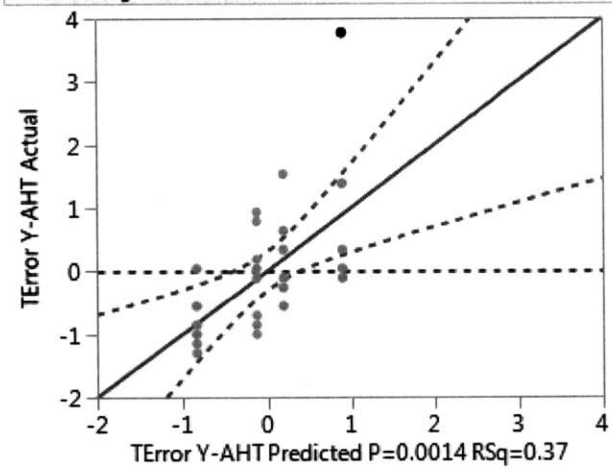
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0282621	0.150644	0.19	0.8528
Z-Orientation (deg from horiz)(0,90)	-0.283125	0.172071	-1.65	0.1135
Distance from Center (mm)(30,90)	0.5027021	0.226169	2.22	0.0363*
Coupon Type[Cylinder]	-0.385815	0.189612	-2.03	0.0536
Z-Orientation (deg from horiz)*Distance Offset (mm)	-0.365599	0.176042	-2.08	0.0492*
Z-Orientation (deg from horiz)*Coupon Type[Cylinder]	-0.484503	0.155812	-3.11	0.0049*
Distance from Center (mm)*Distance Offset (mm)	0.2376358	0.213678	1.11	0.2776
Distance Offset (mm)*Coupon Type[Cylinder]	-0.225488	0.197147	-1.14	0.2645

Figure E-5: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Error X-AHT

Response TError Y-AHT

Actual by Predicted Plot



Summary of Fit

RSquare	0.374661
RSquare Adj	0.329994
Root Mean Square Error	0.83191
Mean of Response	0.003462
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	2	11.610033	5.80502	8.3879
Error	28	19.378078	0.69207	Prob > F
C. Total	30	30.988111		0.0014*

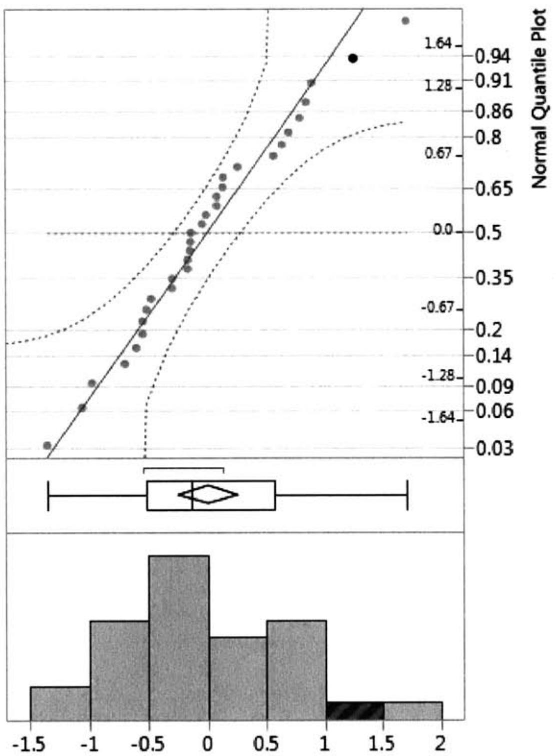
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	0.0313752	0.149576	0.21	0.8354
Z-Orientation (deg from horiz)(0,90)	-0.511979	0.149576	-3.42	0.0019*
Z-Orientation (deg from horiz)*Coupon Type[Cylinder]	-0.353333	0.149576	-2.36	0.0254*

Figure E-6: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Error Y-AHT

Distributions

Residual TError X-AHT



Quantiles

100.0%	maximum	1.7089064352
99.5%		1.7089064352
97.5%		1.7089064352
90.0%		0.8836257522
75.0%	quartile	0.5680506683
50.0%	median	-0.135072733
25.0%	quartile	-0.512738691
10.0%		-0.912648527
2.5%		-1.345855613
0.5%		-1.345855613
0.0%	minimum	-1.345855613

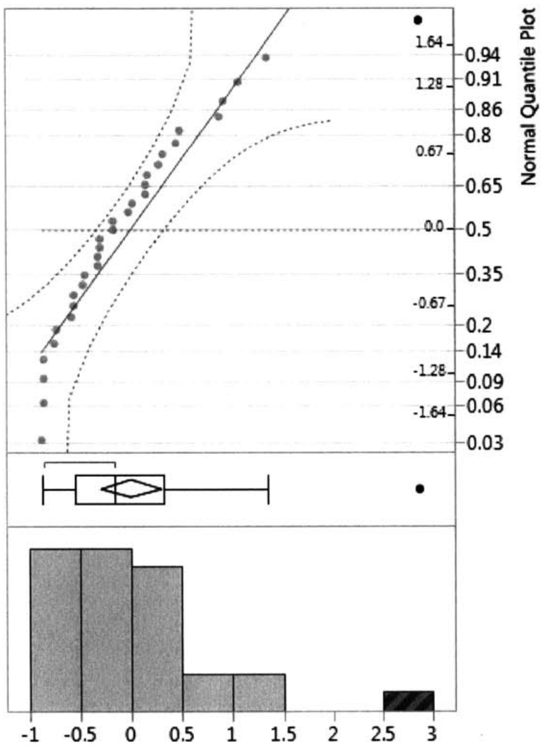
Summary Statistics

Mean	-7.16e-18
Std Dev	0.6835583
Std Err Mean	0.1227707
Upper 95% Mean	0.2507312
Lower 95% Mean	-0.250731
N	31

Figure E-7: Distribution Analysis for the Residuals of the Transformed Error X-AHT Multiple Regression Model

Distributions

Residual TError Y-AHT



Quantiles

100.0%	maximum	2.8781540842
99.5%		2.8781540842
97.5%		2.8781540842
90.0%		1.0352859257
75.0%	quartile	0.31857922
50.0%	median	-0.169265323
25.0%	quartile	-0.556065548
10.0%		-0.854693342
2.5%		-0.875931956
0.5%		-0.875931956
0.0%	minimum	-0.875931956

Summary Statistics

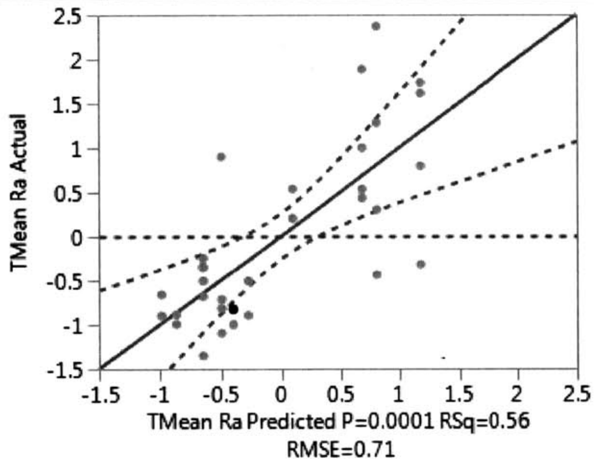
Mean	1.253e-17
Std Dev	0.8037014
Std Err Mean	0.144349
Upper 95% Mean	0.2948001
Lower 95% Mean	-0.2948
N	31

Figure E-8: Distribution Analysis for the Residuals of the Transformed Error Y-AHT Multiple Regression Model

Response TMean Ra

Effect Summary

Actual by Predicted Plot



Summary of Fit

RSquare	0.560889
RSquare Adj	0.495836
Root Mean Square Error	0.710045
Mean of Response	9.02e-17
Observations (or Sum Wgts)	32

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	17.387573	4.34689	8.6220
Error	27	13.612427	0.50416	Prob > F
C. Total	31	31.000000		0.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-0.088961	0.130509	-0.68	0.5013
Distance from Center (mm)(30,90)	0.4789516	0.138118	3.47	0.0018*
Distance Offset (mm)(-15,15)	0.1846835	0.125519	1.47	0.1528
Z-Orientation (deg from horiz)*Distance Offset (mm)	0.3558451	0.142965	2.49	0.0193*
Z-Orientation (deg from horiz)*Coupon Type[Cylinder]	0.2464929	0.138118	1.78	0.0856

Figure E-9: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Mean Ra

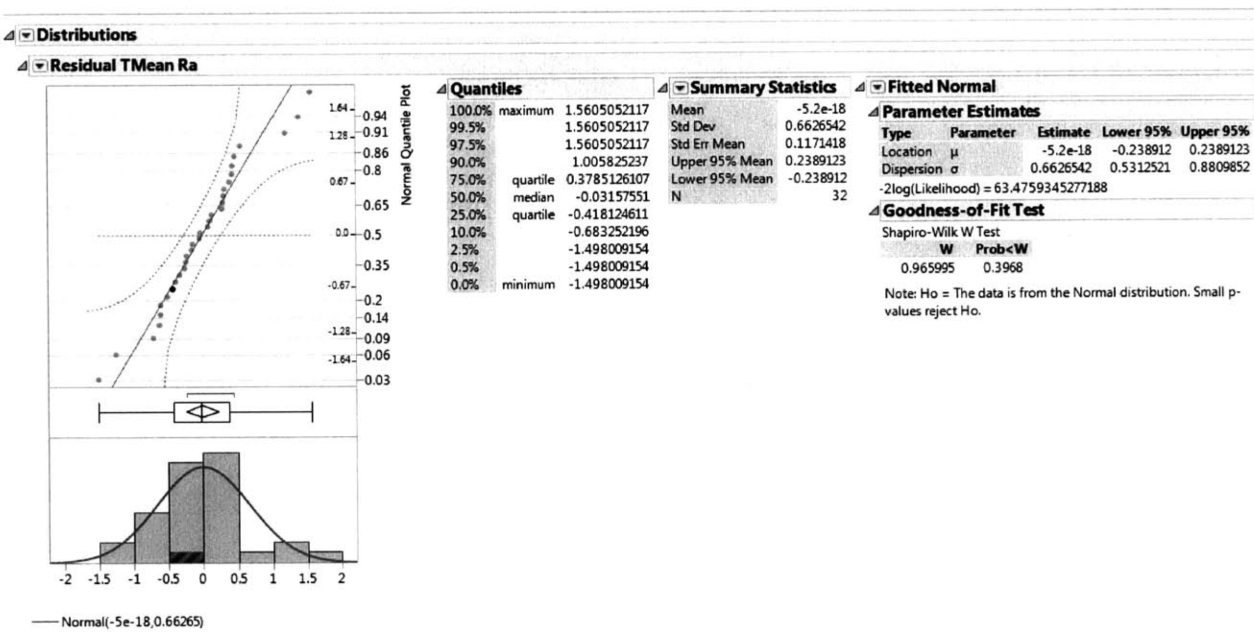


Figure E-10: Distribution Analysis for the Residuals of the Transformed Mean Ra Multiple Regression Model

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Appendix F

Mechanical Properties Data Analysis Tables and Figures

This appendix contains the output from Distribution and Fit Model platforms from SAS JMP for each of the experimental values recorded for yield stress, ultimate stress, Young's modulus and ultimate strain. All data has been transformed through normalizing; therefore, each graph is unitless and values represent the number of standard deviations away from the untransformed mean of the data set. This transformation retains the same distribution and multiple regression results as the untransformed data.

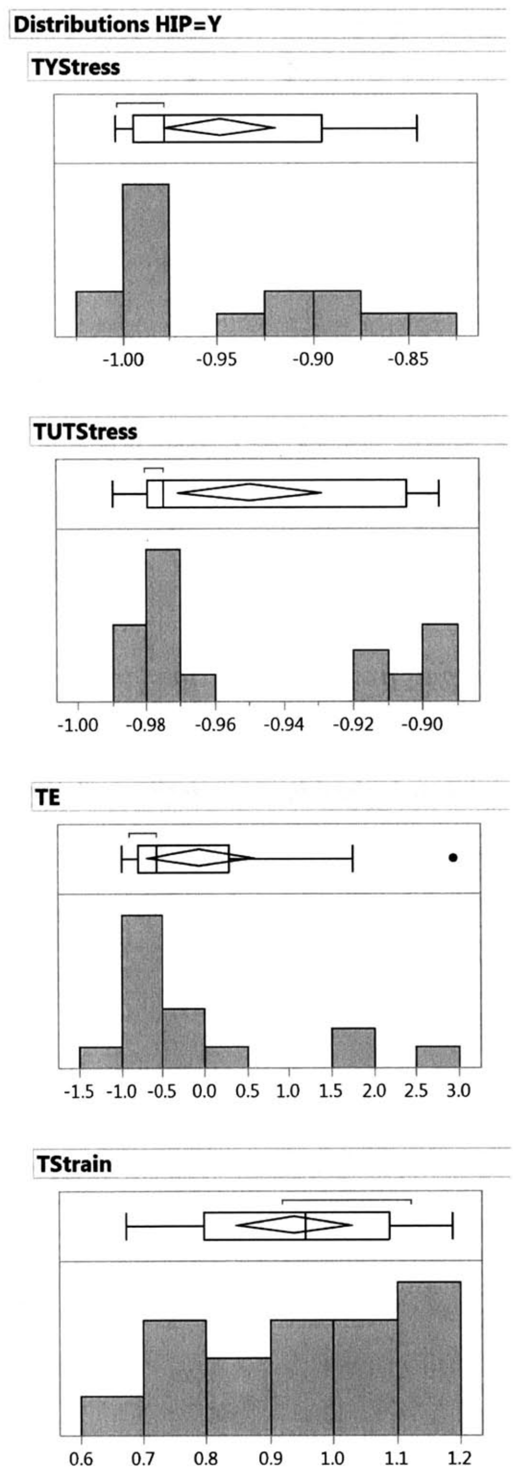


Figure F-1: Distribution Boxplot and Histograms for Transformed Mechanical Properties with HIP

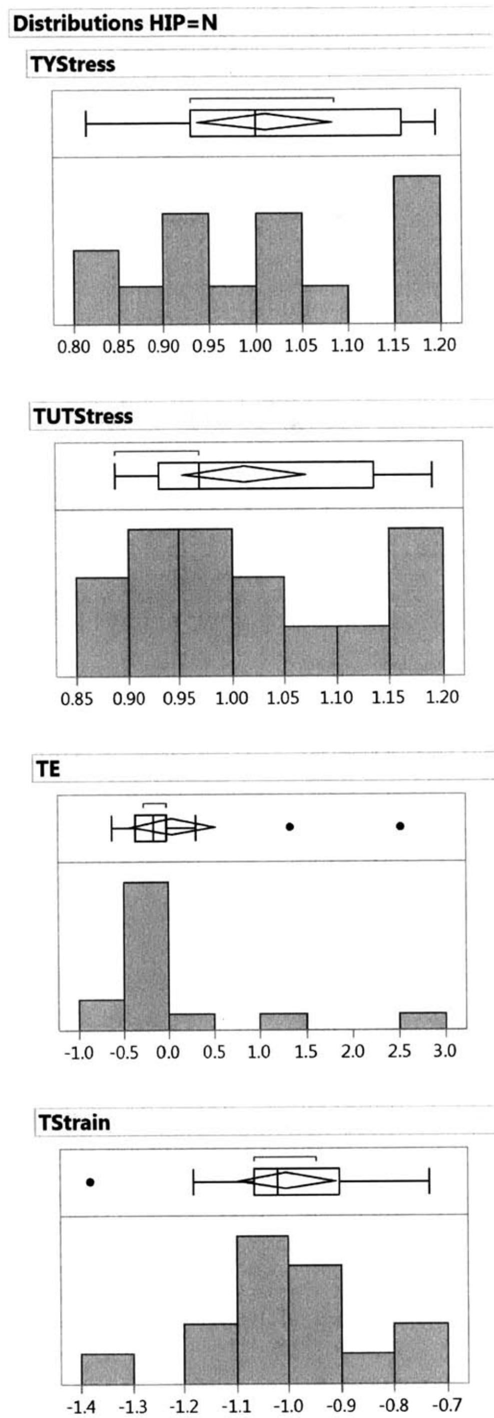


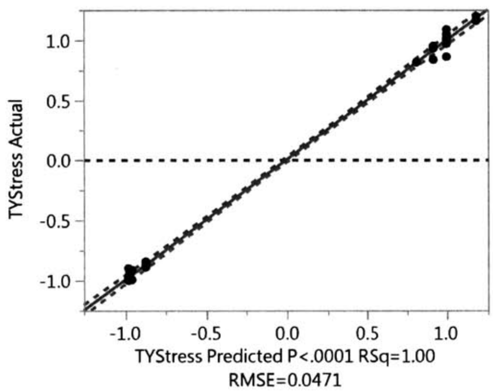
Figure F-2: Distribution Boxplot and Histograms for Transformed Mechanical Properties without HIP

Response TYStress

Effect Summary

Source	LogWorth	PValue
HIP	32.085	0.00000
Z-Orientation (deg from horiz)(0,90)	6.799	0.00000
Coupon Type	4.594	0.00003
Z-Orientation (deg from horiz)*HIP	3.588	0.00026
HIP*Coupon Type	1.719	0.01909
Z-Orientation (deg from horiz)*Coupon Type	1.262	0.05474

Actual by Predicted Plot



Summary of Fit

RSquare	0.998226
RSquare Adj	0.997782
Root Mean Square Error	0.047095
Mean of Response	8.6e-17
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	29.946769	4.99113	2250.344
Error	24	0.053231	0.00222	Prob > F
C. Total	30	30.000000		<.0001*

Sorted Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
HIP[N]	0.9607084	0.009754	98.50	<.0001*
Z-Orientation (deg from horiz)(0,90)	-0.071055	0.009754	-7.28	<.0001*
Coupon Type[Cylinder]	0.0469006	0.00903	5.19	<.0001*
Z-Orientation (deg from horiz)*HIP[N]	-0.04055	0.009471	-4.28	0.0003*
HIP[N]*Coupon Type[Cylinder]	0.0245122	0.009754	2.51	0.0191*
Z-Orientation (deg from horiz)*Coupon Type[Cylinder]	-0.019697	0.009754	-2.02	0.0547

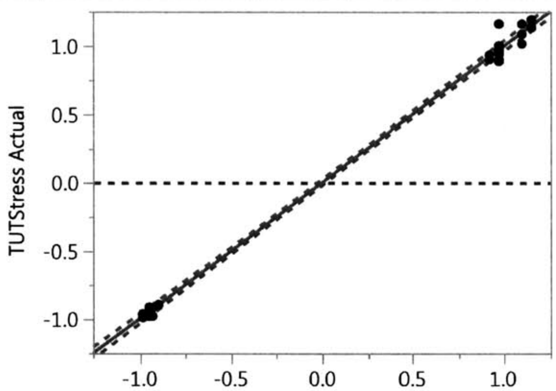
Figure F-3: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Yield Tensile Stress

Response TUTStress

Effect Summary

Source	LogWorth	PValue
HIP	34.491	0.00000
Z-Orientation (deg from horiz)*HIP	4.978	0.00001
Z-Orientation (deg from horiz)(0,90)	2.580	0.00263 ^
Distance from Center (mm)(30,90)	1.615	0.02426

Actual by Predicted Plot



TUTStress Predicted P<.0001 RSq=1.00
RMSE=0.0513

Summary of Fit

RSquare	0.997723
RSquare Adj	0.997372
Root Mean Square Error	0.051262
Mean of Response	1.5e-16
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	29.931679	7.48292	2847.660
Error	26	0.068321	0.00263	**Prob > F**
C. Total	30	30.000000		<.0001*

Sorted Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
HIP[N]	0.9864527	0.009811	100.55	<.0001*
Z-Orientation (deg from horiz)*HIP[N]	0.0525274	0.009654	5.44	<.0001*
Z-Orientation (deg from horiz)(0,90)	0.0356092	0.010704	3.33	0.0026*
Distance from Center (mm)(30,90)	-0.025608	0.010704	-2.39	0.0243*

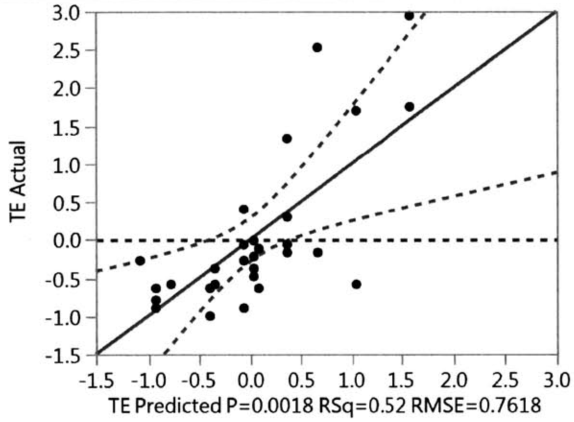
Figure F-4: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Ultimate Tensile Stress

Response TE

Effect Summary

Source	LogWorth	PValue
Coupon Type	1.985	0.01036
Z-Orientation (deg from horiz)*HIP	1.257	0.05531
Distance from Center (mm)(30,90)	1.015	0.09656
Distance Offset (mm)(-15,15)	0.861	0.13759
Z-Orientation (deg from horiz)(0,90)	0.551	0.28091 ^

Actual by Predicted Plot



Summary of Fit

RSquare	0.516364
RSquare Adj	0.419636
Root Mean Square Error	0.761816
Mean of Response	4.71e-16
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	15.490912	3.09818	5.3383
Error	25	14.509088	0.58036	**Prob > F**
C. Total	30	30.000000		0.0018*

Sorted Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Coupon Type[Cylinder]	0.4307553	0.155369	2.77	0.0104*
Z-Orientation (deg from horiz)*HIP[N]	0.3401002	0.169181	2.01	0.0553
Distance from Center (mm)(30,90)	-0.28937	0.167584	-1.73	0.0966
Distance Offset (mm)(-15,15)	0.2664755	0.173713	1.53	0.1376
Z-Orientation (deg from horiz)(0,90)	-0.190527	0.172872	-1.10	0.2809

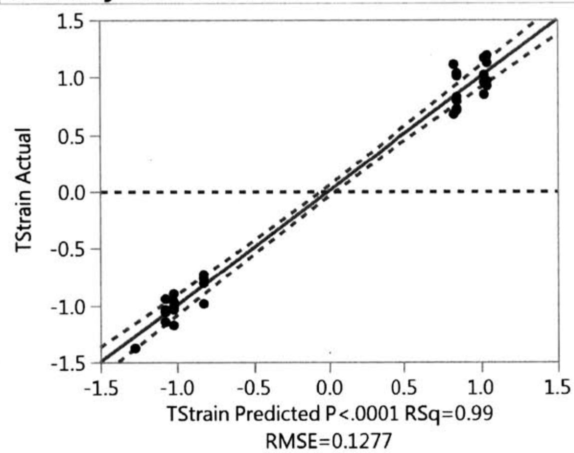
Figure F-5: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Young's Modulus

Response TStrain

Effect Summary

Source	LogWorth	PValue
HIP	24.508	0.00000
Coupon Type	3.403	0.00040
Z-Orientation (deg from horiz)*HIP	1.895	0.01273
Z-Orientation (deg from horiz)(0,90)	1.625	0.02369 ^

Actual by Predicted Plot



Summary of Fit

RSquare	0.985877
RSquare Adj	0.983704
Root Mean Square Error	0.127654
Mean of Response	-4.6e-16
Observations (or Sum Wgts)	31

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	4	29.576314	7.39408	453.7462
Error	26	0.423686	0.01630	**Prob > F**
C. Total	30	30.000000		<.0001*

Sorted Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
HIP[N]	-0.994089	0.024078	-41.29	<.0001*
Coupon Type[Cylinder]	0.0985244	0.02424	4.06	0.0004*
Z-Orientation (deg from horiz)*HIP[N]	-0.067823	0.025346	-2.68	0.0127*
Z-Orientation (deg from horiz)(0,90)	-0.057859	0.024078	-2.40	0.0237*

Figure F-6: Overview of Fit Model Platform containing Multiple Regression Model of Transformed Ultimate Strain