Variable Oxidation & Defects In Ti-6Al-4V Material In Electron Beam Melting Additive Manufacturing

Edward Patton Clark
University of Denver

Follow this and additional works at: https://digitalcommons.du.edu/etd

Part of the Materials Science and Engineering Commons, and the Mechanical Engineering Commons

Recommended Citation
Clark, Edward Patton, "Variable Oxidation & Defects In Ti-6Al-4V Material In Electron Beam Melting Additive Manufacturing" (2017). Electronic Theses and Dissertations. 1243.
https://digitalcommons.du.edu/etd/1243

This Thesis is brought to you for free and open access by the Graduate Studies at Digital Commons @ DU. It has been accepted for inclusion in Electronic Theses and Dissertations by an authorized administrator of Digital Commons @ DU. For more information, please contact jennifer.cox@du.edu,dig-commons@du.edu.
VARIABLE OXIDATION & DEFECTS IN TI-6AL-4V MATERIAL IN ELECTRON
BEAM MELTING ADDITIVE MANUFACTURING

A Thesis
Presented to
the Faculty of the Daniel Felix Ritchie School of Engineering and Computer Science
University of Denver

In Partial Fulfillment
of the Requirements for the Degree
Master of Science

by
Edward Clark
March 2017
Advisor: Dr. Maciej Kumosa
Abstract

Powder-based metal in additive manufacturing (AM) is advantageous for rapid prototyping of parts and components, with the benefit of reusing powder to reduce production costs. A common driver in the aerospace industry is free-form complex geometries which can be created using CAD software to optimize specifications with strength-to-weight ratios in components. Weight optimization of aircraft components using additive manufacturing reduces material, which significantly reduces production cost in comparison to cast and wrought metallic products. Large biomedical and aerospace industries heavily invest in feedstock metal powders that have low density under structural stresses and high temperatures, resulting in superior resistance to corrosion in extreme environments. The high strength-to-weight ratio material with long-term cost affordability obtained by AM process results in cost-efficiency of the Ti-6Al-4V powder by recycling unspent powder material. However, while efficient, the recycling of titanium (Ti) powder in additive manufacturing results in micro-particle characterization, chemical, and mechanical changes due to indirect and direct environmental extremes. Direct exposure to high thermal heat during the electron beam melting (EBM) process after powder reclaiming can result in particle microstructure and chemical variations. Initial assessments of oxygen (O) wt% in bulk powder material was subsequently selected for further investigation as a characteristic for decline in mechanical properties of consolidated materials. A preliminary analysis of virgin, 5x-recycled, and artificially highly oxidized Ti-6Al-4V powder was
conducted using Charpy impact testing on consolidated specimens from each wt% O in the bulk powder batches, and finite elemental analysis (FEA) to understand the build defect effect found in solidified Ti-6Al-4V material.
Acknowledgments

Thank you to my advisor Dr. Maciej Kumosa for his help and driving support during the research. Also, thank you to Monika Bleszynski and Dr. Euri Solis-Ramos for their patience and guidance, and a special thank you to William Grell and Dr. Zach Loftus at Lockheed Martin Space Systems for their support throughout this project.
# Table of Contents

Chapter One:
- Introduction .............................................................................................. 1
- Literature Review .......................................................................................... 1
- Oxidation of Ti-6Al-4V Powder Material ...................................................... 2
- Statement of Problem ..................................................................................... 3
- Summary of Research ..................................................................................... 4
- Publication Synopsis ..................................................................................... 5

Chapter Two: Artificially Oxidizing Ti-6Al-4V Powder ........................................ 7
- Introduction ..................................................................................................... 7
- Material Microstructure Characterization ....................................................... 8
- Experimental Methods .................................................................................... 10
- Artificial Thermal Oxidation .......................................................................... 11
- Results and Discussion .................................................................................. 12
- Surface Oxide Formation ................................................................................. 15
- Oxidized Powder Mixture Preparation ......................................................... 18
- Results and Discussion .................................................................................. 22

Chapter Three: Charpy V-Notch Testing ............................................................... 27
- Introduction ..................................................................................................... 27
- Experimental Methods .................................................................................... 29
- Orientation Effect on Charpy Impact Properties ........................................... 30
- Effects of Oxygen Content in Solidified Powder Composition ....................... 31
- Results and Discussion .................................................................................. 32

Chapter Four: FEM of Charpy V-Notch Specimen with Defect .............................. 39
- Introduction ..................................................................................................... 39
- Experimental Methods .................................................................................... 42
- Limitations of FEA Model ............................................................................. 48
- Notched Body Stress ..................................................................................... 49
- Results ...................................................................................................... 50
- Discussion .................................................................................................... 64

Chapter Five: Summary and Conclusion .............................................................. 67
- Summary .................................................................................................... 67
- Conclusion ..................................................................................................... 68

Appendix ............................................................................................................. 71
- Works Cited .................................................................................................... 71
Chapter One: Introduction

Literature Review

Electron beam melting (EBM) is a powder bed fusion additive manufacturing (AM) based process that utilizes an electron energy beam to melt metallic powders in a layer-by-layer fashion. This process is capable of fabricating 3D objects according to a computer-aided draft (CAD) setup file. Additive manufacturing is a rapid prototyping building process synonymous to 3D printing, which deposits material rather than removes material, such as subtractive manufacturing methods. Some advantageous factors for AM in industry are the abilities to create free-form geometrical complexity with minimum constraints, large production volumes over a long periods of time, and operating cost savings. Metal alloy powder in additive manufacturing process is advantageous for rapid prototyping of parts, and the ability to reuse powder to reduce cost of production parts. A common driver in the aerospace industry is free-form complex geometries, which can be created via CAD software to optimize specifications with strength-to-weight ratios in critical aircraft components. Optimization of aircraft and spacecraft components for weight saves material and significantly reduces production cost in comparison to cast and wrought products [1-4]. Large aerospace industries such as Lockheed Martin heavily invest in feedstock metal powders that have low density and relativity high stability under structural stresses and high temperatures, as well as superior resistance to corrosion in extreme environments. Titanium alloy is a work-horse material widely used in airframe structures and crucial high-
flow jet engine applications. Standard grade 5 Ti-6Al-4V is an optimized metal alloy that meets requirements for aerospace industries. The complexity of aircraft and aerospace structural components can be expensive to build; although traditional casting and wrought fabrication methods are still widely used in specialized industry, EBM additive manufacturing processing greatly enhances production for powder metallurgical air/space applications. Hence, the balance of a high strength-to-weight ratio material with lower long-term cost results in the affordability of the AM process, and many industries such as Lockheed Martin Space Systems utilize cost-efficiency of the Ti-6Al-4V powder by recycling between EBM AM builds [6].

Ti-6Al-4V powder in AM is advantageous for rapid prototyping of parts, and one very important aspect of the process is the ability to reuse of powder to reduce cost of production parts. However, one direct effect of reusing titanium powder in AM results in microstructure characteristics changes of the chemical composition and the particle morphological shape [1-3]. Reuse of Ti-6Al-4V powder for EBM results in elemental content variations, and recycled powder shows advanced stages of distortions of the particle sphere surfaces. Although the sampled flow testing of multiple recycled powders shows improvement compared to virgin powder [4], previous studies conducted on powder-based Ti-6Al-4V additive manufacturing showed little deleterious effects after recycling resulting in increased oxygen (O) wt% and particle morphological changes [4-7]. In this study, the morphology of the powder spheres was observed. Ti-6Al-4V powder data from previous experimental studies suggests relevant changes in the following Ti-6Al-4V powder and consolidated material characteristics: chemical properties, particle morphology, and mechanical behavioral properties of solidified material.
Statement of Problem

A preliminarily discussion with engineers at Lockheed Martin Space Systems unveiled a necessity for investigating improvements in vital aerospace components built by conventional cast and wrought methods. The importance in expanding additive manufacturing technologies was crucial to save manufacturing time and cost in constructing expensive spacecraft components. An investigation was conducted on the thermal influences in Ti-6Al-4V particles, with the recommendation to study the mechanical quality of various solidified material built from recycled powder types in the EBM AM process. Lockheed Martin expressed interest to research the quality degradation effects of recycling grade 5 Ti-6Al-4V powder between builds in the Arcam A2/A2X EBM AM system. Initially, preliminary research was conducted in the oxidization effects of titanium powder and consolidated material by the EBM in additive manufacturing, however the study later evolved to incorporate analysis and the study of artificial isothermal aging of powder and the effects on mechanical impact energy absorption on consolidated parts.

Currently, limited information regarding the high oxygen wt%, advanced-stage oxidization effects of Ti-alloy powder exists. Powder material degradation aging from environmental and within the EBM processes such as residual oxidization, morphological deformations of particles, and exposure to excessive thermal heat causes quality variances as seen in manufacturing and experimentation [1-7]. Nevertheless, readily available research concerning the oxidization effects on consolidated materials is not readily available for scientific collaboration. Thus, it is essential to develop extensive powder oxidization characterization and mechanical behavior assessments in the AM process.
A known powder aging mechanism associated with Ti-64 material recycling is assessed by the accumulation of wt% O of bulk material [1-5]. Oxidation effects, mechanical impact analysis, and finite element (FE) modeling were selected for the research due to the inadequacy of available, present research findings. Ti-6Al-4V powder was the focus material in the study, as severe bulk oxide products integrate into melted, fused consolidated material during the AM building process. Thus, micro-particle characterization, advanced mechanical impact testing related to impact fracture analysis, and finite element analysis (FEA) were used in detail, as significant excessive oxidization in bulk material can compromise the quality of AM finished parts.

Purpose of Research

An effective manufacturing process in the Arcam AM EBM system is the ability to reduce cost by recycling unspent, leftover powder material. Powder-bed fusion based systems allow for collecting and reprocessing powder-base material for future builds [1, 2]. Titanium alloy is a high cost production material, and recycling Ti-6Al-4V powder is essential for significant manufacturing cost savings. The Arcam EBM process operates in a high vacuum environment, and the Ti-6Al-4V material is spread in a layer-by-layer fashion as an electron beam is used to melt the powder. The titanium powder is exposed to prolonged high temperatures exceeding 1000°C [3, 5]. As a result, recycled powder material undergoes severe thermal stresses in which the particle morphology and chemical composition may change during multiple reuses of the powder. A recent study revealed a progressive increase in O content wt% after multiple recycling in sintering the powder material with an increase in ultimate tensile strength (UTS) as powder is recycled [4]. The
Ti-Al-4V material was reused to build tensile specimens concurrently over 16 times. An increase in O was measured, with oxygen levels recorded within the appropriate range for O wt% content \(\leq 0.13\), which is less than the O wt% recorded for powder recycled more than 4 times [4]. ASTM F2924-14 parameters for AM requires O content wt% to be < 0.2 for all standard Ti-6Al-4V powder [9]. However, the powder material was shown in previous studies [4] that powder can be reused more than 16 times by mixing virgin and recycled powder to achieve a O wt% content < 0.2 [4]. ASTM 3049-14 states standard guidelines for wt% O in Ti-6Al-4V powder is wt% O < 0.2 [8]. However, although the wt% O was within guidelines, the Ti-6Al-4V powder chemistry and ultimate tensile strength (UTS) of consolidated material were shown to change from using recycled powder [4]. Thus, it was important to identify the quality variations from virgin to highly oxidized powder beyond the ASTM wt% O limit during the EBM process.

This research studies the effects of oxidization within virgin and consolidated Ti-6Al-4V powder within the additive manufacturing process. The differences in quality of virgin and recycled Ti-6Al-4V powder resulted in characteristic changes in chemical composition, particle morphology, mechanical properties of consolidated material, and finite element modeling. Reuse of standard grade Ti-6Al-4V powder used in an EBM system therefore results in variations elemental content due to continuously recycling the powder material to produce specialized components for specific aerospace industry.

*Publication Synopsis*

A peer-review paper was submitted to Additive Manufacturing Journal on February 3rd, 2017 during the course of this extensive study in conjunction with University of Denver.
research group, Lockheed Martin Space Systems, and National Institute of Standards and Technology (NIST).

“Effect of powder oxidation, hot isostatic pressing, and build orientation on the impact toughness of electron beam melt Ti-6Al-4V”, W.A. Grell$^{1,2}$, E. Solis-Ramos$^1$, E. Clark$^1$, E. Lucon$^3$, E.J. Garboczi$^3$, P.K. Predecki$^1$, Z. Loftus$^2$ and M. Kumosa$^1$

$^1$ NSF Center for Novel High Voltage/Temperature Materials and Structures, University of Denver

$^2$ Lockheed Martin Space Systems

$^3$ National Institute of Standards and Technology
Chapter Two: Artificially Oxidizing Ti-6Al-4V Powder

Introduction

Titanium (Ti) and largely proportional Ti-alloy materials are susceptible to oxidation. A primarily factor in the development of a passive surface oxidation layer is oxygen (O). Due to Ti’s high tendency to oxidize in open-air environments, Ti-alloy materials are significantly influenced by O and thermal heat [5, 7]. Thermal influence on micro-sized Ti particles due to long-term exposure to excessive isothermal heat in open-air conditions can accelerate surface oxidation and bulk oxide accumulation [5, 6-8]. The evaluations of research literature indicate similarities of Ti-alloy high versatility to oxidization and protective qualities from corrosion were used to conduct extreme artificial laboratory aging of Ti-6Al-4V to study the bulk oxygen effects on Ti particles and bulk powder [5, 9-16].

The bulk O content wt% property of Ti-6Al-4V powder and chemical composition will vary depending on the initial quality of the powder material from the manufacture [1, 2, 9]. A direct microscopy observation can be used to analyze the powder particles shape and quality. Several mechanisms for quality changes in the Ti-alloy powder are directly influenced by handling of the material before use in an EBM system and after reclaiming unspent powder for recycling from the EBM AM process. Both environmental factors have direct influence on physical shape of particles, foreign contaminants, and chemical changes. Powder that is reclaimed showed an increase in O wt% and formation of oxides
on particle surface layers. The laboratory oxidization experiment results in this study are comparable to extreme conditions, showing the underlining and potentially damaging of the Ti-alloy powder.

Material Microstructure Characterization

Standard aerospace Grade 5 Ti-6Al-4V powder for EBM system such as Arcam A2/A2X is a created by a plasma-atomized process. Arcam Ti-alloy powder has a density of ~2.70 g/cm³ and particle size distribution (PSD) ~ 40-60 µm from the manufacture [5-10]. Relative morphology of particles is spherical, however some smaller variations of spheres (satellites) and non-spherical shapes are also created during the initial manufacturing process [1, 8]. The approximation of PSD and chemical analysis for EBM Arcam system requirements is consistent with industry standards ASTM F3049-14 and F2924-14.

A majority of engineering applications use light-weight/high-strength ratio Ti and Ti-alloys. Ti-6Al-4V Grade 5 alloy is an α+β twin-phase alloy. The metal consists of balanced Ti 90%, with Aluminum (Al) 6% and Vanadium (V) 4% (table 1). Al and V are stabilizers: Al is the α stabilizer as V is the β stabilizer. Pure Ti heated at 882°C transforms from α to β [22]. Stabilizers metals are added to Ti to manipulate phase changes in the metal. Thus, adding α-phase metals to Ti will raise the α-β transus temperature, and adding β-phase stabilizers metals will lower the α-β transition temperature [21]. The α-phase, Al acts as simple dilution of the crystalline lattice structure of Ti; enhances the mechanical strength of the existing Ti-bonds. The bonding is retained in the hexagonal close packed
(HCP) crystalline structure. The β-phase, V is an isomorphic metastable solute that contributes to strengthening by heat treatment [21-23].

Figure 1. Left: α-phase, Hexagonal Close Packed (HCP) and Right: β-phase Body Centered Cube (BCC). Reformatted from [22].

Heat treatment of Ti-6Al-4V depends on rate and time of direct thermal exposure and cooling rate. Microstructure of the Ti-alloy is dependent on α and β phases. Slow cooling of the alloy forms lamellar structures (β grains). Variability in cooling rate affects the lamellae grain-coarseness, and quenching the alloy leads to hardening [23]. The microstructural mechanical properties are strongly influenced by the processing and direct thermal treatment [20-24]. Recrystallization of the microstructure (equiaxed) can be achieved by heavily cold-working the material [23-24]. Formations of primary grain growth and coarseness are dependent on the annealing time. Thus, the sensitivity of the growth is enhanced by slower cooling than rapid fast cooling. However, transformation of grain structure and morphological distinctions is dependent on the solution heat treatment, annealing temperature, and post final temperature treatment. The dwelling time also contributes to the entirety of the microstructure [21, 23-24]. The chemical composition and
microstructure vary the properties of the Ti-alloy. Ti-6Al-4V, being an α+β metal alloy, is heat treatable and has an advantage with vanadium β-stabilizer, that enhances the mechanical properties at ambient room temperatures, resulting in resistance to fracture and increase toughness [21].

*Experimental Methods*

Thermal oxidation of Ti-6Al-4V plasma atomized powder between 200 - 650°C has been investigated. The oxidation rate of Ti-alloy metal has shown to be exponential at high thermal temperatures above 600°C. In this study, the development of an outer oxide layer was carried out in an open-air environment on 5x-recycled Ti-6Al-4V powder under thermal temperatures between 600 - 650°C. Isothermal temperatures above 600°C also showed oxidation and oxygen diffusion into the Ti-alloy powder [15, 18], changes in microstructure and morphology, and relative variations in mechanical properties of bulk powder. Observation under a scanning electron microscopy (SEM) of cross-section of Ti alloy powder particles showed formation of a heavy oxide layer. This experimental study, along with previous research, suggested that the accelerated oxidization activation growth of oxides and wt% O occurs above 650°C [15, 18, 19], therefore the process of artificially oxidizing Ti-6Al-4V powder and the effects of high temperature in open-air conditions were observed.
Artificial Thermal Oxidation

Commercially available standard grade 5 Ti-6Al-4V plasma-atomized 5x recycled powder was used in this study provided by Arcam AB manufactures. The composition of the powder consisted of Ti ~ 90 %, Al ~ 6%, and V ~ 4%, which is a material widely used in biomedical and aerospace industry [1]. Particle size consisted of ~ 40 – 60 µm, according to the manufacturing specifications [8, 9, 10]. The alpha-beta Ti alloy chemical composition of consolidated Ti-alloy is given in table 1.

<table>
<thead>
<tr>
<th>O</th>
<th>N</th>
<th>C</th>
<th>H</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.11</td>
<td>0.01</td>
<td>0.03</td>
<td>0.003</td>
<td>6</td>
<td>4</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Prior to testing, the powder acquired from the additive manufacturing lab contained a wt% oxygen of ~ 0.16 for the 5x-recycled Ti-6Al-4V powder, which is ~ 0.05 higher in wt% oxygen of virgin powder. Powder specimens of 3 grams each were placed small ceramic crucibles (diameter 3 mm, 2 mm x 3.5 mm) before placed into high-heat rectangle open furnace with a single temperature zone. The oxidation temperatures were 200 – 670°C. Temperature control during the oxidization tests was done using K-type high-temperature tungsten thermocouples with an external digital fluke meter to monitor temperature readings. Variability in temperature range was observed to be ± 30°C with humidity (~ 25 - 50%), due to the influencing open-air laboratory environment. The temperature ranges were set to understand the oxide layer and change in morphology under
non-vacuumed, isothermal conditions. After oxidation treatment, the Ti-6Al-4V powder samples were taken out of the furnace and open-air cooled to room temperature.

Microstructural analysis was completed using an optical microscopy for the 200°C/2h and 200°C/24h test specimens. Specimens were mounted in epoxy followed by hand-sanding using 240-grit silicon-carbon paper to remove loose debris, then hand-sanded with 400-grit paper. Final wet sanding with 600-grit paper was completed on rotational Buehler wet-sander. Polishing of the epoxy specimens using micro-polishing rotation pad with multi-step polishing solution of 5.0 µm, 1.0, 0.05, to 0.03 µm Buehler Alumina micro-polishing compound. Water was applied to prevent heat build up during the sanding process to prevent microstructural changes. Finally, the mounted epoxy specimens were etched with HNO3 solution. The specimens were thoroughly rinsed with deionized water to remove sanding debris before placed in a sonic cleaner. The mounted specimens were dried with low-heat hand held dryer. Additional microstructure characterization was done using SEM for specimens treated at ~ 400 - 670°C/4h. These specimens were prepared by hot-mounting in carbon-based KonductoMet epoxy resin followed by hand-sanding with silicon-carbide paper and similar method used for epoxy mounted low-temperature isothermal treated specimens. Examination of high-heat isothermal treated specimens were completed using a Joel JSM-5800LV SEM.

*Results and Discussion: Isothermal Treated Ti-6Al-4V Powder*

Ti-alloy typically provides excellent resistance to corrosion in extreme thermal environments [21-24]. Advanced surface oxidation was observed after Ti powder was
exposed to various thermal conditions, and relative increases in oxidation were noted in the color changes. Optical microscopy showed the distinct color variations on the immediate surface of the particles. A silver-gray color change from lower heat exposure of \(~ 200°\text{C}/2\text{-}24\text{h}\) indicated minimum surface oxidation (figure 3a), however with increased thermal heat of \(400°\text{C} /4\text{h}\), the Ti-alloy particles changed to a golden-yellow, (figure 2b). A drastic color change to dark-brown was observed at temperatures \(500 - 650°\text{C}/4\text{h}\), figures 3a, 3b. These color changes are due to an increase thickening and growth of the oxide layer. Oxide layer formation on the particle surfaces was evident by additional SEM analysis. Further characterization was conducted with enhanced viewing of particle morphology, samples of Ti-6Al-4V powders placed in epoxy resin for cross-sectional analysis showed changes from lower thermal temperatures to higher thermal temperature exposure. The prepared powder specimens were polished and etched before SEM viewing, and internal microstructures were viewed with the SEM (figure 5).

Figure 2. (a) \(\sim 200°\text{C} /2\text{h}\), (b) \(\sim 400°\text{C} /4\text{h}\). Oxidation of Ti-6Al-4V powder at lower thermal temperatures. Color change indicates mild oxidation layer formation. **Images courtesy of E. Solis-Ramos**
Figure 3. (a) ~ 500°C /2h, (b) ~ 650°C /4h. Thermal oxidation of powder at higher elevated temperatures. Oxidation color change to brown to dark-brown indicates thick surface oxide layer on particles. **Images courtesy of E. Solis-Ramos

Table 2. Artificial oxidation conditions of powder (IGA instrument)

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Duration (h)</th>
<th>O Content (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>~25 (5x-recycled)</td>
<td>--</td>
<td>0.16</td>
</tr>
<tr>
<td>200</td>
<td>2</td>
<td>0.16</td>
</tr>
<tr>
<td>200</td>
<td>24</td>
<td>0.17</td>
</tr>
<tr>
<td>400</td>
<td>4</td>
<td>0.20</td>
</tr>
<tr>
<td>500</td>
<td>4</td>
<td>0.32</td>
</tr>
<tr>
<td>650</td>
<td>4</td>
<td>4.4</td>
</tr>
</tbody>
</table>

Oxidation condition in high-heat furnace (open-air). IGA taken at Lockheed Martin. Values from bulk artificially oxidized powders.

Measurements were taken using instrumental gas analysis (IGA), which detected wt% O > ~ 4.1 at temperatures above 650°C/4h. The bulk Instrumental-gas Analysis (IGA) was performed on the high-isothermal aged powder. Instrumental analysis of the titanium powder was exposed to higher thermal heat showed particle surface changes, which indicates a parabolic oxidation-rate effect [15, 19]. The dramatic rise in thermal temperature 500°C - 650°C resulted in an increase in O wt% levels exponentially from 0.32 to 4.4, table 2 (figure 4). It was evident from table 2 there was an exponential rise in
bulk O content correlation with higher thermal-heat exposure. Titanium has an overwhelming affinity to oxygen, thus under open-air conditions the rate of oxidation on the surface significantly increases, and oxygen diffuses quickly through Ti-alloy particle surfaces [13, 17, 19]. Comparing tested powders characteristics to larger bulk material properties; there were noticeable effects of highly oxidized powder on the EBM consolidated material.

![Figure 4. IGA wt% O of isothermal oxidized Ti-6Al-4V powder.](image)

**Surface Oxide Formation**

External environmental factors may influence the oxidation rate, oxidation state, and composition of Ti-alloys. Therefore, the rate of oxide formations is directly influenced by the material size, shape, and external thermal factors, which may result in different
oxidation compounds on the surface of the Ti-alloy [13, 15, 24]. Ti-6Al-4V is considered an excellent material for highly corrosive environment, as it has strong corrosion resistance [22, 24]. Ti in a natural environment, produces a passive corrosion resistant surface oxide layer that is primarily a rutile TiO$_2$ coating is a crystalline coating, with a relatively stable adhesive quality [20]. Other species of oxides consist of Al$_2$O$_3$, which is considered a stable oxide layer formation. Vanadium-based oxides exist below the outer-most dominant oxide layer and Ti-based oxides. Consisting mainly of V$_2$O$_5$ with lesser existence of V$_2$O$_3$ and VO$_2$, previous research indicates these lesser degree vanadium-oxides occur interstitially or are substitutional ions in the oxide matrix of the predominantly TiO$_2$ [24].

Longer high-heat exposure of 650°C/ > 4h also indicated oxide surface flaking and spallation of oxide layers from surface of particles (figure 9). Oxidation rate for Ti in open-air conditions will contribute to surface growth of TiO$_2$ and oxide species. The rapid diffusion of O into the Ti-alloy particle surface can be characterized as rapid thermal oxidation of the metallic alloy. The observations from the artificially oxidized experiment agree with Fick’s 1$^{\text{st}}$ law of diffusion, $J = -D \frac{d\varphi}{dx}$, where diffusion is a response to the concentration gradient of O. Therefore, the interaction of Ti-alloy particles with high-state kinetic energy of oxygen gas molecules for increased gradient and diffusion rate [19]. The oxidation behavior of Ti-alloy particles in the experimental data suggests the predictive-range of the oxidation-layer formation on particle surfaces. The O diffusion with respect to relative particle size and compaction also indicates a strong oxidation mechanism in temperatures over 650°C [15, 20].
Cross-section analysis indicates there are no significant differences in external oxidation layers between untreated powder and marginally thermal treated powder at 200°C for 4-24 hours. However, increased oxidation on particle surfaces was observed after furnace aging at 650°C for 4-8 hours. An oxidation-zone, which is the oxide layer formation from direct high-heat exposure, appeared ~10 µm thick with the surface oxide-layer thickness of ~3 µm (figure 5d). The oxide-layer is the passive, protective coating, which was observed to increase in thickness from the high-heat. Differences in the internal structures of virgin powder and heat treated powder can be seen in the microstructure of the images.
the particle treated at 650°C/4h. SEM Energy Dispersive X-ray Spectroscopy (EDS) showed a weak increase of O on the outer surface of a particle (figure 6). It can be determined that high temperatures in an open-air atmosphere (no vacuum) accelerated oxidation on particle surfaces and within Ti-6Al-4V particle microstructure. Therefore, high isothermal heat exposure of temperatures between 500-650°C/4h accelerated the oxidation of Ti-6Al-4V particle surface, figure 5b, 5d.

![Figure 6. **SEM EDS line scanning of 650°C/4h oxidized cross-section of Ti-6Al-4V powder particle. Ti, Al, V, and O reading of particle cross-section surface*[43]. **Image analysis courtesy of E. Solis-Ramos](image)

**Oxidized Powder Mixture Preparation**

Ti-6Al-4V powder specimen batches were prepared with 0.13, 0.16, 0.32, and 0.45 wt.% O content. Large quantities of powder were isothermally oxidized in high-heat
furnaces at 645 - 670°C/4 h in open-air conditions. The O wt% of the highly oxidized powder was taken by IGA to evaluate the composition. The highly oxidized Ti-alloy powder consisted of an oxide case formation on the surface of the Ti-alloy particles, which was expected for Ti [17, 22]. Artificial aging treatment of bulk quantity powder was carried out in ceramic evaporating dishes (Diameter depth 100 ml, 90 mm X 35 mm). Two dishes consisting of ~ 0.75 kg/day of Ti-6Al-4V powder were placed in a high-heat furnace (100 mm x 100 mm x 210 mm), which was repeated once a day for 20 days. A proportional–integral–derivative (PID) temperature regulation was used for maintaining 645 - 670°C/4h/day. The Ti-alloy’s exposure to high isothermal-heat for an extensive period of time showed excessive clumping and bonding of the powder particles. A dark brownish oxide formation was observed, as with other temperature color variations within the center of the oxidized bulk powder was indicated advance oxidation properties of Ti-Al-4V [14-20]. Combined aged powder was merged in a larger evaporation dish before crushed in a ceramic mortar using a ceramic pestle (figure 7). Images of the highly oxidized crushed powder were taken with an SEM (figure 8-9). 3 grams of the highly oxidized powder was then analyzed for O wt.%, which subsequently used for creating two distinct Ti-6Al-4V batch mixtures to build consolidated specimens.
Figure 7. ~ 1.0 kg of Ti-6Al-4V powder artificially oxidized in an isothermal high-heat furnace for 650°C/4h in open-air conditions. The powder was placed in three individual ceramic evaporation dishes for treatment, then combined for manual powder separation. Powder sintering was observed due to exposure to high-heat.
Figure 8. Highly oxidized powder measured at 4.4 wt% O, powder agglomeration clumps broken down in mortar before mixing with 0.16 wt% O powder.

Standard aerospace Grade 5 Ti-6Al-4V powder was used for fabrication of consolidated testing specimens in an Arcam A2 and a newer A2X system. The Arcam system requires powder material weight of ~ 45 kg, which was placed in two adjacent gravity fed hoppers within the system. Prior to fabrication of specimens, four distinct powder batches were prepared with specific wt% O (table 3). Two separate powder batches were mixed with highly oxidized powder (~ 4.1 wt% O) with 5x-recycled (~ 0.16 wt% O) to acquire the required ~ 0.32 and ~ 0.45 wt% O. The standard wt% O calculation was used to calculate the appropriate wt% O for each batch (table 4 & 5).
Table 3. Ti-6Al-4V powder batch mixture matrix

<table>
<thead>
<tr>
<th>wt% O Content</th>
<th>0.13</th>
<th>0.16</th>
<th>~ 0.32</th>
<th>~ 0.45</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>Virgin</td>
<td>5x recycled</td>
<td>Mixed 4.1 wt% O w/ 5x recycled</td>
<td>Mixed 4.1 wt% O w/ 5x recycled</td>
</tr>
</tbody>
</table>

Table 4. Powder mixture calculations matrix to create ~ 0.3 wt% O

<table>
<thead>
<tr>
<th>5x-recycled powder wt% O</th>
<th>645 - 670°C/4h</th>
<th>Desired wt% O</th>
<th>Required mass fraction of 4.1 wt % O oxidized powder</th>
<th>Mass of 645 - 670°C/4h wt% O oxidized powder</th>
<th>Mass of 5x-recycled powder</th>
<th>Required mass of 5x-recycled powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.16</td>
<td>4.1</td>
<td>0.32</td>
<td>0.0406</td>
<td>1.827 kg</td>
<td>43.173 kg</td>
<td>45 kg</td>
</tr>
</tbody>
</table>

Table 5. Powder mixture calculations matrix to create ~ 0.6 wt% O

<table>
<thead>
<tr>
<th>5x-recycled powder wt% O</th>
<th>645 - 670°C/4h</th>
<th>Desired wt% O</th>
<th>Required mass fraction of 4.1 wt % O oxidized powder</th>
<th>Mass of 645 - 670°C/4h wt% O oxidized powder</th>
<th>Mass of 5x-recycled powder</th>
<th>Required mass of 5x-recycled powder</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.16</td>
<td>4.1</td>
<td>0.6</td>
<td>0.1117</td>
<td>5.025 kg</td>
<td>39.975 kg</td>
<td>45 kg</td>
</tr>
</tbody>
</table>

Results and Discussion

Isothermal oxidation of Ti bulk particles in open-air was carried out in temperature ~ 645 - 670°C/4h. The significant particle-size reaction with oxygen from a thermodynamic view demonstrates the formation of oxides on particle surfaces and bulking oxide-effect in powder [13, 15, 20]. The micron-sized particles increased surface area, which accelerated the oxidization of the powder. Initial microscopy analysis indicated a variation in color changes. The lower level heat indicated a distinct protective oxide layer with minimum oxide layer growth. However, the advanced stages of higher thermal heat
above 500°C showed thicker growth of oxide layer. From previous research studies, this layer can be isolated to be predominately TiO$_2$ [13, 20, 43]. O wt% analysis of bulk powder results extreme oxygen with was calculated at O wt% 4.4. Advanced oxidation of particles can be attributed to the micronized size of particles, thus the increased surface area [13-17]. Oxidation of different sized particles with Gaussian PSD of 40 – 75 µm means the aging effect can occur at different rates. Therefore, oxidation of smaller particle will achieve greater surface oxides is shorter time at high temperatures [13]. Sintering and agglomeration of particles was observed with the SEM, figure 9. Extensive oxide surface buildup around the particles was achieved through the high isothermal oxidation process. An SEM visual analysis of the particle showed a size-ratio to larger surface area will ultimately generate a thicker oxide layer in shorter aging time. Bulk powder particles agglomeration and sintering is indicative of the combination of larger surface area, figure 9, (right), which increase diffusion into material [13].

![SEM images](image_url)

Figure 9. **SEM images of artificially oxidized bulk Ti-6Al-4V powder in isothermal high-heat furnace at 650°C/4h. Agglomerated (clustered) powder particles with thick surface oxide-layer are visible on highly oxidized particles. **Images courtesy of E. Solis-Ramos
Surface oxide-layer debris was observed with the SEM. The sub-set particle flakes were a result of crushing and compacting the clumped highly oxidized Ti-6Al-4V bulk powder, figure 10. Surface oxide layer detachment was a direct result of manually refining the powder into base PSD 40 – 75 µm; no additional measurements were taken to conclude the PSD was adequate for ASTM standards specification [8, 9]. Spallation and separation may have been a result of impacting and crushing the clustered particles. Previous studies on heavily oxidized particles will result in a thickening layer, which will detach from metal surface from the multilayer oxide buildup. The oxygen rich film modifies into three possible oxides: anatase, brookite, and most likely rutile [13, 15, 20]. Powder was broken down to a base powder by visual observation suitable for mixing with 5x-recycled powder with lower O wt% 0.16.
Research literature data presented on isothermal experimentation was reviewed to base the suggestion on extreme thermal exposure aging of Ti-6Al-4V materials. According to Guleryuz et al [20] formation of oxidation layer on the surface of the Ti-alloy particles to an alternating production of Al$_2$O$_3$ and largely TiO$_2$. The early stages of aging results in nucleates of Al$_2$O$_3$ on the oxide surface with TiO$_2$. Therefore, the most outer casing of the oxide layer is Al-oxide based as the inner most substrate surface is Ti-oxide [20].

Rapid accelerated growth of the oxide layer on the surface of Ti-6Al-4V is accompanied with oxygen diffusion beyond the oxide layer [20]. Research from Guleryuz
et al [20] suggests the growth of the oxide layer has a parabolic-effect of kinetic energy at temperatures greater than 600°C. In combination with particle size, with increased surface area will, nevertheless result in hastened the extreme degradation of the particles [26]. Therefore, the bulk accumulation of O content wt% in the experiment is consistent within findings in supporting literature [20, 26, 43].
Chapter Three: Charpy V-Notch Testing

Introduction

Four distinctive batches of powder were prepared during the course of this study for consolidated chemical characterization and impact toughness testing, see chapter two bulk powder oxidization. As seen in chapter two, bulk powder oxidation of powder was conducted from experimental methods in chapter two. Isothermal-heat expose of Ti-6Al-4V powder to high levels heat in open-air conditions, which resulted in extreme O wt% pickup in bulk powder material. To mimic exaggerated extreme case of bulk oxygen pickup in powder material during recycling, this experimental testing focused on mechanical performance of specimens created from mixed low-level bulk oxygen powder with highly oxidized powder with concentrations exceeding industry standards of O wt% < 0.2 [30]. For mechanical testing, Charpy v-notch impact analysis, following ASTM E23 standards was conducted to test the energy absorbed on impact. Charpy v-notch testing is widely used to evaluate impact toughness in materials [3, 27, 29, 30, 39]. Energy absorbed from instrumental analysis of the complete fracture of the material taken is measured in Joules (J). Impact Charpy specimens are configured to different specification for measuring of fracture of material with specific characteristics. Given the nature of consolidated Ti-6Al-4V, a nonferrous metallic alloy will have a high hardness similar to carbon steels; therefore, a center v-notch was for crack propagation [30]. The brittle and
ductility in metallic materials is typically defined by the energy absorption in impact Charpy testing [3, 27-31].

The impact testing was carried out on fully machined and finished specimens in accordance with (IAW) ASTM E23 specifications. In order to determine change in energy absorption, four distinct O wt% batches and built in the Arcam A2/A2X system under three different orientation axis: XY, XZ, and ZX. Orientation during the build process is important since the additive manufacturing is a layer-by-layer process, which results in unique melting and fusion properties in the EBM AM process [1-7]. Including different orientations of the crack-propagation of the v-notch helped measure layering effect on strength during manufacturing in the EBM AM system [1, 4, 5, 10, 29]. Part built with Ti-6Al-4V powder or powder metallurgy materials commonly undergo post heat-treatment before machining. Portions of the Charpy E23 specimens for all O wt% levels were hot isostatic pressed (HIP) before machining and directly machined from EBM built for comparison. Impact energy absorption from the impact results specimens shown generally better for the Ti-6Al-4V specimen with the post build HIP treatment [30]. As current industry standards practices and literature suggests, the micro-porosity and various underlining internal defects of the solidified material are greatly reduced. The applied production technique provides better fatigue and strength, and stronger toughness in metal powder consolidated materials [29, 30].
Experimental Methods

The extremely aged powder was mixed with low O wt% powder to create specific batch. Several types of specimens were built in the Arcam EBM AM A2/A2X systems for each oxygen wt% level: labeling convention noted as AX, B, BX, C, C1, and D, table 6. The specimen was specifically designed in accordance with ASTM Standard E23 for v-notched bar impact testing of metallic materials (Figure 11). Three specimens for each conventional orientation were designed in SolidWorks CAD program. To assess the mechanical powder characteristics for specified O wt% batches, a total of 27 specimens were built in the EBM AM Arcam A2/A2X systems for each specific batch: virgin, 5x-recycled, and mixed highly-oxidized. Three specimens from each orientation group was selected for mechanical impact testing at National Institute of Standards and Technology (NIST) Laboratory.

Figure 11. The standard ASTM E23 Charpy v-notch impact bar specimen. Modified from [30].
Orientation Effect on Charpy Impact Properties

The orientation-effect in the specimen was observed in the mechanical testing. Three distinct axis orientations: XY, XZ, and ZX, figure 12. In the EBM AM process, solidified materials built in a layer-by-layer fashion, which the microstructure of the Ti-6Al-4V the nucleation of the elongated columnar β-grain structure is the same as the build direction, the Z-axis, therefore yielding the best strength values. [10-14]. The experimental impact results showed a distinct difference in energy absorption (J) compared to the three different orientations. The ZX exhibited larger energy absorption compared to the XY and XZ orientation specimens. The solidified material of the primary growth of grain microstructure orientation is stronger in the transverse, transaxial plane versus the unidirectional orientation of the XY and XZ of the Charpy v-notch specimen (figure 14).

The effect of the primary transaxial to unidirectional build orientation in the specimens were comparable with a considerable energy absorption. Impact mechanics of the Z-axis specimens were shown to have higher energy Joules (J) values than both the YX and XZ specimens.
Figure 12. A CAD schematic layout of the 27 blank EBM AM specimens. Charpy v-notch E23 samples were built in the Arcam A2/A2X system for each O wt% batch. Build direction is Z-axis direction. Three different orientations were marked on specimens: XY, XZ, and ZX, with corresponding number. V-notch location on specimens is specific to crack propagation for orientation effect.

Effects of Oxygen Content in Solidified Powder Composition

The Charpy v-notch impact mechanical testing was conducted at NIST laboratories on the various solidified Ti-6Al-4V material with different O content wt% ranging from virgin to mixed highly oxidized powder; O wt% were 0.11, 0.137, 0.142, 0.34, and 0.525 respectively prepared laboratory before sending bulk powder batches to the Additive Manufacturing Lab at Lockheed Martin to build the specified Charpy v-notch E23 specimens. Additional oxide batches were created to account for defective specimen. Full testing of specimens built from virgin to highly oxidized mixture powder batches, table 6.
Table 6. Specimen batches and O wt% measured before and after build in EBM system

<table>
<thead>
<tr>
<th>Specimen Batch Label</th>
<th>Powder Description</th>
<th>Targeted Oxygen wt%</th>
<th>Oxygen wt% Before Build</th>
<th>Oxygen wt% After Build</th>
</tr>
</thead>
<tbody>
<tr>
<td>(B) 5x-recycled</td>
<td>Single lot of 5x re-used powder</td>
<td>0.16-0.18</td>
<td>0.142</td>
<td>0.157</td>
</tr>
<tr>
<td>(C1) Oxidized Mix 1</td>
<td>96.5% 5x reused, 3.5% aged at 650°C/4hFour</td>
<td>0.25-0.35</td>
<td>0.340</td>
<td>0.292</td>
</tr>
<tr>
<td>(D) Oxidized Mix 2</td>
<td>88.8% 5x reused, 11.2% aged at 650°C/4h</td>
<td>0.40-0.60</td>
<td>0.525</td>
<td>0.455</td>
</tr>
<tr>
<td>(BX) 5x-recycled</td>
<td>Single lot of 5x re-used powder</td>
<td>0.16-0.18</td>
<td>0.137</td>
<td>0.151 (bottom), 0.157 (top)</td>
</tr>
<tr>
<td>(C1X) Oxidized Mix 1</td>
<td>96.5% 5x reused, 3.5% aged at 650°C/4h</td>
<td>0.25-0.35</td>
<td>*N/M</td>
<td>*N/M</td>
</tr>
<tr>
<td>(AX) Virgin</td>
<td>Single lot of virgin powder</td>
<td>0.10-0.15</td>
<td>0.11</td>
<td>~ 0.11</td>
</tr>
</tbody>
</table>

*N/M, not measured at Lockheed Martin facility.

Results and Discussion

The impact results shown in figure 14 clearly clarify the oxidation effect and orientation effect on Ti-6Al-4V powder used in the EBM AM process. The experimental process of artificially oxidizing the powder in open-air conditions severely damaged the bulk powder, in which the increased oxides and bulk oxygen were present. The high percentage of oxygen in bulk powder can be excessively damaging to the Ti-alloy particles.
and bulk powder used in the EBM system. Consolidated Ti-6Al-4V powder with acceptable O content wt% < 0.2 showed energy (J) absorption for both virgin and 5x-recycled powders. The HIP post process showed an increase in energy absorption during impact testing, as with the combination of lower O content wt% and build orientation effect the mechanical impact performance results were significantly higher than specimens with high O content, figure 14.

![Figure 14](image_url)

Figure 14. Results for Charpy v-notch specimens, energy absorbed for three oxygen levels and orientation effect. Modified from *[43]*.

The significant reduction in toughness in solidified Charpy specimens created oxidized mixtures O wt% > 0.27 can be attributed to microstructure chemical composition.
and characterization [14-22]. High amounts of oxygen with the Ti-alloy powder create a decreased ductility and increased brittleness in the consolidated material. SEM observation of fracture surfaces of Charpy specimens show declining surface roughness to smoother flatter surface of the high O content wt% specimens, figures 15-19 [43]. The consolidated material post impact tested fracture surface of virgin, 5x-recycled, and oxidized mixtures exhibit pronounced differences in appearances. Intense granular roughness of virgin and 5x-recycled to smoother, flat ceramic surface in highly oxidized mixtures.

**Images courtesy of E. Solis-Ramos

Figure 15. **Charpy orientation XY, fracture surface of 0.13 wt% O.
Figure 16. **Charpy orientation XY, fracture surface of 0.13 - 0.14 wt% O.

Figure 17. **Charpy orientation XY, fracture surface of 0.27 - 0.29 wt% O.
Enhanced SEM image analysis of post impact testing of orientations XY, XZ, ZX in virgin, 5x-recycled, and both oxidized mixtures indicated visually identical fracture surface roughness appearance. The relative increase in high O content wt% for both oxidized mixtures correlates to decrease in impact fracture toughness, figure 14.

Solidified Ti-6Al-4V material of both HIP and non-HIP treatments specimens presented similar O content wt% from IGA analysis conducted at Lockheed Martin, table 7. As expected from other literature research, the decreasing or leeching of Al and V were observed the chemical composition [5-7]. Identical chemical composition changes were observed in pre-production powder preparation analysis conducted before consolidated EBM building of Charpy specimens.
Table 7. Standard grade Ti-6Al-4V powder, chemical consolidated composition (wt%)

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>H</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>Y</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>AX</td>
<td>0.129</td>
<td>0.04</td>
<td>0.01</td>
<td>0.00</td>
<td>6.01</td>
<td>4.09</td>
<td>0.20</td>
<td>&lt; 5e-4</td>
<td>0.01</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>2</td>
<td>07</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>BX</td>
<td>0.134</td>
<td>0.01</td>
<td>0.01</td>
<td>0.00</td>
<td>5.66</td>
<td>4.05</td>
<td>0.19</td>
<td>&lt; 5e-4</td>
<td>0.01</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>9</td>
<td>06</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>0.137</td>
<td>0.01</td>
<td>0.02</td>
<td>0.00</td>
<td>5.82</td>
<td>4.31</td>
<td>0.21</td>
<td>&lt; 5e-4</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>6</td>
<td>07</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>C1</td>
<td>0.272</td>
<td>0.02</td>
<td>0.02</td>
<td>0.00</td>
<td>5.80</td>
<td>4.04</td>
<td>0.19</td>
<td>&lt; 5e-4</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>3</td>
<td>09</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>C1X</td>
<td>0.288</td>
<td>0.02</td>
<td>0.01</td>
<td>0.00</td>
<td>5.73</td>
<td>4.07</td>
<td>0.19</td>
<td>&lt; 5e-4</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>5</td>
<td>07</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>C2X</td>
<td>0.298</td>
<td>0.02</td>
<td>0.01</td>
<td>0.00</td>
<td>6.06</td>
<td>4.17</td>
<td>0.20</td>
<td>&lt; 5e-4</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>0</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>0.458</td>
<td>0.02</td>
<td>0.01</td>
<td>0.00</td>
<td>5.75</td>
<td>4.09</td>
<td>0.20</td>
<td>&lt; 5e-4</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>4</td>
<td>07</td>
<td></td>
<td></td>
<td></td>
<td>4</td>
<td>2</td>
<td></td>
</tr>
</tbody>
</table>


Four different O content wt% solidified material testing was conducted at NIST. Impact testing was conducted on over 72 E23 specimens for virgin, 5x-recycled, and two oxidized mixtures. The extreme decrease in impact strength > 20 J absorbed was seen in the two oxidized mixtures with O content wt% of 0.27-0.33 and 0.46. Both HIP/non-HIP and orientation showed an effect with the impact strength. Significant microstructure changes were observed in highly oxidized consolidated material of powder batch D (table 7).

Microanalysis SEM of fracture surfaces coincides with decreased ductility and increased brittleness, and corresponds with the NIST Charpy impact testing. Although
the HIP treatment increased impact strength values for all specimens tested. The effect of orientation was negligible with highly oxidized mixture specimens.
Chapter Four: FEM Analysis of Charpy V-Notch Specimen with Defect

Introduction

Previous research on analysis of defects mechanisms is usually established during optimal EBM AM system operations [38]. Therefore, best manufacturing practices are generally recognized for studying powder defects mechanisms [38, 40]. However, defects may occur during poor system performance, such as faulty equipment functionality due to random or systematic errors, which may result in inadequate electron-beam performance, beam instability, or improper temperatures for complete melting [2, 3, 4]. Electron beam performance can influence the melting pool of the powder during the melting of the layers [12-13, 19]. The reduction in melting of a powder layer can cause un-melted, improperly fused particles, which can result in a defect as shown in figure 19.

In the EBM process, the powder bed fusion process can be inadvertently interrupted [2-4]. The under-melt of powder particles within a specimen or part can decrease material strength, resulting in poor quality performance, rendering the specimen defective, therefore, insufficient for use in sensitive industries that rely on high quality, high strength parts. To better understand the quality deficiencies from build defects in the EBM process, a preliminary finite elemental analysis (FEA) was developed to investigate the stress on the v-notch and simulated defect within the specimen.

Stress and strain analysis of metal structures is widely practiced and accepted as standard, reliable engineering examination in industry [35, 36, 42]. Current literature [4,
suggests the unique characteristics of impact strength has a correlation with ductility and brittleness in Ti-6Al-4V solidified materials [1-7]. However, additive manufactured metallic materials have shown to have unique building parameters, which may result in mechanical flaws not found in traditional wrought or cast solidified materials. In the study, the FEM of a standard Charpy v-notch E23 specimen with three different various defect lengths and positions was investigated. The effect of isolated stress and strain on the modeled specimen’s stress concentrated radii of the v-notch and therefore simulated a defect. The characterization of ductility to brittle materials in materials and structural factors influenced the circumstances in which elastic to plastic transition proceeded with regards to crack propagation [39-42]. The Charpy v-notch testing is characterized by the fracture of the material due to impact [40, 42].

Figure 19. Arcam A2 system build defect of ZX Charpy specimen. The specimen was machined and tested at NIST to study the effects of the incomplete melted layer. The Charpy specimen broke (failed) outside the v-notch area as expected. SEM images courtesy of E. Solis-Ramos
Experimental Charpy impact testing data from NIST showed a large decline in energy (J) absorption with defective Charpy v-notch specimens compared to unaffected specimens (figure 13). The defective specimens, which broke outside the v-notch, were built with 5x-recycled wt% O ~ 0.16 powder in the EBM system. This is considered well within the ASTM standards for O content wt% [8, 9]. However, the impact test data shows the drastic decrease in energy (J) absorbed is similar to the decline energy (J) absorbed of the highly oxidized powder mixture with ~ 0.45 wt% O, figure 20.

Industry standards and practices indicate machine defective parts built in the EBM AM Arcam system are generally discarded or destroyed [8, 9, 30]. However, the defective ZX-axis Charpy v-notch specimens with within acceptable O content wt% of 0.16, and were machined and tested for a comparative analysis with the high O content wt% 0.46. This study demonstrates the effect of two notches: the center crack-propagation notch and the simulated variable size notch. The simulated defect location and size effect on stress concentration on the notch radius membrane.

To better understand the mechanical interactions in the Charpy v-notch specimen, a FEA was conducted to investigate the isolated stress and stress concentrations in the Charpy v-notch specimen under static conditions with concentrated force loading. An unpredictable factor in EBM AM building is the resort of a machine defect, which in comparison can be quite close in energy absorption to highly oxidized powder. A model was constructed to quantify the interaction between various simulated defects to the v-notch in the specimen to stress and strain buildup to predictable fracture in the specimen.
under a static load. Calculations were carried out using ANSYS FEM software with isotropic Ti-6Al-4V materials parameters.

![Graph showing Charpy v-notch impact testing results](image)

Figure 20. NIST Charpy v-notch impact testing: build defect specimens with O wt% ~ 0.157, energy absorbed < 10 J (circled left) comparable to highly oxidized (circle right) mixture ~ 0.46 O wt% specimen energy absorbed.

**Experimental Methods**

Using ANSYS FEA software, a 3D Charpy v-notch specimen was designed with the same dimensions as indicated in the ASTM E23 specifications [30]. The FEA of the specimen was designed with a simple, course meshing, and the 3D static structural problem is shown in figures 23 - 24. The analysis was calculated with the Mechanical APDL solver. The Charpy v-notch (CVN) specimen was made of Ti-6Al-4V alloy, with dimensions of
55 x 10 x 10 mm, including the centered, 2 mm deep v-notch with .25 mm radius. The radius of the v-notch (.25 mm) with defect radius (0.065 mm).

The finite element model was designed in accordance with the E23 CVN specification, with a static structure stress and strain analysis performed, using the ANSYS Mechanical APDL solver. The specimen was modeled using a solid isotropic Ti-6Al-4V material specification, which was modified from the ANSYS element library [22, 42]. The automatic course-meshing was applied to the structure with a standard hexahedron and tetrahedron element assignment. The element and nodal assignment was automatically generated with refinement face splitting for modeling the three-dimensional defect (d₃), figures 23a-23f & 24a-24f.

Figure 21. Standard ASTM, E23 Charpy v-notch specimen with no defects.
Figures 22. Charpy v-notch E23 specimen, with 2, 5, & 7.5 mm simulated defect dimensions: height ($d_1$), INNER($d_2$), MID($d_2$), and OUTER ($d_2$): distance from center v-notch and ($d_1 \times d_2$) for three-dimensional cut-in defect ($d_3$).
Figures 23a-23c. INNER (d₂) defect, 2, 5, & 7.5 mm (d₁).

Figures 23d-23f. MID (d₂) defect, 2, 5, & 7.5 mm (d₁).

Figures 23h-23j. OUTER (d₂) defect, 2, 5, & 7.5 mm (d₁).
Charpy v-notch E23 specimens, with 2, 5, and 7.5 mm defect located at one-fourth, one-half, and three-fourths the distance from the center v-notch (d₂).
Figures 24a - 24c. INNER (d₁) defect, 2, 5, & 7.5 mm (d₃).

Figures 24d - 24f. MID (d₁) defect, 2, 5, & 7.5 mm (d₃).

Figures 24h - 24j. OUTER (d₁) defect, 2, 5, & 7.5 mm (d₃).

Charpy v-notch E23 specimens, with 2x2, 5x5, and 7.5x7.5 mm defect located at INNER, MID, and OUTER d₂ region from the center v-notch (d₃).

Using the materials library in ANSYS Workbench, the Ti-alloy (nonlinear) material properties were modified to specifications similar to Ti-6Al-4V metal properties. However, the specifications are valid for cast/wrought alloy material [22] (table 8). The properties for Ti-6Al-4V do not represent the change in high levels of oxidation in the
powder, as characterization and modification of properties will need to be modified in future studies to represent highly brittle material behavior.

Table 8. CVN specimen general material properties for Ti-6Al-4V [22]

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>DENSITY</td>
<td>4430 KG m⁻³</td>
</tr>
<tr>
<td>YOUNG’S MODULUS</td>
<td>96000 MPa</td>
</tr>
<tr>
<td>POISSON’S RATIO</td>
<td>0.33</td>
</tr>
<tr>
<td>BULK MODULUS</td>
<td>94118 MPa</td>
</tr>
<tr>
<td>SHEAR MODULUS</td>
<td>3609 MPa</td>
</tr>
<tr>
<td>YIELD STRENGTH</td>
<td>1100 MPa</td>
</tr>
<tr>
<td>TANGENT MODULUS</td>
<td>2150 MPa</td>
</tr>
<tr>
<td>TENSILE YIELD STRENGTH</td>
<td>1100 MPa</td>
</tr>
<tr>
<td>TENSILE ULTIMATE STRENGTH</td>
<td>1170 MPa</td>
</tr>
</tbody>
</table>

Figure 25. Stress/Strain Bilinear properties for titanium metal, nonlinear material modified for Ti-6Al-4V general specifications [22].
Limitations of FEA model

The model was constructed using similarities to a simple, static structural analysis. The specimen was constrained on the bottom edges, one pinned and the other a roller support, with force applied down the vertical axis (z-axis) to the center of the specimen: 500 N of force at 22°C (ambient room temperature). The boundary conditions were applied at bottom edges were constrained with one pinned with limited mobility displacement related to the nodes on the symmetrical planes: vertical axis with no limitations of movement (free), and x-axis & y-axis = 0 (no movement). and roller axis: A linear analysis was used to calculate the significant change in stiffness, linear-elastic analysis; hence, the nodal stress of both the v-notch and simulated defect was captured by the software. A bilinear analysis would not be compatible with a full explicit analysis because the elastic to plastic transition would not converge properly, and plasticity would be infinitely continuous. This creates improper physical attributes to the material behavior, thus only the static calculations of the nonlinearity of the material were calculated. The FEM for the Ti-6Al-4V CVN isotropic specimens with 18 variations of the simulated defects were created with the automatic course meshing tool in ANSYS. Therefore, the auto-mesh generated a hexahedron dominant element for the CVN with the simulated defect in d₁, (INNER, MID, & OUTER) with variable height (d₂), figures 26 - 29. The auto-mesh generated for the 3D defect, (d₃) was a tetrahedron dominant element type, figures 34 - 36. The bilinear hexahedron auto-meshing drastically reduced computation processing time, which offered reasonable results. The quadratic tetrahedral elements were better; however, the computation processing time was greatly increased for linearized stress analysis.
**Notched Body Stress**

Body loading with notched body-regions will have concentrated stress risers in non-uniform, triaxial stress in the notched location within the specimen body. Thus, an increase in stress concentration will be highest at the notched radius tip. In a typical boundary body setup, the stress and strain at the notch tip can be obtained from an equilibrium equation solution [33-34, 36]. Solid structure components may contain discontinuities in the geometry that cause changes in the state of nominal stress field [34, 36]. Stress risers or raisers can exist in the solid material as interruptions in the continuity of the stress throughout the structure. The distribution of stresses is no longer constant throughout the material, due to the sudden changes in the material geometry. Notches can range from various shapes e.g., grooves, corners cut-outs, or an absences of material [34].

Absorbed energy (J) for CVN specimen is dependent on the dimension and depth of the notch. Notch angle, radius of root, and depth affect the fracture of the materials [33]. When load is applied to the specimen the flow of stress will peak abruptly in discontinues region of the v-notch, which act as a crack initiation point upon load concentration, leading to accelerated fatigue failure. Additional imperfections in the material may interrupt the flow of the stress through the material body. Therefore, the use of finite element modeling (FEM) was used to study the effects of defects in EBM additive manufactured Ti-6Al-4V material.
Results

The stress and strain were calculated in respect to the geometrical discontinuous interrupts in the solid CVN solid isotropic material, with the existence of a v-notch and defect in the structure, there will be a stress riser effect. Therefore, the concentrated mechanical loading applied to the specimens generated secondary forces to the notched radii of the v-notch and defect with the specimen structure. The stress/strain analysis within the v-notch and defect radii was captured the ANSYS software in three distinct finite element models, figure 22. The maximum stress analysis was conducted using labeling convention: INNER, MID, & OUTER regions of the CVN specimen. The CVN geometrical locations of the defects in comparison to the v-notch: d1, d2, & d3 (see figure 22).

Figure 26. CVN v-notch specimen stress analysis using hex dominant course meshing.
Figure 27a-27c. INNER defect 2, 5 & 7.5 mm (d₁) analysis. Stress risers are present in defect height greater than 5 mm.
Figure 28a-28c. MID defect 2, 5 & 7.5 mm (d₁) analysis. Stress risers are present in defect height greater than 5 mm & 7.5 mm (c).
Figure 29a-29c. OUTER defect 2, 5 & 7.5 mm (d₁) analysis. Stress risers are present in defect height greater than 7.5 mm (bottom image).
The CVN specimen model was tested with concentrated force of 500 N for 1 second, with 30 steps capturing. Isolating displacements for y-axis and z-axis on roller edge and accounting for displacement free on x-axis for pinned edge. Stress concentration within the radii of the v-notch and defect are two-dimensional. Therefore, the linearized stress across the radii of the v-notch and simulate defect is consistent. A relative decline in stress was calculated from the v-notch with increase length of crack (figure 33). Respectably, the increased length shows a large increase in von Mises equivalent stress (MPa) > 50% with INNER, MID, & OUTER noted as d_2, with defect length of 2, 5, & 7.5 mm noted as d_1, see tables 14a - 14f. Respectably, the equivalent strain for the v-notch and defect increased. However, the strain of the v-notch increased only slightly with a larger deflection in the defect for the INNER and MID model, table 9a - 9d.

Table 9a. INNER CVN stress equiv. von Mises/strain total equiv. deflection analysis

<table>
<thead>
<tr>
<th>CVN v-notch</th>
<th>von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>v-notch (no defect)</td>
<td>171</td>
<td>1.85</td>
</tr>
<tr>
<td>v-notch w/ 2 mm</td>
<td>73.636</td>
<td>0.78867</td>
</tr>
<tr>
<td>v-notch w/ 5 mm</td>
<td>60.855</td>
<td>0.63944</td>
</tr>
<tr>
<td>v-notch w/ 7.5 mm</td>
<td>64.564</td>
<td>0.72065</td>
</tr>
</tbody>
</table>

Table 9b. INNER CVN stress equiv. von Mises/strain total equiv. deflection analysis of simulated defect

<table>
<thead>
<tr>
<th>CVN Defect (length)</th>
<th>von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>v-notch (no defect)</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>2 mm defect</td>
<td>85.2</td>
<td>0.93669</td>
</tr>
<tr>
<td>5 mm defect</td>
<td>173.2</td>
<td>1.8763</td>
</tr>
<tr>
<td>7.5 mm defect</td>
<td>284.27</td>
<td>3.169</td>
</tr>
</tbody>
</table>
Figure 30. INNER defect analysis. Stress (MPa)/Defect length (mm) indicates length > 1.9 mm.

Table 9c. MID CVN stress equiv. von Mises/strain total equiv. deflection analysis

<table>
<thead>
<tr>
<th>CVN v-notch</th>
<th>von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>v-notch (no defect)</td>
<td>171</td>
<td>1.85</td>
</tr>
<tr>
<td>v-notch w/ 2 mm</td>
<td>69.041</td>
<td>0.74019</td>
</tr>
<tr>
<td>v-notch w/ 5 mm</td>
<td>87.401</td>
<td>0.93249</td>
</tr>
<tr>
<td>v-notch w/ 7.5 mm</td>
<td>77.4</td>
<td>0.92922</td>
</tr>
</tbody>
</table>

Table 9d. MID CVN stress equiv. von Mises/strain total equiv. deflection analysis of simulated defect

<table>
<thead>
<tr>
<th>CVN Defect (length)</th>
<th>von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>v-notch (no defect)</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>2 mm defect</td>
<td>47.006</td>
<td>0.53101</td>
</tr>
<tr>
<td>5 mm defect</td>
<td>96.15</td>
<td>1.0881</td>
</tr>
<tr>
<td>7.5 mm defect</td>
<td>292.04</td>
<td>3.2645</td>
</tr>
</tbody>
</table>
Figure 31. MID defect analysis. Stress (MPa)/Defect length (mm) indicates length > 4.8 mm.

Table 9e. OUTER CVN stress equiv. von Mises/strain total equiv. deflection analysis

<table>
<thead>
<tr>
<th>CVN V-notch</th>
<th>Von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>V-notch (no defect)</td>
<td>171</td>
<td>1.85</td>
</tr>
<tr>
<td>V-notch w/ 2 mm</td>
<td>87.642</td>
<td>0.93544</td>
</tr>
<tr>
<td>V-notch w/ 5 mm</td>
<td>122.71</td>
<td>1.4004</td>
</tr>
<tr>
<td>V-notch w/ 7.5 mm</td>
<td>85.816</td>
<td>1.4045</td>
</tr>
</tbody>
</table>

Table 9f. OUTER Charpy v-notch stress equiv. von Mises/strain total equiv. deflection analyses of simulated defect

<table>
<thead>
<tr>
<th>CVN Defect (length)</th>
<th>Von Mises Equivalent Stress (MPa)</th>
<th>Equivalent total Strain (mm/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>V-notch (no defect)</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>2 mm defect</td>
<td>28.199</td>
<td>0.31137</td>
</tr>
<tr>
<td>5 mm defect</td>
<td>60.001</td>
<td>0.64148</td>
</tr>
<tr>
<td>7.5 mm defect</td>
<td>172.56</td>
<td>1.8752</td>
</tr>
</tbody>
</table>
Figure 32. OUTER defect analysis. Stress (MPa)/Defect length (mm) indicates length > 5.4 mm.

Figure 33. Stress/Defect Length analysis for INNER, MID, and OUTER (d₂) in respect to height (d₁) of defect length (mm).

The CVN specimen under concentrated force of 500 N of force with nine different defects (d₃) within the INNER, MID, & OUTER regions of the specimen. The finite
element models, shown in figure 34-36, represent the variability in geometrical changes in the specimens. Stress linearization differences form across the membrane of the v-notch. The procedure was performed using ANSYS normal linearized stress, shown in figure 37. The v-notch stress concentration decreases with increased height in the simulated defect. The concentration of stress in respect with the size of defect is noticeable with a dramatic shift max. stress > 150 MPa with defect lengths > 6.3 mm, the defect located in the INNER & MID region of the CVN specimen showed to have the maximum stress < 270 MPa, figure 33. The OUTER region showed to have the least effect on stress depletion from the v-notch. Stress concentrations for v-notch area of CVN specimen in model are shown to have the highest equivalent von-Mises stress in models with d<sub>3</sub> < 5 x 5 mm figures 34a, 35a & 36a. However, larger defects with 7.5 x 7.5 mm influence the movement of the structure during loads. The nodal stress concentration within the v-notch decreases as stress concentration in greater in the defect, figure 34c, 35c & 36c.

![Image of CVN v-notch Linearized Normal stress with stress classification line (SCL).](image)

Figure 34.1. CVN v-notch Linearized Normal stress with stress classification line (SCL).
Figure 34a-34c. INNER region of CVN specimen with stress concentration of v-notch. Stress concentration isolated to v-notch with defect > 2 x 2 mm.
Figure 35a-35c. MID region of CVN specimen. Stress concentration decreases with larger defect > 5 x 5 mm (b), (c).
Figure 36a-36c. OUTER ($d_3$) region of CVN specimen.
Figure 36.2. Equivalent stress concentration with $d_3$ defect size of 7.5 x 7.5 mm. Stress-relief is observed in v-notch region with simulated defect. An out of plan shearing effect is seen.

Figure 38. Linearized normal stress across v-notch membrane, INNER location.
Figure 39. Linearized normal stress across v-notch membrane, MID location.

Figure 40. Linearized normal stress across v-notch membrane, OUTER location.

Table 10. Peak normal stress of v-notch membrane with location defect

<table>
<thead>
<tr>
<th>Defect Size (mm)</th>
<th>Stress (MPa) w/ INNER</th>
<th>Stress (MPa) w/ MID</th>
<th>Stress (MPa) w/ OUTER</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 x 2 (1/4)</td>
<td>174.69</td>
<td>176.77</td>
<td>204.12</td>
</tr>
<tr>
<td>5 x 5 (1/2)</td>
<td>266.01</td>
<td>175.74</td>
<td>171.22</td>
</tr>
<tr>
<td>7.5 x 7.5 (3/4)</td>
<td>264.25</td>
<td>253.63</td>
<td>209.98</td>
</tr>
</tbody>
</table>
Figure 41. Primary membrane stresses intensity for linearized stress across the classification line are observed across the v-notch with the variation of the d₃ defect.

Discussion

Electron beam melting layer-by-layer process build defects are unpredictable, which can result in catastrophic, unexpected failure in additive manufactured parts [2, 3, 4, 6]. The potential for EBM AM parts to contain system error defect may be dependent on two main factors: random errors and systematic errors. Errors in the EBM build process, as shown in figure 19, can have a detrimental effect on solidified material. Such damaging effects in the system build can result in comparably low impact-strength of highly oxidized solidified Ti-6Al-4V material, as seen in figure 33. Because the simulated defect is entirely absent of material, stress analysis change in radii of CVN specimen with combination of strain isolation may cause failure, resulting in direct failure outside the crack propagated...
v-notch of the E23 specimen during the mechanical impact testing [39-42]. Un-melted, powder material is removed from the crucial geometries within the specimen during the post EBM AM process of powder reclaiming, which may reveal defects as seen in this research.

The FEM analysis was developed to study the defect size effect on a CVN specimen during impact testing. Defect regions in relation to the v-notch, and conjunction with height to relative failure are determining factors for failure outside v-notch, figures 27-29. Stress analysis from radii of simulated defect in comparison with v-notch radius indicate larger equivalent stress and strain in simulated defects with d₂ > 5 mm, tables 14a -14f. An additional evaluation of modifying the simulated defect to represent a 3-dimensional cut in the CVN specimen. The FEA of this model illustrated the change in stress/strain concentration across the radii of the v-notch (figures 34 -36). However, FE modeling adjustments of refining meshing and comparisons between selective meshing types were not conducted in this study. Thus, future analytical simulation verification benchmark may be conducted. Stress concentrations were variable across the v-notch radius plane. The linearized stress calculated increase within 2 mm of the v-notch for most d₃ defects, figure 38-40. However, specimen failure outside the v-notch became increasing imperative with larger defects and distance from the v-notch (figures 34 – 36). The simulated defect (d₃ size < 2 x 2 mm) shown to introduce the least amount of stress deviations from the v-notch stress-shifting. Therefore, having marginal influence on fracture failure outside the v-notch of the specimen. The defect location closest to the v-notch (INNER & MID), [figures 35b,
35c & 36b, 36c] with a size > 5 x 5 mm was observed to significantly increase defect stress concentrations. Thus, the possibility of the facture failure at the defect is more likely.
Chapter Five: Conclusion

Summary

The electron beam melting process in additive manufacturing creates challenges with quality of solidified titanium alloy parts in the powder-bed fusion system. The metallurgical effects of powder Ti-6Al-4V material during the EBM AM process creates a uniqueness in micro characterization and mechanical properties in comparison with traditional wrought and cast processed solidified materials. Advanced isothermal, open-air oxidation behaviors directly influence the microstructure of powder particles and bulk powder material, which also have shown a strong correlation to deviations in consolidated material qualification in chemical and mechanical analysis. The evolution of the powder characterization, microstructure and mechanical qualification, are directly related to bulk oxygen pickup during powder recycling and post treatment of consolidated material.

The powder bulk oxygen content wt% > 0.3, with a dramatic linear decline in impact strength with oxygen content wt% of 0.46 [figure 4] influenced the microstructure and mechanical impact properties of the Ti-6Al-4V material. The decrease in energy (J) absorbed (< 10 J) can be attributed directly to melting large amounts of oxides within the consolidation EBM AM process. A correlation with the layer-by-layering effect of orientation, parts manufactured in the EBM AM process are therefore susceptible to defects during the additive manufacturing process. In addition, as seen in the supporting literature, the direct high temperature isothermal exposure in open-air and very high surface area of
the bulk powder can account for the exponential, oxidization parabolic-effect of the bulk powder material, table 2.

It has been shown in this study the measure of oxygen content wt% of bulk powder > 0.3, most notably for 0.46, in consolidated material. Evident microstructure characteristic changes of powder particles bulk Ti-6Al-4V material were present during the study of the artificially oxidized effect on the powder. Consolidated manufactured impact Charpy v-notch strength, and advanced FEM analysis of the mechanical effects of defective solidified material were shown to have a significant effect on mechanical impact testing. Thus, there was a reduction in concentration stress in the v-notch as the length of the defect increased in size.

**Conclusion**

The artificial oxidation effect of the Ti-6Al-4V powder at temperatures greater than 500°C and 650°C/4h with calibrated mixtures in batches for manufacturing consolidated material Charpy v-notch impact testing and FEM was evaluated, which was established on micro-characterization of powder chemical analysis, and the additive manufactured Charpy v-notch specimens impact testing. The mechanism of oxidation on the bulk powder material as well with the introduction of highly oxidized powder material in the Arcam electron-beam melting additive manufacturing process was compared with virgin Ti-6Al-4V consolidated material. Impact energy absorption of high oxygen content Charpy specimens were similar to low oxygen content specimen within the EBM AM build defect.
FEM observations for simulated defects created isolated stress/strain greater than the v-notch, thus failure outside notched region was evident.

Major conclusions are as followed:

- The artificial oxidized Ti-6Al-4V powder presented a rapid increase in bulk wt% oxygen with temperatures > 500°C/4h. The surface oxide layer growth showed a dramatic increase in particle surface oxides, and oxygen content wt% in bulk Ti-6Al-4V material.

- Open-air isothermal oxidization of Ti-6Al-4V powder at high heat > 650°C/4h exhibited a parabolic-effect in large accumulation of oxygen in powder material.

- The oxygen content of powder before build process and post manufactured, solidified Ti-6Al-4V material had a relativity consistent oxygen wt% in respect to declining mechanical impact energy absorption (J) during testing.

- Chemical analysis of artificially oxidized bulk powder was consistent with chemical analysis of consolidated high oxidized mixture material.

- A large increase in brittleness of consolidated material was observed in powder batch mixtures with oxygen content wt% 0.3 & 0.46, with slight increase in energy absorption with particular ZX orientation and HIP treatment.

- Post HIP treatment of EBM specimens yielded better impact testing results than non-HIP treated specimens with very insignificant increase in energy absorption for consolidated material with oxygen content wt% > 0.46.

- Defective CVN specimens with unfused EBM build layer was shown to have similar impact energy absorption to high O wt% CVN specimens.
• Defective ZX-axis CVN specimen with O wt% 0.16 failed outside v-notch with energy absorption < 7 J, which was equal to lowest performing ZX-axis CVN specimen with O wt% ~ 0.45.

• FEA of CVN specimen indicated a large influence in dependency of fused, melted material versus absent material during build defect; stress/strain concentrations in defective regions of specimen yield increase impact fracture failure outside of center v-notch.

• Intense stress concentration outside center v-notch with large defects within FEM CVN Ti-6Al-4V structure > 5 mm: isolated stresses were observed in radius of defect.

• Variability in defect size and length in FEM of CVN specimen effect stress & strain across v-notch area.

• Larger simulated defect > 5 x 5 mm closest to the center v-notch in FEM exhibited the greatest isolated stress concentration than simulated defect < 2 x 2 mm.

• Peak membrane linearization v-notch stress increases to proximity of defect location.
Work Cited


74


