MICROSTRUCTURAL CHARACTERIZATIONS AND MODELING OF TI-6AL-4V
PARTS MADE BY ELECTRON BEAM ADDITIVE MANUFACTURING (EBAM)

by

XIBING GONG

Y. KEVIN CHO, COMMITTEE CHAIR

VIOLA L. ACOFF
JOHN BAKER
DANIEL J. FONSECA
LAURENTIU NASTAC
MARK L. WEAVER

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ABSTRACT

The electron beam additive manufacturing (EBAM) technology builds parts layer-by-layer from metal powder using an electron beam. EBAM is capable of producing fully melted metallic parts with fine microstructures and superior mechanical properties. However, the microstructures, determined by the process thermal history, and yet, governing the attainable mechanical properties, are very complex in EBAM. Further, there has been little systematic study of the relationship between the process parameters and the microstructures in EBAM. Therefore, understanding the microstructure evolution is vital to achieve the desired properties of the fabricated components.

The primary objectives of this research are: (1) to characterize Ti-6Al-4V powder in EBAM, (2) to analyze the process parameter effects on the microstructure, (3) to model the microstructure evolution. The research approaches include: (1) characterization of the powder using metallographic method, (2) microstructural characteristics of EBAM solid parts made by various beam scanning speeds, (3) numerical modeling to conduct thermal analysis and phase field modeling to simulate microstructure evolution, and phase transformation kinetics to calculate the phase composition.

The major findings are summarized as follows. (1) Preheating results in metallurgical bonds of the powder during the EBAM. The calculated porosity of the preheated powder is about 50%. (2) The specimens of the part side surfaces show columnar prior β, while the scanning surface specimens show equiaxed grains. A higher beam scanning speed leads to a smaller grain size. The width of the columnar structure decreases with the increase of the scanning speed, 109.7 μm at 214 mm/s vs. 37.1 μm at 529 mm/s. The α-lath thickness is 1.5
μm for the sample with the lowest scanning speed, while the thickness is 1.0 μm for other scanning speed samples. (3) The finite element method is able to simulate the temperature history and melt pool geometry during EBAM. The phase field model is able to simulate the morphology and solute concentration of EBAM parts. In addition, a larger beam scanning speed results in a higher percentage of martensitic structures, and the fraction of martensites is about 30% for the highest scanning speed sample compared to around 10% for the lowest scanning speed sample.
LIST OF ABBREVIATIONS AND SYMBOLS

\( \beta \)  The body centered cubic beta phase of titanium and its alloys
\( \alpha \)  The hexagonally closed packed phase of titanium and its alloys
\( \alpha + \beta \)  A two phase mixture
\( \alpha' \)  Martensitic structure in titanium or titanium alloys
\( \alpha_m \)  Massive shape of \( \alpha \) phase
\( \alpha_p \)  Primary \( \alpha \) phase
\( k \)  Thermal conductivity
\( x \)  Direction of electron beam travel
\( y \)  Direction of layer addition (depth)
\( C_0 \)  Initial concentration,
\( \beta \)-transus  \( \beta \) phase transus temperature
\( M_S \)  Martensite start temperature
\( T \)  Temperature of the thermal model
\( \dot{Q}_{(x,y)} \)  Absorbed heat flux from the thermal model
\( T^* \)  Temperature at the solid-liquid interface
\( c \)  Specific heat capacity
\( T_L \)  Liquidus temperature
\( T_S \)  Solidus temperature
\( \rho \)  Density
\( v \)  Constant speed of the moving heat source in the \( x \) direction
\( L \)  Latent heat
\[ dU \] Internal thermal energy
\[ H \] Enthalpy
\[ T_{preheat} \] Preheating temperature in EBAM
\[ \Delta T \] Undercooling, difference between \( T^* \) and \( T_{eq} \)
\[ f_S \] Solid fraction
\[ f_L \] Liquid fraction
\[ P_B \] Beam power
\[ \eta \] Absorption efficiency
\[ U \] Acceleration voltage
\[ I_b \] Beam current
\[ t_{layer} \] Powder layer thickness
\[ \varphi \] Porosity
\[ d_P \] Beam penetration depth
\[ \phi \] Phase field
\[ f^S \] Free energy of solid
\[ f^L \] Free energy of liquid
\[ c_L \] Liquid concentration
\[ c_S \] Solid concentration
\[ c^*_L \] Liquid concentration at the interface
\[ c^*_S \] Solid concentration at the interface
\[ \varepsilon \] Gradient energy coefficient
\[ w \] Height of double-well potential
\[ \mu^L \] Chemical potential of liquid
\[ \mu^S \] Chemical potential of solid
\[ D(\phi) \] Solute diffusion coefficient
\( K \)  
 Partition coefficient

\( m \)  
 Liquidus slope

\( \nu \)  
 Magnitude of anisotropy

\( \eta_a \)  
 Magnitude of anisotropy

\( \theta \)  
 Angle between normal direction of interface and horizontal direction

\( \chi \)  
 A random number distributed uniformly between -1 and 1

\( \omega \)  
 Amplitude of the fluctuations

\( M \)  
 Interface mobility

\( \lambda \)  
 Interface width

\( \sigma \)  
 Interface energy

\( f(\alpha) \)  
 Volume fraction of \( \alpha \)

\( f(\beta) \)  
 Volume fraction of \( \beta \)

\( t \)  
 Time duration

\( k_r \)  
 Reaction rate constant

\( n \)  
 Avrami constant

\( c_m \)  
 Material constant in martensitic phase transformation
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CHAPTER 1

INTRODUCTION

1.1 Additive Manufacturing

Additive Manufacturing (AM), also known by various terms, e.g., direct digital manufacturing (DDM), rapid prototyping (RP), or solid freeform fabrication (SFF), is an emerging technology based on “layer-by-layer” fabrications, by which physical solid parts are made directly from electronic data (Murr et al., 2009). AM is a relatively novel idea to fabricate complex, net-shaped metal components (Baufeld et al., 2010). According to the description from Figure 1.1 (Gibson et al. 2010), any AM process can be generally divided to 8 stages: CAD, STereoLithography (STL) convert, file transfer to machine, machine setup, build, remove, post-processing, and application.

Figure 1.1. Generic process of CAD to part, showing all 8 stages (Gibson et al., 2010).
There are now a large number of technologies which employ this method of manufacture, and some of the more widely used, such as SLA, fused deposition modeling (FDM), selective laser sintering or melting (SLS/M), 3D printing (3DP), and electron beam additive manufacturing (EBAM), etc. Metallic raw materials for AM usually come under powder state. Generally, two different powder-based AM techniques for metals or alloys are currently under investigation: laser based technologies, electron beam technologies. Some of the systems in this group, such SLS/M and EBAM, have a wide usage in industry. In SLS/M and EBAM, a high intensity energy beam (laser or electron) is applied to melt powder and form a solid component after solidification. Compared to SLS/M, the beam intensity and build speed is much higher for EBAM technology (Vayre et al., 2012).

The most important advantages of additive fabrication are:

(1) High speed of the manufacturing process and allows possibility to quickly change design (Holmström et al., 2010).

(2) Part geometry flexibility: unlike conventional processes, AM can produce parts of almost any desired form and can almost be free from geometrical manufacturing constraints.

(3) No tooling cost and maximum material savings. Material is added and not subtracted (Petrovic et al., 2011). The part is obtained directly from its 3D CAD model; an additional advantage is the almost absolute absence of human errors in production.

(4) Full-density of final parts or cellular components. Fabrication of free-form enclosed structures. Additive technologies are capable of fabricating free-form channels as well as different forms of latticed structures (Gong et al., 2012).

AM has been advanced significantly, but its growth in production applications has been limited by some factors. The AM technologies encounter some pending challenges in the near future:
(1) Process failures and surface finishing: the observed errors of the AM parts are significantly larger than those of typical machined parts, even for the solid part, by at least an order of magnitude (Cooke and Soons, 2010).

(2) Process control: need to improve the precision and reliability of the manufacturing process and to increase throughput while maintaining consistent quality.

AM technologies have vast applications in industries, especially in aerospace and medical industries. For aerospace, complex manufacturing processes must be developed to meet the industry’s stringent requirements and to ensure that products can achieve the robust performance levels (Lyons, 2012). The costs for high performance materials are high due to tooling, materials, and labor needed to produce the finished component by manufacturing processes. AM approach would eliminate the need for tooling, allow design flexibility, reduce or eliminate the amount of machining, permit on-demand design changes and greatly shorten the time to production (Mahale et al., 2007).

For medical applications, custom designed implants are preferred or required and AM components have been used as joint replacements, bone plates, etc. (Murr et al., 2009). AM methods permit to scan and build a physical model of defective bones from patients and give doctors a better idea of what to expect and plan better the procedure; this will save cost and time and help achieve a better result (Petrovic et al., 2011). The research of medical applications of AM has been reported frequently, and some of the technologies includes SLS (Vandenbroucke and Kruth, 2007; Giannatisis and Dedoussis, 2009; Gebhardt et al., 2010), and EBAM (Christensen et al., 2007; Murr et al., 2009).

1.2 Background and Motivation

In recently developed AM technologies, EBAM has been widely used to manufacture metal or alloys, especially for titanium (Ti) alloys. Ti and Ti alloys are widely used in many industries due to their desirable and versatile combination of good mechanical and chemical
properties, especially in aerospace and medical industries. Ti-6Al-4V alloy is the most widely used Ti alloy. The excellent combination of specific weight and mechanical properties has promoted its use in the aerospace, automotive, and marine equipment (Sha and Malinov, 2009).

EBAM is one of the few AM technologies capable of making full-density metallic parts and has dramatically extended the applications of EBAM. The inside of the EBAM machine could be seen from Figure 1.2. This process represents a promising opportunity to increase the use of Ti alloys due to its process flexibility, non-reactive environmental requirements and fast processing. Additionally, this technique offers a more efficient and economical way to manufacture Ti parts, by a continuous effort to improve the manufacturing practice itself delivering a higher-quality product and decreasing the cost on post-processing once the part is completed. Therefore the use of the EBAM process for processing Ti alloys such as Ti-6Al-4V, represents a viable alternative to manufacture fully functional parts for aerospace and medical applications at lower cost and lead times (Alderson, 2012). Figure 1.3 presents an example of part manufactured by EBAM. In addition, the use of EBAM on Ti-6Al-4V alloy has the ability to produce fine microstructure with strengthening phase, which makes the components stronger than those from conventional manufacturing methods.
However, since it is a relatively new technology, there are still many process aspects to be fully understood, e.g., the thermal history of the EBAM technology. Also, the microstructure evolution and phase transformation of Ti-6Al-4V are complicated. In addition,
some possible defects, such as layers delamination, are observed from the EBAM components, which limit the application of the EBAM technology on Ti-6Al-4V alloy. Understanding and controlling the microstructure evolution are of great importance to achieve the desired mechanical properties of the components.

First, the thermal history is complicate. The influence of the addition of the powder layers should be also considered to reflect the accurate temperature history. Few literatures were focused on the thermal history of the whole process. The influence of the build parameters has also not been studied yet.

Second, the influence of the thermal history on the microstructure evolution is still unknown. The preheating, which serves to aggregate the precursor powder and reduce the residual stress for the final parts, is critical to influence the subsequent melting and solidification process. The study of preheating helps to control the thermal history and microstructure evolution of the EBAM components.

Third, the prediction of the microstructure evolution of the EBAM components has not been studied yet. The morphology of the microstructure is critical to determine the properties of the components. Models could be adopted to simulate the microstructure evolution.

1.3 Research Objectives and Scope

The main focus of this study is to evaluate the thermal history and microstructure evolution of Ti-6Al-4V alloy produced by EBAM. The objectives of this research include the following:

(1) Investigating the microstructure of the Ti-6Al-4V powder after preheating process, and study the difference of morphology and microstructure among different conditions, such as the powder-bed sample and the powder-enclosed sample. The microstructural
characterization of the sintered powder will help to control the thermal history and microstructure evolution of the EBAM.

(2) Applying a numerical finite element (FE) model of EBAM to investigate the temperature history during the EBAM process. A moving heat source will be applied on two successive powder layers. The influence of the process parameters, such as beam diameters and scanning speeds, will be investigated.

(3) Applying the phase field method to model the columnar structure growth during the solidification process (liquid phase to primary solid phase of $\beta$). The growth of columnar structure will be studied, and the morphology and concentration field will be investigated. In addition, the influence of the undercooling, noise and anisotropy on the columnar structure growth will be studied.

(4) Computing the distribution of phases ($\alpha$, $\beta$, and $\alpha'$) in the final microstructure of Ti-6Al-4V alloy, and conduct the microstructure experiments for the solid EBAM parts to verify the simulation and computation models. The phase transformation kinetics will be applied to make quantitative analysis of the phases.

This work would contribute to the understanding of microstructure formation and resulting mechanical properties of EBAM-built parts as well as provides a fundamental basis for optimization of the EBAM process.

First, the powder particles which are processed after preheating will be investigated to study the influence of the preheating process in EBAM. Then, due to the complex thermal history and microstructure evolution of the EBAM on Ti-6Al-4V alloy, a thermal model will be applied and a microstructure evolution model will be constructed. For the microstructural characterization, solid components with different scanning speeds parameters will be studied. For the thermal history, a FE model will be applied to simulate the temperature distribution and history during the EBAM. To simulate microstructure evolution, phase field model will
be developed to study the columnar structure growth during the rapid solidification. Subsequently, for the solid state transformation, the numerical calculation using phase transformation kinetics will be conducted to study the phase constitution according to the temperature history and cooling rate.

1.4 Outline of Dissertation

The layout of this proposal is as follows.

Chapter 2 presents a thorough literature review of the powder-based EBAM technology. The chapter introduces the general aspects of EBAM, and includes the unique characteristics, advantages and challenges of EBAM. Moreover, it includes extensive discussions of microstructures, mechanical properties, and geometric attributes, which impact the application ranges of EBAM parts, with focus on commonly used Ti alloys.

In Chapter 3, the introduction of Ti and its alloys, especially Ti-6Al-4V, is studied. It provides an overview of microstructure and phase transformation of Ti-6Al-4V alloy. Also, the microstructure modeling is reviewed.

In Chapter 4, the preheating of the Ti-6Al-4V alloy powder is studied. The microstructure characterization, size distribution and thermal conductivity of the preheated powder are investigated.

In Chapter 5, the influence of the scanning speed on the microstructure of the Ti-6Al-4V parts processed by EBAM has been investigated. The relationship of the scanning speed and resulted microstructure is studied.

Chapter 6 studies thermal analysis, microstructure modeling, and phase transformation kinetics in EBAM. The morphology and solute concentration of Ti-6Al-4V during EBAM is investigated by using a phase field model, and the phase constitution in EBAM. The phase fractions are calculated by applying phase transformation kinetics.

The last chapter proposes the future work of this research.
References


CHAPTER 2
LITERATURE REVIEW

EBAM is a relatively new AM technology (Arcam.com, 2011). Similar to electron-beam welding, EBAM utilizes a high-energy electron beam, as a moving heat source, to melt and fuse, by rapid self-cooling, metal powder and produce parts in a layer-building fashion. Moreover, EBAM is one of a few AM technologies capable of making full-density functional metallic parts, drastically extending AM applications. In particular, the ability of direct fabrications of metallic parts can significantly accelerate product designs and developments in a wide variety of metallic-part applications, especially for complex components, e.g. fine network structures, internal cavities and channels, which are difficult to make by conventional manufacturing means (Arcam.com, 2011; Biamino et al., 2010).

EBAM machines were first commercialized, around 1997, by Arcam AB Corporation in Sweden. Because EBAM has many unique characteristics such as high energy efficiency, high scan speed, and moderate operation cost, the technology has attracted, in recent years, increased interests from different industries. The use of an electron beam offers extensive features such as higher build rates due to increased penetration depths and rapid scanning speeds. Since then, many research groups have been studying the EBAM technology from different aspects and for various applications. Despite the potential benefits over conventional manufacturing technologies, EBAM still has a few process deficiencies, such as process stability, part defects and quality variations (Zäh and Lutzmann, 2010), etc. As EBAM technology is relatively new, there have not been detailed reviews. The objective of this paper is to offer a survey of various investigations into EBAM, especially for the study of the Ti alloy parts, along with the EBAM part microstructures and associated mechanical properties.
In addition, modeling and simulation of the EBAM process, though rare, for process understanding and advancements are also discussed.

2.1 EBAM Process

2.1.1 Working Principle

A conceptual schematic of an EBAM machine is shown in Figure 2.1 (Biamino et al., 2010). The principle is similar to that of a scanning electron microscope. A heated tungsten filament, in the upper column, emits electrons which are collimated and accelerated to a kinetic energy of about 60 keV. The electron beam is controlled by two magnetic coils, which are housed in the lower column. The first one is a magnetic lens which focuses the beam to the desired diameter, and the second one deflects the focused beam to the desired point on a build platform. The electron-beam gun itself is fixed, no moving mechanical parts involved in beam deflections. The beam current is controlled in the range 1 to 50 mA and the beam diameter can be focused down to about 0.1 mm. The raw materials used in EBAM are metallic powder and the characteristics and quality of powder strongly affect the process performance. In general, fine powder, with a diameter between 45 and 100 µm, are used. Figure 2.2 shows scanning electron microscopic (SEM) images of Ti-6Al-4V powder: (a) low magnification showing a population of particle sizes and (b) high magnification of an individual powder. In the chamber of the lower part of the machine, the metal powder layers are formed by a raking mechanism. The typical layer thickness is in the range 0.07 to 0.15 mm. The computer-controlled electron beam scans over the powder layer in a predefined pattern and consolidates the desired areas into solid and dense metals. The beam has to first scan at a high speed (order of 2 m/s) in multiple passes to preheat powder to a sintered state, while a beam scan on the order of ~0.5 m/s is used during the melting cycle. Then, a new powder layer is laid on top and the scanning process is repeated until all layers are completed.
Figure 2.1. Schematic drawing of an Arcam EBAM machine (Biamino et al., 2010).

Figure 2.2. SEM images of Ti-6Al-4V powder: (a) particles with different sizes and (b) an individual particle.
The entire process takes place under a high vacuum. The typical pressure of residual gases in an EBAM machine is $10^{-1}$ Pa in the vacuum chamber and $10^{-3}$ Pa in the electron gun (Biamino et al., 2010). During the melting process, a low pressure of inert helium gas ($10^{-1}$ Pa) is added to the vacuum chamber to avoid build-up of electrical charges in powder. When all layers have been completed, the built part is allowed to cool inside the process chamber, which is then filled up with helium as to assist cooling. Because of radiation from electrons, the process observation is not as accessible as other AM technologies, only through a leaded-glass viewport. Therefore, what exactly happens inside the build chamfer is not as well perceived as other AM processes. Recently, Oak Ridge National Laboratory published a video animation of the EBAM process (2011) that offers good illustrations of the process details, especially to those without access to an EBAM machine.

2.1.2 Applications and Challenges

Due to EBAM’s unique capability, it is especially beneficial to such industries as the aerospace sector, creating new opportunities for both prototyping and low volume productions. The time, cost, and challenges of machining or other processes are eliminated, which makes the components readily available for functional testing or installation on a system, e.g. aircraft (Yu et al., 2009). Additionally, the additive process opens a door to new design configurations (e.g., cellular structures) and weight-reduction alternatives.

The energy density of the electron beam is high enough to melt a wide variety of metals and alloys. The EBAM processes have the potential to work with many material classes, for example, aluminum alloys (Yu et al., 2009), tool steel (H13) (Cormier et al., 2004), and cobalt-based superalloys (Gaytan et al., 2010), etc. However, Ti alloys, in particular, Ti-6Al-4V, were the first material extensively researched, also widely used in EBAM technologies. Ti alloys have numerous potential applications as a consequence of their superior properties: low density, high mechanical strengths, corrosion resistance, human
allergic response, and good biocompatibility (Murr et al., 2009; Gaytan et al., 2010; Parthasarathy et al., 2010). The industrial processing routes for Ti alloys include ingot casting, powder processing, ingot forging, and sheet production by hot-rolling. Using traditional processes makes it difficult to manufacture highly complex and functional Ti-alloy parts, such as artificial knee joints, hip joints, and bone plates, etc. EBAM has been used for fabrications of specific net-shaped parts for use within both aerospace and medical implant industries. The fabrication of implants from patient specific data with adaptation to the region of implantation is made possible with EBAM (e.g., temporomandibular joint prosthesis in Figure 2.3(a), eliminating expensive secondary processing such as machining or forming and related lead times. Moreover, with geometric freedom, EBAM has enabled one step fabrications of intricate architecture (meshed, porous, cellular), e.g., porous custom implants with controlled porosity to meet the requirements of the anatomy and functions at the region of implantation. In addition, EBAM has been used to assist the Environmental Control and Life Support System (ECLSS) group of NASA for a new design of hardware, Figure 2.3(b), to pack zeolite materials for improving CO₂ removal from the International Space Station environment (Goods, 2008). Using EBAM, the total production time is 1 week including machining the matted surfaces in contrast to 13 weeks when using traditional processes. In addition, the ability to produce a negative Poisson’s ratio structure, the so-called “auxetic behavior” is another unique capability of EBAM. A negative Poisson’ ratio leads to higher impact and shear resistances, and fracture toughness. Schwerdtfeger et al. (2010) found negative Poisson’s ratios in the range of -0.2 to -0.4 depending on the orientation.
Since Ti-6Al-4V is the most widely used material in EBAM, the major effort of this review is focused on this group of materials, its usages, applications and EBAM part characteristics.

2.2 Microstructures

Typically, Ti-6Al-4V samples from EBAM show an ordered lamellar microstructure, consisting of extremely fine grains, as can be expected by the thermal characteristics of the EBAM process: small melt pool and rapid cooling. The phases of EBAM Ti-6Al-4V samples include some α phase at the β grain boundaries, which is finer than that obtained by metal casting (Gulzar et al., 2009). Facchini et al. (2009) investigated microstructures by x-ray diffratometry (XRD) and reported that the main constituent is α HCP phase with only a small contribution of β phase in EBAM Ti-6Al-4V. The HCP pattern can be attributed to both α phase and the α'-martensite, which is spatially smaller than the α-phase platelets. Koike et al. (2011) compared the EBAM microstructures to counterparts from cast and wrought Ti specimens. For the cast specimen, a typical α-case microstructure consisting of columnar α-crystals can be clearly observed. The microstructure of the wrought Ti-6Al-4V specimen consisted of slightly elongated α grains and intergranular β grains which is a typical microstructure from heat treatment conditions. On the other hand, no α-case was observed.
near the surface of the EBAM specimens. However, Murr et al. (2009) observed that the EBAM Ti-6Al-4V prototypes exhibit the \( \alpha \)-phase, acicular platelet microstructures similar to commercial wrought products, while the mesh and foam prototypes exhibit mainly \( \alpha' \)- martensitic platelets or mixtures of \( \alpha \) and \( \alpha' \) platelets, giving rise to harder structures and consequently greater strengths. Figure 2.4 below compares the microstructures of Ti-6Al-4V specimens from (a) EBAM and (b) casting (Murr et al., 2009). There are also studies reporting different microstructures (grain size and morphology) obtained from the EBAM, attributed to noticeably different cooling rates (Murr et al., 2009; Bontha et al., 2009) and re-melting (Wanjara et al., 2005).

![Figure 2.4. Microstructures of Ti-6Al-4V specimens made by (a) EBAM vs. (b) casting (Murr et al., 2009).](image)

As to chemistry specification, Heinl et al. (2007) tested the element composition of the EBAM parts. The authors claimed that the composition of the EBAM parts conforms to
the standard specifications for Ti-6Al-4V alloy castings for surgical implants. However, Gaytan et al. reported that the chemistry of EBAM made Ti-6Al-4V parts may have 10 to 15% reduction in Al content (Gaytan et al., 2009) possibly due to a higher vapor pressure of Al.

The gas voids or porosities are a typical defect in EBAM parts, which is exemplified in Figure 2.5 below. Gaytan et al. (2009) investigated porosity defects and control during EBAM and attempted using hot-isostatic-pressing (HIP) to eliminate gas voids in built samples. The result showed that while the voids can be largely eliminated by a single standard HIP cycle, remnants sometimes persist. In addition, some gas bubbles actually came from recycled powder and stay in EBAM-built parts. Because of the melt and liquid phase surface tension as well as the low gas pressure, it is essentially impossible to eliminate the intrinsic gas bubbles in EABF parts. Reported by Gaytan et al. (2009), however, because of the small void size (order of 10 µm), they may not impact the mechanical properties of EBAM-built parts.

![Figure 2.5. An example of gas void: optical metallograph of a polished and etched section (Gaytan et al., 2009).](image)

In addition, electron beam-material interactions reduce the EBAM process stability. The melt pool instability is affected possibly by an inadequate energy density transmitted
from the electron beam into the powder, and it may result in the balling effect, when the surface tension of molten liquid exceeds the wetting ability of the previously solidified layer (Cansizoglu et al., 2008). The melt balls further prevent the process from continuation due to rough textures of the top layer. Moreover, as EBAM relies on selective solidification of the top powder layer, energy is inserted into the material in a non-uniform way. Large temperature gradients may emerge due to selective heating of powder areas and thus, residual stresses may be induced. If the residual stresses exceed the bonding abilities between layers, it results in delamination, which depends on the scanning strategy (Schwerdtfeger et al., 2009). Specifically, the orientation of the scan vectors has a considerable influence to delamination (Zäh and Lutzmann, 2010).

It has been reported that operating parameters have significant effects on the part characteristics (Murr et al., 2009; Bontha et al., 2009), quality consistency and process performance. Murr et al. reported that variations in melt scan, beam current, and scan speed affect the EBAM built defects such as porosity (Murr et al., 2009), and may cause significant property-performance variations (Murr et al., 2009). In general, the beam power, diameter, and speed, as well as the pre-heat temperature are four major process parameters; the first three are tied to the thermal cycle variables, temperatures and cooling rates, and the pre-heat temperature governs the sintering state of powder prior to the melting scans.

Rapid self-cooling, which results in the EBAM layer building/bonding mechanism, has been noted to be different upon comparing different melt-scan parameters. Gaytan et al. reported that in Ti-6Al-4V, the differences in melt-scan parameters create microstructural variations characterized by sizes or dimensions as well as phase differences and dislocation density variations (Gaytan et al., 2009). Bontha et al. (2009) investigated the effects of process parameters on solidification microstructures in beam-based fabrications. It shows that variations in the beam power and speed can alter both solidification cooling rates and thermal
gradients by several orders of magnitude, which have a significant effect on resulting microstructures. Results specifically for Ti-6Al-4V suggest that process size-scale can have a significant effect on microstructures, including a transition from columnar to equiaxed microstructure at higher powers.

2.3 Mechanical Properties

EBAM part properties have been frequently investigated. Some studies indicated that properties of EBAM parts are comparable to those from conventional processes (wrought) (Murr et al., 2009; Svensson and Ackelid, 2010). However, other research indicated improved hardness of EBAM parts (Gaytan et al., 2009). Changes in local chemistry and different microstructures have been suggested as possible causes.

2.3.1 Tensile Testing

Tensile testing has been widely used to characterize the mechanical properties of EBAM parts. Some researchers (Facchini et al., 2009) found that the ultimate tensile strength (UTS) of EBAM built specimens is higher than the wrought or annealed ones, with a lower ductility. However, others (Koike et al., 2011) presented that the UTS and ductility of the cast and wrought Ti-6Al-4V specimens were higher than those of EBAM counterparts. The reason for the difference could be attributed to the variation in the built parameters, which result in different structures such as composition, structures, pore size, and porosity distribution, etc. Koike et al. (2011) investigated and reported that the yield strength (YS) and UTS of the EBAM specimens were 735 and 775 MPa, respectively. The ductility was 2.3% elongation. These values were comparable to those of cast specimens, which exhibited slightly higher UTS and ductility. These differences were considered to be due to rippled, rough specimen surfaces and possibly higher oxygen content in the EBAM specimens (0.34 vs. 0.22%), originated from the alloy powder. Other researchers showed better ductility (elongation) in EBAM parts. Murr et al. (2009) studied that the elongation of the EBAM
samples shown is about 16–25% compared to the elongation of 12–14% for wrought alloys. Table 2.1 below summarizes the mechanical properties of EBAM vs. wrought Ti-6Al-4V specimens.

Table 2.1. Comparisons between EBAM and wrought Ti-6Al-4V

<table>
<thead>
<tr>
<th></th>
<th>YS (GPa)</th>
<th>UTS (GPa)</th>
<th>Ductility (%)</th>
<th>H (GPa)</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>EBAM</td>
<td>1.10–1.15</td>
<td>1.15–1.20</td>
<td>16–25</td>
<td>3.8–4.1</td>
<td>(Murr et al. 2009)</td>
</tr>
<tr>
<td></td>
<td>0.735</td>
<td>0.775</td>
<td>2.3</td>
<td>3.619</td>
<td>(Koike et al., 2011)</td>
</tr>
<tr>
<td></td>
<td>0.83</td>
<td>0.915</td>
<td>-</td>
<td>3.2</td>
<td>(Facchini et al., 2009)</td>
</tr>
<tr>
<td>Wrought</td>
<td>1.17–1.22</td>
<td>1.23–1.29</td>
<td>12–14</td>
<td>3.8–4.3</td>
<td>(Murr et al. 2009)</td>
</tr>
<tr>
<td></td>
<td>0.860</td>
<td>0.931</td>
<td>14</td>
<td>3.207</td>
<td>(Koike et al., 2011)</td>
</tr>
<tr>
<td></td>
<td>0.79</td>
<td>0.87</td>
<td>-</td>
<td>-</td>
<td>(Facchini et al., 2009)</td>
</tr>
</tbody>
</table>

2.3.2 Compressive Strength

Compressive testing has also been used to evaluate EBAM parts, mainly for meshed, porous, or cellular structures in biomedical applications. Li et al. (2009) utilized EBAM to fabricate porous Ti-6Al-4V parts with fully interconnected, controlled internal pore architecture. The compressive test showed that a linear elastic deformation stage, followed by a long plateau stage with a nearly constant flow stress to large strains, in which cells collapse due to buckling and plastic-yielding, and the final stage with the stress reaching the maximum value. The authors reported that 66% porosity Ti-6Al-4V parts has a compressive strength approximately 116 MPa and the Young's modulus of 2.5 GPa, close to the human cancellous bone.

Parthasarathy et al. (2010) reported the stiffness and elastic modulus for the open cellular Ti-6Al-4V foams varied with density consistent with the Gibson-Ashby foam model (Gibson and Ashby, 1988). Moreover, the stiffness varies inversely with porosity or pore density consistent with literature values for a number of metal and alloy systems, especially
aluminum. The authors also reported that both compressive stiffness and strength decrease with an increase in the porosity. Heinl et al. (2008) investigated cellular Ti-6Al-4V structures with a controllable interconnected porosity by EBAM. The mechanical properties of the structures were examined under compression load and compared with the properties of human bone. It is found that cellular solids have an important influence to their stiffness and strength.

2.3.3 Other Testing

Other types of mechanical testing such as hardness tests and flexural tests have also been used in studying mechanical properties of Ti alloys processed by EBAM. Koike et al. (2011) investigated EBAM Ti-6Al-4V specimens by Vickers microhardness testing. The hardness of EBAM specimens were higher than that of the cast or wrought specimens, which is probably attributed to finer α/β lamellar microstructures. Additionally, Cansizoglu et al. (2008) investigated EBAM Ti-6Al-4V bars using a three-point bending tester. The results of the flexure tests showed that the elastic properties of the structures are relatively consistent between builds.

2.4 Geometric Aspect

Even with impressive advantages over conventional manufacturing technologies, EBAM still exhibits several process challenges, such as dimensional accuracy and surface finish. Despite an intense interest in attainable accuracy and strengths by EBAM (Liu et al., 1998), few studies have emphasized the geometric aspects in EBAM. Cooke and Soon (2010) studied the geometric accuracy of metallic test part manufactured by EBAM and other powder-based processes. The model was a circle-diamond-square test part with an inverted cone that is used to evaluate the performance of five-axis milling machines. The authors reported that overall, the observed errors of EBAM parts are significantly larger than those of typical machined parts by at least an order of magnitude. The errors seem to be repeatable,
providing opportunities for compensation strategies. In addition, errors of parts may be due to the process; cyclic thermal effects, including deformations due to residual stresses, are most likely the cause.

In research conducted by Koike et al. (2011), an enlarged view of the gauge section of the EBAM Ti-6Al-4V specimen shows a rough exterior appearance compared to as-cast counterparts. The surface of the EBAM specimens are covered by rippled layers. On the other hand, the exterior surfaces of cast specimens are much smoother. Figure 2.6 below shows the surfaces of EBAM Ti-6Al-4V parts (tensile specimens), exhibiting the scanned pattern (Figure 2.6(b)) and surface morphology from a white-light interferometer (Figure 2.6(c)). The surface has a roughness about 20 µm of Rₐ. On the other hand, it has also been suggested that as-fabricated rippled surfaces of EBAM processed alloys may be beneficial in some biomedical applications where an irregular surface is desired.

Figure 2.6. Exterior surfaces of (a) EBAM Ti-6Al-4V samples showing: (b) the scan path and (c) surface morphology.
2.5 Other Materials Used in EBAM

In addition to widely used Ti alloys, EBAM has also been used with other metal-based materials including intermetallics, tool steels, superalloys and copper, etc.

Titanium aluminide, a TiAl-based intermetallic, has been used for propulsion exhaust system components and other aerospace applications. Murr et al. (2010) characterized the microstructures of TiAl powder and solid TiAl components fabricated by EBAM. The EBAM process results in a phase transformation: the powder was $\alpha_2$-phase-rich (HCP), while the EBAM components were largely $\gamma$-TiAl (FCC). The EBAM specimens exhibited an equiaxed $\gamma$-TiAl grain structure with a lamellar $\gamma/\alpha_2$ colony structure within the $\gamma$-grains. A relatively high dislocation density contributed to residual hardness, due in part to rapid cooling, associated with EBAM process. Heat treatment is generally required to adjust the microstructure and increase mechanical properties of EBAM TiAl parts. Sabbadini et al. (2010) studied the effects of HIP treatments. It was shown that HIP produces a tempered microstructure, with near $\gamma$ equiaxed morphology, resulted from an almost full recrystallization and little grain growth. Biamino et al. (2010) also investigated the mechanical properties of both as-fabricated EBAM TiAl specimens and after HIP. After HIP, YS appears to be fairly temperature independent up to 815 °C.

H13 steel is a popular and versatile, hot-work steel, providing good balance of toughness, thermal crack resistance, and high temperature strength needed for forming tooling. Cormier et al. (2004) studied the microstructure and properties of H13 steel produced by EBAM. The authors reported that EBAM H13 parts exhibit full interlayer bonding with virtually no porosity. The as-fabricated material was martensite having a hardness of 48-50 HRC. After annealing, HRC value was under 20. A small number of isolated shrinkage cracks confined within specific layers could be observed.
Co-based alloys are a group of bone implant materials. Murr et al. (2011) investigated the microstructure and mechanical properties of EBAM processed Co-29Cr-6Mo alloy. The EBAM fabricated solid, mesh, and foam Co alloy prototypes all exhibited a directional, columnar \( \text{Cr}_2\text{C}_6 \) precipitate architecture parallel to the EBAM build direction intermixed with some stacking faults in the FCC matrix. The columnar precipitates were spaced within textured, directional grains. Following HIP-annealing, the columnar precipitates dissolved and were replaced by a higher density of intrinsic stacking faults, resulting in hardness essentially constant, while there was a slight drop in YS, but UTS increased by 20%. Gaytan et al. (2010) also investigated the EBAM process on Co-Cr-Mo alloys. The tensile testing of specimens fabricated by EBAM produced average UTS of 1.45 GPa, YS of 0.51 GPa, and an elongation of 3.6 %, which are better than wrought or cast Co-Cr-Mo alloys.

Ramirez et al. (2011) investigated the effects of precipitate (\( \text{Cu}_2\text{O} \)) on the microstructure and mechanical properties of EBAM Cu parts. These precipitate-dislocation architectures create increased hardness, ranging from a base-plate hardness of HV 57 to an EBAM part hardness of HV 88, referenced to the precursor powder containing \( \text{Cu}_2\text{O} \) precipitates that has a hardness of HV 72.

Nickel-based superalloys present prominent high-temperature, corrosion and oxidation resistance applications including jet engine components. Murr et al. (2011) investigated Ni-based superalloys regarding to the effects of heat treatment on the mechanical properties of EBAM built parts. Tensile properties of EBAM cylinders at 538 °C didn’t significantly alter micro-hardness from as-fabricated cylinders tested at room temperature, but YS dropped from 0.33 to 0.30 GPa, and UTS decreased by 23 %. The corresponding elongation increased from 44 to 53 %. Similarly, the fabricated and HIP-ed cylinders tested at 538 °C also illustrated no significant change in micro-hardness, but both YS and UTS decreased marginally with little increase in ductility comparing to tests at room temperature.
By comparison, wrought Ni superalloy tested at 538 °C exhibited a YS of 280 MPa and a UTS of 830 MPa. Table 2.2 below briefly summarizes different materials that have been tested in EBAM, their corresponding applications and properties.

Table 2.2. Property and application of other alloys processed by EBAM

<table>
<thead>
<tr>
<th>Material</th>
<th>Application</th>
<th>Mechanical properties</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiAl</td>
<td>High temperature, Propulsion exhaust system</td>
<td>YS/UTS (700 °C): ~300 MPa, 360 MPa</td>
<td>(Biamino et al., 2010; Murr et al., 2010; Sabbadini et al., 2010)</td>
</tr>
<tr>
<td>H13 Steel</td>
<td>Tool, die/mold</td>
<td>As-processed hardness of 48-50 HRC</td>
<td>(Cormier et al., 2004)</td>
</tr>
<tr>
<td>Co-Cr based</td>
<td>High temperature bearings, biomedical implants</td>
<td>Micro-hardness: 4.4 to 5.9 GPa</td>
<td>(Murr et al., 2011; Gaytan et al., 2010)</td>
</tr>
<tr>
<td>Cu</td>
<td>Electrical</td>
<td>Micro hardness: 88 HV</td>
<td>(Ramirez et al., 2011)</td>
</tr>
<tr>
<td>Ni based alloys</td>
<td>Jet engine parts</td>
<td>YS/UTS (538 °C): 300 MPa, 610 MPa</td>
<td>(Murr et al., 2011)</td>
</tr>
</tbody>
</table>

2.6 Process Simulations

Despite the potential benefits of EBAM technologies and increasing studies/reports of this process, mostly applications, case studies, microstructures/properties, there has been little literature in process modeling/simulations of EBAM. EBAM is a rather complicated process and fundamental understanding of process physics is a key to improve the process performance and part quality consistency. Due to temperature dependent material properties and a moving heat source, an analytical solution of the thermal process model is considerably
demanding. Zäh and Lutzmann developed a simplified heat transfer model of EBAM to study the scan speed and beam power effects on the weld-pool geometry (Zäh and Lutzmann, 2010). Therefore, the mathematical-physical model is being transferred into a simulation software based on the finite-element (FE) method and thus, melt pool instabilities as a function of various input parameters can be predicted. Three different melt pool conditions are compared with each other. The melt pool shapes at the beam power (PB) of 150 W vary significantly with the applied scanning speed, $v$. Within the compared results, the parameter combination $v = 50$ mm/s and PB = 150 W yields the minimum length-to-width ratio of approximately 1.3. The authors were able to define a suitable process window, however, acknowledged a more comprehensive model is needed if new materials are to be utilized.

Shen and Chou recently initiated a study to investigate the thermal phenomenon of the EBAM process. A FE model incorporating Gaussian heat flux distribution, fusion latent heat, temperature-dependent thermal properties was developed to study the thermal response when subject to a moving source of heat of high intensity (Shen and Chou, 2012). Figure 2.7 below shows an example of temperature contours in the part during the process. The results are temperature contours of a half part modeled (because of symmetry) around the beam center at the quasi-steady state. High temperatures with large gradients around the maximum temperature area are noted. It is also noted because the powder porosity strongly affects the thermal conductivity, inclusion of powder as the top layer in the model is required for modeling accuracy. Further, since this is a moving heat source condition, the temperature spatial distributions can be translated to temporal responses to evaluate the corresponding cooling rates. The model, once validated, may be applied to systematically study various issues such as the process parameter effects and the raw material effects.
Moreover, process variable measurements such as temperatures have seldom been reported for EBAM despite the need to validate the simulation model and to monitor the process. Zäh and Lutzmann applied thermocouples, attached to the build plate, to evaluate temperature response during EBAM to evaluate the model developed by this research group (Zäh and Lutzmann, 2010). However, because of high temperatures and steep gradients, thermocouple techniques are limited in accuracy and resolutions. On the other hand, the authors of this paper have attempted to use a near-infrared thermal camera for process temperature measurements in EBAM. A preliminary result is shown in Figure 2.8 below, displaying examples of thermal images at particular frames collected during the EBAM experiment that built a 25.4 mm square block. Figure 2.8(a) was taken during the contour melting and the square boundary being heated can be observed. Figure 2.8(b) was during the hatch melting; at this particular time frame, the electron beam was moving from the left side toward the right. A localized high temperature zone (over 1600 °C) with a rapid temperature diminishing tail, similar to, qualitatively, the simulation result (Figure 2.7) can be noted.
2.7 Conclusions

EBAM, an enabling technology for design and manufacturing integration, can efficiently assist developments and modifications of products, especially for complex, difficult-to-fabricate components. However, the EBAM process physics is complex, and the part characteristics seem to be sensitive to the process parameters, the effects of which may not be well understood. There has been increased literature of EBAM, and this study intends to provide an overview of the EBAM technology, process principles, applications, part microstructures and mechanical properties, and process simulations. The collected and analyzed information from this review can be summarized as below.

(1) Though Ti-6Al-4V is the most widely used materials for EBAM, other materials such as intermetallics, tool steels, Co-Cr alloys and Ni-based superalloys, and Cu, etc., have
been attempted for various applications popularly in the aerospace and biomedical industries, some with unique geometry such as cellular structures.

(2) EBAM parts of different raw alloys have been characterized in microstructures and mechanical properties. In general, EBAM parts have fine microstructures with some porosity, which can be mostly eliminated by HIP. EBAM parts typically have comparable (to wrought counterparts) or superior mechanical properties: high YS and UTS and higher hardness, but a lower ductility.

(3) Dimensional errors of EBAM parts are significantly larger than those of typical machined parts by at least an order of magnitude, and yet, surface finish is poorer than cast parts.

(4) A thorough literature survey shows limited published studies of the EBAM process simulations. To effectively use the EBAM technology, it is necessary to model the process phenomenon and correlate part characteristics with process parameters and variables.
References


3.1 Phase Transformation of Ti-6Al-4V Alloy

3.1.1 Introduction of Ti and Ti Alloys

Ti and Ti alloys are widely used in many industries, especially in aerospace and automobile, due to their desirable and versatile combination of good mechanical and chemical properties (Sha and Malinov, 2009). The mechanical properties of Ti alloys are essentially dependent on the microstructure, which is determined by the thermo-mechanical processing and thermal treatment procedures (Boyer and Furrer, 2004). In order to understand the microstructure in Ti alloys, it is important to study the nature of phase transformations taking place at different thermal conditions.

Ti is stable only at certain temperature ranges. The stable phase at low temperature is $\alpha$, which has hexagonal close packed (HCP) structure. At a temperature of 882 °C, pure Ti undergoes a phase transformation from $\alpha$ to a body centered cubic (BCC) phase ($\beta$) that remains stable up to the melting point. The unit cells of $\alpha$ and $\beta$ phases could be seen from Figure 3.1. Depending on the predominant phase or phases in their microstructure, Ti alloys are categorized in four groups: $\alpha$, $\beta$, $\alpha+\beta$ alloys, and intermetallics (Wessel, 2004). Adding alloying elements to Ti provides a wide range of physical and mechanical properties of Ti alloys. Typically, elements of Al, C, N, and O are $\alpha$-stabilizers, while V, Mo, Nb, and Ta are $\beta$-stabilizers (Collins, 2004).
3.1.2 Ti-6Al-4V Alloy

Ti-6Al-4V alloy is the most widely used high-strength Ti alloy (Sha and Malinov, 2009; Lutjering, 2003; Safdar, 2012). The alloy presents excellent mechanical properties, like high fatigue strength even at high temperature capability, and good biocompatibility. It has wide usage in automotive, aerospace, and biomedical implants (Gaytan, 2009; Gong et al., 2012).

Ti-6Al-4V alloy has the alloy elements additions of both α stabilizer element (Al) and β stabilizer element (V). It falls in the category of α+β alloys. The binary-alloy phase diagram of Ti-V and Ti-Al could be seen from Figure 3.2 (Elmer, 2004). Figure 3.2(a) illustrates the influence of V on the α+β binary alloy equilibria, and the Figure 3.2(b) shows the influence of Al on the α+β binary alloy equilibria. The α+β Ti alloys contain a mixture of α and β phases, may combine the properties of strength of the α phase and ductility of β phase (Zhang, 2008).
Figure 3.2. Binary-alloy phase equilibrium calculated by thermocalc showing the influence of: (a) the V content on the $\alpha$ and $\beta$ phase equilibrium, and (b) the Al content on the $\alpha$ and $\beta$ phase equilibrium. The vertical lines indicate the nominal V and Al contents of the alloy (Elmer et al., 2004).
The weight percentages of Al and V are about 5.5-6.75% and 3.5-4.5%, respectively. It should be noted that, the chemical composition for the Ti-6Al-4V alloy used in EBAM (Arcam Ti-6Al-4V) shows some differences with other commercial usages, as can be seen from Table 3.1.

**Table 3.1. Chemical specification of Ti-6Al-4V alloy (available from www.arcam.com)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Required for Cast</th>
<th>Required for Wrought</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum, Al</td>
<td>6%</td>
<td>5.5-6.75%</td>
</tr>
<tr>
<td>Vanadium, V</td>
<td>4%</td>
<td>3.5-4.5%</td>
</tr>
<tr>
<td>Carbon, C</td>
<td>0.03%</td>
<td>&lt;0.1%</td>
</tr>
<tr>
<td>Iron, Fe</td>
<td>0.1%</td>
<td>&lt;0.3%</td>
</tr>
<tr>
<td>Oxygen, O</td>
<td>0.15%</td>
<td>&lt;0.2%</td>
</tr>
<tr>
<td>Nitrogen, N</td>
<td>0.01%</td>
<td>&lt;0.06%</td>
</tr>
<tr>
<td>Hydrogen, H</td>
<td>0.003%</td>
<td>&lt;0.015%</td>
</tr>
<tr>
<td>Titanium, Ti</td>
<td>Balance</td>
<td>Balance</td>
</tr>
</tbody>
</table>

*ASTM F1108 (cast material)  **ASTM F1472 (wrought material)*

The microstructures of Ti alloys are primarily described by the amount and arrangement of α and β phases. The microstructures at room temperature of Ti-6Al-4V consist mainly of HCP and BCC phases. The chemical compositions of α and β phases is concluded in Table 3.2 (Elmer et al., 2004). According to the phase compositions, the Al element is enriched in α phase and is the stabilizers for α, and so is the V element in β phase. In addition to α and β phases, another common phase is the martensitic phase (α'). It is either the HCP phase or the orthorhombic phase depending upon the composition of the β phase before quenching.

A vertical section of phase diagram for Ti alloys of Ti-6AL-xV (x is wt%) is presented in Figure 3.3 (Leyens, 2003; Safdar 2012). According to the phase diagram, the
phase will be pure β when the temperature is above 1000 °C. Some of the thermal and mechanical properties are listed in Table 3.3.

Table 3.2. Chemical composition of the α and β phases in the base metal as measured by microprobe analysis (Elmer et al., 2004)

<table>
<thead>
<tr>
<th></th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>β phase</td>
<td>2.92±0.11</td>
<td>15.43±0.86</td>
<td>1.32±0.11</td>
<td>80.7±0.70</td>
</tr>
<tr>
<td>α phase</td>
<td>6.73±0.33</td>
<td>1.42±0.73</td>
<td>0.04±0.02</td>
<td>91.2±0.47</td>
</tr>
</tbody>
</table>

Figure 3.3. Vertical section of phase diagram for Ti alloys of type Ti-6AL-xV (Leyens, 2003; Safdar, 2012)
Table 3.3. Some thermal properties and mechanical properties of Ti-6Al-4V (Lütjering and Williams, 2009; Elmer et al., 2004).

<table>
<thead>
<tr>
<th>Physical property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquidus temperature, $T_L$ (K)</td>
<td>1928</td>
</tr>
<tr>
<td>Solidus temperature, $T_S$ (K)</td>
<td>1878</td>
</tr>
<tr>
<td>Density of liquid metal, $\rho$ (kg/m³)</td>
<td>3890</td>
</tr>
<tr>
<td>Thermal expansion coefficient (K⁻¹)</td>
<td>$1.1 \times 10^{-5}$</td>
</tr>
<tr>
<td>Thermal conductivity of liquid, (W/m-K)</td>
<td>32.5</td>
</tr>
<tr>
<td>Thermal conductivity of solid, (W/m-K)</td>
<td>8.3-24.2</td>
</tr>
<tr>
<td>Specific heat of solid (J/kg-K)</td>
<td>725</td>
</tr>
<tr>
<td>Specific heat of liquid (J/kg-K)</td>
<td>872</td>
</tr>
<tr>
<td>Tensile strength</td>
<td>900-1200 MPa</td>
</tr>
<tr>
<td>Elastic modulus</td>
<td>115 GPa</td>
</tr>
</tbody>
</table>

3.1.3 Phases Transformation of Ti-6Al-4V Alloy

The β-transus defines the temperature above which the equilibrium microstructure will consist only for the β phase. The β-transus for Ti-6Al-4V alloy is approximately 1000 °C (Charles, 2008). At temperatures above β-transus, Ti-6Al-4V is composed of equiaxed β grains (Zhang, 2008). During cooling process, the primary phase β transforms to other phases. The cooling rate of the process is critical to determine the microstructure in the room temperature. Lütjering (2003) investigated that, for slow cooling rates from above β-transus temperature, the β phase transforms into globular type of α. In the case of moderate to high cooling rates the prior β phase transforms into α platelets growing from grain boundary of prior β grain to the inside of grain. The length and thickness of the platelets are determined by the cooling rate. Charles (Charles, 2008) investigated that the β phase forms Widmanstätten plate-like or basketweave acicular α for slower cooling rates from high
temperature. Widmanstätten α takes different morphologies depending on the cooling rate, spanning from aligned platelets in colonies to a “basketweave” type of structure. The basketweave structure is assumed to be a finer form of Widmanstätten morphology interpreted to be colonies of α-plates formed with specific orientations to each other.

While, in the case of rapid cooling with cooling rate above the 410 °C/s (Ahmed, 1998; Charles, 2008), the prior β phase transforms to the martensitic α’ phase. The transformed volume is usually plate shaped or disk shaped for most Ti alloys. Generally, the martensitic plates contain a high dislocation density and sometimes twins.

Kelly concluded the relationship between the cooling rate and the resulting microstructures. The β phase may transform into a variety of α morphologies as it cools through the β-transus in Ti-6Al-4V. The α morphologies include diffusion-controlled allotriomorphic and Widmanstätten alpha, massive (a_m), and martensitic (α’). Typical cooling rates for these transformation products have recently been studied and can be found in the Table 3.4 (Kelly, 2004). Typical phase transformations during a heating and cooling thermal cycle involving a peak temperature above the β-transus are schematically shown in Figure 3.4. The AB leg represents heating above the β-transus which leads to complete dissolution of α phase. The CD and CE legs represent decomposition of β upon rapid cooling to nonequilibrium products α’ and a_m. A diffusional transformation of β to a two phase structure consisting of primary-α (α_P) and β is shown by CF (Kelly 2004).

Table 3.4. Characteristics of the β to α + β Transformation (Kelly, 2004)

<table>
<thead>
<tr>
<th>Transformation product</th>
<th>Cooling rate (°C/s)</th>
<th>Start temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Allotriomorphic (a_GB)</td>
<td>CR &lt; 20</td>
<td>970-1000 °C</td>
</tr>
<tr>
<td>Widmanstätten (α)</td>
<td>CR &lt; 20</td>
<td>900-950 °C</td>
</tr>
<tr>
<td>Massive (a_m)</td>
<td>20&lt; CR &lt;410</td>
<td>970-1000 °C</td>
</tr>
<tr>
<td>Martensitic (α’)</td>
<td>CR &gt;410</td>
<td>M_S = 848 °C</td>
</tr>
</tbody>
</table>
3.2 Modeling of Microstructure Evolution

Currently, the rapid development of computer technology and a thorough understanding of the thermodynamics and kinetics of microstructure evolution have impelled the simulation of microstructure evolution. The emergence of simulation methods enables prediction on grain structure and morphological evolution. Among the models for microstructure evolution, Monte Carlo (MC) simulations, cellular automata (CA), and phase field (PF) are most widely used (Xiao et al., 2012).

3.2.1 MC Method

MC applies the atomistic simulation techniques to predict the behavior of materials. The simulations are usually performed on a collection of atoms or molecules. MC is an effective tool to model the microstructural evolution of complicated systems and has been widely used in the domain of physical metallurgy such as grain growth, recrystallization and phase transformation (Mordechai, 2011).
The MC technique is based on the minimization of the interface energy of a grain assembly. MC technique presents a tempting alternative since each calculation is concerned with a new configuration. Between each step, an atom is moved to a new position or, possibly, a pair of atoms are swapped. The total energy of this new configuration is then calculated and compared with the energy of the old configuration. On this basis, a decision is made as to whether the system should be updated to the new configuration or remain unaltered (Atkinson et al., 1999).

As summarized by Stefanescu (2008), the microstructure was first mapped on a discrete lattice. Each lattice site was assigned an integer (grain index), $I_j$, from 1 to some number. Lattice sites having the same $I_j$ belong to the same grain. The grain index indicates the local crystallographic orientation. A grain boundary segment lies between two sites of unlike orientation. The initial distribution of orientations is chosen at random and the system evolves to reduce the number of nearest neighbor pairs of unlike crystallographic orientation. This is equivalent to minimizing the interfacial energy. The grain boundary energy is specified by defining an interaction between nearest neighbor’s lattice sites. When a site changes its index from $I_j$ to that of its neighbor, $I_k$, the variation in energy can be calculated from the Hamiltonian describing the interaction between nearest neighbor lattice sites:

$$
\Delta G = -\gamma \sum (\delta_{i,j} - 1) \quad (3.1)
$$

where $\gamma$ is the interface energy, and $\delta_{i,j}$ is the Kronecker delta. The sum is taken over all nearest neighbors. Thus, unlike nearest pairs contribute $\gamma$ to the system energy, while like pairs contribute zero. The change of the site index is then decided on the basis of the transition probability, which is:

$$
W = \begin{cases} 
\exp(-\Delta G / k_B \cdot T) & \Delta G > 0 \\
1 & \Delta G \leq 0 
\end{cases} \quad (3.2)
$$
where $k_B$ is the Boltzmann constant. A change of the site index corresponds to grain boundary migration. Therefore, a segment of grain boundary moves with a velocity given by:

$$V = C(1 - \exp(-\frac{\Delta G_i}{k_B T}))$$

(3.3)

where $C$ is a boundary mobility reflecting the symmetry of the mapped lattice, and $\Delta G_i$ is the local chemical potential difference.

### 3.2.2 CA Method

Another method used to simulate the grain growth is CA. The CA technique becomes a feasible approach for the modeling of microstructural evolution. It can handle complex microstructure evolutions in two and three dimensions and saves the computation time. The CA method was first proposed by von Neumann and Burks (1966). Since then, it has had numerous diverse applications, including microstructure evolution during solidification (Yin, 2010).

The CA model was first developed by Rappaz and Gandin in 1993 (Rappaz and Gandin, 1993) for meso-scale modeling of grain growth during solidification. The model includes the physics of nucleation, growth kinetics, and crystallographic orientation. The mechanism of competitive dendrite growth is directly embedded in the CA algorithm. The model can perform the time-dependent simulation of typical equiaxed structure, columnar grain structure, and columnar-to-equiaxed transition. Nastac (1999) developed the equations and rules for the CA model for simulating the evolution of dendritic crystals during the solidification of binary alloys. The model includes time-dependent calculations for temperature distribution, solute redistribution in the liquid and solid phases, curvature, and growth anisotropy without further assumptions on the nucleation and growth of dendritic crystals.
For the solidification model, each CA cell has three possible phase types, the liquid, solid, and interface cell. The state of a cell at the next time-step is dependent on its current state and the current states of its immediate neighbors. In the CA models for solidification, the solid fraction change of each cell at the solid/liquid interface is determined based on the temperature and composition of its neighbors. Each solid and liquid cell has one solute concentration $c_S$ or $c_L$, respectively, while the interface cells have both a solid and a liquid solute concentration. A sample two dimensional cell configuration is shown in Figure 3.5 (Choudhury et al., 2012).

At the beginning, an initial state is selected by assigning a state for each cell. Then, a new generation is created according to the transition rule that determines the new state of each cell in terms of the current state of the cell and the states of the cells in its neighborhood.

Figure 3.5. Example 2D CA cell configuration (Choudhury et al., 2012).

The CA method can treat arbitrary grain shapes, and it is also convenient to describe the grain competition growth, morphology transition, and the merging between two arms. In addition, the important feature of the method is that all cells are considered at the same time to define the state of the system in the following time step. Thus the computational time step can be directly related to the physical time step.
3.2.3 PF Method

The phase field method has recently emerged as a powerful computational approach to model and predict mesoscale morphological and microstructure evolution in materials (Chen, 2002). It has been widely used to simulate the microstructure evolution both in liquid and solid. The PF method was firstly developed by Langer (Langer, 1978), and it simulates the microstructure by solving the equations governing the evolution of the PF variable and heat or solute. The Ginzburg-Landau theory of phase transitions shows a wide range of applications. The basis of the theory is the functional of the local free energy density depending on the order parameter of the system and its spatial derivatives (Steinbach et al., 1996).

It describes a microstructure using a set of conserved and nonconserved field variables that are continuous across the interfacial regions. The temporal and spatial evolution of the field variables is governed by the Cahn-Hilliard nonlinear diffusion equation and the Allen-Cahn relaxation equation. With the fundamental thermodynamic and kinetic information as the input, the phase field method is able to predict the evolution of arbitrary morphologies and complex microstructures without explicitly tracking the positions of interfaces (Chen, 2002).

The PF method produces results similar to those of the CA method by obtaining the temperature and solute fields and then determining the solid/liquid (S/L) interface. The method employs a phase field variable, e.g., $\Phi$, which is a function of position and time, to describe whether the material is liquid or solid. The behavior of this variable is governed by an equation that is coupled to equations for heat and solute transport. If the point lies in the liquid region, $\phi=0$; if the point lies in the solid region, $\phi=1$. Values of $\Phi$ between 0 and 1 represent points that lie in the interface. Interfaces between liquid and solid are described by smooth but highly localized changes of this variable between fixed values that represent solid
and liquid (Boettinger et al., 2002). The phase variable can be obtained by solving the kinetics equation:

\[
\frac{\partial \phi}{\partial t} = -\Gamma \cdot \frac{\delta F}{\delta \phi}
\]  

(3.4)

where the \( \Gamma \) is interface kinetic coefficient. The free energy function \( F \) is:

\[
F = \int_{V} \left[ f(\phi) + \frac{1}{2} (\varepsilon_\phi)^2 \cdot (\nabla \phi)^2 \right] dV
\]  

(3.5)

where \( f(\phi) \) is free energy density and \( \varepsilon_\phi \) is gradient energy coefficient.

The PF method simulates the phase types by solving differential equations that govern the evolution of the PF variable. It has been applied to simulate the microstructural evolution of pure metals and multi-component systems. The main advantage of this approach is that PF is easy to be implemented, capable of reproducing most of the phenomena associated with microstructure formation. The disadvantage of this method is that the PF method requires significant computer resources, and the computation time is about two orders of magnitude slower than CA.

### 3.3 Conclusions

Ti-6Al-4V is a typical \( \alpha + \beta \) Ti alloy. The morphology and phases are determined by the thermal history. The microstructure at the room temperature is the combination of \( \alpha \) and \( \beta \) phases, while the martensitic \( \alpha' \) could be formed during rapid cooling process. Since the solidification rate is high in EBAM, the fine \( \alpha \) phase or \( \alpha' \) is expected to be achieved in the microstructure.

The microstructure models could be effective to model the microstructure evolution. CA and PF are both commonly applied to model the morphologies of the alloys. The PF models could achieve the high accuracy of the interface and morphologies. In addition, the quantitative results from PF calculations are therefore usually found to be superior to those achieved by CA. However, the computation of PF is expensive and slower than CA.
References


CHAPTER 4
CHARACTERIZATION OF SINTERED Ti-6Al-4V POWDER IN EBAM

4.1 Introduction

EBAM, using an electron source to melt and fuse powder, is a relatively new AM process, which can produce full-density metallic parts directly from the electronic data of a part design (Murr et al., 2009a). Despite of a short history of the EBAM technology, it has attracted ever increasing interest from aerospace, military and biomedical industries because of several unique advantages such as unique geometries/structures, rapid scan speeds, and a moderate operation cost, etc.

The EBAM process involves powder spreading, preheating, as well as contour melting and hatch melting. The preheating serves to lightly sinter the precursor powder layer by using an electron beam at a low power, but a rather high speed. Preheating-induced sintering can hold metal powder in place during subsequent melting and reduce the thermal gradient between the melted layer and the rest of the build part (Cormier et al., 2004; Rodriguez et al., 2012). Syam et al. (2012) reported that preheating helps the subsequent build process to maintain a high power density and a very rapid scanning speed. Sintered powder surrounding the part also helps support downward facing surfaces during the building process. Yang pointed out that preheating results in a reduced amount of energy needed in melting, and therefore, improves the distribution of the heat (Yang, 2011). Kahnert et al. (2007) claimed that preheating of powder in the EBAM has an important influence to the avoidance of the spreading effect, and preheating affects the results of the subsequent melting and solidification steps.
In spite of advantages and potential benefits of EBAM, there exist several challenges for effective usage and widespread applications. A common process deficiency of EBAM is the delamination of build layers, as can be seen in Figure 4.1. When the residual stresses exceed the binding abilities between the adjacent layers, layer delaminations occur (Zäh and Lutzmann, 2010). In order to eliminate EBAM part defects, the reduction of process-induced residual stresses is necessary. Although preheating in EBAM is critical to subsequent melting processes, there has been little systematic research concerning about powder characteristics due to preheating. In addition, the mechanism of layer-building process affected by preheating may not be clearly understood. It is known that the thermal properties of metallic powder are significantly different from those of the corresponding solid bulk material, and yet, temperature dependent (Lin et al., 1985; Sih and Barlow, 2004; Neira Arce, 2012). In addition, the EBAM process operates at a high vacuum, and thus, the thermal conductivity of powder will not be the same as in a gas surrounding (Roberts et al., 2004). Tolochko et al. (2003) investigated the mechanisms of selective laser sintering of Ti powder and heat transfer in vacuum conditions. It was reported that accurate powder properties are required to better model and simulate the thermal process.

The objective of this study is to better understand the Ti-6Al-4V powder characterization in EBAM, in a loose or sintered form. The morphology, porosity, size distribution and thermal properties of Ti-6Al-4V powder in EBAM were studied experimentally. The intent is to quantify metal powder characteristics resulted from preheating in the EBAM process.
4.2 Experiment

Pre-alloyed Ti-6Al-4V powder was used as feedstock material. The chemical composition are listed in Table 4.1. The experimental specimens were fabricated, per designed part models, using an EBAM system (S12 from Arcam) at Marshall Space Flight Center. The scan speed in preheat was on the order of 10 m/s with a beam current on the order of 30 mA. The target preheat temperature for Ti-6Al-4V powder is around 730 °C and the process lasts for about 10 s. Two kinds of samples with sintered powder particles were prepared for microstructural characterization: powder-bed samples and powder-enclosed samples, as shown in Figure 4.2. Both Z-plane (scanning surface) and X-plane (side surface) were investigated, Figure 4.2(a). Moreover, the microstructure of the solid Ti-6Al-4V part from the EBAM process (Figure 4.2(b)) was also examined with both top and bottom
portions analyzed. Samples were prepared for microstructural observations with standard metallographic procedures including sectioning, mounting, grinding with SiC papers up to the grit size of 1000, and then polished using diamond suspension down to 0.5 µm. Kroll’s reagent (92 ml Distilled water, 6 ml nitric acid, and 2 ml hydrofluoric acid) was applied for etching (Al-Bermani et al., 2010). The samples were immersed in the solution for about 30 s and immediately rinsed with water, then air dried. The metallographic samples were examined using a Leitz optical microscope (OM) and a Philips XL-30 scanning electron microscope (SEM). In addition, to examine particle sizes and distributions and porosity from preheating, specimens from a hollow model (about 4.8 mm in length/width and 6.6 mm in height), designed from CAD software, were further fabricated using the same EBAM system with Ti-6Al-4V powder and default process parameters (70 µm layer thickness). The hollow specimens, with sintered powder enclosed, were scanned using a micro-CT (µCT) system, SkyScan 1172 from Microphotonics, by conducting scans with the following parameters: image pixel size of 2 µm, source voltage of 100 kV. The image analysis using µCT images was conducted to determine the porosity of the sintered powder, measured by ImageJ software (NIH). Moreover, the particle size distributions were measured by quantitative image analyses using the Image-Pro Plus software.

In addition, an approach has been attempted to investigate the thermal properties of sintered powder, as in the powder-bed condition, using a thermal analyzer. Specimens of a hollow model (40 mm by 40 mm by 13 mm with 0.8 mm shell thickness) were fabricated using the same EBAM system with Ti-6Al-4V powder, with 13 mm dimension along the Z direction (build direction). A solid piece of the same overall dimensions was fabricated as well to evaluate the thermal conductivity of solid Ti-6Al-4V. The hollow and solid specimens were measured with thermal properties at different temperatures (up to 750 °C), using a T2500S thermal analyzer from Thermtest Inc.
Table 4.1. Chemical analysis for Ti-6Al-4V powder (Arcam).

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>V</th>
<th>C</th>
<th>Fe</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arcam (wt. %)</td>
<td>6</td>
<td>4</td>
<td>0.03</td>
<td>0.1</td>
<td>0.15</td>
<td>0.01</td>
<td>0.003</td>
<td>Bal.</td>
</tr>
<tr>
<td>Standard (wt. %)</td>
<td>5.5-6.75</td>
<td>3.5-4.5</td>
<td>&lt;0.1</td>
<td>&lt;0.3</td>
<td>&lt;0.2</td>
<td>&lt;0.05</td>
<td>&lt;0.01</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Figure 4.2. Illustration of the samples after preheating: (a) powder-bed sample, (b) powder-enclosed sample.

4.3 Results and Discussions

In general, fine raw powder is, order of 10 to 100 µm, used in the EBAM. Figure 4.3 presents the SEM images of Ti-6Al-4V loose powder; Figure 4.3(a) shows different particle sizes from backscatter electron (BS) image and Figure 4.3(b) shows a few particles from secondary electron (SE) image. Very small-sized particles, also called satellite powder, are found attached to some larger particles. During the EBAM process, a layer of metal powder is formed by a rake uniformly distribute powder supplied by hoppers, followed by preheating for powder sintering, prior to each layer build. The typical layer thickness in EBAM is in the range of 0.05 to 0.2 mm.

After preheating, a certain extent of inter-particle cohesion or aggregation is expected to be formed. Figures 4.4 and 4.5 show SEM images of the aggregated powder from both the Z and X-planes. The neck formations are clearly evident in both planes. The sample shows “elongated” powder which are caused by the connected powder with necks or partial partially melting. He et al. (2011) found that the satellite particles in interspace are connected to large
spherical particles, which forms a skeleton to bond the large particles together. Also, they stated that the connection mode is frangible, but tough enough to hold powder and resist the impact from the electron beam during the EBAM process. The diameter of the necks is on the order of 1 to 10 µm. Tolochko et al. (2003) found that the sintering kinetics affects heat transfer in the powder bed through the dependence of thermal conductivity on the neck size. It can be expected that the size of the necks may also affect the subsequent melting.

Figure 4.3. SEM images of Ti-6Al-4V powder particles: (a) BS image of powder population, and (b) SE image of a few particles.
Figure 4.4. SEM images of sintered particles on Z-plane: (a) low magnification, and (b) high magnification.
Figure 4.5. SEM images of sintered particles on X-plane: (a) low magnification, and (b) high magnification.

Figure 4.6 presents morphologies of the preheated powder of powder-bed sample after fine polishing. The necks (circled area) can be noted on samples from both X and Z-planes. Some powder particles are connected with surrounding powder and the chain-like structure is formed due to the preheating. Generally, it is observed that more neck formations are presented on the Z-plane. It aligns with the phenomenon that the electron beam power density will quickly diminish along the depth from the build surface. He et al. also found that the preheating only heats the upper thin layer powder and brings them to bond with formed
aggregation (He et al., 2011). Figure 4.7 shows the morphologies of preheated powder from the powder-enclosed sample. The interface of the powder-solid (top section of the sample) is also presented. The neck formations can be observed from both the central and top sections. It is also interesting to note that powder particles are connected to the undulated boundary of the down-facing surface of the solid part.

Figure 4.6. OM images of preheated powder from powder-bed sample after polishing: (a) Z-plane and (b) X-plane.
The prepared metallographic samples were also etched to reveal their microstructures. In general, the microstructure of Ti-6Al-4V powder (loose or sintered) is common Widmanstätten (α+β), as can be seen in Figure 4.8. Ti-6Al-4V is an α-β alloy and its microstructure is strongly dependent on the process thermal history. The Widmanstätten morphology is a typical feature for Ti-6Al-4V alloy (Fan et al., 2011). During solidification, the primary phase is body-centered cubic β. When the temperature is below β transus temperature, the solid phase transformation, into the hexagonal closed-packed α phase, begins (Eschey et al., 2009). In the metallographic images, the light phase is α phase and the
dark phase is $\beta$ under OM, while the contrast under SEM is the opposite. For the Ti-6Al-4V powder, the primary phase is the bulk $\alpha$ phase with a small amount of $\beta$ in the $\alpha$-lath boundaries. Figures 4.8(b) and 4.8(c) show that intergranular structure is also present, which is consistent with the microstructure observed from OM images.

Qi and Yang (2010) studied preheating in EBAM and reported that preheating may result in the increase of critical scanning speed needed from the sintering neck theory (Tolochko et al., 2003), which is shown in Figure 4.9. In a single powder, there will always exist some surfaces with higher surface energy, which will be more easily melted than other portion by electron beam preheating. If the preheating scheme can ensure partial melting of the powder, the sintering necks between neighboring particles will be formed (Qi and Yang, 2010). Although the effects of preheating parameters have not investigated yet, a longer
preheating time is expected to produce more neck formations in the sample. Qi and Yang (2010) investigated the effects of the preheating time on 316L stainless steel from 60 s to 120 s. The authors observed that sintering necks become visible and then necks start to form continuous networks, and eventually many powder particles connect and form a mass clump.

On the other hand, some researchers reported different results about preheating. Regarding to the spreading effect of the EBAM process, Eschey et al. (2009) found that a powder layer is able to be exposed to the electron beam at room temperature without spreading when a beam spot diameter of 0.4 mm or less is applied. The preheating can be neglected and a considerable saving of time can be achieved. Furthermore, efforts in terms of the removal of unmelted powder can be significantly reduced as the formation of sintering bonds is eliminated. Although these results are different from other reports described earlier, it is only specific for some designed process parameters.

In order to observe and quantify the sintered powder resulted from preheating, micro-CT scanning was applied, which is a nondestructive inspection technique to characterize internal features of a large volume. The CT scan image example from a horizontal section (Z-plane) is shown in Figure 4.10. ImageJ was used to determine the porosity of the sintered powder in EBAM. The powder portion image was then cropped from Figure 4.10(a) and applied with a threshold value to distinguish between porosity and powder material (Figure

Figure 4.9. The sintering neck between two powder particles (Qi and Yang, 2010).
4.10(b)). The porosity of powder was calculated based on the area ratio between the porosity portion and the whole area in Figure 4.10(b). The results show that the powder porosity was about 52.0% for the Z-plane section. In addition, different locations (top, middle and bottom) of the Z-planes sections were also analyzed, showing very similar results, less than 3% difference. The CT scan images for Z-plane and X-plane sections are noted to be comparable, as shown in Figure 4.11. The particle morphologies of both sections look similar. The analyzed porosity of the X-plane section is 50.6%, which is close to the porosity of the Z-plane section. It can be inferred that the enclosed powder particles are distributed homogeneously. It has been argued earlier that the thermal properties of Ti-6Al-4V are porosity dependent. The results from micro-CT image analysis of preheated powder specimens imply that the powder from preheating may significantly affect the thermal properties of the powder bed in EBAM (Neira Arce, 2012).

Figure 4.10. Images of sintered powder from Z-plane section: (a) CT-scan image, (b) threshold binary image of enclosed powder.
In order to achieve quantitative analysis of the powder particle size and size distribution, Image Pro Plus was applied to study the powder distribution in both the Z-plane and X-plane sections. The minimum diameter was set as 10 μm in order to exclude the attached tiny particles (Murr et al., 2009b) and noise. The distribution of the particles was obtained as shown in Figure 4.12. For the powder diameter in the range of 20 to 90 μm, the particles show a normal distribution. It can also be noted that the major range of powder diameters is around 30 to 50 μm, while the mean overall diameters of the powder particles is 36.6 μm for the Z-plane section and 34.1 μm for the X-plane section. The similar mean particle diameters between both planes indicate a homogeneous distribution of powder particles. Murr et al. (2009) also studied the distribution of the powder diameters (Ti-6Al-4V
for EBAM) and reported the ranged from 1 to 100 μm. The powder diameter shows a bimodal distribution; the mean overall powder diameter is about 30 μm, while the mean diameter of large powder is about 60 μm.

Figure 4.12. Histograms of powder particles: (a) Z-plane section and (b) X-plane section.

The hollow and solid specimens were measured with thermal conductivity at different temperature (up to 750 °C), using a T2500S thermal analyzer by Thermtest at Micro Photonics Inc. The measured thermal conductivity of preheated Ti-6Al-4V powder and solid Ti-6Al-4V are summarized in Gong et al. (2013). Ti-6Al-4V powder has significantly lower thermal conductivity than that of a solid counterpart. Also, the thermal conductivity is highly temperature dependent. For both solid piece and powder-enclosed specimen, with increased temperatures, thermal conductivity values increase (Gong et al., 2014).
4.4 Conclusions

In this study, preheated Ti-6Al-4V powder from the EBAM process has been characterized. Metallographic techniques were applied to observe and characterize sintered powder by both OM and SEM. The porosity of the preheated powder was evaluated using acquired images and analyses from micro-CT scanning of the specimens containing preheated powder. The powder particle sizes and size distributions were also analyzed. In addition, the thermal conductivity of preheated Ti-6Al-4V powder has been measured and analyzed. Following conclusions can be made from this study.

(1) Preheating results in metallurgical bonds or even partial melting of the powder during the EBAM process. The phenomenon of neck formations is evident in both the Z-plane and X-plane sections. The diameter of the necks is on the order of 1 to 10 μm. In addition, the X-plane seems to have less particles of a sintering result because of the energy penetration limit.

(2) The micro-CT scan of preheated powder shows a similar porosity level between the Z-plane and X-plane surfaces. The calculated porosity of the preheated powder is about 50%. Moreover, the major diameter range of the powder is from 30 to 50 μm on both planes.

(3) The microstructure of a Ti-6Al-4V preheated powder exhibits Widmanstätten (α+β) structure.
References


CHAPTER 5
BEAM SCANNING SPEED EFFECTS IN EBAM

5.1 Introduction

The powder-bed EBAM using an electron beam to melt and fuse powder, is one of a few AM technologies for direct digital manufacturing of metal products (Murr et al., 2009a). Because of several unique advantages such as high energy efficiency, rapid scan speed, and moderate operation cost, etc., EBAM has attracted ever increasing interest from aerospace, military and biomedical industries, etc.

Research subjects in EBAM have spread across a wide spectrum ranging from material and microstructural characterization (Murr et al., 2009a; Gong and Chou, 2013a), process modeling and simulation (Zäh and Lutzmann, 2010; Gong et al., 2013b), process metrology (Price et al., 2012), and geometric attributes (Koike et al., 2011), etc. Even with many advantages over conventional manufacturing technologies, EBAM still exhibits several process challenges such as part accuracy and property scattering. Cooke and Soons (2010) studied the geometric accuracy of EBAM parts. It is observed that the errors of EBAM parts are significantly larger than those of typical machined parts.

Ti-6Al-4V is one of the most commonly used alloys in EBAM. Microstructures of Ti-6Al-4V samples from EBAM contain a mixture of phases such as α (HCP), β (BCC) and α’ martensite (HCP). The columnar prior β structure formed during initial solidification has been observed, which is a result of very high temperature gradients, along the build direction (Al-Bermani et al., 2010; Safdar et al., 2012a). Antonysamy et al. (2013) studied the prior β grain texture of EBAM components and reported that the columnar structure shows strong fiber texture of \(<001>\) β, which is normal to the deposited powder layers. Safdar et al.
(2012a) reported typical Widmanstätten ($\alpha+\beta$) inside of the prior $\beta$ grains. Facchini et al. (2009) investigated microstructures and showed that the main constituent is $\alpha$ with only a small fraction of $\beta$. Christensen et al. (2007) compared the EBAM microstructures to counterparts from cast specimens. For the cast specimen, a coarse acicular ($\alpha+\beta$) and thick prior $\beta$ grain boundaries can be observed; while the microstructure of the EBAM specimen consisted of fine acicular ($\alpha+\beta$) and thin prior $\beta$ grain boundaries. The thickness of the $\alpha$-lath is around 1.4-2.1 $\mu$m for different EBAM samples (Murr et al., 2009a). Due to high cooling rates during the solidification, $\alpha'$-martensitic platelets exist in EBAM parts, which may contribute to increased strength and hardness but lower ductility (Murr et al., 2010a; Gong et al., 2012). In summary, Ti-6Al-4V samples from EBAM show a fine Widmanstätten ($\alpha+\beta$) microstructure combined with $\alpha'$, which can be expected by the thermal characteristics of the EBAM process: small melt pool and rapid cooling. However, the microstructures of EBAM Ti-6Al-4V vary with the changes of process conditions.

It has been reported that EBAM process parameters may have significant effects on the part quality (Murr et al., 2009b; Bontha et al., 2009). Murr et al. (2009b) studied that variations in melt scan, beam current, and scan speed affect the EBAM built defects such as porosity, which may be used for microstructure-property variations in the final product (Murr et al., 2009c). The electron beam scanning speed is one of critical parameters to the EBAM process affecting the process condition (Zäh and Lutzmann, 2010). Jamshidinia et al. (2013) applied an FEM model to study the heat distribution in EBAM using Ti-6Al-4V powder. It was reported that the scanning speed of 1000 mm/s has a cooling rate of more than 135 and 11 times faster than those of the 100 mm/s and 500 mm/s, respectively. In a research conducted by Bontha et al. (2009), thermal process maps are developed to predict solidification microstructures in the wire-feed electron beam freeform fabrication process. It was suggested that increasing of the scanning speed may result in a predominant decrease in
grain size of Ti-6Al-4V build parts. Despite of research on modeling and experiments of EBAM, there has been very little systematic study on the relationship between the scanning speed and the microstructure of the EBAM parts.

The objective of this experimental study is to better understand the microstructural variations, in terms of phases and characteristic length, affected by the scanning speed in EBAM built Ti-6Al-4V. A commercial EBAM system was used to fabricate specimens with different speed functions (that are related to beam speed) during the same build cycles. Moreover, a thermal imager was applied to acquire process temperatures during the build. The intent is to correlate part microstructures with process thermal characteristics.

5.2 Experimental Procedure

An Arcam S12 EBAM machine, at NASA’s Marshall Space Flight Center (Huntsville, AL) was used to fabricate samples, which were simple blocks (60 mm long, 5.5 mm wide and 25 mm high) modeled from CAD software. Fine pre-alloyed Ti-6Al-4V powder, with a diameter between 45 and 100 µm, was used as feedstock material. The layer thickness was 70 µm. The EBAM process involves powder spreading, pre-heating, as well as contour and hatch melting. During the melting stages, an electron beam move across the powder-layer surface tracing the model cross-section boundary and then raster-scan through the inside of the contour.

The electron beam scanning in an EBAM system is controlled by magnetic deflection coils, which enables high scanning speeds, e.g., 15 m/s. For Arcam EBAM systems, the speed function (SF) is a process parameter setting related to the actual beam scanning speed (Mahale, 2009). In this study, four different SFs were tested (20, 36, 50 and 65) to investigate the beam speed effects on build part microstructures. Figure 5.1 shows the EBAM fabricated samples processed with the corresponding speed function.
Figure 5.1. EBAM parts built with different speed functions.

A LumaSense MCS640 near-infrared (NIR) camera was used for temperature measurements during EBAM builds. The NIR camera has a spectral range of 0.78-1.08 µm and a 640 × 480 pixel uncooled focal plane array sensor. The image capturing system has a maximum frame rate of 60 Hz and a detectable temperature range of 735-2446 °C. The NIR camera was mounted on a tripod and positioned to look downward through the EBAM machine’s viewport onto the build platform. The emissivity of Ti-6Al-4V at high temperatures and liquid state is not unknown, so was assumed as 0.35 in the camera setting. The details of the camera setup and settings with the EBAM system can be found in Price et al. (2012). For the current study, the acquired thermal images were analyzed to obtain average temperature profiles around the beam location and to estimate melt pool sizes, which were determined by the liquidus temperature of Ti-6Al-4V (1655 °C), for each speed function case at the same build height of 24.43 mm. Moreover, the thermal videos acquired were used to calculate the actual beam speed values during melt scans, by estimating the molten pool movement between consecutive frames. The scanning speed decreased with the increased build height, however, it became stable when it achieves a certain build height.
Fabricated Ti-6AL-4V samples were prepared for microstructural observations with standard metallographic procedures including sectioning, mounting, grinding with SiC papers up to the grit size of 1000, and then polished using diamond suspension down to 0.5 µm. Specimens close to the top surface of the build parts were used for analysis. Specimens of different cross-sections (scanning surface: Z-plane, build side surface: X-plane) were prepared to examine the anisotropic conditions in microstructures. To reveal the microstructures, polished specimens were then etched with a hydrofluoric acid-based solution: 1 ml hydrofluoric acid (50 wt. %) and 3 ml (60 wt. %) nitric acid in 7 ml distilled water (Murr et al., 2009b). The etched metallographic samples were examined using a Leitz optical microscope (OM) and a Philips XL-30 scanning electron microscope (SEM). In order to quantify the size of columnar β, equiaxed β and α-lath, a measurement method, from Wang et al. (2009), was applied. For each size measurement, three corresponding images were used for a statistical study.

5.3 Results and Discussions

5.3.1 Typical Microstructures

Figure 5.2 shows typical microstructures from the X-plane of an EBAM sample, SF 36. It is obvious that the prior β grains grew along the build direction and across multiple layers in the sample. The solidification of Ti-6Al-4V alloy involves two steps: liquid to primary solid phase of β and solid phase transformation (β to α or α′) depending on the cooling rate. The nucleation and growth of columnar grains of prior β take place during the initial rapid solidification when the temperature is above the β-transus temperature (about 980 °C). The columnar prior β grains are typical in high-energy materials processing, and upon rapid cooling from the melt, the growing grains align themselves with the steepest temperature gradients (Kelly, 2004) and result in a columnar shaped morphology (Wu et al., 2004). According to Antonysamy et al. (2013), in EBAM, the nucleation of β grains occurred
heterogeneously from the boundary layers at the build plate or the part surfaces. The authors also studied the prior β-grain texture of EBAM parts and the columnar structure shows strong fiber texture of <001> β along the build direction, which could be attributed to the elongated shape of the moving melt pool.

Another feature in typical EBAM Ti-6Al-4V microstructures is the martensitic phase, α’, which appears as plates, as can be observed in Figure 5.2. The α’ is transformed from the β phase due to a very high cooling rate. According to Ahmed and Rack (1998), for Ti-6Al-4V, a cooling rate of more than 410 °C/s, from the single β to the (α+β) region, will induce α’ formations. In addition, to trigger the formation of martensite, the temperature must be lower than the martensite starting temperature ($M_S$) of 575 °C for Ti-6Al-4V (Kelly, 2004). Other researchers, however, reported the $M_S$ as 650 °C and the martensite finishing temperature as 400 °C (Elmer et al., 2004). The α’ phase is commonly observed in Ti-6Al-4V alloy subject to rapid solidifications such as selective laser melting (Baufeld et al., 2011) and electron beam welding (Lu et al., 2012).

The width of the columnar prior β grains was measured in this study. Figure 5.2(a) illustrates an example of the width measurements. The image is overlaid with straight lines which are normal to the boundaries of the columnar structures and the intersections of the lines with grain boundaries were examined and used for width estimates. Different images from different specimen areas were measured to obtain statistical data. The average width of the columnar structure is about 41.6 µm for this case (SF 36). This result is smaller than the columnar width reported by Al-Bermani et al. (2010), 75 to 150 µm.
Figures 5.3 and 5.4 illustrate typical microstructures of the Z-plane specimen, also from the SF 36 sample. Different from the microstructure from the X-plane (Figure 5.3), equiaxed grains are noted on Z-plane. It can, thus, be concluded that the prior β grains are of a rod shape. Similar equiaxed grains at the Z-plane have also been reported by other researchers for Ti-6Al-4V alloy (Al-Bermani et al., 2010) and other alloys (Amato et al.,
Figures 5.3(b) and 6.4 show higher magnification images of the Z-plane specimen from OM and SEM, respectively; both α and β phases can be identified. Upon cooling from the β-transus temperature, the initial α that nucleates is “grain boundary” α (α_{GB}) because of its location on a β boundary. Eventually, the β boundaries will be replaced by α_{GB} in a continuous fashion (Kelly, 2004), as can be noted from Figure 5.4(a). In addition, fine Widmanstätten (α+β) structures are shown inside of equiaxed grains, indicating a rapid cooling rate in EBAM. Widmanstätten (α+β) is a typical microstructure of Ti-6Al-4V alloy produced by EBAM, as can be clearly identified from high magnification images, Figures 5.3(b) and 5.4(b). In solid phase transformation, the prior β columnar grains are transformed into fine α laths. The (α+β) structure is formed by diffusion-controlled solid phase transformation, in which V diffuses into β while Al diffuses into α (Safdar et al., 2012a). Further, the classical α-lath structure is surrounded by a very small amount of β in α boundaries. The result is similar to studies from Safdar et al. (2012a) and Murr et al. (2010a). Compared with the wrought or cast Ti-6Al-4V, which shows coarse α laths or equiaxed α/β (Murr et al., 2009a), EBAM Ti-6Al-4V parts show a finer α. Al-Bermani et al. (2010) reported that fine α laths have no preferred texture which is different from strong texturing of prior β grains.

Similar to the measurement method for the columnar width, intersections of the lines with prior β grain boundaries were marked and used for grain size estimates. The microstructural image was overlaid with several random lines, as shown in Figure 5.3(a). From the measurements, the estimated size of the equiaxed grains is 50.1 µm in this case. The width of α laths was also quantitatively analyzed using SEM images; an example is shown in Figure 5.4(b). The average thickness of α laths is 1.1 µm for this particular sample, SF 36.
Figure 5.3. Microstructure of Z-plane specimen from an EBAM sample (SF 36): (a) low magnification showing grain size analysis, and (b) high magnification image.
Figure 5.4. Scanning electron micrographs from Z-plane of an EBAM sample: (a) 1000 × image, and (b) 5000 × image showing α-lath analysis.

5.3.2 Beam Speed Effects

The estimated beam speed values using the method described above were 214 mm/s, 376 mm/s, 529 mm/s and 689 mm/s for SF 20, 36, 50 and 65, respectively. It needs to point out that the beam speed actually changed with the build height, in a reverse relation; however, reached approximately an asymptote after 15 to 20 mm build height. The reported
speed values above were at the build height of 24.43 mm, close to the end of the build. The acquired thermal images (temperature contours) were processed, using MATLAB software, to obtain temperature profiles, along the beam scan path. Multiple frames were processed and analyzed to obtain statistical information. A typical exam of the temperature profiles from different speed functions are shown in Figure 5.5. The SF 20 case has the highest maximum temperatures and noticeably higher temperatures in general. On the other hand, SF 36 and SF 50 cases have close temperature distributions and SF 65 has slightly lower temperatures compared to SF 36 and SF 50. In the temperature profiles, a near-zero slope (low cooling rate) area can be identified as the liquidus-solidus transition. It also needs to point out that because of assumed Ti-6Al-4V emissivity setting for imaging (0.35), the transition zone is deviated from the theoretical liquidus/solidus temperatures of Ti-6Al-4V. However, the identified liquidus temperature \( T_L \) is still valid for melt pool size estimates, e.g., the distance between 2 locations with \( T_L \) as the melt pool length. The average molten pool dimensions, as well as standard deviations, for each speed function were calculated at the build height of 24.43 mm, shown in Figure 5.6. The melt pool length and width are in the range of 1.25 mm to 1.75 mm, and 0.75 mm to 1 mm, respectively. The melt pool result also shows noticeable variations, probably because both the process and measurements are sensitive to any change in the system. However, the trend clearly shows that both the molten pool length and width decreases with an increase of the speed function, also the beam speed.
Figure 5.5. Typical example of average temperature profiles from different speed functions at build height of 24.43 mm (Price, 2014).

Figure 5.6. Melt pool size vs. speed function at build height of 24.43 mm (Price, 2014).

The influence of the scanning speed to the microstructures from the X-plane specimen is shown in Figure 5.7. Generally, the width of columnar structure decreases with the increase
of the scanning speed, 109.7 µm at 214 mm/s vs. 37.1 µm at 529 mm/s, shown in Table 5.1. However, the exception is SF 65, in which the width of columnar structure is slightly larger than that of SF 50. For a given beam power, increasing the scanning speed would increase the cooling rate and the thermal gradient, which will form smaller columnar β grains (Bontha et al., 2009). Gockel and Beuth (2013) also reported that increasing the scanning speed will have a greater tendency to form columnar grain structures. Wu et al. (2004) also investigated the scanning speed effect on the grain morphology in laser AM of Ti-6Al-4V. At a higher scan speed, more nuclei can be generated due to rapider cooling with finer grains in long and narrow columnar morphology.

Table 5.1. Measured characteristic sizes from samples with various speed functions (SF).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Columnar β size (µm)</th>
<th>Equiaxed β size (µm)</th>
<th>α-lath thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>Standard deviation</td>
<td>Average</td>
</tr>
<tr>
<td>SF 20</td>
<td>109.7</td>
<td>30.2</td>
<td>85.2</td>
</tr>
<tr>
<td>SF 36</td>
<td>41.6</td>
<td>6.1</td>
<td>50.1</td>
</tr>
<tr>
<td>SF 50</td>
<td>37.1</td>
<td>6.3</td>
<td>48.7</td>
</tr>
<tr>
<td>SF 65</td>
<td>48.6</td>
<td>11.6</td>
<td>50.8</td>
</tr>
</tbody>
</table>
Figure 5.7. OM microstructures from X-plane specimens: (a) SF 20, (b) SF 50, and (c) SF 65.

Figure 5.8 shows microstructures, from OM for the Z-plane specimen, which is characterized by equiaxed grains for all SF values tested. The estimated size of equiaxed grains are also listed in Table 5.1. The SF 20 (lowest) sample has the largest grain size of 85.2 μm, while the grain sizes for SF 36, SF 50 and SF 65 are similar, all less than 50 μm. Gil et al. (2001) studied the effects of the cooling rate on the grain size in Ti-6Al-4V. The authors observed that the grain size is generally smaller at a faster cooling rate; however, further increasing over a certain cooling rate would not further reduce the grain size. In this study, it is also noted that the SF 65 sample show a larger percentage of $\alpha'$, as evident in Figure 5.9, SEM micrographs from the Z-plane specimens. Upon cooling, the transformation of equiaxed $\beta$ grains that exist at high temperatures produces the Widmanstätten ($\alpha+\beta$) structure. The $\alpha_{GB}$ is also found to form along the grain boundaries of prior $\beta$ grains.
As illustrated in Figure 5.9, SEM micrographs exhibit α_{GB} and primary-α colony morphologies. The size of the α-lath ranges from 0.5 µm to 5 µm depending on different process conditions (Al-Bermani et al., 2010; Xi et al., 2013). When Ti-6Al-4V alloy is cooled down from β-transus, the key factor to control the dimensions of α laths is the cooling rate (Bontha et al., 2009; Gockel and Beuth, 2013). The average thickness of the α-lath from different SFs is listed in Table 5.1. The SF 20 sample has the α-lath thickness of 1.5 µm, while the α-lath thickness is around 1.0 µm for other SF cases. Murr et al. (2009c) reported that the thickness of the α-lath is around 1.4 to 2.1 µm for different EBAM parts. It is evident that a higher cooling process results in a smaller lath size. At a higher cooling rate, a larger undercooling may lead to the formation of many α nuclei and result in smaller α-laths. Moreover, the size of α-lath is also affected by the size of the prior β (Safdar, 2012b; Gong et al., 2014). The larger the prior β columnar structure, the larger the α-lath during the subsequent solid state transformation. Further, the α phase grows in a lamellar fashion inside of β grains, and thus, the final length is limited by the prior β grain sizes, while the thickness of α-lath is controlled by diffusion and additional coarsening is possible provided with a slower cooling rate (Tiley, 2002). Since a higher cooling rate will result in increased martensite, as expected, more α′ is observed in the samples with a higher scanning speed, (SF 65), as shown in Figure 5.9.
Figure 5.8. OM microstructures from Z-plane specimens: (a) SF 20, (b) SF 50, and (c) SF 65.

Figure 5.9. SEM micrograph from Z-plane specimens: (a, b) SF 20 and (c, d) SF 65.
The scanning speed also affects the build defects such as part porosity. In this study, the SF 65 sample shows a much higher percentage of pores, which were found in both X-plane and Z-plane samples, with an example shown in Figure 5.10. Pores are also noticeable on the final build surface (as in Figure 5.1). The porous regions are also accompanied with unmelted particles and unconsolidated materials. The porous condition was also observed by Gaytan et al. (2009) and Puebla et al. (2012). Puebla et al. (2012) explained that the percentage of the porosity is related to the cooling rate and the melt pool size, all affected by the beam speed. A higher scanning speed results in a lower build temperature, a smaller melt pool, and a higher cooling rate, shown in Figures 5.5 and 5.6. Gong et al. (2013c) statistically evaluated the effects of the process parameters on the porosity of EBAM Ti-6Al-4V parts. The authors reported that the scanning speed has the most significant effect on the build part porosity, which increases greatly with increasing the scanning speed.

Figure 5.10. SEM micrograph showing porosity and unmelted powder in SF 65 sample: (a) unmelted powder and (b) pores.

5.4 Conclusions

Powder-bed EBAM is one of metal AM technologies that is capable of making full density metallic components of complex geometries and/or structures, greatly enabling design freedom and manufacturing flexibility. However, the EBAM process is very sensitive to the process parameters, among other factors such as powder characteristics, which may cause scattering of build part quality. In this study, the effects of the beam scanning speed on
build part microstructures from EBAM were experimentally studied. Four levels of speed function (SF), a parameter setting, were tested in building EBAM samples, which were subsequently used to prepare metallographic specimens for observations and analysis. During the experiment, an NIR thermal imager was also employed to acquire build surface temperature distributions, which was used for melt tool size estimates. The major findings are summarized as follows.

(1) The X-plane (side surface) specimens show columnar prior β grains with martensitic structures. The measured width of columnar grains is in the range of about 40-110 µm for the beam speeds tested in this study. With the increase of the beam scanning speed, the width of the columnar grains tends to be smaller because of a higher cooling rate during solidification.

(2) The Z-plane (scanning surface) specimens show equiaxed grains, in the range of 50-85 µm. The grain size from the lowest-speed sample (SF 20 case) is much larger than that of samples from higher beam speed cases. The microstructure inside of equiaxed grains is Widmanstätten (α+β).

(3) In addition, increasing the scanning speed from 214 mm to 376 mm/s will result in finer α-laths. However, further increasing the beam speed, e.g., to 689 mm/s, will not further reduce the α-lath size.

(4) From this study, a SF between SF 36 and SF 50 may result in fine microstructures without severe surface pores in the EBAM Ti-6Al-4V parts.


CHAPTER 6

MICROSTRUCTURE EVOLUTION IN EBAM

6.1 Introduction

During the EBAM process, metallic powder particles are selectively molten by an electron beam, and then rapidly self cooled and solidified. First, the substrate is placed underneath the build plate and used for a support and thermal isolation during the build plate pre-heating stage. Later as the additive process continues, additional layers of powder are placed on the top of the substrate. A thermal model is required to investigate the thermal history of the EBAM process. However, the thermal process simulation of EBAM is still a challenging task because of the complex heat transport and physical mechanisms involved. Multiple thermal models are applied on different manufacturing processes, such as electron beam welding (Liu et al., 2010; Luo et al., 2010), laser deposition or melting (Wang and Felicelli, 2008; Roberts, 2009; Yin, 2010), and laser welding (Lankalapalli et al., 1996; Tsirkas et al., 2003). According to these studies, the heat source is generally modeled as a conical volumetric body heat flux under the top surface of the workpieces. Due to substantially high computational costs and limited commercial code access, finite element analysis (FEA) modeling is still the most efficient way to simulate the EBAM process.

The dendritic structure is commonly formed during the solidification of alloys (Friedli et al., 2013). The growth of morphology has a strong link with the mechanical properties and performance of final products. In order to control the solidification structure and achieve the desired properties, fundamental knowledge about the mechanism of microstructure formation is required. The experimental and theoretical work has been carried out to characterize dendritic growth behavior. Nevertheless, because of its complexity, it is not yet well
understood and further research would be necessary. The emergence of simulation methods enables prediction on the grain structure and morphological evolution. The phase field model was originally proposed for simulating the dendrite growth in undercooled pure melts and has been extended to solidification of alloys. Despite the advantage of the phase field method, it still requires considerable computation time and can only simulate very small domains with a few dendrites. Modeling of solidification microstructures in rapid solidification requires understanding of different aspects of the physical phenomena occurring during the process, which are affected by both processing and material parameters (Fallah et al., 2012). There is some research about the simulation of dendritic morphology growth during the rapid solidification process (Yin and Felicelli, 2010; Tan et al., 2011). However, the microstructure evolution of the rapid solidification of the Ti-6Al-4V alloy is not studied yet. In addition, the effects of the phase field parameters on the morphology are also not investigated.

The type of phases present, structure size, morphology, and distribution of the fine microstructure ($\alpha+\beta$ colonies) all determine the properties and application of the Ti-6Al-4V alloy. The phase constitution of the Ti-6Al-4V depends strongly on the processing history. It is important to know the thermodynamics and kinetics of the phase transformations taking place during the EBAM process.

The previous experimental study has investigated the microstructure evolution of Ti-6Al-4V parts processed by EBAM with different scanning speeds in Chapter 5. The objective of this numerical study is to better understand the microstructural evolution, in terms of phase transformation and solute distribution, in EBAM built Ti-6Al-4V. A phase field model was applied to model the dendritic and columnar structure formation in EBAM. Moreover, the effect of the phase field parameters on the microstructure evolution is also studied. The intent is to predict the microstructure evolution during the solidification process in EBAM.
6.2 Thermal Analysis in EBAM

6.2.1 Thermal Model

A 2D FE model is applied to simulate the transient temperature field during the deposition of two consecutive layers of Ti-6Al-4V. The schematic of the geometry is shown in Figure 6.1.

![Figure 6.1. FEA model configuration: substrate and powder layers.](image)

The model is divided into two basic domains, (a) the two layers of metallic powder on the top and (b) the solid substrate. Domain A (green and above) includes two thin powder layers on the top of the substrate; domain B (brown and below) is the substrate. For the multi-layer scan, two sequential layers are simulated. The distance for a single scan is 8 mm. The first scan which moves from left to right on the first powder layer, and then right to left during the second scan on the second powder layer. In this study the layer thickness is 0.1 mm, and the substrate thickness is 8.8 mm.

The microstructure evolution during EBAM is determined by the thermal history of the materials, which is the result of energy absorption by the materials, heat conduction
within the built part, and heat losses. With the assumption of negligible molten flow during the solidification process, the governing equation of heat transport during the EBAM process becomes thermal conduction based (Hemmer and Grong, 1999):

\[
\nabla (k \nabla T) + \dot{Q} = \frac{d(\rho c_p T)}{dt} + v \frac{\partial(\rho c_p T)}{\partial x}
\]

(6.1)

where \( k \) is thermal conductivity, \( T \) is temperature, \( \rho \) is density, \( \dot{Q} \) is the absorbed heat flux, \( c \) is specific heat capacity, and \( v \) is the constant speed of the moving heat source on the scanning surface. The latent heat of fusion, \( L \), was considered in this model to track the solid/liquid interface of the molten pool.

The latent heat of fusion, \( L_f \), was considered in this model to track the solid/liquid interface of the molten pool. When the temperature drops between the liquidus and solidus temperatures, \( T_L \) and \( T_S \), respectively, the latent heat of fusion is modeled as an additional term of the internal thermal energy per unit mass, \( dU \). Hence, the enthalpy is defined as:

\[
H(T) = \int c dT + L_f f_e
\]

(6.2)

where \( f \) is the volumetric liquid fraction, which is defined as

\[
f_e = \begin{cases}
0 & T < T_S, \\
\frac{T - T_S}{T_L - T_S} & T_S \leq T \leq T_L, \\
1 & T > T_L
\end{cases}
\]

(6.3)

In this study, the heat source is modeled as Equation (6.4), which is slightly modified from Rouquette et al. (2007). For the 2D model, the equation of the intensity is described as:

\[
S(x, y) = f(y) \frac{8\eta U I_b}{\pi \Phi^2} \exp \left\{ -\frac{8(x-x_S)^2}{\Phi^2} \right\}
\]

(6.4)

With \( f(y) = \frac{2}{h} \left( 1 - \frac{y}{h} \right) \)

(6.5)

where the parameters are: efficiency coefficient \( \eta \), voltage \( U \), current \( I_b \), penetration \( h \), and beam diameter \( \Phi \). \( x_S \) and \( y_S \) are the position of the heat source (electron beam) center.
This subroutine of DFLUX can read the simulation time and determine the beam center position and raster direction before each calculation, so that the domain of the volumetric heat flux can be determined. The magnitude of the heat flux at each node will be interpolated with the heat source equation (Hemmer and Grong, 1999).

The preheating plays an important role in the EBAM process. The preheating takes place within the powder bed during EBAM, and it is usually preheated to about 700-800 °C before melting the powder. The substrate and powder layer are assigned with a uniform temperature distribution of $T_{\text{preheat}}$ as the initial thermal condition. The temperature of solid substrate bottom is held at a constant temperature of $T_{\text{preheat}}$ as the thermal boundary condition. In the model set up, the initial temperature of the entire model is set to 750 °C ($T_{\text{preheat}}$).

The geometric boundary conditions are shown in the model configuration (Figure 6.1). The bottom of the substrate is set as stable. Therefore only the top section of the actual substrate is simulated; all the mechanical degrees of freedom are confined at the bottom of the substrate due to the bottom being bound by the solid bulk material which is not included in this study. The contact between the solid bulk material and powder is assumed to be the same as the solid, since all the powder are preheated to be sintered before each scan.

There are two cooling steps: the first cooling stage, which lasts a few seconds, occurs during a pause between the first and the second powder laying scans; the second cooling state to room temperature commences after both powder layers have been scanned. The time for the first cooling step is set at 3 s, which approximates the break for the new powder spreading in EBAM machine. The thermal boundary conditions are the same as those in the melting step. The second cooling step is defined in the same way as the final cooling step in the single straight scan simulation, which is 50 s in this simulation.
The final temperature of the model is assumed to be room temperature (20 °C). Due to the vacuum condition of the chamber, the convective heat transfer between the powder layer and environment is ignored. Hence, only radiative heat transfer is considered between the powder/substrate and its surroundings.

The materials used in this study have temperature dependent properties. Figure 6.2 shows the temperature dependent density, thermal conductivity and specific heat (Yang et al., 2010) below the melting point for the solid substrate. The emissivity of Ti-6Al-4V was estimated from literature. Yang et al. (2010) experimentally calibrated the emissivity of Ti-6Al-4V as 0.7. Further properties not covered here are enclosed in Table 6.1.

Due to the application of metallic powder in EBAM, the effects of powder porosity cannot be neglected. It is well known that most thermal properties of metallic powder materials are significantly different from those of the corresponding solid bulk material (Sih and Barlow, 2004). Researchers have indicated though that both the specific heat and latent
heat of fusion of powder can be considered the same as those of the solid material (Sih and Barlow, 2004; Zäh and Lutzmann, 2010). The emissivity of powder is modeled as the combination of the solid bulk emissivity and the emissivity of the gaps or cavities between adjacent powder particles. The weight of each component is determined by the area fraction of the surface that is occupied by the radiation emitting holes (Shen and Chou, 2012). The thermal conductivity is only considered an “effective” thermal conductivity due to thermal radiation and heat transfer through powder necks.

Table 6.1. Parameters used in thermal simulations

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solidus temperature, $T_S$ (°C)</td>
<td>1605 (Boyer et al., 1998)</td>
</tr>
<tr>
<td>Liquidus temperature, $T_L$ (°C)</td>
<td>1665 (Boyer et al., 1998)</td>
</tr>
<tr>
<td>Latent heat of fusion, $L$ (kJ/Kg)</td>
<td>440 (Boyer et al., 1998)</td>
</tr>
<tr>
<td>Electron beam diameter, $\Phi$ (mm)</td>
<td>0.4</td>
</tr>
<tr>
<td>Absorption efficiency, $\eta$</td>
<td>0.9 (Zäh and Lutzmann, 2010)</td>
</tr>
<tr>
<td>Scan speed, $v_s$ (mm/s)</td>
<td>200, 400, and 600</td>
</tr>
<tr>
<td>Acceleration voltage, $U$ (kV)</td>
<td>60 (Gaytan et al., 2009)</td>
</tr>
<tr>
<td>Beam current, $I_b$ (A)</td>
<td>0.002 (Gaytan et al., 2009)</td>
</tr>
<tr>
<td>Powder layer thickness, $t_{layer}$ (mm)</td>
<td>0.1 (Zäh and Lutzmann, 2010)</td>
</tr>
<tr>
<td>Porosity, $\phi$</td>
<td>0.45</td>
</tr>
<tr>
<td>Beam penetration depth, $d_p$ (mm)</td>
<td>0.1</td>
</tr>
<tr>
<td>Preheat temperature, $T_{preheat}$ (°C)</td>
<td>750 (Gaytan et al., 2009)</td>
</tr>
</tbody>
</table>

6.2.2 Effects of the Beam Scanning Speed

Since a faster scanning velocity may cause lower beam energy density per unit area per unit time, a lower temperature distribution on the scanning path will be anticipated.
Figure 6.3 shows temperature contours and molten pool geometries at various levels of scanning speeds (200, 400 and 600 mm/s) on the powder layers. Based on Figure 6.3 and results in Table 6.2, a higher maximum molten pool size occurs at a lowest speed (200 mm/s). The maximum temperatures of the three speeds of 200, 400 and 600 mm/s are 3419 °C, 2792 °C and 2492 °C, respectively. Increasing the scanning speed results in less energy dissipation due to conduction and radiation. All the depths of the molten pool are larger than the powder layer thickness used in this model (100 µm).

Table 6.2. Simulated molten pool sizes with different beam scanning speeds

<table>
<thead>
<tr>
<th>Φ (mm)</th>
<th>Speed (mm/s)</th>
<th>Length (µm)</th>
<th>Depth (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4</td>
<td>200</td>
<td>1687</td>
<td>274</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>845</td>
<td>143</td>
</tr>
<tr>
<td></td>
<td>600</td>
<td>735</td>
<td>117</td>
</tr>
</tbody>
</table>

6.3 Phase Field Modeling

6.3.1 Phase Field Model

In the phase field model, the free energy density, \( f(c, \phi) \), where \( \phi \) is the phase field, is defined as the sum of the free energies of liquid and solid phases with different
compositions of \(c_L\) and \(c_S\), respectively, and an imposed double-well potential, \(w \cdot g(\phi)\). The chemical potentials (\(\mu\)) are defined as the difference between the chemical potentials of solute and solvent (Kim and Kim, 2001; Suzuki et al., 2002; Kobayashi et al., 2000).

\[
f(c, \phi) = h(\phi) f^S(c_S) + (1 - h(\phi)) f^L(c_L) + w \cdot g(\phi) \tag{6.6}
\]

\[
c = h(\phi)c_S + (1 - h(\phi))c_L \tag{6.7}
\]

\[
\mu^S(c_S(x,t)) = \mu^L(c_L(x,t)) \tag{6.8}
\]

where \(h(\phi) = \phi^3(6\phi^2 - 15\phi + 10), \ g(\phi) = \phi^2(1 - \phi)^2\), the superscripts or subscripts of \(S\) and \(L\) indicate solid and liquid phases, respectively. Note that the chemical potentials, \(\mu^S\) and \(\mu^L\), are derived from the free energy densities, \(f^S\) and \(f^L\). The free energy density at the interface without the \(w \cdot g(\phi)\) is defined as the fraction-weighted average values of the free energy in solid and liquid phases.

The phase field model for solidification is based on the Ginzburg-Landau free energy functional. The phase field and diffusion equations are described as (Kim et al., 1999):

\[
\frac{\partial \phi}{\partial t} = M[\varepsilon^2\nabla^2 \phi - h'(\phi) [f^S - f^L] - w \cdot g'(\phi)] \tag{6.9}
\]

\[
\frac{\partial c}{\partial t} = \nabla \cdot \left( \frac{D(\phi)}{\partial^2 f / \partial c^2} \nabla \frac{\partial f}{\partial c} \right) \tag{6.10}
\]

where \(M\) and \(\varepsilon\) are phase field parameters, \(D(\phi)\) is the solute diffusion coefficient. The phase field parameters of \(\varepsilon\) (gradient energy coefficient) and \(w\) are related to interface energy (\(\sigma\)), interface width (\(\lambda\)). The detailed equation for the height of double-well potential is described (Feng, 2007):

\[
w = \frac{6.6\sigma}{\lambda} \tag{6.11}
\]

The equation for the gradient energy coefficient \(\varepsilon\) is calculated by (Zhang, 2012):

\[
\varepsilon = \varepsilon_0(1 + \nu \cdot \cos(4\theta)) \tag{6.12}
\]
where $\nu$ is the magnitude of anisotropy, and $\theta$ is the angle between normal direction of interface and horizontal direction in the model. $\varepsilon_0$ is the mean value of $\varepsilon$ and could be calculated by the following equation (Feng, 2007):

$$
\varepsilon_0 = \sqrt{\frac{6\lambda\sigma}{2.2}}
$$

(6.13)

The influence of the noise could be put in a solutal function and phase function (Zhang, 2012). In the current model, the noise is used in the phase function, and it is defined as:

$$
\frac{\partial \phi}{\partial t} \rightarrow \frac{\partial \phi}{\partial t} + 16g(\phi)\chi\omega
$$

(6.14)

where $\chi$ is a random number distributed uniformly between -1 and 1, and a new number is generated for every point of the mesh at each time step; $\omega$ is an amplitude of the fluctuations.

In phase field modeling, the phase field $\phi=0$ means the alloy is liquid, while $\phi=1$ means the alloy is solid. Interface cells also possess a solid fraction between $\phi=0$ and 1, whereas all liquid and solid cell have zero and unity solid fractions, respectively. For the solute concentration field, each solid and liquid cell has one solute concentration, while the interface cells have both a solid and a liquid solute concentration.

The local equilibrium at the S/L interface is described as (Nastac, 1999):

$$
C^*_L = \frac{\Delta T}{m}
$$

(6.15)

$$
K = \frac{C^*_S}{C^*_L}
$$

(6.16)

where $C^*_S$ and $C^*_L$ are the concentrations of the solid interface and liquid interface, respectively; $m$ is the liquidus slope, $\Delta T$ is the undercooling, and $K$ is the partition coefficient. In this model, the comprehensive undercooling, including curvature undercooling, thermal
undercooling, and constitutional undercooling are applied. In addition, undercooling is set as a scalar factor and it is kept the same for different directions in the model.

In this model, the ternary Ti-6Al-4V alloy is treated as binary and the solute is the combination of Al and V. The thermo-physical parameters used in the phase field modeling are listed in Table 6.3. It is assumed that the initial temperature and concentration of liquid is uniform in the calculated region. The programming was written in Matlab to simulate the microstructure evolution. The multiple dendrites growth is also simulated to validate the phase field modeling.

Table 6.3. Parameters used in the phase field modeling on Ti-6Al-4V alloy (Nastac, 2012; Kim et al., 1999; Suzuki et al., 2002).

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquidus slope, ( m_L ) (K wt. %(^{-1}))</td>
<td>-0.088</td>
</tr>
<tr>
<td>Initial concentration, ( C_0 ) (wt. %)</td>
<td>10.0</td>
</tr>
<tr>
<td>Equilibrium partition coefficient, ( K )</td>
<td>0.5</td>
</tr>
<tr>
<td>Diffusion coefficient in liquid, ( D_L ) (m(^2)s(^{-1}))</td>
<td>9.5E-9</td>
</tr>
<tr>
<td>Diffusion coefficient in solid, ( D_S ) (m(^2)s(^{-1}))</td>
<td>5.0E-13</td>
</tr>
<tr>
<td>Interface energy, ( \sigma ) (J/m(^2))</td>
<td>0.5</td>
</tr>
<tr>
<td>Interface mobility, ( M )</td>
<td>0.3</td>
</tr>
<tr>
<td>Magnitude of anisotropy, ( \nu )</td>
<td>0.01-0.07</td>
</tr>
<tr>
<td>Noise factor, ( \omega )</td>
<td>0.01-0.07</td>
</tr>
</tbody>
</table>

6.3.2 Phase Field and Concentration Field

The 2D zone with the size of 80 μm × 80 μm is considered for the study of single dendrite growth. The simulated results of the phase field and solute concentration field at different time steps are presented in Figures 6.4 and 6.5, respectively. The grain growth morphologies and solute concentration at 0.01 ms, 0.2 ms, 1 ms and 1.2 ms are shown,
respectively. The initial seed is put in the center of the domain and applied to simulate the dendrite structure growth, as can be seen in Figure 6.4(a). The preferential growth orientation of the primary dendrites is parallel or perpendicular to the $x$-direction. In the beginning of growth, arms are not obvious. With continuous growth, solutes accumulate at solid/liquid (S/L) interface more intensely. The primary arms begin to grow from the horizontal and vertical directions, as shown in Figure 6.4(b). With the time increase, the dendritic structure is formed. When the solidification time is 1.6 ms, the continuous growth of the dendritic structure can be seen in Figure 6.4(d). It is noted that the current model is applied to model the growth of primary $\beta$ phase, and the phase has a body centered-cubic crystal structure with four-fold symmetry.

![Figure 6.4. Simulated dendrite structure growth at different time: (a) 0.01 ms, (b) 0.2 ms, (c) 1 ms, and (d) 1.6 ms.](image)

The solute concentration profiles shown in Figure 6.5 are consistent with the phase field profile. During the dendritic structure growth, the interface is found to have the highest
concentration. In the current study, $K$ is less than 1 and it is obvious that the $C^*_S$ is much smaller than $C^*_L$. It is also noted that the interface is described by a smooth transition of the phase field variable, which extends in thickness over the range of several grid nodes. The smooth interface in the phase field leads to a much higher computational cost compared to cellular automata which consists of one layer of cells (Kobayashi et al., 2000).

![Figure 6.5](image)

Figure 6.5. Simulated solute concentration during dendrite structure growth at different time: (a) 0.01 ms, (b) 0.2 ms, (c) 1 ms, and (d) 1.6 ms.

6.3.3 Effect of Process Parameters

The phase field parameters, such as the noise and anisotropy, have a great influence on the morphology or solute concentration distribution. The interface anisotropy coefficient represents the tension of interfacial surface, interface thickness and anisotropy degree of interface kinetics (Nakajima et al., 2006).
Two magnitudes of anisotropy ($\nu$) are applied to investigate the influence on morphology, and the result of the morphology at the step time of 2 ms is listed in Figure 6.6. The increased anisotropy parameter leads to the morphology of the dendrite structure and growth velocity. To quantitatively evaluate the growth rate of the dendrites under different magnitudes of anisotropy, the tip velocities are calculated based on the growth morphology. The velocities are 14 mm/s and 23 mm/s for the lower and higher $\nu$, respectively. Under the same conditions, the larger the anisotropy coefficient is, the faster the dendrite grows. When the anisotropy coefficient value is 0.065 or more, the dendrite variation occurs. This is because as anisotropy coefficient increases, the thermal noise is likely to be amplified, the front interface will become unstable, and the grain shape may become complicated (Zhao and Hou, 2013). Zhao et al. (2003) studied that if no anisotropy exists, no dendrite appears; nevertheless a large anisotropic coefficient complicates the grain pattern.

![Figure 6.6. Dendrite growth at different magnitudes of anisotropy: (a) 0.02, and (b) 0.04.](image)

For considering the fluctuations at the interface, stochastic noise introduced into the phase field model causes fluctuations at the solid/liquid interface that leads to the development of a dendritic structure. Two noise factors ($\omega$) are applied to investigate the influence on morphology when $\nu$ is kept as 0.03, and the result of the morphology at the step
time of 2 ms is listed in Figure 6.7. It is found that the higher noise in the phase field modeling is responsible for the side branching dendrites. Some scholars had studied the effects of incorporated noise on the grain patterns, and concluded that the conversed noise was the determinate reason of side-branching (Zhao et al., 2003). It is noted that, the change of the noise factors does not result in the large variation of the tip growth velocity: 19.4 mm/s and 18.2 mm/s for the $\omega$ of 0.01 and 0.04, respectively.

![Figure 6.7. Dendrite growth with different noise factors: (a) 0.01, and (b) 0.04.](image)

According to the principle of crystallization, undercooling has an important impact on growth processes of dendritic structure. To demonstrate the influence of undercooling on the dendrite growth, simulations are conducted with different amounts of undercooling, with corresponding simulation times of 0.6 ms. A nucleus is placed at the center of the calculation domain. Figure 6.8 illustrates the effect of undercooling on the average velocity of primary arm growth.
Figure 6.8. Dendrite growth at different undercooling up to 0.6 ms: (a) 47.5 K; (b) 52.5 K; and (c) 55 K.

The larger undercooling helps the formation of the primary arms. Smaller undercooling leads to lower velocity of grain growth, which results in more time for the transfer of the solute from the S/L interface to the bulk liquid region. This is the reason that small undercooling results in lower maximum composition. Lipton et al. (1984) developed an analytical model (the LGK model) which described free dendrite growth at a given melt undercooling. The tip growth velocity with various undercoolings calculated by the LGK theory is shown in Figure 6.9. The influence of undercooling on tip growth velocity follows well with the analytical model, which describes free dendrite growth at a given melt undercooling (Lipton et al., 1984). Zhu and Stefanescu (2007) compared the dendritic
morphologies for Al dendrites grown at different undercoolings. They found that the dendrite arms at the smaller undercooling were thicker than those at the larger undercooling. In the present model, similar results are obtained. In addition, it can be seen that a larger undercooling increases the growth speed of the dendrite and causes the formation of an increased number of secondary dendrite arms. A similar phenomenon is also observed by Zhao et al (2013). The increase of secondary dendrite arms reflects the further instability of the S/L interface at large undercoolings, while the quicker growth of the dendrite at larger undercooling is mainly due to the larger driving force. The increase of the interface instability, along with the undercooling, is also within the solidification theory predicted by Mullins and Sekerka (1964).

To quantitatively evaluate the effects of different parameters in the phase field modeling, the range of the tip growth velocity is listed in Figure 6.10. Both the lower values and high values of the tip growth velocity corresponding to different parameters are shown. It could demonstrate that all of the parameters have influence on the growth velocity. However, compared to the anisotropy and noise factors, the undercooling has a much greater influence on the dendritic structure growth.

![Figure 6.9. Tip growth velocity vs. undercooling calculated by the phase field model.](image)
6.3.4 Multiple Columnar Structure Growth

In EBAM, the columnar prior β grains are formed from the liquid. The build plate or the finished layers in EBAM provide the preferential sites for heterogeneous nucleation. The solidification of the Ti-6Al-4V alloy involves two steps: liquid to primary solid phase of β and solid phase transformation (β to α or α’) depending on the cooling rate. The nucleation and growth of columnar grains of prior β takes place during initial rapid solidification when the temperature is above the β-transus temperature (about 980 °C). The size of α after solid phase transformation is also affected by the size of the prior β (Safdar, 2012). The larger the prior β columnar structure, the larger the α-lath during the subsequent solid state transformation. The morphology and the spacing of the phases are critical to the properties of the EBAM Ti-6Al-4V alloy, and it is of great interest to simulate the columnar β growth during the EBAM process.

The thermal model is able to model the temperature history during the EBAM process. After the modeling, the temperature history of the EBAM process is achieved. Under the equilibrium condition, the cooling rates are calculated when the temperature is below the melting temperature and above the solid phase transformation temperature (about 980 °C). In
one EBAM powder layer, three cooling rates (top, middle, and bottom) are calculated. The mean value of the cooling rate is shown in Table 6.5. A faster cooling rate results in a larger undercooling and subsequently contributes to a higher density of the nucleation sites in EBAM. The relationship between the cooling rate and nucleation sites is found based on this regression equation (Luo and Zhu, 2013):

\[
\rho = 28.8 \exp\left\{-\left(\frac{C_r - 68970}{60120}\right)^2\right\}
\]  

(6.17)

Table 6.4. Calculated cooling rate from thermal model.

<table>
<thead>
<tr>
<th>SF</th>
<th>SF 20</th>
<th>SF 36</th>
<th>SF 50</th>
<th>SF 65</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actual speed (mm/s)</td>
<td>214</td>
<td>376</td>
<td>529</td>
<td>689</td>
</tr>
<tr>
<td>Simulated cooling rate (°C/s)</td>
<td>11,078</td>
<td>34,965</td>
<td>65,780</td>
<td>106,815</td>
</tr>
</tbody>
</table>

To model the growth of the columnar grains, nuclei sites are placed at the bottom wall at the beginning of the simulation. The calculation domain has a 2000 by 2000 mesh. Figure 6.11 shows the simulated columnar dendrites under different speed functions (related to scanning speeds and cooling rate), respectively, at the corresponding simulation time of 10 ms. The primary arms, whose morphology orientations are not parallel to the temperature gradient direction, are stopped by the growth of the arm which is parallel to the thermal gradient direction. The growth of some of the main arms is suppressed by nearby dendrites. High liquid composition between the two columnar grains makes the secondary arms comparatively short due to the small separation between them. The simulation results agree qualitatively well with the experimental studies of columnar microstructure in regard to different speed functions. The current model can not only simulate the dynamic growth of the columnar dendrites, but can also be applied to the branching and competition growth processes.
During the EBAM process, the cooling rate is high, so the grain growth velocity is correspondingly high. The columnar dendritic grains grow rapidly from the bottom boundary to the center. At the beginning of the growth, the secondary arms are not distinct and do not generate on the surfaces of some columnar grains. This results from not only the undercooling conditions, but also the situation of remaining space that is required for the growth of the secondary arms. This remaining space can be quantitatively correlated to the primary dendrite spacing. The following results indicate that the secondary arms are distinct as the primary dendrite spacing increases.

Figure 6.11. Comparison of the phase field contours between modeling results with different speed functions: (a) SF 20; (b) SF 36; (c) SF 50; (d) SF 65.
Note that the microstructure shown in Chapter 5 represents the Ti-6Al-4V alloy after primary solidification and then phase transformation. However, the columnar structure produced from the primary solidification is still visible. To quantify the size of the columnar structure, the width of the columnar prior β grains was measured. According to the previous studies in Chapter 5, for a given beam power, increasing the scanning speed would increase the cooling rate and the thermal gradient, which will form smaller columnar β grains. At a higher scan speed, more nuclei can be generated due to rapider cooling with finer grains in long and narrow columnar morphology. The results could demonstrate that the phase field model is able to model the columnar structure growth during the EBAM process.

6.4 Solid State Phase Transformation Kinetics

6.4.1 Introduction to Johnson-Mehl-Avrami Theory

The thermodynamics of the phase equilibrium in Ti alloys are generally well studied. However, the computation of the kinetics has not yet been equally developed. There are few studies that have calculated the quantitative composition of the phases in the alloy. Understanding the phase fractions may help to predict the mechanical properties of the EBAM parts. One of the most effective methods is the Johnson-Mehl-Avrami (JMA) theory (Avrami 1939; Avrami 1940; Avrami 1941).

6.4.2 Phase Transformation in EBAM

The Ti-6Al-4V alloy is an α+β Ti alloy which presents two equilibrium phases, the HCP α and the BCC β. The melting structure of the alloy consists of 100% β. During the cooling from the β-transus temperature to room temperature, β may transform into two different phases, depending on the cooling rate. If the cooling rate is lower than 410 °C/s, a diffusion controlled transformation will take place, and the β phase will transform to α phase as it cools to room temperature. The JMA theory is valid for the Ti-6Al-4V alloy (Charles,
In an isothermal condition, the kinetics of the phase transformation is described by the JMA equation:

\[ f_\alpha(T) = 1 - \exp(-k_r t^n) \quad (6.18) \]

\[ f_\beta(T) = 1 - f_\alpha(T) \quad (6.19) \]

where \( f_\alpha(T) \) and \( f_\beta(T) \) is the volume fraction of \( \alpha \) phase at time \( t \), \( t \) is the time duration in seconds, \( k_r \) is the reaction rate constant, and \( n \) the Avrami constant, both \( k \) and \( n \) are dependent on the type of phase transformation and grain growth (Malinov et al., 2002; Katzarov et al., 2002). The \( n \) and \( k \) values are suggested as 1.15-1.6 and 0.033-0.045 for the Ti-6Al-4V alloy (Malinov et al., 2001; Fan et al., 2005).

For cooling rates higher than 410 °C/s the \( \beta \) to \( \alpha \) transformation is suppressed and the \( \beta \) transforms by a martensitic transformation into \( \alpha' \) martensite. The proportion of \( \beta \) transformed into martensite depends essentially on the undercooling below the \( M_s \) and is given by this empirical formula (Elmer et al., 2004):

\[ f(\alpha') = f'(\beta)\{1 - \exp[-c(M_S - T)]\} \quad (6.20) \]

where \( f'(\beta) \) is the volume fraction of the available \( \beta \) phase for martensitic transformation; \( M_S \) is the martensitic transformation starting temperature, and \( c \) is a material constant and the value for the Ti-6Al-4V is set as 0.015 (Crespo et al., 2009).

It is known that the mechanical properties of different phases are quite different and the mechanical properties of Ti-6Al-4V alloy may be calculated from the phase constitution of the alloy using the rule of mixtures. The Young’s modulus and hardness were calculated from the phase constitution of the alloy using the rule of mixtures. The Young’s moduli of \( \alpha \), \( \beta \) and \( \alpha' \) are 117, 82 and 114 GPa, respectively, and the related Vickers hardnnesses are 320, 140 and 350 HV, respectively (Crespo et al., 2011).
Kelly studied the influence of cooling rates on the laser deposition process on the Ti-6Al-4V alloy, in which a JMA model was used to predict the fraction of $\alpha$ in the microstructure (Kelly, 2004). Fan et al. (2005) also investigated the effect of phase transformations on laser forming of the Ti-6Al-4V alloy. The $\beta$ to $\alpha$ transformation during the decomposition of $\beta$ phase, producing either martensite $\alpha'$ or $\alpha$ depending on the cooling rate, is numerically investigated. The spatial distribution of volume fractions of phases is obtained by coupling thermal and phase transformation kinetic modeling.

6.4.3 Validation of Phase Transformation in EBAM

The volume fraction of the phases ($\alpha$, $\beta$ and $\alpha'$) may be determined using a regular grid of points overlaid onto the high magnification SEM images (Tiley et al., 2004). Dividing the number of points that lay within each phase by the total number of points gives an estimate of the phase fractions. Examples of etched samples are shown in Figure 6.12; different colors represent different phases: blue for $\alpha$, green for $\beta$ and red for $\alpha'$. For each SF sample, two high magnification images were used for a statistical study. The results of the phase fractions are shown in Figure 6.13. Generally, increasing the scanning speed, the fraction of $\alpha$ decreases while the fraction of $\alpha'$ noticeably increases. The trend of the volume fractions is correspondent to the theoretical study by the phase transformation kinetics. The phase transformation from $\beta$ to $\alpha$ is diffusional and time dependent. Thus, a slower cooling rate leads to the diffusion-controlled nucleation and growth process of $\alpha$. While a higher cooling rate provides a shorter time for the transformation, and results in a smaller fraction of $\alpha$. For $\beta$, since it locates at the boundaries of $\alpha$ laths, the volume fraction of $\beta$ decreases with the decrease of $\alpha$ when the scanning speed is increased. For $\alpha'$ martensites, a higher scanning speed results in a higher cooling rate which produces more martensites, i.e., the $\alpha'$ fraction increases with the increase of the scanning speed.
Figure 6.12. Examples of volume fraction measurements: (a) SF 36 and (b) SF 65.

Figure 6.13. Result of volume fraction measurements with different SFs.

To quantitatively predict the phase fraction during solid state phase transformation in EBAM, the cooling rates were first calculated from a thermal model. During the cooling in...
EBAM, a diffusion controlled transformation will take place and the β phase will progressively transform to α phase as it cools from 980 ºC (known as the β-transus temperature) (Crespo et al., 2009) to $M_S$ (725 ºC). When the temperature is lower than $M_S$, whether the diffusion-controlled β to α transformation or the martensitic transformation would take place was then governed based on the cooling rate. From the calculation of the thermal model simulations, the cooling rates from the four scanning speed cases are much higher than 410 ºC/s. Hence, it means that the martensitic transformation will take place when the temperature is lower than $M_S$, and the diffusion-controlled transformation (β to α) stops. For the JMA equation in the current study, the $n$ and $k$ used were as 1.5 and 0.04, respectively. For the martensitic transformation, the value for $c$ is set as 0.015 (Gong et al., 2014).

Figure 6.14 presents the phase fractions changed with the scanning speed, and Figure 6.15 shows the difference of the phase volume fraction results between analysis and experiment. The estimated phase constitution is in a reasonable agreement with the experimental results. This indicates that the modeling approach is able to predict the microstructure evolution induced by EBAM processing of the Ti-6Al-4V alloy.
6.5 Conclusions

A phase field model was developed to model the microstructure evolution in the EBAM built Ti-6Al-4V alloy. As an input to the phase field model, the cooling rate was extracted from the thermal simulation. Matlab code was used to implement and solve the phase field equations, and achieve the phase field and solute distribution in EBAM. A thermo-kinetic model coupling heat transfer and phase transformation kinetics, which predicts the phase constitution in the Ti-6Al-4V by EBAM, was developed. In addition, the phase volume fractions were evaluated by experimental and computational methods. The temperature history and cooling rate from the thermal model are incorporated well into the phase transformation modeling. The major findings are summarized as follows.

(1) The thermal model was applied to study the scanning speed and beam diameter effects. With the increase of the beam scanning speed, temperatures are lower in the molten pool, which not only decreases in the depth direction, but also in the direction the beam is moving.

(2) The phase field model is able to model the morphology and solute concentration during solidification. The phase field parameters, such as anisotropy, play an important role.
in the morphology evolution during EBAM. The dendrite morphology at various undercoolings is simulated. The undercooling is shown to affect the dendrite growth significantly, and the larger undercooling results in the greater growth velocity. The columnar dendritic spacing and the width of dendrites decreases with the increase of beam scanning speed. The columnar dendritic arm spacing values, estimated from the phase field simulations, match reasonably well with those obtained in the experimental observation.

(3) From the experimental study of the phase constitution, increasing the scanning speed leads to the increase of the $\alpha'$ and the decrease of $\alpha$, while the variation of $\beta$ is not obvious. The variation of the phase volume fractions at different scanning speeds could be attributed to the different cooling rates during solid phase transformation. The result of the phase transformation model is consistent with the experimental results.
References


Nastac, L. (2012). Solute redistribution, liquid/solid interface instability, and initial transient regions during the unidirectional solidification of Ti-6-4 and Ti-17 alloys. CFD Modeling and Simulation in Materials Processing, Orlando, FL, March 11-15.


CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

7.1 Conclusions

The EBAM technology builds parts layer-by-layer from metal powder using an electron beam. EBAM technology is capable of producing fully melted and 100% dense alloy parts with superior properties. The use of EBAM on Ti-6Al-4V alloy has the ability to produce fine microstructure with strengthening phase, which makes the components stronger than those from conventional manufacturing methods. The thermal history, which includes the preheating, melting and solidification, is critical to determine the microstructure and mechanical properties of the EBAM parts. Understanding and controlling the microstructure evolution is vital to achieve the desired properties of the components. The major findings are summarized as follows.

(1) Preheated Ti-6Al-4V powder from the EBAM process has been characterized. Preheating leads to metallurgical bonds or even partial melting of the powder during the EBAM process. The phenomenon of neck formations is evident in both the Z-plane and X-plane sections. The diameter of the necks is on the order of 1 to 10 µm. In addition, the X-plane seems to have less particles of a sintering result because of the energy penetration limit. The micro-CT scan images of preheated powder show a similar porosity level between the Z-plane and X-plane surfaces. The calculated porosity of the preheated powder is about 50%. Moreover, the major diameter range of the powder is from 30 to 50 µm on both planes. Ti-6Al-4V powder has significantly lower thermal conductivity than that of a solid counterpart. Also, the thermal conductivity is highly temperature dependent: about 0.63 W/m·K at room
temperature and less than 2.44 W/m·K at 750 °C for the preheated powder. The microstructure of a Ti-6Al-4V preheated powder exhibits Widmanstätten (α+β) structure.

(2) The effects of the beam scanning speed on built part microstructures from EBAM were experimentally studied. The X-plane specimens show columnar prior β grains with martensitic structures. The width of columnar grains measured is in the range of about 40 to 110 µm. With the increase of the scanning speed, the width of the columnar grains tends to be smaller because of a higher cooling rate during solidification. The Z-plane specimens show equiaxed grains, in the range of 50 to 85 µm. The grain size from the lowest-speed sample (SF 20) is much larger than samples from higher SF cases. The microstructure inside of equiaxed grains is Widmanstätten (α+β). In addition, increasing the scanning speed will also result in finer α-lath. With the increase of the scanning speed, the porosity defects in the built part also become more severe. From this study, both SF 36 and SF 50 may be workable beam speeds that result in fine microstructures without severe porosity.

(3) A 2D thermal FE model was applied to simulate the temperature history and melt pool geometry in EBAM. With the increase of the beam scanning speed, temperatures are lower in the molten pool, which not only decreases in the depth direction, but also in the direction the beam is moving. For the microstructure modeling, a phase field model was developed to model the microstructure evolution in EBAM built Ti-6Al-4V alloy. The phase field model is able to model the morphology and solute concentration during solidification in EBAM. The undercooling is very critical to the growth velocity. The larger undercooling results in the higher growth velocity. The columnar dendritic spacing and the width of dendrites decrease with the increase beam scanning speed. To achieve the phase constitution in the EBAM Ti-6Al-4V alloy, a thermo-kinetic model coupling heat transfer and phase transformation kinetics was developed. From the study of the phase constitution, increasing the scanning speed results in the increase of α’ and decrease of α. The variation of the phase
volume fractions at different scanning speeds could be attributed to the different cooling rates during solid phase transformation.

7.2 Contributions of this Study

The contributions of this study are summarized below:

(1) The study correlates thermal history, microstructure evolution and mechanical properties in EBAM.

(2) This study investigates the scanning speed effects in EBAM components. The study would help to optimize the process parameters in EBAM.

(3) The phase field modeling has been developed to model the morphology and solute concentration during the grain growth. The thermal kinetic studies on phase transformation would help to predict the phase constitution of EBAM components by incorporating a thermal model.

7.3 Recommendations for Future Research

This study provides a better understanding of the microstructure evolution of EBAM components. Furthermore, the thermal modeling, microstructure modeling and thermal kinetics study provide better understanding of the microstructure and phase transformation in EBAM. Future research can be pursued in the following directions:

(1) For the sintered powder research, to investigate the preheating effect, further studies will be needed to use different preheating conditions (e.g., the beam power, beam speed, and scanning time) in EBAM and then to examine the sintering level by metallurgical characterizations. In addition, the influence of preheating conditions to the microstructure of built parts should also be investigated in order to optimize the process parameters in EBAM.

(2) The current study has investigated the scanning speed effects on the microstructure. Future studies may include other process parameters such as beam diameters,
input power and etc, and this study may help to have a better understanding of process parameters in EBAM and contribute to the process optimization in EBAM.

(3) In the microstructure characterization, simple geometry blocks are applied for microstructure evolution studies. Thus, its application may be limited in EBAM components with simple geometries. Therefore, investigating the thermal history and microstructure evolution for complex geometry components will be more beneficial in the understanding of EBAM technology.
Appendix

(1) Flow chart for phase field microstructure modeling
(2) Matlab Codes for phase field modeling

clc;clear all;close ; % Initialize

% Materials properties
N=800; % Domain matrix
T0=1888; % Initial temperature for the solidification
Tm=1938; % Melting point
Me=250; % Liquidus slope, K/(mol%)
Ke=0.5; % Partition coefficient
cle=(Tm-T0)./Me; % Equilibrium liquid concentration based on undercooling
cse=Ke*cle; % Equilibrium solid concentration
c0=0.138; % Initial concentration
cs0=1/((cle/cse*(1-cse)/(1-cle))*(1-c0)/c0+1); % Initial solid concentration

% Initial phase field and solute concentration field
S=zeros(N,N); % Initialize the phase field to zero
cl=c0*ones(N,N); % Initialize the liquid concentration
cs=cs0*ones(N,N); % Initialize the solid concentration
C=c0*ones(N,N); % Initialize the solid concentration
R=round(1/50*N); % Radius of the initial nucleation, R=1/50*N
d0= 1.0e-6; % Fundamental size in the model

% Set of nucleation
for x=1:N
    for y=1:N
        if (x-N/2)^2+(y-N/2)^2<=R^2;

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S(x,y)=1;
C(x,y)=cse;
cl(x,y)=cle;
cs(x,y)=cse;
end
end
end

% Simulation parameters

dx=0.1*d0; % Mesh in x direction
dy=0.1*d0; % Mesh in y direction
lambda=2*dx; % Interface thickness
sigma=0.5; % Interfacial energy
W=6.6*sigma/lambda; % Height of double-well potential
epsilon0=sqrt(6*lambda/2.2*sigma); % Phase field parameter
gamma=0.03; % Magnitude of Anisotropy
omega=0.02; % Noise factor
Dl=9.5e-9; % Liquid diffusion coefficient
Ds=5e-13; % Solid diffusion coefficient
Rg=8.31; % Gas constant
Vm=1.0e-5; % Molar volume
M=0.3; % Interface mobility
dt=dx^2/5/Dl; % Step time increment
max=1000; % Total steps
epss=1e-12; % Setting of a minimum number
% Initial conditions

for V=1:max;
    Mr=-1+2*rand(N,N);  % Random number between -1 and 1
    aa=zeros(N,N);
    bb=zeros(N,N);
    a=zeros(N,N);
    b=zeros(N,N);
    Xs=zeros(N,N);
    Ys=zeros(N,N);

% Phase field districts
    S1=S([2 1:end-1],:);
    S2=S([2:end end-1],:);
    S3=S(:,[2 1:end-1]);
    S4=S(:,[2:end end-1]);
    S5=S1(:,[2 1:end-1]);
    S6=S1(:,[2:end end-1]);
    S7=S2(:,[2 1:end-1]);
    S8=S2(:,[2:end end-1]);

% Solute concentration field districts
    C1=C([2 1:end-1],:);
    C2=C([2:end end-1],:);
    C3=C(:,[2 1:end-1]);
C4=C(:,[2:end end-1]) ;
C5=C1(:,[2 1:end-1])  ;
C6=C1(:,[2:end end-1]) ;
C7=C2(:,[2 1:end-1])  ;
C8=C2(:,[2:end end-1]) ;
cl1=cl([2 1:end-1],:) ;
cl2=cl([2:end end-1],:) ;
cl3=cl(:,[2 1:end-1]) ;
cl4=cl(:,[2:end end-1]) ;
cs1=cs([2 1:end-1],:) ;
cs2=cs([2:end end-1],:) ;
cs3=cs(:,[2 1:end-1]) ;
cs4=cs(:,[2:end end-1]) ;

% Derivatives of phase field
X=(S2-S1)/2/dx;
Y=(S4-S3)/2/dy ;
XX=(S2+S1-2*S)/(dx^2)  ;
YY=(S4+S3-2*S)/(dy^2)  ;
XY=(S5+S8-S6-S7)/4/(dx^2) ;

% Derivatives of solute concentration field
CX=(C2-C1)/2/dx;
CY=(C4-C3)/2/dy ;
CXX=(C2+C1-2*C)/((dx)^2) ;
\[ CYY = \frac{(C4 + C3 - 2C)}{(dy)^2}; \]
\[ CXY = \frac{(C5 + C8 - C6 - C7)}{4(dx^2)}; \]
\[ clX = \frac{(cl2 - cl1)}{2/dx}; \]
\[ clY = \frac{(cl4 - cl3)}{2/dy}; \]
\[ csX = \frac{(cs2 - cs1)}{2/dx}; \]
\[ csY = \frac{(cs4 - cs3)}{2/dy}; \]

\[ EE = (\text{abs}(S - S1) + \text{abs}(S - S2) + \text{abs}(S - S3) + \text{abs}(S - S4)) < \text{epss}; \]
\[ PP = (EE <= 0 \& S < 0.999); \]
\[ DD = XX + YY; \]
\[ xy = (X == 0).*(Y == 0); \]
\[ FF = PP.* (xy <= 0); \]

\[
\text{for } m = 1:N; \\
\quad \text{for } n = 1:N; \\
\quad \quad \text{if } FF(m,n) >= 1; \\
\quad \quad \quad a(m,n) = 4*(Y(m,n).* (X(m,n).^3)-X(m,n).* (Y(m,n).^3))./((X(m,n).^2+Y(m,n).^2).^2); \% \sin (4\theta); \\
\quad \quad \quad b(m,n) = 1 - 8*(X(m,n).^2).* (Y(m,n).^2)./((X(m,n).^2+Y(m,n).^2).^2); \% \cos (4\theta); \\
\quad \quad \quad Xs(m,n) = (X(m,n).* XY(m,n) - Y(m,n).* XX(m,n))./(X(m,n).^2+Y(m,n).^2); \% d\theta/dx \\
\quad \quad \quad Ys(m,n) = (X(m,n).* YY(m,n) - Y(m,n).* XY(m,n))./(X(m,n).^2+Y(m,n).^2); \% d\theta/dy \\
\quad \quad \text{end} \\
\quad \text{end} \\
\text{end} \\
\text{end} \]
% Anisotropy in phase field model
pa=(1+(gamma^2).*b.*b+2*gamma*b).*DD+(16*gamma*b+16*(gamma^2)*b.*b-...
…16*(gamma^2).*a.*a)*(Xs.*Ys.*X)-...
(8*(gamma^2).*a.*a+8*gamma*a)*(Xs.*X+Ys.*Y);

% h(Φ), g(Φ), D(Φ), and related derivatives
hp=S.^3.*(10-15*S+6*S.^2);
gpp=4*S.^3-6*S.^2+2*S;
hpp=30*(S-S.^2).^2;
gp=(S-S.^2).^2;
hppp=60*(2*S.^3-3*S.^2+S);
Dp=Ds*hp+Dl*(1-hp);
Dpp=(Ds-Dl)*hpp;
Dpx= Dpp.*X;
Dpy= Dpp.*Y;

fp=zeros(N,N);   % Random zero matrix
Z=zeros(N,N);    % Random zero matrix
% Solve cs and cl
for m=1:N;
    for n=1:N;
        if  FF(m,n)>=1;
            Z(m,n)=((1-cse).*(1-cl(m,n))./(1-cle)./(1-cs(m,n)));
            fp(m,n)=-Rg*T0/Vm*hpp(m,n).*log(Z(m,n))+W*gpp(m,n);
        end
    end
end
end

end

% Update the solute concentration field
DC1=Dp.*(CXX+CYY)+Dpx.*CX+Dpy.*CY;
DC2a=Dp.*hpp.*(cl-cs).*DD;
DC2b=(Dpx.*X+Dpy.*Y).*hpp.*(cl-cs);
DC2c=Dp.*hppp.*(cl-cs).*(X.^2+Y.^2);
DC2d=Dp.*hpp*((clX-csX).*X+(clY-csY).*Y);
DC2=DC2a+DC2b+DC2c+DC2d;
DC=dt*(DC1+DC2).*PP;
C=C+DC;

% Noise in phase field model
pb=omega*16*Mr.*gp;%= noise

% Update the phase field
FA=dt*((epsilon0).^2*pa-fp);
Eta=(M*FA-pb).*PP;
S=S+Eta;

% Limitation of phase field
S(S>1)=1;
S(S<0)=0;
% Random zero matrix
A=zeros(N,N);
B=zeros(N,N);
Cc=zeros(N,N);

% Solute concentration for liquid and solid
for m=1:N;
    for n=1:N;
        if FF(m,n)>=1;
            A(m,n)=(1-K).*(1-hp(m,n));
            B(m,n)=hp(m,n)+K.*(1-hp(m,n))-(1-K).*C(m,n);
            Cc(m,n)=-K.*C(m,n);
            cl(m,n)=(-B(m,n)+sqrt(B(m,n).^2-4*A(m,n).*Cc(m,n)))./(2*A(m,n));
            cs(m,n)=1./(K*(1-cl(m,n))./cl(m,n)+1);
        end
    end
end
end