Optimization of Electron Beam Melting for Production of Small Components in Biocompatible Titanium Grades

JOAKIM KARLSSON
Additive manufacturing (AM), also called 3D-printing, are technologies where parts are formed from the bottom up by adding material layer-by-layer on top of each other. Electron Beam Melting (EBM) is an AM technique capable of manufacturing fully solid metallic parts, using a high-intensity electron beam to melt powder particles in layers to form finished components. Compared to conventional machining, EBM offers enhanced efficiency for production of customized and patient specific parts such as e.g. dental prosthetics. However, dental prosthetics are challenging to produce by EBM, as their small sizes mean that mechanical and surface properties may be altered as part sizes decreases.

The aim of this thesis is to gain new insights that could lead to optimization for production of small sized components in the EBM. The work is focused to understand the process-property relationships for small size components production.

To improve the surface resolution and part detailing, a smaller sized powder was used for production and compared to parts made with standard sized powder. The surface-, chemical and mechanical properties were evaluated for parts produced with both types of powders. The results indicate that the surface roughness may be influenced by powder and build layer thickness size, whereas the mechanical properties showed no influence of the layer-wise production. However, the mechanical properties are dependent on part size. The outermost surface of the parts consists of a surface oxide dominated by TiO$_2$, formed as a result of reaction between the surface and residual gases in the EBM build chamber. The surface oxide thickness is comparable to that of a conventionally machined surface, but is dependent on build height.

This work concludes that the surface resolution and component detailing can be improved by various measures. Provided that proper process themes are used, the EBM manufactured material is homogenous with properties comparable to conventional produced titanium. It has also been shown that the material properties will be altered for small components. The results point towards different ways of optimizing manufacturing of dental prosthetics by EBM, which will make dental prosthetics available for an increased number of patients.

Keywords: Additive Manufacturing, 3D-printing, Electron Beam Melting, Titanium alloys, Chemical properties, Mechanical properties, Surface properties

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“3D printing has the potential to revolutionize the way we make almost everything”

-President Barack Obama

Till Thomas
List of Papers

This thesis is based on the following papers, which are referred to in the text by their Roman numerals.


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**Author’s Contribution**

My contribution to the papers included in this thesis was:

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### Abbreviations

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<th>Description</th>
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<tr>
<td>2D</td>
<td>Two-Dimensional</td>
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<td>3D</td>
<td>Three-Dimensional</td>
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<td>AES</td>
<td>Auger Electron Spectroscopy</td>
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<td>AM</td>
<td>Additive Manufacturing</td>
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<td>CAD</td>
<td>Computer Aided Design</td>
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<td>CAM</td>
<td>Computer Aided Manufacturing</td>
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<td>CT</td>
<td>Computer Tomography</td>
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<td>DIC</td>
<td>Digital Image Correlation</td>
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<td>E-beam</td>
<td>Electron Beam</td>
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<td>EBM</td>
<td>Electron Beam Melting; is a registered trademark of Arcam AB.</td>
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<td>EELS</td>
<td>Electron Energy Loss Spectroscopy</td>
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<td>FDM</td>
<td>Fused Deposition Modeling</td>
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<tr>
<td>ICP-OES</td>
<td>Inductively Coupled Plasma – Optical Emission Spectroscopy</td>
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<tr>
<td>PH</td>
<td>Pre-Heating</td>
</tr>
<tr>
<td>PRS</td>
<td>Powder Recovery System</td>
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<td>SEM</td>
<td>Scanning Electron Microscopy</td>
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<td>SLM</td>
<td>Selective Laser Melting</td>
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<td>SLS</td>
<td>Selective Laser Sintering</td>
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<tr>
<td>STEM</td>
<td>Scanning Transmission Electron Microscopy</td>
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<td>TEM</td>
<td>Transmission Electron Microscopy</td>
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<tr>
<td>Ti6Al4V</td>
<td>Titanium-6Aluminum-4Vanadium (wt%)</td>
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<tr>
<td>ToF-SIMS</td>
<td>Time-of-Flight Secondary Ion Mass Spectrometry</td>
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<tr>
<td>UTS</td>
<td>Ultimate Tensile Strength</td>
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<tr>
<td>UV</td>
<td>Ultraviolet</td>
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1. Introduction

Additive manufacturing (AM), commonly also named 3D-printing, rapid prototyping and/or freeform fabrication, has emerged as an efficient way to produce customized and fully functional parts from various materials [1]. AM is a revolutionary technique that is able to produce near-net-shaped parts from three-dimensional computer aided design (3D CAD) models from most kinds of materials, such as polymers, ceramics and metals [2]. Contrary to conventional machining, AM produces parts from the ground up by adding material in a layer-by-layer fashion rather than subtraction of material by i.e. milling or turning [3]. AM has progressed from only prototyping in polymeric materials in the late 80’s, to production of fully functional parts in metals and other materials. From this development, completely new areas of applications have emerged for use of the various AM-techniques [2, 4].

One AM technique that has found increased industrial use is Electron Beam Melting (EBM). EBM is an AM technique that uses a highly intense electron beam to selectively melt metal powders [5, 6]. In the following, the term EBM will be used to refer to this AM process. The EBM process is capable of producing fully solid parts from numerous materials such as Ti-6Al-4V [7], Ti-48Al-2Cr-2Nb [8], CoCr alloys [9] and H13 steel [10]. Today, EBM is used to produce, among other applications, customized titanium biomedical products with unique geometrical features [11]. EBM is also used for mass production of acetabular cups for hip replacements, containing a designed porosity that is claimed to result in improved bone ingrowth and fixation [12]. Other applications are production of fully functional components and titanium parts used in service today [13, 14].

The many possibilities with AM have made it a flexible production method, compared to conventional machining, with enhanced efficiency to produce customized and patient specific parts out of titanium for numerous medical devices. The need for customized and patient specific medical devices originates from the simple fact that we are all different, both in personality, but also in our external and internal physical appearance. Since diversity exists between individual patients, standard sized medical products aimed to replace and/or support our existing body parts, may not give a perfect fit. Customization, or special made for an individual patient, of parts for biomedical implants and/or body part replacements has become more common. Custom-
ized parts have gained increased use instead of standard sized mass-produced parts in areas such as hip, knees and dental [9, 12, 15-18]. If a patient has lost his or her teeth, new teeth are custom made just for a patient's specific needs [19]. Hence, this is time consuming and tedious work performed by technicians, where experience and practice give the technician the skills to produce a dental crown or dental bridge with the needed fit and suitable appearance [20].

By use of AM, the possibility to produce dental replacements in a more cost efficient and effective manner has emerged since the possibility to simultaneously produce differentiated products in a single build has become available [21]. For dental replacements, the level of detailing is of great importance to achieve a suitable fit into the dental implant connections as well as a suitable representation of the esthetics for the individual patient. Therefore, the accuracy of the AM process to produce these replacements is essential. Multiple studies have investigated the possibility to produce dental crowns and bridges using AM [22-24]. Dental crowns and bridges are relatively small in size with dimension in the range of 1-2 cm in height, and wall thicknesses as thin as 0.1-1 mm. At the same time a high level of detailing is needed to model all the features of the patient's own teeth. Therefore the surface resolution is of vital importance for these types of products [25].

Today, production of larger parts often several centimeters in all dimensions are, or are close to be, in serial production utilizing the EBM technology [26]. Examples of such products are acetabular cups for hip joint replacements [26] and turbine blades for aerospace engines [27]. For these types of parts, the overall sizes are large with dimensions of several centimeters where the bulk properties are dominating. These properties are well studied and are found to be similar to conventionally machined products [18, 28-32]. As the parts are scaled down from bulky parts, in dimensions of several centimeters to sub centimeter dimensions, material properties are altered as microstructural and compositional effects come into play. Simultaneously, surface properties will affect the material properties to a larger extent as the ratio between the surface area versus bulk portion will drastically be minimized. It is therefore of vital importance to be aware of the material properties in small sized parts. Nevertheless, AM has the potential to revolutionize the way we design, construct and produce metallic components in the near future, including constructing and manufacturing of shapes and designs that are impossible to produce today with conventional methods [3, 4].

Dental prosthetics treatments are often expensive to buy for the individual patient. The high prices are mainly due to the high demand of manual work and excessive generation of expensive dental prosthetic material waste. By the use of EBM for production of these types of parts, costs can be de-
creased. However, post-processing is still needed due to the poor surface morphology of as-built parts. If the resolution and surface properties for these prosthetics can be improved, the production of dental prosthetics can be streamlined and the cost decreased even further. This will result in an increased availability for this type of treatments for an increased number of patients.
2. Aim of Thesis

Additive manufacturing has been recognized as a revolutionary production technique, but has also received extensive criticism for its poor surface quality, detail resolution and material properties. The work in this thesis aims to provide new insights that could help overcoming these drawbacks during production of small sized components (less than 1 cm in at least one dimension) in the Electron Beam Melting (EBM) process for biomedical applications. This work investigated how EBM process parameters influence different material properties such as mechanical, chemical and surface properties.

Focus has been to use a new type of small sized stock powder material to improve surface resolution and detail resolutions. The function and process parameters of the EBM process have also been investigated in regards of their impact on the finished parts.

In this work the following questions have been addressed:

- Is it feasible to use a smaller sized Ti6Al4V powder in the EBM process and how does it affect the mechanical and chemical properties as well as surface morphology?
- How are the tensile properties affected as the component size decreases below 1 cm in at least one dimension and how does the surface morphology impact the mechanical properties as the components become smaller?
- Will the tensile properties be dependent on the layer-by-layer wise method?
- How is the surface resolution affected when the size of the components decreases below 1 cm in at least one dimension and how can it be improved?
- How does the oxide formation in the EBM process progress and are the oxide properties dependent on parameters such as raw material powder size and build height?

Based on the work of this thesis, the aim is to be able to produce dental crowns and bridges in a more efficient manner, minimizing post-processing, with controlled material properties utilizing the EBM process.
3. Additive Manufacturing (AM)

Additive Manufacturing (AM) is the generic term for technologies usually referred to as Additive Fabrication [4], 3D-printing [33], Freeform Fabrication [34] or Rapid Prototyping [3]. AM is defined by the ASTM International (formerly American Society for Testing and Materials) standard as [35]:

“The process of joining materials to make objects from 3D-model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies, such as traditional machining.”

The main benefit with these technologies is the freedom to construct complex shapes and geometries, where some are impossible to manufacture by conventional manufacturing technologies [36, 37]. Also, the economic benefits by the use of AM for low series production are substantial as customization and single part production are easy to accomplish [38]. The AM technologies were initially used for production of prototypes and visualization models only, but today focus has shifted towards manufacturing and production of finished and functionalized parts, rather than prototyping [4, 39-41]. Today various materials can be used in AM processes i.e. polymers [42], ceramics [43], metals [44] and organic tissues and cells [45, 46]. As of 2013, AM was mainly used in the consumer products and electronics market (21.8 % of the total market), followed by motor vehicles (18.6 %) and biomedical and dental applications (16.4 %) [4]. Examples of parts produced through AM are shown in Figure 1.

![Figure 1. Examples of parts produced by additive manufacturing in both polymeric and metallic materials.](image)

In the late 80’s the first commercial AM system was commercialized by 3D Systems [47]. The first system was a polymer based, so called Stereolithog-
raphy apparatus (SLA) system, that cures an ultraviolet (UV) light sensitive polymer using a laser [48]. Following SLA, Selective Laser Sintering (SLS) was introduced in the beginning of the 1990’s where thermoplastic powders are melted and fused with a high intense laser to produce the finished parts. Also in the same time period the Fused Deposition Modeling (FDM) technology spurred out from the company Stratasys [49]. FDM is the technology that boosted the low end consumer machine market, as it is a simple technique where a polymer filament is melted similar to a glue gun [3, 4, 48]. Apart from the polymeric processes mentioned here, during the 1990’s many other techniques evolved for all kinds of materials, such as plaster, ceramics and waxes. The laser based metal machines were also launched in the 1990’s by multiple vendors that set a new direction for the AM technologies [48, 50].

3.1. Additive manufacturing terminology

The AM technologies have gained acceptance through multiple terminologies for different applications, usage and time. As of 2008 the ASTM committee F-42 was founded to handle the standardization of the emerging technologies. Apart from the terminologies, the committee has worked on standardization for the CAD file format (.amf) used by all machines, to be able to transfer drawings between different vendor’s machines [51]. Also, the material properties of the finished AM products have been in focus for the committee.

The meanings for some various AM terminologies and its technologies are described below:

**Additive Manufacturing** – is defined by the ASTM committee as the word to use for all of the various techniques that build parts in an additive manner from 3D CAD-models [3, 51].

**3D-printing** – refers to the printing technology that was first invented by Massachusetts Institute of Technology (MIT), which is based on ink-jet printing where small droplets of material are printed in layers [33]. 3D-printing has also become a popular word for description of all AM techniques, mostly due to its clear relation to 2D home printing [3].

**Freeform Fabrication** – Refers to the ability for all AM processes to produce complex and advanced geometries and shapes. Many of the shapes and geometries that are possible to produce using AM would be virtually impossible to manufacture by conventional machining.

**Rapid Manufacturing** – This definition relates to the fact that fully functional parts in its finished shapes are possible to produce, where
no post processing is needed [51]. This name can also take into account non-additive based processes, such as high speed machining/cutting for prototype production [21].

In this thesis the terminology Additive Manufacturing (AM) will be used, and will refer to all types of AM production techniques in their general functionality.

The various AM processes can, for process description purposes, be divided into subgroups in respect of their functionality. However, multiple classification systems have been presented and a perfect one is hard to present. Due to the varying amount of classification suggestions, a brief classification is presented to give the reader an idea of what types of AM machines are available.

**Powder bed processes** – Used for both polymers and metallic materials. The powder bed processes fuse a powder based stock material using an energy source. Usually the powder is spread out in a thin layer, and the energy source scans over the unmelted powder and fully melts the powder according to the 2D model layer.

**Polymerization processes** – Polymers are chemically polymerized using either an energy source, such as a laser or other light source, or a chemical inhibitor that is sprayed according to the 2D model layer, to cure the substrate.

**Extrusion Based Processes** – Similar to a heated glue gun, a filament of, usually, polymeric material is melted using heat and extruded through a nozzle. The nozzle moves around a build table in a continuous pattern.

**Printing Processes** – A nozzle prints, or sprays droplets, of either a liquid material that later hardens or a binder that consolidates a substrate. Similar to ink-jet printing used for home printers.

### 3.2. The additive manufacturing production cycle

From the ATSM definition it is stated that additive manufacturing technologies function in the opposite way compared to conventional machining. Parts are built when thin cross-section layers of material are added on top of each other to grow the parts according to a 3D CAD-model, rather than removing material [1]. Although the material or process used may vary, the steps to move from the 3D CAD drawing to a finished part are similar for all types of techniques. The different process steps are visualized in Figure 2 and described below. The description is a simplification based on Gibson et. al. [3] and Gebhardt [51] unless stated otherwise.
1. The first step in an AM process is to draw the part to be built in a CAD program to generate a 3D model. This 3D model is the basis for the production of the part. Internal features and other shapes can be designed directly in this step; even moving parts in the finished component can be directly created.

2. The 3D CAD-model is converted into a file of .stl, or the upcoming .amf, format. The .stl format converts all external closed surfaces of the model into an array of triangles [35].

3. The array of triangles is the base for the next step, where the 3D-model is sliced into thin 2D layers stacked on top of each other. During the slicing a build file is generated from the manufacturer’s specific program that is later transferred to the AM machine.

4. After the machine is setup, the build process is taking place as material is added in thin layers according to the discrete layers generated in the build file. A detailed description of the EBM build process is found in chapter 4.

5. As the build process is finished, the part is removed and to some extent post processed. The amount and the method of post processing vary depending on the AM process that is used. A common post processing method is removal of supports. Support struts are usually used for many of the AM processes for reasons such as gravity support when building overhangs and for heat transportation.

*Figure 2. A visual representation of the basic additive manufacturing principle.*
The freedom of design is often highlighted as an advantage for AM processes, and multiple examples of complex and abstract geometries have been presented [52] (see also Figure 1). So-called 3D lattice structures can be produced using multiple AM techniques [53]. These lattice structures feature low density, with almost intact mechanical properties, where the rigidity can be tailored to fit specific demands [36, 54-56]. To make full benefit of these advantages with the AM techniques, it is important for the designer and constructor to learn and understand these possibilities. The constraints that usually exist for conventional machining such as, i.e. tool diameters and fit, or release angles for injection molding, do not apply in the same manner for AM and have to be learned by the designer [4, 56]. Some constraints affecting the AM processes are the layer thickness and the size of the stock material used, i.e. powder grain size. Dependent on process, other constraints are nozzle diameter, ink-jet droplet size and/or energy source spot size [3, 41, 56].

3.3. Benefits and drawbacks of additive manufacturing

Hederick described that AM can be applied as a manufacturing technology for parts that cannot be manufactured by conventional machining and this is considered to be a major benefit of the AM processes [57]. Other suggested benefits have been summarized by Holmström et. al. [58], and Grote and Antonsson [21], and are presented below:

- No tooling is needed, significantly reducing production ramp-up time and expense.
- Small production batches are economically feasible.
- Possibility to quickly change design.
- Allows products to be optimized for function (for example optimized cooling channels).
- Possibility to reduce waste.
- Potential for simpler supply chains; shorter lead times, lower inventories.
- Design customization.
- Manufacturing efficiency possibilities as multiple individual parts can simultaneously be produced.

Dimensional accuracy and poor surface finish are, however, drawbacks for many of the AM-processes. The stair stepping effect is a natural phenomenon in all AM-processes and refers to the stair case shape caused when a curved shape is built. When layers are deposited on top of each other, the layer resolution will shape the surface as stair steps similar to low resolution pixels in a circular image. This causes some of the problems regarding the poor surface finish, and in combination with other factors, is responsible for
the poor dimensional accuracy [18, 59]. Today, only the layer height and melt pool temperature are controlled to solve this problem in the literature [3, 59].

Material properties of the materials produced in AM processes usually have inferior mechanical, electrical and thermal properties compared to conventionally machined parts [60]. For metallic AM parts however, material properties similar or better than for conventional machining have been reported [30, 61].

Another drawback with AM processes is the speed of part production, since tall parts require multiple layers to be fused on top of each other, which is costly and time consuming. The cost for a single unit, using mass production technologies, i.e. injection molding, will become lower as the number of identical units increases. For the example of injection molding, the tool cost per unit decreases as the total number of produced parts increase [62]. AM on the other hand will have an almost constant cost regardless of the number of units produced. For single unit production, the AM will be more advantageous [4]. See Figure 3, as a representation.

Another drawback that should be considered is the lack of feedback loop in many of the AM processes today. Since the material is dependent on build history, the detection of defects is of great importance and is extensively researched for all types of AM processes [39, 44].
3.4. Examples of applications for additive manufacturing

AM has from its benefits found use in a number of different applications. Today many of them are short series units, with high level of complexity, and/or a high level of customization.

3.4.1. Biomedical

AM techniques are beneficial for production of hard tissue replacements. The main advantages are the possibilities to customize the implant to the specific patient. Parts can be customized at almost no additional cost. Also the possibility to manufacture lattice structures is advantageous as the mechanical strength can be designed to mimic the bone strength, and therefore minimize stress shielding [12, 16, 63-65].

By the use of computer tomography (CT) and other imaging techniques, CAD models for AM production are easy to produce and customized parts can be additively manufactured from these scan data [7, 66, 67].

3.4.1.1. Dental

Dental prosthetics, such as dental crowns and bridges, braces and aligners are customized for the specific patient. By the use of AM, these types of products may be manufactured in a cost efficient manner as multiple patients wearables may be produced in the same build, each individual completely unique. The manufacturing of these kinds of products has moved more and more into serial production [58]. This is done both for polymeric and metallic parts [68-70].

3.4.2. Aerospace

In the aerospace sector weight savings and structural integrity are of essence. By the use of AM, new types of shapes, i.e. organic shaped parts or lattice structures, makes it possible to save weight as material is put only where needed. Also, the aerospace industry often uses expensive and hard to produce material where the buy-to-fly ratio becomes high as materials are traditionally removed. By the use of AM, these materials can be more efficiently processed [4, 8, 27, 38, 71].

3.4.3. Rapid tooling

Rapid tooling is the term where tools, for example for injection molding, use the benefits of AM. The advantages to produce these tools through AM are the fast production of the tools, where the design can easily be iterated. Also,
the possibility to produce complex surface shapes and complex cooling channels has become great a benefit by the use of AM for rapid tooling production [72].
4. Electron Beam Melting, EBM

Electron Beam Melting, shortened (EBM) is an additive manufacturing (AM) technology that was developed at Chalmers University of Technology in the late 1990’s and commercialized by the Swedish company Arcam AB in the early 2000’s [3, 73]. Fully dense metallic parts are built up from thin layers of powder particles that are fused together utilizing a high-intensity electron beam. The beam scans and fully melts the powder according to the 3D CAD-drawing. The EBM technology is similar to the laser based AM processes, but with the difference that an electron beam (e-beam) is used instead of a photon based laser as energy source to fuse the powder particles [74]. EBM as a process has found extensive use for multiple types of metallic materials such as titanium–6aluminum–4vanadium (Ti6Al4V) [7], titanium-48aluminum-2chrome-2niobium (Ti–48Al–2Cr–2Nb) [27], CoCr alloys [9] and H13 steel [10]. Today the EBM technique is used to produce, among other applications, customized titanium biomedical products with unique geometrical features [11]. In Figure 4, examples of EBM manufactured parts are presented.

Figure 4. Examples of parts manufactured by Electron Beam Melting (EBM) in titanium.

The most widely used material in the EBM process is the Ti6Al4V alloy, where optimized process parameters are available and extensive research has been conducted [1, 5]. The mechanical properties are as good as, and to some extent superior compared to a cast and/or wrought counterpart. The microstructure is also similar to a wrought material, with the traditional basket-weave like Widmanstätten structure found in rapidly quenched Ti6Al4V. Although, the grain size is usually seen to be somewhat smaller in size in the EBM produced material compared to traditionally produced material. [18, 28-30, 32, 75-79].
4.1. Electron Beam Melting technology

A schematic illustration of the EBM process including all vital parts of an EBM machine is presented in Figure 5. The following description of the EBM process is information based on Gibson et. al. [3] and material from Arcam’s Level 1 and 2 courses.

![Schematic of the EBM process](image)

**Figure 5.** Schematic of the EBM process including descriptions and text reference numbers.

The major difference between the EBM process and the various other powder bed laser fusion processes is the use of an e-beam instead of a laser to melt the stock powders. The overall EBM process is partly similar to that of
a scanning electron microscope (SEM). In similarity to an SEM, the e-beam is generated in the electron gun (1). A tungsten filament is placed in a grid-cup, or anode. A current is applied to the filament and the tungsten strand is heated to 2200-2700 K. The electrons are collimated and accelerated, by an electric field between the filament (cathode) and the anode, to an energy of 60 keV and an e-beam is generated that is running down the so called drift tube.

In the drift tube magnetic lenses are located. The first lens is to correct for astigmatism (2) and to generate a circular e-beam with a Gaussian energy distribution. The second lens is the focus lens (3) which focuses the beam into a small 0.1 mm spot. Finally, the deflection lens (4) scans the e-beam across the build area.

In the build chamber (5) the build process is taking place. Inside the build chamber two powder hoppers (6) are placed. The hoppers hold the powder stock material that is used in the process.

Below the hoppers is the build table (7). The build table is the area where powder is spread over by the rake (8). The rake fetches powder from the hoppers and scrapes the powder over the build table. In the middle of the build table the build tank (9) is located. Inside the build tank, the build platform (10) moves down the z-axis as the build progresses. The actual build area, where the beam melts the powder, is on top of the build tank in the same height as the build table. See also all parts of the build tank in Figure 6.
The whole EBM build system, build tank and e-beam column, is under vacuum during the process. Vacuum is needed since electrons interact with the gaseous atoms, if present, and will be deflected. The use of vacuum is however advantageous for the process, since the vacuum will prevent reactions between reactive metals, e.g. titanium, with atmospheric gases, such as oxygen. Also the vacuum acts an insulator in helping to keep the process temperature at an elevated temperature.

The EBM process is run at an elevated temperature. Due to the high power of the e-beam (up to 3 kW) and the rapid movement of the beam, the beam is scanned rapidly in a defocused mode over the powder surface to keep an elevated temperature. The elevated build temperature will help to prevent residual stress build up in the finished part. Also, the elevated temperature will lightly sinter the powder particles surrounding the fully melted finished part.

A slightly sintered powder will help to increase the conductivity between the individual powder grains. There is always a risk of so-called “smoke” when running the EBM process. Due to the negative charge of the electron that
bombards the surface, the powder bed will be charged up if inferior conduction exists between the powder particles and the ground. If the charge-up becomes too high and the negative repulsing forces between the powder particles will become higher than the gravitational force and friction forces holding them in place, the powders will create a powder cloud inside the chamber, “smoke”. Another effect of the negative charge buildup is the risk of e-beam deflection, resulting in a decrease in beam accuracy and spot spreading.

4.1.1. Process description

A build in the EBM process starts with pumping down the build chamber to a vacuum of approximately $10^{-5}$ mbar. As vacuum is created, the heating of a stainless-steel plate, so called start plate, using the e-beam takes place. The beam is rapidly scanned across the start plate in a highly defocused mode. As the temperature reaches 600-750 °C the build table is lowered a distance of usually between 100 and 50 µm, dependent on the operator setting. The rake spreads a layer of powder over the build table. The newly laid powder layer is heated to 80 % of the melting temperature [74] and slightly sintered using a defocused beam that raster over the build area at speeds between 14 700 and 25 000 mm/s with currents between 10 and 40 mA.

Following sintering, or so called pre-heating (PH), the contours of the present 2D-layer are melted using a so called MultiBeam™ melting strategy. During MultiBeam™ melting the electron beam is deflected instantaneously and in a discontinuous pattern between individual points along the contour, creating the illusion of multiple spots simultaneously melting the contour. The use of magnets to control the beam offers the possibility to move the beam instantaneously between adjacent points in the build chamber. A fully melted contour is created as multiple points will be fused together. The individual point melting during MultiBeam™ will enhance the surface morphology.

As the contours are finished, the beam melts the bulk, or inner part, inside the contours using a so-called hatch melting. During hatch melting the beam, with a high focus, raster in a snake shaped pattern back and forward to fill the contours. As a single layer is melted, some additional heating might be needed to keep the build temperature at a constant elevated temperature.

After a layer has been melted according to the strategy described above, the process starts over to create a new layer and the build table is lowered. This process repeats several times to add the required amount of layers to complete the build. This gives a building rate of approximately 3-6 mm in height per hour [77].
As the build is finished the build chamber is cooled from the service temperature to 100°C in helium at 400 mbar, followed by cooling in air. Slightly sintered particles will surround the fully solid and melted part. This excess powder will be removed using the so called Powder Recovery System (PRS). In the PRS, the same powder as used for the build is used to blast away the sintered powder surrounding the finished part. All powder blasted off from the part is recycled into the process once again and the material loss is minimal.

4.1.1.1. Key process parameters
The EBM machines hold over 100 process parameters that the experienced operator may adjust. Some of the key process parameters are briefly described below.

EBM produced surfaces are reported to have a quite rough morphology, containing a rippled structure with visible sintered powder grains [80]. For products where a smooth surface is essential, the as-produced surface roughness resulting from EBM may be unfavorable. Also, when producing smaller components (<1 cm) with high levels of detailing the accuracy of the product dimensions will be affected and details might be lost [81]. Since EBM uses fused layers stacked on top of each other to buildup parts, the layer thickness influences the resolution of the build as described by [81]. Early versions of EBM process equipment used 100 µm as the standard layer thickness. The current standard layer thickness has been reduced to 50–70 µm. A powder particle size of 45-100 µm is currently used [5]. For Selective Laser Melting (SLM), which is an AM process that uses a laser as the energy source, a finer surface morphology can currently be obtained than with EBM. Yasa et al. have studied the appearance of parts manufactured by laser [82] where Murr et al. have compared surfaces from both EBM and laser manufactured parts [18]. In SLM a thinner layer thickness compared to EBM, normally 20-30 µm is used [50] together with a powder size of 25-45 µm [83]. Layer thicknesses down to 2 µm have, however, been reported [40]. For SLM, the use of powder sizes as small as 1-10 µm has also been reported [84]. Gibson et al. [3] have stated that the use of powders with smaller sizes will make it possible to use a thinner layer thickness and thereby obtain a finer surface morphology.

The reason for the rougher surface morphology in the EBM process is that it is also affected by the spot size [85]. The spot size is in turn affected by the functionality of the electron gun and the magnetic coils that shape the e-beam [86]. The size of the e-beam will also be affected by the negative charge build up that is taking place in the process as described in section 4.1.
4.2. Applications

Today the two main application areas of EBM are medical devices and aerospace. These applications demand a high level of complexity and customization. Titanium is frequently used for products within these areas. Some examples of parts for these applications manufactured by EBM can be seen in Figure 4.

4.2.1. Medical devices

Customized medical devices by the EBM system have been extensively researched and today acetabular cups for hip joint replacement are in serial production utilizing the EBM process. EBM porous structures have been tested in animals for biocompatibility by Palmqvist et. al. with promising results [87]. A similar porous structure is used on some acetabular cups manufactured with EBM and is seen as an advantage compared to conventionally machined hip joints.

EBM has also been utilized for manufacturing of dental replacements, such as a full set of teeth, including roots, and wearables as crowns and bridges [24, 70, 80, 88].
Dental prosthetics, also named dental restorations, are used for dental restorations where patients have lost their own teeth. Common dental replacements are crowns, bridges or dentures [89, 90] produced mainly from three different groups of materials; metals, plastics and silicates [91]. Metals used are, e.g., titanium (Ti), gold (Au), cobalt-chrome alloys (CoCr) and platinum (Pt) [91]. Many patients are reluctant to use dentures, whereas fixed dental replacements, such as anchored crowns are bridges, have become popular [89]. These replacements consist of a complete system of multiple components and include oral implants, abutment and dental prosthetics [89]. A schematic figure of a dental replacement system is presented in Figure 7.

*Figure 7. Schematic illustration of a dental implant system and the surrounding healthy teeth and bone showing, 1) dental implant, 2) abutment, 3) dental prosthetic, 4) surrounding healthy bone, 5) mucosa, and 6) healthy teeth.*
Dental implants (lower screw shaped part (1) in Figure 7) are commonly produced by machining of titanium-6aluminum-4vanadium (Ti6Al4V) rods [92]. These types of implants can be implanted in both the upper and lower jaw bones. A benefit with these types of implants is that they support the prosthetics (3) without sacrificing healthy teeth (6). Due to osseointegration, the dental implants have a strong attachment to the supporting bone (4) [93]. As the implant is located in the bone underneath the mucosa (5) in the oral cavity, an abutment (flat middle metal part penetrating the mucosa (2) in Figure 7) is used to distance the dental prosthetics through the mucosa [89].

Dental prosthetics (3) is the visible part of the dental replacement system and is designed to mimic the patient’s own teeth. Since the prosthetics are fastened in the abutments and supposed to mimic teeth, high detailing and excellent fit are important factors [89]. When the prosthetics are manufactured out of titanium, porcelain or composite polymers are applied as coatings to mimic teeth and cover the grey metallic color of titanium. However, due to the thermal coefficient expansion and the oxidation properties of titanium, adhesion of these materials to the titanium surface may be difficult [94, 95]. The adhesion is also affected by the surface roughness. Titanium is a frequently used material for dental prosthetics due to its favorable chemical properties and high strength to weight ratio [89]. The dental prosthetics will be load bearing during, e.g., chewing, wherefore mechanical properties are important. Maximum bite forces of 200 to 1500 N have to be considered when designing dental prosthetics [20].

### 5.1. Manufacturing dental prosthetics using additive manufacturing

By the use of AM, titanium dental prosthetics can be produced in a cost efficient and effective manner [22, 96]. Due to the complex geometry, low volume, strong individualization and high aggregate prices of dental prosthetics, AM technologies are suitable for the direct manufacturing of these components [19]. Instead of conventional processes with multiple labor intensive process steps, a highly automated process can be obtained through AM. In Figure 8, a comparison of the process steps for dental prosthetics production, using both AM and traditional manufacturing processes, is presented.

In the AM process, the first step is to create a digital file of the patient’s teeth. This is made either by a hand scanner, directly scanning the patient’s teeth, or by scanning an impression taken by the dentist [97]. The digital file is imported along with multiple other files into the AM process (preferably EBM or SLM) and numerous individual customized prosthetics are simulta-
neously manufactured [19, 20]. An example of a digital file and the finished AM produced prosthetic is presented in Figure 9.

**Figure 8.** Visualization of the various process steps for dental prosthetics manufacturing with either casting, CAD/CAM or Additive manufacturing.

Production of dental prosthetics from titanium, using traditional processes, is usually done through high-speed milling, often referred to as CAD/CAM methods [20]. During this process, a digital CAD-file is created from a plastic mockup of the patient’s teeth. From the digital file, the prosthetics is produced by subtracting material from a titanium slab [98]. This procedure is time consuming and costly since the production times can be long and the generation of scrap material substantial.

Another method of producing dental prosthetics in titanium is through casting. From an impression of the patient’s teeth, a technician is creating a model by hand in a wax material. From the wax model a cast mold is formed and the final prosthetics is casted from liquid titanium. The cast procedure is a tedious process that requires skilled technicians to secure a suitable fit of the prosthetics to the fixed implants [20]. Also, the material properties must be considered since titanium is hard to cast due to its reactivity with atmospheric gases such oxygen and nitrogen, as well as the mold materials.
As seen in Figure 8, the process steps for production by the use of AM will be much more effective compared to the traditional methods described above.

*Figure 9.* Example of a digital model of a dental prosthetics (left) and the additively manufactured counterpart (right).
Titanium and titanium alloys have become important as structural materials. Applications such as medical technology, chemical processing, sports, leisure, marine, and aerospace all make use of the advantageous combination of properties of titanium materials [13]. Compared to other metals, titanium and titanium alloys exhibit high corrosion resistance and high specific strength-to-weight ratio [99]. Today the most widely used titanium alloy is Ti6Al4V [100]. This alloy has a balanced combination of mechanical properties and workability and has been extensively researched [101]. Due to a relatively low thermal conductivity, titanium and its alloys are somewhat complicated to process through traditional methods such as milling and turning [102].

Ti6Al4V is a so called two-phase material, consisting of the hexagonal close packed (hcp) α phase and the body center cubic (bcc) β phase [102]. The transition temperature between the two phases for Ti6Al4V is 995 °C [103, 104].

The mechanical properties of the two-phase Ti6Al4V alloy are dependent on the microstructure and the distribution of the two phases throughout the material [103, 105]. The required properties of wrought Ti6Al4V for surgical implant applications as standardized by ASTM 1472 are summarized in Table 1 [106].

Table 1. Summarized key properties of wrought Ti-6Al-4V, based on [106].

<table>
<thead>
<tr>
<th>Elemental composition</th>
<th>wt [%]</th>
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<tr>
<td>Titanium</td>
<td>Balance</td>
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<tr>
<td>Aluminum</td>
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<tr>
<td>Vanadium</td>
<td>3,5-4,5</td>
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<td>Iron</td>
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<tr>
<td>Oxygen</td>
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<tr>
<td>Nitrogen</td>
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<tr>
<td>Hydrogen</td>
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<table>
<thead>
<tr>
<th>Mechanical Properties</th>
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<tr>
<td>Tensile strength</td>
<td>930 [MPa]</td>
</tr>
<tr>
<td>Yield strength</td>
<td>860 [MPa]</td>
</tr>
<tr>
<td>Elongation</td>
<td>10 [%]</td>
</tr>
</tbody>
</table>
6.1. Titanium surface oxide

The high corrosion resistance of titanium is due to a dense surface oxide layer that protects the underlying metal from further reactions, such as corrosion [107-109]. Due to high reactivity, metallic titanium will, in just microseconds, form a protective oxide layer, usually 2-7 nm thick, when exposed to atmospheric or other oxidizing conditions [110-114]. The composition and structure of a titanium surface are important factors that influence the biological response and are therefore especially important for its use in biomedical applications. Material degradation in body environment and adverse effects on bone and soft tissues are usually low with titanium due to the surface oxide properties.

At higher temperatures, above 400 °C, the oxidation growth rate will increase drastically [102]. Since oxide growth is controlled by thermodynamics and kinetics an increased temperature will increase the oxidation growth rate. Simultaneously the diffusion rate of oxygen within the material will increase as the temperature becomes higher [13]. The kinetics of oxide growth is described in Figure 10.

Figure 10. Oxidation of titanium: a) Adsorption of oxygen at the surface. b) oxygen dissociation. c) titanium/oxygen reaction to form titanium oxide. d) oxide growth through diffusion. Based on [115].

The first step (a) is adsorption of the atmospheric oxygen or water molecules to the pure metallic titanium surface. Once on the surface, the oxygen and/or water must dissociate to atomic oxygen (b) for the reaction to start. On pure titanium, the dissociation of oxygen and/or water molecules progresses extremely fast. Following dissociation, the reaction between the titanium and atomic oxygen (Ti+O) occurs (c), and a monolayer of titanium oxide is formed quickly. This step is controlled by thermodynamics and driven by the minimization of the free energy for the Ti+O reaction. As the monolayer has been formed, further oxide growth takes place through diffusion of oxygen.
atoms to the pure titanium subsurface, and/or through diffusion of titanium metal ions to the surface (d). The rate of titanium oxide growth is limited to these diffusion rates, which in turn are dependent on temperature.

6.2. Osseointegration of titanium

Osseointegration is the term describing the ability for bone tissue to grow into direct contact (on the light microscopy level) with a foreign material [116]. For titanium, this is manifested as a stable long term anchorage of the implant, resulting in fracture in the surrounding bone upon removal of the implant [117]. This favorable property is considered to be, at least partly, due to the surface oxide present on titanium materials [118, 119]. Therefore, titanium surface properties are important for the osseointegration process and this process can be expected to be affected by the specific properties of the surface oxide layer [120, 121]. To enhance the osseointegration of titanium, several studies have reported on surface modification of titanium surfaces, for example addressing surface roughness [122], both on the micrometer and nanometer scale [123, 124], surface chemical composition and surface oxide thickness [125], and surface coatings such as hydroxyapatite [126]. Such treatments of the titanium surface before insertion and usage as an implant are claimed to be beneficial for controlling the surface characteristics and the biocompatibility of the material [127].

6.3. Titanium powder

Metallic powders are commercially produced through chemical electrolytic, mechanical or atomization methodologies. For production of titanium powder, atomization processes are the most common to use [128]. All atomization processes have the basic functionality to melt a base metal which forms spherical droplets, solidifying into powder [128]. There are several different atomization processes, where two are described briefly here.

In gas atomization a stream of gas is used to break up melted metal into spherical droplets. For titanium production, an inert gas such as argon must be used due to the reactivity of the titanium [128].

Plasma rotating electrode process (PREP) is another atomization process for powder production. In the PREP procedure, a metal, in this case titanium, rod is melted in one end using a plasma arc. The rod is spun and by the centrifugal forces, spherical titanium droplets are thrown out to the sides and collected [128].
For accurate size distribution of the powder grains, sieving is performed to separate the various powder fractions created [128].
7. Materials and Methods

The work performed and described in this thesis is aimed to gain knowledge that can be used for optimizing the manufacturing of small titanium components in general, and dental restorations in particular, using the Electron Beam Melting (EBM) technology. To optimize the production of small components, a smaller sized powder stock material has been utilized, characterized and compared to the standard sized powder stock material normally used for EBM production. To increase the understanding of the EBM process and the resulting material properties, the process characteristics and their correlation to the resulting material properties have been studied. Work has been focused on the material properties achieved when producing small components. The techniques used to investigate these properties are described in the sections below.

7.1. Sample manufacturing

All samples and powders used in this work are made from titanium-6aluminum-4vanadium alloy (Ti6Al4V). For the manufacturing of samples, pre-alloyed Ti6Al4V powders of two different size fractions were used. The first powder used was the standard powder, which is regularly used in the EBM process today. This powder is gas-atomized and has a fraction size of 45-105 µm. The second powder is a plasma rotating electrode processed (PREP) powder and is finer with a fraction size of 25-45 µm. Scanning electron microscopy (SEM) images of the two powders are presented in Figure 11. In papers I, II, III and IV, a small sized titanium powder was used for the purpose to decrease the surface roughness and improve surface detailing.
Figure 11. Scanning electron microscopy micrographs of the two powders used for sample production. a) standard 45-105 µm powder and, b) finer 25-45 µm powder.

All manufacturing in this work was performed in Arcam A1, A2 or S12 machines. The key process parameters used are summarized in Table 2. More details of the process parameters regarding melt strategy are presented in the corresponding papers.

Table 2. Summary of the key process parameters used for manufacturing of samples.

<table>
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7.2. Analysis techniques

To analyze the built parts and the powder stock material various analysis techniques were used. Since focus has been on surface properties, many of the techniques are used for characterization of various surface properties. Also mechanical properties have been studied on small components. Short descriptions of the techniques used are presented below.

7.2.1. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) uses the interaction between electrons and the sample material to form images of the sample surface. In SEM, a focused beam of electrons is generated through extraction of electrons either by thermal emission from a filament or by field emission from a sharp tip. The electrons are normally accelerated to energies between 1 and 30 keV. The electrons will interact with the sample atoms, leading to emission of secondary electrons, which are detected by a secondary electron detector. The intensity of secondary electrons is dependent on the surface topography (and also to material composition), so by recording the secondary electron intensity from each spot of the scanned area, information about the topography (and material composition) is obtained. Since the electron beam can be focused to very fine diameters (~1 nm), high-resolution images can be recorded [86].

SEM was used extensively for visual inspections of sample appearances and morphology in all papers I-V. SEM was also used for microstructural investigations in papers I & II. In paper III & V SEM was used to study the fracture surfaces.

7.2.2. Surface chemical analysis

When changing the powder in the EBM process from the standard 45-105 µm sized powder to the finer 25-45 µm powder, it is important to be aware of the surface chemical compositions. Specifically for use in biomedical applications, where the surfaces will come into contact with human tissues, the understanding of surface properties, and the surface oxide layer in particular, is of importance. Therefore an understanding of the formation and properties of the surface oxide layer formed in the EBM is needed. Various surface sensitive techniques were used in this work to study the surface chemistry and surface properties of various EBM manufactured parts.

7.2.2.1. X-ray photoelectron spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS) irradiates the sample surface with a focused beam of X-rays. The X-rays will result in emission of electrons, so-
called photoelectrons from the outermost 1-10 nm of the material. The photoelectrons will be emitted with different kinetic energies, which in turn reflect different binding energies of the atoms they originate from. The XPS spectrum therefore contains peaks at binding energies representing the different elements present at the surface, where the intensity of each peak reflects the concentration of the element within the analyzed volume. Since the binding energy is slightly shifted depending on the chemical state of the element, information can also be obtained about e.g. oxidation state of elements or functional groups present in organic materials [129].

XPS was used in paper I to detect the variation in surface chemical composition between the parts produced by the two different powders.

7.2.2.2. Auger electron spectroscopy (AES)
Auger Electron Spectroscopy (AES) uses a focused electron beam to irradiate on a sample surface. From the sample surface, so called Auger electrons are emitted from the outermost 1-10 nm of the material due to a series of electronic transitions within an atom. The kinetic energy of the emitted Auger electrons is element specific, so the kinetic energy spectrum will contain peaks at specific energies representing the elements present at the surface. Like for XPS, the intensity of the peaks reflects the concentrations of the elements. AES therefore provides information about the elemental composition of the surface. By scanning the electron beam over the surface, element specific maps, showing the lateral distribution of elements within the scanned area can also be obtained. AES can also be used for depth profiling, in which an ion beam is used to etch (sputter) away material from the surface, providing information about the depth distribution of elements in the material [130].

AES was used in paper V to measure the thickness of the surface oxide layer in EBM produced parts, by depth profiling. Due to the appearance of EBM surfaces, where both fully melted and partially melted powder grains may be present, AES was useful for point analysis of various sample points.

7.2.2.3. Time-of-flight secondary ion mass spectrometry (ToF-SIMS)
Time-of-flight secondary ion mass spectrometry (ToF-SIMS) is a surface sensitive technique that gives information regarding the chemical composition in the top 1-10 nm of a surface. Ions are used to bombard the sample surface, where secondary ions of the sample material are emitted from the surface. The secondary ions are collected and analyzed in a time-of-flight mass spectrometer, where the ions are separated in regards of their mass. From the measurements a mass spectrum is acquired that represents a “fingerprint” of the chemical composition of the analysis area. A second ion source can also be used to continuously etch (sputter) away the outermost
surface during the measurement, to record depth profiles representing the distribution of different elements from the surface and into the material. The drawback with ToF-SIMS is the difficulty to measure quantitative values of the composition present. However, for relative comparisons the technique is very useful [131].

ToF-SIMS was used in paper I and V to evaluate the surface oxide thickness of different samples. During the analysis, depth profiles of titanium oxide (TiO⁺) secondary ions from the top 1-50 nm of the surface were measured.

### 7.2.2.4. Transmission electron microscopy (TEM)

In similarity to SEM, Transmission Electron Microscopy (TEM) uses electrons as an information source. The main difference between SEM and TEM is that the electrons are transmitted through the sample in TEM, since the sample is made very thin. During analysis, the electrons will interact with the sample and collect information as they pass through the sample. Examples of information acquired in TEM are high resolution images at nanometer resolution, elemental and compositional information, and crystal structure information [132].

In this work, TEM was used to study the surface oxide layer present on an EBM manufactured surface. High resolution images and electron energy loss spectroscopy (EELS) maps were acquired to measure the thickness of the surface oxide layer. The results are presented in paper IV.

### 7.2.3. Surface morphology measurement

Surface roughness has been an issue for effective usage of small components manufactured by EBM, in particular for the dental prosthetics where detail resolution and adhesion are key properties. Apart from the surface sensitive techniques used to investigate the surface chemistry of EBM produced components, various techniques were used to measure the surface roughness of EBM samples made with different powders and layer thicknesses.

#### 7.2.3.1. Focus variation profilometry

For focus variation profilometry, multiple images are taken of the same surface area with different focal heights. In the optical lens, the depth of focus is short and multiple images with different focus depth are taken to have all heights of the sample in focus. The images are stacked together and a 3D representation of the surface is generated. From the 3D reproduction, lines of the surface profile are taken from where the surface roughness values, such as $R_a$, are calculated. A 3D representation of an EBM surface by this method is seen in Figure 12. $R_a$ is a one dimensional measure of the arithmetic aver-
age (absolute values) of the surface roughness variations from a simulated straight base line over the surface.

![Image](image.png)

**Figure 12.** Representative 3D generation from focus variation profilometry of an EBM manufactured surface. The surface profile in the lower left corresponds to the line in the top of the 3D image.

This technique was used in *paper IV* as one of the methods to measure the impact of wall thickness, powder size and build layer thickness on the surface roughness $R_a$ values of EBM surfaces.

### 7.2.3.2. White light interferometry

This non-contact method uses white light to measure the surface roughness by interferometry. As white light is irradiating the surface, the reflected light waves will be combined with the primary light wave and the two light waves will go through interference as they have the same phase. The differences in amplitude will, by software, generate a 3D representation of the surface from where surface roughness values may be calculated. A 3D representation of an EBM surface recorded by this method is seen in **Figure 13.**
This technique was used to evaluate the impact of wall thickness, powder size and build layer thickness surface structure on various EBM produced surfaces in *paper IV*.

### 7.2.3.3. Stylus profilometry

Stylus profilometry is a contact measurement technique to measure the surface structure of a sample. A small tip stylus is in contact with the surface and mechanically follows the contours of the surface to measure the surface morphology.

Stylus profilometry was used in *paper IV* to investigate the impact of wall thickness, powder size and build layer thickness on surface roughness of EBM produced parts.

### 7.2.4. Inductively coupled plasma – optical emission spectroscopy (ICP-OES)

Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES) is an analysis method that gives information regarding the quantity of different elements in a solution. For a metallic sample the sample is first dissolved using various reagents. The dissolved solution is sprayed as a mist into a plasma. The plasma is generated by flowing argon gas through an electromagnetic field. As the solution droplets pass through the plasma the solvent evaporates and the metal atoms become ionized and electronically excited. De-excitation of the ions leads to the emission of element-specific light wavelengths. As the light intensities are related to the concentration of re-
spective element, measurement of the emitted light can be used to quantitatively measure elemental composition [133].

ICP-OES was used in *paper I* to quantitatively characterize the bulk metal composition of both the two stock material powders and the components built from these two powders.

### 7.2.5. Tensile test

In tensile tests, a sample is put under load to test its tensile durability. During testing, a constant displacement speed is applied on the sample and the load and elongation of the sample are measured. Numerous information values are extracted from this measurement, for example the ultimate tensile strength, which represents the maximum load, irrespective of the sample size, a material can take. Other values are the yield strength, which gives information on what load a material can take in its elastic condition, the amount of total energy the material can withstand and the material stiffness (Young’s modulus).

In this work, tensile properties were investigated in *paper II*. The effect of component size and surface morphology on mechanical properties was studied. The tensile properties were also investigated in *paper III* with addition of digital image correlation (described below) measurements to investigate the integrity of EBM produced material in regards of the layer-by-layer dependency of the EBM process.

### 7.2.6. Nanoindentation

A nanosized tip is pointed at a polished sample and a force is applied. From the load put on the nanosized tip and the displacement the sample renders, information about material hardness and material stiffness is obtained.

This technique was used in *paper I* as a method to compare mechanical properties between the samples built with various build layer thicknesses and powders.

### 7.2.7. Digital image correlation (DIC)

Digital image correlation (DIC) is an analysis technique used in conjunction with tensile tests. The technique is an optical measurement technique that uses algorithms to track shifts in image datasets. The specific technique used in this thesis is used to track local strain changes by following facet packages during tensile testing in comparison to the unloaded condition [134, 135].
This technique was used in paper III to find the layer-by-layer dependency of local strain fields in EBM produced material. For a first analysis, two digital cameras were aimed at the tensile bar to track the facet movement of the full tensile bar. The resolution was approximately 340 µm. In a second test, an in-house tensile stage was constructed for micro level investigation. Here, a 5 X microscopic lens was used in conjunction with a camera and the microstructure was used as the facet pattern. An area of approximately 2x2 mm$^2$ was analyzed where the resolution was increased to approximately 23 µm. A schematic representation of this setup is presented in Figure 14.
8. Results and Discussion

8.1. Feasibility study of fine powder in Electron Beam Melting (EBM) - Paper I

Important objectives of this study were to explore the feasibility of using a finer stock powder material and to investigate possible differences in EBM built materials, as compared to materials built from a standard stock powder material. Blocks of fully consolidated titanium-6aluminum-4vanadium (Ti6Al4V) were produced with both the standard 45-105 µm (referred to in the coming text as standard powder) and the finer 25-45 µm powder (referred in the coming text as finer powder). For production of blocks with the finer powder some modifications in the Electron Beam Melting (EBM) process were necessary. The process parameters were changed, mainly with regard to pre-heating parameters such as beam speed, focus offset and beam current. The melt parameters were only corrected for the layer thickness as the current was lowered.

To compare the bulk metal composition of the standard powder with the finer powder and parts produced by the respective powder, inductively coupled plasma optical emission spectroscopy (ICPS-OES) was used. The results from ICP-OES measurements are presented in Table 3.

Table 3. ICP-OES measurements of titanium (Ti), aluminum (Al), vanadium (V) and iron (Fe) for the standard and fine powder and their respective builds. All values are atomic % and mean values of two measurements. ASTM F-1472 specifications are added for comparison [106]

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>45-105 µm</td>
<td>89.01</td>
<td>6.78</td>
<td>4.12</td>
<td>0.09</td>
</tr>
<tr>
<td>25-45 µm</td>
<td>90.12</td>
<td>5.84</td>
<td>3.72</td>
<td>0.08</td>
</tr>
<tr>
<td>Build 1a</td>
<td>89.73</td>
<td>6.09</td>
<td>4.09</td>
<td>0.09</td>
</tr>
<tr>
<td>Build 2b</td>
<td>89.79</td>
<td>5.95</td>
<td>4.14</td>
<td>0.12</td>
</tr>
<tr>
<td>Build 3c</td>
<td>89.60</td>
<td>5.90</td>
<td>4.28</td>
<td>0.16</td>
</tr>
<tr>
<td>Build 4d</td>
<td>89.75</td>
<td>6.00</td>
<td>4.10</td>
<td>0.15</td>
</tr>
<tr>
<td>ASTM</td>
<td>Balance</td>
<td>5.50-6.75</td>
<td>3.50-4.50</td>
<td>Max 0.30</td>
</tr>
</tbody>
</table>

a Powder 45-105 µm, build layer thickness 70 µm.
b Powder 45-105 µm, build layer thickness 50 µm.
c Powder 25-45 µm, build layer thickness 70 µm.
d Powder 25-45 µm, build layer thickness 50 µm.
To investigate and compare the surface chemical composition of the finished builds, X-ray photoelectron spectroscopy (XPS) was used. Similar results were obtained for all of the four samples regarding the XPS measurements and are discussed below. A representative survey spectrum for the results obtained is presented in Figure 15.

![Figure 15](image)

**Figure 15.** Representative XPS survey spectrum for all surface measured.

From the bulk chemical measurements of the powders in Table 3, the two powders had similar compositions and were both within the ASTM F-1472 standard specifications. The dominating elements, as from the ICP-OES measurements, were Ti, Al, V and Fe. Some traces were found of potassium (K), magnesium (Mg) sodium (Na) and silicon (Si), but their concentrations were close to the detection limit and well below the ASTM standard specifications. These are important results, since they showed that the powder stock material used to produce all samples was similar, and should, if processed equally result in similar elemental composition in the finished components.

The bulk composition for all four built samples was shown to be similar to one another. The same was observed for the surface chemical composition measured by XPS. Here, the spectra for all samples were dominated by Ti, carbon (C) and oxygen (O). Signals from nitrogen (N), calcium (Ca), Al and V were also detected. The abundance of Ti and O indicates that a surface oxide was present on the sample surface, as expected. The C signal corresponded to what is normally found on Ti surfaces, and was most likely due to surface contamination. An important observation from these results was that the build process in itself did not alter the chemical composition of the material.

The resulting microstructures from the builds with a build layer thickness of 50 µm manufactured by the standard and finer powder respectively are presented in Figure 16. From this figure, the microstructures were similar for both builds with a Widmanstätten basket-weave like structure, commonly found in rapidly quenched Ti6Al4V. These findings indicated that the micro-
structure will not be strongly influenced by powder size fraction. The results were also in agreement with what was expected, since some remelting of each layer occurs in the EBM build, which will “homogenize” the final microstructure.

![Figure 16](image)

Figure 16. Scanning electron microscopy micrographs representing the microstructure of builds with (a) 45-105 µm powder and (b) 25-45 µm powder.

The main objective of using the finer powder was to decrease the surface roughness of EBM built components. Therefore, a comparison of the surface morphology between the two powders and the two build layer thicknesses was conducted. Scanning electron microscopy (SEM) images were taken of all four build surfaces in the build (z) direction, as presented in Figure 17.
The general surface appearance of all four builds was similar, with a rippled structure perpendicular to the build direction. On all surfaces, partly melted powder particles were sintered to the surface, as commonly seen on EBM manufactured parts. By a comparison of the builds that have the same build layer thickness but different powders, Figure 17a&c and Figure 17b&d, differences were noted. For the naked eye, the parts made from the finer powder (surfaces in Figure 17c&d) appeared to have a smoother surface. However, it appeared that the parts made from the finer powder had a rougher surface, at least on a finer scale. This was due to an increased amount of powder particles sintered to the surface. However, a more regular pattern was observed for the underlying peak-to-valley ratio of the parts produced by the finer powder. From these results it was concluded that the surface morphology will be altered by the use of a finer powder.

Mechanical properties were also studied for the four samples prepared at the four process conditions with varying powders and build layer thicknesses.
Information about the hardness and stiffness (Young’s modulus, or E-modulus) was acquired through nanoindentation, see Figure 18.

![Figure 18: Hardness and stiffness (E-modulus) values with standard deviation error bars of 12 measurements respectively. Build 1, powder 45-105 µm, build layer thickness 70 µm, Build 2, powder 45-105 µm, build layer thickness 50 µm, Build 3, powder 25-45 µm, build layer thickness 70 µm, Build 4, powder 25-45 µm, build layer thickness 50 µm.](image)

From the results in Figure 18, no differences were found in mechanical properties between the builds. The hardness values were considered to be similar although some difference was seen. The difference was however neglected due to the large error margins found in build 1 and 2, which indicated that the powder size and build layer thickness did not influence the mechanical properties on samples in the same size.

Time-of-flight secondary ion mass spectrometry (ToF-SIMS) was used to investigate the surface oxide thickness. The same block shaped parts, as used for the previous analysis, were analyzed by depth profiling. Depth profiles for all four samples are presented in Figure 19 where each curve represents the evolution of the TiO$^+$ signal, thus representing the surface oxide profile.
Figure 19. TiO$^+$ signal evolution from depth profile measurements by ToF-SIMS in paper I. The build number corresponds to: Build 1, powder 45-105 µm, build layer thickness 70 µm, Build 2, powder 45-105 µm, build layer thickness 50 µm, Build 3, powder 25-45 µm, build layer thickness 70 µm, Build 4, powder 25-45 µm, build layer thickness 50 µm.

Based on these profiles the actual oxide thicknesses were estimated by the use of a reference sample with a known oxide thickness. The oxide thickness was estimated as the depth where the measurement reached the oxide-metal interface, here defined when the TiO$^+$ has decreased 50 % between its maximum value in the oxide and that in the bulk metal. The estimated surface oxide thicknesses are presented in Table 4.

Table 4. Estimated surface oxide values from ToF-SIMS measurements from samples in paper I.

<table>
<thead>
<tr>
<th>Build</th>
<th>Oxide thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Build 1</td>
<td>46</td>
</tr>
<tr>
<td>Build 2</td>
<td>42</td>
</tr>
<tr>
<td>Build 3</td>
<td>35</td>
</tr>
<tr>
<td>Build 4</td>
<td>43</td>
</tr>
</tbody>
</table>

$^a$ Powder 45-105 µm, build layer thickness 70 µm.
$^b$ Powder 45-105 µm, build layer thickness 50 µm.
$^c$ Powder 25-45 µm, build layer thickness 70 µm.
$^d$ Powder 25-45 µm, build layer thickness 50 µm.

The four EBM built surfaces had relatively similar oxide thicknesses, typically around 40 nm, which was thicker than native oxides typically found on titanium (normally less than 10 nm, see e.g. [136]). Although measurement errors may have contributed to this discrepancy, two possible explanations for the thicker oxide of the EBM built surfaces could be proposed. They were both connected to the elevated temperature during the process, which can: (i) in combination with small amounts of oxidative gas species (e.g., H$_2$O) in the vacuum chamber lead to oxide growth, or (ii) oxide growth by oxygen diffusion from the bulk to the surface during the melting process. This behavior was therefore further investigated in paper V.
8.2. Mechanical properties of small sized components and layer dependency on mechanical tensile properties for methodology development of EBM manufactured material – Paper II & III

As the thickness of a part becomes smaller, the material properties may become altered for reasons such as difference in cooling rate and altered melting strategy in the EBM process. Surface properties will also impact the mechanical properties as the size is decreased. To investigate these effects, specimens with a wall thickness range of 5, 4, 3, 2, 1 and 0.5 mm were produced. The samples were produced with the finer 25-45 µm powder and build layer thicknesses of 50 µm and 25 µm respectively. These specimens were investigated with regard to their microstructure and mechanical properties. Microstructures for samples with a build layer thickness of 50 µm are presented in Figure 20. The thickest sample wall thickness (4 mm) in Figure 20e&f shows a representative appearance for all samples thicker than 1 mm.

![Figure 20. SEM micrographs of the microstructure from the batch with 50 µm build layer thickness for three different wall thicknesses; a) 0.5 mm, 5 000 X; b) 0.5 mm, 10 000 X; c) 1 mm, 5 000 X; d) 1 mm, 10 000 X; e) 4 mm, 5 000 X; f) 4 mm, 10 000 X.](image)

Just as observed in paper I, see section 8.1, a Widmanstätten microstructure was observed for the samples in Figure 20. However, the grain size and lamellar structure differed for the thinner samples compared to the thicker ones. Average α grain widths for the thinner samples (0.5 and 1 mm) were approximately 700 nm, whereas the α grain widths for all thicker samples ranged between 1200 and 1300 nm. The shift in microstructure around 1 mm wall thickness was believed to be the result of faster cooling rate from liquid to the stable process temperature of approximately 700 °C in the EBM system during the build process. The more rapid cooling was probably enabled by the lower required energy input into the parts with smaller wall thick-
nesses. An altered melting strategy in the EBM process for the sample 0.5 mm was also believed to be a contributing factor, as this also will increase the cooling rate.

The tensile properties were hypothesized to be altered as the part size decreased in one dimension. To test this hypothesis, dog-bone shaped specimens were manufactured with the finer 25-45 µm powder with build layer thicknesses of 50 µm and 25 µm respectively. Samples were produced with wall thicknesses of 5, 4, 3, 2, 1 and 0.5 mm and the ultimate tensile strength (UTS) values were tested in their as-built condition; see the scatter plot in Figure 21.

![Figure 21. Scatter plot over the UTS values for tensile test of as built parts with 25-45 µm powders and build layer thickness of 50 µm and 25 µm respectively.](image)

The UTS values in Figure 21 showed the same overall behavior for both sample batches. For both batches, the increase in UTS was of a parabolic shaped growth as the sample wall thickness increased from 1 mm to 4 mm. A change from the parabolic appearance was noted as the wall thickness was decreased from 1 mm to 0.5 mm for both sample batches. The UTS value for 0.5 mm wall thickness was about two thirds of the UTS compared to the sample of 1 mm wall thickness, and a factor 2 lower compared to the maximum UTS at 4 mm. For samples thicker than 4 mm, the trends pointed towards constant UTS values. Since, by definition, UTS values were independent of sample cross-section areas, and therefore sample thicknesses, the differences in UTS reflected the changes in material mechanical properties.

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and/or surface appearance between the samples. As a finer microstructure often gives better mechanical properties, the lowering of UTS for the samples thinner than 4 mm, and especially 0.5 mm, will presumably be an effect of the fact that surface effects (roughness) become significant relative to the bulk properties. Therefore the surface structure will affect the mechanical properties of small components.

The lower UTS for one of the 3 mm samples with a layer thickness of 50 µm and for two of the 5 mm samples with a layer thickness of 25 µm were explained by the presence of pores. On the fracture surface for sample 3 mm pores were seen as shiny areas in the dull gray fully melted matrix, see Figure 22. Unmelted powders were also observed inside some of the pores.

![Figure 22. Fracture surface of the sample with lower UTS, built using a build layer thickness 50 µm, 25-45 µm powder and a wall thickness of 3 mm.](image)

All parts manufactured by AM, and also EBM, have successive layer fused on top of each other. Therefore, insufficient fusion between the individual layers is a potential concern, which is of interest to investigate. Digital image correlation (DIC) was used for visualization of the local strain fields. It was especially interesting to investigate if the strain fields are co-located with the layer boundaries and causes fracture between individual layer boundaries. The basic investigation was a setup where two cameras were used to analyze a full length cylindrical tensile bar with a gage length of 42 mm, 7 mm in diameter. The tensile bars were machined and manufactured according to the description of paper III. The DIC strain fields for two samples representing the general behavior are presented in Figure 23, just before fracture of each bar.
Local strain fields were observed to be scattered over the entire analysis area, as areas of strains of 2 % and below were seen to be associated with areas of strains of 7 % and higher, see Figure 23a. The other sample, Figure 23b, on the other hand, showed a homogenous behavior where the strain fields were evenly distributed around 7 %. The randomly distributed strain in Figure 23a was most likely due to porosity found on the sample surface caused by non-optimized EBM process parameters. However, the strain fields observed were in the range of millimeters, so the individual build layers of 70 µm would not be possible to resolve with the set-up used. Therefore DIC measurements were conducted using a microscopic lens to track the strain fields with an increased resolution. The results from the micro DIC measurements are presented in Figure 24.
Figure 24. DIC strain fields showing the same sample. Lower row shows, the micro level strain fields. Upper row shows the corresponding full length global strain field. The images were recorded at different stages of the tensile test: (a) Elastic deformation zone. (b) Plastic deformation zone. (c) maximum applied force, (no micro level image available).

The resolution of the DIC was improved by the use of the microscopic lens. From the appearance of both the global (top part of Figure 24) and local micro-level (lower part in Figure 24) strain fields, the material did not show a layer-wise strain distribution. Especially for the local micro level strain fields, where the area measured was approximately $2 \times 2 \text{ mm}^2$ and the spatial resolution 22.65 µm pixels, each 70 µm layer would be possible to be resolved. If the strain fields were to be located in the layer boundaries, straight red lines would be present in Figure 24b. Since the local strain fields were irregularly distributed over the entire analysis area the material was presumed to be homogenous and no correlation was found between the layer thickness and the strain fields.
8.3. Surface properties on small sized components, oxidation formation and surface chemical properties of EBM manufactured material - Paper IV & V

As been described earlier, the surface roughness will impact the material properties of small sized components, when surface roughness becomes significant relative to the nominal dimensions of the cross-section. Since EBM produced parts have a characteristic surface morphology the appearance of the surface is of importance. To investigate the effect of powder size and layer thickness, samples were produced in three batches, where batch one had a build layer thickness of 50 µm and used the standard 45-105 µm powder, batch two a build layer thickness of 50 µm and the finer 25-45 µm powder, and batch three a build layer thickness of 25 µm and the finer 25-45 µm powder. Samples from all baths were manufactured with wall thicknesses of 5, 4, 3, 2, 1 and 0.5 mm. These samples were investigated with respect to surface roughness parameters. Representative images of the surface appearance of the side surfaces (perpendicular to build direction) for all samples are presented in Figure 25.

![Figure 25](image)

Figure 25. SEM micrographs of the surface appearance for samples of 0.5 mm wall thickness built with (a) 45-105 µm powder, layer thickness 50 µm; (b) 25-45 powder, layer thickness 50 µm; (c) 25-45 µm powder, layer thickness 25 µm.

The surface morphology appeared to be different for the samples with the same build layer thickness but with different powder sizes (50 µm layer thickness and 45-105 µm or 25-45 µm powder respectively, see Figure 25a&b). Differences were also observed for samples with the same powder but different build layer thicknesses (25-45 µm powder and 50 µm or 25 µm build layer thickness respectively, see Figure 25b&c). The surface roughness of samples from all three batches was analyzed using focus variation profilometry, white light interferometry and stylus profilometry. The resulting mean surface roughness $R_a$ values for samples with different wall thicknesses in each batch are presented in Figure 26 for the respective surface measurement technique used.
The surface roughness values in Figure 26 varied for each sample, dependent on the measurement technique used. As expected, stylus profilometry gave underestimated values, since the stylus was not able to correctly follow the surface contour. White light interferometry on the other hand used a single focus depth, where dead, undetected, pixels are extrapolated by the software. Due to the amount of undetected pixels, the white light interferometry values of the surface features were presumably over-estimated. From these results a trend was seen where both powder particle size and build layer thickness influenced the surface roughness. Comparing the two samples with the same build layer thickness of 50 µm, but with different powder sizes, it was observed that the sample with the finer powder had a 15% lower Ra value according to the focus variation profilometry measurement. This was most likely due to the finer powder structure, as the stair-stepping size during melting will be decreased and the surface resolution increased. If the build layer thickness was altered, but keeping the same powder in Figure 26, a small 4% decrease in Ra value was indicated which was due to the finer melting and smaller stair-stepping size used for the parts with the smaller build layer thickness. This behavior will increase the surface resolution.

The earlier findings in paper I, indicated that EBM manufactured surfaces had a thicker surface oxide than conventionally machined parts. Therefore work was conducted in paper IV and V to further investigate the surface oxide thickness and its formation in the EBM process. In paper IV the cross section of an EBM produced surface in the z-direction was analyzed with transmission electron microscopy (TEM) to visualize the surface oxide and to obtain an independent measure of its thickness. A scanning transmission electron microscopy (STEM) spectrum and elemental maps of the O and V
distribution obtained by electron energy loss spectroscopy (EELS) measurements are presented in Figure 27. The surface oxide layer, seen as a region of elevated signal in Figure 27b, was measured to be 5-6 nm thick. A 12-13 nm thick vanadium-enriched region at the surface was also detected (Figure 27c).

Figure 27. High resolution images from cross section of surface of EBM material (z-direction), showing a) TEM image, b) EELS map of oxygen (O), c) EELS map of vanadium (V).

To confirm these measurements, the surface oxide thickness was also measured using ToF-SIMS and Auger electron spectroscopy (AES) in paper V. The general appearance of the surfaces in the build direction has been discussed earlier. Due to the EBM process setup, the top surface of an EBM built component showed a different appearance compared to the build direction (side) surfaces, see Figure 28.
AES depth profiles were measured from both fully melted areas and sintered powder grains on the side surface, from various points on a top surface, and from stock powder particles. Representative AES profiles for the powder and a melted side surface are presented in Figure 29.

These depth profiles were seen to have similar general qualitative features. As the contaminations (carbon signal) were etched away, the shape of the titanium (Ti) and oxygen (O) signals reflected the presence of a surface oxide. The change in Ti and O signals reflected the removal of the surface oxide during etching and that the oxide-metal interface was gradually reached. The AES depth profiles can be used to estimate the depth at which the oxide–metal interface was reached in the measurement. In this case, the thickness was defined as the depth of half the initial decrease of the O signal. Considering the complexity of the oxide layer, with an outermost TiO₂ layer beneath which suboxides or dissolved O were present and possible profile
broadening due to topography, it was not, however, straightforward to define an oxide layer thickness.

From this definition of the surface oxide, surface oxides were estimated for a sample located on the start plate, 0 mm into the build height, see Figure 30.

![Surface Oxide Thickness](image)

*Figure 30. Surface oxide thicknesses at different location on a sample located 0 mm into the build and the powder used for production, as measured by AES. The values are averages of two measurements for the Top and Side-Grain values, three for the Side-Melt, and ten for the powder.*

From these values, it was observed that the surface oxide of the as-built parts was thicker than the powder stock material. These values were also slightly thicker than what is normally found on traditionally machined titanium surfaces. The difference between the oxide thicknesses on the top surface and the side surface was presumed to be due to an insulating effect of the surrounding powder bed around the side surfaces. This will result in an elevated temperature around these surfaces, and the elevated temperature will promote the oxide growth, which was presumed to take place in the EBM system. To evaluate how the oxide growth evolve over the build time and build height, ToF-SIMS analyses were made on surfaces located 0 mm, 80 mm and 120 mm in the build direction. The ToF-SIMS analysis consisted of depth profiling, similar to the procedure described in section 8.1. The relative oxide thicknesses for the different samples, normalized against a polished reference sample, are presented in Figure 31.
Figure 31. Relative oxide thicknesses of EBM samples at different build heights, as measured by ToF-SIMS. All values are normalized against a polished reference sample.

The samples built at a lower height had a thicker oxide compared to samples built at elevated heights. The samples at lower build heights only had slightly thicker oxide thickness than the polished reference sample. The surface oxide of the sample at the start plate (0 mm) had a surface oxide which is approximately two times thicker than the sample built at 120 mm. Comparing the oxide thicknesses of different surfaces within each build height showed that the side surfaces tended to have thicker surface oxides than the corresponding top surface, in agreement with the AES results.

From the results of the surface oxide thickness measurements from both AES and ToF-SIMS, the surface oxides were presumed to be formed in the EBM build chamber after melting of the current layer. The difference in oxide thickness between the top and side surfaces for parts on the same build height was most likely due to temperature difference. For the top surface, no surrounding powder was present, and the surface cooled down fast in comparison to the side surface which was lodged in surrounding unmelted powder. Water and hydroxyl groups were present on all un-melted powder particles. Upon heating, the water and hydroxyls were released and can therefore react with the titanium. The presence of moisture was also a tentative explanation for the formation of a thicker oxide on parts at low build heights. As these parts were exposed to an elevated temperature for a longer time, the oxide will grow thicker than for surfaces higher in the build. Also, the moisture will reach steady state as the build progressive and a constant oxide thickness, comparative to a native oxide will be formed. The work presented here has increased the knowledge of the oxide formation process on EBM built parts.
9. Conclusion

The work in this thesis concludes that the surface morphology of EBM built parts can be improved in various ways. An improvement of the surface resolution will result in the possibility to produce parts with a higher level of detailing. The following facts have been answered in the scope of this work:

- It is feasible to use a finer 25-45 µm Ti-6Al-4V powder in the Electron Beam Melting (EBM) process. The chemical and mechanical properties of material built with the finer powder were similar as for standard powder.
- The tensile properties of small components will be negatively affected by the surface roughness for parts smaller than 4 mm.
- Provided that proper process parameters are used to avoid pore formation and incomplete melting, EBM produced components do not display layer dependency on mechanical strength. The developed digital image correlation methodology is a powerful tool for investigating tensile properties analysis of additive manufactured parts.
- By the use of a finer powder, and a smaller build layer thickness, the surface resolution can be improved.
- There is a dependency of the surface oxide thickness on the build height, where the oxide thickness is thicker at low build height. The surface oxide is formed in the build chamber after melting the powder.

A dental prosthetic bridge was manufactured, applying the results found here, to show the improvements made in this work. This dental bridge was compared to a dental prosthetic bridge manufactured using the standard EBM process settings and powder. The final result can be seen below.

*Figure 32. Dental bridge manufactured in EBM using the standard process setting (left) and with the settings optimized in this work (right).*
The results found in this work can help to rationalize the manufacturing of dental prosthetics using the EBM process. This will give an increased number of people the opportunity to a better life as they gain access to affordable dental prosthetics with known material properties.
10. Future Perspective

Although the work performed in this thesis has broken new grounds to improve the production of small sized components in the Electron Beam Melting (EBM) system, much more work needs to be done. For the work presented in this thesis, further optimization of process parameters is needed to secure good quality parts. An optimized process theme will give pore free components with an increased surface resolution. Investigations regarding how other parameters affecting the surface resolution and material properties, such as melt strategies of small components are also desired. This is tedious work since reliable simulation models for the EBM process do not yet exist. However, the potential benefits of using a finer powder in the EBM are persistent.

For the use of additive manufacturing in general, as a competitive manufacturing method, a great deal of understanding of both the processes and material properties are still needed for these additive manufacturing technologies before they can gain final acceptance in industry. Although the additive manufacturing processes are still in a state of infancy for industrial use, the technique has great potential to revolutionize the manufacturing industry.
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Svensk sammanfattning


Dock finns idag begränsningar med EBM-tekniken där ytstrukturen är för ojämn. Detta gör att mycket detaljer på tänderna inte fås med och att det blir svårt att fästa den ytbeläggning av porslin eller polymer som ofta används. Det gör att detaljerna måste efterbearbetas innan de kan användas. Man har idag en relativt god kunskap när det gäller materialegenskaperna på större detaljer tillverkade med EBM. För tandersättningar, som oftast har tunna väggar och storlekar mindre än 1 cm, kommer dock materialegenskaperna att bli påverkade under tillverkningen, eftersom ytans egenskaper i högre grad än för stora komponenter påverkar materialegenskaperna.

Målet med arbetet har varit att studera hur yttegenskaperna för små komponenter tillverkade med EBM kan förbättras. Det har också undersömts hur materialegenskaperna ändras och hur ytan påverkar de slutliga egenskaperna.
I en första studie har ett finare pulvermaterial använts och jämförts med den pulverrvaror som normalt används i processen. Egenskaper såsom kemisk sammansättning i bulk och yta ha studerats, både på de två pulverrvarornas och på tillverkade detaljer. Dessutom har mekaniska egenskaper testats för de tillverkade detaljerna. Det visar sig att ingen nämnvärd skillnad finns för egenskaper hos vare sig pulverrvarorna eller de färdigbyggda detaljerna.

För att undersöka hur ytegenskaperna påverkar små komponenter, testades de mekaniska egenskaperna hos små komponenter i obearbetat material tillverkat med EBM. Det visar sig att för komponenter med en väggtjocklek på större än 4 mm finns en liten, eller ingen, påverkan av ytan på materialets brottstyrka. För material med dimensioner mindre än 4 mm kommer brottgränsen att minska i takt med att den nominella väggtjockleken minskar. En drastisk minskning i brottstyrka fås när den nominella väggtjockleken minskar från 1 mm till 0.5 mm.

För alla typer av AM-tillverkat material finns en risk att materialet i varje lager inte fäster tillräckligt vid lagret under, och att lagren tenderar till att spricka ifrån varandra. För att testa detta på EBM-tillverkat material användes så kallad digital image correlation, vilket enkelt kan beskrivas som ett sätt att digitalt analysera bilder för att studera mikroskopiska förändringar i ett material under, till exempel, ett dragprov. Från tester på både makro- och mikronivå syns tydligt att materialet inte påvisar något lagerberoende, utan är homogent i sina mekaniska egenskaper.

Ytans råhet har blivit uppmätt med flera metoder för att undersöka hur ytråheten påverkas vid användande av de två pulverrvarorna och olika bygglagertjocklekar. Ytans råhet förbättras avsevärt när det finare pulvret, med mindre pulverkorn, används. Dessutom blir ytan mer slät när bygglagertjockleken minskas, med samma typ av pulverstorlek. Detta innebär att ytans egenskaper kan förbättras om ett finare pulver används och bygglagertjockleken minskas.


Resultaten från detta arbete pekar på flera sätt hur tillverkningen av dentalersättningar med hjälp av EBM-tekniken kan förbättras. Det kommer resultera i att fler patienter får tillgång till tandersättningar och att dessa patienters livskvalitet kommer att förbättras.
References


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