NAVAL MEDICAL RESEARCH UNIT SAN ANTONIO

TECHNICAL REPORT

DENSITY DETERMINATION AND METALLOGRAPHIC SURFACE PREPARATION OF ELECTRON BEAM MELTED Ti6Al4V

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<th>Abbreviation</th>
<th>Description</th>
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<tr>
<td>AM</td>
<td>Additive Manufacturing</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
</tr>
<tr>
<td>CMG</td>
<td>Commercial Medical Grade</td>
</tr>
<tr>
<td>EBM</td>
<td>Electron Beam Melting</td>
</tr>
<tr>
<td>D</td>
<td>Density</td>
</tr>
<tr>
<td>D_L</td>
<td>Density of Water at Recorded Temperature</td>
</tr>
<tr>
<td>DI</td>
<td>Deionized Water</td>
</tr>
<tr>
<td>SAMMC</td>
<td>San Antonio Military Medical Center</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SiC</td>
<td>Silicon Carbide</td>
</tr>
<tr>
<td>Ti6Al4V</td>
<td>Titanium-6Aluminum-4Vanadium</td>
</tr>
<tr>
<td>WRNNMC</td>
<td>Walter Reed National Military Medical Center</td>
</tr>
<tr>
<td>W_d</td>
<td>Dry Weight</td>
</tr>
<tr>
<td>W_w</td>
<td>Wet Weight</td>
</tr>
</tbody>
</table>
EXECUTIVE SUMMARY

Background: Electron Beam Melting (EBM) is an additive manufacturing technique that is currently used to produce customized titanium-6aluminum-4vanadium (Ti6Al4V) cranial implants for wounded warfighters who require cranial reconstruction surgeries due to loss of skull tissue. Infections can occur in the surgical site, which may be due to the surface topography of the implant that favors bacterial contact and adhesion.

Objective: The objective of this study was to establish density determination and metallographic surface preparation procedures for Ti6Al4V coupons fabricated using the EBM process.

Methods: Densities of EBM Ti6Al4V coupons were measured using the Archimedes method and compared to those of commercial medical grade (CMG) Ti6Al4V coupons. Surfaces of coupons were treated using serial mechanical polishing with silicon carbide (SiC) papers and colloidal silica suspension to produce samples with varying surface topographies. Surfaces were polished to 60 grit SiC, 120 grit SiC, 400 grit SiC, and colloidal silica finishes were analyzed using scanning electron microscopy (SEM) and compared to CMG Ti6Al4V coupons that were polished to the same finishes. Statistical analysis to compare density measurements of EBM and of CMG Ti6Al4V was performed using a t-test.

Results: The average density of EBM Ti6Al4V coupons was 4.26 ± 0.04 g/cm³, while that of CMG Ti6Al4V coupons was 4.41 ± 0.01 g/cm³, which was close to the theoretical density of Ti6Al4V (4.43 g/cm³). The difference in densities between EBM Ti6Al4V and CMG Ti6Al4V were statistically significant (p < 0.05). SEM analysis of polished EBM Ti6Al4V samples showed a progressive change in surface topography from polishing with 60 grit SiC paper to polishing with colloidal silica suspension.

Conclusions: One factor contributing to an average lower density of EBM Ti6Al4V compared to CMG Ti6Al4V may be due to the presence of porosity, which is known to be introduced during the EBM process. Conventional metallographic surface preparation can be used to engineer the surface of EBM Ti6Al4V to produce different surface topographies.
INTRODUCTION

Additive manufacturing (AM) is gaining much interest for the development of near-net shape medical implants because AM requires reduced machining and post-processing to fabricate customized implants compared to conventional implant fabrication techniques. Electron beam melting (EBM) is an AM technique currently used to produce titanium-6aluminum-4vanadium (Ti6Al4V) cranial implants for wounded warfighters who require cranioplasty procedures at Walter Reed National Military Medical Center (WRNMMC) and San Antonio Military Medical Center (SAMMC). Electron beam melting is a powder bed AM process involving consolidation of metallic powder layers in a vacuum chamber by focused heating of each powder layer using an electron beam to build a component, based on a computer aided design (CAD) file [1, 2]. An example of an EBM Ti6Al4V implant is shown in Figure 1A.

Figure 1. (A) Example of an EBM Ti6Al4V cranial implant. (B) SEM image of the surface of EBM Ti6Al4V. Examples of incompletely melted powder particles and pores are indicated by boxes and arrows, respectively. (C) X-ray micro computed tomography cross section image showing pores, indicated by yellow boxes, which penetrate deep into the EBM material.
EBM Ti6Al4V surfaces are known to be rough with many incompletely melted powder particles and large pores (Figures 1B and 1C). Previous studies [3-5] have found that surface topographies and porosity can influence bacterial contact and attachment to titanium implant surfaces leading to biofilm growth and infection. The presence of porosity within a material results in a decrease in its density. The objectives of this investigation were to: (1) develop a procedure based on Archimedes’ method to determine the density of EBM Ti6Al4V, and (2) determine whether conventional metallographic surface preparation techniques can be used for post-processing of EBM Ti6Al4V to obtain different surface topographies.

**MATERIALS AND METHODS**

*Materials*

Ti6Al4V coupons, approx. 25 mm in diameter and 2.6 mm thick, were fabricated from Arcam Ti6Al4V powder (Arcam AB, Mölndal, Sweden) ARCAM A1 EBM system equipped with Arcam EBM Control software (Arcam AB, Mölndal, Sweden) using the EBM process at the 3D Medical Applications Center at WRNMMC. Commercial medical grade (CMG) Ti6Al4V coupons (Vulcanium, Northbook, IL) were obtained for comparison.

*Density Measurement via Archimedes’ Method*

Archimedes’ method for density determination is based on Archimedes’ principle, which states that the buoyant force on an object when it is immersed in a liquid is equal to the weight of the liquid that it displaces [6]. The volume of the object can therefore be calculated based on the difference between the weight of the sample in air (or dry weight, \(W_d\)) and the weight of sample when immersed in the liquid (or wet weight, \(W_w\)), which is the buoyant force, and the density of the liquid (\(D_L\)). Density (D) of the object can be determined from \(W_d\) and the calculated volume. Therefore, according to Archimedes’ method,

\[
D = \frac{W_d}{(W_d - W_w)} \times D_L
\]

(1)

In this investigation, \(D_L\) is the density of water at the recorded temperature.
The densities of 20 EBM Ti6Al4V coupons were determined using a Torbal AGCN200 scale fitted with a Density Measurement Kit (Torbal, Bohemia, NY) shown in Figure 4A. Different components of the kit (labeled in Figure 4B) include a 400 ml beaker for the weighing liquid, beaker support frame, thermometer and thermometer hanger, upper pan for measuring dry weight ($W_d$), lower pan for measuring wet weight, and lower pan hanger. Before starting experiments, the beaker was placed on the beaker support frame pan. The thermometer was inserted through the holes of the hanger and hung on the inside of the beaker. The upper pan, lower pan hanger, and lower pan were assembled and hung from the top support of the beaker support frame. The beaker was filled with MilliQ water (the weighing liquid) above the 300 ml level to ensure that the lower pan and the hinge connecting the lower pan hanger to the lower pan were fully submerged. The scale was powered on and set to the density measurement function by following instructions provided in the Torbal AGCN200 manual. The weighing liquid was selected as “H2O” and the temperature entered into the display panel was read from the thermometer inside the beaker.

Figure 4. (A) Torbal AGCN200 scale with density measurement kit. (B) Detailed view of density measurement kit.
For the determination of density of each coupon, the following steps were taken. The scale was tared by pressing the tare button ‘↔T’. The dry weight was measured by placing the coupon on the dry weight pan. Once the display on the scale stabilized, the print button ‘P’ was pressed to store the \( W_d \) value. The coupon was removed from the upper pan and submerged in MilliQ water in a separate beaker. The coupon was removed from the water and brushed with a wet Q-tip to completely wet and remove any air bubbles on all surfaces of the coupon. The lower pan was lifted out of the 400 ml beaker and the coupon was placed on the lower pan and lowered down into the 400 ml beaker. Once the display on the scale stabilized, the print button ‘P’ was pressed to store \( W_w \) value. ‘P’ was pressed again to obtain the value of \( D \) of the coupon according to Equation 1.

Figure 5. Flow chart outlining procedure to measure density using the Torbal AGCN200 scale with attached density measurement kit. Steps outlined in black were performed before starting density measurements. Steps outlined in red were repeated to obtain the density measurement for each coupon.
A flowchart summarizing the density measurement procedure is shown in Figure 5. The steps outlined in red were repeated 3 times for each EBM Ti6Al4V coupon to determine reproducibility of the results. Densities of 20 CMG Ti6Al4V coupons were also measured for comparison.

Mounting of EBM Ti6Al4V Coupons for Surface Preparation

Before polishing, coupons were pressed into plastic mounts that were custom made in house based on the diameters of the coupons and diameter of the specimen holes in the Buehler 1.25” x 6 barrel central force specimen holder (catalog #602483, Buehler, Lake Bluff, IL). Two versions of mounts were made. Mount 1 is shown in Figure 6A. Coupons were pressed into these mounts using a small table top press. Once coupons were pressed into the mounts, they were loaded into the holes of the specimen holder, which was sitting on a custom made stand (Figure 6C). The stand was designed so that the mounted coupons protruded from the bottom of the specimen holder by 3 mm to ensure that the bottom of the holder was not abraded during polishing and the coupons were level on a horizontal surface. Mount 2 is shown in Figure 6B. In the case of these mounts, the mounts were first loaded into the central force specimen holder, followed by pressing the coupons into the mounts by hand.
Metallographic Surface Preparation of EBM Ti6Al4V

Metallographic surface preparation procedures consist of a series of steps that vary depending on the type of material and the desired application of the material. One of the most common surface preparation procedures is mechanical grinding/polishing. During each step, the surface of interest is subjected to mechanical abrasion to remove the top layer of material that has defects such as scratches. The abrasive media consists of particles of a specific average size that are affixed to a paper or film backing or in liquid suspension. Grinding is the first step and involves using a coarse abrasive media to remove damage on the surface of the sample, which is usually the result of sample cutting or the manufacturing process. In the case of EBM Ti6Al4V the initial rough surface, shown in Figure 1B, resulted from the manufacturing process. For titanium alloys, the grinding media typically used is silicon carbide (SiC) paper. Table 1 lists grades of SiC papers that are available commercially along with the average size of SiC particles.
adhered to the surface of the papers. Mechanical treatment with grit papers 60 – 600 is generally considered as grinding. Grinding is followed by polishing. Polishing involves either using finer grades of SiC papers (grit 800 – 1200) or suspensions, such as alumina or diamond, containing finer particles sizes. To polish with a suspension, the suspension is introduced onto a lapping cloth and the surface of the material is abraded with the “charged” cloth. In the case of titanium alloys, diamond suspensions are typically used for polishing steps. However, in this study, different grades of SiC paper were used for polishing to reduce the difference in surface chemistry resulting from different polishing media. A ‘final polish’ step follows the last polishing grit size to produce a “mirror” finish on the sample surface. For titanium, a colloidal silica suspension (as the abrasive media) with hydrogen peroxide (as an attack-polish agent) is typically used to produce the characteristic “mirror” finish after final polishing. However, in our study, hydrogen peroxide was removed from this step to reduce changes in surface chemistry that may result due to the change in polishing media. Particle size for colloidal silica is also listed in Table 1. Other techniques may also be used for surface preparation. [7, 8, 9] Throughout this report, the word “polishing” will be used to refer to the grinding and polishing steps.

Table 1. List of SiC grinding/polishing paper grades available commercially. [9] Each grade is classified by a grit number based on the average particle size on the paper. The particle size for MasterMet Colloidal Silica suspension is also listed. SiC grits used to prepare coupons in this study are highlighted.

<table>
<thead>
<tr>
<th>Grit Number</th>
<th>Size (µm)</th>
<th>Step #</th>
</tr>
</thead>
<tbody>
<tr>
<td>60</td>
<td>268.0</td>
<td>1</td>
</tr>
<tr>
<td>80</td>
<td>188.0</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>148.0</td>
<td></td>
</tr>
<tr>
<td>120</td>
<td>116.0</td>
<td>2</td>
</tr>
<tr>
<td>180</td>
<td>78.0</td>
<td>3</td>
</tr>
<tr>
<td>220</td>
<td>66.0</td>
<td></td>
</tr>
<tr>
<td>240</td>
<td>51.8</td>
<td>4</td>
</tr>
<tr>
<td>280</td>
<td>42.3</td>
<td></td>
</tr>
<tr>
<td>320</td>
<td>34.3</td>
<td>5</td>
</tr>
<tr>
<td>360</td>
<td>27.3</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>22.1</td>
<td>6</td>
</tr>
<tr>
<td>500</td>
<td>18.2</td>
<td></td>
</tr>
<tr>
<td>600</td>
<td>14.5</td>
<td>7</td>
</tr>
<tr>
<td>800</td>
<td>12.2</td>
<td>8</td>
</tr>
<tr>
<td>1000</td>
<td>9.2</td>
<td></td>
</tr>
<tr>
<td>1200</td>
<td>6.5</td>
<td>9</td>
</tr>
<tr>
<td>Colloidal Silica</td>
<td>0.06</td>
<td>10</td>
</tr>
</tbody>
</table>
Mechanical polishing was performed using a Buehler EcoMet 6 grinder/polisher with AutoMet automatic polishing head attachment (Buehler, Lake Bluff, IL), shown in Figure 7A. The EcoMet 6 has two 8” diameter polishing wheels and two water spouts, one for each wheel, which can be controlled individually via valves. The steps listed below were followed for surface preparation. For the silicon carbide (SiC) papers, an 8” diameter double-sided adhesive MetGrip liner (catalog #308508, Buehler, Lake Bluff, IL) was carefully smoothed down onto the polishing wheel to prevent air bubble entrapment. For steps 1 – 9 listed below, the respective grade of 8” diameter CarbiMet 2 SiC polishing paper (Buehler, Lake Bluff, IL) was carefully smoothed down onto the liner before polishing and carefully peeled off and disposed after polishing.

Figure 7. (A) Buehler EcoMet 6 polisher with Automet polishing head. (B) Set-up of polisher during polishing with SiC paper. (C) ChemoMet cloth mounted for polishing with colloidal silica suspension.
Polishing time, rotation direction, force, and polishing fluid were programmed on the AutoMet head, and polishing wheel speed (RPM) was programmed on the EcoMet base. Polishing parameters vary based on the class of materials undergoing polishing. For titanium, if an optimum combination of rotational speed and force are not used, the coupon surface can either become ‘burned’ (or oxidized) or polishing may not occur effectively and longer polishing times are required [8]. The parameters listed below were chosen specifically for Ti6Al4V based on discussions with Buehler technical service staff. Figure 7B shows the set-up of the polisher during polishing with SiC paper. Table 2 is a summary of the steps listed below.

1. Coupons were first polished with 60 grit SiC paper for 3 min with complementary rotation, 24 lb force (4 lb/sample), and 250 RPM. To reduce heat from friction and remove debris of polished material and loose SiC particles, “water” was selected as the polishing fluid and aimed onto the paper throughout the duration of polishing. The control valve for the water spout was turned on by ½ turn of the knob, while the other water control valve was kept closed. Polishing was stopped after 3 minutes to check the sample surface and clean the polishing paper. The paper was washed and the specimen holder was removed from the polisher head and the side with the coupons was dried with compressed air. Polishing was repeated for another 3 min. The paper was then switched out to a new 60 grit paper and polishing was repeated until all coupons were flat and the original EBM surface was removed.

2. The coupons were then polished with a 120 grit SiC paper for 1.5 min with contra-rotation, 24 lb of force, and 250 RPM with water as the polishing fluid.

3. Step 2 was repeated with 180 grit SiC paper.
4. Step 2 was repeated with 240 grit SiC paper.
5. Step 2 was repeated with 320 grit SiC paper.

6. The coupons were then polished with a 400 grit SiC paper for 30 sec with contra-rotation, 24 lb of force, and 250 RPM with water as the polishing fluid. To enhance the removal of debris from the paper, a folded paper towel was held on the SiC paper to catch debris and prevent it from revolving back under the coupons.

7. Step 6 was repeated with 600 grit SiC paper.
8. Step 6 was repeated with 800 grit SiC paper.
9. Step 6 was repeated with 1200 grit SiC paper.
10. The second polishing wheel on the EcoMet was used. A ChemoMet cloth with adhesive backing (catalog #407918, Buehler, Lake Bluff, IL) was smoothed down carefully onto the polishing wheel (Figure 3C). Water was run onto the cloth until it was damp. A disposable pipette was used to transfer 2 ml MasterMet colloidal silica suspension (catalog #406370064, Buehler, Lake Bluff, IL) uniformly onto the cloth. Polishing was performed for 10 min with contra-rotation, 30 lb of force (5 lb/sample), and 150 RPM. The polishing fluid was selected as ‘other’ because water was not used as the polishing fluid. During polishing, a 0.5 – 1 ml of MasterMet suspension was pipetted onto the cloth every 30 sec to ensure that the coupons were always in contact with fresh suspension. For the last 20 seconds of polishing, water was run onto the cloth to clean the colloidal silica suspension from the surfaces of the coupons.

To achieve different surface finishes, polishing was stopped at Step 1, 2, 6, or 10 for different sets of samples. Figure 8 shows the appearance of the grit papers and colloidal silica suspension used in steps 1, 2, 6, and 10.

Table 2. List of parameters used in each step of the metallographic surface preparation procedure.

<table>
<thead>
<tr>
<th>Step</th>
<th>Grit Number</th>
<th>Force (lb)</th>
<th>Rotation Direction</th>
<th>Polishing Time</th>
<th>Wheel Speed (RPM)</th>
<th>Polishing Fluid</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>60</td>
<td>24</td>
<td>complementary</td>
<td>3+ min</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>2</td>
<td>120</td>
<td>24</td>
<td>contra-rotation</td>
<td>1.5 min</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>3</td>
<td>180</td>
<td>24</td>
<td>contra-rotation</td>
<td>1.5 min</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>4</td>
<td>240</td>
<td>24</td>
<td>contra-rotation</td>
<td>1.5 min</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>5</td>
<td>320</td>
<td>24</td>
<td>contra-rotation</td>
<td>1.5 min</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>6</td>
<td>400</td>
<td>24</td>
<td>contra-rotation</td>
<td>30 sec</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>7</td>
<td>600</td>
<td>24</td>
<td>contra-rotation</td>
<td>30 sec</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>8</td>
<td>800</td>
<td>24</td>
<td>contra-rotation</td>
<td>30 sec</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>9</td>
<td>1200</td>
<td>24</td>
<td>contra-rotation</td>
<td>30 sec</td>
<td>250</td>
<td>water</td>
</tr>
<tr>
<td>10</td>
<td>Colloidal Silica</td>
<td>30</td>
<td>contra-rotation</td>
<td>10 min</td>
<td>150</td>
<td>MasterMet suspension</td>
</tr>
</tbody>
</table>
Figure 8. Appearance of the different grades of polishing media used to create the different EBM Ti6Al4V finishes: (A) surface of the 60 grit paper for Step 1, (B) surface of the 120 grit SiC paper for Step 2, (C) surface of the 400 grit SiC paper for Step 6, and (D) colloidal silica suspension for Step 10.

Unmounting and Cleaning of Coupons

Coupons were removed from Mount 1 by leveraging the coupons at the side grooves (Figure 6A) of the mounts. For Mount 2, coupons were removed by pushing the bottom of the coupon to force them out from the top of the mount. Coupons polished with only SiC papers were dried with compressed air and stored in individual plastic zipper bags after removal from the mounts. Coupons polished with MasterMet suspension required an additional cleaning step to remove any colloidal silica adhered on the surface. A mixture of 5 drops of dishwashing soap in 20 ml of deionized (DI) water was prepared. Using gloved hands, each coupon was cleaned individually by gently swabbing the polished surface with a Q-tip soaked with the soap-water mixture. The entire polished surface was then rubbed gently with a gloved finger to aid in removal of silica particles. The coupon was washed with DI water while rubbing the surface
with a gloved finger to remove the soap and dried with compressed air. If any colloidal silica remained on surface after drying (appeared as blue streaks), the cleaning procedure was repeated until the surface appeared clean.

**Scanning Electron Microscopy (SEM)**

Polished EBM Ti6Al4V coupons were secured on an electron microscope stage using double-sided copper tape and imaged using a Zeiss Sigma VP-40 SEM (Zeiss, Jena, Germany) operated at 2 kV in high vacuum mode. Unpolished EBM Ti6Al4V and CMG Ti6Al4V polished to the same surface finishes were also imaged for qualitative comparison.

**Statistical Analysis of Density Measurements**

The total average densities and standard deviations for EBM Ti6Al4V and CMG Ti6Al4V were calculated. A *t*-test was performed to compare the densities of the EBM and CMG coupons.

**RESULTS**

**Density Measurement via Archimedes’ Method**

The total average density of EBM Ti6Al4V coupons was 4.26 ± 0.04 g/cm$^3$, while the total average density of CMG Ti6Al4V coupons was 4.41 ± 0.01 g/cm$^3$. The difference in the average densities of EBM and CMG Ti6Al4V was statistically significant (p < 0.05).

**Metallographic Surface Preparation of EBM Ti6Al4V**

Figure 9 shows the different surface finishes of unpolished and polished EBM Ti6Al4V coupons. Figure 10 is an SEM image of the surface of an unpolished EBM Ti6Al4V coupon. Figures 11 – 14 show comparative SEM images for the surfaces of EBM and CMG Ti6Al4V coupons polished to 60 grit, 120 grit, 400 grit, and colloidal silica surface finishes. No apparent differences were observed between the surfaces of EBM Ti6Al4V and the surface of the CMG Ti6Al4V samples. In addition, surfaces of coupons of the same grade of Ti6Al4V polished with the same parameters had similar surface finishes.
Figure 9. EBM Ti6Al4V coupons polished to different steps to produce different surface finishes: (A) unpolished, (B) polished to Step 1 with 60 grit paper, (C) polished to Step 2 with 120 grit paper, (D) polished to Step 6 with 400 grit paper, and (E) polished to Step 10 with colloidal silica. Topography on the surface of coupon A results from the EBM manufacturing process. Scratches on coupons B – D result from polishing with SiC grit paper. The “mirror” surface finish of coupon E results from final polish with colloidal silica.

Figure 10. SEM image of an unpolished EBM Ti6Al4V coupon. A label ‘P’ indicates a large pore.
Figure 11. SEM images of EBM (A) and CMG (B) Ti6Al4V polished to Step 1 with 60 grit SiC paper. Streaks in the images are scratches from polishing with 60 grit paper.

Figure 12. SEM images of EBM (A) and CMG (B) Ti6Al4V polished to Step 2 with 120 grit SiC paper. Streaks in the images are scratches from polishing with 120 grit paper.

Figure 13. SEM images of EBM (A) and CMG (B) Ti6Al4V polished to Step 6 with 400 grit SiC paper. Streaks in the images are scratches from polishing with 400 grit paper.
Figure 14. SEM images of EBM (A) and CMG (B) Ti6Al4V polished to Step 10 with colloidal silica suspension. Minimal topographies on the surfaces in the images are due to polishing with colloidal silica.

**DISCUSSION**

Theoretical density of Ti6Al4V is 4.43 g/cm$^3$. CMG Ti6Al4V coupons were found to have an average density close to theoretical density; however, EBM Ti6Al4V coupons had an average density that was significantly lower. The presence of porosity in the EBM Ti6Al4V coupons, which resulted from build-defects, such as incompletely melted/sintered regions and gas bubble inclusions [10], resulted in the reduced density of the EBM coupons. The average porosity of each EBM coupon can be calculated relative to the CMG coupons using Equation (2). The average porosity of the EBM coupons was found to be 3.21 ± 0.85 %. Porosity has been found to enhance bacterial contact and biofilm formation, which can lead to infections at implant sites [5]. Therefore, adjustment of EBM parameters to produce Ti6Al4V coupons with greater densities would be beneficial to reducing bacterial contact and biofilm growth.

$$P = \frac{D_{\text{comm}} - D_{\text{EBM}}}{D_{\text{comm}}} \times 100$$  \hspace{1cm} (2)

Mechanical polishing created scratches on the surfaces of coupons, due to abrasion with the polishing media. Grit papers with coarser particle sizes created deeper and wider scratches on the coupon surface compared to papers with finer particle sizes. Scratches on the surfaces of EBM Ti6Al4V coupons became less pronounced as polishing was performed with the 60 grit
paper (Figure 9B and Figure 11) to the 400 grit paper (Figure 9D and Figure 12). The most observable change in surface topography was seen between the unpolished EBM Ti6Al4V surface (Figure 9A and Figure 10) and the surface polished up to the colloidal silica suspension (Figure 9E), which had the characteristic “mirror” finish (Figure 14). Surface finishes on the EBM Ti6Al4V were comparable to those of CMG Ti6Al4V coupons polished using the sample parameters. Therefore, conventional metallographic surface preparation techniques can potentially be used for post-processing of EBM Ti6Al4V implant surfaces with the goal of preventing infection at implant sites.

In conclusion, this study evaluated density measurement and mechanical surface preparation of Ti6Al4V produced using the EBM process. The total average density of EBM Ti6Al4V was found to be lower than that of CMG Ti6Al4V. Mechanical surface preparation of EBM Ti6Al4V coupons produced surface topographies similar to those produced on CMG Ti6Al4V coupons polished to the same surface finish. Future work will include measurement of surface roughness values for the EBM Ti6Al4V and CMG Ti6Al4V coupons using confocal laser scanning microscopy.

**MILITARY RELEVANCE**

EBM Ti6Al4V cranial implants are used in the treatment of cranial defect injuries of warfighters wounded during recent operations in Iraq and Afghanistan. Occurrence and recurrence of infections at implant sites require multiple surgeries to replace implants resulting in prolonged periods of recovery, extended hospital stays, physical pain, and emotional distress to patients as well as increased costs to the military healthcare system. Extended hospital stays for patients may also lead to hospital acquired infections, which further prolong recovery. Implementing surface modification of implants to eliminate or reduce the occurrence of implant failure due to infections will promote enhanced healing and recovery of the wounded warfighters and enable them to resume active military service or begin active civilian lifestyles.
REFERENCES


Electron Beam Melting (EBM) is currently used to produce customized Ti6Al4V cranial implants for wounded warfighters who require cranial reconstruction surgeries due to loss of skull tissue. Infections occurring at implant sites may be due to the surface topography of the implants, which favor bacterial contact and adhesion. The objective of this study was to establish density determination and metallographic surface preparation procedures for EBM Ti6Al4V coupons. Densities of EBM Ti6Al4V coupons were measured using the Archimedes method and compared to those of commercial medical grade (CMG) Ti6Al4V coupons. Surfaces of EBM Ti6Al4V and CMG Ti6Al4V coupons were polished to 60 grit silicon carbide (SiC), 120 grit SiC, 400 grit SiC, and colloidal silica finishes and analyzed using scanning electron microscopy (SEM). Statistical analysis to compare density measurements of EBM and of CMG Ti6Al4V was performed using a t-test. The average density of EBM Ti6Al4V and CMG Ti6Al4V were 4.26 ± 0.04 g/cm³ and 4.41 ± 0.01 g/cm³, respectively. The difference in densities between EBM Ti6Al4V and CMG Ti6Al4V were statistically significant (p < 0.05). SEM analysis of polished EBM Ti6Al4V samples showed a progressive change in surface topography from polishing with 60 grit SiC paper to polishing with colloidal silica suspension. The density of CMG Ti6Al4V was close to the theoretical density (4.43 g/cm³). One factor contributing to an average lower density of EBM Ti6Al4V compared to CMG Ti6Al4V may be due to the presence of porosity, which is known to be introduced during the EBM process. Conventional metallographic surface preparation can be used to engineer the surface of EBM Ti6Al4V to produce different surface topographies.