Microstructure, defects and mechanical behavior of beta-type titanium porous structures manufactured by electron beam melting and selective laser melting

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Abstract

This study investigates the differences in the microstructure, defects and mechanical behavior of a β -type Ti-24Nb-4Zr-8Sn porous structures manufactured by electron beam melting (EBM) and selective laser melting (SLM). The phases, size and shape of melt pool, volume and distribution of defects are analyzed and correlated to the compressive and fatigue properties. Due to different powder bed temperatures, EBM and SLM samples consisted of $\alpha+\beta$ phases and a single β phase, respectively. The faster cooling rate during SLM promotes the formation of fine β dendrites, which leads to a higher compressive strength (50 ± 0.9 MPa) and lower Young's Modulus (0.95 ± 0.05 GPa) in comparison to the EBM parts (45 ± 1.1 MPa and 1.34 ± 0.04 GPa respectively). The large defects present within solid strut are likely a result of tin vaporization. The tin vapor is more easily trapped during the SLM process due to a

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smaller laser spot size and a faster cooling rate. This results in a 10 times increase in the number of defects. These defects have a limited influence on both the static properties and low stresses level fatigue strength, but it causes a reduced and variable fatigue life at high stresses level.

Keywords: Selective laser melting; electron beam melting; Titanium alloys; Mechanical properties; Porous structures.

1. Introduction

Recently, the demand for implants has been increasing as more people are suffering from joint problems caused by aging population and obesity [1]. It is therefore becoming necessary to produce high quality, artificial joints in order to reduce the risk of revision surgery. Several desirable requirements such as customized complex shape to fit the surrounding bone, interconnecting porosity with suitable size to facilitate bone in-growth, high strength and low Young's Modulus are needed to produce a successful implant [2]. Fortunately, additive manufacturing (AM) techniques such as selective laser melting (SLM) and electron beam melting (EBM), are emerging as advanced manufacturing technologies that are capable of manufacturing porous implants with optimal properties to meet these requirements, using medical grade metallic powder materials [3, 4]. These AM technologies, which build components using a layer-wise method directly from 3D CAD models, have attracted increasing interest in the past decade.

Compared to conventional processing methods, SLM/EBM can create

complicated geometries (such as porous structures) in a shorter time and with less cost [5]. The as-fabricated samples typically contain a finer, and often different, microstructure compared to those produced by conventional processing technologies. As such, the SLM/EBM-produced porous components have been reported to exhibit outstanding properties including low density, high strength, toughness and ductility [6-9].

Both EBM and SLM have similar working principle. A focused heat source selectively scans a powder bed. The scanned powder is melted and then rapidly solidifies. Once a layer is completed, the build platform descends by one layer thickness and a new layer of powder is deposited on top. The layer-by-layer process continues until the entire component has been completely produced [6]. The main difference between the two processes origin of the heat source used; EBM is equipped with a tungsten filament to generate electron beam while SLM uses a laser. In addition, there are difference in the working conditions between the two techniques, including the chamber pressure and the pre-heating procedure. These can significantly alter the microstructure of the samples manufactured by the two technologies [12].

As a result of its density, low Young's Modulus and high strength and corrosion resistance, titanium alloys are regarded as the most appropriate implant materials for load bearing applications [13, 14]. Currently, the majority of studies on AM-produced titanium alloys have been focused on the processing and mechanical properties of the traditional ($\alpha+\beta$)-type Ti-6Al-4V. Although it has been reported that the

SLM-produced Ti-6Al-4V porous structures exhibit high biocompatibility, good mechanical properties and good corrosion resistance [15], there is a concern that the toxic elements Al and V in Ti-6Al-4V might lead to allergic reaction and Alzheimer's disease [16]. Furthermore, the large mismatch in Young's Modulus between Ti-6Al-4V implants and the surrounding bone can lead to the well-known "stress-shielding" phenomenon [10]. In addition, α' martensite usually forms in AM-produced Ti-6Al-4V components, which is detrimental to their ductility and fatigue life [3] and decreases the corrosion resistance [17]. Therefore, it is imperative to find alternative titanium alloys to eliminate the above drawbacks.

β -type titanium alloys, such as Ti-29Nb-13Ta-4.6Zr, Ti-35Nb-5Ta-7Zr and Ti-24Nb-4Zr-8Sn (abbreviated as Ti2448), are attracting increasing research interest due to their advantages of low modulus and the presence of only non-toxic elements [13, 18]. For example, β -type Ti2448 exhibits a low modulus of ~42-50 GPa (compared with α + β -type titanium alloys ~100-120 GPa) coupled with high biocompatibility and mechanical properties [9, 19-21]. Ti2448 has been successfully manufactured into dense and porous components via both EBM and SLM [10, 22]. Ti2448 porous structures with designed porosity of 85% have been produced using SLM. These parts exhibited high relative density (~99.3%), low Young's Modulus (~1 GPa) and high compressive strength (51 MPa) [8]. Ti2448 solid parts obtained via EBM at a preheating temperature of ~200 °C consisted of large columnar grains aligned with the build direction and possess high hardness (~2.5 GPa) [22], which is higher than that of SLM-fabricated sample (~2.3 GPa) [10]. This suggests that EBM- and

SLM-produced components display significant differences in their microstructure.

However, there have been no reports on comparing the performance of porous β -type titanium alloys structures manufactured by EBM and SLM. Such work could further underpin the understanding of the influence of microstructure and defects produced by these two well-known methods on the resultant mechanical properties. In this study, a 75% porosity structure was made from Ti2448 by using both EBM and SLM. The phases were determined and melt pool characterized in terms of size and shape. The number and distribution of defects were analyzed and a mechanism for their formation is proposed. The role of the defects on compressive strength and fatigue property was also examined.

2. Experimental procedures

2.1. Powder material

The powder used was argon gas atomized from a Ti-24Nb-4Zr-8Sn ingot. The chemical composition of the powder is given in Table 1. The Ti2448 powder was spherical in shape (Fig 1 (a)), with a nominal particle size distribution between 45 and 106 μ m and an average particle size (d_{50}) of 80 μ m (Fig 1 (b)). Powder from the same batch was used for both SLM and EBM.

2.2. Electron beam melting process

The EBM samples were made using an Arcam A1 System, with a layer thickness of 70 μ m and a processing voltage of 60 kV. The build plate was preheated to 500 °C to avoid smoking during the process. Samples were manufactured directly on a

titanium substrate plate, which was maintained at a temperature between 450 and 500 °C by the electron beam. The whole build process was performed under a vacuum of 2×10^{-3} mBar, which was controlled via the injection of high purity helium. The input power was 180 W and electron beam scan speed, 130 mm/s, which has been shown to produce high density parts [20]. When the build process was completed, the as-fabricated samples were cooled to room temperature in the chamber.

2.3. Selective laser melting process

Selective laser melting was performed on a Realizer SLM 100 machine with a 200 W Yb:YAG fiber laser. The laser wavelength was 1.06 μm with a spot size of 40 μm . Prior to build samples, the chamber was purged with high purity argon until the oxygen content was $<0.1\%$ to minimize oxygen contamination. The laser speed was 750 mm/s and the power was 175 W, which have reported to result in the best properties [8]. The powder layer thickness was fixed at 50 μm and the scan spacing at 100 μm , while the substrate plate was heated to 200 °C. The scan strategy was set as zigzag pattern, the direction of which was rotated 90 ° between layers.

2.4. Microstructure and mechanical properties characterization

The 3D CAD model of a single unit cell created by Magics software (Materialise, Belgium) is shown in Fig. 2. This was a rhombic dodecahedron structure, with a porosity of 75%. Scaffold structures were produced using a $3 \times 3 \times 3$ array of unit cells; each unit cell was 3.33 mm $\times$ 3.33 mm $\times$ 3.33 mm in size (i.e. the overall part

was a 10 mm cube). The meshes were analyzed using a Zeiss Versa500 Micro-CT using an accelerating voltage of 120 kV and power of 10 W. 1600 projections were collected on a charge-coupled device detector using a 1s exposure time. The voxel size was 6.13 μm and, therefore, defects with equivalent diameter of $>\sim 20\ \mu\text{m}$ were able to be detected. Avizo 8.0 was employed to analyze the micro-CT data. The defect count and distribution were also studied base on the 3D raw data. X-ray diffraction (XRD; D/Max-2500PC source with Cu $K\alpha$ radiation), transmission electron microscopy (TEM; JEOL-2100; 200 kV) and electron probe microanalysis (EPMA-1610 microprobe, consisting of 4 tunable wavelength dispersive spectrometers conducted at 52.5 degrees angle, with a beam energy of 15 keV and current of 20 nA for a dwell time of 10 ms per pixel) were used to examine the phase constituent and elemental distribution of as-built Ti2448 parts. Microstructural features were also investigated by using an Olympus PMG-3 optical microscope (OM) and a JSM-6301F field emission scanning electron microscope (SEM). OM and SEM samples were etched using Kroll's Reagent (2 vol% HF, 6 vol% HNO_3 , and 92 vol% H_2O) for ~ 10 s. Compression testing was performed using an Instron 5869 machine at strain rate of 0.5 mm/min. Young's Modulus was also measured via compression testing. All mechanical property data shown were averages of at least 3 tests. The compression fatigue tests were carried out using an Instron 8800 machine at a stress ratio R of -0.1 and a frequency of 10 Hz in air at room temperature. Solution treatment of the samples was performed at 750 $^{\circ}\text{C}$ for up to 1 h, followed by air cooling. All specimens were sealed in vacuum tube during the whole heat treatment process. The

oxygen contents of the EBM as-produced and annealed specimens were measured with value of 0.19 wt% and those for the SLM as-produced and annealed specimens were 0.20 wt%.

3. Results

3.1. Phase analysis and microstructure

Fig. 3 compares the XRD patterns of the EBM and SLM samples in their as processed and annealed condition. As seen from the XRD patterns, the as-fabricated EBM sample contained both α and β phases. After annealing the EBM sample contained a single β phase, as does the SLM part for both conditions. The as-fabricated OM and SEM micrographs in the X-Y (i.e. build plane) and X-Z (i.e. build direction) plane are shown in Fig. 4. The different processing techniques result in differences in grain morphology and melt track characteristics. In the EBM samples, the α grains (labeled in the higher magnification images of Figs. 4 (b) and (d)) appear to originate from the β grain boundaries. The EBM samples also exhibit a continuous melt track (Figs. 4 (a) and (c)), whereas it was conical-shape in the SLM samples (Fig. 4 (g)). The size of the melt pool was measured in the as-processed material using optical microscopy as shown in Fig. 4. Ten measurements were conducted for each width and depth and found to be $\sim 280 \pm 23$ μm and $\sim 152 \pm 15$ μm , respectively, for the EBM sample and $\sim 146 \pm 17$ μm and $\sim 172 \pm 21$ μm , for the SLM parts. The SLM samples also contain small parallel dendrites with a width of ~ 1 μm within the β grains (Fig. 4 (h)). Vrancken et al. [23] suggested that such small elongated β grains

are generated by the steep thermal gradient that exists during the rapid solidification of the melt pool. The orientation of dendrites appears to be perpendicular to melt pool contour.

Defects (in the form of voids) were found in both EBM and SLM samples. It was observed that in the SLM samples, the defects were often located at the edge or bottom of the melt pools (Figs. 4 (e) and (g)) and were irregular in shape. In contrast, most defects in the EBM samples were spherical and distributed randomly (Fig. 4 (a)).

Fig. 5 displays the OM micrographs of samples annealed at 750 °C. A significant difference is observed between the EBM and SLM microstructures in terms of the size and shape of the grains. The shape of β grains in the annealed EBM samples was irregular and ~10-150 μm in size. By contrast, the β grains in the annealed SLM samples were squarer shaped and with a more uniform size, ranging between ~100 μm and ~140 μm .

3.2. Defect distribution

In order to quantify the defects, micro-CT was used to study the EBM and SLM specimens. The relative density calculated based on micro-CT 3D data was 99.94% and 99.63% for the EBM and SLM specimens, respectively. The micro-CT images of the single unit cell used for the EBM and SLM samples are shown in Fig. 6. Because of the resolution of the micro-CT, only those defects with size more than ~20 μm can be detected. As such, the actual relative density is likely to be lower than the value determined by micro-CT. It is apparent that the SLM samples have a smoother strut

surface than the EBM counterparts. Figs. 6 (c) and (d) show the location and distribution of the defects (marked in red) inside the solid struts. Nearly all the defects in the EBM samples were spherical in shape, while those made using SLM had an irregular shape, including some which had a conical shape. Fig. 6 (e) shows the defect distribution in the EBM and SLM specimens according to the Figs. 6 (c) and (d) respectively. It is noted that the number of defects inside the EBM samples is less than that of SLM samples; this is especially true for pores with an equivalent diameter (EqDiameter) of 30 to 90 μm . The EqDiameter indicates the equivalent diameter of the spheres which have the same volume as the measured defects.

Fig. 7 shows the EPMA maps elemental tin near a defect for both EBM and SLM samples. All the other elements had a uniform distribution and their maps are therefore not shown. It is clear that tin concentration is high on the inside surface of the pores. Fig. 7 (c) also shows that big the defects inside the SLM sample appear to have formed as result of the joining of two smaller defects.

3.3. Surface morphology

The temperature variation of plate during EBM and SLM process is plotted schematically in Fig. 8 (a). Based on the microstructure shown in Fig. 4, the schematic diagrams of EBM and SLM have been developed and are shown in Fig. 8 (b). This Figure illustrates the differences in the melt pool size, keyhole formation and defects generation between the two processes. It is apparent that the size of the melt zone during EBM is larger than that during SLM, while SLM results in a deeper

keyhole.

The surface morphology of the two methods is compared in the SEM images shown in Fig. 9 (a). Based on this, a schematic diagram comparing the two methods was developed and is shown in Fig 9(b). Both samples display a relatively coarse strut surface, with obvious “stair stepping” present. However, the surface of the EBM sample is rougher with more pronounced stair stepping.

3.4. Mechanical properties

The Young’s Modulus for as-fabricated and annealed samples is shown in Fig. 10 (a). In the as-fabricated state, the EBM samples had a higher Young’s Modulus ($\sim 1.34 \pm 0.04$ GPa) than the SLM-produced one ($\sim 0.95 \pm 0.05$ GPa). This is likely to have been a result of the presence of α phase in the microstructure of EBM samples (Fig. 3). After annealing, the Young’s Modulus of the EBM samples had decreased to $\sim 1.04 \pm 0.04$ GPa, which was very close to that of the SLM samples ($\sim 1.09 \pm 0.03$ GPa).

The typical compressive behavior of the EBM-/SLM- samples in the as-fabricated condition and after annealing is shown in Fig. 10 (b). It should be noted that only the as-fabricated EBM sample experienced a layer-wise fracture, while the other three samples exhibit completely ductile deformation.

In order to remove the effect of the presence of different phases, the fatigue performance of the scaffolds was measured on annealed samples, which both contained a single β phase, Fig. 10 (c). At lower stress levels (i.e. high cycle fatigue region), both manufacturing methods resulted in very similar fatigue strengths.

However, with increasing the applied stress, the fatigue life of the annealed SLM samples became worse and more variable than the EBM samples.

Fig. 11 shows the fatigue fracture surface morphologies for the highest stress level samples (applied stress of 12 MPa). It is apparent that the crack initiated from either the surface region which contained unmelted powder or from close to large defects (arrowed in Fig 11). The fatigue striations were also visible in most crack regions for both EBM and SLM samples. A large defect, greater than 100 μm in size (and marked by a dotted line) was also observed on the fracture surface of the annealed SLM samples. Such defects would significantly decrease the fatigue life of the samples.

4. Discussion

4.1 Microstructural comparison for the as-fabricated samples

It is known that both EBM and SLM manufacturing processes are very complex and highly dynamic, where the thermal and temperature distribution of the melt pool has significant effect on the resulting microstructure. However, until now, there is no accurate method to measure the temperature of melting zone in the two technologies [24]. Only the substrate temperature is measured using a sensor underneath the plate. From the temperature data collected from the plate, the powder bed temperature of processing area can be assumed. The plate temperature variation curves are plotted in Fig. 8 (a). As seen, there is a significant temperature difference in the substrate temperature during the melting phase between the two technologies. In this work, the

preheating temperature of $\sim 500\text{ }^{\circ}\text{C}$ (note that this temperature is material-specific) in the EBM process was much higher than that in the SLM process ($\sim 200\text{ }^{\circ}\text{C}$). During the EBM build phase, each molten layer was cooled relatively slowly due to the presence of a vacuum, and was kept between $400\text{--}500\text{ }^{\circ}\text{C}$ for several hours while subsequent layers were created. This is essentially identical to an aging treatment for Ti2448 alloy, which has been reported to result in the formation of α precipitates [20, 25]. This explains the presence of α phase in the as-fabricated EBM samples (Fig. 3). The initial α grains nucleate at the β grain boundaries and then grow toward the center of β grains. Although most α grains are distributed along the β grain boundaries, a small fraction of α grains are located in the interior of β grains (Figs. 4 (b) and (d)).

In contrast, the SLM samples were manufactured with a much lower substrate temperature of $200\text{ }^{\circ}\text{C}$. In addition, the Ar atmosphere within the build chamber facilitates convective cooling of the build surface [26]. Hence, the cooling rate of in SLM is much higher than that in EBM. The result of this higher cooling rate is that α formation is suppressed and the as-fabricated samples consist of a single β phase. Interestingly, the fine parallel dendrites form are aligned with the direction of the heat flow and are uniformly distributed (Fig. 4 (h)). As the α phase increases the Young's Modulus of titanium alloys [18], as-fabricated EBM samples have a higher modulus ($\sim 1.34 \pm 0.04\text{ GPa}$) than the SLM ones ($\sim 0.95 \pm 0.05\text{ GPa}$).

According to our previous work for Ti2448 alloy, the α phase can dissolve after annealed treatment at $750\text{ }^{\circ}\text{C}$ [27]. Accordingly, both EBM and SLM parts consist of a single β phase (Fig. 5) after annealing at 750°C for 1h. Dissolution of the α is most

likely the reason for the drop of the Young's Modulus of the EBM parts after annealing. The similarity of the EBM and SLM microstructure after annealing also explains their near identical Modulus values. The Young's modulus of SLM annealed specimen increases slightly, most likely as a result of a change in the β grain size variation and dendrite elimination.

Fig. 8 (b) schematically displays the process of EBM and SLM. There are several important differences in the characteristics of the heat source between EBM and SLM. The main differences are the input energy and momentum of the electron beam or the laser. Both of these can directly affect the size of melt pool and the volume of defects. Following the suggestion of Simchi [28], the energy density (Q) can be affected by processing parameters:

$$Q = \frac{\pi\eta P}{4dv} \quad (1)$$

where P is the input power, η is the coupling efficiency, d is the diameter of electron/laser beam spot and v is the scan speed. All the values of the parameters used in the EBM or SLM process are summarized in Table 2. According eq. (1), the energy density in the EBM process is greater than that in the SLM process. Associated with higher initial temperature of EBM powder bed, the pre-heating procedure and the spot size, the electron beam melting area of single scanning track is greater than that of laser beam. This result explains a larger value of scan track width ($280\pm 23 \mu\text{m}$) in EBM than that in SLM ($146\pm 17 \mu\text{m}$) (Figs. 4 (a) and (e)).

It is reported that the formation of a keyhole has a significant effect on the melt zone depth [9, 29-31]. Semak et al. [32] showed that a recoil pressure forms as a result

of metal vaporization and acts to push the liquid away from the melt zone, thereby generating a keyhole. The electron/laser beam can be reflected by the inner surface of the keyhole and the power is then concentrated at the bottom of keyhole (this process is shown in Fig. 8 (b)). This causes a higher temperature at the bottom of the melting pool than the top. Thus, it is possible for the electron/laser beam to penetrate deeply into the material. In this work, the laser beam has a similar power but has a much smaller spot size than the electron beam. Therefore, it is possible for the laser beam to penetrate further, causing a deeper melt pool ($172\pm 21\text{ }\mu\text{m}$ for SLM versus $152\pm 15\text{ }\mu\text{m}$ for EBM).

4.2 Defect and surface roughness generation

The defects generated during EBM/SLM process will affect the performance of the samples. The mechanism of defects formation during additive manufacturing process is complex and not yet fully understood [24]. For most metallic powders, the reported possible reasons for defects formation include powder defects [33], insufficient energy [10, 34], balling [31, 35], material vaporization [36] and imperfect collapse of the keyhole [37, 38]. Gong et al. [39] pointed out that the defects can have a negative influence on the tensile and fatigue properties of EBM/SLM-produced samples. Therefore, a detailed understanding is needed on mechanism(s) of defect formation.

During the EBM/SLM build process, the heat source has an approximate Gaussian distribution and is focused on the powder bed [40, 41]. There are many

physical effects occurring inside the melting zone including the interactions between solid, liquid and gas phases [42, 43], and the evaporation of the material which generates a recoil pressure and vapor capillary (named keyhole) [38, 44]. For Ti2448 alloy, the boiling point of each element is different. Of most interest is tin as it has the lowest boiling point of the elements used (2600 °C) and would therefore be the first to vaporize. Indeed, high concentrations of tin have been detected on the surface of pores by using EPMA (Fig. 7), indicating that the vaporization of tin is playing a critical role in the formation of these pores.

Since the shape of the defects differ between EBM and SLM (Fig. 6 and 7), it seems reasonable to assume that the defect formation mechanism is also different. EBM samples tend to contain spherical defects, whilst the SLM sample contains both conical and spherical pores. It has been reported [33] that in EBM, spherical voids are related to both defects in powder used and metal vaporization. Similarly, there are several possible mechanisms of defect formation in SLM samples [8]. Liu et al. [45] pointed out that the intensity of spatter (ejected material) fluctuates periodically and can interact with the laser radiation, resulting in shadowing of the laser and defect formation. Similar to the EBM samples, the defects formed during SLM contained a high concentration of tin on their surface (Fig. 7). Hence, it is likely that the vaporization of tin plays a key role in the formation of these conical-shaped defects.

During powder melting and solidification process, Geiger et al. [42] revealed that the balance of recoil pressure (metal vapor pressure) and the surface tension of liquid metal is the main factor that determines defect formation. In addition to this, the

energy input, temperature distribution and cooling rate can also affect the depth of the keyhole and the position of defects [38, 46, 47].

During both SLM and EBM, the temperature of melt pool increases rapidly as a result of the high energy input, increasing the vapor pressure of molten metallic liquid. The apparent melting temperature (T_m) can be determined by the processing parameters as followed [28]:

$$T_m = T_p + \frac{1}{c} \left[\left(\frac{\pi \eta}{4 \rho} \right) \left(\frac{P}{h v w} \right) - \Delta H \right] \quad (2)$$

where T_p is the preheating temperature, C is the specific heat capacity, ρ is the density of the powder, h is hatch space, w is the layer thickness and ΔH is the enthalpy of the powder.

The deformation of melt pool and formation of a keyhole is determined by recoil pressure [47, 48]:

$$P_V(T) = P_o e^{\frac{\Delta H_m}{R} \left(\frac{1}{T_v} - \frac{1}{T} \right)} \quad (3)$$

where the P_o is the recoil pressure at the boiling temperature T_v , R is the ideal gas constant, and ΔH_m is the enthalpy change due to vaporization.

The relationship between the temperature and the surface tension is defined by Ramsay-Shields' equation [49]:

$$\gamma V_m^{2/3} = K(T_b - T - 6.0) \quad (4)$$

where γ is the surface tension, V_m is the molar volume, K is the universal constant, T_b is the powder boiling temperature.

Eqs. (2)-(4) indicate that a larger energy input leads to a faster temperature rise thereby resulting in a greater recoil pressure. The surface tension will also drop with

increasing temperature and therefore tin vaporizes more easily from the melt pool during EBM under vacuum environment. Such defects tend to be spherical shape. On the other hand, SLM has a lower energy density which results in a lower temperature in melt zone. Furthermore, energy concentrates the underpart of keyhole due to ray reflection (Fig. 8(b)) and therefore the lower part of the melt pool will be at a higher temperature. Thus the tin vapor is more likely to be generated at the bottom of melt pool. If the surface tension is larger than the recoil pressure (tin vapor pressure), tin vapor bubbles are trapped, thereby generating defects [46]. Due to the temperature distribution in the melt pool, it is likely that that solidification occurs from the top of melt pool; the bottom region will be at a higher temperature and will solidify last. The melt pool boundary created by laser beam during melting process has a conical shape in the X-Z plane. As the tin vapor bubble is kept at the bottom of the melt pool, its shape will be influenced by the shape of the melt pool, which is conical. As such, the defects that form during SLM process usually occur in the lower part of the melt pool and are conical in shape.

The surface roughness is mainly determined by the heat source spot size and the layer thickness. The schematic diagram in Fig. 9 clearly displays the melting process for both EBM and SLM. The electron beam spot size (200 μm) is much larger than the laser spot size (40 μm), which leads to a larger melt pool of EBM than that of SLM. Consequently, the EBM as-produced surface with 70 μm layer thickness is rougher than that of SLM with 50 μm layer thickness.

4.3 The mechanical properties

The uniaxial compression testing results show a significant difference between the as fabricated EBM and SLM (Fig. 10 (b)). The as-fabricated SLM samples exhibit excellent compressive properties, i.e. high ultimate stress of $\sim 50 \pm 0.9$ MPa and good ductility. The parallel fine dendrites that are present inside β grains (Fig. 4 (h)) are similar to the previous observations of nanoscale growth twins [50] and strengthen the β titanium via preventing dislocation motion cross the numerous coherent dendrites. In contrast, the as-fabricated EBM samples have a lower compressive stress of $\sim 45 \pm 1.1$ MPa due to coarser β grains despite the presence of hard α phase precipitate in the microstructure. Both EBM and SLM samples annealed at 750 °C are composed of a single β phase (Figs. 3 and 5) and exhibit a very similar compressive strength $\sim 42 \pm 0.5$ MPa and $\sim 41 \pm 1.1$ MPa for the EBM and SLM samples respectively (Fig. 10 (b)). Except for the as-produced EBM sample whose ductility is reduced due to the presence of α , the traditional layer-wise failure mechanism of the scaffolds does not occur. Instead, failure occurs via the gradual plastic deformation of the struts. This indicates that this material has very high damage tolerance. In addition, the high strength and ductility of the SLM sample (despite it containing the highest level of defects) indicates that the defects have a limited influence on the static properties. Similar results have been reported in our previous study [8].

For long term application of metallic cellular structures in the human body, it is important to understand their fatigue behavior. Recently there has been some work aimed at studying the fatigue behavior of EBM and SLM meshes. However, these

have been focused on commercially pure titanium or Ti-6Al-4V meshes [3, 21, 51-54]. For a given porous structures, the factors that influence the fatigue behavior of AM-produced titanium meshes mainly include surface roughness, internal defects, microstructure and loading condition of the struts. The underlying fatigue mechanism of the meshes is the interaction of cyclic ratcheting and fatigue crack growth on the struts and the former is shown to be the dominant mechanism [3]. For these two mechanisms, the cyclic ratcheting is closely related to the microstructure and loading condition of the struts, while the surface roughness and internal defects in the struts mainly contribute to the fatigue crack initiation in the struts [55]. For the annealed SLM and EBM samples in this work, the surface roughness, cell shape and microstructure are similar; the only significant difference is the distribution of porosity. Thus the variation of fatigue behavior between the annealed SLM and EBM samples can be related to this difference in the characteristics of the pores contained within the struts.

The effect of pores on the fatigue behavior of dense metallic materials has been investigated extensively. The prominent role of porosity is that it acts as a preferential crack initiation site [56-58]. The number of cycles to failure for a given stress level can be correlated to the size of the pores at the crack initiation site [58]. If the porosity is considered equivalent in size to an initial crack, the probability for failure at different stress amplitudes is determined by critical stress intensity amplitude, K_{cr} [58-60]. Specimens fail if critical stress intensity amplitude K_{cr} is reached. Below K_{cr} , fatigue cracks may initiate at the porosity but do not propagate until failure. The

statistical distribution of defects is linked to the fracture probability at different stress amplitudes. In this work, at the lower stress level (i.e. high cycle fatigue region), the stress intensity around pores does not reach the K_{cr} and the fatigue crack does not propagate. Thus the cracks initiate and propagate from the much sharper notches in rough surface [58, 60]. In this case, the fatigue behavior of the meshes is mainly determined by the cyclic ratcheting and surface properties of the struts. Thus, at low stress levels, the annealed SLM and EBM samples have similar fatigue strengths. However, at high stress levels, the probability of crack initiation and propagation from the pores contained in struts become high. As such, since the SLM samples have a higher porosity level, they exhibit a lower and more variable fatigue life compared to the EBM samples. Such behavior is the typical fatigue characteristics of metallic alloys with high porosities [59]. The above results indicate that for the reticulated meshes containing pores in the struts, the fatigue behavior is sensitive to the stress level. If the stress is high enough to initiate the crack propagation from the pores, then the fatigue life will be closely related to the porosity contained in the mesh struts.

5. Conclusion

In this work, a β -type biomedical Ti2448 alloy powder was used to fabricate porous structures via electron beam melting (EBM) and selective laser melting (SLM). The microstructure, defects and mechanical properties were systematically studied and analyzed. Based on these experiments, the results are summarized as follows.

1. Due to the high temperature ($\sim 500^\circ\text{C}$) of powder bed and relatively slow cooling

rate during EBM, the as-fabricated parts form an $\alpha+\beta$ microstructure, which increases the Young's Modulus of the porous samples ($\sim 1.34 \pm 0.04$ GPa). In contrast, low substrate plate temperature (~ 200 °C) and high cooling rate results in the formation of very fine β phase dendrites in the SLM porous samples, reducing the Modulus of the structure to $\sim 0.95 \pm 0.05$ GPa.

2. The laser beam spot size of ~ 40 μm is much smaller than the electron beam spot of $\sim 200\mu\text{m}$ which leads to differences in width and depth of the melt pool. The width of melt pool in the EBM sample ($\sim 280 \pm 23$ μm) is larger than that in SLM ($\sim 146 \pm 17$ μm). This is a result of the higher pre-heating temperature and energy density input in EBM process. The smaller spot size of the SLM process results in a deeper keyhole caused by radiation reflection, thereby leading to a deeper melt pool ($\sim 172 \pm 21$ μm) in SLM than that in EBM ($\sim 152 \pm 15$ μm).
3. The porosity formed within both EBM and SLM samples is mainly caused by the tin vaporization during the melting process. Due to continuous scan track, the as-fabricated samples by EBM contain defects in a spherical shape. The defects with a conical-like shape in SLM-produced samples are caused by the discontinuous conical shaped melt pool. Smaller recoil pressures and higher surface tension inside the deeper keyhole during SLM tends to promote defect formation. Hence the number of defects in the SLM samples was ~ 10 times that of EBM samples.
4. The defects in the EBM and SLM samples appear to have limited impact on the compressive properties of the parts. The fine single β phase dendrites in the

as-fabricated SLM samples results in high compressive strength ($\sim 50 \pm 0.9$ MPa).

During annealing of the SLM samples at 750 °C, the β grains coarsen and the fine dendrites dissolve. This decreases the strength of the SLM samples by nearly 20%.

For the EBM samples, annealing dissolves the α phase, which, along with grain growth, decreases the strength.

5. The fatigue strength of the annealed EBM or SLM samples is sensitive to the stress amplitudes. At the lower stress levels, the fatigue behavior of the meshes is mainly determined by the cyclic ratcheting and surface properties of the struts, resulting in similar properties for both manufacturing processes. However, at higher stress levels, the crack initiation and propagation from the pores tends to occur and therefore the SLM samples (which contain a higher number of defects) have a lower and more variable fatigue life.

Acknowledgement

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Table

Table 1 The chemical composition and particle size of Ti2448 powder used for additive manufacturing

Composition (wt%)					Particle size(μm)		
Ti	Nb	Zr	Sn	O	d ₁₀	d ₅₀	d ₉₀
Bal	23.9	3.90	8.20	0.19	47.2	79.4	130.2

Table 2 The processing parameters used in the EBM and SLM

Sample	Power(W)	Scan speed (mm/s)	Electron/laser spot size (μm)	Layer thickness (μm)	Initial temperature ($^{\circ}\text{C}$)	Hatch space (μm)	Chamber pressure (mBar)
EBM	180	150	200	70	500	200	2.0×10^{-3}
SLM	175	750	40	50	200	100	10

Figure Captions

Fig. 1 (a) The morphology and (b) particle size distribution of the as-received Ti–24Nb–4Zr–8Sn powder.

Fig. 2 The CAD single unit cell of the rhombic dodecahedron used.

Fig. 3 The XRD patterns of the EBM and SLM-produced samples in the as-fabricated condition and annealed for 1h at 750 °C.

Fig. 4 The OM and SEM micrographs of at X-Y cross-sections and X-Z cross-sections for the EBM and SLM samples.

Fig. 5 The OM micrographs of the annealed (a) EBM and (b) SLM samples.

Fig. 6 The micro-CT reconstructed images showing the strut outside surface of the (a) EBM and (b) SLM samples, the defects inside of the solid struts of the (c) EBM and (d) SLM samples, and (e) the size and count distribution of the defects inside the samples as a function of equivalent diameter.

Fig. 7 (a) and (c) The SEM microstructure, and (b) and (d) EPMA quantitative chemical analysis maps for elemental tin near a defect for the EBM and SLM samples. Tin appears to concentrates on the surface of the defects.

Fig. 8 (a) Temperature variation of the substrate plate during EBM and SLM process, and (b) the schematic diagram of the EBM and SLM melting process.

Fig. 9 The SEM surface morphologies of EBM and SLM samples and their melting schematic diagram.

Fig. 10 (a) The Young's Modulus, (b) the typical compressive stress–strain curves for EBM or SLM samples, and (c) the fatigue of samples annealed at 750 °C for 1 hour.

Fig. 11. The fatigue fracture morphologies for the high stress level of the annealed EBM- and SLM-produced samples.

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SLM	175	750	80	40	50	250	100	1.0×10^4

Figure Captions

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(a)

(b)

Fig 1. Y.J. Liu *et al*

Fig 2. Y.J. Liu *et al*
Needs scale bar

Fig 3. Y.J. Liu *et al*

Fig 4. Y.J. Liu *et al* replace boards with boundary

Fig 5. Y.J. Liu *et al* These are not great images – add EBM and SLM to (a) and (b) respectively. What is the rectangle in the middle of (b)?

Fig 6. Y.J. Liu *et al*

Fig 7. Y.J. Liu *et al*

(a)

(b)

Fig 8. Y.J. Liu *et al*

(a)

(b)

Fig 9. Y.J. Liu *et al*

(a)

(b)

(c)

Fig 10. Y.J. Liu *et al*

Fig 11. Y.J. Liu *et al*