Title: Selective Laser Melting of Titanium Alloy with 50 wt% Tantalum: Effect of Laser Process Parameters on Part Quality

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Abstract:

Selective laser melting (SLM) is a powder bed fusion additive manufacturing (AM) technique that produces three-dimensional (3D) parts by fusing metallic powders with a high-energy laser. SLM involves numerous process parameters that may influence the properties of the final parts. Hence, establishing the effect of the SLM processing parameters is important for producing parts of high quality. In this study, titanium-tantalum alloy was fabricated by SLM using a customized powder blend to achieve *in situ* alloying. The influence of processing parameters on the microstructure and properties such as relative density, microhardness and surface roughness was investigated. The results show that fully dense titanium-tantalum parts can be obtained from SLM. With laser power of 360 W, scan speed of 400 mm/s, powder layer thickness of 0.05 mm and hatch spacing of 0.125 mm, the titanium-tantalum alloy produced by SLM has relative density of 99.85 ± 0.18 %. Despite the variation in process parameters, titanium-tantalum shows laminar β grains in random directions in both xy and yz-plane from optical microscope (OM) analysis in all the parts produced. This observation is further confirmed using x-ray diffraction (XRD).

1. Introduction

Selective laser melting (SLM) is a powder bed fusion additive manufacturing (AM) technique [1]. AM is also commonly known as 3D printing. SLM uses laser as energy source to completely melt and fuse powder material to form parts that can be fully functional directly. Details of SLM have been described previously [2, 3]. The significant SLM process parameters have been identified, with the major controlling parameters being scan speed, hatch spacing, laser power and powder layer thickness [4]. These are shown in Fig. 1.

Fig. 1. Controlling parameters in SLM process

Due to the numerous variable parameters involved in SLM, several studies have been done on the effect of process parameters on the properties of SLM fabricated parts. Kasperovich *et al.* studied the correlation between the part porosity and process parameters in Ti6Al4V fabrication. It is concluded that SLM part porosity can be significantly reduced by optimizing the processing parameters. Excessive energy density results in circular or spherical pores while insufficient energy density results in elongated voids, similar to narrow cracks, oriented perpendicularly to the build direction [5]. Similar conclusions were also obtained by other studies [6, 7]. Despite the studies conducted, little is known about how the SLM process parameters affect part properties fabricated from powder mixtures. This is because SLM has been pre-dominantly using pre-alloyed powder as feedstock, and the opportunity to use powder mixtures has only been recently explored [8, 9]. Whenever a new material is introduced to be processed by any powder bed fusion system, there must be identification of the process parameters so as to manufacture components with appropriate mechanical properties and surface finish [10].

Titanium alloys are superior biomedical materials due to their excellent properties such as high mechanical strength, high corrosion resistance, good biocompatibility and low density. There has been extensive research done on SLM processed titanium alloys [3, 11, 12]. For biomedical applications, Tantalum is a superior choice for alloying with titanium as like titanium, it has high biocompatibility, corrosion resistance and good mechanical properties. Furthermore, tantalum is a β stabilizing element for titanium alloys. SLM processed β titanium alloys display superior properties with lower modulus [8, 9, 13]. As such, titanium-tantalum (TiTa) alloys are promising materials for biomedical applications because of high strength to modulus ratio.

In this paper, titanium-tantalum parts were manufactured using SLM with various combinations of process parameters. The feedstock used is a blend of commercially pure titanium and tantalum

powder with equal weight percentages. Microstructures of the SLM produced titanium-tantalum parts under the effect of different process parameters were analysed and discussed. Their relative density and mechanical properties, such as microhardness and surface roughness, were also investigated.

2. Experimental procedure

2.1. Powder preparation

Gas atomized commercially pure titanium and tantalum powders are used. The commercially pure titanium powder particles (Grade 2 ASTM 348, LPW Technology Ltd, United Kingdom) have average particle size of 43.5 μm and are spherical in shape. The tantalum powder particles (Singapore Demand Planner Ltd, Singapore) have average particle size of 44 μm and are irregular in shape. The two powders were mixed at a rate of 60 rpm for 12 hours using a tumbler mixer (Inversina 2L, Bioengineering AG) with weight ratio of 1:1.

2.2. Selective laser melting

Fabrication of all samples was carried out using a SLM 250HL machine (SLM Solutions Group AG, Germany). The SLM machine is equipped with a Gaussian beam fiber laser with maximum power of 400 W and a focal diameter of 80 μm . All processing is done in argon environment with less than 0.05 % oxygen to prevent oxidation and degradation of the material during the fabrication process [14]. Sectorial, also known as island or chessboard, scanning is used to minimize thermal stresses formed during the process [15, 16].

Energy density, E_D , is often used as a metric to quantify the different process parameters needed to fabricate parts using SLM and it is given by the equation [17-19]:

$$E_D = \frac{P}{v \cdot h \cdot l} \quad (1)$$

where P is the laser power (W), v is the scan speed (mm/s), h is the hatch spacing (mm) and l is powder layer thickness (mm).

In order to study the effect of different parameters on the resulting properties of titanium-tantalum parts, the combinations of process parameters used are shown in Table 1.

Table 1 Combinations of process parameters

	Laser Power, P (W)	Scan Speed, v (mm/s)	Layer thickness, l (mm)	Hatch spacing, h (mm)	Energy density, E_D (J/mm ³)
1	120	400	0.05	0.125	48
2		800			24
3		1200			16
4	240	400			96
5		800			48
6		1200			32
7	360	400			144
8		800			72
9		1200			48

The layer thickness is fixed due to the powder size distributions. The hatch spacing was fixed as it was found to be the least sensitive in affecting the part quality [5]. Build direction is along the z-axis (ISO / ASTM52921 – 13, Standard Terminology for Additive Manufacturing - Coordinate Systems and Test Methodologies), as shown in Fig. 2.

Fig. 2. Build orientation of samples fabricated using SLM

2.3. Parts characterization

The density of the as-built SLM samples was measured using Archimedes' Principle (XS204, Mettler Toledo, Switzerland). The method of using Archimedes' Principle to measure density is

detailed in ASTM B311-17 Standard Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity. Relative density, $\rho_{relative}$ of the samples are calculated using the equation:

$$\rho_{relative} = \frac{\rho_{absolute}}{\rho_{theoretical}} \times 100 \% \quad (2)$$

Where $\rho_{absolute}$ is the measured density and $\rho_{theoretical}$ is the calculated density, taken to be 7.10 g/cm³ [8].

Energy-dispersive x-ray spectroscopy (EDS, Oxford Instruments) was used to ascertain the chemical composition of the as-built SLM samples. The weight percentage (wt%) of tantalum in the titanium-tantalum parts was determined.

Surface roughness of the as-built samples is measured using laser scanning (VK-X130K, Keyence Corporation, Japan).

The SLM samples were subjected to standard metallographic procedure which involves grinding with 320, 800 and 1200 SiC papers and then polished by diamond suspensions of 9, 3 and 1 μ m sizes. The samples were then etched with Kroll's reagent (ASTM E407). The microstructure study was conducted using optical microscopy (OM, VK-X130K, Keyence Corporation, Japan) and X-ray diffraction (XRD, Empyrean, Panalytical, Netherlands). The microhardness test of the samples was carried out using Vickers hardness test (FM-300e, Future-Tech Corp., Japan) under 1 kg of load with 10 s load time. Ten readings at randomised position of each surface of the polished samples were measured.

3. Results and discussion

3.1. SLM part relative density and melted tantalum content

The process parameters are shown to influence the part relative density and its variation is shown in Fig. 3.

Fig. 3. Variation in relative density of SLM TiTa samples with (a) scan speed (b) laser power (c) energy density

It is observed that the variation in relative density is higher for lower energy density values. This is attributed to the instability of melt tracks formed during the SLM process. This lower energy

densities also lead to insufficient melting and lack of fusion between the layers. Similar energy density can be achieved by very different combinations of laser power, scan speed, hatch spacing and powder layer thickness. However, the processing parameters have interdependent relationships which can cause the use of energy density alone to be misleading [5, 20]. Nonetheless, it gives a good quantity for comparison of results for a complex process such as SLM.

From the results, it can be observed that lower scan speed and higher laser power lead to higher relative density. At $P = 360$ W, $v = 400$ mm/s, $l = 0.05$ mm and $h = 0.125$ mm, the SLM produced titanium-tantalum are fully dense with relative density of 99.85 ± 0.18 %. However, at lower scan speed, the laser beam dwelling time on the melt pool increases, resulting in higher energy input into the melt pool. This leads to higher temperature of the melt pool, which leads to higher viscosity of the overheated liquid. The instability of the melt pool increases, and liquid droplets tend to splash from the liquid front that is being solidified. This is due to reduction in the surface energy of the liquid at a shorter length scale, resulting in balling [21]. The insufficient melting and balling result in pores formation within the SLM titanium-tantalum samples, as shown in Fig. 4. It is seen that there are traces of relatively large pores and unmelted tantalum particles in the samples.

Fig. 4. OM images of SLM TiTa samples in xy-plane (a) 120 W, 1200 mm/s, 50x (c) 240 W, 800 mm/s, 150x (e) 360 W, 400 mm/s, 150x and yz-plane (b) 120 W, 1200 mm/s, 50x (d) 240 W, 800 mm/s, 150x (f) 360 W, 400 mm/s, 150x

The variation of melted tantalum in the SLM titanium-tantalum parts to process parameters is shown in Fig. 5.

Fig. 5. Variation in melted tantalum composition in SLM TiTa samples with (a) scan speed (b) laser power (c) energy density

From the results, it can be observed that lower scan speed and higher laser power lead to higher melted tantalum content in the samples. As EDS is carried out in the titanium-tantalum matrix, it is inferred that the difference between the powder blend composition and melted tantalum content is the amount of unmelted tantalum particles. At $P = 360$ W, $v = 400$ mm/s, $l = 0.05$ mm

and $h = 0.125$ mm, the SLM titanium-tantalum produced has melted tantalum content of 47.23 ± 0.88 wt%. This is closest to the tantalum content of the powder mixture. It is expected that a further increase in energy density can lead to further increase in melted tantalum content, due to the high melting point of tantalum (3020°C). However, a further increase in energy density can also lead to increase in vaporization of titanium during SLM process, which results in material wastage [22].

There is presence of particles in the material due to the incomplete melting of tantalum. In general, secondary phase particles can lead to several advantages in alloys. For example, small and hard particles are used in composites to increase the strength and hardening of the materials [23, 24]. Hence, in the case of tantalum particles, it is possible to lead to an increase in strength and hardness of TiTa alloys as the tantalum content increases. However, Vrancken *et al.* concluded that unmelted molybdenum particles have no visible effect on the mechanical properties of the SLM parts when processed using a blend of Ti6Al4V and molybdenum powders [13]. Hence, there is a need for further study on the effect of these particles on the performance of the materials.

3.2. Microstructure of titanium-tantalum parts

XRD was performed on the samples with energy density of 16, 48 and 144 J/mm^3 . The XRD spectrums are shown in Fig. 6.

Fig. 6. XRD patterns of SLM TiTa with different energy densities

After the SLM process, the peaks of β -titanium and tantalum can still be observed for the chosen range of energy densities applied during the SLM process. At ambient temperature, titanium exists as hexagonal close packed (HCP) structure, i. e. α -titanium. During the SLM process, the temperature is raised to be above 883 °C, which is the phase transformation temperature for pure titanium, resulting in α to β transformation. Due to tantalum being a beta stabilizer, the body centered cubic (BCC) β crystal structure is retained during the rapid cooling and solidification of SLM process. The stability of the BCC structure depends on the amount of alloying elements, and is given by the Molybdenum Equivalency, Mo_{eq} [13].

$$Mo_{eq} = 1.0Mo + 0.67V + 0.44W + 0.28Nb + 0.22Ta + 1.6Cr + \dots - 1.0Al \quad (3)$$

Based on the Molybdenum Equivalence and the tantalum content, the titanium-tantalum alloys formed are classified as shown in Table 2.

Table 2 Conditions of β titanium in SLM TiTa with different energy densities

Energy density (J/mm ³)	Tantalum content (wt%)	MO _{eq}	
16	23.80 ± 1.71	5	Unstable
48	42.23 ± 5.05	9	Close to metastable
144	47.23 ± 0.88	10	Metastable

The addition of tantalum in the alloy suppresses the transformation of β phase to the α' phase despite none of the β phase being fully stable. Coupled with the rapid solidification during SLM process, the β phase are retained in all the samples. However, the samples fabricated with low energy density shows β island structure instead of β laminae due to the lower tantalum content within the alloy, which restricted laminae formation. The samples fabricated using high energy density exhibited similar microstructure which shows β laminae in the SLM titanium-tantalum, as described in previous study [8]. As the content of tantalum increases, the critical cooling rate to retain β phase decreases, which favors the β laminae formation instead of β island formation. Furthermore, the addition of tantalum in titanium suppresses the transformation of β to α' phase by lowering the martensitic start temperature. Hence, all the SLM titanium-tantalum samples exhibit β phase structures.

3.3. Microhardness of titanium-tantalum parts

The microhardness of SLM TiTa samples in the xy- and yz-planes are shown in Fig. 7.

Fig. 7. Variation of microhardness of SLM TiTa samples in xy-plane with (a) scan speed (c) laser power (e) energy density and yz-plane with (b) scan speed (d) laser power (f) energy density

Similar observations are found for both xy- and yz-planes. Slow scan speed and high laser power result in higher microhardness, which corresponds to the trend obtained for relative density of the samples. It was concluded that lower porosity in the sample will result in higher microhardness, which will lead to higher strength [25]. It was suggested that highest microhardness can be achieved by ω -titanium, followed by α' -titanium, α'' -titanium, β -titanium and lowest microhardness is achieved from α -titanium [26]. From the XRD results, it is concluded that all the samples exist only as β -titanium, hence, the variation in microhardness can be due to the difference in porosity of the samples, as well as due to the variation of tantalum content in the samples.

3.1. Surface roughness of titanium-tantalum parts

The surface roughness of the SLM TiTa samples for the xy- and yz-planes are shown in Fig. 8.

(a)

(b)

(c)

(d)

(e)

(f)

Fig. 8. Variation of surface roughness of SLM TiTa samples in xy-plane with (a) scan speed (c) laser power (e) energy density and yz-plane with (b) scan speed (d) laser power (f) energy density

It is observed that in the xy-plane, an increase in scan speed or decrease in laser power will lead to an increase in surface roughness. However, no observable trend is obtained for these two parameters in the yz-plane. This is due to the melt pool formation. In the xy-plane, the melt pool dimension is significantly affected by laser power and scan speed. The specimens processed with high laser power and low scanning speed have more stable melt pools. This results in continuous scan tracks, and hence, lower surface roughness [10]. Furthermore, at high energy density, the melt pool width increases, which create a recoil pressure that minimize the surface tension in the melt pool. This enhances the flattening effect on the solidified surface [27]. Furthermore, as the melt pool width increases, the overlap between each scan tracks increases, more remelting occurs, which improves the surface finish [28].

A contradictory observation is seen in the yz-plane. In the yz-plane, as the energy density increase, it increases the melt pool depth, and this leads to greater variation in the thermal properties of the melt pool. Furthermore, it creates a larger difference in the surface tension across the melt depth [27, 29]. As the melt pool tries to decrease the surface tension differences, it causes the melt pool to separate and transformed into smaller balling (coalesce). Hence, these small balling solidifies at the side surface of the melt pool which causes the surface roughness in yz-plane to increase [30]. The increase in surface roughness can also be due to partially fused particles that adhere to the yz-plane of the part [31]. Furthermore, the surface finishing in yz-plane is also affected by the layer thickness more significantly compared to xy-plane. Hence, there is difference in the trend observed between the two planes.

As a result, proper optimization of the process parameters is needed to control the composition of alloy formed by SLM using powder mixtures and their part properties.

4. Conclusions

This study shows that TiTa alloy can be formed *in-situ* successfully using SLM. Fully dense titanium-tantalum alloy can be obtained using the SLM process parameters shown in Table 3.

Table 3 Optimized SLM process parameters for TiTa

Process Parameter	
Laser Power (W)	360
Scan speed (mm/s)	400
Layer thickness (mm)	0.05
Hatch spacing (mm)	0.125

Based on the results, the key findings can be summarised:

- 1) Laser power and scan speed have significant effect on the density of SLM titanium-tantalum samples. High energy density is needed to achieve full dense parts. As a result, the SLM process parameters have effect on the microhardness of the as-built titanium-tantalum samples due to their influence on the porosity of the samples.
- 2) Laser power and scan speed have significant effect on the tantalum content of SLM TiTa samples. High energy density is needed to fully melt the tantalum in the powder mixture

to obtain SLM titanium-tantalum parts that have same composition as the powder mixture.

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