## INFLUENCE OF PROCESS PARAMETERS ON LAYER BUILD-UP AND MICROSTRUCTURE OF TI6AL4V (ELI) ALLOY ON THE OPTOMEC LENS

## N. Arthur<sup>1\*</sup>, K. Malabi<sup>1</sup>, P. Baloyi<sup>1</sup>, H. Moller<sup>1</sup> & S. Pityana<sup>1</sup>

<sup>1</sup>CSIR, NLC, Laser Enabled Manufacturing Group, Pretoria Campus, 0001, South Africa

\*NArthur@csir.co.za, KMalabi@csir.co.za, PBaloyi1@csir.co.za, HMoller@csir.co.za, SPityana@csir.co.za

# ABSTRACT

In this study, 3D cylindrical components were laser printed by additive manufacturing. The cylindrical components were 15 mm in diameter and 11 mm in height. The varied laser process parameters used in the layer by layer build-up were the laser power, scanning speed and powder flow rate. These parameters were varied in order to determine their influence on the physical properties, as well as microstructure of Ti6Al4V Extra-Low Interstitials (ELI) build-ups conducted on the LENS<sup>TM</sup> system for the qualification of additive manufacturing of aerospace and biomedical components. Results showed that at higher laser powers used, the desired shapes were not achieved, but produced higher build heights. The typical microstructure that evolved appears to be two-phase  $\alpha$ -B lamellar structures, consisting of parallel  $\alpha$ -phase lamellae found in the primary B-phase matrix as observed from SEM imaging. The hardness values obtained suggest an increase in cooling rate and thus refinement in microstructure, which is consistent with the "Basket-weave" structure that was observed in SEM imaging.

Keywords: LENS™; 3D Printing; Laser Metal Deposition; Ti6Al4V ELI; Aerospace; Biomedical Components

## INTRODUCTION

The Optomec Laser Engineered Net Shaping (LENS<sup>M</sup>) 850-R system is a laser-based platform that relies on computer-aided design (CAD) files for the design and manufacturing of three dimensional (3D) components [1]. The LENS system at CSIR-NLC is comprised of a 1 kW laser system and a co-axial powder delivery nozzle housed in a controlled atmosphere process chamber (enclosure) to maintain an inert atmosphere with oxygen content below 10 ppm, so as to reduce oxidation of the part being manufactured [2].

The manufacturing of 3D parts is done by blowing spherical metal powders through co-axial nozzles, and melting the powders with high power laser. The powders are melted onto a substrate and quickly cool down and solidify into the desired shape of the component. The component/part is thus built up layer by layer until the final part is achieved. Typically, high solidification rates in the range of approximately  $10^3 - 10^5$  K/s are experienced in this process, and newly built parts exhibit microstructural changes that can be tailored based on the process parameters used [2]. Slight variations in process design and parameters allows for complex shapes and structures to be fabricated out of various powder materials, based on the application and expected requirements. A wide range of powder materials and alloys, such as titanium, stainless steels, copper, nickel, and various others can be employed in the manufacture of 3D parts with the LENS<sup>™</sup> system. This system makes use of spherical particles with a minimum and maximum diameter of 36 and 150 µm respectively (typically, a size range of 45 to 100 µm is preferred) to prevent damage to the powder delivery system, and due to the added benefits in manufacturing owing to their shape and size.

An investigation performed by Kummailil et al. [1] to determine the effect of process parameters on powder deposition showed that increasing either the powder flow rate or laser power would result in an increase in build height, while a decrease in build height was observed due to an increase in scan velocity or hatch spacing. Hatch spacing is the distance between two adjacent tracks when determined from the center of one track to the center of the next track [1]. The observation made from designed experiments was that the mass flow rate and scan velocity seemed to have a greater influence on how much material was deposited onto the substrate. An increase in mass flow rate would deposit higher amounts of material, whereas an increase in scan velocity would see less material being deposited along each track. The obtained results were consistent with expectations, and thus it was concluded that as the magnitude of effects of the powder flow rate and scan velocity by one unit would approximately be equivalent to a decrease in the mass flow rate by the same unit within the range tested.

The objective of this study was to determine the effects process parameters have on the quality, build rates and microstructure of cylindrical components built. Knowledge of parameters that produce good builds allows the user to make more informed decisions in process design when fabricating more complex parts for various applications on the LENS<sup>M</sup> system. The laser power and powder feed rates can be manipulated to produce the desired builds, and to an extent the desired microstructures in materials being processed.

## 1 EXPERIMENTAL PROCEDURE

#### 1.1 Laser Deposition

Figure 1 shows the laser system that was used for these experiments. The LENS<sup>TM</sup> 850-R system mounted with a 1 kW IPG fiber laser as shown in Figure 1 (a) was used in processing of materials. A spot size of 1 mm was used. The material used was Ti6Al4V Extra-Low Interstitials (ELI) with a particle size range of 40 - 100  $\mu$ m as it is typically used for biomedical implants and aerospace components. The powder flow rates at the employed rpm values were determined by timing the amount of powder delivered in a plastic container over a 1 minute interval. Titanium plates of 4.5 to 5 mm thickness were prepared by sandblasting and cleaned with acetone to remove any oils and impurities from the surface of the plate, and prevent laser reflection for processing.



Figure 1: CSIR Laser Engineered Net Shaping (LENS™) system used for experimental work

Various process parameters were used in determining the effect on physical properties of builds. The reported results are of experiments conducted within the range indicated in Table 1. In these experiments, the first 3 layers of coupons fabricated were built at a laser power setting of 450 W, which was then reduced to the laser power setting as indicated in Table 1 for remainder of processing, in order to allow for proper fusion of material and substrate. This approach also aids in limiting the development of residual stresses that can lead to crack formation [3]. Parts were fabricated in an argon atmosphere to reduce oxidation.

Sample/Parameter	Laser Power (W)	Scan Speed (mm/s)	Powder Flow Rate (g/min)		
SumplerFulumeter	Luser Fower (W)	scun speeu (mm/s)			
a	360	10.58	1.46		
b	360	8.47	1.46		
c	360	6.35	1.46		
d	400	8.47	1.46		
e	320	8.47	1.46		
f	360	10.58	1.42		
g	360	10.58	1.49		

#### Table 1: Process parameters

#### 1.2 Metallurgical Sample Preparation

After the samples had been fabricated, cross sectioning was done across the build-up layers and mounted using a conductive resin. Samples were ground using SiC grinding papers with water as a lubricant. Three different SiC grit size grinding papers were used, namely 80, 320 and 1200 grit size. After grinding, two polishing stages were applied in order to obtain a mirror finish surface. Samples were polished using Md-Largo cloth with a 9  $\mu$ m diamond suspension and then final stage of polishing was done using Md-Chem cloth with a 0.04  $\mu$ m suspension. Kroll's reagent was used to etch the samples in order to reveal microstructure. An Olympus BX51M colloidal silica suspension microscope together with Stream Essentials software was used for sample analysis.

A Matsuzawa Vickers micro-hardness tester was used to evaluate the hardness of the Ti6Al4V ELI samples. A load of 300 grams and dwelling time of 10 seconds was applied, while employing a spacing of 500  $\mu$ m between the indentations. Indentations were done at the center of the samples from the top of the surface to the base metal.

## 2 RESULTS AND DISCUSSION

## 2.1 Effect of scanning speed and Powder Flow Rate

Figure 2 illustrates Ti6Al4V ELI test coupons fabricated on the LENS<sup>M</sup> system. The parts built showed physical properties more closely related to the CAD model, and were fabricated at scanning speeds of 10.58, 8.47 and 6.35 mm/s for *a*, *b* and *c* respectively as seen in Table 1. A thin film of oxidation can be seen on the surface of the builds due to oxygen content in the range of 60 to 90 ppm reported during processing. The laser power was set at 360 W, while the powder flow rate setting of 2.8 rpm delivered 1.46 g/min of material. Build heights in excess of 11 mm were achieved, with (*a*) standing at 11.7 mm, (*b*) at 12 mm and (*c*) at 12.7 mm. The observation made was that a decrease in scanning speed resulted in an increase in build height of the structures, which is consistent with observations made by Kummailil et al. [1], and an increase in hardness of samples fabricated (as observed with samples *a*, *b*, and *c* in Table 2). This could also mean that a decrease in scanning speed is equivalent to increasing the powder flow rate, which leads to longer interaction time between powder and the laser as more material is available for melting. The increasing Vickers hardness values of samples can further be attributed to the longer interaction times.



Figure 2: Test coupons indicating influence of scan speed on build properties of laser fabricated Ti6Al4V ELI parts, with scan speed of 10.58 mm/s (*a*); 8.47 mm/s (*b*); 6.35 mm/s (*c*). Powder flow rate is 1.46 g/min and laser power is 360 W

There was no significant influence on the microstructure observed with a variation in powder flow rate. It is postulated that the difference between 1.42, 1.46 and 1.49 g/min (which corresponded to a machine setting of 2.6, 2.8, and 3 rpm) of powder delivered was not significant, and did not produce a significant difference in hardness values or microstructure of samples f, a, and g (as seen in Table 2) when considering powder flow variations. The influence of powder flow variation was more evident when looked at in terms of the physical structures of parts built as compared to CAD model. Higher powder flow rates meant more material was delivered to the melt pool to allow for increased stability of builds, and prevent over-melting of powders to deform the built samples.

# 2.2 Effect of laser power

Figure 3 shows the optical micrographs of typical microstructures obtained for Ti6Al4V ELI samples fabricated on the LENS<sup>TM</sup> system at laser power of 360 W, scanning speed of 10.58 mm/s, and powder flow rate of 1.49 g/min (*sample g* as seen in Table 1). The micrographs in Figure 3 indicate the growth of stable  $\alpha$  and  $\beta$ phases, which constitute the characteristic microstructure formed during most laser fabrication techniques [4]. When Ti6Al4V alloys are fabricated and then cool to below the  $\beta$  transus temperature, which is the lowest temperature at which 100% beta phase can exist, the result is the formation of a martensitic microstructure consisting of  $\alpha'(\alpha')$  phases when higher cooling rates are experienced, i.e. 18 °C s<sup>-1</sup> and above [5]. Low to intermediate cooling rates give rise to  $\alpha$ - $\beta$  lamellar structures that appear in groups called colonies (as seen in Figure 3 and Figure 4), with  $\alpha$ -phase lamellae found in a  $\beta$ -phase matrix [5]. These colonies are composed of parallel  $\alpha$ -phase lamellae in primary  $\beta$ -phase grains, which are indicated by Figure 4. Figures 4a, 4b, and 4c represent samples d, b, and e as indicated in Table 1 The evolution of  $\alpha$ - $\beta$  lamellar structures as seen in Figure 4 suggests that low to intermediate cooling rates for samples fabricated were achieved, thus resulting in a refinement of microstructure as the cooling rate increases slightly.



Figure 3: Micrographs of typical martensitic microstructures observed in LENS™ fabricated Ti6Al4V ELI samples, at 5x (a); 10x (b); 20x (c); magnification. Only one power setting of 360 W is reported.

In an investigation by Gasper [6], it was observed that the  $\alpha$ -lamellae experience a decrease in thickness and length as the cooling rate increases, and consequently an increase in yield stress of the material. This suggests that a decrease in grain size results in an increase in hardness values obtained for samples fabricated. The evolution of new colonies typically results in formation of the Widmanstatten microstructure. Figure 5 indicates the characteristic Widmanstatten microstructure or "Basket-weave" (at 3500x magnification) that develops perpendicularly to other lamellae when new colonies nucleate on  $\beta$ -phase boundaries as well as the boundaries of other colonies [5]. This sample was fabricated at a laser power of 360 W, scanning speed of 10.58 mm/s, and powder flow rate of 1.42 g/min (sample f as seen in Table 1), and displays the typical microstructure that evolved for Ti6Al4V ELI samples fabricated on the LENS<sup>TM</sup> system.



Figure 4: SEM images indicating the presence of α-phase lamellae found in a β-phase matrix. Ti6Al4V ELI samples fabricated at laser power of 400 W (a); 360 W (b); 320 W (c). Scanning speed was 8.47 mm/s and powder flow rate was 1.46g/min.



Figure 5: SEM image indicating the Widmanstatten ("Basket-weave") microstructure: a - parallel α-lamellae, b - perpendicular growth due to nucleation of new colonies, c - thickness of α-lamellae.

#### 2.3 Hardness

Table 2 indicates the hardness values obtained for samples fabricated at different powder flow rates. Table 2 indicates the relationship between the various parameters and the hardness of the samples fabricated. Hardness measurements done on sample a, b and c shows an increase in hardness as the coupons are built up layer by layer. On average, an increase in hardness is observed in samples as a result of scanning speed. The difference in hardness of samples with variation in laser power was attributed to an increased amount of oxidation build-up during fabrication of *samples d* and *e*. This evidently had an undesired impact on sample hardness as compared to other samples fabricated. It is postulated that the difference between 1.42, 1.46 and 1.49 g/min (which corresponded to a machine setting of 2.6, 2.8, and 3 rpm) of powder delivered was not significant, and did not produce a significant difference in hardness values of samples *f*, *a*, and *g* to determine a meaningful relationship.

Sample	Scanning	HV	Sample	Laser	HV	Sample	Powder	HV
	Speed			Power			Flow Rate	
	(mm/s)			(W)			(g/min)	
a	10.58	439.7	d	400	346.3	f	1.42	456.1
b	8.47	449.1	b	360	449.1	a	1.46	439.7
С	6.35	482.1	е	320	380.1	g	1.49	455.6

Table 2: Summary of process parameters versus sample hardness

#### 3 CONCLUSION

The typical microstructure observed to evolve from fabrication of Ti6Al4V ELI samples on the LENS<sup>TM</sup> system appear to be two-phase  $\alpha$ -B lamellar structures, consisting of parallel  $\alpha$ -phase lamellae in primary B-phase grains. The formation of these structures is controlled by the rate at which the parts fabricated cool down to below the beta transus temperature. The presence of the stable  $\alpha$ -phase lamellae grouped in colonies found in the B-phase matrix suggest cooling rates in the region of 2°C s<sup>-1</sup> were achieved. This increase in cooling rate is attributed to be as a result of the build strategy, and leads to refinement of the microstructure, and the formation of the "Basket-weave" microstructure. It is when these microstructures are obtained that elevated hardness values are achieved. Manipulation of the scanning speed towards slower speeds allows for longer laser/material interaction time, which promotes a refinement in microstructure of the material, while more moderate laser powers promote microstructural refinement than higher laser powers. The effect of powder flow variations is still inconclusive, and it is recommended for further investigations to be conducted to draw better inferences.

#### REFERENCES

- [1] Kummailil, J.; Sammarco, C.; Skinner, D.; Brown, C. A. and Rong, K. 2005. "Effect of select LENS™ processing parameters on the deposition of Ti6Al4V." *Journal of Manufacturing Processes (v7, n1)*, pp.42 50.
- [2] Bandyopadhyay, A.; Krishna, B.V.; Xue, W.; Bose, S. 2009. "Application of Laser Engineered Net Shaping (LENS) to manufacture porous and functionally graded structures for load bearing implants." *Journal of Materials Science: Materials in Medicine*; 20:S29-S34.
- [3] Vrancken, B.; Buls, S.; Kruth, J-P.; Van Humbeeck, J. 2015. "Influence of preheating and oxygen content on selective laser melting of Ti6Al4V." *Proceedings of the 16th RAPDASA Conference, RAPDASA, Annual International Conference on Rapid Product Development Association of South Africa*, edition 16, Location: Pretoria, South Africa, date: 4 6 November 2015.
- [4] Han, Y.; Lu, W.; Jarvis, T.; Shurvinton, J. and Wu, X. 2015. "Investigation on the microstructure of direct laser additive manufactured Ti6Al4V alloy." *Materials Research*. 2015: 18 (Suppl. 1): pp 24 28.
- [5] Sieniawski, J.; Ziaja, W.; Kubiak, K.; Motyka, M. 2013. "Microstructure and Mechanical Properties of High Strength Two-Phase Titanium Alloys." *Titanium Alloys: Advances in properties control*, 2<sup>nd</sup> Edition, Jan Sieniawski and Waldemar Ziaja, pp. 69 80.
- [6] Gasper, G. 2012. "Microstructural Characterisation of Ti6Al4V and its relationship to sample geometry." Materials Engineering Department, California Polytechnic State University - San Luis Obispo. <u>http://digitalcommons.calpoly.edu/matesp/32</u> (Date accessed: 24/08/2016)