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Process parameter interaction effect on the evolving properties of laser metal deposited titanium for biomedical applications

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A R T I C L E I N F O

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The laser power interaction effects on the evolving properties of commercially pure titanium during Laser Metal Deposition were analyzed. The optimized processing parameters obtained for this research study were, spot size of 4 mm, powder flow rate of 2 g/min, gas flow rate of 2 l/min, and the scanning speed set at 0.002 m/s. A total of seven samples were fabricated by depositing titanium powder onto a Ti–6Al–4V base metal; using an Nd-Yag laser by varying the laser power from 400 to 1600 W while keeping all the other parameters constant. The deposited samples were characterised through the evolving microstructure, microhardness, wear and the corrosion behaviour. The microstructural evaluation revealed that the ratio of dilution increased with an increase in the laser power. Furthermore, it was found that as the dilution increased, the wear resistance behaviour of the deposits decreased due to the increased foreign elements (Al and V) from the substrate which inhibited smooth fusion as the molten deposit cooled. Also, the microstructural evaluation showed that finer martensitic microstructures were obtained at lower laser power rating which was associated with inter-layer porosity and due to the low laser-material interaction. However, Widmanstätten structures were observed at higher laser power settings together with the presence of intra-layer porosity which is desirable for osteointegration. For biocompatibility, immersion tests in the Hank's solution were conducted for 14 days. The atomic absorption spectroscopy analyses showed that no leaching happened during the immersion process for all the samples hence, confirming the desirable properties expected of biomedical implants. An overall overview on the effects of the laser power which has a significant effect on the evolving properties is essential in order to know how this process parameter can be controlled to attain certain properties of the material for specific and tailored functions.

1. Introduction

Additive manufacturing (AM) is amongst the latest technologies being employed in the manufacturing industry for a number of parts. Unlike olden days, the modern day consumers are requesting more customized products than standardized ones [1,2]. This has led to a greater demand in a manufacturing process that produces more customized goods in less manufacturing lead time [3,4]. There are different types of additive manufacturing techniques and Laser Metal Deposition (LMD) is one of the techniques that are still being developed to meet bulk production standards in the flexible manufacturing industry.

Laser Metal Deposition (LMD) process uses Computer Aided Design (CAD) model and a laser beam source to fabricate the parts through layer by layer addition. In essence, the solid materials are delivered either in powder or wire form into a pool of molten material generated on a substrate by an incident laser beam. The powder materials melt and dissolve together with the molten substrate material. The laser beam is scanned over the surface of the substrate following a path generated from the produced CAD file. After which the molten materials solidify forming a deposited layer. For subsequent layers to follow, the same process is repeated [5].

Through LMD, complex shaped parts can be fabricated at ease and at low cost [6]. Most metals have their surfaces modified by the use of laser beams because of the high coherence and directionality of the beams [7]. Because of the poor machinability property of titanium, LMD is mostly used in the fabrication of titanium parts [7,8]. LMD amongst other Laser Additive Manufacturing (LAM) methods is also used in the fabrication of a number of metals from the super alloys (e.g. titanium and nickel) to steels which are found in commonly made industrial goods. The Laser Additive Manufacturing methods are mostly preferred for the production of fully dense and near net-shaped parts which are difficult to produce using most additive manufacturing methods [9]. Titanium and its alloys have been used in the aerospace and the medical fraternities for a while now because of their high strength light weight properties. However, the use of titanium and its alloys in the manufacturing industry is limited with the materials being hard to machine using conventional methods as they react with the tools at high

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temperatures causing massive tool wear. Also, with the repeatability problems being experienced in the LMD processing of titanium, a lot of research interest has been developed in the Laser Metal Deposition of titanium and its alloys for biomedical applications.

Kobryn et al. [10] investigated the effect of the laser power and traverse speed on the microstructure, porosity, and build height in laser-deposited Ti–6Al–4V using a Laser Engineered Net Shaping (LENS) system. In their study, they discovered that high temperature gradients and high cooling gradients result in the formation of fine transformed and columnar microstructures. Also, they found out that the width of the columnar grains decreases with the increase in the cooling rates. They also noted that the laser power has no direct influence on the build height, however, they highlighted that porosity affect the solidification conditions of the deposit which in turn affect the build height.

A comparative study conducted by Wu et al. [11] on the microstructures of laser-deposited Ti–6Al–4V showed that the morphology of the deposited alloy is greatly influenced by directional heat extraction and a tendency of forming long columnar grains. It was observed that the length of the columnar grains decreased with an increase in the laser power and that large equiaxed grains were formed. The same phenomenon was observed by Shukla et al. [12] at higher laser power setting of 3000 W. They attributed this to the fact that the temperature gradient reduces with the increase in the laser power. This is as result that at higher laser power more heat is delivered to the substrate hence the substrate remains hot for a longer period of time and therefore the decrease in the temperature gradient of the heat from the deposit to the substrate.

Brandl et al. [13] in their research work on the mechanical properties of additive manufactured titanium (Ti–6Al–4V) blocks deposited by a solid state-state laser and wire they discussed that a finer or equiaxed microstructure increases the strength and ductility whilst the lamellar microstructure reduces the strength and ductility. Brandl et al. [14,15] did further studies on microstructural and mechanical properties of wire deposited Ti–6Al–4V using laser. In these studies they observed that the increase in the laser power resulted in the sizes for the grains in the melted and heat affected zones to increase. In these studies, they observed that the increase in the laser power resulted in the sizes for the grains in the melted and heat affected zones to increase. They also observed that the hardness on the deposited material increased with the increase in the laser power; as more substrate fused with the molten powders.

The same observation was shown by Mahamood et al. [6], whilst studying the effect of laser power on the microstructure and microhardness of the laser metal deposited Ti–6Al–4V. These studies attributed the impurities or alloying constituents from the substrate as the ones responsible for the increase in the hardness characteristics. Brandl et al. [16] attributed these findings to the post heat treatment that happens to the deposit, as more heat is delivered to the molten area. Then, there tends to be some work hardening effect as the molten deposit cools.

Oldani and Dominguez [17] discussed the use of titanium as a biomaterial for implants. The problems they highlighted that hinder the use of titanium in the medical industry are that the cost of processing is too high, due to the difficulty in the machining of titanium. Hence, they suggested powder metallurgy as an alternative. Also, the other problem was that although the elastic modulus of titanium is relatively low compared with that of other metallic biomaterials in its raw form, the elastic modulus of titanium is 4 to 6 times higher than that of the cortical bone. This causes stress shielding, and ultimately, the failure of the implants. Nonetheless, the solution to this problem can be reduced by controlling the porosity and morphology of the titanium during processing. The porosity of the titanium has to be controlled; because, if fully porous, the implant would not be able to sustain the physiological loads [17].

From the previous studies it is imperative that some research work be done on titanium powder which is laser metal deposited onto a Ti–6Al–4V substrate, with the intention of producing a candidate material for biomedical application. For this research study samples were fabricated by varying the laser power and keeping all the other operating parameters constant.

2. Experimental technique

2.1. Fabrication of samples

A preliminary study was first conducted to ascertain the process window for optimisation. These include the laser scanning speed, powder flow rate and gas flow rate. Preparation of the substrate was done by sandblasting it using a guyson sand blaster and cleaned with acetone to remove the dirt. This was done to remove foreign particles on the surface of the substrate and also to increase the adsorption capacity of the substrate.

The fabrication of the samples was done using a Kuka robot to deliver a 4.4 kW Nd-YAG laser. Also on the robot end effector, was a coaxial nozzle which was used to deliver the powder onto the melt pool as shown in Fig. 1. Argon gas was used as a delivery mechanism for the powder which was used as a shielding gas for the powder as well. Also, a plastic cover box was used to cover the deposition platform in order to prevent oxidation from taking place during the deposition. From the results of the deposited samples in the preliminary study, final processing parameters were chosen which are shown in Table 1.

The Laser Energy Density is described as the laser energy available on a specific area of concern for a specific amount of time. Hence, it mainly affected by the laser spot size which has an effect on the focused area and the laser scanning speed which determines the amount of time the laser is focused on that area [18].

The Laser energy density (LED) was calculated using Eq. (1)

$$LED = \frac{Laser\ power\ (KW)}{Spot\ size(m) \times Scanning\ Speed\ \left(\frac{m}{s}\right)} \tag{1}$$

From Eq. (1), it can be seen that the smaller the spot, the higher the LED. Mahamood et al. [18] maintained that the smaller the spot the size, the higher the LED available to melt the powder. This is mainly because the laser power becomes more energy intensive, as the laser energy available is concentrated on a smaller area. This improves the material efficiency; as there is more powder melted, because of the higher LED.

2.2. Material characterization

2.2.1. Metallurgical preparation and characterization of the deposited samples

The samples for microstructural characterizations were cut into 10 mm lengths. The sample preparation was conducted according to the ASTM E3–11 standard guide for preparation of metallographic specimens and the application notes from Struers for the metallographic preparation of titanium [19,20]. The sample preparation for microscopy included cutting off 10 mm in length samples from each deposit. They were then mounted, grinded and polished according to the ASTM E3–11 standard. Before the samples could be viewed under an optical microscope they were etched using the Kroll's reagent. The same procedure without the etching part was followed for the samples on which a feasible microhardness study was done.

The microscopy of the etched samples was first done under an Olympus BX51M optical microscope to characterize the zones. In addition to view and characterize the microscopic zones of the samples, the dimensions of the different properties of these microscopic zones were also measured under the microscope using the stream essentials software. The same software was used to estimate the porosity on the samples. Also, for further accuracy and characterization of the morphology of the samples, microscopy was also done using a Tescan Scanning