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Morphological characterization of recycled powder and microstructures of Ti-6Al-4V components synthesized by LENS additive manufacturing

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Abstract. Direct Metal Laser Deposition (DMLD) processes such as LENS are gaining ground in commercial manufacturing processes of functional end-products. This is attributed to this process being popularized as a sustainable technology with the potential to substitute the energy intensive, wasteful and environmentally unfriendly traditional techniques. However, during processing a significant fraction of the injected powders do not end up as part of the final component. The properties of these powders change due to heating resulting in coalescence and possible chemical reactions with the surrounding environment. A potential way to overcome this is by adopting an effective powder recycling procedure by which material use efficiency can be improved. Even so, these changes may have detrimental impact on the mechanical properties of the end-product. The objective of this paper is to investigate the effect of powder recycling on mechanical properties as a consequence of a change in powder characteristics. The potential for improving the LENS process by powder recycling process has been evaluated by comparatively studying the feedstock characteristics, i.e. morphology, particle size distribution and microstructure of powders in as received state and after laser exposure during repeated deposition cycles. Microstructures, micro-hardness and Density of the built samples were measured after the first deposition-normalization-reuse cycle. The results revealed a correlation between the powder characteristics and the properties of the corresponding built sample used for that specific deposition.

1. Introduction

The significance of the development of new and existing fabrication techniques in attempt to provide the world with cleaner and cheaper engineering materials comprised of advanced characteristics is forever on the rise. Laser Engineered Net Shaping (LENS) is a cutting edge advanced additive manufacturing technique possessing the potential to change the perception of design and revolutionize manufacturing as a whole [1]. This process involves fabrication of complex 3-D Near Net Shape metallic components directly from CAD models by layer upon layer deposition of metal powders which are transferred into the melt pool created by a laser on the metallic substrate or preceding layers [2], [3]. The operational principle of additive manufacturing processes is in contrast with the conventional subtractive and formative approaches which are wasteful of feedstock and energy intensive as well as limited in freedom of design. Additive fabrication techniques provide various benefits including elimination of forming equipment, high material use efficiency and the flexibility of producing customized parts with high precision which require minimal or sometimes no post process machining; these makes a direct contribution to both the reduction of production cost and delivery time [4]–[6].

Due to the its advantages, LENS is well suitable for critical building and repairing applications such as aerospace, biomedical and architectural industries. Titanium and its alloys are considered expensive materials due to difficulty in refining as a result they are a perfect candidate for near net shape manufacturing techniques [7]. Ti-6Al-4V, the most used titanium alloy has been reported to have applications in many industries because of the excellent combination of attractive properties such as high strength to weight ratio, excellent corrosion resistance and biocompatibility among others. Over the recent years there has been a growing interest in developing additive manufacturing processes of Ti-6Al-4V. The driving force behind this interest is the attempt to reap the benefits provided by this material as it holds a promising future for an extended application scope of Titanium alloys which are commonly limited due to high fabrication cost [8]. However, widespread adoption of LENS fabrication of Ti-6Al-4V is hindered by having most studies concentrating only on process parameters and ignoring the development of powder recycling methods; which can contribute a deal in giving additive manufacturing technologies an upper hand over conventional techniques [9].

According to Renderos et al., [10] the development of recovering, treating and re-using procedures for the powders that remain unfused during deposition eliminates the formation of agglomerations and reduces the risk of clogging as well as maintain the characteristics of the built component while reducing waste. Another study carried out by Asgari et al., [11] involved comparing the characteristics of the virgin, condensate and recycled powder on DMLS process of AlSi10Mg_200C alloy. The authors' observations show that both the virgin and the recycled powder retained relatively identical characteristics, their respective components also revealed similarities in microstructure and mechanical behaviour. From the above literature, it is evident that not much attention has been given to the exploration of recycling methods for the LENS fabrication of Titanium alloys (Ti-6Al-4V in particular) in further assisting in industrialization of the process. The key objective of the present research is to investigate the influence of powder recycling in LENS additive manufacturing.

2. Experimental

2.1. Materials

The present study employs the LENS fabrication for the most widely used titanium alloy, Ti-6Al-4V, which dominates in the aerospace applications. Despite titanium being known as the 4th most abundant metal on the earth's crust, it is still considered expensive due to high refining costs and being difficult to fabricate by subtractive traditional methods which are wasteful of feedstock as machining creates scraps. The above mentioned makes this material and it's based alloys good candidate for exploration of additive manufacturing techniques such as LENS in attempt to reduce fabrication costs and therefore expand their scope application scope in engineering. In this study, the atomized Ti-6Al-4V ELI spherical powders (size range 45-100 microns) were used which were relatively identical in terms of chemistry to the workpiece used as the build platform. The chemical composition of the virgin powder is shown in Table 1.

Table 1. Supplier's chemical composition of the virgin powders.

Element	H_2	N_2	O_2	Fe	C	Al	V	Ti
Composition (%)	0.001	0.006	0.078	0.25	0.006	6.34	3.94	Bal.

2.2. LENS deposition and powder recycling procedure
The Optomec LENS 850-r system mounted with a 1KW IPG fiber laser from CSIR was used for the fabrication of Ti-6Al-4V test samples. The laser deposition processing parameters are presented in Table 2. Ti-6Al-4V plates used as substrates were initially sand blasted and cleaned with acetone in order to remove of dirt/contamination and minimize laser reflection which enhances absorption of the laser energy. The square test samples of 5mm x 5mm x 5mm dimensions were fabricated using virgin powder and then the powder that didn't form part of the test sample was sieved to a particle size range of 45-100 microns using a sieve shaker before it was reused to fabricate the subsequent sample. The samples fabricated from virgin and recycled powder were classified as sample "VP" and sample "RP", respectively. The schematic methodology for the LENS powder recycling procedure followed in this work is shown in Figure 1.

Table 2. Table Showing the Processing parameters, microhardness and the densification of the Ti-6Al-4V deposits using new and aged powder.

Sample	**Laser power (kW)**	**Scan speed (mm/s)**	**Powder feed rate (g/min)**	**Average hardness (HVN)**	**Density (g/cm^3)**
VP	**300**	**16,93**	**1,6**	**367,5**	**4,42**
RP	**300**	**16,93**	**1,6**	**356,22**	**4,32**

The fabricated samples and a powder sample from the first build cycle were taken for characterization in attempt to investigate the changes in both powder and build properties with a successive powder re-use cycle.

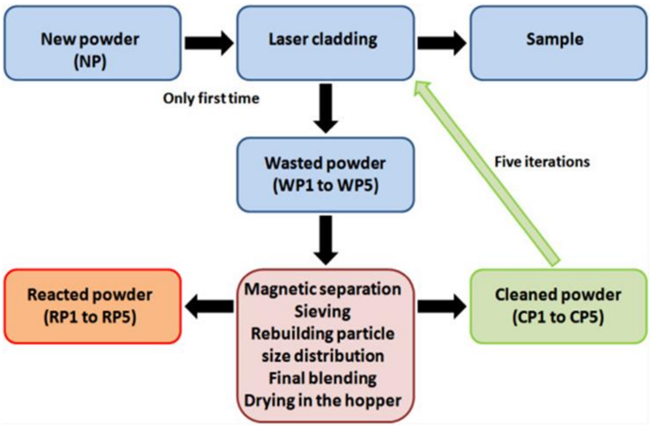

Figure 1. Schematic illustration of the powder recycling procedure.

2.3. Characterization of Powder materials
The characterization of powders carried out after the first re-use cycle has been focused on morphology and particle size distribution of re-used powders in comparison with the fresh virgin powders. The morphological studies and particle size analyses were carried out using SEM/EDS and laser diffraction, respectively.

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2.4. Characterization of Built Components
Samples were ground and polished using P320 SiC abrasive/water, Aka-allegran/6micron diamond lubricant and Aka-chemal/Fused Silica Lubricant. Microstructural characterization was carried out by an SEM/EDS. Microhardness and density measurements were done using Vickers micro-hardness tester and the density test rig following Archimedes Principle.

3. Results and Discussions
3.1. Powder Morphology and Particle Size Analyses
The particle size distribution results of the new and re-used powder are presented in the plots in Figure 2 and summarised in Table 3. The particle size of the new powder was found to be different from the particle size range data provided by the supplier (40-100µm) which is confirmed by the presence of particles of up to 119.5 µm size found above the 95th percentile. The analyses of the powder after the exposure to laser radiation shown an increase in particle size of the recycled powder. This serves as proof that despite the potential offered by powder recycling on saving cost through reduction of feedstock required per part built there are issues that may arise due to alterations in powder properties. The particle size found at 95th percentile increases from 119.5 µm to 139.6 which translates to 16.82% increase in particle size.

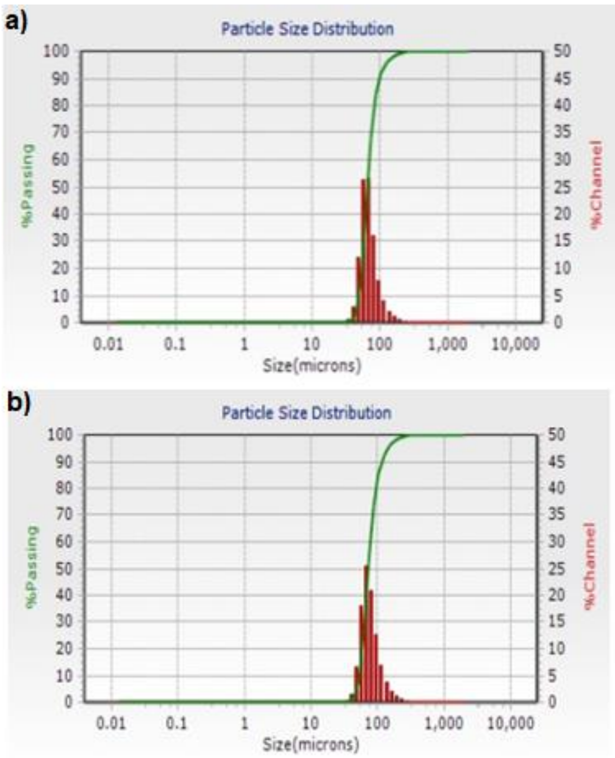

Figure 2. Particle size distribution of a) as-received virgin powder and b) recycled powder.

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Table 3. Percentile summary of the particle size distribution

a)

Summary Data	Value	Percentiles %Tile	Size(um)
MV(um):	71.57	10.00	49.51
MN(um):	58.01	20.00	54.32
MA(um):	65.64	30.00	58.06
CS:	9.10E-02	40.00	61.70
SD:	17.66	50.00	65.56
Mz:	68.73	60.00	69.88
si:	19.98	70.00	75.25
Ski:	0.367	80.00	83.32
Kg:	1.335	90.00	99.32
		95.00	119.5

b)

Summary Data	Value	Percentiles %Tile	Size(um)
MV(um):	80.64	10.00	53.31
MN(um):	62.44	20.00	59.04
MA(um):	72.68	30.00	63.79
CS:	8.30E-02	40.00	68.26
SD:	22.48	50.00	72.98
Mz:	77.28	60.00	78.52
si:	24.98	70.00	85.50
Ski:	0.378	80.00	95.73
Kg:	1.304	90.00	115.9
		95.00	139.6

In addition, the average particle size at the 10th percentile which is generaly referred to as D10 and also the lowest in the presented results was seen to increase from 49.51 µm to 53µm after after the first use cycle which suggests that the number of fines is reduced in feedstock therefore flow properties are improved. The d50 and d90 which are represented as the 50th and 90th percentiles, respectively were also found to increase with the former increasing from 65.56 to 72.98µm and the latter increasing from 99.32 to 115µm. This is evident in the micrographs in Figure 3 whereby the smaller particles are seen to have fused onto the larger particle after the first use cycle, which could be the work of coalescence due to the exposure to the laser irradiation.

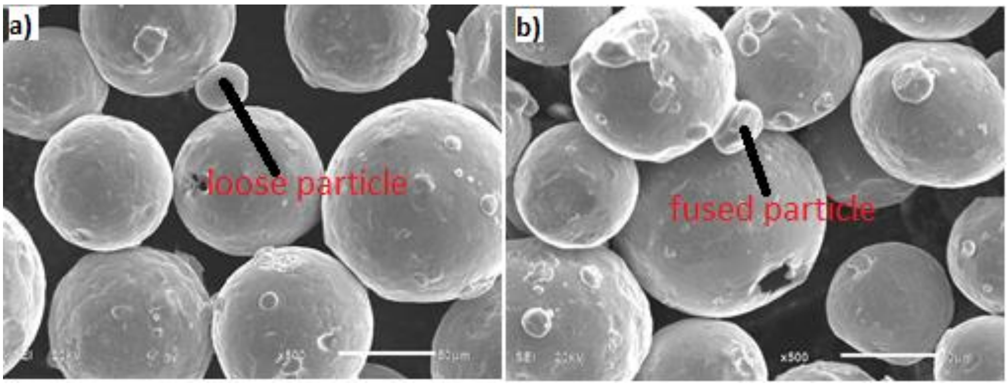

Figure 3. Morphological representation of a) virgin and b) recycled Ti-6Al-4V ELI powder.

This observation serves as evident to the changes in particle size which was seen to increases after the powder was recycled. This can be attributed to the decreasing number of fine sized particles as they are coalesced to the larger particles.

3.2. Microstructures of LENS As-Built Parts

The SEM micrographs of the specimen manufactured using the new and recycled powders in the LENS system are presented in Figure 4. The microstructural features are seen too be relatively similar which is expected since aging of feedstock have very little if not nothing to do with the thermal input and distribution in the system which are determinants of the microstructural evolution during fabrication. This observation is in line with findings reported in literature [12], [13]. However, the re-use of powder induced the formation of defects such as the crack seen in Figure 4b). This observation is attributed to the change in particle size and shape of the feedstock which alter their flowability property. These newly evolved defects may have detrimental effects on the mechanical properties of the built component because they act as the point of weakness where failure propagation is initiated.

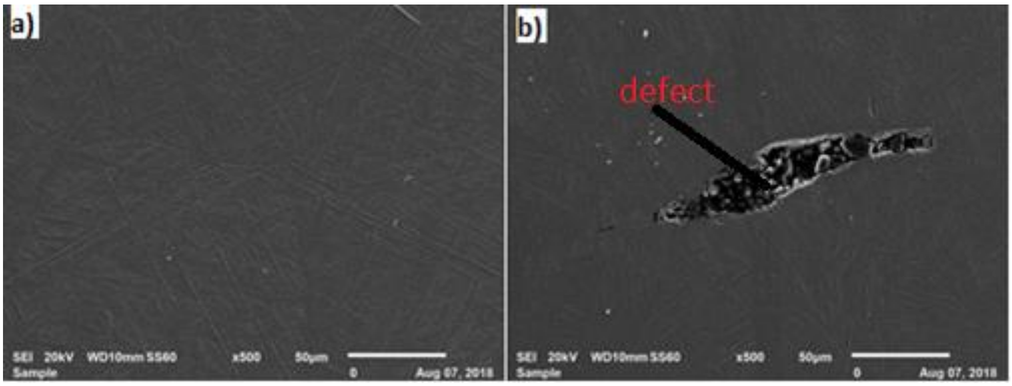

Figure 4. SEM micrographs of the sample built with virgin powder a) and built with recycled powder b).

3.3. Microhardness and Density Measurements

In Table 2, the microhardness and density characteristics of specimen fabricated with feedstock of different characteristics as described above are presented. The results revealed a decrease in both microhardness and density after the first reuse cycle. This decrease in the abovementioned properties can be attributed to an increase in porosity and the existence of defects (e.g. crack in Figure 4b) which are both well known for degrading the properties of the material regardless of the chemical composition.

4. Conclusion

Ti-6Al-4V samples were successfully deposited using recycled powder and results have been reported. The average particle size of powder was found to increase after the first use cycle i.e d10, d50 and d90 increased after exposure to laser radiation during deposition. The morphological micrographs revealed the attachment of fine particles to larger particles which was attributed to coalescence. The decrease in microhardness and densification of deposits after the first re-use cycle was attributed to the evolution of defects as a result of the change in quality of the feedstock after the first re-use cycle. However, a more vigorous study entailing and describing the mechanism of aforementioned changes in powder characteristic is yet to be undertaken in order to correlate the evolution of these changes in powder quality and corresponding deposits.

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