

# A technical review of the challenges of powder recycling in the laser powder bed fusion additive manufacturing process

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## Funding information

Engineering and Physical Sciences Research Council, Grant/Award Number: EP/R043973/1; Lloyd's Register Foundation, Grant/Award Number: GS012018COV

## Abstract

Laser powder bed fusion (L-PBF) is one of the most widely used additive manufacturing techniques for fabrication of components with complex geometries for various industrial applications including aerospace, medical and automotive. The unconsumed powder after part manufacturing is often recovered and recycled to improve process efficiency. However, some of the particles in the recycled powder can have different physical and chemical properties from those in the virgin powder owing to their exposure to the complex environment during the manufacturing process. In addition, some contaminants can be introduced in the recycled powder due to poor process control. A number of studies have been published in the past few years revealing the effects of powder recycling on the build properties. The present work aims to highlight the key phenomena during the manufacturing process that caused degradation to the recycled powder. Further to this, some comments, gaps and areas that deserve further detailed studies are also highlighted.

## 1 | INTRODUCTION

The interest in additive manufacturing (AM) processes such as laser powder bed fusion (L-PBF) stems from their capability to manufacture complex functional parts with materials like titanium, which are difficult to fabricate via conventional subtractive manufacturing routes like casting, forging and machining [1–3]. In L-PBF, components can be produced without the need for fixtures, tools or moulds. This offers high design freedom and therefore near-net-shaped custom parts for automobile or aerospace applications can be produced rapidly with relatively low buy-to-fly ratio [4–6]. A schematic of the L-PBF process is shown in Figure 1. The equipment consists of a powder delivery system that loads the powder into the build chamber. A thin layer of powder is then spread over the build platform based on the layer height set by the manufacturer (generally less than 70  $\mu\text{m}$  to build fine geometrical features [7]). The laser beam (operating in continuous or pulsed-wave mode) is programmed to

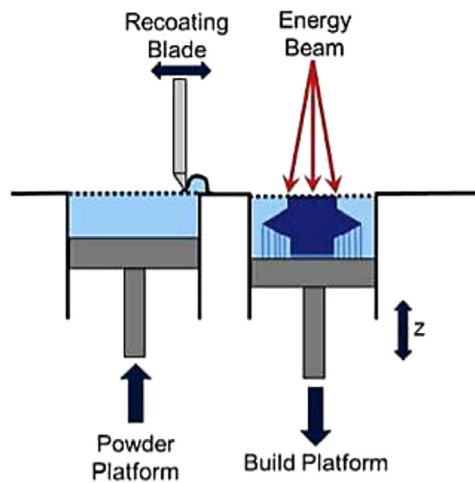
selectively melt certain regions of the powder layer corresponding to the input 3D computer automated design (CAD). After the layer is melted, the build platform retracts downwards and a fresh powder-layer is again spread, and the process continues in a layer-wise fashion until the desired part is manufactured [8, 9].

Although the market for L-PBF parts is rapidly growing, one of the major issues faced by manufacturers is the consistency in part properties [10]. Optimisation and standardisation of the process parameters and powder feedstock quality can lead to an improvement in the reproducibility of part properties [11]. While much attention has been given to the development of suitable process parameters subject to different machines and materials, the quality of feedstock powder material is given less attention. The powder feedstock quality plays a crucial role in controlling the mechanical properties of the parts [12, 13].

Several studies have reviewed different techniques that are currently available to characterise the powders used in the

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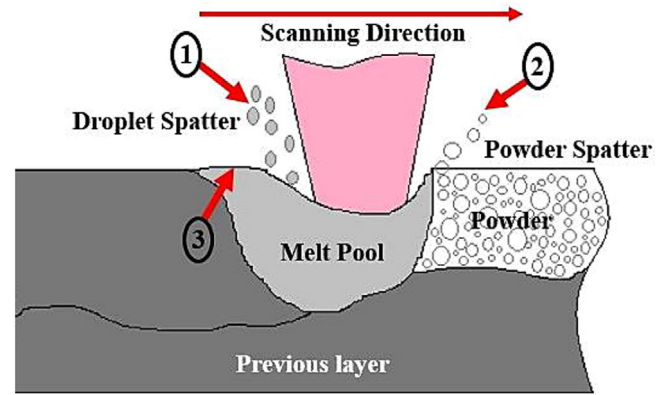
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**FIGURE 1** Schematic of laser powder bed fusion (L-PBF) process (reproduced from [14] with the permission of AIP publishing)

L-PBF process [15–17]. The key powder physical properties include powder shape and particle-size distribution (PSD) [18]. Spherical powder particles with optimum PSD are desired as irregular shaped, odd-sized particles yield poor powder-layer packing density [19, 20], which in turn affects the part density [21]. Powder shape is generally studied by imaging-based methods like optical or scanning electron microscopy (SEM) and X-ray computed tomography (XCT) techniques. PSD is estimated by three commonly used methods: Sieving, imaging, and diffraction. Sieving is a simple, rapid method for analysing large quantities of powder. However, limitations arise for powders less than 50  $\mu\text{m}$ . Fine powder particles tend to agglomerate due to cohesive forces leading to clots in the mesh apertures. Therefore, particles smaller than the mesh sizes tend to get retained in the sieve resulting in inaccurate results [22]. Therefore, imaging and diffraction-based techniques are widely used for testing L-PBF metal powders. In terms of chemical composition, some of the widely used techniques are inert gas fusion, inductively coupled plasma optical emission spectroscopy, energy-dispersive X-ray spectroscopy, X-ray plasma spectroscopy and so forth [23].

The unconsumed powder after the L-PBF process can be recycled to improve process and cost-efficiency. However, the recycled powder might have some process-affected particles that possess different physical and chemical properties compared to the virgin powder [24]. Therefore, it is important to check the quality of the recycled powder feedstock before reuse. A number of studies have been published in the past few years that looked at the effect of powder recycling in some of the most commonly used L-PBF metal powders such as titanium [21, 25, 26], aluminium [25, 27], stainless steel [10, 28] and nickel [25, 29, 30] alloys. These studies have reported deterioration in the metal powder properties caused by the complex environment during the manufacturing process. Therefore, the current study aims at highlighting the different phenomena that occur during the L-PBF process that affect the recycled powder properties.



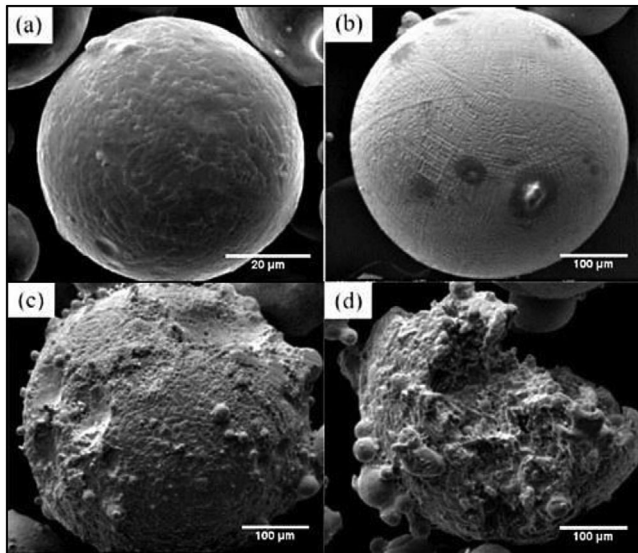
**FIGURE 2** Laser spatter during the L-PBF process (adapted from [36]). Regions 1, 2 and 3 indicate potential sites for oxygen diffusion in reactive alloys

## 2 | DEGRADATION DUE TO LASER SPATTER

Spatter, a solid or liquid ejection from a melt region, is a phenomenon that can be observed in any laser manufacturing process such as drilling, welding or machining [31–33]. In L-PBF, the process is relatively complex, as along with a significant part of the molten droplets from melt pool, powder particles are also ejected that tend to fall back into the powder bed, deteriorating the powder-layer chemistry and PSD [34, 35].

A schematic representation of the spatter phenomenon is shown in Figure 2. It can be seen that there are two types of spatter: Liquid spatter from the melt pool, and powder spatter. When the laser is focussed on the powder bed surface, heating of the powder particles creates an initial melt pool that grows by absorbing the powder particles it makes contact with by a conduction mode of melting [37, 38]. With increasing power density and relatively low thermal conductivity in a powder bed compared to the same bulk material alloy [39], the melt-pool surface temperature reaches the boiling point. When the surface temperature of the melt pool exceeds the boiling point, due to rapid vaporisation, recoil pressure is generated that causes melt expulsions (droplet spatter) [40]. Powder spatter occurs due to the entrainment of powder in the vapour jet, generated due to intense vaporisation caused by a keyhole mode of melting [41].

The formation and motion of the spatter particles were investigated by conducting *in situ* studies using a high-speed [42], infrared [43] and X-ray cameras [40] followed by image-based methods for motion detection. Spatter particles are generally larger than the parent powder particles [44]. The size, quantity, and distribution of the spatter particles vary with laser power, scan speed, and flow rate of the shielding gas [45, 46]. In terms of spatter particle distribution, increased numbers of spatter particles have been found in the region closer to the melting area [44]. Wang et al. [5] classified spatter particles based on their morphology as shown in Figures 3(b)–(d). Type-I (Figure 3(b)) are smooth spherical particles. These spatter particles were ejected from the melt pool, suffered rapid cooling upon exposure to the shielding gas flow but had sufficient time to solidify as a smooth spherical particle before



**FIGURE 3** Scanning electron microscopy (SEM) image of powder morphology: a) virgin powder; spatter particles; b) Type-I; c) Type-II and d) Type-III (reproduced from [5])

falling into the powder bed. Type-II (Figure 3(c)) are rough spherical particles. These spatter particles collided with the nano-particles in the condensate before falling into the powder bed resulting in the sintering of fines on the particle surface [7]. Type-III (Figure 3(d)) are agglomerated particles with an irregular shape. These spatter particles ejected from the laser-front had short flight time and, therefore, fell into the powder bed in the half-molten condition resulting in the formation of agglomerates.

As can be seen in the micrograph (Figure 3), the spatter particles are coarser than the sieve mesh size ( $<63 \mu\text{m}$ ) typically used in the L-PBF process. Therefore, sieving should remove most of the laser spatters. However, Sutton et al. [7] observed laser spatters smaller than  $45 \mu\text{m}$  (smaller than the sieve mesh size generally used in the L-PBF process). These particles can pass through the sieves and settle in the powder bed when the powder is recycled. These non-spherical particles can significantly affect the powder flow and packing behaviour by creating local voids in the powder bed that can result in lack of fusion (LoF) defects in the built parts [47]. Furthermore, the spatter particles have different phase and chemical composition compared to virgin powder. For example, in 316L and 304L stainless steel, it has been reported that the  $\delta$ -ferrite content increased with the increasing spatter size with some particles being completely ferrite with islands of oxides rich in manganese and silicon content over the powder surface [7, 10]. Due to the reactive nature of the spatters, the spatter particles might also pick-up minute amounts of residual oxygen left in the chamber, thereby increasing the oxygen level of the recycled powder, especially in case of reactive alloys like titanium or aluminium [48].

Some of the spatter particles get mixed with the unconsolidated powder and are spread over the previously melted layer. As the spatters have different surface chemistry and size distribution, their attenuation to the laser beam is different

resulting in unmelted particles becoming embedded in the part [5] and also over the part surfaces [49]. An example of unmelted laser spatters deposited on the part surface is shown in Figure 4. The unmelted particles on part surfaces lead to poor surface quality, and an increase in the average surface roughness from  $14.8$  to  $28 \mu\text{m}$  has been reported in parts manufactured from clean and spatter-rich regions of the powder bed, respectively [38]. The non-spherical, high-aspect-ratio spatters present in the powder bed can create local voids in the powder layer. The embedded unmelted spatters inside the part create LoF defects. Pore sizes larger than  $28 \mu\text{m}$  were found in the parts produced from spatter-rich regions [38]. Effects of defect morphology and population in L-PBF parts on tensile and fatigue properties have been widely investigated by various researchers [50–53].

As spatter particles can cause potential deterioration to the part and powder properties, efficient reduction and removal methods should be employed. Argon gas flow used during the process can carry the spatter particles and condensate outside the powder bed. However, large-size spatters are hard to remove as they possess relatively high inertia resulting in high gravitational forces acting on them. For efficient prevention of spatters from falling back into the powder bed, Ladwig et al. [54] made some key recommendations: (a) Homogenous gas flow at a high velocity close to the build surface is required, and care must be taken to avoid disturbing the powder layer, as it may affect the powder-layer density; (b) upward turbulence in the flow should be kept to a minimum as this could disturb the laser beam. In addition, Khairallah [55] proposed preheating the powder bed using a lower laser energy density before selective melting (similar to the approach used in the electron beam powder bed fusion (EB-PBF) process) that would sinter the powder particles, restrict their mobility and reduce the number of spatter particles.

### 3 | NEAR-MELT ZONE REGION OF THE POWDER BED

During laser melting, some of the powder particles present closer to the melting area (near-melt zone) are exposed to the latent heat from the melt pool. The size of the near-melt-zone region varies with laser power, spot size, and part geometry and powder properties. Several in situ [56, 57], analytical and numerical modelling experiments [58–60] have been conducted to study the temperature distribution in the L-PBF process. However, these studies focussed mainly on thermal gradients within the melt pool and solidified part but did not consider the heat dissipation outside the melting area, that is, the effect of high temperatures on unconsolidated powder near the melt pool. As the powder particles in the near-melt zone region would experience high temperatures, changes in the powder particle shape, size, chemical composition and phase constitution may occur. In addition, laser spatters are found in the highest concentration in the near-melt-zone region [44]. Therefore, maximum degradation occurs in the near-melt-zone region [61]. Most powder recycling studies have reported an increase in the

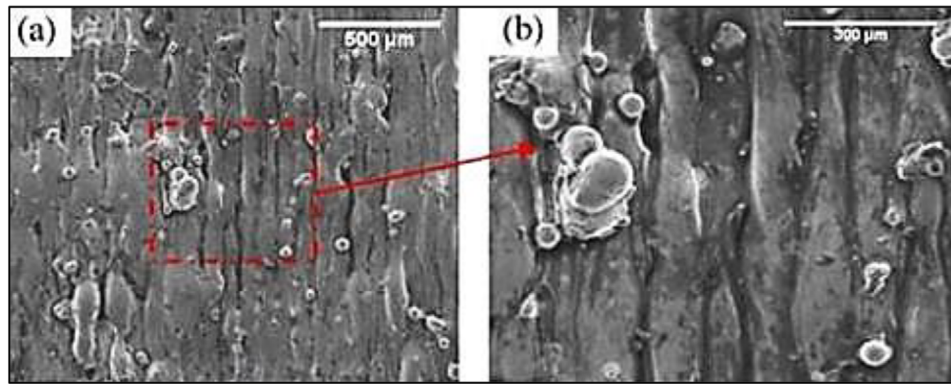


FIGURE 4 SEM image of unmelted laser spatters on the part surface (reproduced from [5])

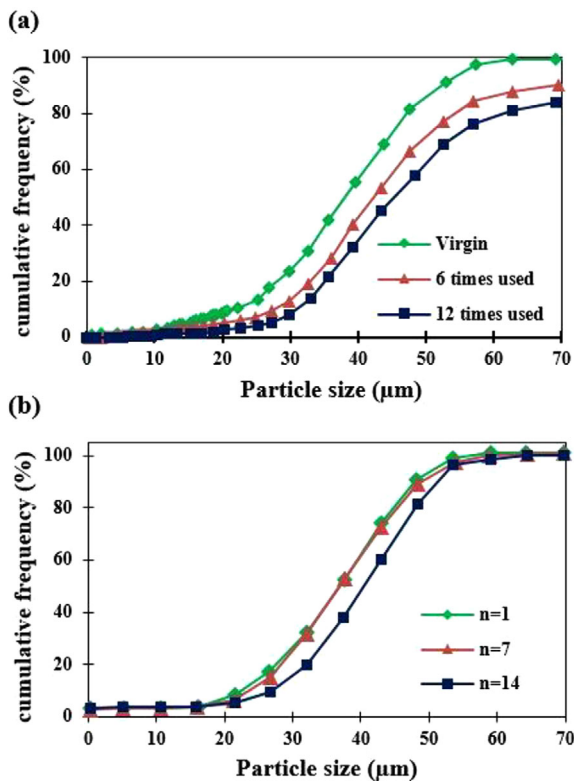


FIGURE 5 Particle-size distribution of virgin and recycled powder: (a) Ti6Al4V, (b) IN718 measured by laser diffraction (reproduced from [21] and [29], respectively)

volume fraction of coarser particles with recycling. An example of the changes in the powder PSD of Ti6Al4V and IN718 with recycling is shown in Figure 5. The cumulative fraction of coarser particles increased after 12 and 14 times of recycling in Ti6Al4V and IN718 powders (Figure 5) even though the powders were sieved after each build cycle. This could be due to some spatter particles or some of the particles in the near-melt-zone region forming agglomerates or becoming sintered such that their aspect ratio was high enough to enable them to pass through the sieve mesh in a certain orientation [10]. For any powder bed AM process, the powder particles should be spher-

ical with optimum size distribution [62], and the presence of particles with a high aspect ratio affects the powder flowability and packing behaviour, which thereby affects the part density [18, 21].

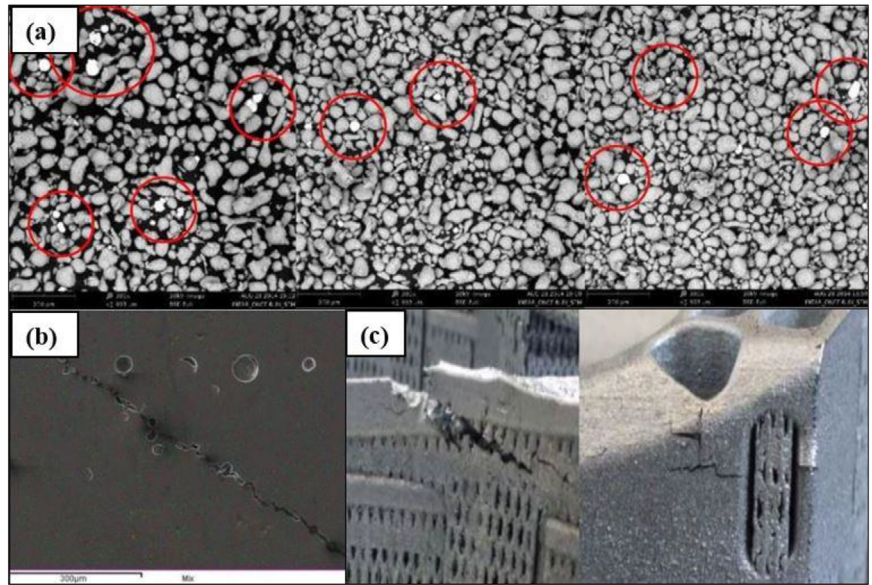
In reactive alloys like titanium, the powder oxygen level increases with recycling [63]. Powder temperature is a major factor in oxidation, and particles experiencing a high temperature can oxidise relatively fast [64]. As the near-melt-zone region experiences high temperatures, the powder particles in that region have higher oxidation rates than the particles away from the melting zone. In addition, it has been reported that the temperature of the melt pool could reach 3700°C for Ti6Al4V even though the melting point of Ti6Al4V is only ~1668°C [65]. As the temperature of the melt pool can rise to two to three times the melting point of the material, some of the light elements present in the particles located closer to the melt pool might be vaporised. Most of the powder degradation studies conducted to-date have demonstrated powder degradation relative to the number of times powder is reused. To the best of the author's knowledge, powder degradation with respect to different areas of the powder bed has not been explored so far and, therefore, deserves attention.

#### 4 | FOREIGN ELEMENT CONTAMINATION

Powder purity is a critical factor when it comes to safety-critical, high-performance applications like aerospace, defence and space parts. Powder contamination occurs when there is poor quality control. As L-PBF is a layer-by-layer manufacturing process, when a foreign element is introduced in any one layer during manufacturing, it can become sealed in by the subsequent layers. The presence of foreign contaminants in the part can cause deleterious effects to the part properties. Therefore, it is important to investigate the powder purity using robust techniques. Some of the sources of powder contamination are as follows:

1. Powder production: Gas and plasma-based atomisation processes.

**FIGURE 6** Cross-contamination: Nickel particles in aluminium powder lead to part failure (reproduced from [71])



2. Powder handling during recycling or storage.
3. Cross-contamination from previous builds: Contaminated by other AM powders.

In a gas atomisation (GA) process, the pre-alloyed ingot is melted and the molten stream is broken down into droplets by high-pressure inert gas upstream. The melt droplets solidify into pre-alloyed spherical powders during the flight. A detailed description of the gas atomisation process can be found in [66]. Although GA is one of the widely used powder production techniques for AM, there are several sources from which contaminants can be introduced into the powder. Some of the sources of contaminations are (i) refractory elements in the molten state could be highly reactive and, therefore, can erode atomiser nozzles [67] and crucibles [68]; (ii) partially solidified powder particles, impinging on stainless steel walls of the atomiser, can pick up iron; (iii) gas entrapment inside powder particles [3]; and (iv) improper cleaning of the atomiser, when changing the alloy, can lead to powder cross-contamination. In plasma atomisation, a pre-alloyed wire is melted and broken into droplets by plasma torches. In this case, tungsten (W) nozzles used in the plasma torches can wear-out creating tungsten inclusions in the powder. As W particles possess a higher melting temperature than most of the commonly used AM powders such as titanium, stainless steel or aluminium, it can remain unmelted and create inclusions in the manufactured part, which act as preferred sites for crack initiation [69].

When the unconsumed powder post-manufacturing is recycled or recovered and stored, there are a number of opportunities for contaminants to entrain into the powder such as fragments from hair, gloves, cleaning brushes, other powder-handling accessories and so forth [70]. In addition, cross-contamination from previous builds is a serious issue when the same machine is used for processing different alloys. The machine and its accessories should be thoroughly cleaned before loading a different powder material, as even a small quan-

tity of contaminant particles in the powder feedstock can cause a catastrophic failure of components as shown in Figure 6, where nickel particle contaminants created inclusions in the aluminium part, which led to its failure.

This is particularly important for small and medium-sized enterprises where the powder recovery process is not automated. Montazeri et al. [70] have pointed out that the ease of contaminant detection depends on the type of contaminant and the powder feedstock. For instance, tungsten contaminants in titanium powder feedstock are easily detectable by XCT techniques, but finding aluminium contaminants could be more difficult as the difference in the material density is marginal. Therefore, SEM/EDX technique could be used. In sum, suitable characterisation techniques should be employed to check the cleanliness of the powder feedstock.

## 5 | CONCLUSIONS

In the L-PBF AM process, the unconsumed powder is recycled to improve process efficiency. However, some of the particles in the recycled powder are subject to changes in physical and chemical properties due to spatter or heat conduction in the powder bed. Sieving has been proved to be an effective method to remove large sized spatters and agglomerates. However, process-affected particles that are smaller than the size of the sieve mesh, and particles with a high aspect ratio, tend to pass through the sieves and become embedded in the powder bed in future cycles when the powder is recycled. The process-affected particles degrade the powder layer and part properties. There are several sources from which foreign contaminant can entrain into the powder. Therefore, powder quality must be checked at each stage of the process before using. The powder purity examination could be established via techniques like SEM or X-ray imaging as the presence of contaminants in the powder can cause localised heterogeneity in part properties.

## ACKNOWLEDGEMENTS

This publication was made possible by the sponsorship and support of the Lloyd's Register Foundation, a charitable organisation that helps to protect life and property by supporting engineering-related education, public engagement and the application of research. The work was enabled through and conducted, at the National Structural Integrity Research Centre (NSIRC) managed by TWI through a network of both national and international Universities. The financial support from the EPSRC Fellowship EP/R043973/1 awarded to Bo Chen is acknowledged to facilitate this research collaboration.

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**How to cite this article:** Soundarapandiyana G, Johnston C, Khan R, Chen B, Fitzpatrick ME. A technical review of the challenges of powder recycling in the laser powder bed fusion additive manufacturing process. *J Eng.* 2021;2021:97–103. <https://doi.org/10.1049/tje2.12013>