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Abstract

A powder capture capsule has been designed to capture thin regions (3 mm, Z height) of powder at multiple heights within a powder bed of laser powder bed fusion (LPBF) equipment. This design is stackable, support-free, and allows powder capture from a targeted region (stacked layers) of the powder bed. Extraction of powder from the capsule is ergonomic, quick, and does not require any special tools; it also ensures sufficient powder for chemical analyses using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). An approximate powder bed density pertaining to the region under investigation has also been evaluated in this test case.

Keywords

Metal powder; Powder capsule; Laser powder bed fusion; Additive manufacturing.

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Acknowledgments

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1. Introduction

In situ, powder capture from a powder bed is crucial to study powder quality in powder bed fusion (PBF) additive manufacturing (AM) [1]. In literature, there have been a few approaches to capturing powder from the bed. The applicability of each technique depends on the end objective of the study. In the simplest case, if a random powder sample is required to be extracted from any location of the powder bed, a known quantity of powder can be scooped out of the bed post-printing [2–4]. However, there are reports of powder heterogeneity across the AM powder bed spanning all three dimensions [5, 6]. To tackle these cases, more sophisticated powder collection strategies are required. These include (a) targeted extraction devices and (b) powder capsules. The extraction devices surveyed for this document offer less flexibility in powder capture than a powder capsule in terms of the exact coordinate volume of powder extraction. Therefore the word targeted refers to the accuracy or a lower uncertainty of the region of powder capture. The word capsule has been used in literature as a reference to a part that encapsulates powder [7]. These are parts built in the powder bed to capture powder and they may or may not possess a capsular shape [7]. The advantages and disadvantages of each strategy are presented in **Table 1**.

Table 1. Powder collection strategies along with pros and cons.

Powder collection method	Objective	Pros	Cons
Scooping from top [2–4]	Quickly collect sample powder	Speed	Least accurate
Targeted extraction [8, 9]	Quickly collects samples from specific zones	Speed, more accurate than scooping	Less accurate than capsules
Powder capsules [1, 10–13]	Collection of powder from exact zones and layers	Most accurate	Requires more effort

Various designs are available for powder extraction devices and powder capture capsules. Muzzio et al. patented a general method and a corresponding apparatus for extracting powder from a powder bed [8]. In this case, powder collection probes are required to be inserted into a powder bed through a template [8]. Specifically for laser powder bed fusion (LPBF), Whiting and Fox designed a custom slot sampler to extract powder for particle size distribution (PSD) measurements by inserting a probe-like collecting device inside the powder bed from the surface [9]. This device is useful if it can be inserted with no obstruction or can be directly used in a powder bed where no parts have been built. Ejecta produced during the LPBF process can land at specific locations within the powder bed (e.g., adjoining parts or zones near the gas outlet) [14]. Powder capsules have been employed by Snow et al. to capture ejecta-rich powder from these locations for imaging and PSD analyses [14]. Furthermore, the powder bed density of the specific captured region within a powder bed can also be evaluated from captured powder. Jacob et al. designed cylindrical-shaped powder capsules to capture powder from specific locations of the powder bed [10, 15]. This design has also been used by other researchers for evaluating powder bed density [16, 17]. Lopez et al. patented a design for a powder capsule and an integrated part intended for quality control post-printing [1]. Whiting et al. also created a multi-cavity powder capsule to measure thermal diffusivity using a laser flash system [11]. In certain cases, powder capsules provide the basis for hot isostatic pressing (HIP). Hernandez-Nava et al.

created a thin-walled pill shaped capsule for HIP of Ti6Al4V powder after electron beam additive manufacturing [18]. Finally, capsules can also be designed to study the chemistry of captured powder provided enough powder is captured. Since Ti6Al4V feedstock is susceptible to a rise in oxygen content with reuse, this method was used by Grainger et al. and Derimow et al. for laser and electron beam powder bed fusion, respectively [12, 13]. Since feedstock powder sinters during electron beam powder bed fusion, powder cannot escape. Therefore the capsule designed by Derimow et al. contained an open top and bottom intentionally as there was no chance of powder escaping unlike capsules used in LPBF AM. A quick comparison table for various powder capsules and capture devices is shown below in **Table 2**.

Table 2. Comparison of previous powder capture devices or capsules used in powder bed fusion.

Reference	Type	Shape	Principal purpose
[9]	Extraction device	Conical with a handle, the shaft has multiple cavities (slots)	Capture powder for PSD
[11]	Capsule	Cylinder with multiple cavities	Thermal diffusivity measurement
[10]	Capsule	Cylinder with a single cavity	Powder bed density measurement
[16]	Capsule	Cylinder with a single cavity	Powder bed density measurement
[17]	Capsule	Cylinder with a single cavity	Powder bed density measurement
[14]	Capsule	Cylinder with a single hollow conical cavity	Capture powder and spatter for PSD
[13]	Capsule	Double cone with a single cavity	PSD and chemical analysis
[12]	Capsule	Cylinder with a single cavity, the top and bottom are open	Chemical analysis (Oxygen content)
Current work	Capsule	Box-shaped with multiple cavities	Chemical analysis

Since variations can arise in three dimensions, a universal design would aim to collect powder from particular regions in three dimensions within the powder bed. Therefore, the design of powder capture capsules is flexible, meaning it can be modified depending on the amount and location of powder capture. Ideally, the design of the powder capture capsule requires accuracy in powder capture and guarantees ease of powder extraction. The capsule in the current work at the National Institute of Standards and Technology (NIST) was designed specifically to extract unfused powder from a region consisting of stacked layers within the powder bed. Powder was extracted from three different heights using this capsule-based design. Specifically, this design is suited for capturing enough powder for chemical constituent analysis using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Additionally, this capsule design also allows measurement of the PSD of the captured powder and the powder bed density.

2. Method

Criteria and design of the powder capsule

For optimized powder collection, powder capsules should (a) contain the requisite amount of powder and (b) allow easy extraction of the powder. Provided these two criteria are met, capsules of different shapes can be designed with various types of cavities. The current capsule design combines a multi-stack of capsules into a single capsule enabling powder collection from three different heights as depicted in **Figure 1**. To remove the requirement for support structures of overhanging features, triangular channels were selected as shown in **Figure 1** (b, c). Designers can also choose other cross-section shapes; however, it would be convenient to eliminate any need for support structures. The ends of the channels are thin walls designed to be punched to extract powder. These thin walls should not be too thick that they are difficult to punch. To facilitate punching, the end walls of the triangular channels have a thickness of 0.4 mm and are recessed by 0.4 mm from the outer wall. A wall thinner than 0.4 mm (400 µm) was avoided to mitigate the chances of part failure [19]. This allows an alloy steel transfer punch with a 12.7 mm thickness and a flat-end tip dimension of 2.2 mm (0.09 in) to punch a hole. The punched hole diameter is close to 2.5 mm (≈ 0.1 in), which is used in the Hall flow meter (ASTM B213-20) [20]. Theoretically, it ensures ease of draining the powder capsule by gravity and tapping.

An EOS M290[1] LPBF machine was employed with unused nickel alloy 718 (IN718) powder feedstock (size range 20-55 µm with $d_{10} = 17.53$ µm, $d_{50} = 31.98$ µm, $d_{90} = 53.79$ µm) to print capsules on subplates. There are several techniques for chemical analysis. Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) was selected due to its relevance for future work with these powder capsules. The designers should choose the capture amount based on the technique of their choice. For this case, chemical analysis using ICP-OES requires powder in the range of > 10 g per measurement. Allowing a safety factor of 1.5, the goal amount of captured powder per stacked region (row) or from 8 triangles was set to 15 g. A density of 8.15 gcm^{-3} was used for IN718 according to the supplier's datasheet [21], and an average powder bed density of 40 % was assumed to calculate the volume of the channels. The powder bed density is intentionally a lower value since the unpunched channels can be kept intact if the desired quantities are extracted. Thereafter, the capsule was designed with eight triangular channels per stacked region. They possess isosceles triangle-shaped cross-sections with a 3 mm height, 6 mm base, and 65 mm channel length. As seen in **Fig.1**, the powder is captured from three different heights spaced at 2 mm between them.

[1] Certain commercial equipment, instruments, software, or materials, commercial or non-commercial, are identified in this paper in order to specify the experimental procedure adequately. Such identification does not imply recommendation or endorsement of any product or service by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

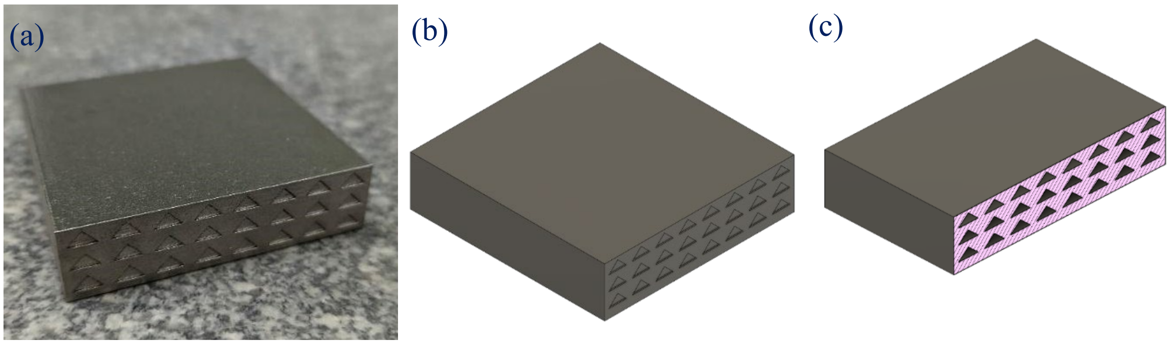

Fig.1. Design of the capsule, presenting (a) the actual print of the sample, (b) computer-aided design (CAD) for the capsule, and (c) CAD for its cross-section.

Procedure for powder extraction

The capsule separated from the build plate using electrical discharge machining (EDM) is shown in **Fig.1** (a). Subsequently, the powder capsule was mounted on a vise as shown in **Fig. 2** (a). Using a hammer and a transfer punch, holes can be easily made on the thin depressions as seen in **Fig. 2** (b). No plume formation or powder loss occurs when punching. After each hole is punched, the capsule was unlocked from the vise, inverted, and tapped over a glass vial for powder extraction. As predicted, each orifice was large enough for the powder to flow smoothly. To extract any remnant powder out of the capsule, a transfer pipette was filled with isopropanol and the powder was rinsed out of the capsule onto a glass dish. It was found that a negligible amount of powder (less than 1 mg) not detectable by the weighing scale was rinsed out of the capsule. It is also important to note that no debris of any kind was found after punching so chances of contamination were minimal. Thereafter the weight of the glass vial was measured to estimate the quantity of powder extracted from each hole. The glass vial was cleaned with isopropanol once weight is measured. Subsequently, the next hole is punched, and the described procedure is used to measure the quantity of powder extracted. Eventually, all holes were punched, and the weight of powder contained in each channel was measured. The powder capsule after punching all the holes is depicted below in **Fig. 2** (c).

Fig. 2. Depiction of the capsule (a) before punching shown with the hammer and transfer punch, (b) one hole is punched (the isopropanol bottle is shown for reference) then powder is extracted, and (c) after punching all holes.

3. Results and Discussions

The amount of powder extracted from every stacked region is shown below in **Table 3**. The extracted amount of powder is higher than the estimated amount (15 g) of powder by roughly 5 g. This is a result of using a low value of powder bed density used for estimation. Therefore, there is room to reduce the channel dimensions and still capture > 10g of powder. Assuming uniform powder bed densities and using the minimum solid density value of 8.15 g/cm ^{3}, the maximum powder bed density values were also evaluated for each region. Using theoretical values to approximate the volume of the triangular cross-sectioned cavities, the maximum powder bed density was found to be 54%. This is higher than the capsule design value of 40% and is close to that measured by Jacob et al. at NIST using nickel alloy 625 (IN625) powder [15].

As a follow-up, the internal triangular cross-sectional area was measured to determine the validity of the assumed channel volume used in the powder bed density values as presented in **Table 3**. The powder capsule was sectioned laterally into two parts and one section was examined using an optical microscope. An optical image of one channel is shown in **Fig. 3**. The edge lengths of four random triangles were measured and used to calculate the area. The mean calculated area was 8.67 % more than the assumed area. The actual volume of each channel could be measured following the procedures by Jacob et al. [15]. This was not performed in this study for two reasons. First, the powder capsule was designed to capture a specific amount of powder at a specific location and was not aimed toward accurate measurements of powder bed density. Second, once the punched holes are filled with deionized water, clearing it from the meniscus of a punched capsule is difficult and it leads to erroneous weight measurements.

Table 3. Details of amounts of powder extracted from the capsule.

Amount extracted (g)						Powder bed density (%)
Region	Channel Mean	Std. dev.	Total extracted per stacked region	Stacked region Mean	Std. dev.	
Bottom	2.597	0.011	20.773	20.713	0.058	50.106
Mid	2.591	0.011	20.73			50.003
Top	2.579	0.011	20.635			49.774

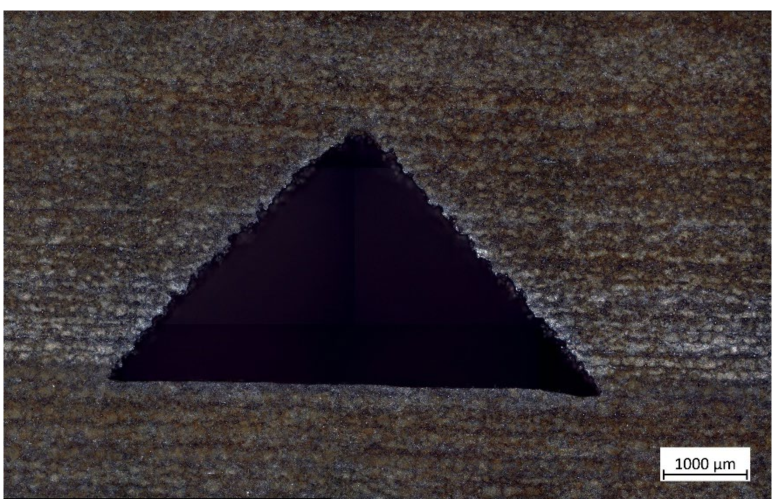

Fig. 3. Optical micrograph of a single channel cross-section.

4. Conclusion

The capsule effectively captures powder from three separate height regions (Z) and allows easy extraction of powder. The extracted powder can be analyzed for chemistry. An adequate amount of powder required for Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) was extracted. The average collection quantity of IN718 powder was approximately 20 g per region of the capsule. Subsequently, the average approximate powder bed density was evaluated to be around a maximum of 54%. As per user requirements, the dimensions of the channels can be adjusted to capture various powder amounts.

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