Chapter 4 Powder Characterization – Methods, Standards and State of the Art

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Abstract

Laser Powder Bed Fusion (L-PBF) is a technique in which a layer of metal powder is deposited, melted and solidified using a laser to create a solid layer of metal, with consecutive 2D layers being micro welded to preceding layers to build up fully solid 3D components. This technique has been the focus of considerable interest from research groups and industrial sectors where it is acknowledged to be a multi-variate process capable of producing highly complex geometries in low volumes. One of the most important factors to control is the quality and properties of the powder feedstock used. In this chapter, the various properties of metal powders that have been shown to affect the quality of fabricated parts are discussed. How the powder flows and spreads within the L-PBF process, and how the shape, size and composition of powders are investigated and can lead to different defects and porosities in parts are presented. This chapter provides an overview of how the mechanical and thermal properties of powders can impact the L-PBF process. The sustainability and recycling of such material feedstock within the powder life cycle are discussed. The international standards for analysis and safety considerations of powder which are relevant to additive manufacturing are also presented.

Key Words: Powder Rheology, Powder Shape and Morphology, Powder Composition, Mechanical Properties, Laser Powder Bed Fusion (L-PBF), Powder Sustainability

4.1. Introduction

Powder is a complex material form, composed of solid (the powder particles), liquid (moisture or solvent on the particle surface) and gas (usually air, however, as we will see later, this can also be inert gases such as argon or nitrogen) entrained between the particles. Therefore, we can expect a complex interplay of properties such as shape, size and flow as well as humidity, thermal conductivity and mechanical strength, all of which will be affected by the process in which the powder is utilized. The focus of this chapter is to give an understanding of how powder properties are investigated and quantified, and how these are relevant to additive manufacturing. For the scope of this chapter, additive manufacturing will be taken to mean L-PBF, however, other processes such as Direct Energy Deposition (DED) and Electron Beam Melting (EBM) also use a powder feedstock. In earlier chapters, the process and parameters of the L-PBF operation were discussed and will not be repeated here.

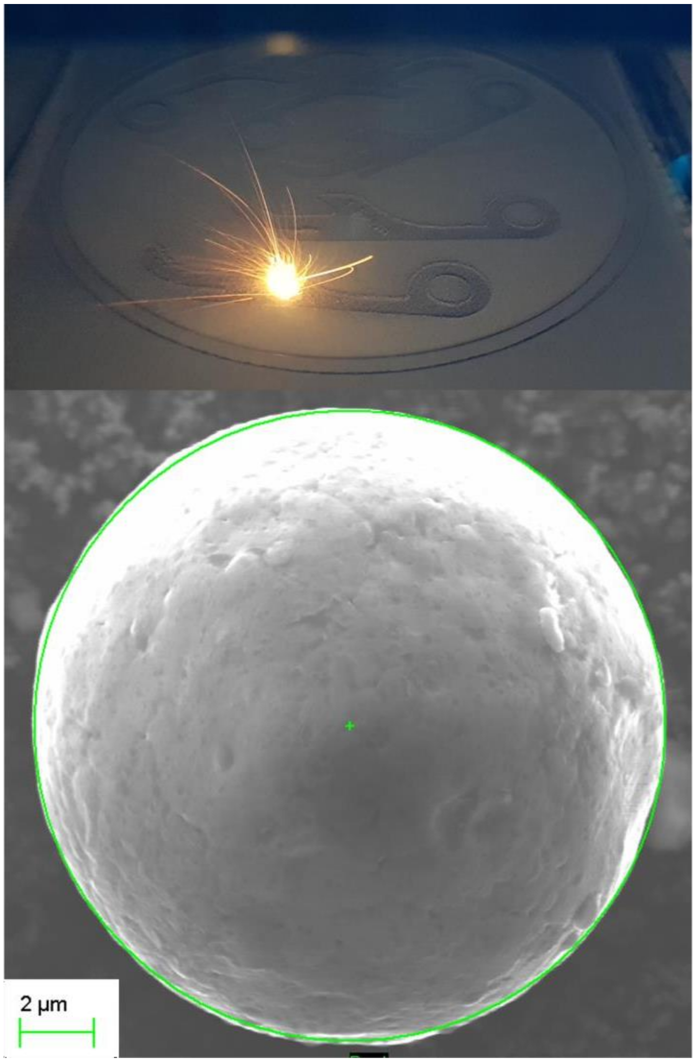


Figure 1: A microscope image of a single 316L stainless steel powder particle, magnified 10960 times.

Metallic powders can be produced from a number of different methods, yet they all involve atomization of a solid metallic feedstock, for example and ingot. The methods differ in the medium of atomization, namely water, gas or plasma. In our experience, powder produced from water atomization are less spherical and have a wider size distribution. Gas and plasma atomization methods both yield more spherical and uniform powder particles. The production methods are discussed in greater detail in section 4.6.1 below. Given that L-PBF has around 200 parameters which can affect the quality of the parts fabricated, it is widely agreed that it is a very complicated process. Therefore, it is essential that a thorough understanding and quantification of numerous powder properties be obtained, prior to a powder feedstock being used in the process. It is still however, a matter of some debate as to the "ideal" powder properties, this is likely due to the number of available materials (pure metals and alloys), variability between suppliers, batch-to-batch variability, variability in how the same powder from the same batch will behave in different L-PBF machines, and also how different machine operators store, handle and use the powders. This makes the characterization of the powder properties all the more important, since, if they can be quantified, then one source of variability can be, if not controlled, then at least limited and understood within the process. In this chapter, the following powder properties will be discussed; rheology or flow, size shape and morphology (shape, circularity and aspect ratio of individual particles), elemental composition and thermal, mechanical and hygroscopic characteristics. In each section, a discussion of the relevant international standards of analytical methods is presented along with a consideration of how these powder properties pertain to additive manufacturing. Important industrial and academic contributions to these methods and to the overall powder life cycle and sustainability of the L-PBF process will be highlighted and discussed. This chapter is not intended to be an exhaustive review of these areas, but a high-level snapshot of the current best practices and standards.

The standards noted are from the ASTM International, Metal Powder Industries Federation (MPIF), International Organization for Standardization (ISO) and DIN (German national organization for standardization), etc. standard databases, where appropriate and available for each analytical technique.

4.2. Powder Rheology

4.2.1. Methods

Powder flow and powder spreadability are complex multi-variate phenomena. The former has been investigated over the last number of decades, and a number of standard methods exist to quantify and compare powders of similar materials or batches. Powder flow methods can be static or dynamic. For example, the angle of repose is a static measurement, since the powder is allowed to stabilize prior to the measurement whereas the application of a moving blade within the powder while recording torque would be considered as a dynamic measurement. It is still a matter of discussion as to the relevance of each method for a particular process. Some testing methods yield a quantity and a unit, while others provide a unitless quantity or empirical value, which on comparison with that of another powder can be used to evaluate which is more suitable for a given process.

Hall Flowmeter

This method was first developed in 1945 and is documented in the MPIF and ASTM standards (ASTM -B213, 2014; MPIF, 2019)[1,2]. The procedure involves passing 50 g of powder through a funnel of specific geometry and size, the hole in the funnel is of 2.5 mm diameter. The time required for the powder to pass through the funnel is measured, and from this, the flow rate is determined. The test may be run in static (where the flow of the powder is initially blocked) or dynamic (where the powder is poured into the funnel and allowed to flow right through it) into an empty weighing dish. The apparent density of a powder can also be determined using a Hall apparatus (ASTM - B964-16, 2016)[1] and an Arnold Meter (ASTM - B855-17, 2017; MPIF, 2019). The Carney method is a similar procedure and is used when the powder does not pass through the Hall funnel orifice (and is therefore not considered free-flowing (ASTM - B964-16, 2016). Additionally, there are a number of other standardized methods of evaluating tapped and bulk densities of powders. Tap density is defined as the density of a powder when the receptacle of known volume is tapped or vibrated under specified conditions. Tapping or vibrating a loose powder induces movement and separation and lowers the friction between the powder particles. This short-term lowering in friction results in powder packing and in a higher calculated density of the powder mass. Tap density is a function of particle shape, particle porosity, and particle size distribution.

A number of standards are available, collected in the MPIF standard publication (MPIF, 2019), for tapped density, consult standard 46, and for apparent density measurements, standards 4 (using Hall apparatus) and 28 (using a Carney funnel) are most relevant. Their ASTM counterparts are (ASTM -B213, 2014; ASTM B527-20, 2020). ISO standards for this measurement are codified in ISO 3953 (ISO, 2011).

The tapping mechanism is important, and a calibrated mechanical tapping machine should be used. A graduated cylinder should be used to measure the volume of the powder under investigation.

In the initial test, the number of taps, N, should be that required such that no further decrease in the volume of the powder is observed. In practice, once N is established, a tap number value of 2N should be used, or a value based on experience with the particular powder. However, for reproducibility purposes, the value should be documented and periodically re-checked.

For apparent density measurements using a Carney funnel (of 5 mm orifice), a test sample of powder is loaded into the funnel and allowed to flow through and fill the density cup container, see Figure 1. The volume of the density cup is accurately known. The mass of the powder in the density cup after levelling of the powder on the top of the density cup is then determined. Replicates can be carried out and an average obtained. The experimental setup is shown in Fig 2, (MPIF, 2019).

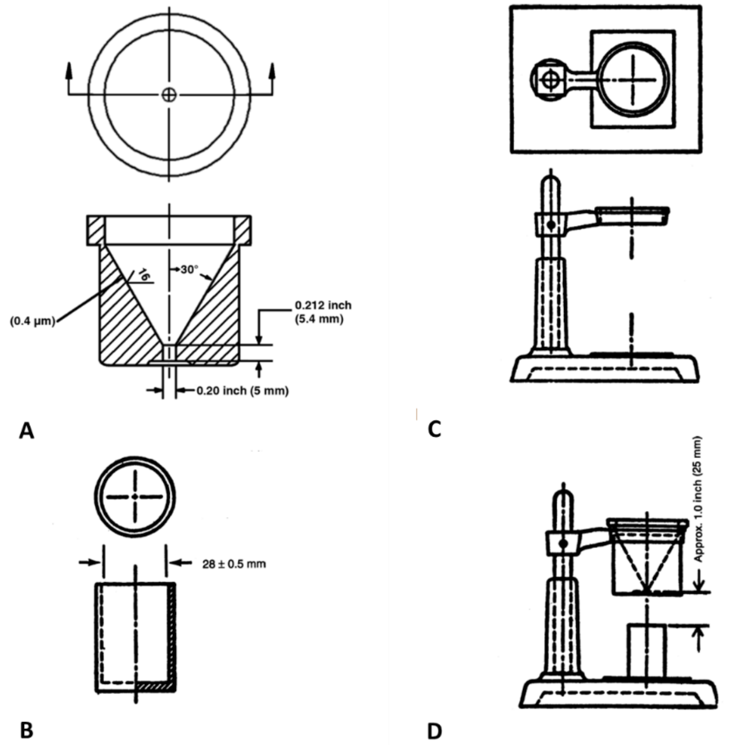


Figure 1: A: The schematic drawing of the Carney Funnel, B: schematic drawing of the density cup, C: stand required for the funnel and cup, maintaining the correct distance between both, and D: the complete setup; adapted from (MPIF, 2019).

A method known as Carr Indices (ASTM D6393-14, 2014) is used to quantify a number of bulk powder properties such as cohesion, angle of repose, bulk densities, and powder dispersibility. This standard method is suitable for free flowing and moderately cohesive powders, and granular materials of up to 2 mm diameter, and must be able to flow through a nozzle of 6 mm to 8 mm in diameter. Angle of repose is defined as the maximum angle a mound of powder makes with the surface it is deposited on, at which it is stable and does not fall (no powder movement on slope) (ASTM D6393-14, 2014). There are a number of other methods which can be used for determining the angle of repose of a powder, which can lead to confusion amongst researchers, however, since this method is mainly for powders of larger particle size (sands), it is not as widely used in L-PBF powder research as the other methods described here. Powders are cohesive if they clump or aggregate during flow. In general, metal powders are not considered cohesive under a flow regime, given their high density and aeration behavior.

The Arnold meter is a technique which requires a higher degree of operator training, as the powder deposition method and filling method of the stainless-steel die is difficult and as such is more prone to variability and error.

In recent years, a number of other techniques have been developed to analyze powder in both static and dynamic regimes and are applicable to a wide range of material types and particle sizes. Two will be discussed in detail here and are considered the current best practice in additive manufacturing labs around the world for powder flow analysis. They use different methods to induce a flow in the powder sample, and yield different, yet somewhat complementary results.

Dynamic testing flow regime 1

The Freeman Technology FT4 (Freeman Technology, 2016) instrument uses a precisely machined 23.5 mm stainless steel blade to measure a number of properties (dynamic flow, shear and bulk properties) of a powder sample (see Figure 3). These include basic flowability energy (BFE), specific energy (SE), flow rate index (FRI), minimum aeration velocity, as well as bulk and tapped densities. These tests are conducted on precise masses of powder, and the blade is rotated and lowered through the powder at a defined rotational and vertical velocity. The blade experiences a torque as it passes through the powder. Bulk, dynamic, wall friction and shear force tests can be performed. The wall friction test is in accordance with ASTM Standard D7891 (ASTM - D7891, 2015).

The stability of a powder can be measured with the procedure as follows. In passing through the powder, the blade measures the resistance to flow exhibited by the powder over several repetitions (tests 1-7) and the velocity of the blade is varied to discrete values for each remaining test (tests 8 - 11). This variation in torque as a function of powder height and blade velocity is calculated as the BFE while the blade is moving downwards, known as the confined regime. When the blade moves back up through the powder it is in the unconfined regime, and in this test the SE is calculated. These can be expressed as mJ/g of powder (Freeman Technology W7013, 2007; Freeman Technology W7030, 2008; Freeman Technology W7031, 2008).

The free-flowing nature of a powder, or how easily fluidized it is can be quantified by a similar method, except that during this test, compressed air isallowed to flow upwards through the vessel and the powder via the mesh base of the vessel. The velocity of the air is precisely controlled and the variation in the BFE is plotted as a function of the air velocity. The velocity of the air at which the BFE is at or near zero is taken to be the minimum fluidization velocity. This is therefore a measure of how easy the powder is to fluidize and therefore of how free flowing it is.

The compressibility of the powder can also be calculated using the FT4, using a vented piston in place of the blade. The height of the piston is measured precisely as incrementally increasing kinematic forces are applied to the powder. The compressibility percentage of the powder is thereby calculated. This is influenced by packing efficiency, hardness, chemistry, particle shape, and size. If a powder possesses a large number of satellite particles, the breaking of these particles from the larger ones can potentially be seen in the variation of the compressibility, if a large non-linear shift is observed, particularly at higher applied forces.

Interpretation of the results is based on the values of the various calculated parameters, and in which range of values they fall. Powders can be identified as cohesive or non-cohesive, free flowing or aggregating, stable or unstable. However, it should be pointed out that reliance on just one test or calculated value for the determination of the powder properties is not recommended. Values should not be considered in isolation, and may in fact provide conflicting interpretations of the properties. The interpretation of rheological properties is a complex science, and additional characterization tools should also be employed to better understand the results.

Dynamic flow testing regime 2

An alternative and complementary measurement device to the FT4, is the Revolution device (Mercury Scientific, 2020) which utilizes a rotating drum in which the powder is placed. Figure 3 illustrates the experimental setup. A camera is placed at one end of the drum and the drum is rotated at a defined rpm. As the powder rotates, it undergoes what is termed as an "avalanche event". The precise surface of the powder as each avalanche occurs is imaged and a number of parameters such as surface fractal, avalanche energy, as well as rest and avalanche angles are measured and averaged over a series of such events. This is a different flow regime to that of the Freeman device, yet is also appropriate for powder in an additive manufacturing application. Again, interpretation of the results is difficult and requires operator experience. The flowability of the powder is interpreted as a function of the avalanche angle. The lower the angle, the higher the flowability, i.e. the better the powder flows. The rest angle is comparable to the angle of repose of a powder sample. The rotation speed can be varied to account for different flow regimes under investigation. The Revolution device can also be used to investigate the packing efficiency of the powder after it has been subjected to a vibrational energy from the rotating drum (Mercury Scientific, 2020).

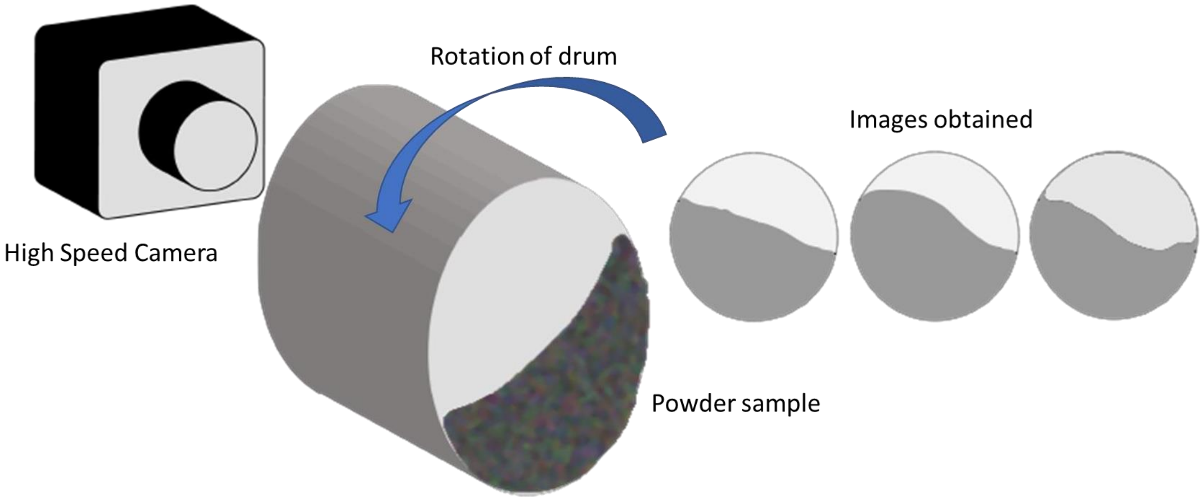


Figure 3: Experimental setup of the Revolution powder rheology analyser. A high-speed camera captures images of the rotating powder, and the avalanche events it undergoes. On the right hand side, a set of typical images of the

avalanche event from the camera point of view are shown. Imagine supplied by the author Robert Groarke.

4.2.2 Applications of Powder Rheology Measurement in Additive Manufacturing

The understanding of how a powder flows and spreads is of critical importance in many AM techniques, but in particular in L-PBF. Part density, microstructure and surface finish are some of the part properties that rely on the formation of a well packed, evenly distributed layer of powder, and necessitate layers to be consistently formed in this way. Powder flow is affected by particle size and shape, as well as by cohesivity, density, packing efficiency, permeability. Various research groups have investigated the influence of powder properties on resultant part properties in L-PBF processes, as well as the interplays of various powder parameters on each other. Much of the research has been focused on 316L stainless steel which is one of the most commonly used materials in metallic additive manufacturing, however other materials have also been studied. (Klausner, *et al.*, 2000; Clayton *et al.*, 2015; Strondl *et al.*, 2015; Hausnerova *et al.*, 2017; Liverani *et al.*, 2017; Kurzynowski *et al.*, 2018). Increasingly, a different interpretation of flow is being proposed as an area of study, particularly for AM, but also as a regime which may be suitable for certain other powder applications. It focusses on how a powder is delivered across a flat surface, mimicking a build plate in a L-PBF machine. The effect of powder rheology and powder delivery dynamics on the AM process, and in terms of the basic science, has been increasingly a source of interest (Lyckfeldt *et al.*, 2013; Spierings *et al.*, 2016; Hausnerova *et al.*, 2017; Escano *et al.*, 2018; Chen *et al.*, 2019; Snow *et al.*, 2019). The two rheological devices discussed in the previous section 4.2.2 that are the most relevant to L-PBF processes are the FT4 and the Revolution devices, though in differing ways. We must consider how the powder spreads and flows, upon its interaction with itself, the surrounding boundaries, and the recoating mechanism in the AM device. It may be argued that the FT4 blade rotating through the powder is one way of simulating the flow of the powder under the applied force of the moving re-coater mechanism, on a quasi-bulk scale. The Revolution may be considered to yield important information regarding the nature of the "leading edge" of the powder, investigating as it does the formation of an avalanche event, and the angle at which the powder starts to move downward and become less stable (beyond the rest angle). This may be important in order to understand why powders may not form stable layers of consistent height, depending on recoating velocity, re-coater height and particularly for larger layer heights. The Revolution sample drum can also be filled with an inert gas for powders which are hygroscopic or air sensitive. The FT4 can give information about how resistant a powder is to flow, how likely aggregation is to occur, how compressible a powder is, which will inform how well a powder will pack. Therefore, it is readily seen that both techniques have a place in the characterization of powder behavior in an additive manufacturing process. However, powder rheology should not be studied in isolation. There are many other properties of powders which must also be understood in the context of their relevance and application to L-PBF, which will be addressed in the following section.

4.2.3 Powder Rheology Standards

Table 2 lists the important international standards for powder rheology and flow. It is important to note that two other standards are being developed which are related to the characterization of powder rheology. These pertain specifically to additive manufacturing and are given the working designations ASTM WK55610 and ASTM/ISO DIS 52907 (America Makes and AMSC, 2018). While there is no specific standard for powder delivery, a shear cell test can be used to approximate this effect but a quantitative standard is still required (America Makes and AMSC, 2018).

Table 2: International standards used for powder rheology assessment.275

TEST/METHOD	ASTM	ISO	MPIF
Hall Flowmeter	ASTM - B213, 2014	ISO 4490	MPIF, 2019 Page 17
Apparent Density	ASTM - B964-16 ASTM - B855-17 ASTM - B212	ISO 3923/1	MPIF, 2019 Page 21
Tapped Density	ASTM - B213, 2014 ASTM – B527, 2020	ISO 3953	
Carr Indices	ASTM - D6393-14		
Angle of Repose	ASTM - D6393-14	ISO 902 (1976)	
Shear Cell Tests (Spreadability)	ASTM - D6128-16 ASTM - D6773-16 ASTM - D7891-15		

4.3 Powder Shape, Size and Morphology

4.3.1 Methods

As discussed in the preceding section 4.0, the flow behavior of powder is a complex phenomenon, and is very relevant to the success and reproducibility of a L-PBF process. This flow behavior can be influenced by the shape, size and morphology of the powder particles. In this section we will discuss how such characteristics are analyzed and quantified. The basis of most techniques is a microscope and image analysis software. The difference between techniques is generally a case of throughput, how many individual particles can be analyzed in a reasonable amount of time, while still allowing for statistically relevant deductions to be concluded about the bulk sample. A sample of powder which has sampled correctly can be considered a representative sample of the whole. The sampling techniques which are considered best practice as well as appropriate tools required are codified in international standards such as (ASTM B215-15, 2012). In this section, several standards and somewhat novel methods for characterization of powder shape, dimensions and morphology are considered.

There are a number of methods by which the average dimensions of the particles in a powder sample may be measured. The simplest means of measuring the particle size distribution of a sample is by using a series of sieves of calibrated mesh sizes (pore sizes) and passing the powder through the sieves using a vibratory motion. The amount of material remaining in each sieve plate at the end of the test is tabulated relative to the total mass of the sample. This approach is codified in the MPIF standard number 5 (MPIF, 2019) and is also dealt with in an ASTM standard (ASTM B214-16, 2016). For additional guidance, ASTM F3049-14 can also be used (ASTM F3049-14, 2014). For this method, the powder is measured as a solid however, the measurement can also be carried out in a solvent matrix. The conventional wisdom is that the powder should be measured in the form in which it is utilized in the process. In the case of additive manufacturing, therefore, the particle size measurement should be carried out on the powder in the solid form. The type of technique employed is somewhat dictated by the expected size range of the particles, for example Dynamic Light Scattering (DLS) would be ideal for nanoparticles, but less suited to powder particle size ranges typically found in L-PBF processes, which are generally of the order of 10-100 µm. For particles in the latter range, Laser Diffraction (LD) is more appropriate. According to the definition from Malvern Panalytical, DLS is recommended for particles and dispersions in the range of 1 nm to 10 µm, whereas LD has a broader particle size range of application (sub-micron to mm) (Malvern Panalytical, 2020). This technique also has the advantages of rapid measurement time, large particle sampling, ease of interpretation, and can be integrated at or online to the process. In terms of standards it is codified in ISO 13320 (2020). It is suited to both spherical and non-spherical particles. The results are reported as either a volume-based distribution or a number-based distribution. The results are summarized as D_{10}, D_{50} and D_{90}, which is the particle size below which 10, 50 and 90% of the total volume or total number of particles lies. Modern LD systems will give an indication of the reliability of the result or results, and can be configured to report the values in accordance with various standards or industrial settings for statistical analysis, and to ensure compliance for regulatory testing environments. Care must be taken during the experiment that the powder feed is controlled and constant, to ensure a consistent occlusion of the beam by the particles. A third approach is to examine the particles using a Scanning Electron Microscope (SEM), along with image analysis software such as ImageJ. The analyst then selects individual particles and adjusts the contrast of the image within the software to yield a grayscale (for example a 16-bit scale version of the image) where the selected particles are seen. The software then calculates the dimensions of the particles based on scaling data provided by the analyst. This approach is not designed for high-throughput applications as it is a time-consuming process and is not designed to allow a large number of particles to be analyzed, not least because the SEM image itself even at low magnification will show perhaps a few hundred particles. However, with the advent of AI, this technique may see a resurgence, as it may allow a vast number of images and particles to be analyzed, but these images must still be acquired, therefore it is still only ideal for small scale samples. This technique is similar to the basis of operation of the Malvern Morphologi G4 instrument (https://www.malvernpanalytical.com/en/products/product-range/morphologi-range/morphologi-4). This uses compressed air to deposit a precise volume of particles on to a glass plate. This is then imaged using an optical microscope. This is essentially a 3D microscope, as vertical “stacking” of images can be performed to clarify if a particle is indeed a single, mis-shaped particle or in fact two particles fused or touching. The proprietary software allows for upwards of 400,000 particles to be individually imaged per sample, and their dimensions to be calculated. Specific analysis criteria for the size and shape of the particles can be set, to remove certain unwanted particles (or dust) from the calculation. This instrument reports particle shape data in the form of a large number of parameters.. As with all microscopic based methods, care must be taken to ensure that particles are not touching each other, which is why the SEM approach is more prone to errors. The data allows for detailed quantitative comparisons to be made between powder samples and can be correlated with SEM images. 4.3.2

Applications of Powder, Shape, Size and Morphology Measurement in Additive Manufacturing

As with other powder processing methods, knowledge of particle size and shape is important process information for L-PBF. The lower limit of layer height chosen for a build is often determined by the D_{50} of the powder sample with the layer thickness selected not being lower than this. This will in turn dictate the laser power parameters, in order to ensure melting and partial re-melting of previous layers. Particle shape is important as this is a key factor in how a powder will pack within the layer or layers and will affect the contact between powder particles, both in the plane of the build plate, but also vertically through the build. This in turn determines the heat affected zone and the thermal conductivity through the powder. Taken together, these factors will influence the level of powder melting, defect formation and porosity. The powder particles can also be analyzed post-build, to see if their shape or size has been changed, invariably there are fused particles which have been ejected from the build layer by the laser energy. This spatter phenomenon has recently been examined and shown to be more significant for altering particle shape and size though agglomeration and coalescence than change of the bulk particle crystal structure (Obeidi *et al.*, 2020). The effect of powder shape on packing and flow, and subsequent part properties using micro-CT has also recently been examined (Brika *et al.*, 2020). In this work it was found that spherical particles resulted in parts with better mechanical properties, while higher layer heights within the range examined also produced parts with improved mechanical properties. Interestingly, they also found that samples manufactured from powders with differing morphologies and rheological characteristics did not have measurably different mechanical properties. This illustrates how complex the L-PBF process is, and while certain characteristics may not lead to significantly different part properties, a quantitative analysis of the feedstock is still an important research topic to allow for improved process control and sustainability.

4.3.3 Powder Morphology Standards

The international standards for powder morphology assessment are shown in Table 2. Further progress in these methods is required to improve repeatability and reproducibility of results (America Makes and AMSC, 2018).

Table 2: International standards in particle size and shape analysis

NAME / TEST	ASTM	ISO	MPIF
Standard Test Method for Sieve Analysis of Metal Powders	ASTM – B214-16		
Standard Practices for Sampling Metal Powders	ASTM – B215-15		
Estimating Average Particle Size of Metal Powders Using Air Permeability			Standard 32
Particle Sizing using Light Scattering	ASTM – B822-17		
Particle Sizing using Laser Diffraction		ISO13320 - 2009	

Particle Size Result Presentation		ISO 9276, Parts 1-6	
Standard Guide for Characterizing Properties of Metal Powders Used in Additive Manufacturing Processes	ASTM F3049-14		

4.4 Chemical Composition of Powders

The chemical composition of the powder samples (powder chemistry) is critical in determining properties of final L-PBF produced parts. Impurities may be introduced during the manufacture and handling of the powder feedstock and thus will be incorporated into the melt pool during processing. These impurities can remain as discrete particulates or non-fused interfaces in the produced parts which then can act as stress concentrators and may reduce fatigue life by increasing the probability of fatigue crack initiation. Similarly, the presence of elements such as carbon, oxygen, nitrogen, sulphur and hydrogen can influence the physical properties of the final product. Methods used for the powder chemistry analysis can be divided into three types, surface, micro and bulk analysis techniques. Bulk chemistry analysis and validation are particularly important to ensure that recycled, as well as virgin alloy powders, meet their purity standards and alloy designation. Many techniques are available for powder chemistry analysis and suitable methods can be used depending on the elements of interest and level of accuracy needed for the final applications (Samal *et al.*, 2015).

4.4.1 Methods

X-ray photoelectron spectroscopy

The X-ray photoelectron spectroscopy (XPS) technique is an extensively used method for surface chemical composition analysis. It can be used to measure both the presence and bonding state of elements near the surface (typically <10 nm for lab based XPS; and <100 nm High Energy XPS) of the powder particles. This technique is based on the photoelectric effect, in which the material is irradiated/bombarded with X-rays and the kinetic energy of the ejected core-level electrons are measured. The binding energy of the ejected photoelectrons from the powder samples can be calculated using the knowledge of the kinetic energy of the ejected electrons (using electron analyzer), energy of the x-rays and the work function of the spectrometer. XPS analysis will provide information on the elemental composition as well as the chemical state of the powder surfaces, as the core-electron binding energy represents the characteristics of an element in a particular chemical environment. Thus, it is possible to determine quantitative information of the elements present as well as their oxidation states on the surface layers of the powder particles. XPS can detect all elements except hydrogen and helium with a detection limit of <0.1 atomic percentage (Slotwinski *et al.*, 2014), however, it depends on the elements and the matrix in which it is present

(Shard *et al.*, 2014). It requires the use of ultrahigh vacuum for the sample analysis and the measurement area can range from 70 μm^2 to 1 cm^2 and the lateral resolution of commercial XPS instruments is typically about 10 μm (Slotwinski *et al.*, 2014).

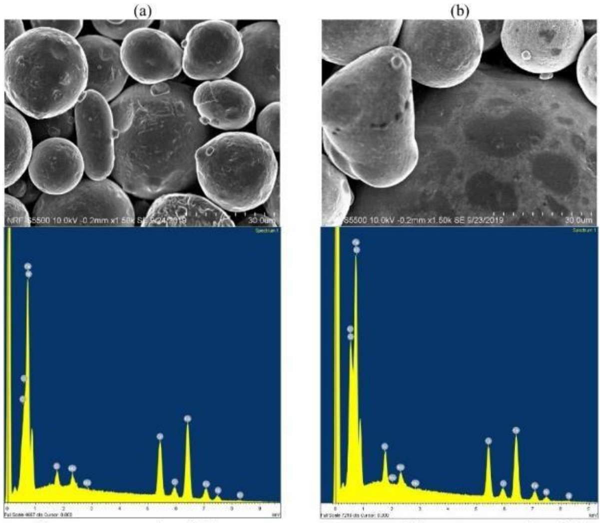
The XPS technique can also be utilized to extract elemental analysis at a particular depth from the surface by combining it with an ion sputtering capability. Thus a depth profile of elemental composition versus sputtering time can be obtained, in which the sputtering time can be correlated to the depth (Gruber *et al.*, 2019). For example, this technique has been used to determine oxide layer thickness in powder samples, however, the elemental composition analysis may not be very accurate due to the possible effects of (i) ion beam damage, (ii) preferential elemental sputtering, and (iii) the curved nature of the powder particle surface. ASTM E 1829 represents Standard Guide for Handling Specimens Prior to Surface Analysis (ASTM E1829, 2014)

Auger electron spectroscopy

Auger Electron Spectroscopy (AES) is a surface sensitive quantitative elemental analysis technique, in which L-level (auger electrons) electrons will be ejected after a series of electron transitions, from the material, by the irradiation of an electron beam. Similar to XPS, this technique can be used for the quantitative detection of all elements except hydrogen and helium, along with some information on the chemical state, within a depth of 2 nm. While both AES and XPS are surface analysis techniques, changes in the electron escape depth results in differences in the sample volume analyzed using the two methods. AES has an advantage of higher spatial resolution (compared to the XPS). Similar to XPS, AES also can be used for depth profile analysis of elements and to determine oxide layer thickness on the powder particles (Gruber *et al.*, 2019).. AES has a depth resolution of 5–25 Å. AES also require the use of ultra-high vacuum for the analysis as in the case of XPS. ASTM E1127 represents a Guide for Depth Profiling in Auger Electron Spectroscopy (ASTM E1127, 2015).

SEM – Energy Dispersive X-ray Spectroscopy (micro analysis)

Energy dispersive X-ray analysis (EDS or EDX) is a widely used analytical technique, generally performed in combination with SEM or TEM, to carry out semi-quantitative elemental or compositional analysis of the powder particles. EDS utilizes the X-ray signals produced due to the interaction of the SEM’s electron beam with the powder samples. Primary electrons when incident on the powder sample surface eject inner shell electrons, and x-rays are produced by the transition of outer shell electrons to fill up the vacancy in the inner shell. Each element produces a characteristics X-ray emission pattern due to its unique atomic structure, and hence can be used to perform chemical/compositional analysis with an energy dispersive spectrometer. The analysis of these peaks provides qualitative as well as semi-quantitative information on the material. The position of the peaks in the resulting spectrum gives information on the type of elements present in the sample and area/peak height measurement provide semi-quantitative information on the concentration of the element in the sample. A more refined quantitative result can be obtained by measuring a standard of known chemistry. The area under the peaks can be generally correlated to the weight percentage of the elements and this semi-quantitative information is very useful to make a comparison between different particles (Mussatto *et al.*, 2019, Obeidi *et al.*, 2020), see a particle EDS result example in Figure 4. Nevertheless, more accurate compositional analysis on spherical powder particle can be difficult to perform as the EDS technique has been found to work better on flat surfaces (Sutton *et al.*, 2016).



(a)

Element	Atomic%
Fe	55.37
Ni	17.55
Cr	13.15
O	4.20
Si	1.51
Mo	1.12

(b)

Element	Atomic%
Fe	52.30
Ni	13.82
Cr	16.27
O	14.17
Si	2.48
Mo	0.96

Figure 4: EDX comparison between the surface chemical composition of a (a) virgin stainless steel powder particle, and (b) spattered particles (Obeidi *et al.*, 2020)

Chemical composition analysis in a microscopic area is possible using EDS due to the capability to focus electron beam to an area of this size. The interaction volume of the EDS X-ray microanalysis can be varied by changing the accelerating voltage used for imaging the sample. The interaction volume is approximately 1 μm in steel at 15 kV accelerating voltage (Slotwinski *et al.*, 2014). Since EDS is a semi-quantitative composition analysis technique, appropriate reference standards should ideally be used for the system calibration in order to extract more accurate quantitative information from the sample.

EDS suffers from difficulties such as overlapping peaks (poor energy resolution) and inability to detect light elements. The use of wavelength dispersive spectroscopy (WDS) can improve the energy resolution and increase the accuracy of elemental quantification compared to EDS. ASTM E 1508 is the “Standard Guide for Quantitative Analysis by Energy-Dispersive Spectroscopy” (ASTM E1508, 2012). ASTM E 1078 is the standard guide for “Specimen Preparation and Mounting in Surface Analysis”.

Inductively coupled plasma optical emission spectroscopy

Inductively coupled plasma optical emission spectroscopy (ICP-OES) or atomic emission spectroscopy (ICP-AES) is used for the identification and quantitative determination of elements present in the powder samples. In this technique the sample in the liquid form, for example metal powder dissolved in acid solutions, is injected into the plasma, which is used as the excitation source. The plasma excites electrons in the elements and their de-excitation results in the emission of characteristic wavelengths, which can be used for the composition analysis. The emitted wavelengths are measured using a spectrometer. This technique can be used to measure major as well as trace elements simultaneously. ICP-OES produces qualitative elemental information by measuring the intensity of the emission peaks which correspond to the various elements. Since the elements generally have numerous emission peaks, specific emission lines will be used for different elements in order to avoid any peak overlapping (Sutton *et al.*, 2016). The concentration detection accuracy of this technique can be improved (up to 3 decimal places) by using an internal standard. X-ray fluorescence spectroscopy The X-ray fluorescence (XRF) spectroscopy technique is used for the qualitative and quantitative analysis of powder samples. XRF identifies elements in the sample by detecting characteristic X- rays emitted from the respective elements after the irradiation with high energy primary X-rays. XRF generally detects elements with accuracy at the ppm (parts per million) level.

X-Ray Diffraction (bulk)

X-ray Diffraction (XRD) is an analytical technique used to characterize phase, crystal structure and composition of the bulk powder particles. A beam of X-rays is directed on the crystalline powder materials, in which atomic planes are arranged in a regular manner, thereby scattering the X-rays in a regulated manner. The interatomic distances in crystalline solids (few angstroms) are of the same order as that of the X-rays and the scattered X-rays produce the diffraction pattern where the Bragg’s Law condition ($n\lambda=2d\ \mathrm{Sin}\theta$) is satisfied. Thus, the X-ray diffraction patterns, which consists of diffracted X-ray intensity as function of diffraction angle, observed from a material will be the “fingerprint” of that material. These XRD patterns are used to identify crystal structure and phases, and can be used to measure micro strain, grain size, crystal orientation etc. Powder sample chemistry can be measured by comparing with diffraction patterns from materials of the same chemistry, which are available for many materials in the powder diffraction data base (ICDD). ASTM E975 is a standard practice for the X-ray determination of austenite in steel (ASTM E975, 2013). Rietveld refinement analysis of the XRD data can provide a quantitative estimation of different phases, if more than one crystalline phases are present in the powder samples (Rietveld, 1967, 1969; Slotwinski *et al.*, 2014). Inert gas fusion (bulk) Inert gas Fusion (IGF) is a quantitative analytical technique used to determine the amount of hydrogen, oxygen and nitrogen in the metal powders. The presence of H, O and N has a significant influence in determining the mechanical properties, shelf life and quality of the metallic parts/materials. Hence their identification and quantification in the metal powder samples are important for the quality control process. To meet specification for a given application, the determination of these impurity levels is essential.

In this technique, the powder sample is melted in a graphite crucible at very high temperature over which an inert carrier gas is flowed. As the powder sample melts, the hydrogen present in the sample is released as molecular hydrogen, nitrogen as molecular nitrogen, and oxygen present in the sample can react with the carbon in the graphite crucible and produce carbon monoxide (CO) and carbon dioxide (CO_2). The gases produced are swept by the inert gas flow onto a detector where they are analyzed separately to yield a weight percentage of the elements present. This technique described by ASTM E1409-08 (ASTM E1409, 2008), ASTM E1447-08 (ASTM E1447, 2016) and E2792-11 (ASTM-E2792, 2016).

4.4.2 Applications of Composition Measurement in Additive Manufacturing

Quality and chemistry of powder feedstock plays a crucial role in determining the properties of the parts and are key to the additive manufacturing quality control process. The quality of the powder feedstock influences features such as (i) manufacture of defect-free parts, (ii) build-to-build consistency, (iii) manufacturing defects on surfaces, and (iv) reproducibility between additive manufacturing machines (EPMA, 2019). The chemical composition of the powder can change the melting and solidification behavior during the PBF process and affect parts properties. The non-metallic elements like oxygen, nitrogen, carbon, sulfur and hydrogen, which may be present in the powder feedstock or introduced during the manufacturing process, will significantly influence the physical properties of the additive manufactured parts.

Some powder surfaces are more susceptible to oxidation, moisture adsorption and hydroxide layer growth. The powder properties may also change with re-use due to the repeated exposure to the build chamber conditions. In all the above situations, powder composition analysis is crucial for the quality control of the final product.

Studies show that oxidation of the powder particles are detrimental due to their ability to maintain a low level of porosity in the parts produced. For example, Simchi *et al.*. (2004) found an increase in the porosity of steel manufactured parts with an increase in the initial oxygen content in the powder. Similarly, Leung *et al.* (Leung *et al.*, 2019), investigated the effect of powder oxidation on the molten pool dynamics and defect formation during laser additive manufacturing of Invar 36 powder. In a different EBM study, Tang *et al.* (Tang *et al.*, 2015) observed an increase in the oxygen content of Ti6Al4V due to powder re-use, which eventually exceeds the maximum specification for oxygen content in the material, indicating that the powder may not be suitable for use after more than four re-cycles.

It is important to recognize that, depending on the analytical method used for the chemical analysis of additive manufacturing powders and the element of interest, each method has its own limitations to perform accurate elemental analysis. For example, EDS cannot detect the lightest elements and has poor energy resolution. More reliable quantitative information can be extracted using destructive bulk chemical analysis..

4.4.3 Powder Material Composition Measurement Standards

The international standards for powder composition measurement are shown in Table 3.

Table 3: International standards, specification and methods in material characterization of powders

NAME / TEST	ASTM	M PIF
Chemical analysis of stainless, heat-resisting, maraging, and other similar chromium-nickel-iron alloys	**ASTM E353-14**	
Determination of C, S, N and O in steel, iron, nickel, and cobalt alloys by various combustion and fusion techniques	**ASTM E1019-11**	
Analysis of low-alloy and stainless steels, cast irons and Nickel-Base alloys by wavelength dispersive XRF	**ASTM E322-12 ASTM E572-13 ASTM E1085-16 ASTM E2465-13**	
Guide for Minimizing Unwanted Electron Beam Effects in AES	**ASTM E983**	
Analysis of nickel alloys by ICP-AES	**ASTM E2594-09 ASTM E2823-17**	
Analysis of titanium alloys by XRF	**ASTM E539-11**	
Analysis of Titanium and Titanium Alloys by ICP-AES	**ASTM E2371-13**	
Determination of O and N in Titanium and Titanium Alloys by Inert Gas Fusion	**ASTM E1409-13**	
Chemical Analysis of Aluminium and Aluminium-Base Alloys	**ASTM E34-11e1**	
Guide for Depth Profiling in Auger Electron Spectroscopy	**ASTM E1127**	
Method for Determination of Acid Insoluble Matter in Iron and Copper Powders		**MPIF Standard Test Method 06**
Method for sample preparation for the determination of the total carbon content of powder metallurgy (pm) materials		**MPIF Standard Test Method 66**
Guide to Charge Control and Charge Referencing Techniques in XPS	**ASTM E1523**	
Guide for Handling Specimens Prior to Surface Analysis	**ASTM E1829**	

4.5 Thermal, Mechanical and Humidity Properties

4.5.1 Methods

Thermal Conductivity

Thermal conductivity of powder feedstock is a key parameter affecting the consolidation characteristics of powder particles in PBF which are very important in determining the L-PBF produced part quality (Cooke and Slotwinski, 2012).

There are a number of techniques used for the measurement of thermal conductivity of powder samples and they are generally classified into two groups: steady state and transient (Sih and Barlow, 1992). The steady state approaches include (i) plate (or disk) method, (ii) cylindrical method, and (iii) spherical and ellipsoidal methods. The Guarded-Hot-Plate Method, which is described in ASTM C177-10, is an example of the plate method. In this approach, two sample test specimens are sandwiched between a guarded-hot-plate and two isothermal cold plates. The thermal conductivities are calculated using measurement of various surface temperatures, area and thickness of the sample. In the cylindrical method a heater is located along the axis of the cylindrical sample specimen, while in the spherical and ellipsoidal method, a spherical heater is placed in the center of the spherical or ellipsoidal sample. In both the cylindrical and spherical/ellipsoidal methods, thermal conductivities are calculated using heat transfer principles and the measured temperature values at different radii (Sih and Barlow, 1992; Cooke and Slotwinski, 2012).

Examples for the transient thermal conductivity measurements include techniques such as the transient hot wire method, thermal probe method, transient hot strip method and the flash method. In the transient hotwire method, a long thin heater wire is embedded in a large powder sample specimen. The heater is turned on and the temperature at a point in the specimen is recorded as a function of time, and thermal conductivity can be calculated using heat transfer principles. The thermal probe works similar to the transient hot wire method, but the heat source is enclosed inside a probe for easy insertion into the sample. In the transient hot strip method, a thin metal strip with a known temperature coefficient of resistance, which acts as the heat source as well as a resistance thermometer, is placed within the sample specimen. Resistance as well as the output voltage of the strip varies as a function of temperature. When a constant current is applied to the strip, the temperature of the strip and the surrounding material increases. The thermal conductivity of the surrounding material is calculated by monitoring the output voltage of the strip. (Gustafsson, *et al.*, 1979; Sih and Barlow, 1992; Cooke and Slotwinski, 2012). In another transient approach called the flash method, a high intensity light pulse is focused onto the surface of the powder specimen (powder bed) and the temperatures on the sample surfaces are determined, from which the heat capacity can be calculated. The thermal conductivity of the powder sample can be calculated by multiplying the heat capacity, thermal diffusivity, and the density (Parker *et al.*, 1961; ASTM E1461-13, 2013).

Nano-indentation

Nano-indentation is a technique in which a diamond tip of precise geometry is pressed into a sample surface under a controlled known constant or varying load or force. Based on the deformation of the sample, the modulus and hardness of the surface can be determined. For powder, this experiment is very challenging, in terms of sample preparation and addressing the powder particle with the indenter tip. Care must be taken to use the correct type of material in which to embed the powder particles, it should not deform itself under the applied load. Particle concentration must be such that powder particles are present at the surface, but are not densely packed such that the packing of the particles would affect the reaction of the powder particle to the applied indenter load.

Porosity

Porosity within the powder particle is an important characteristic which can negatively affect the final produced part properties. Porosity within the particles can lead to porosity within the final L-

PFB produced part. A high level of porosity can lead to poor melting, gas entrainment and outgassing during the L-PBF process. Porosity can also lead to lower part density, cracks and lower part strength. Powder porosity can be measured using micro-Computed Tomography (µCT), however this can be a relatively time consuming and costly technique, with significant expertise required for its application and significant data post processing required. It does however have the capability of providing porosity data for a large number of particles. µCT hardware and software has also progressed significantly in recent times providing higher accuracy measurements in shorter periods (Du Plessis *et al.*, 2018). A simpler and faster approach is pycnometry, where a powder sample is placed in a sample holder, and an inert gas is introduced into the sample, filling the voids and surface pores. Based on the volume of gas introduced, a measure of porosity can be obtained. It can be used for micropore and mesopore analysis. However, fully closed pores within the part will not be measured by this approach, and the reader is directed to the NDT chapter for more details on µCT based techniques. The ASTM standard for pycnometry for skeletal density of metal powders is codified in ASTM B923-20 (ASTM B923-20, 2020). Specific surface area, related to porosity, can be measured using ASTM standard B922, (ASTM B922-20, 2020).

Humidity

Humidity in a powder sample can play a very important role in safety, stability, reliability, and fluidity of the powder (Matthes *et al.*, 2020). One method used to quantitatively measure the moisture content utilizes the Relequa MP-1000 moisture analyzer (http://www.relequa.com/). This instrument uses a sealed chamber which contains a small amount of powder to calculate the amount of moisture present in the powder sample. The specific starting relative humidity (RH) % is set as a baseline, and the amount of moisture lost per unit time is then calculated. In practice, a number of different starting %RH values are chosen, one for each new aliquot of powder. The correct %RH value to begin with is the one where the final Water Vapor Equilibrium Point (WVEP) value is the same as the starting %RH. A second approach is to choose the same starting %RH for every sample and material and compare the moisture loss under similar conditions.

Phase transition temperature and type

Thermogravimetric Analysis (TGA) is used to determine the phase change temperatures and also determine the composition of the solid material. It is detailed in the ASTM standard (ASTM E1131-20, 2020).

4.5.2 Application of Thermal, Mechanical and Humidity Measurements in Additive Manufacturing

Due to the presence of weak conduction through the gas voids between the powder particles, thermal conductivity of powder will be significantly lower than that of the bulk material. The packing density of the powder can influence the contact area between the particles and the thermal conduction path. Thus, the thermal conductivity of the powder samples are found to increase with packing density (Field *et al.*, 2020). Similarly, Alkahari *et al.* (Alkahari *et al.*, 2012) found that the thermal conductivity of 316L metal powder increases with increase in the bulk density and particle diameter, while the thermal conductivity of the consolidated metal decreases with increased porosity. In general, thermal properties of the powder, especially their reduced effective thermal conductivity compared to the bulk, influence the melt pool characteristics and hence the mechanical properties of the parts produced using PBF additive manufacturing.

Understanding the mechanical properties of metal powders on the other hand allows for a greater appreciation of powder flow, packing, and for more accurate predictions of the interactions between powders and the re-coater blade, roller or other powder distribution mechanism employed in the L-PBF tool. Density and hardness are also important when mixtures of powders are used, such as when a metal powder is mixed with a reinforcing agent such as silicon carbide or tungsten carbide. Hardness and mechanical properties obtained via indentation methods such as nano-indentation may also inform the operator about density and porosity, and the presence of defects with the powder particle.

One potential way of integrating the moisture testing into the additive manufacturing workflow is shown in Figure 5 below. In this concept, the powder would be tested upon receipt from supplier. This powder sample is then stored (Sample A). After each build or at monthly/weekly intervals, the virgin powder is tested and compared with Sample A (red arrows). After each build, the used and sieved powder is tested and compared with the original Sample A (yellow arrows).

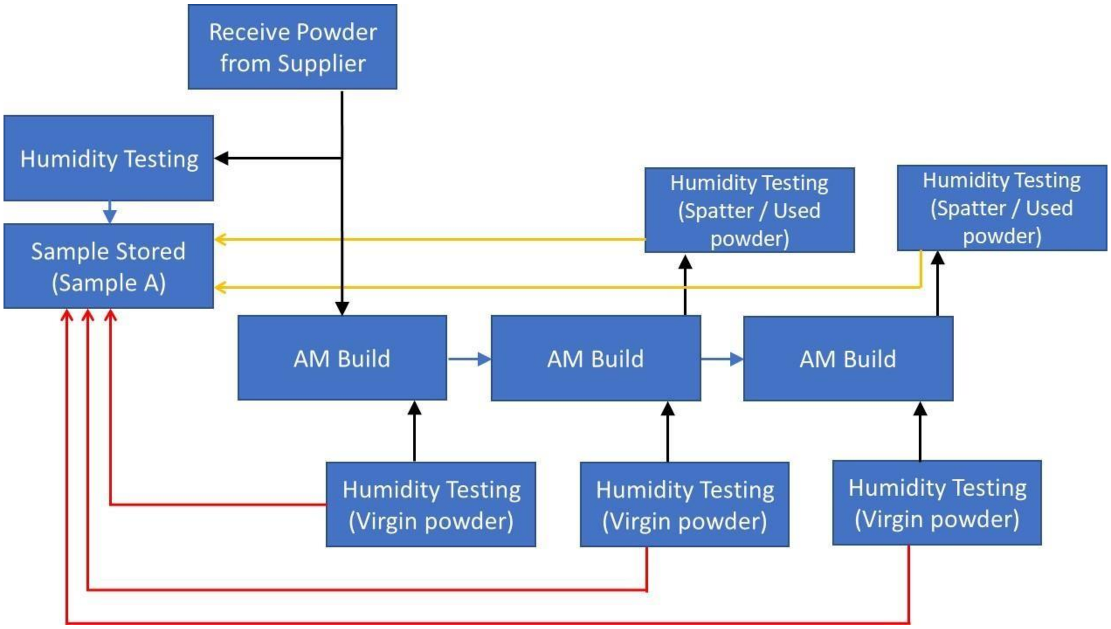


Figure 5: Schematic illustration of the integration of humidity testing into AM quality control workflow.

4.5.3 Powder Thermal Conductivity and Porosity Assessment Standards

The international standards for powder conductivity and porosity assessment are shown in Table 4.

Table 4: International standards, specification and methods in powder thermal conductivity and porosity.

NAME / TEST	**ASTM**
Standard Test Method for Steady-State Heat Flux Measurements and ThermalTransmission Properties by means of the Guarded Hot Plate Apparatus	ASTM C177-10
Standard Test Method for Thermal Diffusivity by the Flash Method.	ASTM E1461-13
Standard Test Method for Metal Powder Specific Surface Area by Physical Adsorption	ASTM B922-20
Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry	ASTM B923-20
Standard Test Method for Compositional Analysis by Thermogravimetry	ASTM E1131-20

4.6 Powder Life Cycle and Sustainability Analysis

In order to ensure the economic feasibility of powder-based additive manufacturing, powder is almost always used multiple times in L-PBF processes. Sartin *et al.*, 2017 found that only 6.7% of powder fed into the L-PBF process was consumed; the remaining 93.3% was recovered and could be reused in future builds. A case study by LPW Technology Ltd (Rushton, 2019) showed that a 92% reduction in material costs could be achieved if powder was reused up to 15 times versus just one use as virgin powder. However, some or all of the reclaimed powder will have been subjectedto high-temperature melt pools, spatter particles, partial melting of adjacent particles, sub-optimal atmospheric environments and human handling (introducing oxygen and humidity contamination and contamination from powders in the build chamber from previous builds). These factors can contribute to powder degradation through debris contamination, changes in particle morphology/size and changes in the chemical composition of the powder.

As can been seen from the preceding sections 4.0 to 4.3, a “powder” is a generic term that encapsulates a wide range of properties. If even small changes are made to just one of these properties, a different powder is formed. This can be seen in Figure 6; the Particle Size Distribution

(PSD) of powders can vary greatly within a relatively small size range, forming a potentially infinite number of powders. Two powders with different PSDs are unlikely to produce the exact same component properties from the L-PBF process. However, other properties also make up anyone powder, such as particle morphology, chemical composition and flowability. If many of these properties change simultaneously, as is typical when powder is recycled (Powell *et al.*, 2020), it can become very difficult to determine whether a powder is suitable for use in AM. Controlling powder quality and being aware of powder degradation is therefore paramount in L-PBF.

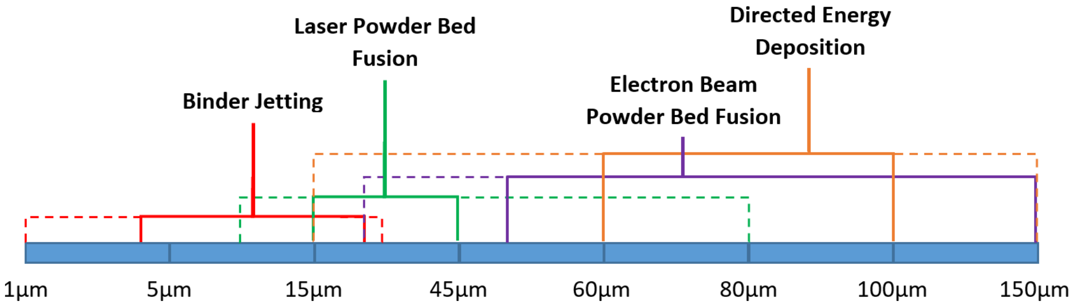


Figure 6 Particle sizes used by the different metal powder-based additive manufacturing techniques. Thick lines indicate the desirable particle sizes for each process, whilst dashed lines indicate usable but less acceptable particle sizes. (Powell, 2020).

The recycling process should be strictly controlled to minimize the risk of powder degradation, attempting to keep the properties as similar to the virgin powder as possible. Whilst standards exist to characterize metal powders (ASTM F3049-14, 2014), there is no standard methodology for powder reclamation or powder recycling. This leads to a wide variation in the rate of powder degradation when recycling metal powders across the industry. Equipment such as the AMPro Sieve Station by Russell Finex (Russell Finex, n.d.) offers fully automated and closed-loop recycling of powders under inert atmospheric conditions. After automatically extracting powder from the build chamber, it sieves out unsuitable particles and returns the remaining powder back into the feed hopper. This reduces degradation by eliminating manual handling of powders and minimizing exposure to the atmosphere. However, many additive manufacturing users still manually remove loose powder from the completed build (either by hand or the use of a vacuum cleaner) and sieve the powder in a separate machine, reintroducing the sieved powder to the material feed hopper. This results in increased exposure to the atmosphere and the potential inclusion of contaminants such as dust or other powder particles, accelerating powder degradation.

4.6.1 Powder Reuse Methods

An important question is raised: "What can be done with the remaining End-of-Life (EoL) powder?" Powell *et al.* (2020) investigated methods to improve the resource efficiency of powders in additive manufacturing, identifying several potential solutions. The suggested approach to achieving this was to both reduce the need for new virgin powder to be created and increase the longevity of metal powders. Several potential solutions were investigated, with a technique called "plasma spheroidization" showing the most promise.

A rudimentary understanding of powder creation is necessary to appreciate potential powder upcycling methods. Powder is typically generated through "atomization" (shown schematically in Figure 7). Metal is melted, allowing it to flow through a nozzle. High-pressure jets of fluid are aimed at the end of this nozzle, rapidly dispersing the metal upon contact and creating tens of thousands of small particles. The fluid chosen greatly influences the powder properties. For example, using a jet of water rapidly cools the metal, resulting in less spherical particles, whilst using a jet of inert gas creates highly spherical particles and minimizes the risk of changes in the chemical composition (Dawes *et al.*, 2015). The metal flow rate and jet velocity can be altered to tailor the size of particles created, allowing many different specifications of powder to be produced.

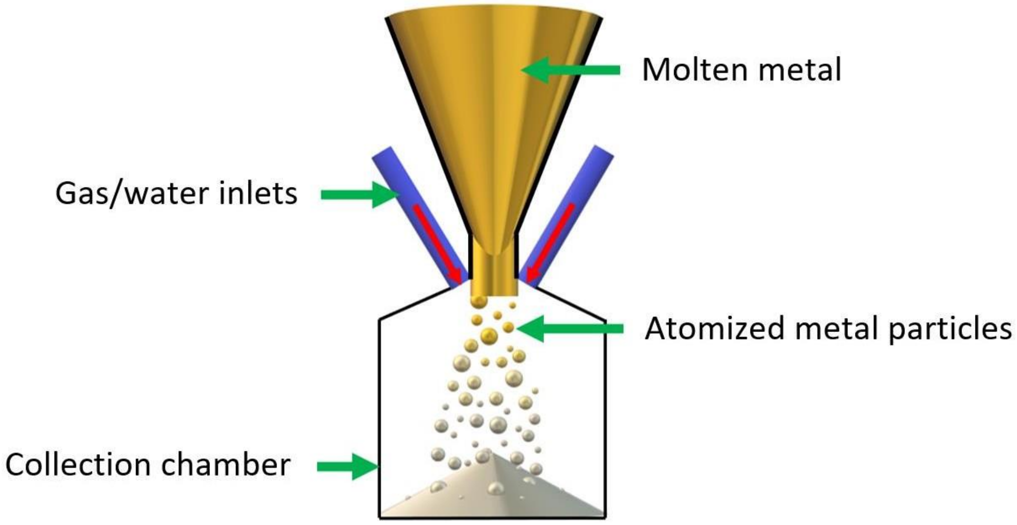


Figure 7: Schematic showing the material and processing elements within the atomization process.

Plasma spheroidization uses a similar principle to gas and water atomization, however, rather than atomizing molten metal, the feedstock material is heated rapidly inside the nozzle. This has two distinct advantages over gas or water atomization. The first is that the metal spends less time at high temperatures, reducing the likelihood of chemical reactions with the surrounding atmosphere (even an "inert" atmosphere contains small quantities of oxygen and other reagents). The second advantage is that the feedstock material can be small in volume, such as a wire or powder, which will melt faster than an ingot, bar or similar The latter advantage makes plasma spheroidization a viable technique to upcycle poor-quality powders that have been reused numerous times and are no longer suitable for use in L-PBF. This principle has been demonstrated by Kelkar and co-workers, (Kelkar *et al.,* 2019), where low-quality oversized non-spherical water-atomized powder particles were converted to smaller high-qualityspherical powder particles through plasma spheroidization. Perhaps more impressive was the ability of the plasma spheroidizer to alter the chemical composition of the particles through introducing reducing agents to the gas inlets, reducing the oxygen content by 97%. Oxygen is considered to be a likely contributor to the formation of pores (Pal *et al.,* 2020), so coupling the improvement in chemical composition with the superior morphology suggests that plasma spheroidization could sufficiently upcycle EoL powders. This would both reduce the waste output in the form of EoL powders from L-PBF whilst reducing the necessity to create virgin powders from raw materials, greatly improving the sustainability of L-PBF. Powell *et al.*. (Powell, 2020) conservatively estimated that powder production through plasma spheroidization could reduce energy consumption by 18.3%, if not more, when compared with gas atomization to create high-quality powders.

An alternative and simpler (albeit less effective) solution to improve powder longevity is to blend recycled powders with virgin powders. Whilst this is common practice within industry and can be empirically demonstrated to work, there is little research into this technique. Vock *et al.* (Vock *et al.* 2018) found that mixing equal quantities of virgin and recycled powders resulted in no changes being observed in the powder properties, suggesting that the components produced from blended powder may also be unchanged. Jacob and co-workers (Jacob *et al.* 2017) used another blending technique that introduced virgin powder at regular intervals to a number of consecutive builds, finding that both the powder properties and properties of the produced components remained relatively constant, further demonstrating the viability of blended powders. It should be noted that blending powders does not remove the contamination that may result from the existence of spatter particles. Whilst it does dilute their concentration in the powder mixture, a single heavily oxidized spatter particle could still cause porosity in a fabricated component.

4.6.2 Effect of Powder Recycling on Additive Manufacturing

Research has been undertaken into the impact of recycling powders in various powder-based additive manufacturing techniques. This is well summarized by (Powell *et al.*, 2020) and (Vock *et al.,* 2019), offering an overview of the trends that are witnessed as powder is repeatedly reused. Different powder recycling techniques were applied, emphasizing the lack of standardization in powder recycling in additive manufacturing. Changes observed when comparing the properties of virgin and recycled powder tended to be small, indicating that powder can be recycled effectively. However, the changes were also found to be gradual; the magnitude of these changes increased as the powder was continuously reused/processed. As powder properties can have a considerable influence on component properties in L-PBF, it has been found that recycled powders often influence the component properties such as surface roughness, strength/hardness, chemical composition and porosity (Renderos *et al.* 2016; Tang *et al.,* 2015, and Seyda *et al.* 2012).

One of the biggest problems found in recycled powders was “spatter”. Spatter particles can be seen as “sparks” flying off in the build chamber of the AM machine. An example of a spatter particle can be seen in Figure 8. These particles have a higher oxygen content than the virgin powder (Liu *et al.*, 2015) and can vary in morphology and oxide coverage (Gasper *et al.*, 2018; Obeidi *et al.*, 2020). After sparking off, the spatter particles often fall back into the build chamber powder bed, either becoming incorporated into the build or are extracted through the recycling process. LPW Technology Ltd (2018) state that a “significant amount” of contaminated spatter particles are small enough to pass through sieves in the recycling process, resulting in them being present in future builds, a clear explanation of how this can negatively impact future builds through the creation of pores, in turn creating regions of weakness in fabricated components, is offered by Pal *et al.* (202

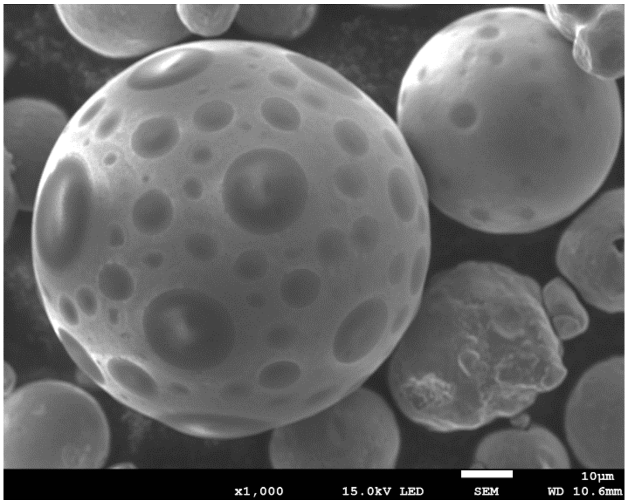


Figure 8: A partially oxidised spatter particle. The darker spots are heavily oxidised.

Spatter is not currently measured in ASTM F3049-14 as it is not present in virgin powders. It would be incredibly difficult to measure the presence of spatter particles quantitatively. Their effect is unlikely to be detected through standard powder quality measurements such as PSD or bulk chemical composition, as oversized spatter particles will be sieved out and the quantity of spatter particles will be small in comparison to normal particles. However, it is reasonable to assume that spatter forms at a constant rate per minute of “laser on” time (provided build parameters are unchanged). Therefore, as powder is reused repeatedly over time, the quantity of spatter particles present in a powder will inevitably increase. This leads to an increased probability of spatter particles being incorporated into components and potentially forming pores, as demonstrated by Pal *et al.* (2020).

As the powder degrades gradually, it is difficult to confidently determine an EoL point where it is no longer suitable for use in L-PBF AM processes. Due to the numerous L-PBF systems on the market, there is no minimum standard for powder quality; it is up to both the machine user and manufacturer to decide what powder is suitable for use, and more importantly, when powder is no longer suitable. Some materials used in high-end industries are highly reactive, notably Al- and Ti-based materials, resulting in them being recycled fewer times before falling out of specification (Daraban *et al.*, 2019). In industries using less reactive materials, the powder is likely to have a considerably longer lifespan. Either way, there is eventually going to be a quantity of powder that is no longer useful as a material feedstock for additive manufacturing.

4.7 Powder Safety

It would be remiss to discuss the analysis of powder for L-PBF processing without addressing safety and the potential risks and hazards associated with the powder feedstock and its use within the L-PBF chamber. This is not intended to be an exhaustive discussion, and the reader is encouraged to seek out the Safety Data Sheet for the particular powder feedstock they are using, including from their preferred supplier for the most up to date information.

Briefly however, the risks include those arising from the chemical nature of the metals or alloys, their reaction with water (aluminium, titanium), the laser (hence the necessity of an inert environment in the L-PBF process chamber), itself (is it self-igniting), and the presence of static charges (operator, tools). Many of the most commonly used alloys such as stainless steel and

Inconels (nickel super alloys) contain carcinogenic or suspected carcinogenic materials. Therefore, when working with such materials, it is important to plan how much of the material is to be used, how to contain this material (for example by using a glove box), transfer steps, how to avoid the formation of a dust cloud, choosing the correct personal and respiratory protective equipment (PPE and RPE) and using engineering controls (O_2 sensors, Class D powder fire extinguisher, and anti-static mats) to limit the risks associated with working with these materials. Appropriate risk assessments need to be completed for an L-PBF process tool prior to installation and commissioning.

As mentioned above, certain powders will react violently with water, therefore, iso-propyl alcohol wipes (70 or 100%) should be used to clean up powder spills and for general housekeeping and cleaning.

One important aspect of working with metal powders is the concept of ATEX compliance. For powder usage, there are three Zone (20, 21 and 22) classifications. These are defined as below:

- Zone 20 – A place in which an explosive atmosphere in the form of a cloud of combustible dust in air is present continuously, or for long periods or frequently.
- Zone 21 – A place in which an explosive atmosphere in the form of a cloud of combustible dust in air is likely to occur in normal operation occasionally.
- Zone 22 – A place in which an explosive atmosphere in the form of a cloud of combustible dust in air is not likely to occur in normal operation but, if it does occur, will persist for a short period only.

This allows for an understanding of the risks involved in each element of the process, for example, in the absence of the inert atmosphere, the L-PBF process chamber would be considered Zone 20, the vacuum cleaner for removal of powder from the process chamber needs to be ATEX rated, as it is considered a Zone 22. It is important to understand the air flow of the laboratory in which powder is manipulated, to avoid strong air currents in the area where powder is open, for example in the process chamber during loading. A dust explosion requires five contributing factors: turbulence, confinement, heat, material or fuel, and oxygen. Hence it is described as the explosion pentagon. If any of these factors are removed, the explosion is not possible, or the risk is greatly diminished. When planning an installation, it is important to consider the room volume, the number of air changes per hour, the flow of inert gas required for the safe operation of the L-PBF tool and the maximum inert gas outflow rate in the event of a leak.

Metallic powders should always be stored in cabinets which are rated as fire resistant for 90mins (EN 14470-1). Powder should be stored in a laboratory with low relative humidity, and should be handled/poured in an isolation cabinet or glove box. An inert atmosphere should be considered but is not essential based on our experience in DCU. Powder that has already been processed via L-PBF should be sieved prior to being used again. It can be mixed with virgin powder as needed. Experience with specific materials suppliers and batches is required in order to develop a robust workflow for use and recycling of powder in L-PBF, as discussed above.

Health and Safety Standards

Some standards related to powder combustibility include ASTM E2019-03, 2013; ASTM E1515-14, 2014; and ASTM E1226-19, 2019. The reader is also directed to the local regulations of the country they are working as these can be different between regions.

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