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POWDERS FOR ADDITIVE MANUFACTURING PROCESSES: CHARACTERIZATION TECHNIQUES AND EFFECTS ON PART PROPERTIES

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Abstract

Powder-bed based Additive Manufacturing is a class of Additive Manufacturing (AM) processes that bond successive layers of powder by laser melting to facilitate the creation of parts with complex geometries. As AM technology transitions from the fabrication of prototypes to end-use parts, the understanding of the powder properties needed to reliably produce parts of acceptable quality becomes critical. Consequently, this has led to the use of powder characterization techniques such as scanning electron microscopy (SEM), laser light diffraction, x-ray photoelectron spectroscopy (XPS), and differential thermal analysis (DTA) to both qualitatively and quantitatively study the effect of powder characteristics on part properties. Utilization of these powder characterization methods to study particle size and morphology, chemical composition, and microstructure of powder has resulted in significant strides being made towards the optimization of powder properties for powder-bed based AM processes. This paper reviews methods commonly used in characterizing metallic AM powders, and the effects of powder characteristics on the part properties in these AM processes.

1 Introduction

Additive manufacturing (AM) is a layer-based approach used for the creation of parts directly from Computer-Aided Design (CAD) files. Rather than utilize subtractive methods to remove material from a larger piece, parts are built by bonding successive layers of material typically through heat input, a binder, or by chemical means [1–3]. This new approach facilitates the production of components with high geometrical complexity that would otherwise be impossible to create through conventional manufacturing processes [4,5]. Although many different AM process variations exist [6–10], it is important to realize that all can be categorized according to the state of the starting material: liquid, solid, and powder [3]. Yet, the existing literature indicates that many of the successful attempts to produce functional AM components stem from using powder as the raw input material [11].

This is due to the advantages that powdered material has when compared to liquid and solid starting materials. One of the major benefits is its ability to serve as a support structure [1], as is the case in the Selective Laser Sintering/Selective Laser Melting (SLS/SLM) and Electron Beam Melting (EBM) processes. Other reasons stem from its flexibility in powder mixing to produce parts of various chemical composition [12], and the potential for direct recycling to yield little to no waste material [13].

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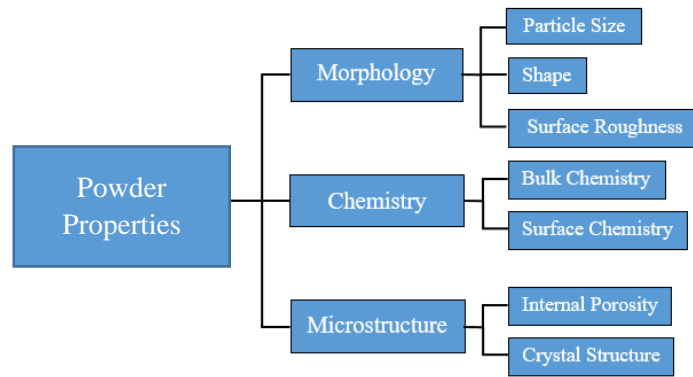


Figure 1: Classification of Powder Properties

Due to these reasons as well as the overwhelming desire to produce functional components, a great deal of focus has been centered on the use of metallic powders in the SLS/SLM, EBM, and Direct Metal Deposition (DMD) processes. Among others, Ti-6Al-4V [17–19], 316L stainless steel [20–22], 17-4 PH stainless steel [23–25], IN625 [26–28], and IN718 [29–31] are some of the most commonly studied materials. The use of Ti-6Al-4V is prominent in aerospace and medical applications where the combination of its strength and lightweight properties prove extremely attractive for components such as gas turbines and airfoils [32] as well as hip joints and bone plates [33]. IN718 is also used in aerospace applications in addition to pumps and tooling where high temperatures and fatigue are often encountered [29]. Due to its versatility, 316L stainless steel is used in a large variety of areas including aircraft, automotive, and medical industries [20].

Although the use of powder in AM is quite common, optimization of powder properties (such as those shown Figure 1) to yield desired performance characteristics has posed a serious challenge to researchers. This complication not only stems from the dependence of powder behavior on both the bulk solid properties and their interaction with the AM system during the build process [31], but also the underlying physics of particle to particle consolidation [2]. The impact of particle size, shape, and surface roughness in powder-bed fusion processes has been observed in several studies [11,32–36] to conclude that morphological properties of powder influence the density of powder after deposition onto the build platform, sintering kinetics between particles, and the surface roughness and density of manufactured parts. Thus, it is critical that powder properties be studied and controlled to ensure reliability and repeatability of the components that are produced. Although many studies have been conducted, a lack of understanding regarding the effects of initial particle characteristics on the properties of built parts still exists. As the AM industry matures and the desire to produce functional AM components increases, the need to close this knowledge gap becomes clear [2,37–39].

Accomplishing this task warrants the use of powder characterization techniques. Table 1 gives a listing of commonly used powder characterization methods in AM, and organizes each into three categories: particle morphology, particle chemistry, and particle microstructure. When selecting and/or optimizing a powder for any given process, it is imperative that each is considered. In AM, this approach is rarely ever taken. Instead, a powder that is deemed suitable

in terms of size and chemistry is chosen whereby the process parameters are then optimized to yield parts of acceptable quality. However, it must be realized that if AM processes are ever to rival traditional manufacturing methods, tuning of powder properties must also be regarded as necessary. This can only be accomplished through the use of powder characterization methods to correlate powder characteristics to material properties. In this paper, an overview of the powder characterization efforts in AM of metallic components aimed at understanding the effect of material characteristics on part properties is given, as well as pertinent research needs regarding future characterization of powders

2 Powder Morphology

Particle morphology refers to the size, shape, and surface roughness of particles. In AM, it is well known that all of these characteristics play a significant role in powder performance including flowability and packing efficiency, and thus impact the final component properties. The focus of this section is to discuss the most commonly used powder characterization techniques used in the morphological characterization of powders in AM, and to highlight the research that has been performed which utilizes these methods to quantify and predict powder performance characteristics in powder-bed fusion processes.

2.1 Sieve Analysis

Size determination by sieving is one of the oldest methods to measure particle size, albeit it is one of the most widely used due to its simplicity and low cost. In practice, a stack of sieves with decreasing mesh size from top to bottom is shook or mechanically vibrated. Depending on the size of the particles, each mesh will retain powder comprised of particles larger than the mesh size. In order to extract particle size distribution data, the mass of powder on each mesh is measured and reported corresponding to the size range, or bin size, in which it was found [40].

Although this method is simple requiring minimal sample preparation, it is important to note that there are inherent drawbacks with the overall approach. First and foremost, the resolution of the particle size distribution curves generated is determined by the differences between adjacent mesh sizes. Therefore, if it is desired to increase the accuracy of particle size distribution data obtained through sieving, the bin size must be decreased. This will not only increase the cost of the measurement process, but also the time necessary to perform the size analysis. Exacerbation of this problem also occurs when particle size ranges are below 50 μm where meshes become considerably more expensive and the propensity for particles to agglomerate drastically increases yielding inaccurate results [41]. The problem of agglomeration can cause blinding of mesh apertures where particles smaller than a given mesh size are no longer able to pass through due to clogging of the mesh. If this problem persists unbeknownst to

Table 1: Common Powder Characterization Techniques used in Additive Manufacturing

Summary of Powder Characterization Techniques					
	Characterization Technique	Resolution	Particle Size Range	Advantages	Disadvantages
Particle Morphology	Sieve Analysis	Bin Size: Dependent on size separation of sieves	20 μm - 125 mm	Applicable to broad size range, Minimal sample preparation, Low cost	Does not aid in shape analysis, Longer measurement times, Prone to blinding, Particles with large aspect ratios can produce large uncertainties
	Microscopy	$d = \frac{0.61\lambda}{NA}$ <i>d</i> – resolution distance λ – light source wavelength <i>NA</i> – numerical aperture	Determined by system resolution	Allows qualitative and quantitative observation of particle shape, Great flexibility for particle size and shape analysis	Cost increases with decreasing particle size, SEM and TEM sample preparation require more effort than optical microscopy
	Laser Diffraction	Dependent on instrument design and data analysis algorithm	0.04 μm - 8000 μm	Short analysis time, Does not require skilled labor, Highly repeatable results	Error can result if particle shape differs from spherical, Measurements dependent on instrument design, Detection of agglomerates is difficult
Particle Chemistry	X-Ray Photoelectron Spectroscopy	1 μm	Any	Determination of overall composition as well as chemical bonding	Sputtering through spherical particles can be difficult
	Auger Electron Spectroscopy	0.5 - 2 μm	Any	Elemental analysis at a lower penetration depth compared to XPS	Destructive
	Energy Dispersive Spectroscopy	2 μm	Any conductive particles	Very fast, Allows for point scans, line scans, and mapping elemental analysis	Semi-quantitative
	Inductively Coupled Plasma - Atomic Emission Spectrometry	20 - 50 μm	Particles must be completely dissolved	Quantification of multiple elements at a wide range of concentrations	Destructive and Expensive; Unable to detect C, N, O, F, and H
	Inert Gas Fusion	Dependent on resolution of cells used to analyze H, N, and O	Any	Allows for the detection of O, N, and H	Destructive
Particle Microstructure	X-Ray Diffraction	Can detect phases that are present down to 5%	Best when powder particles are < 150 μm	Able to determine crystal structure of phases;	Only able to measure phases that are present > 5%
	Thermal Analysis	1- 2°C	Any	Allows insight to the endo- and exothermic transitions associated with the sample	Destructive

the operator, a large quantity of powder can be collected on a single mesh where its weight can potentially increase the size of the mesh openings thereby causing erroneous results [42].

Due to these limitations coupled with the common use of fine powder in powder-bed fusion processes, sieve analysis is not as commonly used as other size determination methods, such as microscopy and laser diffraction. However, it is more likely to be utilized in EBM and SLS with typical particle size ranges of 45 μm - 106 μm and 20 μm - 150 μm , respectively, rather than SLM (15 μm – 80 μm) since the particle size ranges are typically more coarse.

2.2 Microscopy

Microscopy is a powerful tool that is commonly used in AM due to its capability to quantitatively measure particle size. This is accomplished by the use of microscopy-based instruments such as optical light microscopes, scanning electron microscopes, and transmission electron microscopes [42] to directly observe particles. Due to this very reason, this technique is often treated as a reference method to other size distribution techniques when studying powder morphology [43].

Typically, the images obtained are post-processed to calculate equivalent diameters of particles based upon the two-dimensional projected area of the particles in the collected micrographs (Figure 2). Although a two-dimensional approach to calculate particle size may not be the most accurate, it is widely accepted in the AM industry because it provides the most versatile approach in particle analysis. This is due to the ability to study powder particles not only in terms of size, but shape and surface roughness [44] both qualitatively and quantitatively. Due to the sizes of particles used and the reliability of the method, analyzing particles from SEM micrographs is a widely used method in AM to determine particle size distributions, although post-processing of the images can be very time consuming. SEMs equipped with automated feature analysis have offered the advantage of reducing the time for analyzing these micrographs. There is, however, a lack of agreement on the minimum number of particles needed in order for these results to be acceptable [43].

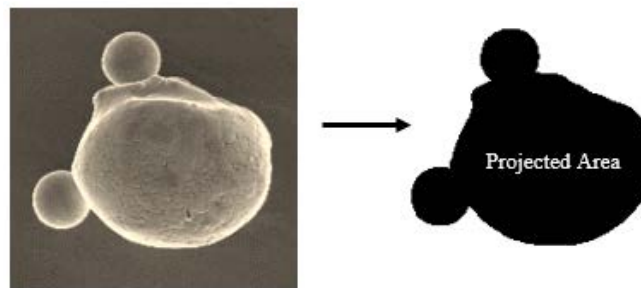


Figure 2: Method of projected area for particle size determination by microscopy. The micrographs containing the particles under observation are converted to binary where the projected area for each particle is measured by the amount of black pixels present.

2.3 Laser Light Diffraction

Another widely used particle size determination technique is laser diffraction, which is a class of non-image based instruments that analyze the diffraction pattern of laser light when shining through a dispersed medium of particles (Figure 3). In order to process the diffraction pattern, it is assumed that the scattering pattern on the detectors is comprised of the superposition of scattering patterns formed by each particle in the sample. Deconvolution of the obtained data is then performed by various algorithms including Mie Theory, Fraunhofer approximation, and Rayleigh scattering which are all based on the solution to Maxwell's equations for electromagnetic radiation for a perfect sphere. The algorithm utilized depends on the relative size of the particles in the sample with respect to the wavelength of the incident light [42]. Although this method provides the ability to measure a large number of particles in a relatively short amount of time to obtain a statistically significant particle count, it's assumption of a perfect sphere can be misleading in the case of highly irregular particles or those with high aspect ratios. However, this is rarely a problem in powder bed fusion processes since highly spherical particles are the most commonly used.

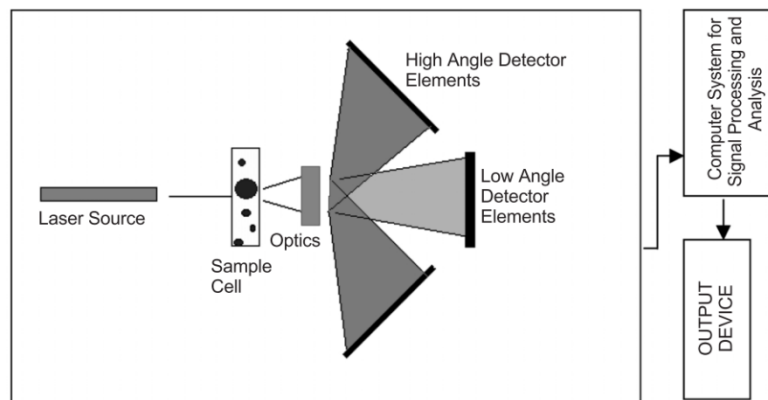


Figure 3: Typical Schematic of a Laser Diffraction [42]

2.4 Influence of Particle Size and Size Distribution on Part Properties

In any powder-based manufacturing process, the determination of particle size and size distribution is essential since these powder characteristics have a large effect on the properties of the parts built including the mechanical strength, porosity, and surface finish. While the chemistry and microstructure of powder can influence part quality as well, powder morphology is typically the most studied in AM when initial input material characteristics are correlated to the manufactured component properties.

The selection of particle size for any given powder-based manufacturing process stems from the desired properties of the components that are to be produced. In powder-bed fusion of metals, some of the most sought after part properties are a near one-hundred percent relative density and a smooth surface finish, both of which are directly dependent upon the size and size distribution of the particles used. In turn, the particle size determines the minimum layer

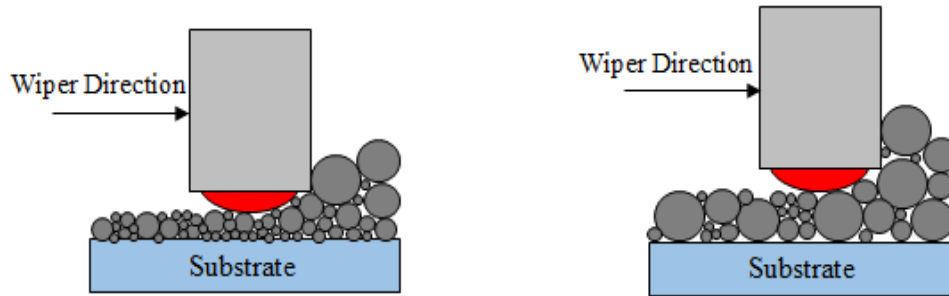


Figure 4: Image reconstructed from Abd-Elghany et al. [47], (a) Layer thickness of $30\ \mu\text{m}$ corresponding to a high density of powder particles and (b) Layer thickness of $70\ \mu\text{m}$ corresponding to a low density of powder particles

thickness and the minimal size of the part features that can be built [45]. If the layer thickness is greater than the maximum particle size, all of the powder particles regardless of size will be deposited into the build volume. Conversely, a layer thickness that is smaller than the maximum particle size leads to a preferential deposition of a finer distribution of particles. Therefore, the desired layer thickness serves as a limit to the maximum particle size that should be specified within a given powder [46]. Abd-Elghany et al. [47] describes this interaction between the wiper and powder particles and ultimately its effect on the solid density of 304L stainless steel parts produced by SLM. The results showed that an increasing layer thickness leads to a decrease in the density of manufactured parts due to a reduction in density of each powder layer as illustrated in Figure 4. It was also found that an increased layer thickness led to rougher surfaces on parts due in part to the larger particles that were located in the powder bed and were unable to be fully melted.

In order to combat the issues of low density and high surface roughness, fine powders within the size range of $15\ \mu\text{m}$ to $150\ \mu\text{m}$ are often employed. In addition to there being a limit to the maximum particle size, constraints should be imposed on the minimum particle size as well. Although a decreasing particle size can have advantages in regards to increased particle packing [48,49] and reduced surface roughness [50,51], one of the major drawbacks of fine powders is its propensity to agglomerate [11,52,53]. This can prove to be detrimental in powder-bed fusion processes since agglomerates can impede flow behavior [54,55] of powder by increasing interparticle friction during the recoating process. Both a decreased density and inhomogeneity in each of the layers can therefore result where more material may be deposited in one region than others. Such an effect can cause balling in regions where there is locally more powder leading to an increased likelihood of undesired porosity [56–59]. Since agglomerates can assume irregular shapes, the laser or electron beam interacts differently when compared to spherical particles. In fact, it was demonstrated by Olakanmi [33] that pore formation in the SLS of Al, Al-Mg, and Al-Si powders was increased by the appearance of irregularly shaped particles in the density of sintered parts in the SLS process as a result of the differences in laser-material interaction of irregularly shaped particles compared to spherical particles. This difference in laser-material interaction was also observed by Simchi [11] who found that the gradual decreasing of particle size does not always lead to higher densification of as-built parts, as what

is most commonly believed; in addition to agglomeration of powder, it was apparent that the reflectivity of the powder bed increases with decreasing particle size causing less absorption of laser power.

Although small particles can cause problems with flowability of material, they can also lead to higher packing efficiencies. In powder-based AM, the density of each layer should be as high as possible to reduce residual stresses in parts. One way to optimize powder bed density is through tailoring the particle size distribution of a powder so that the smaller particles can fill the voids between the larger particles. However, optimization of particle size distribution to yield superior packing characteristics [60] ex-situ of the build environment does not directly translate to a maximum powder bed density [48]. This is a direct result of the interaction between the powder and the wiper during the recoating process and is denoted as the flowability of a powder. Although powders with wide particle size distributions can increase the packing efficiency, it is known that they also have decreased flowability [61]. Therefore, when compared to conventional powder metallurgy processes, narrow particle size distributions are used in AM to decrease interparticle friction and maximize flowability while also retaining high apparent densities [62].

In the SLM process, Bochuan Liu et al. [51] observed the effect of particle size distribution on the mechanical properties of parts including the following: solid density, surface roughness, and ultimate tensile strength some of which were created from a narrow (15 – 60 μm) particle size distribution and others from a wide (1 – 60 μm) particle size distribution. It was found that parts made from the narrow size distribution have a higher ultimate tensile strength and hardness while those with the wide size distribution have higher density and less surface roughness. Similarly, A.B. Spierings et al. [34,50] found that the particle size distribution ultimately determines the density of the developed parts, and thus has a major impact on the mechanical properties. In particular, powders containing more fines correlated to smoother part

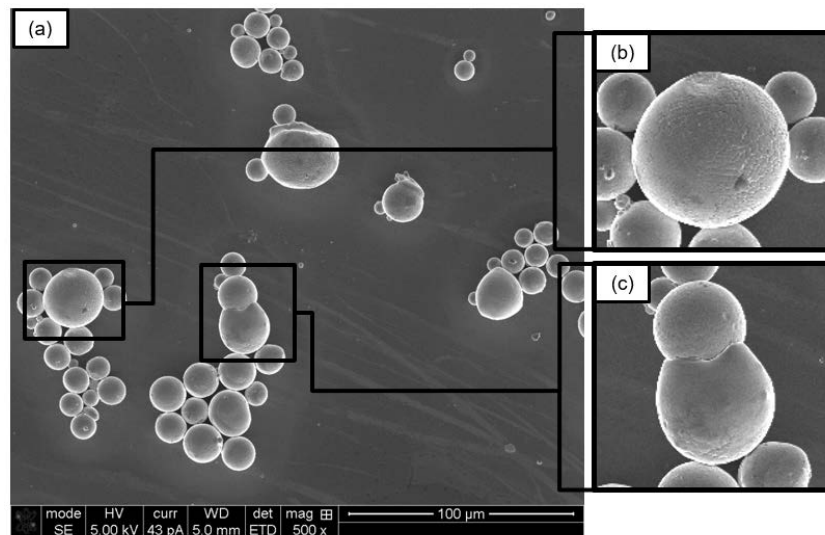


Figure 5: 304L Stainless Steel Powder, (a) Micrograph of 304L Stainless Steel Powder, (b) Spherical Particle, and (c) Oddly Shaped Particle

surfaces while those with larger particles showed higher elongations during tensile testing. Simchi [11] also studied the effect of differing particle size distributions of iron powder on the properties of selectively laser sintered parts. It was found that particle size has a profound effect on the porosity of SLS manufactured iron parts with their being an optimum mean particle size to produce parts with the highest fractional density.

Even with the information generated from the aforementioned research, specification of an exact particle size distribution for a given material is extremely difficult. Not only does the optimal size distribution of a powder depend on the machine and process parameters, but also on the method of size measurement as well. In fact, it is important to realize that measurements of particle size are highly dependent upon the measurement technique as well as the particle shape [53]. For instance, consider Figure 5 which shows an SEM image of a 304L stainless steel powder sample. Most of the particles shown are spherical (5b) with a diameter no greater than 50 μm . The size of spherical particles is rather easy to describe since only the diameter of the particle is needed. However, deviation from a spherical shape (Figure 5c) immediately complicates the measurement procedure as no one single dimension can completely describe its size. To simplify the problem, irregularly shaped particles are either 1) transformed to a sphere typically by measuring the projected area of particles through microscopy, or 2) assumed to be spherical so data obtained through laser light diffraction or gravitational sedimentation techniques can be used to calculate equivalent diameters [42]. Therefore, particle size determination is subjective thereby making it imperative that the measurement procedure is explained whenever size information is reported.

2.2 Effect of Particle Shape and Surface Roughness

The size of particles is not the only morphological factor when looking to achieve high density parts through powder-based AM; rather, it is also a combination of the particle shape and surface roughness. Describing the shape of particles both qualitatively and quantitatively can be extremely challenging, especially if the particles are highly irregular and show no resemblance to primitive shapes. Nevertheless, qualitative descriptions of powder particles have been standardized [63] and are used as a means to describe the general shape of powder particles. On the other hand, quantification of particle shape still proves to be a challenge. Many researchers approach this problem by formulating equations that yield dimensionless ratios, more commonly known as shape factors. Throughout the literature there exists numerous shape factors [64], some of which are listed in Table 2.

Although it is common knowledge that particle shape and surface roughness influences part properties, the exact effects of these initial powder characteristics on additively manufactured components are still not fully understood. To combat this issue, Strondl et al. [65] studied the effect of particle shape on the flowability of bulk powder in both the selective laser melting (SLM) and electron beam melting (EBM) processes. It was determined that a more spherical particle shape yielded less internal friction in the powder allowing it to be more easily deposited to obtain higher layer densities. By continuously recycling powder and tracking its

Table 2: Various Shape Factors used for Particle Shape Characterization

Name of Shape Factor	Equation	Reference
Circularity	$\frac{4\pi A}{P^2}$	[66]
Aspect Ratio	$\frac{l}{L}$	[67]
Elongation	$\log_2\left(\frac{a}{b}\right)$	[41]
Dispersion	$\log_2(\pi ab)$	[41]
Roundness	$\frac{r_1}{R}$	[68]
Flatness	$\frac{r_2}{R}$	[68]
Perimeter to Area Ratio	$\frac{P^2}{A}$	[69]

A – Projected area of particle

P – Perimeter of particle

l – Minor axis length perpendicular to major axis

L – Major axis length connecting two most distant point on the projection area A

a – Major axis length of Legendre ellipse of particle

b – Minor axis length of Legendre ellipse of particle perpendicular to a

r_1 – Radius of curvature of sharpest developed edge of particle

r_2 – Radius of curvature of most convex direction of particle

R – Mean radius of particle

evolving shape characteristics after reuse, it was also found that even slight changes in the aspect ratio of the particles can drastically change the flow behavior and degrade the properties of the manufactured parts.

Similarly, Sun et al. [31] used the circularity shape factor to study the evolving morphology characteristics after continual reuse of powder in the EBM process. It was found that the average circularity of the powder decreased due to the appearance of agglomerates after successive build iterations. This resulted in more interparticle friction decreasing the flowability of the recycled titanium powder. Additionally, an external powder deposition system was created to study the influence of the recycled powder on its deposition in the powder bed; virgin powder

was determined to provide the most uniform layers where the recycled powder showed powder density irregularities in the build area.

The results of the aforementioned studies both imply that powder-bed based AM processes can be very sensitive to the appearance of irregularly-shaped particles since they directly affect how the particles interact and flow together as a bulk solid. Therefore, highly spherical particles with smooth and dry surfaces [48] are desired as these are known to provide the least amount of interparticle friction equating to the best flowability characteristics [70,71]. Consequently, the circularity and aspect ratio are by far the most commonly used shape factors in powder-based AM since most of the powder utilized is spherical. Although, it is important to note that the degree of sphericity depends on the powder manufacturing process (Table 3).

Table 3: Powder Characteristics Observed from Different Manufacturing Processes [72]

Table of Powder Manufacturing Processes			
Manufacturing Process	Particle Size Range	Advantages	Disadvantages
Water Atomization	0 - 500 μm	High production rate, Large particle size range, Feedstock in ingot form	Post-processing for water removal, Irregular particle shape, Satellites present, Wide particle size distribution
Gas Atomization	0 - 500 μm	Wide range of alloys available, Suitable for reactive alloys, Feedstock in ingot form, High throughput, Large particle size range, Spherical particles	Satellites present, Wide particle size distribution
Plasma Atomization	0 - 200 μm	Highly spherical particles	Feedstock must be in wire or powder form, High cost
Plasma Rotating Electrode Process	0 - 100 μm	High purity powders, Highly spherical particles	Low productivity, High cost
Centrifugal Atomization	0 - 600 μm	Large particle size range, Narrow particle size	Difficulty in making extremely fine powder
Hydride-dehydride Process	45 - 500 μm	Low cost	Irregular particle shape, Limited to metals which form a brittle hydride

Surprisingly, the means by which a powder supplier creates powder for its customers is not always initially known and may require further inquiry. Since morphological characteristics of powders differ between manufacturing processes, it is important for quality control purposes to ensure that the powder ordered with the given specifications is the powder received.

3 Powder Chemistry

Powder chemistry refers to the elemental composition of the powder samples. Characterization of a powder particle's chemistry is an essential property to study as it can have a significant effect on the properties of the final part. Powder chemistry can be broken down into surface chemistry and bulk chemistry. The surface chemistry is seldom the same as the bulk chemistry, making its accurate determination critical to comprehensive powder characterization. The surfaces of powder can also differ greatly from the surfaces of the same material in bulk form. For example, stainless steels are known to have a protective Cr_2O_3 oxide film of approximately 1-3 nm thick [73]. However, gas-atomized stainless steels have been shown to have some Cr_2O_3 on their surfaces, but mostly manganese oxides and iron oxides [74]. Bulk chemistry validation is necessary to accurately characterize powder chemistry. This becomes exceedingly important when undergoing recycling studies or using processes that can change the bulk chemistry of the powder. Bulk chemistry validation is needed to ensure that the recycled or tailored powders stay within their alloy designations or have acceptable purity. Many techniques are available to determine the chemistry of the surface layers as well as the bulk of the powder. The choice of an appropriate technique is made in regards to the required accuracy of the data as well as the elements that are present that need to be detected [75].

3.1 X-ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), is a surface elemental analysis spectrographic technique. In an XPS analysis, a sample is bombarded with x-rays causing K level electrons to be ejected and the kinetic energy of these resultant photoelectrons are measured. The binding energy of the photoelectron is determined and it is characteristic of the element, orbital, and chemical environment that the photoelectron was ejected from [76]. In addition to XPS's ability to acquire quantitative information about the elements that are present, it can also give quantitative information about the oxidation state of those elements. XPS is able to detect all elements except for hydrogen and helium. Due to the detection of photoelectrons, the depth of analysis is generally 1-3 nm [75].

XPS has been used to determine the elemental composition and oxidation states of the surface layers of powder particles [38]. XPS instruments also allow for ion sputtering to a particular depth for analysis. A depth profile can be constructed where spectra are taken sequentially after set times of sputtering, yielding a graph of elemental composition versus sputtering time. The sputtering time can then be correlated to a depth of sputtering and the oxide layer thickness can be determined. Although this is complicated with the use of alloys and non-flat surfaces, the method has still seen use with powders [77].

3.2 Auger Electron Spectroscopy

Auger electron spectroscopy (AES) is another surface sensitive, quantitative technique that has been applied to metal samples [78]. In AES, an electron beam is used to probe the surface of the sample, where a series of electron transitions results in the ejection of an L level electron, i.e. the Auger electron. Figure 6 shows the difference between the electron that is analyzed in AES versus XPS. AES is primarily used to determine elemental information about the sample. AES offers the ability to quantitate all elements apart from hydrogen and helium where the analysis depths is typically less than 2 nm [75]. Traditionally, both XPS and AES can be used as a means to determine the thickness of the oxide layer by depth profiling and they are often used in conjunction [77,79].

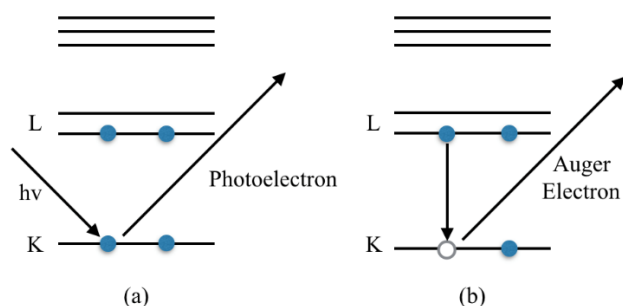


Figure 6: The Photoelectron Emission During (a) XPS and an (b) Auger. In XPS (a), the incident x-rays cause the ejection of a K level electron and the resulting photoelectron is detected. In Auger (b), an electron is excited (by an electron beam) to a higher energy level and the transition of the L level electron to the open K level position causes an Auger electron to be emitted.

3.3 Energy Dispersive X-Ray Spectroscopy

Energy dispersive x-ray spectroscopy (EDS or EDX) is a widely used technique as a semi-quantitative elemental analysis for a variety of materials and samples, including powders [79–82]. Commonly, EDS instruments are inside an electron microscope, meaning samples must be polished and etched prior to the analysis. During an EDS analysis, either an electron beam (electron microscope) or an x-ray beam (x-ray fluorescence) is used to excite the sample surface. The resulting ejected x-rays are detected that can be assigned to specific elements. EDS serves as a qualitative elemental analysis when using the peak locations to determine the elements present. Semi-quantitative information can be determined by measuring the peak heights while quantitative information requires the use of a standard of known chemistry. EDS offers several advantages including their relatively simple construction and ease of use. However, EDS is generally seen as an estimation tool and more rigorous techniques are required if accurate elemental analysis is needed. This technique also has been found to work better for flat surfaces, making accurate chemistry determination on powders difficult to achieve.

3.4 Inductively Coupled Plasma Optical Emission Spectroscopy

Inductively coupled plasma optical emission spectroscopy (ICP-OES, ICP-AES, or ICPS) is a spectrographic technique that uses plasma as the excitation source. Plasma is used as it offers significantly better quantitative data compared to other excitation sources, such as arcs or flames. For an ICPS analysis, metallic samples must be dissolved in an acid solution and fed into the plasma where the sample is converted into free atoms [83]. Due to the high temperature of the plasma, most elements can have good emission spectra under a single set of conditions, offering simultaneous detection of these elements. ICPS measures the intensity of the emission lines of the elements in the plasma and is able to produce quantitative elemental data. It can also detect all metal elements and because elements generally have several emission lines, specific emissions lines for different elements can be used to avoid overlapping of peaks. ICPS is also able to concurrently measure major and trace elements. Typically an internal standard is used to further improve the accuracy of this technique, where concentrations can be determined up to 3 decimal places [76]. However, there are several elements that cannot be accurately detected with this technique (most notably C, O, N, H, and F) [75]. The ICPS is also a relatively expensive and complex instrument, requiring a skilled operator to run the instrument and analyze the data.

Many researchers use ICPS to determine the bulk chemistry of their powders [84–86]. Typically these researchers are adding alloying elements to their material (such as bulk metallic glasses and high entropy alloys) and need to characterize the tailored powders prior to their use in a particular process. This is in contrast to most work with powders, where the manufacturer (or an outside testing laboratory) usually performs the bulk chemistry analysis and the consumer simply confirms that using a less rigorous technique such as EDS. For future recycling studies however, the verification of the chemistry of the powders at each recycling step will be necessary, where a more accurate instrument like the ICPS should be employed.

3.5 Inert Gas Fusion

Inert gas fusion is a quantitative technique used to measure the contents of hydrogen, nitrogen, and oxygen in metal samples [87]. Samples are first weighed and placed in a graphite crucible where they are heated to a molten state. At this point, molecular hydrogen, nitrogen, and oxygen are released from the sample. These released gases are separated and analyzed separately to yield a weight percent of each of the elements. Inert gas fusion is often used in conjunction with ICPS to get a comprehensive determination of the chemistry of a sample [85]. Again, the use of an inert gas fusion instrument is often found in studies where new powder chemistries are being evaluated and their chemistry has to be determined prior to processing.

3.6 Effect of Powder Chemistry

It is well known that different alloying elements can have advantageous or detrimental effects to a material under certain processes. Consider stainless steel, a steel alloy that was developed specifically for corrosion resistance. The main alloying elements that are added to

stainless steel to improve the corrosion resistance are Cr and Ni. Chromium allows for passivation, meaning that at certain conditions an oxide layer forms on the surface that inhibits further corrosion. However, at high temperatures, this chromium can migrate and react with carbon and have a detrimental effect on the steel, a process known as sensitization. Nickel is added to stainless steel, among other reasons, in order to combat the sensitization issue by driving the carbon out of solution.

With any new process, chemistries specifically designed for the particular application are developed in order to optimize the process. Some work has been done on finding additives that aid the laser-based additive manufacturing process. Fe_3P has been found to have several beneficial effects. Iron phosphide forms a eutectic with iron, so the addition results in a lower melting point and therefore a lower input energy is required. It has also been shown to improve the surface finish and density due to the fact that it lowers the surface tension of the melt pool [58,88,89]. In addition, Averyanova et al. [20] performed a study on 17-4 PH steel with powder of slightly different chemistry. They showed that this change in chemistry had a significant difference on the final part's microstructure and therefore their mechanical properties as well [90].

Another study involving 17-4 PH steel was by Slotwinski et al. [38] who used XPS to reveal high oxidation on the stainless steel particles being used in various additive manufacturing processes, although the effects of such were not investigated. In regards to metal powder-based AM, oxidation is viewed as a contaminant and should be avoided due to its ability to increase part porosity. Simchi [11] observed this phenomenon by correlating the initial oxygen concentration in the form of oxides on the powder to the parts that were built. The results indicated that an increase in the initial oxygen content in the powder led to higher porosity in the SLS manufactured components. Tang et al. [91] also observed that an increase in the oxygen content of Ti-6Al-4V in the EBM due to powder reuse can force the material to accrue oxygen to the point of exceeding the maximum specifications for oxygen content of the material. Due to this reason, it was found that the increasing oxygen contaminant creates a situation where the powder cannot be used more than four times.

Ardila et al. [29] used EDS technique to track the evolving chemistry of IN718 powder as it was recycled in the SLM process. It was concluded that the chemistry of the powder does change with continual reuse, although only slightly. In the end, no significant change was observed in the measured fracture toughness of the parts manufactured. However, it is important to note that care must be taken when using EDS solely to characterize chemical composition of a material, especially a powder. This technique is often thought to produce quantitative results; yet, as previously mentioned, it is primarily a qualitative method unless a sample with known chemistry is used for calibration purposes. This information coupled with only the slight variation in chemical composition observed points to the fact that the sole use of EDS is not able to completely detect any minute differences between the virgin and recycled powder. Therefore, the chemical results produced by Ardila leave the reader without a definitive look at the change

in chemical composition as a result of reuse simply because a lack of characterization was performed.

It is important to note that the chemical composition of metals often includes upper and lower limits for the amounts of each element that can be present within the bulk material. Therefore, in order to reduce variability in part properties, powder chemistries must be tailored specifically for the application. Work in this area is still in its infancy in powder-based AM. It has been suggested that due to the similarities of AM and welding (intense power source and high cooling rate) that materials that work well with welding or weld wire chemistries may be a good place to begin the testing for AM alloys. More studies are necessary to pinpoint these materials.

4 Powder Microstructure

Powder microstructure corresponds to the phases present in the powder samples and inevitably effects part characteristics. Since porosity influences crystal structure, it is often studied in conjunction with microstructure. In AM, porosity in parts is often quantified and related back to process parameters. However, porosity can also arise as a result of voids within powder particles. Therefore, it is important to identify if internal particle porosity exists in AM-ready powder in addition to potential microstructure effects the powder has on the final component properties. This section highlights various methods that are commonly used to study the microstructure of powder, and how these techniques have been used in AM for characterization of input material properties.

4.1 Metallography

Studying the internal microstructure of powder particles can be accomplished using traditional metallographic techniques, however some adjustments have to be made due to the small size and circularity of AM powders [62]. A powder sample can be mounted in bakelite or, preferably, epoxy and subjected to a polishing procedure. The procedure for polishing the mounted powder particles differs from that for bulk material. In the grinding steps, only 600-1200 grit SiC paper should be used, as any smaller grits will result in removing the particles from the mount with little grinding of the actual particles occurring. Fine polishing often results in edge rounding leading to loss of surface details. This problem can be avoided by using vibratory polishing. Once a satisfactory polish is achieved, etching of the surface is carried out to reveal microstructure and potentially enlarge internal pores. Detailed information on the selection of etchants is provided in reference [92]. It is necessary to use a swab/immersion etchant for the etching of the mounted powder particles, as electrolytic etching has inherent problems given the separation of the particles and the conductivity of the mounting material. The cross-sections obtained by polishing the mounted particles also offers a means to determine the morphology of the particles as previously described, as you already have a two-dimensional image of the powder.

4.2 X-ray diffraction

X-ray diffraction (XRD) is another x-ray spectrographic technique primarily used in identification of crystal structures. During an XRD analysis, a sample is bombarded with x-rays and the intensity of the diffracted x-rays are measured. Constructive interference of the diffracted x-rays is needed in order to have a distinguishable peak. Constructive interference occurs when the distance between points A and B in Figure 7 is equal to the distance between B and C. Trigonometry yields Bragg's law as the condition for constructive interference:

$$n \lambda = 2 d \sin \theta$$

where n is an integer, λ is the wavelength of the incident radiation, d is the d-spacing of the crystal, and θ is the degree between the incident x-rays and the sample surface [76], [93]. All seven crystallographic systems have expressions for their d-spacings. With this information and the knowledge of allowed reflections for specific Bravais lattices, the crystal structure can be determined by indexing the peak positions. XRD can also be used as a means to characterize the powder chemistry, but only if known diffraction patterns of that same chemistry are in the powder diffraction library.

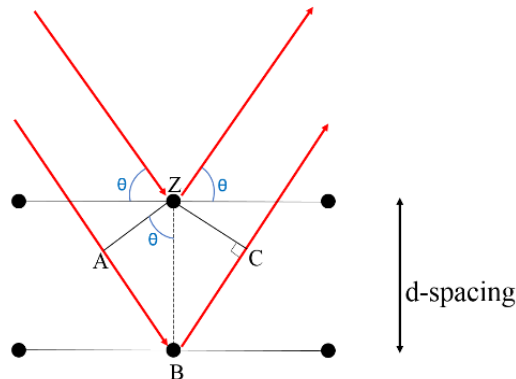


Figure 7: Principle of X-Ray Diffraction. The black dots indicate atoms in their lattice positions, red lines are the x-ray path, and the angle, θ , is shown. In order for constructive interference, $n\lambda$ must equal $AB + BC$, or $2AB$. Observing the triangle ABZ shows that AB is equal to $\sin(\theta)d$. Substitution yields Bragg's Law of $n\lambda=2d\sin(\theta)$.

XRD has been extensively used as a characterization method for metal powders [22,94–97]. Many powder diffraction standards are available that generally enable the use of XRD for chemical verification. However, advanced materials sometimes have intermetallic compounds with complex spectra that are not necessarily well known. In these cases, XRD can only be used as a means to determine the crystal structure of the elements present. XRD has also shown use in the characterization of amorphous materials where it can be used to semi-quantitatively and quantitatively determine the amount of crystallinity of materials.

4.3 Thermal Analysis Methods

Thermal analysis techniques are primarily described as tracking chemical properties as a function of temperature. Rather than offer a direct observation of the microstructure, this class of methods yields information about the exothermic and endothermic events that are occurring in the sample. This offers the ability to determine events such as glass transition and crystallization temperatures, therefore giving information about the microstructure. In general, there are three main techniques that are used: thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential scanning calorimetry (DSC).

TGA measures the mass of a sample as a function of temperature. Since it is often a question of how a material behaves under elevated temperatures, this approach allows insight into loss of volatiles and oxide formation. In many powder-bed fusion processes, the temperature of the powder bed is often raised. Thus, this technique allows ex-situ analysis of how the powder responds when subjected to increased temperatures. Differential thermal analysis (DTA) is a qualitative technique that measures the temperature difference between a sample and a reference material as a function of temperature. This temperature difference is plotted against time and can be used to study transitions to different phases. Often, TGA and DTA are often coupled for simultaneous analysis. DSC is a quantitative technique that measures the difference in energy between a sample and a reference material as a function of temperature. DSC is a popular technique in the characterization of metallic glasses [98,99].

4.5 Effect of Powder Microstructure

In powder metallurgy, it is well known that the initial microstructure of powder plays a key role in the properties of manufactured components. For instance, consider the sintering of a pressed compact. Not only does internal porosity and the size of the initial grains in the powder determine the sintering activity of the compact, but also the formation of pores in the final product. Although the manufacturing processes in press-and-sinter powder metallurgy are considerably different than those found in AM, the potential impacts of particle microstructure need to be studied especially since the current AM literature contains very little research in this particular area.

As mentioned previously, the method of powder manufacture will often determine the morphological characteristics of particles. However, it is also important to note that the powder manufacturing process also has a significant impact on the developed particle microstructure [37]. Starr et al. [100] proved this dependence by performing XRD on two gas-atomized 17-4 PH powders where one was atomized in a nitrogen atmosphere and the other being argon. It was shown that the nitrogen-atomized powder contained primarily an austenitic microstructure, while the other being manufactured under argon contained mostly martensite. In order to observe the effect of different powder microstructure and build atmosphere on the phase composition of as-built materials, both powders were used to build tensile specimens where XRD was performed before tensile testing. The XRD results for the as-built parts showed that the argon-atomized

powder produced fully martensitic products regardless of the gas used during the build process. However, the nitrogen-atomized powder showed a different behavior where the parts built under a nitrogen atmosphere retained more austenite than those produced in the presence of argon.

Similarly, Murr et al. [22] used XRD to determine a possible dependence of 17-4 PH powder microstructure on the phases present in parts in both argon and nitrogen atmospheres. In the end, it was determined that building under an argon atmosphere produces martensitic products, whereas using nitrogen for gas in the build chamber produced a phase in parts identical to the precursor powder phase. This major difference was attributed to the large difference in thermal conductivity of the gas used during the build process.

Internal particle porosity can also have a major effect on the microstructure of powder in addition to the density achieved by the final part. Therefore, it is necessary to quantify this powder characteristic as well for quality control purposes. However, this is often not practiced in many studies warranting further characterization of AM-ready powder to be done in this particular area.

5. Conclusion

As additive manufacturing reaches a stage of maturity, the realization of these processes for potential production becomes ever more so apparent. A significant amount of research has focused on the development of process parameters to manufacture parts having strength comparable to wrought materials, low surface roughness, and minimal porosity. However, both intra-build and inter-build part variability are often noticed forcing researchers to now look at the input material properties and their effects on the build process. This has led to the use of different powder characterization techniques to quantify and predict the behavior of AM-ready powder.

When characterizing a powder, it is important that the following three main areas are researched: particle morphology, chemistry, and microstructure. In AM powder-bed fusion processes, research is primarily focused on the morphological characterization of powders and their effect on part properties. Techniques that are typically used to perform morphological characterization of AM powders include microscopy and laser diffraction, although sieve analysis is occasionally employed as well. Microscopy not only allows the characterization of particle size and size distribution through micrographs, but also shape and surface roughness. Therefore, it is an extremely versatile tool making it attractive in powder-based AM. Unfortunately, the analysis times are long and the amount of particles needed in order to obtain statistically significant results is still currently under debate warranting further investigation. Conversely, laser light diffraction is able to measure the size of a very large amount of particles in a short amount of time. Due to the large particle count and automation of the entire procedure, particle size distributions are highly repeatable. Yet, particles that deviate from a spherical shape can give erroneous results.

As a whole, the effect of particle size and size distribution is the most studied when input material properties are correlated to part quality. From these findings, it is clear that the particle size and size distribution affect the flowability of the powder thus influencing layer homogeneity. Moreover, the porosity of as-built parts is also influenced where the use of large particles and agglomerates can produce undesired voids due to a decreased packing density in the powder bed. Some of the same phenomena have been noticed when irregular particles are used as these tend to produce more interparticle friction and impact layer uniformity and density. In order to maximize flowability while also increase the powder bed density, narrow particle size distributions that contain spherical particles with little surface deformities are often employed.

Characterization methods to determine the chemical composition of powders fall under two categories: surface analysis and bulk analysis. XPS is a quantitative surface sensitive technique that takes advantage of the photoelectron effect to identify percentages of elements that are present. High-resolution spectra can even be obtained to determine binding information for each element in a sample. As for bulk chemistry, EDS is commonly utilized; however, it is important to note that this is only a semi-quantitative technique meaning that the results should not be interpreted as absolute unless appropriate reference standards are used for machine calibration. Unfortunately, many studies only use EDS for chemical characterization of powder and treat it as a quantitative method. ICPS is a much more accurate technique that can be used to determine the bulk chemistry of a sample. This technique paired with inert gas fusion can give the concentrations of all metallic elements as well as the oxygen content.

A significant amount of research will need to be performed in order to determine the effect of chemistry on part properties. A number of studies in this particular area focuses on powder contaminants, namely, oxides. Few studies discuss the potential of optimizing powder chemistries to yield desired part properties. This research will inevitably need to be strengthened in the future as the AM industry grows.

The most widely used methods to characterize a powder particle's microstructure are using XRD and thermal analysis. As seen with particle chemistry, a lack of research is also noticed in terms of particle microstructure. Currently, most of the work aimed at characterizing the microstructure of powders uses XRD with cross-sectioned and polished samples. Although this is a great first step, the exact influence of particle microstructure on as-built parts is unknown. A few aforementioned cases not only stated the importance of crystal structure, but also its dependence on the type of gas flow during the build process and its relevance in the microstructure of parts.

Overall, much research work is still needed in order to fully understand the influences of powder in AM powder-bed fusion processes. The current approximate knowledge of necessary morphological powder characteristics coupled with an insufficient understanding of particle chemistry and microstructure on the manufacturing processes necessitates a significant need towards a more complete characterization of AM-ready powders. Therefore, the primary focus of research should be not only on optimizing process parameters, but also on the interplay between

these variables and the powder properties. This will result in reducing variability between builds and parts, and allowing the process to be more controllable. Only then will AM processes be able to rival traditional manufacturing methods in terms of being recognized as a viable means for the creation of reliable components.

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