

Powder Metal Testing for 3D Printing (Additive Manufacturing): Characterization and Safety

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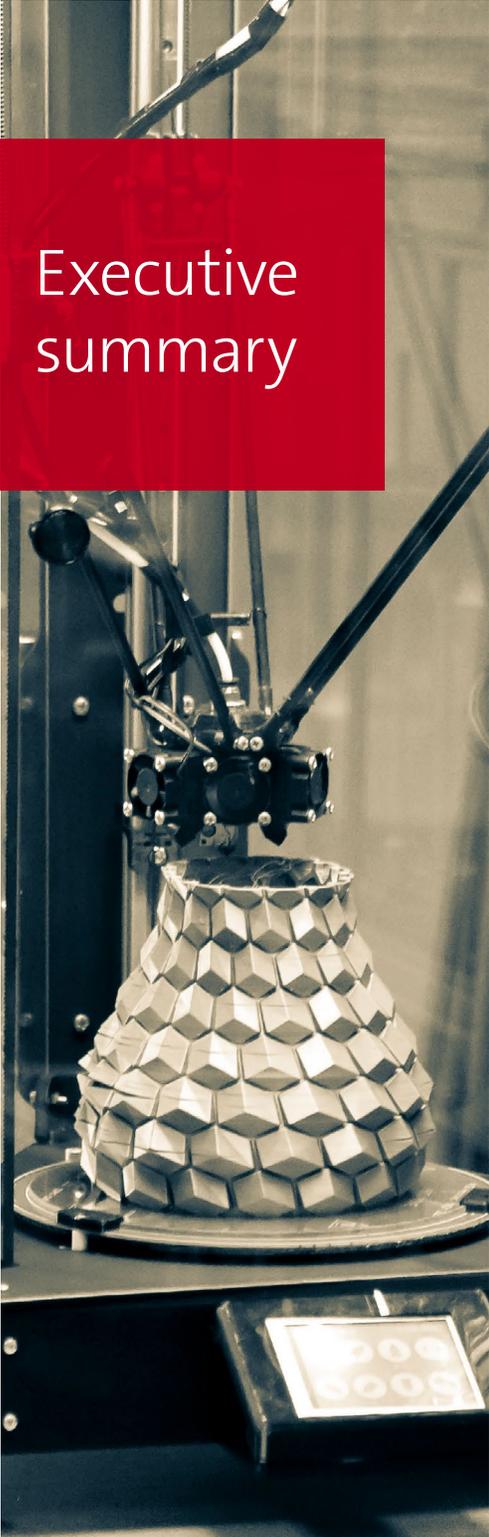
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Executive summary

The general goal of producing a 3D-printed metal part is to use it in production applications that take advantage of the properties of the metal used. The most common form of 3D printing (also known as additive manufacturing) with metal consists of sintering or melting together layers of powder metal in precise designs.

This is performed in subsequent layers, one on top of the other, until the fully formed part has been manufactured. The heat source for sintering or melting is a high-energy beam, such as a laser or electron beam. These parts can then go through post-processing, which can include heat treatment, surface refining and porosity impregnation.

3D printing allows the manufacture of parts with intricate designs that may have been difficult or impossible with conventional manufacturing processes, and it does so directly from a design file without complicated tooling preparation. The nature of the technique permits the production of cavities within the parts that were previously not otherwise possible, and these effectively decrease the weight of a part. It also enables the production of parts that, in the final state, are made up of one piece of metal, whereas more traditional fabrication methods require multiple pieces to be made for the same part and then joined; such joints add manufacturing steps and can act as points of weakness for a part. The usefulness of metal parts produced by 3D printing is evident in many fields, including automotive and aerospace applications. The medical field benefits from using 3D-printed metal parts because they can be made selectively porous in areas to increase the biological acceptance of surgical implants; the porosity provides pathways for bio-integration. They can also be used to quickly produce intricate dies and molds for use in more conventional manufacturing processes.

The properties of the powder metal being used can have a strong effect on a finished part and can be used to set various parameters in the 3D-printing process. The shape and size of the powder particulate can potentially set the minimum dimensional accuracy of the part. The shape, flow characteristics (flowability) and packed density of the powder can affect the potential porosity in the finished part. The powder's thermal properties and absorptance/reflectivity can be used to determine the heat source, energy, and travel speed needed to accomplish sintering or melting of the desired spot size and depth. Perhaps even more important than the characteristics affecting the finished part are those that ensure safer work environments. It is important to know whether a powder is flammable or explosive and, if it is, whether there are safe conditions that can help prevent this hazard so that such substances can be handled accordingly. Besides providing information on the possible mechanical and electrical properties of a manufactured part, a complete understanding of the powder's chemistry can help determine whether a powder poses a health hazard, such as being toxic, caustic, mutagenic or carcinogenic. This paper summarizes potential tests that can help characterize powder metals for use in additive manufacturing and identify potential hazards.

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Powder metal testing for additive manufacturing: Characterization and safety

Background and motivation

Additive manufacturing, also referred to as 3D printing, is an established approach advanced by the rapid acceleration of innovations, such as using powder metal in 3D printing. 3D-printed parts now perform critical functions in industries that rely on quality parts, from aerospace and defense to medical and dental to consumer goods and electronics.



3D printing is a computer-aided process in which a part is manufactured by adding material in increments (usually in layers) to form a part.¹ This technique supports the manufacture of parts with intricate designs that may have been difficult or impossible through more conventional manufacturing processes. By working directly from a design file, 3D printing eliminates the hassle and potential errors from a complex tooling preparation. Other advantages include the manufacture of lighter-weight parts that use less material, as 3D printing permits the production of cavities within parts that previously were not otherwise possible. Because 3D-printed parts consist of one single piece of metal, they lack joints that can be weak points in the part.

The powder metals used in 3D printing can enhance the overall product's performance but may require precautions

for safe use during manufacturing. These precautions are determined by the chemistry of the powder and the production process used by its supplier; users should not assume that performance of powder metals will be uniform across multiple suppliers. Powder versions of metals may be more chemically reactive than in bulk form—even pyrophoric—and can pose fire and explosion hazards if not handled properly.

In this paper, we will outline the characteristics of powder metals that may affect their use in additive manufacturing, define their hazards and identify tests to evaluate powder metals for 3D printing.

Key powder metal 3D-printing processes: Direct laser sintering and direct laser melting powder metal

While additive manufacturing and computer numerical control (CNC) machining both rely on computers to produce parts, the key difference is the method used. CNC machining begins with a bulk piece of metal and uses subtractive methods to cut material away to shape the desired part.

Numerous methods for using metal in 3D printing are used, and this area is rich with innovation. Typically, this approach consists of sintering or melting thin layers of powder metal onto preceding layers, known as direct laser sintering (DLS) and direct laser melting (DLM), using a high-energy beam source such as a laser. Within the apparatus, the bulk of the metal powder is stored in a powder bed from which the thin layers of powder are spread onto a flat workstation.

After the desired pattern is sintered or melted into a layer of powder, a new thin layer of powder is spread over it. A pattern is then sintered or melted into this layer, ensuring that the conglomerated material bonds to the preceding layer. This is repeated until the layers build up to form the desired part.

Table 1: Characteristics of powder metal printing to consider when assessing their respective processing parameters for 3D printing

3D-printing parameter	Powder metal characteristics
High-energy beam source	Thermal characteristics, absorptance/reflectivity, size and shape
Beam energy	Thermal characteristics, absorptance/reflectivity, size and shape
Beam traveling speed	Thermal characteristics, absorptance/reflectivity, size and shape
Powder layer thickness	Size and shape, chemistry, flowability, densities
Compaction of the powder layer	Size and shape, chemistry, flowability, densities
Chamber atmosphere	Flammability/explosibility, size and shape, chemistry
Powder handling/storage	Flammability/explosibility, size and shape, chemistry

The temperature of the powder bed is critical during 3D printing, as the difference in temperature between the molten and cooler layers may create warping through residual stresses, which can weaken the part. The powder bed may be heated to reduce this difference in temperature, depending on the design and size of the part. A variety of options and parameters can be controlled in these processes, and proper process design is essential to producing a part of the desired dimensions, detail and porosity while maintaining safe working conditions.

Table 1 illustrates the impact of powder metal characteristics on 3D-printing parameters.

Manufacture of powder metal

The process used to manufacture powder metal directly influences the size distribution and shape of the particles.

Atomization

This is the most common method used to produce powder metals, but the term is imprecise, and a variety of distinctly different methods fall under this classification.

Water and inert gas atomization: This method transforms a thin, falling stream of molten metal into powder through disintegration by impingement of high-velocity jets of liquid or gas. Water is generally used for ferrous material and produces irregularly shaped particles. When inert gas is used, the shape of the particles varies based on the time allowed for surface tension to take effect before solidification. Low heat capacity gases, such as nitrogen and argon, extend this time and enable the creation of spherical particles.

Atomizing jets can be positioned to strike the molten stream at the source. Called close-coupled or confined atomization, this approach eliminates free-fall height and is useful for producing fine powders.^{2,3}

Plasma atomization: This process employs argon plasma torches at >10,000°C to melt metals into fine droplets. It results in a fine, highly flowable powder of pure metal suitable for materials with high melting points, such as superalloys.

Centrifugal atomization: In this process, a container of molten metal is rotated at high speed, causing droplets to propel outward. Alternatively, molten metal may be poured from an opening onto a rotating disk or cone. In the rotating electrode process (REP), another form of centrifugal atomization, a bar of metal rotates in an arc and melts the free end, forming



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droplets as the molten metal is propelled away from the bar due to centrifugal force. The arc source can be a tungsten electrode or a plasma source. When a plasma source is used, this process is referred to as the plasma rotating electrode process (PREP). This process is the leading method for the production of titanium powder metal.^{2,3}

Vibration-based atomization: This technique features a stream of liquid metal striking an internally cooled surface vibrating at ultrasonic frequency. This causes fine particles to be repelled.⁴

Electrolysis

Electrolysis is similar to metal plating, but instead of depositing a solid, well-adhered layer of metal onto a substrate, this method deposits a powdery layer. This is most commonly used in the manufacture of copper powder.³ In electrolysis, a pure metal anode is submerged in a high-acid electrolyte with a low-ion concentration. A high cathode current density is applied, and the powder is deposited loosely onto a cathode substrate submerged in the electrolyte. Particulates formed using electrolysis have a dendritic (pine tree-like) shape and high purity.

Mechanical comminution

Powder metal can also be produced mechanically from larger-scale pieces of metal. Mechanical comminution is the production of powder through pulverization in ball mills, hammer mills and attritor mills. The particle shapes produced by mechanical comminution vary depending on the relative ductility and brittleness of the material. Ductile powders are often flat with high aspect ratios, while brittle materials are generally angular and regularly shaped. The size distribution is fairly uniform, with the size controlled by the size of the pulverizing elements in the mills.^{2,4} Figure 1 shows a diagram of various mechanical processes used to produce powder metals. Notice that, as in this figure, lists of mechanical processes sometimes include atomization.

Additional chemical methods of production

Two of the most notable chemical conversion processes that can be used in producing powder metals are metallic oxide reduction and the carbonyl processes.

A metallic oxide reduction process of particular note is the sponge iron process. Magnetite (Fe_3O_4) is placed in a sealed tube made of refractory material, typically silicon carbide (SiC), along with coal, coke and limestone. The tube is then placed in a furnace at around $1,200^\circ\text{C}$, at which point the combined materials in the tube decompose to release a reducing atmosphere.² The magnetite is converted into a porous iron cake and a layer of slag, which is subsequently removed. The iron is then crushed into powder. Iron powder produced by this method has highly irregular and angular-shaped particles with substantial internal porosity.^{2,3,4}

The carbonyl process is used to manufacture fine iron and nickel powders. The raw metal is exposed to carbon monoxide under pressure to form a carbonyl, which is gaseous at the reaction temperature. The pressure is then decreased and the temperature is raised such that the carbonyl gas decomposes to deposit the fine metal powder.

An additional chemical conversion process includes metals precipitated from a soluble salt, such as precipitating silver by adding a reducing agent to a silver nitrate solution. The Sherritt-Gordon process is used to precipitate nickel powders using hydrogen reduction of a nickel salt under pressure. In general, chemical processes produce particulate with a full spectrum of shapes, ranging from spherical to angular.²

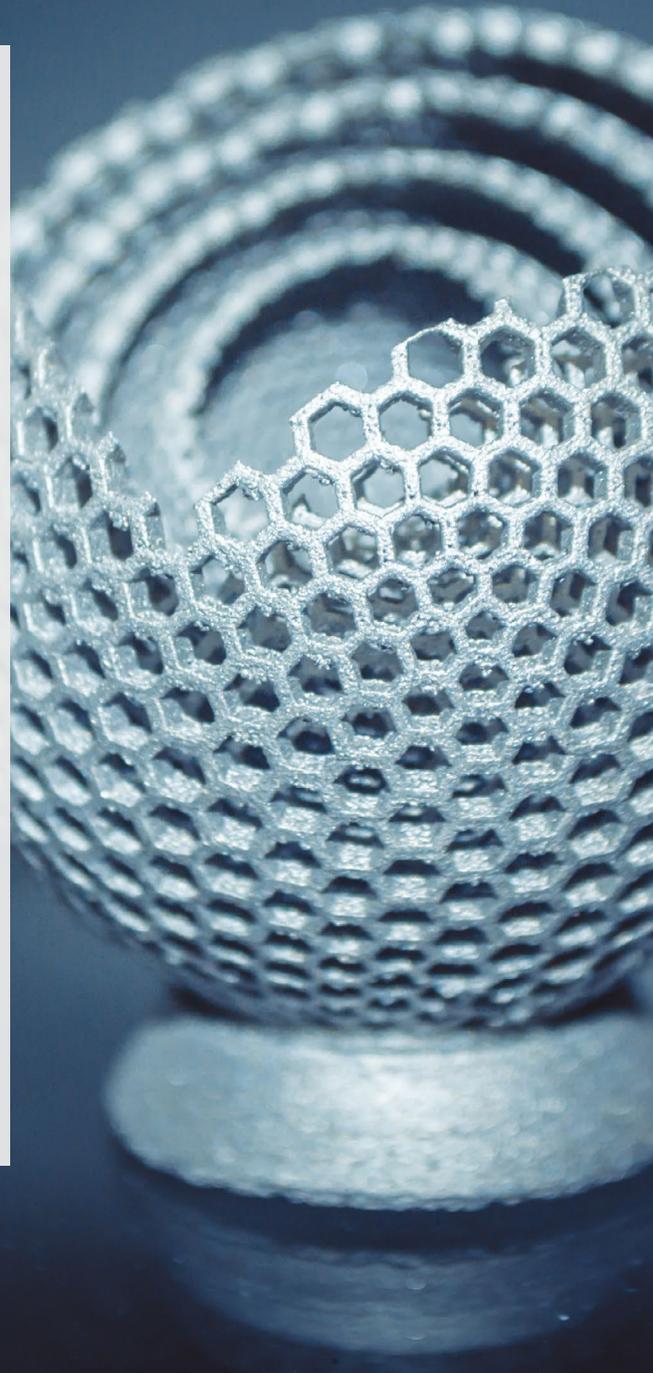


Figure 2 shows a diagram of various chemical and electrochemical methods used to produce powder metals.

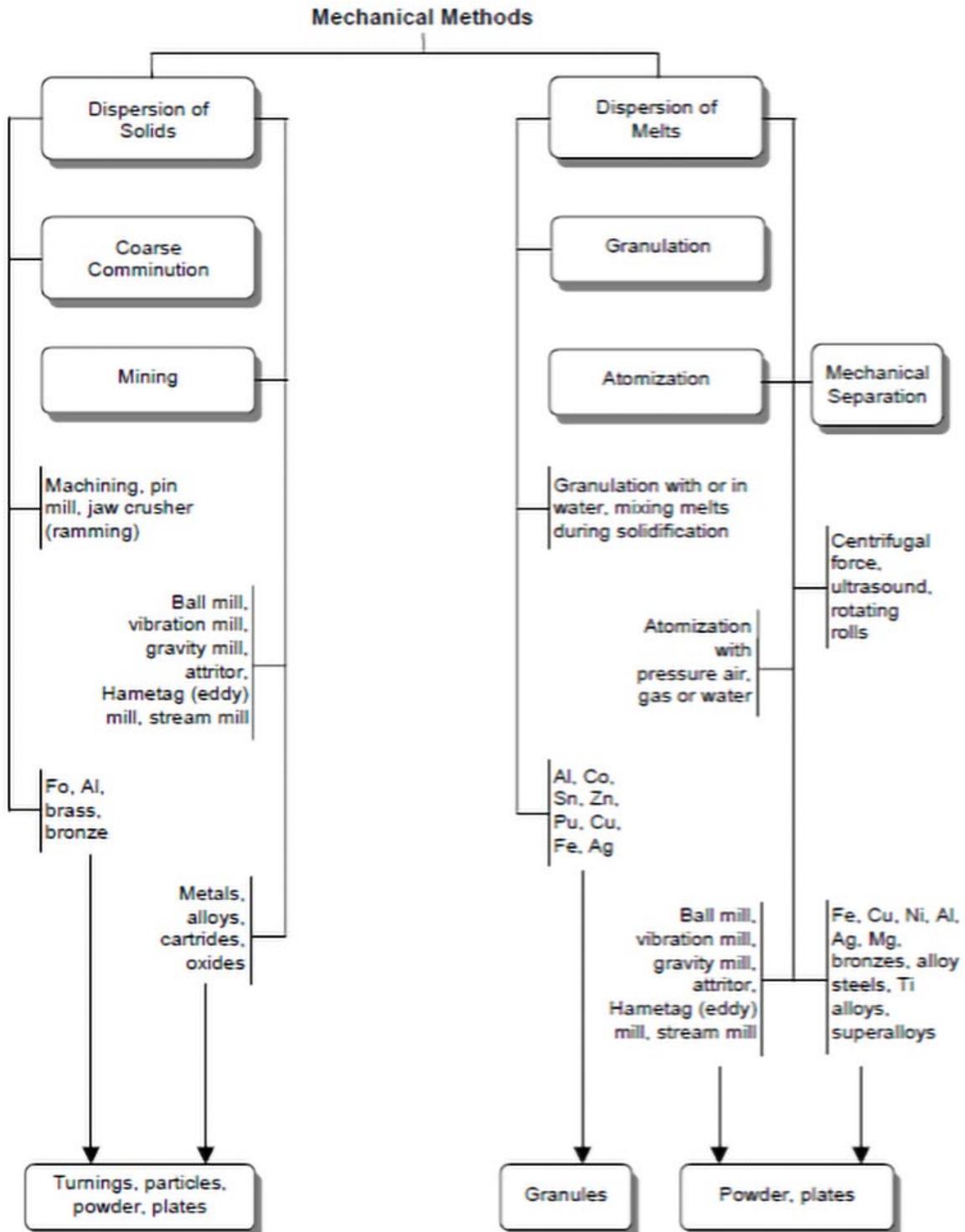


Figure 1: Mechanical processes, including atomization, used to make powder metals
 It should be noted that particulates produced through granulation may not truly be considered powders due to their large size and are not being considered for use in 3D printing.⁴

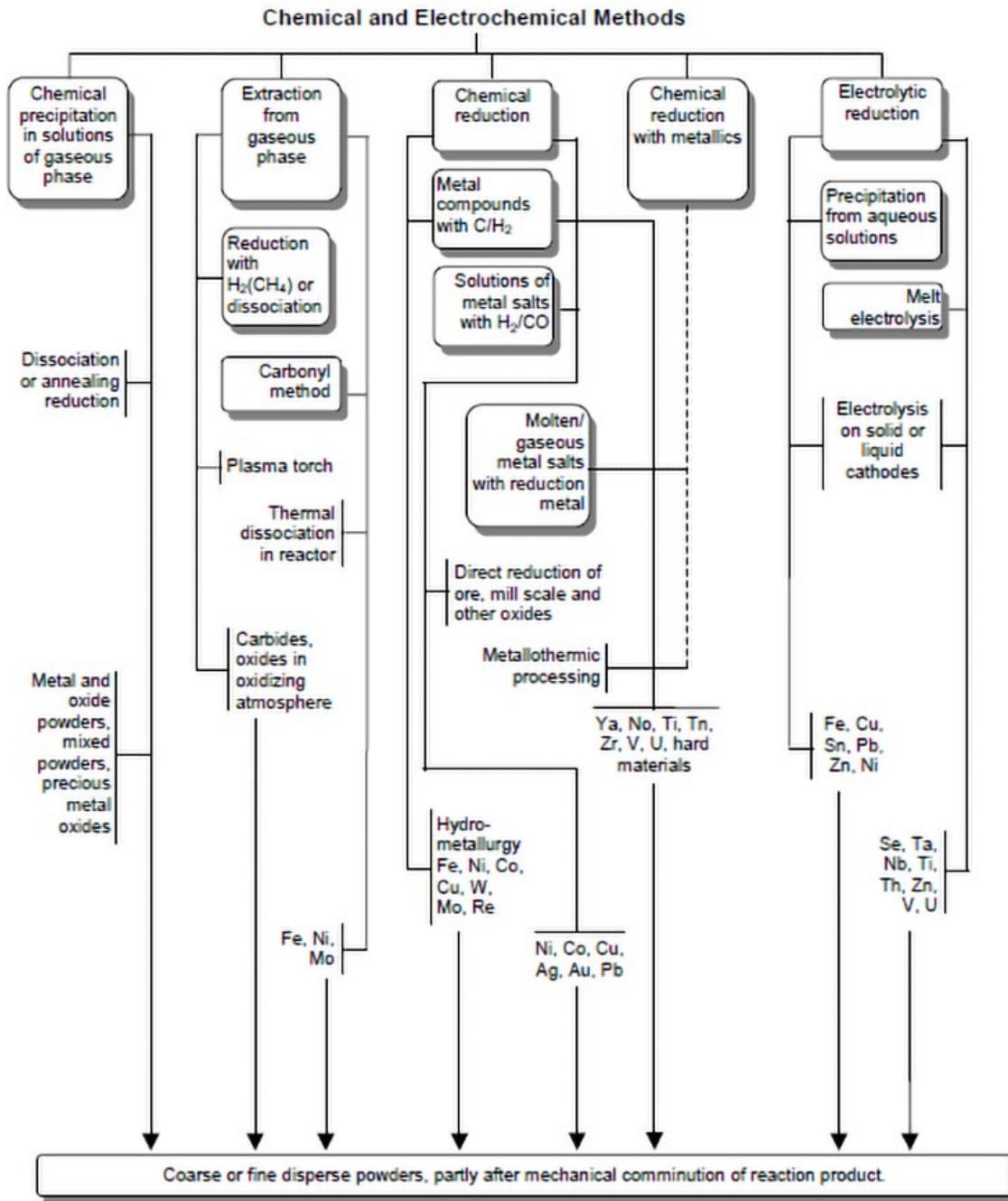


Figure 2: Chemical and electrochemical processes used to produce powder metals⁴

Powder sampling

Representative samples are required to test powder metal as batches are large. To ensure an appropriate sample, measures must be taken to avoid a heterogeneous particle distribution caused by particle settling. It is preferable that the bulk sample be taken while the powder is in motion, but a sample may also be taken from a static batch using a specialized tool called a Keystone sampler, or “sample thief” — see Figure 3 for an illustration.⁵ With flowing powder, material should be sampled from different stages of the batch, such as the beginning, middle and end, when transferring to a different container. Static batches may be sampled from the bottom, middle, top, front and back.



Figure 3: Illustration of a Keystone sampler, also known as a sample thief⁵

After mixing the bulk sample, typically in a blender designed for the purpose, the powder metal must be split into more manageable sample sizes. Bulk product may be split into equivalent samples using a variety of tools outlined by different standards. ASTM B215 calls for a chute-based sample splitter or a spinning riffler, as shown in Figure 4. For a more detailed description of the sampling procedure for powder metals, refer to ASTM B215.⁶

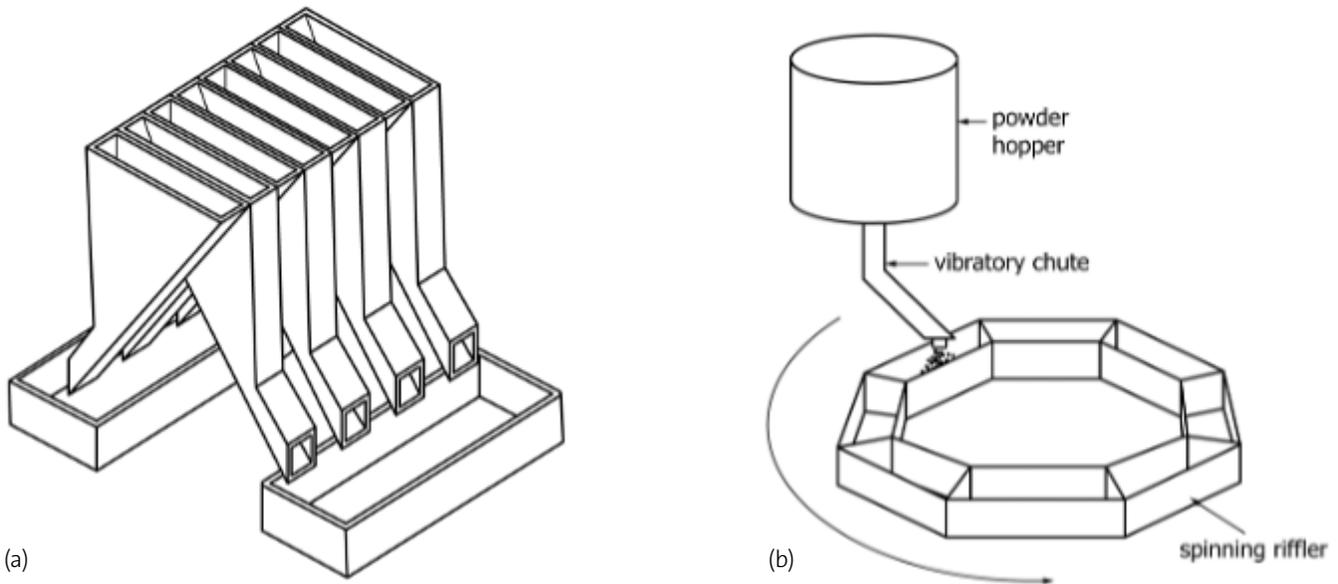


Figure 4: Illustrations of a sample splitter (a) and a spinning riffler (b) for splitting a bulk sample of powder into portions for testing⁶

Particle size

The size of powder metal particles can serve as the minimum part layer thickness and the minimum printed dimension for any detail of the part. Particle size also affects the thermal conductivity of the powder, which is described in more depth later in this report.

Quantitative information regarding particle size is invaluable in determining a given powder’s design and processing capabilities and viability for a desired application. Powder metals used for making alloy products may be made from the atomized alloy or by mixing individual powder metals, in which a variety of particle sizes and shapes may be present.

The ASTM standard designed as a guide for characterizing powder metal for additive manufacturing, ASTM F3049,⁷ suggests using ASTM B214⁸ or B822⁹ to determine the size distribution. ASTM B214 describes passing the powder

through a series of sieves for this purpose. However, this standard only uses sieves with openings from 45 to 1,000 µm, making it unsuitable for particles outside of this range.⁸ ASTM B822 allows the measurement of particles between 0.4 and 2,000 µm. In this method, the powder disperses in a circulating medium, which can be a nonreactive liquid or gas, and passes through a light beam. A particle size analyzer evaluates the light scattering from the particles to provide a size distribution.⁹ The light source is typically a laser, so this method is often called the laser diffraction method.¹⁰ Research has shown, however, that ASTM B214 can be modified with the addition of electroformed micromesh sieves down to 5 µm, which produces more reliable results than those gained through light scattering analysis.¹¹ ASTM F3049 states that nonstandardized methods such as image analysis can be used for size distribution measurements.⁷

Particle shape

ASTM F3049 notes that no standard currently describes a method to quantify the particle shape (also referred to as morphology) of powder metals. It does, however, point to ASTM B243¹² for qualitative definitions to describe powder shapes (which can be found in Table 2) and indicates that both quantitative and qualitative analyses are possible through light scattering and image analysis.⁷ As discussed previously, particle shape is directly related to the powder processing method. Figure 5 illustrates this connection.

Table 2: Definitions from ASTM B243 used to categorize particle shapes¹²

Shape	Definition
Acicular powder	Needle-shaped particles
Flake powder	Flat or scale-like particles whose thickness is small compared with the other dimensions
Granular powder	Particles having approximately equidimensional non-spherical shapes
Irregular powder	Particles lacking symmetry
Needles	Elongated, rod-like particles
Nodular powder	Irregular particles with knotted, rounded or similar shapes
Platelet powder	Composed of flat particles of considerable thickness (as compared with flake powder)
Spherical powder	Globular-shaped particles

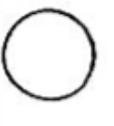
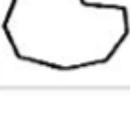
One-dimensional			
<i>Acicular</i> Chemical decompositions		<i>Irregular Rod-like</i> chemical decompositions mechanical comminution	
Two-dimensional			
<i>Dendritic</i> Electrolytic		<i>Flake</i> mechanical comminution	
Three-dimensional			
<i>Spherical</i> atomization carbonyl (Fe) precipitation from a liquid		<i>Irregular Rod-like</i> atomization chemical decomposition	
<i>Irregular</i> atomization chemical decompositions		<i>Porous</i> reduction of oxides	
<i>Angular</i> mechanical disintegration carbonyl (Ni)			



Figure 5: Particle shapes along with manufacturing processes typically associated with them¹³

Flow and density characteristics

The fluidity of a powder, or how easily a powder flows, is an important factor for many additive manufacturing processes. Powder flow and compaction are highly dependent on interparticle friction, which is directly influenced by chemistry and particle shape, largely a result of the production process.¹⁰

Two ASTM standards are available for measuring the mass flow rate of powders: ASTM B213 (Hall Flowmeter funnel method)¹⁴ and ASTM B964 (Carney funnel method).¹⁵ The test procedures described in each standard are identical except for a single part of the test fixture: a calibrated funnel. With a 2.54 mm diameter, the Hall Flowmeter funnel has a smaller opening than the Carney funnel at 5.08 mm.

ASTM B213 is the default method used, while the Carney funnel method is only used when the powder will not freely flow through the Hall Flowmeter. The tests measure the amount of time it takes a sample of powder of known mass to pass through a funnel of standardized dimensions. See Figure 6 for an illustration of the test setup.

The ASTM standards describe static and dynamic methods for taking these measurements. The static method involves blocking the funnel's opening with a dry, possibly gloved finger before pouring the powder sample into the funnel. The finger blockage is then removed and the time recorded until the funnel empties. The dynamic method involves pouring the powder sample into the open funnel and recording the time between when the powder starts and stops coming out of the opening in the funnel.^{5,14,15} Another option is to record the volume flow rate using ASTM B855, which is equivalent to the Hall Flowmeter funnel method in ASTM B213 but uses a sample of known volume (using an Arnold Meter) instead of known mass.^{7,14,16}

Calculating various densities

Characterizing the apparent, skeletal and tapped densities of a metal powder is valuable for understanding the expected behavior of a powder in additive manufacturing. These measurements not only give the density of the metal but also describe the probable porosity present in the powder as it is laid on the printing bed. This information can be used to predict the final porosity in a manufactured part and can be used to adjust the manufacturing process, such as whether to use a roller to compact the powder of each additive layer. As with the flow of the powder, the apparent and tapped densities are highly dependent on interparticle friction, influenced by material, particle shape and production process.

Apparent density is the mass of a unit volume of powder. This is the ratio of the mass to a given volume of powder, taken up by the particles of powder, the spaces in between them and any porosity in the particles. ASTM B212 and

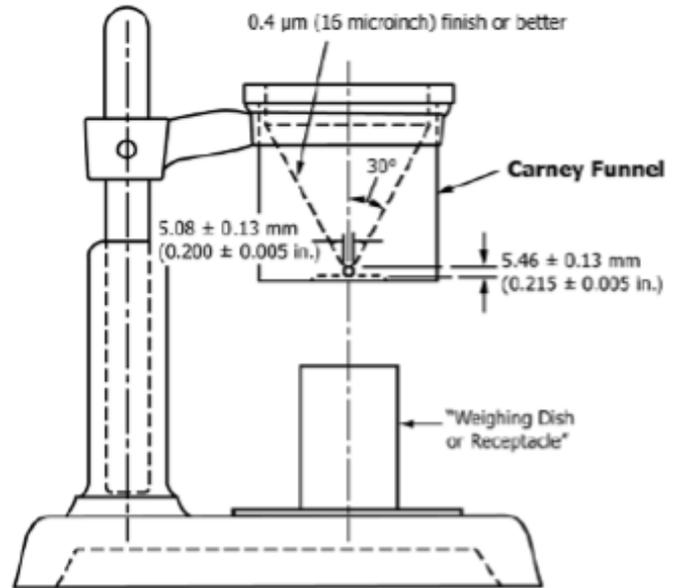


Figure 6: Illustration of the test setup for the Carney funnel method (ASTM B964) for measuring the flow rate of metal powder
ASTM B213 and ASTM B855 use an equivalent setup with a Hall Flowmeter.^{14,15,16}

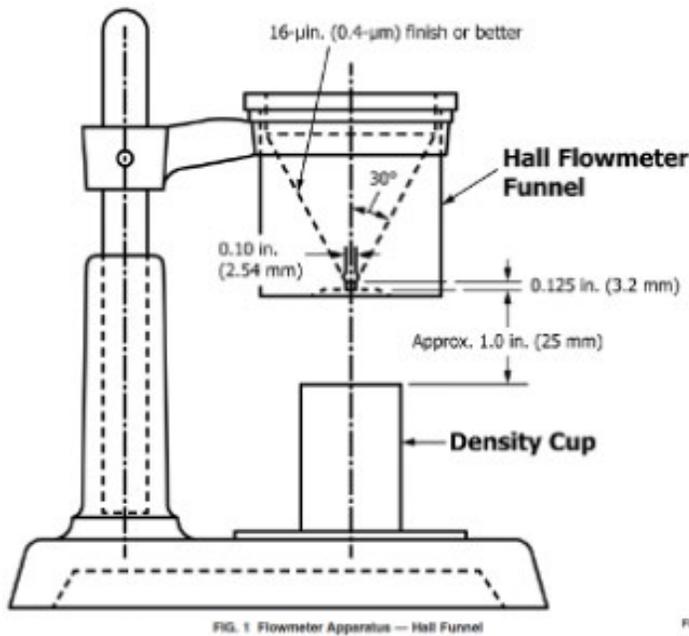


FIG. 1 Flowmeter Apparatus — Hall Funnel

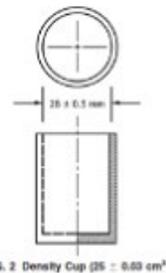


FIG. 2 Density Cup (25 ± 0.03 cm³)

Figure 7: Setup for measuring apparent density used in ASTM B212
The same setup is used in ASTM B417, but a Carney funnel is used instead.¹⁷

ASTM B417 use a Hall Flowmeter funnel and a Carney funnel, respectively, to measure this. The powder sample is passed through the funnel to ensure that the particles are not compacted and are collected in a cup of known volume at the other end until it overfills. A nonmagnetic straight edge is then used to level off the cup before measuring the mass.^{17,18} The test setup can be seen in Figure 7. The process described in ASTM B329, the Scott Volumeter method, utilizes the same process, but a Scott Volumeter (comprising two funnels and a series of baffles) is used instead of the Hall Flowmeter or Carney funnels.¹⁹ Yet another measurement method available is ASTM B703, which uses an Arnold meter. An Arnold meter is a steel block with a cavity of known volume filled with a powder delivery sleeve that ensures that the cavity is completely and levelly filled. The mass of the powder in the cavity is then measured and used with the known volume to calculate the apparent density.^{5,20}

ASTM B527 describes a method for measuring the tap density of metallic powders, which is the density of a powder that has been tapped to settle contents in a container. The process consists of pouring a sample of powder of known mass into a graduated cylinder that is then raised and lowered a 3-mm stroke at a frequency between 100 and 300 times per minute in a way that does not loosen the powder at the surface. This setup is called a tapping apparatus. The test continues until the volume stops decreasing. This final volume is used in calculating the tap density.²¹

The skeletal density refers to the density of the actual particulate. ASTM B923 determines this using helium or nitrogen pycnometry.²² In the process, a powder sample of known mass is placed into a chamber of known volume. The chamber is then evacuated of air (also known as outgassing), creating a vacuum. The sample may be heated during outgassing to facilitate the removal of air from pores and eliminate any moisture. Subsequently, helium or nitrogen is allowed into the chamber until it reaches equilibrium pressure. The apparatus measures the volume of gas added to the chamber and uses the known volume of the chamber to determine the volume taken up by the particulate, which is then used to calculate the skeletal density.⁵

Chemical composition

Metal powder can consist of a pure metal, but in the case of metal alloys, the powder can be made from an atomized alloy ingot or through the mixing of constituent elemental powders. Both pure and alloy powder metals can also have impurities. The potential mechanical properties of a finished part strongly depend on the chemistry of the raw material. The chemistry of the powder can also be used to determine if it is prone to oxidation and whether it should be handled and processed in inert gas atmospheres. It can also be used to determine whether a powder poses a health hazard — i.e., is toxic, carcinogenic or mutagenic — or if reactions with other chemicals should be avoided, and to predict whether it will pose a fire or explosion hazard. For instance, UL 3400, the Outline of Investigation for Additive Manufacturing Facility Safety Management, considers the chemistry of powders used in 3D printing to address toxicity, environmental concerns related to the disposal of waste powder and exhausting of inert gases to the atmosphere, air quality, and volatile organic compounds (VOCs). This Outline also addresses health concerns including breathing hazards, skin exposure and neurological and carcinogenic effects.²³

Testing methods to determine chemical composition

Chemical composition may be determined through numerous methods. Depending on the elements involved and the desired accuracy, multiple methods may be necessary to get definitive quantitative results for a material.

X-ray fluorescence: This versatile method is nondestructive and requires no sample preparation for analyzing powder. The sample is irradiated by a beam of x-rays and some of its energy is absorbed by an electron in the innermost electron shell of the sample atoms. If enough energy is absorbed, it will cause the ejection of this electron (photoejection). An electron from an outer electron shell then drops into the vacancy created by the photoejection, which results in the emission of x-ray radiation. This proceeds for as many electron shells as a particular atom has. This phenomenon is called the photoelectric effect. The x-ray energies emitted due to this phenomenon are unique for each element. The energy of each x-ray emitted and the number of x-rays at each energy level are used to determine the composition of the sample.²⁴

The national standards for chemical analysis using XRF include ASTM E539,²⁵ E572,²⁶ E1085,²⁷ E2465²⁸, and E2626²⁹ for titanium alloys, stainless steels and alloy steels, low-alloy steels, nickel alloys, and reactive and refractory elements, respectively. Other materials can also be evaluated: The technique can detect elements from beryllium to uranium on the periodic table within the parts-per-billion range.²⁴

Inductively coupled plasma optical emission spectroscopy

(ICP-OES): Also called atomic emission spectroscopy (ICP-AES), this technique requires that the sample be dissolved into a solution, typically using an acid, and then diluted. Plasma is generated inside the apparatus by flowing an argon torch through an induction coil. A spark within the torch acts along with the induction coil to strip electrons from the argon gas, causing high-energy collisions resulting in a stream of plasma. The sample in solution is sprayed into this plasma. The plasma excites the sample into an ionized gas. Upon exiting the plasma stream, the atoms of the sample revert to a lower-energy, relaxed state by emitting light that has elementally characteristic wavelengths. The intensities and wavelengths of this emitted light are used to determine the composition of the sample.²⁴

National standards for chemical analysis using ICP-OES include ASTM E2371,³⁰ E2594³¹ and E2626²⁹ for titanium alloys, nickel alloys, and reactive and refractory elements, respectively. These standards do not cover the full capability of this method, which can detect at least 73 elements, including all metals, with typical limits in the range of 1 to 10 parts per billion. Inductively coupled plasma mass spectrometry (ICP-MS) uses the same concept with added filters and different detectors that enable it to analyze at least 82 elements with limits on the order of parts per trillion. However, this added capability would provide little to no benefit in the analysis of conventional powder metals and would come at an increased cost for equipment, upkeep and clean-room laboratory conditions to make use of the full capability of ICP-MS.^{32,33}

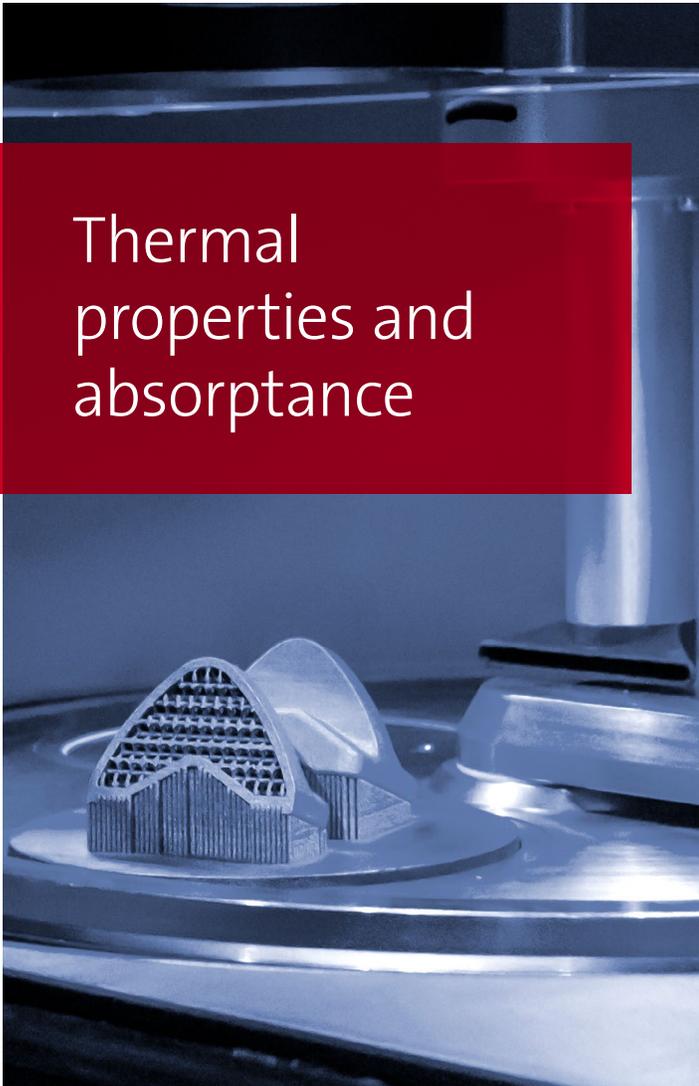
High-temperature combustion: This method can be used to determine the amounts of carbon and sulfur in a variety of materials, including most powder metals. High-temperature combustion uses an accurately measured amount of the sample placed within a ceramic crucible, which is then inserted into a high-temperature furnace. The furnace is flooded with oxygen and the temperature is raised such that the carbon and sulfur in the sample combust to form CO, CO₂ and SO₂. Infrared absorption or thermal conductivity sensors are used to analyze these gasses and determine the amount of carbon and sulfur in the sample.²⁴ ASTM E1019³⁴ describes how this method is used for steel, iron, nickel and cobalt alloys, and ASTM E1941³⁵ describes its use for refractive and refractory metals.

Inert gas fusion: This method can be used to determine the amounts of nitrogen, oxygen and hydrogen in metallic samples. Similar to the combustion method, an accurately measured amount of the sample is placed within a crucible, which is then inserted into a high-temperature furnace. However, this method requires a crucible made from pure graphite. The furnace is flooded with an inert gas that is not being analyzed, such as argon, and the temperature is raised until the sample melts. In this melted state, the hydrogen and nitrogen are released as gasses and are carried away to detectors. The oxygen in the sample disassociates and reacts with the carbon in the crucible to form CO and CO₂ gasses that are also carried away to detectors. The detectors use thermal conductivity sensors to determine the amounts of nitrogen, oxygen and hydrogen in the sample.

ASTM E1019³⁴ describes how this method is used for steel, iron, nickel and cobalt alloys. ASTM E1409³⁶ describes how it can be used to determine the oxygen and nitrogen content of titanium alloys, while a separate standard, ASTM E1447,³⁷ describes how it can be used to determine hydrogen content, though both can be performed simultaneously in the same setup. ASTM E1569³⁸ describes its use in determining the oxygen content of tantalum powder, and ASTM E2792³⁹ describes its use in determining the hydrogen content in aluminum alloys. The functionality of the inert gas fusion method extends beyond the purposes described in these standardized methods.



Thermal properties and absorptance



The majority of current additive manufacturing methods using powder metals employ some form of concentrated high energy source to melt layers of powder. The thermal properties of the powder are highly influential in the performance of this process. Significant properties include the specific heat capacity, melting temperature, enthalpy of fusion, energy absorption rate and rate of transfer of heat to the surrounding powder. The energy source in the processes needs to be strong enough and travel slowly enough to melt the layer at a temperature that allows it to adhere to the underlying layer without porosity. Yet, it must also not be too strong or travel too slowly to the point where the effective spot size of molten material is larger than desired due to the viscosity of the molten material and thermal conductance to surrounding powder. The thermal conductivity of a powder can be affected by such characteristics as chemistry, size and shape.

Size and shape affect conductivity because paths of lower resistance lead to higher thermal conductivity. Larger particles provide a path of lower resistance. This is also the case with particles with lower inter-particle friction because a layer of them will be more compact.⁴⁰ Also, rolling the powder prior to melting increases compaction and thermal conductivity.

ASTM E1269 is a standardized procedure that describes how differential scanning calorimetry (DSC) can be used to determine the specific heat capacity of the metal powders. The apparatus used, called a differential scanning calorimeter (also shortened to DSC), compares the heat energy absorbed by the sample to a reference material as the temperature in the chamber increases at a controlled rate. Samples are contained in a crucible made of a material that is stable at test temperatures, such as quartz or alumina, and testing is performed in an inert gas atmosphere.⁴¹

Melting point and solidification temperatures can be determined using the methods described in ASTM E794.⁴² The test can be used to determine the temperature at the onset of melting as well as the temperature where the sample is completely liquid. This is particularly valuable when considering alloys with different constituent metals and phases that melt at different temperatures. DSC or differential thermal analysis (DTA) can be used. DTA uses the same test conditions as DSC, but the apparatus measures the difference in temperature between the sample and reference material as the chamber temperature is increased rather than heat energy absorbed. Because of this, DTA cannot be used to determine the specific heat capacity of a material. The operating range for a typical DSC is -120 to 600°C, whereas the range of a

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typical DTA is 25°C to 1,500°C. DTA would be necessary for most metals. For example, titanium has a melting point of around 1,700°C. However, there are commercially available DSCs that have an operating range up to 2,000°C, and their use is permitted in ASTM E794.⁴² Because DSC provides useful information beyond what DTA is capable of, such as specific heat capacity, it is recommended that tests for metal powder for 3D printing use a high-temperature DSC such that a DTA would not be necessary.

Taniguchi et al.⁴³ and Sih and Barlow have demonstrated a method for determining the thermal conductivity of metal powders using a laser as a heat source. Because high-energy beams are the most common sources of heat in today's powder metal 3D printing, this method can closely approximate the conditions seen in service. This method can also be used to determine the absorptances of powders, which is the proportion of incident radiation energy absorbed (not reflected) by a powder. It should be noted that each powder may have differing amounts of reflectivity with different energy beam sources or lasers due to differences in their emitted wavelengths. The absorptances of powders have been shown to be valuable in determining the energy needed to melt the desired thickness of material to its underlying layer and the rate of melting.^{45,46} Figure 8(a) shows the design used by Taniguchi et al., which was treated as a finite slab configuration for their sample.

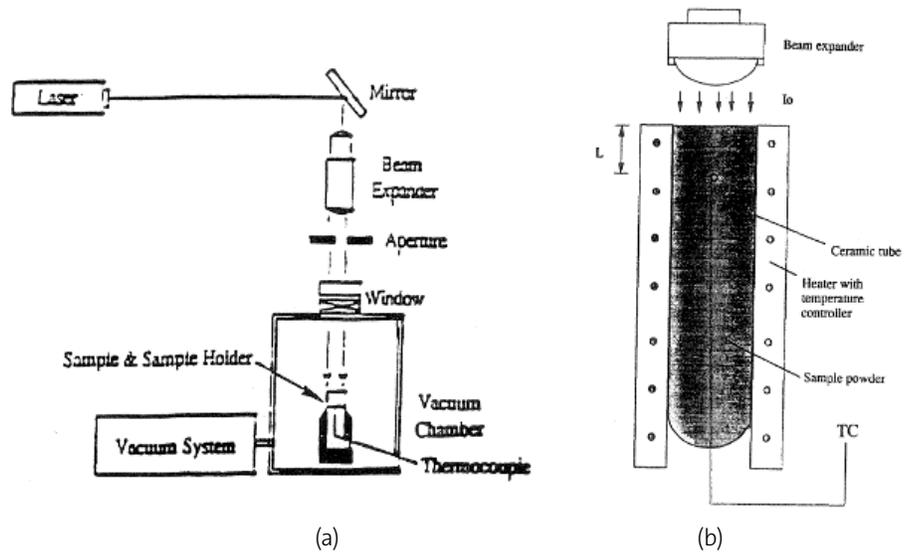


Figure 8: The finite slab (a) and semi-infinite slab (b) configurations used to determine thermal conductivity with a high-energy beam source⁴⁴

Figure 8(b) shows the semi-infinite configuration that Sih and Barlow used because it is easier to use with powder samples and simplifies calculations. The semi-infinite configuration can be approximated as one-dimensional heat flow from the surface exposed to the energy source toward the opposite side.

There is approximately no heat loss to the environment if the experiment is done at room temperature in a vacuum. The powder sample is held in a ceramic tube surrounded by a heater with a temperature controller to simulate the starting powder bed temperature and inhibit heat escaping to the sides. A thermocouple positioned a known distance below the exposed surface is used to record change in temperature as a function of time. The high-energy beam used as the heat source should be chosen to mirror what would be used in service, such as a CO₂ or Nd-YAG laser. The change in temperature versus time can be used with the Fourier relationship for semi-infinite slab/bed configurations (see Figure 9) to determine a powder's thermal diffusivity, which can then be multiplied by its bulk density and specific heat to determine its thermal conductivity. The test should then be repeated with a thin layer of material with known absorptance. The absorptance of the powder sample can then be determined using the difference in temperature rises as a function of time under both conditions.⁴⁴

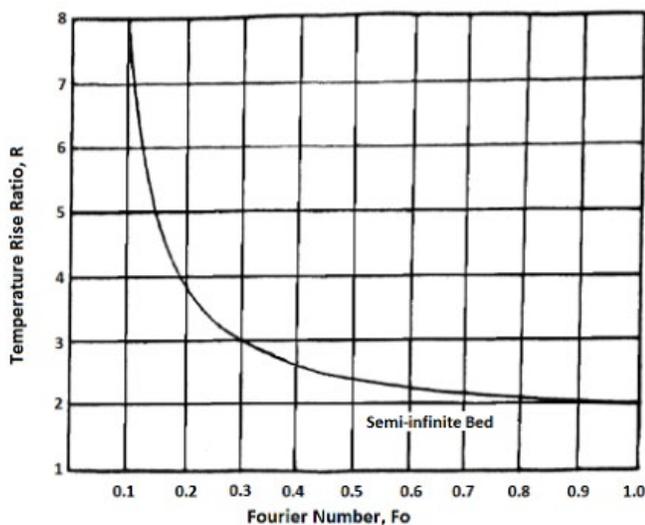
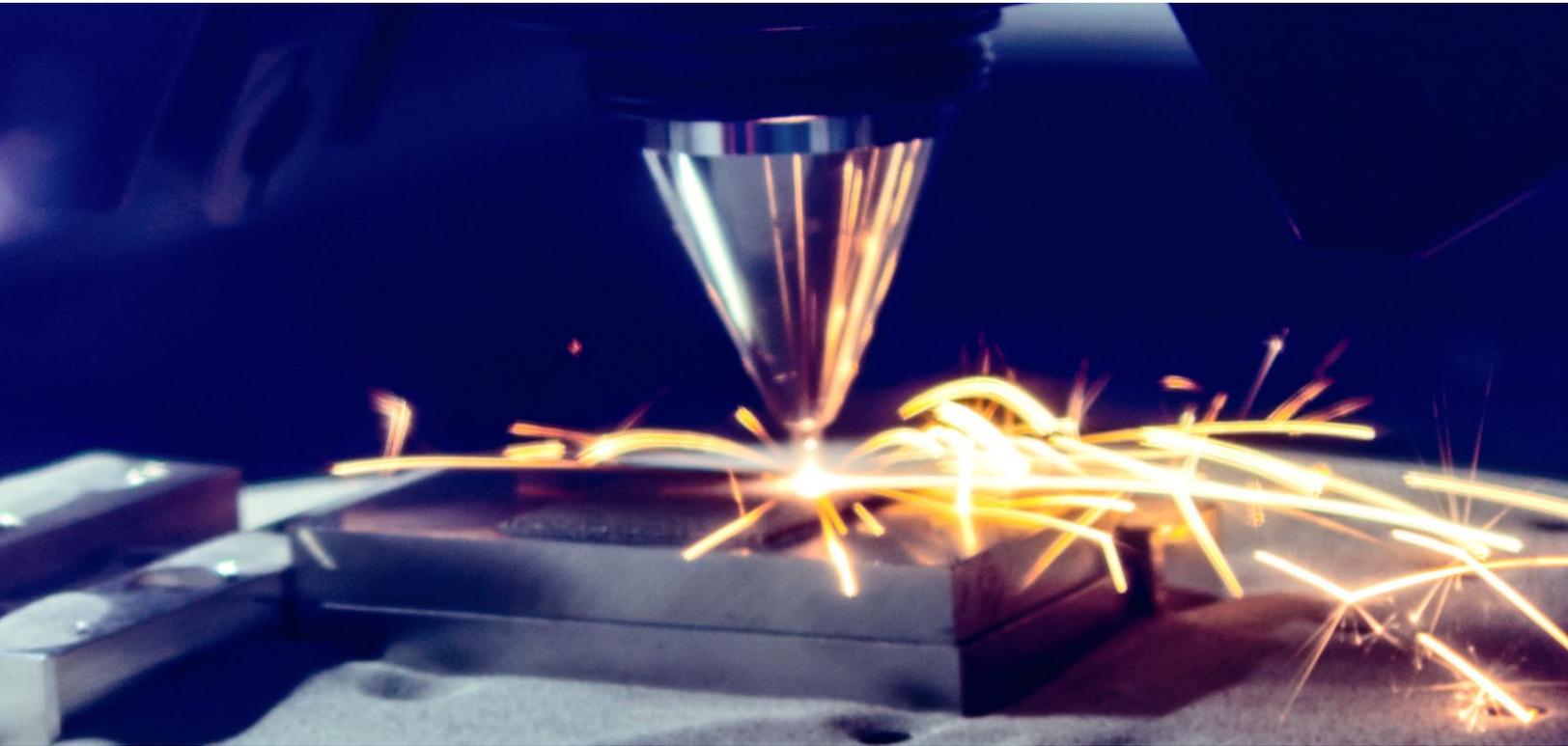


Figure 9: Temperature rise ratio (R) versus Fourier number (Fo) relationship for the semi-infinite slab configuration⁴⁴



Flammability/explosion hazards

A hazard that is sometimes overlooked is that some materials not typically flammable may become highly flammable in the form of a powder or dust. This is true of approximately 80% of organic dusts and many very fine metal powders. OSHA defines dust as particulate that has a dimension smaller than 420 μm .⁴⁷

In general, the smaller the particulate, the more likely it will become airborne and possibly ignite. Powders and dusts are particularly dangerous when airborne because ignition may lead to an explosion. For an explosion to take place, the following conditions must be met: The dust must be suspended in a gas that supports combustion, the dust must have a particle size capable of spreading a flame, the dust concentration must be in an explosive range, and an ignition source must be present that can bring dust in its immediate vicinity above its ignition temperature. After a first explosion, there is typically a second explosion because the initial explosion causes powder that had been resting on surfaces to become airborne.^{48,49} These explosions may cause serious injuries and fatalities, such as a titanium dust explosion that killed three workers in West Virginia in 2010, and a sugar dust explosion that killed 14 workers in Georgia in 2008.⁵⁰ Additive manufacturers should note an explosion

and fire at Powderpart Inc. in 2013. This company used titanium and aluminum powders, both of which are reactive and combustible, without taking safety measures such as eliminating sources of ignition and providing protection for employees. The accident caused an explosion that resulted in third-degree burns for one employee.⁵¹

To better ensure safety when working with powders that may pose a combustion hazard, it is necessary to understand and mitigate the risks. Standards exist for exactly that purpose. UL 3400 addresses topics such as proper powder storage, inert gas sensors, personal protective equipment (PPE), electrostatic discharge flooring and appropriate fire suppression systems. It also covers how to prevent explosion hazards through proper material handling, material transfer (loading/unloading the machine) and related process operations.²³

Other standards that pertain to practices when working with powders include NFPA 77⁵² for controlling static electricity to prevent fires, NFPA 654⁵³ for preventing fire and dust explosions and NFPA 704 Annex E for thermal hazard evaluation techniques.

The flammability of a metal powder depends on its composition, particle size and particle shape.

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Table 3 provides a list of some of the metals that have the potential for creating a flammable dust risk. The smaller the particle size, the more flammable a powder can be. The shape of the particles plays a factor in that fire ignites more easily on sharp edges than on round surfaces.⁵⁵

Table 3: Hazards attributed to metal powders.^{56,57,58,59}

Metal powders	Combustible with ignition source	Spontaneous ignition in air	Release hydrogen if exposed to water	Exothermic reaction with water
Aluminum	●		●	
Titanium	●		●	
Iron	●			
Copper	●			
Chromium	●			
Tin	●			
Molybdenum	●			●
Manganese	●			
Zinc	●			●
Lithium	●			
Antimony	●			
Beryllium	●		●	
Bismuth	●			
Tantalum	●			
Tellurium	●			
Tungsten		●		
Zirconium		●		
Magnesium		●	●	
Sodium		●	●	
Potassium		●	●	
Barium			●	
Calcium			●	
Cadmium			●	
Strontium			●	
Thallium			●	

Explosibility tests are valuable for helping to determine the minimum concentration and minimum amount of energy necessary to cause an explosion when working with potentially flammable powders.⁶⁰

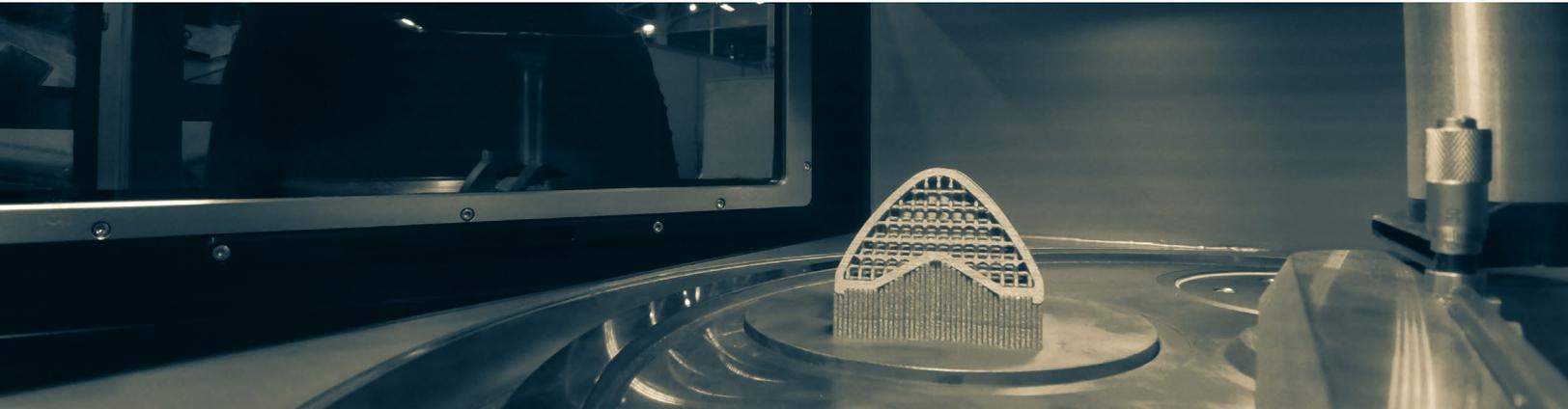
ASTM E1226 can be used to determine whether a powder can form an explosible dust cloud (meaning that it can propagate a deflagration/incident of burning) and, if so, to what degree. It assesses maximum explosion pressure and rate of pressure rise and assigns an explosibility index, K_{st} , to compare explosion severity to other dusts.

The test is performed by generating a dust cloud in an enclosed steel chamber that creates a uniform dispersion of particulate, such as a Siwek 20-L vessel. The amount of oxygen in the chamber is controlled. An ignition source at the center of the chamber is used to attempt to ignite the cloud. If an explosion is established, the pressure rise versus time is recorded for evaluation. The test procedure incorporates a go/no-go screening test to determine whether a dust or powder should be classified as explosible. It is important to note that even if a powder is found not to be explosible, this does not mean it is not combustible. A powder characterized as non-explosible as a result of this test may still smolder or burn as a dust cloud or layer and ignite surrounding materials. It also does not assess the effects of elevated temperature and moisture on the susceptibility of ignition. Additional analysis should be considered.⁶¹

The minimum explosible concentration (MEC) for a dust cloud can be evaluated using ASTM E1515. Because the MEC values attained are specific to the test conditions, such as particulate dispersion, ignition energy and propagation criteria, the values should be considered a relative measurement. These values can be used as part of a fire risk assessment that takes into account all relevant factors present in the facility and equipment where the powder is to be used. Tests per ASTM E1515 are performed in an enclosed chamber, as described in ASTM E1226. A 2,500 J or 5,000 J pyrotechnic igniter attempts to ignite varied amounts of powder that are uniformly dispersed within the chamber until the lowest amount of powder for an explosion is determined. This information is used with the volume of the chamber to calculate the particulate concentration. The maximum pressure and rate of pressure rise after ignition are used to determine whether an explosion took place. The MEC is defined as the lowest concentration where the pressure ratio (determined using pressures before and after ignition) is greater than or equal to two.⁶²

The test method described in ASTM E2019 is used to determine the minimum ignition energy (MIE) of a dust cloud in air. This is useful in identifying and eliminating possible ignition sources during processing and handling to ensure safe practices. The MIE determined from this test is the electrical energy stored in a capacitor that, when released as a high-voltage spark, is just enough to cause ignition in a dust cloud at its most easily ignitable particle concentration — a worst-case scenario. The test must be performed in an enclosed chamber as in ASTM E1226, or a glass Hartmann





tube can be used. It is important that the chamber be able to withstand any potential rise in pressure and that the interior of the chamber can be viewed during operation. The powder to be tested is dispersed in the air in the chamber and an attempt is made to ignite the dust cloud using a spark discharge between electrodes from a charged capacitor. Ignition is determined by visual observation of whether a flame propagates away from the spark gap. The procedure must be iterated, finding the minimum spark energy for a particulate concentration, then repeating at higher and lower concentrations until an MIE is found for the optimum powder concentration for ignition.⁶³

If a vacuum or inert gas is to be used to prevent combustion of metal powders, such as within the storage of metal powders and within the apparatus used for 3D printing, it is important to know the limiting oxygen concentration (LOC) where combustion is possible. ASTM E2931 gives a standardized method for determining the lowest oxygen concentration (L) that permits a flame to propagate in the optimum concentration of

particulate in a dust cloud to combust and the highest oxygen concentration (H) where a flame does not propagate. The enclosed chamber as described in ASTM E1226 is once again used for containment and powder dispersion. The optimum concentration of particulate for combustion (worst-case scenario) is found in the same manner as ASTM E2019. Ignition is attempted with a pyrotechnic igniter. It is suggested that a 2,500 J ignition source be used, but ideally, the ignition source would increase until the measured LOC is independent of ignition energy. A mixture of oxygen with an inert gas such as nitrogen or argon should be varied in the chamber until the L and H values are determined and should be no more than 1.0 volumetric percent

between one another. The LOC is the oxygen concentration in the middle of L and H. The criterion for ignition is a pressure ratio (determined using pressures before and after ignition) greater than or equal to two.⁶⁴ NFPA 69⁶⁵ provides guidelines for the practical use of the measured LOC.

Metal powders that can combust or self-heat may not be able to form an explosible dust cloud because particles will not stay suspended in air, or combustion of ignited particles is unable to propagate to adjacent suspended particles. However, these combustible powders can still pose a fire hazard. The combustibility of powders can be determined using a test such as the one found in the

The burning rate test is not required if the powder is found not to be combustible. Before testing, it is suggested that powders be checked to determine whether they are explosive or highly flammable by exposing a very small sample (1 gram or less) to an ignition source under safe conditions.

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UN Manual of Tests and Criteria, Section 33.2, Division 4.1 (also known as UN 4.1). Sometimes called the “Fire Train Test,” it is also used in NFPA 484 Chapter 4 (Determination of Combustibility) and EPA Method 1030 (Ignitibility of Solids).⁶⁸

This two-part test examines both preliminary screening and burning rate. The burning rate test is not required if the powder is found not to be combustible. Before testing, it is suggested that powders be checked to determine whether they are explosive or highly flammable by exposing a very small sample (1 gram or less) to an ignition source under safe conditions. For the preliminary screening test, an unbroken 250-mm-long line of powder with a triangular cross-section (height: 10 mm x width: 20 mm) is made using a mold on a cool, impervious, non-combustible, low heat-conducting base plate such as a ceramic tile. Testing is performed under a fume hood with an air speed of approximately 0.7 m/s to prevent fumes from escaping into the laboratory. A gas flame (such as from a Bunsen burner) with a tip temperature of at least 1,000°C and a flame diameter of at least 5 mm is applied to one end of the line of powder. For metal powder to be classified as flammable, it must ignite within 20 minutes of flame application and propagate combustion a length of 200 mm, either by smoldering or burning with an open flame, within 20 minutes of ignition.

Once a powder is classified as flammable, a burning rate test must also be conducted. The line of powder is once again formed, but this time the mold is filled loosely, then dropped onto a solid surface three times from a height of 20 mm to settle the powder before inverting to deposit the powder onto the base plate. An ignition source capable of reaching a minimum temperature of 1,000°C is used to ignite the powder at one end. Once the line has burned a distance of 80 mm, a stopwatch is started to record the time needed for the combustion to propagate another 100 mm. This process is repeated six times to determine the burning rate of the material for comparison with other substances. Powder metals are considered highly flammable if the entire length of the sample burns in 10 minutes or less.⁶⁶

The minimum temperature at which a layer of metal powder will ignite can be determined using ASTM E2021. The test apparatus consists of a hot plate, a circular metal plate positioned on top of the hot plate approximately at its center, and a metal ring placed on the approximate center of the metal plate. The metal plate, typically made of aluminum or stainless steel, has thermocouples mounted radially from its center at a depth of approximately 1 mm from its surface.



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The hot plate must be capable of heating and maintaining the temperature across the hot plate at 400°C without the test sample present. The metal ring is stainless steel and is approximately 100 mm in diameter with slots in its sides to accommodate a Type K bare thermocouple positioned at its center. The standard ring is 12.7 mm in depth, but other ring depths are acceptable, particularly if they better represent the depths relevant to the application for which the powder is intended. It is recommended that various layer thicknesses be tested to attempt to generate a trend that can be extrapolated to other powder thicknesses.

After heating the metal plate to the desired test temperature, the ring is filled with powder to slightly overflowing without compacting. A straight edge is then drawn across the top to create a level layer and the excess is removed. The temperatures at the thermocouples are monitored throughout the test. The typical test time is two hours but is continued if any self-heating is observed from a temperature rise or heavy smoke. The test is stopped if the powder has completely melted, ignited or reached a maximum temperature and is cooling down. Criteria for ignition include a measured powder temperature of at least 50°C above the hot plate temperature or when evidence of combustion such as a red glow or flame is observable.

Tests are repeated with fresh powder samples until an ignition temperature is found that is no more than 10°C above a temperature without ignition. Testing is discontinued



if a set temperature of 450°C fails to ignite the sample or the sample melts.⁶⁹ This method provides a lower, more conservative ignition temperature than ASTM E1491,⁷⁰ which is for dust clouds. The procedure in ASTM E2021 exposes the dust layer to an elevated temperature for an extended period on the order of minutes to hours. In ASTM E1491, the dust cloud is exposed only for a matter of seconds.⁶⁹

Outline of potential test capabilities for facilities to test powder metals for use in additive manufacturing

Table 4 lists the tests (ASTM standardized tests except where noted) that can be used to characterize the properties of powder metals specifically for consideration for use in 3D printing, including identifying any hazards posed by the material.

Table 4: List of tests to characterize powder metals for use in additive manufacturing, including those potentially relevant to processing parameters and hazard mitigation

Test parameter	Description	ASTM standard	Sample size	Notes
Sampling		B215		
Size determination	Sieving (45-1,000 μm)	B214	90-110 g, density ≥ 1.50 g/cm ³ , 40-60 g, density < 1.50 g/cm ³	Non-standardized methods such as image analysis may be used
	Light scatter (0.4-2,000 μm)	B821/B822	Refer to recommendations of light scatter apparatus manufacturer	

Test parameter	Description	ASTM standard	Sample size	Notes
Morphology	Light scatter and/or image analysis	B243 (terminology)	N/A	No standard describes a method to quantify particle morphology
Chemical composition	Hydrogen in Ti and Ti-alloys — inert gas fusion technique	E1447	0.15-0.30 g	Trace element tests chosen based on predominant elements in samples reported by the manufacturer
	Hydrogen in Al and Al-alloy — inert gas fusion technique	E2792	4-7 g	
	Oxygen in tantalum powder — inert gas fusion technique	E1569	0.08-0.15 g	
	Oxygen and nitrogen in Ti and Ti-alloys — inert gas fusion technique	E1409	0.100-0.150 g	
	Carbon content in refractory and reactive metals — combustion analysis	E1941	Size recommended by carbon determinator manufacturer	
	Carbon, sulfur, nitrogen and oxygen in steel, iron, nickel and cobalt alloys using combustion and fusion techniques	E1019		
	Inductively coupled plasma atomic/optical emission spectroscopy (ICP-OES)	E2371 (Ti alloy), E2594 (Ni alloy) and E2626 (reactive/ refractory)	Size recommended by spectrometer manufacturer	Pick one standard from either of these methods depending on available equipment and material being analyzed
	X-ray fluorescence (XRF)	E539 (Ti alloy), E572 (SS and alloy steel), E1085 (low-alloy steel), E2465 (Ni alloy) and E2626 (reactive/ refractory)	Size recommended by spectrometer manufacturer	
Flow characteristics	Hall Flowmeter funnel methods	B213 and B855	150 g and 20 cm ³ , respectively	Methods may not work with a significant fraction of small or irregularly shaped particles due to inconsistent and/or greatly reduced flow rates
	Carney funnel method; used only when powder will not flow through the Hall Flowmeter	B964	150.0 g for ferrous and copper-based materials, 200.0 g for tungsten-based powder; for other materials, experimentation used to determine the appropriate quantity	

Test parameter	Description	ASTM standard	Sample size	Notes
Density	Apparent density (free-flowing particles) — Hall Flowmeter funnel	B212	30-40 cm ³	Pick one. “Non-free-flowing particles” does not mean they do not flow at all; it means they do not flow freely through the orifice in the Hall Flowmeter.
	Apparent density (non-free-flowing particles) — Carney funnel	B417	30-40 cm ³	
	Apparent density — Scott volumeter	B329	60 cm ³	
	Apparent density — Arnold meter	B703	50 cm ³	
	Tap (packed) density	B527	See Table 1, ASTM B527	
	Skeletal density — helium or nitrogen pycnometry	B923	Representative of whole	Density of a solid calculated by excluding all open pores and internal void volume
Thermal properties	Differential scanning calorimetry, specific heat capacity and enthalpies of fusion	E793, E1269	Size recommended by DSC manufacturer	-
	Thermal conductivity and absorptance	(Sih and Barlow)*	Depends on selection of ceramic tube and heating apparatus	-
Explosibility	Explosibility of dust clouds	E1226	250 g/m ³ in the chamber; increases with each trial	-
	Min. explosible concentration of combustible dusts	E1515	100 g/m ³ in the chamber; varied each trial	Not necessary if found not to be combustible in ASTM 1226
	Min. ignition energy of a dust cloud in air	E2019	250-500 g/m ³ in the chamber; varied each trial	Not necessary if found not to be combustible in ASTM 1226
	Limiting oxygen concentration where combustion is possible in a dust cloud	E2931	~500 g/m ³ in the chamber per trial, same as concentration for most easily ignitable concentration in E2019	Not necessary if found not to be combustible in ASTM 1226
Flammability	Determine whether a powder is flammable — f, Fire Train Test	(UN 4.1)*	200 cm ³	Powder metal can be flammable even if found not to be combustible in dust cloud form
	Min. temperature to ignite a layer of dust	E2021	100 cm ³ per trial	Perform if found to be flammable in UN 4.1

*Not ASTM standardized tests

Summary and potential applications



Numerous properties of powder metals directly influence their performance in additive manufacturing processes. The variety of methods used to produce powder metals each result in particulate of different sizes, shapes and purity levels. Consequently, the performance of a powder metal can vary between manufacturers, even if it is described as having the same composition.

The size of the particulate used can determine the smallest attainable layer thickness and dimension of a 3D printed part. Size and shape affect how powder flows when spread as a layer, and this must be assessed to ensure uniform and consistently formed layers. These characteristics also determine the spaces between particles, which influences both effective porosity in the finished part and thermal conductivity through the powder during localized sintering or melting. Determining the bulk and tapped densities of powders

will identify how the particles lay once they are spread into a layer and whether they should be mechanically pressed prior to printing.

The thermal properties of a powder, including specific heat capacity, melting temperature, heat of fusion and thermal conductivity, can be used to determine the energy needed during heating and the speed at which the focused heat source should be moved across each layer. The energy must be high enough to melt and bond the layer to the underlying layer. However, the energy should not be too high or moved too slowly; otherwise, the melted spot size would be larger than desired due to heat conducted to surrounding particles or a low viscosity of molten material. When lasers are used, these parameters are also affected by the absorptance/reflectivity of the powder, which determines how much of the energy emitted by the focused heat source is absorbed

by the powder. The reflectivity of a powder is affected by the chemistry and surface/shape of the particulate. Different lasers emit high-energy beams of different wavelengths. As a result, the absorptance of a powder is affected by the type of laser used.

It is important to identify the potential hazards of working with a powder metal before use. Once hazards are identified, one can plan accordingly to use practices and equipment to ensure safe handling and processing conditions. Information that can be evaluated to assess the hazards associated with powder metals includes their chemistry, flammability and whether they can be explosive. Chemistry can be used to determine whether a powder presents a health hazard to employees and if it will react in an unwanted manner with other substances, but it is inadequate in determining whether a powder is flammable or explosive because particle shape and size are also influential factors. Therefore, specialized tests using various ignition sources and possible workplace/processing conditions are needed.

The tests in this paper provide information that can be used in the characterization of powders from any manufacturer for use in 3D printing. Using standardized, well-designed tests can help manufacturers identify the quality and suitability of powders and safe, appropriate printing parameters to produce consistent parts. This knowledge can be used to promote proper handling and processing conditions for safer use and to help expedite the ongoing innovation in printing equipment through more thorough technical understanding.

Learn more at [Additive Manufacturing | UL Solutions](#).

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