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THEORY, PRODUCTION TECHNOLOGY, AND PROPERTIES OF POWDERS AND FIBERS

REQUIREMENTS FOR METAL AND ALLOY POWDERS FOR 3D PRINTING (REVIEW)

O.K. Radchenko[1,2] and K.O. Gogaev[1]

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There are five 3D printing methods that use metal or alloy powders. The most promising methods are powder bed fusion, directed energy deposition, and binder jetting. General requirements for the powders and their most important characteristics (particle size and shape, powder flowability), as well as the chemical composition of nickel alloy powders from two manufacturers, are addressed. Features peculiar to the behavior of powders in use of two types of recoater (as a blade or a roller) are analyzed. It is shown that the d_{90} *size does not meet the actual requirements and* d_{max} *needs to be taken into account instead. Powders with nonspherical particles (mixtures of spherical and nonspherical particles) are known to be reused, but there are still no clear recommendations for their use. Inadequate attention is paid to the shape of powder particles. In additive manufacturing processes, powders with nonspherical particles (produced by grinding and other methods) have been already used but, in most cases, the shape indicators or their dispersion are not determined. Basic criteria for the particle shape that correlate with the powder flowability should be identified. The standard flowability value (determined by flow test) does not adequately characterize the dynamic behavior of powders, nor does it allow the powders with significantly different bulk densities and particle material to be compared, and thus requires adjustment. The most important characteristic for the processes considered is the ability of powders to form a thin flat layer in certain conditions. A new characteristic of the powder dynamic behavior has been proposed: spreadability. It includes two criteria: build plate coverage ratio and powder dynamic flow angle, each having its drawbacks. To date, there is no accepted technique for testing spreadability, nor is there an agreed indicator that would characterize it. There is only an understanding that a research method should best reproduce the powder behavior in a 3D printer in operation. Methods such as powder drum rotation (GranuDrum instrument) or long-established classification of pharmaceuticals by flowability, which was tried to be applied to metal powders, are involved. According to the classification, excellent flowability is inherent in powders having an angle of repose varying from 25 to 30 deg, Hausner ratio lower than 1.11, and Carr index lower than 5–15. The validity of this application requires thorough verification. The advantages and disadvantages of the following basic methods for producing powders of various metals and alloys used in 3D printers are addressed: gas atomization of melts in*

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[1] Frantsevich Institute for Problems of Materials Science, National Academy of Sciences of Ukraine, Kyiv, Ukraine.

[2] To whom correspondence should be addressed; e-mail: arradch@gmail.com.

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crucibles without vacuum and with vacuum melting or induction melting, plasma atomization using feedstock rods, rotation electrode gas or plasma atomization, etc. Gas atomization as a commercial method remains the most popular. Powders of greater quality made from reactive elements allow the production of new high-quality parts but also involve additional costs.

Keywords: *3D printing, metal and alloy powders, binder jetting, powder bed fusion, directed energy deposition, particle size, particle shape, flowability, recoater, spreadability.*

INTRODUCTION

Additive manufacturing (AM)[1] has become a new, rapidly growing industry allowing the fabrication of parts at low costs and in complex shapes that are not afforded by other technologies [1]. There is a great number of additive manufacturing options, each intrinsically involving 3D printing with various materials. 3D printing with metal powders is the most complex option [2, 3]. The high prime cost of the process partially restrained its development but has been scaling down [4]. 3D printing machines using metal powders and wires have entered the market. These machines incorporate advanced knowledge in metallurgy, laser technology, optics, electronics, control systems, mechanical engineering, vacuum engineering, and instrumentation [5].

The origination and development of 3D printing with metal powders dates back to more than two decades [6, 7]. Selective laser melting (SLM) is the most advanced method for 3D printing of metal powders. The first commercial printing was undertaken only in 2004 by SLM Solution GmbH, Germany [8, 9].

The development of additive manufacturing posed new challenges for the powder metallurgy, both theory and practice. Eason believes that [10] additive manufacturing brought a renaissance to powder metallurgy research. The growing industry interest in metal parts manufactured additively urged the academic and scientific world to develop new alloys, optimize process parameters and geometries of parts, and warrant the reliability of responsible parts of new generation. Unfortunately, to understand the interaction between the various phenomena associated with 3D metal printing is a challenging problem [11]. The lack of confidence in the quality of products prevents the transition of this technology from the manufacture of prototype models to the manufacture of crucial parts. The confidence can be restored through a fundamental understanding of the physics behind the process. This understanding is widely recognized to be achieved by simulation. However, wide ranges of sizes, lengths, and temperatures for the process pose certain issues for computations [12]. Much effort has been already made to understand the processes accompanying 3D printing [2, 3, 13]. An array of accumulated experimental data would allow the development of standards for some processes [14]. Nevertheless, there is still no full understanding of the interrelation between the properties possessed by the powder and the part produced [15, 16]. There are still no requirements for some powder characteristics. This can be explained by the lack of ultimate agreement on what methods for determining the powder characteristics would be most acceptable in the context of additive manufacturing.

This review is intended to find scientifically grounded values for parameters of metal and alloy powders that would be used in additive manufacturing processes and ensure that the products are of appropriate quality.

3D PRINTING PROCESSES THAT MAY USE METAL AND ALLOY POWDERS

There are seven categories of additive manufacturing processes determined by the ASTM F2792-12a Standard, but only the following five categories can be used for the printing of metal powders [5, 6]:

1. Material extrusion (ME).
2. Material jetting (MJ).
3. Binder jetting (BJ).
4. Powder bed fusion (PBF).
5. Directed energy deposition (DED).

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[1] The term approved by ASTM F2792-12a Standard Terminology for Additive Manufacturing.

All these categories come from the names of 3D printing processes and are thus called '3D printing processes' throughout the paper. All these processes are commonly controlled by computers with software that includes a model developed to describe a part as multiple layers and set operating conditions for all 3D printer components (build plate, powder feeder, recoater, laser, etc.).

Process one (ME) is as follows. The heated powder mixture with a plasticizing binder is extruded layer by layer through a nozzle onto a platform to build a green part [5, 17] to be then dried and sintered. The drawback of this method is that it can produce only rough parts of simple shape. The fabrication process includes several stages and results in a porous item that can be further subjected to infiltration with another metal or nonmetal.

Process two (MJ) involves selective jetting (onto specific platform areas) of the powder mixture with a binder but has not found wide application with metal powders[2] because of high rheological requirements for the material to be jetted. Spray deposition and spray forming of semi-solid metal and alloy droplets [18] are methods that are closest to MJ, but they do not yet belong to additive manufacturing processes.

Process three (BJ) is as follows. A powder layer is first spread onto the platform and a binder adhesive is deposited onto the required areas and then the next powder layer is deposited and the adhesive is jetted once again. The process is repeated until the entire part has been built. The part solidifies together with the powder in the build box. Then the build box is removed from the machine and the powder is blown with compressed air in a protective atmosphere. Some parts can be used in green form, some are subjected to infiltration, and some are sintered. This method allows more complex and precise parts to be manufactured [19]. The 316L stainless-steel powders used with this method have particle sizes $d_{10} = 3.7$, $d_{50} = 9$, and $d_{90} = 21.4$ μm [20].

Process four (PBF) [21] includes melting and fusion of a powder layer preliminarily deposited onto the build platform. This process has found the widest application with some variations. Selective laser sintering (SLS) or otherwise called direct metal laser sintering (DMLS) was developed first [22]. This method was used to make parts with varying porosities (commonly from 30 to 50%). Further infiltration was required to produce compact parts. This method was gradually replaced by selective laser melting (SLM) [23], allowing the buildup of parts in virtually any geometries and properties close to those possessed by cast products. A powder layer 20 to 80 μm thick is first deposited on the platform with both methods. After the required powder regions are melted with a laser or electron beam (electron beam melting (EBM) [24]), the platform lowers accordingly in a rectangular box and then a new powder layer is deposited, spread with a roller or a blade (the importance of these operations is shown below), and melted and fused. The procedure is repeated until a part has been produced. As of 2019, the PBF products constituted 54% of the market value in the manufacture of metal additive parts.

Process five (DED) [25] adds the material layer by layer onto the platform or a part being repaired through a nozzle mounted on a multi-axis (4 to 5) arm. The metal powder[3] is fed to the nozzle and is melted upon deposition with a laser, electron beam, or plasma arc. The procedure is repeated until a part or a coating layer has been produced. In the case of electron-beam systems, the process is performed in a vacuum chamber to prevent the electrons from interacting with air molecules. Laser-based systems require a fully inert chamber for work with reactive metals and alloys to prevent oxidation. The process is disadvantageous in that the material is unevenly distributed when deposited: more material accumulates at the center than on the periphery. For metals, almost any metal that is weldable (titanium and titanium alloys, Inconel, tantalum, tungsten, niobium, stainless steel, aluminum, etc.) can be 3D printed with DED. The powder particle sizes are approximately 50 μm (laser) and 150 μm (electron beam and plasma) [26]. The DED processes also include laser engineered net shaping (LENS) [27], which is an

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[2] Nanoparticle jetting technology (NPJ) has been developed. This proprietary material-jetting technology from XJet jets a liquid that contains nanoparticles. The liquid is loaded into the printer via cartridge and is jetted onto the plate with extremely thin layers to build up a part. The liquid evaporates under the action of high temperatures, leaving parts fabricated from the required material. This technology is acceptable for metals and ceramics, but there are no data on its commercial use.

[3] This process may use metal wire instead of powder (as, for example, in Sciaky Inc.'s electron beam additive manufacturing (EBAM)).

additive manufacturing technique developed to make metal parts directly from computer-aided design solid models [28].

Nowadays, the technology for fabricating each part is tested separately with a specific 3D printer and specific powder (whose characteristics should be ensured to be stable by the manufacturer and guaranteed by the supplier). The transition to another printer or powder does not ensure the same quality of parts. The powder layer thickness, laser type and power, exposure time at one point, laser path, and a computer model that may eliminate or reduce machine drawbacks are extremely important for a 3D printer [29, 30]. The 3D printers used in the above processes are described in detail in [5, 17] and the manufacturers' promotional material.

METHODS OF PRODUCING METAL AND ALLOY POWDERS USED IN 3D PRINTERS

Metal powders used in 3D printers [31] are produced by many methods. Below is an overview of the basic methods.

The most widespread method is *gas atomization of liquid metal* without preliminary evacuation of the melting chamber [32]. This method is used to produce powders of nonreactive metals and is described in sufficient detail in [33]. The gas atomization process has three stages: initial, operating, and final. The powder reaches stable properties only at the second stage and is not of required quality at the first and third stages. For this reason, greater length of the second stage increases the amount of appropriate-quality powder. The process is disadvantageous in that the powder has high gas impurity content (O_2, N_2, etc.).

Vacuum melting is used to produce powders with lower impurity content (with $d_{50} = 10–40$ μm). With this method, the melting chamber is evacuated to minimize contact of the melt with oxygen and nitrogen. The process is called *vacuum induction melting and gas atomization* (VIGA) [34]. This process is employed to produce high-temperature creep-resistant nickel alloys (Inconel, Rene, etc.) to make parts of aviation and stationary turbines, cobalt alloys to make medical instruments and implants in dentistry, and high-alloy steels (for example, tool and high-speed cutting steels) with great carbide content.

To prevent the liquid metal from contacting with the crucible, *electrode induction guide inert gas atomization* (EIGA) is used, like vacuum induction melting [35]. This process produces highly reactive metals, Ti, Zr, Hf, Gd, and Cr, and associated alloys, TiAl and FeGd. Induction heating melts feedstock rods. The metal droplets enter the nozzles and are atomized with inert gas. The EIGA equipment allows several kilograms to tens kilograms of metal to be atomized within a single melting cycle.

To produce high-quality spherical powders from Mo, Ti, Ni, Ta, and Co–Cr alloys, *plasma atomization* (PA) is employed [36, 37]. This process produces particles of required size and narrow grain-size distribution. The process is disadvantageous in that rods 1–5 mm in diameter need to be preliminarily made and its effectiveness is low.

There is atomization with gas dissolved in the melt [5]. Melts that can dissolve gas (hydrogen, helium, nitrogen) are used in this process. The process is called *soluble gas atomization* (SGA) and is also known as vacuum atomization. The atomizer consists of two chambers: one is to melt the metal and create excess gas pressure for greater gas dissolution and the other, vacuum chamber located above is to accept the metal moved there under excess pressure. The gas dissolved in the metal breaks the jet into droplets that spheroidize, cool down, and fall into the powder receiver. There are no data on the effectiveness of this process or the stability of particle sizes.

Centrifugal atomization has two varieties. One is rotation electrode process (REP) for atomizing the melt formed when an arc passes between the feedstock rod and tungsten electrode. The rod rotates at a high speed. The melt is atomized under a high-speed ionized inert gas flow [38]. The method produces especially pure fine powders, for example, such as VT-6 titanium alloy powders [39]. The other is plasma rotation electrode process (PREP), which differs from REP in that plasma is used instead of ionized inert gas. This method produces high-quality powders from reactive metals and alloys with almost perfect particles. Spherical Ti, FeAl, TiAl [40], and VT20 [41] powders were prepared with this method. The production of spherical titanium alloy powders is an individual problem described in the review [42].

Methods involving additional powder processing constitute a large group. Spherical VT-6 alloy powders were produced by radiofrequency plasma spheroidization of nonspherical powders. With appropriate process plasma parameters, the spheroidized powders had almost 100% spherical particles, smooth surface, favorable dispersion, and narrow particle-size distribution. The average particle size of the spheroidized powders was somewhat larger than that of the starting powders. In addition, the spheroidized powders showed greater bulk density and improved flow properties (including dynamic ones) [43, 44].

Spherical metallic powders were also produced by high-temperature remelting spheroidization (HRS). When flied through the induction furnace hot area, nonspherical particles melted and became spherical [45]. Spheroidized copper powders were prepared by this method.

The Equispheres Company[4] (Canada) produced AlSi10Mg aluminum alloy powders with spherical particles of equal size. This increased the laser power and reduced the production time. The powder production process is not specified.

The ATO Lab Company (Poland) designed an installation in 2018 to produce small amounts of powders (hundreds of grams per cycle) from different metals and alloys, including reactive metals (titanium, magnesium, aluminum, etc.) with narrow grain-size distribution, ranging from 20 to 100 μm [46]. The process details are not described. The installation is advantageous in that it is compact and mobile and suitable for office applications. However, electrodes from an appropriate metal need to be made first and the process has low effectiveness (though the authors do not expect commercial production but recommend the installation for research institutions).

Near-spherical powders are also produced by *water atomization* [47–50]. They can be used in the AM technology if they have appropriate chemical composition and low surface contamination (resulting from additional processing).

Melt atomization processes are most effective among all the techniques described and produce approximately 90% of the AM powders [5].

POWDERS FOR AM

There are numerous metal and alloy powders used for 3D printing [5, 16, 31, 51]. The manufactures of 3D printers commonly propose powders that they purchase from well-known powder producers [52]. Advice was developed on how to better choose a powder supplier [53, 54]. A specific powder production technique is recommended for a specific process. Hence, gas or plasma atomized powders are recommended for the SLM and EBM processes. Centrifugal, gas, or plasma atomized powders are recommended for the LMD and DED processes. Some companies produce powders suitable for several processes. The LPW powders (Great Britain) were optimized for 3D printers of most famous companies (EOS, Conzept Laser, Phoenix, Renishaw, SLM Solutions, Realizer, Optomec, DM3D, Trumpf, etc.). The powders are generally grouped according to the base metal: Al, Co, Ni, Ti, W, or Fe. The powders are supplied with a quality certificate, indicating the powder chemical composition and properties. The following characteristics are determined for the powders[5]: flowability, angle of repose, apparent density, tap density, particle size (laser diffraction), sieve (granulometry) analysis data, moisture content (thermal gravimetry data), and particle morphology and porosity (optical or electron microscope).

Consider powders for 3D printers such as nickel-based powders produced by LPW (Great Britain) [55] and Höganäs (Sweden) [56, 57]. Table 1 summarizes the chemical composition of the powders.

Neither Höganäs (VIGA process) nor LPW specifies oxygen content of the nickel alloy powders. Only does Höganäs provide standard properties of the Inconel 625 powder (particle size, flowability, and apparent density) [52]. The promotional material indicates that the powders are intended for the PBF process; for the SLM process,

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[4] Press release: Equispheres and TRUMPF team up to improve production speed for additive manufacturing. https://equispheres.com/ trumpf_additive_manufacturing_partnership/

[5] Most of these characteristics have been standardized in Ukraine.

TABLE 1. Content of Chemical Elements in 3D Printing Nickel Alloy Powders Produced by Höganäs (Sweden) under the AMPERPRINT Trade Name and LPW (Great Britain) under the LPW Trade Name

Company grade	International grade	Chemical composition, wt.%						
		Cr	Co	Al	Mo	Nb	C	Others
Höganäs (Sweden)								
0151		16	8.5	3.5	1.75	0.9	0.1	3.4 Ti, 2.6 W, 1.7 Ta, 0.02 Zr, 0.01 B
0153	Inconel 625	21	–	–	9	4	<0.01	
0152		22	19	2	–	1	<0.2	4 Ti, 1.9 W, 1.4 Ta
0181	Inconel 718	19	–	0.5	3.1	5.2	0.04	1 Ti
0211		22	–	0.35	2	–	0.07	0.5 Si, 0.6 Mn
0228		22	1.5	–	9	–	0.07	18 Fe
LPW (Great Britain)								
625	Inconel 625	20.0–23.0	1.0	0.4	8.0–10.0	3.15–4.15	0.1	0.4* Ti, 5.0 Fe, 0.05 Ta, 0.50 Mg, 0.50 Mn, 0.30 Cu, 0.50 Si, 0.015 S, 0.015 P
718	Inconel 718	17.0–21.0	1.0	0.3–0.7	2.8–3.3	4.75–5.5 (Nb + Ta)	0.02–0.08	0.75–1.15 Ti, 15.0–21.0 Fe, 0.01 Mg, 0.35 Mn, 0.30 Cu, 0.35 Si, 0.015 S, 0.015 P, 0.01 Ca, 0.005 Se, 0.006 B
247LC		7.50–8.50	8.5–9.5	5.4–5.8	0.4–0.6	–	0.04–0.08	0.60 Ti, 9.5–10.5 W, 3.0–3.5 Ta, 0.004 Mg, 0.05 Fe, 1.20–1.6 Hf, 0.05 Si, 0.003 S, 0.006–0.015 Zr, 0.0010–0.018 B, 0.015 P
X-2LC		20.5–23.0	1.5–2.5	–	8.0–10.0	0.25	0.05–0.08	0.60–1.0 W, 0.25 V, 0.2 Cu, 17.0–20.0 Fe, 0.25 Hf, 0.1 Si, 0.015 S, 0.010 B, 0.015 P
WASP		18.00–21.00	12.00–15.00	1.20–1.60	3.50–5.00	–	0.02–0.10	2.75–3.50 Ti, 2.00 Fe, 0.10 Mn, 0.003–0.010 B, 0.10 Si, 0.010 S, 0.010 P
263		19.00–21.00	19.00–21.00	0.30–0.60	5.60–6.10	–	0.04–0.08	1.90–2.40 Ti, 0.70 Fe, 0.60 Mn, 0.40 Si, 0.007 S, 0.015 P

*If one number is indicated, it corresponds to the maximum value.

the powders are to be sieved to have 15–45 μm particles (there may be maximum 5 wt.% oversize or undersize). The sifting through such fine sieves is time- and labor-consuming, and the sieves often fail. For the DED process, powders with 45 to 90 μm particles are sieved (there may be maximum 3 wt.% oversize and maximum 0.2 wt.% undersize). The Hall flowability should be 15–18 sec/50 g.

REQUIREMENTS FOR POWDERS

The development of uniform requirements for metal powders suitable for 3D printing is challenging because it requires that the mutual impact of individual properties of powders [14] and the variety and parameters of 3D printers for different metals [5, 17] are considered. Therefore, a single powder for each printer cannot be proposed. It is only possible to identify some common features for specific types of machines classified into different groups and only after that establish the requirements for powders. The paper [5] also states that there are no general requirements for metal and alloy powders used for 3D printing.

In the proposed review, the properties of powders are addressed in relation to the group of methods that are categorized as PBF (printers used in SLM, SLS, and EBM processes), DED, and BJ [59].

The requirements established for this group are quite general. In accordance with EMPA requirements[6], metal powders should have:

- spherical shape to ensure good flow and coating ability;
- particle size usually below 50 μm or below 150 μm[7] depending on machine type and surface finish required;
- particle size distribution tailored to the application and properties of the final product;
- controlled chemical composition and gas content.

In addition, it is indicated [60] that the powder properties such as flowability, particle size, chemical composition, and spherical shape should be standardized for typical grades. To date, only requirements for the VT6 alloy powder have been standardized [61]. The standard establishes the powder production method and properties of the AM material. To ensure high effectiveness of the AM process and stable properties of metal powders used in complex cases, powder rheometry is recommended [62].

The paper [14] overviews metal powders used in the PBF process. The powder quality was found to be key to the final properties of parts fabricated. The powder flowability, grain-size distribution, shear test, and powder properties at elevated temperatures and in reuse were addressed in the paper. The properties of 17-4SS stainless-steel and nickel/cobalt alloy powders used for direct laser metal sintering (DLMS [21]) were analyzed. The powders were produced by gas atomization. The particle density of the powders was shown to be determined by helium pycnometry and the particle size by laser diffraction and X-ray computed tomography accompanied by spherical harmonic analysis. The phase composition was studied with X-ray diffraction. Scanning electron microscopy with energy-dispersive elemental analysis and X-ray photospectroscopy was employed. It was emphasized that the PBF powder could not be assessed comprehensively only with standard procedures (to determine the tap density, apparent density, particle size distribution, and flowability).

For more accurate identification of powders, a combination of different methods for determining their characteristics may be proposed or a new, improved method for assessing whether the powder is suitable for printing with the above processes may be developed.

There are attempts to automate (computerize) the requirements for powders [63–65]. Hence, a method for computer identification of properties possessed by titanium alloy powders for additive manufacturing was developed [63]. The paper analyzed the titanium alloy powders and showed that they could be divided into four classes by their characteristics: 1) powders with perfect properties, 2) powders with optimal properties, 3) powders that may cause defects in material, and 4) powders that may result in defective material.

Besides requirements for powders, there are requirements for the powder feeder (recoater).

REQUIREMENTS FOR RECOATERS AND ASSOCIATED REQUIREMENTS FOR POWDERS

The recoater distributes a thin powder layer on the build plate and then all subsequent layers and consists of a blade or roller mechanism, linear rails for its movement, and a powder supply platform. The powder is sometimes on another platform and the recoater moves it to the build plate to manufacture a part [66].

To improve the powder layer homogeneity, the ratio between the blade shape and speed is proposed to be controlled and the rounded shape of the lower blade part is considered appropriate [67]. Moreover, coarser particles may be jammed when a hard blade is used [68, 69] and disturb the powder layer surface.

Rollers may form a denser and smoother powder layer than blades do. Note that a roller is counter-rotating with respect to its translational movement. The roller systems have weaker adhesion effects than those observed with the blade systems. A roller may better spread a powder layer than a hard blade because they differently distribute the particle contact forces and speeds in the powder bed [70]. The effect of particle elongation ratio on the recoater performance and powder layer quality was studied [71]. Particles of different elongation were simulated as

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[6] Additive Manufacturing (https://www.epma.com/powder-metallurgy-process).

[7] For Arcam electron-beam melting (EBM) machines (Sweden).

two, three, or four sintered particles located in one direction. Elongated particles in a powder layer demonstrated segregation. Greater particle elongation ratios or higher recoater translational speeds were found to decrease the powder layer quality, causing greater surface roughness[8] and smaller powder volume content per layer. The surface roughness increases monotonically with the elongation ratio. Nevertheless, the powder volume content per layer is maximum if the elongation ratio is 1.5; particles with a higher elongation ratio tend to accumulate in the layer upper part. To improve the powder layer quality, double smoothing is proposed [72] though it significantly reduces the 3D printer speed.

PARTICLE SIZE

The particle size is of primary importance for AM since all powder particles must be smaller than the powder layer height. The quality of powder layers, particularly their packing density and surface uniformity, are critical factors that influence the quality of parts manufactured.

The average particle diameter, d_{50}, is a parameter that characterizes the powder particle size. The Phenix machines (3D Systems) [5] use powders with d_{50} = 10 μm, Conzept Laser machines may use powders with a particle size ranging from 25 to 52 μm at d_{50} = 26.9 μm, Arcam machines operate with powders with particle sizes from 45 to 100 μm, and powders for SLM Solutions machines have d_{50} that may vary from 10 to 30 μm.

However, the d_{50} characteristic alone is not sufficient, the upper and lower distribution limits and the size distribution itself are required. Generally, d_{90} and d_{10} are indicated, though they do not suggest what would be the $d_{\max}$ value associated with the powder layer thickness. Thus, the VT6 powder model gives $d_{\max}$ = 50 μm at d_{90} = = 44 μm. The differential and integral size distribution [73] permits $d_{\max}$ to be established. The lower limit is correlated with the laser power and exposure time to avoid the spattering of small particles and the increase in porosity and also to direct a gas flow to remove the spatter from the buildup area. The upper limit is correlated with the powder layer thickness. If this thickness is exceeded, the previous layer would be disturbed and the part would be of lower quality [69]. The particle size distribution should be chosen appropriately to promote the densest particle packing in a powder layer [74]. Dense packing is important not only because it reduces porosity but also because it improves the thermal conductivity in the powder layer and increases the absorption of laser energy [75].

Analysis of the effect exerted by a greater number of particles in the powder layer on the packing quality showed that there was a very small difference between the cases when the layer thickness (t_0) was three and four maximum particle diameters ($d_{\max}$). Hence, the layer thickness can be optimally accepted to be equal to $3d_{\max}$ and the maximum diameter of one particle to be equal to $t_0/3$ [76].

A discrete-element computer model was developed in [77] to study the critical effect of powder cohesion and particle size on the powder recoating in AM. The VT6 alloy powder was chosen for the study. At an average particle size of 17 μm, the cohesion forces were found to be two orders of magnitude greater than the forces of gravity, which resulted in lower quality of the powder layer. The effect of cohesion forces is especially noticeable for spherical powders of light metals and alloys (van der Waals forces) and nonspherical powders (interparticle adhesion) [78, 79]. The accurate determination of dimensional parameters for fine powders remains an individual problem [80].

PARTICLE SHAPE

Although most descriptions of requirements for powders declare that the particles should be spherical, many particles are nonspherical in actual conditions, especially when the powders are reused, which is evidenced by associated photographs [81–84]. The quantitative indicators of particle shape are rarely used, and only elongation [71] and sphericity [85] or, its flat analogue, roundedness are in use among many shape factors. However, this is not enough. As a rule, a particle shape indicator alone is determined, but it is important to know the particle shape

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[8] The powder layer surface roughness in 3D printing needs to be additionally defined and described (a layer whose thickness constitutes several maximum particle diameters is concerned).

distribution and one of its average values (weighted average, median, arithmetic mean, etc. [86]) and dispersion. For this purpose, there are indicators for flat or shadow images of a particle taken when the particles are illuminated. Today accurate shape indicators can be determined by processing a dynamic image of a particle flow (for example, with a Bettersizer S3 plus analyzer). The correct choice of particles for analysis and their number in the sample are important. But the most important is to choose a particle shape criterion. A standardized criterion will be the best choice; i.e., it will be equal to 1 for spherical powders. Then the particle shape distribution factor needs to be established and analyzed [87, 88]. It should be also determined how the particle shape changes with variation in particle size. Such aspects were addressed when the properties of metal powders were studied. Thus, when the effect exerted by particle shape of the gas-atomized high-speed steel powder on its properties was studied, four standardized shape indicators were applied: roundness, compactness, irregularity, and elongation [89]. They were determined on shadow outlines of particles with metallographic image processing software. The average value of the four factors was found to be stabilized with a sample size ranging from 60 to 100 particles for electrolytic nickel and reduced iron powders. The following tendency was revealed: the more complex and more scattered the particle shape was, the greater was the sample to be processed.

In reuse of powder containing nonspherical particles, its proportion in a mixture with the starting powder may differ and is determined individually in each case [15]; such data sometimes are knowhow of the 3D printer manufacturer.

The papers [90–93] demonstrate that nonspherical powders, particularly titanium powders, can be used in additive manufacturing and believe that this would make the production less expensive [94]. The process for producing nonspherical titanium powders has been tested. Powders of irregular shape are shown [95] to be packed better (a rotating roller was used as a recoater). Test samples were built up layer by layer. High-density model samples without pores were made of nonspherical titanium powders [96]. There is still no quantitative estimate of the particle shape factor for the powders used, nor are there other characteristics important for additive manufacturing. It is not clear what 3D printer was used. Nonspherical particles are characteristic of composite materials whose mixtures are produced by grinding. Thus, 99.5% density samples of nonspherical Ti–TiB_2 composite powders were printed by SLM [97]. On the other hand, the disadvantages of nonspherical powders remain obvious. The flow of such powders is complicated because the particles mechanically adhere to each other. There is a high probability that inhomogeneous powder layers with different packing densities would form, which may cause porosity or incomplete fusion [98]. When nonspherical polymer particles are applied as a thin layer, they collect into clusters [99]. Metal nonspherical powders may be subjected to clustering as well.

There is an understanding that the particle shape plays an important role [100] and particle shape control can help avoid defects in 3D printing.

METAL POWDER FLOWABILITY

The adequate powder flowability, along with appropriate particle size and shape, belongs to key parameters in applying thin powder layers. The flowability of metal powders depends on many factors (powder parameters), the coefficient of friction between individual particles (or powder internal friction coefficient) being the main factor [101]. The flowability of powders deteriorates with the presence of moisture, greater specific surface area, higher particle roughness, more complicated particle shape, and greater fine content [73]. The powder flowability can be improved by drying and rolling the particles and optimizing the particle-size distribution. The standard[9] flowability determination (flow test) was borrowed from the rheology of liquids (as suggested by the test name). The method involves measuring the time of pouring 50 g of powder through a stainless-steel Hall flowmeter[10] with a polished

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[9] ISO/ASTM 52907-2019 *Additive Manufacturing—Feedstock Materials—Method to Characterize Metal Powder* recommends four methods to determine the powder flowability: flowability measurement with a calibrated Hall funnel (ISO 4490:2018), Carney funnel (ASTM B964-16), and Gustavsson funnel (ISO 13517:2020) and repose angle measurement (ISO 4324-1977).

[10] The method was developed for testing iron powders of intermediate particle size.

surface, a 60° angle of opening, and a hole 2.5 mm in diameter [102]. According to the Höganäs promotional material [52], the flowability of Amperprint 0153 powders found with a Hall funnel is 15–18 sec/50 g, but it is unknown whether this value is optimal or how it is associated with the quality of parts. The paper [103], studying the titanium powder, concludes that the flowability found with a Hall funnel does not allow establishing the difference between powders in their application in the EBM process. The paper [104] points out that flow through the funnel opening depends on powder aeration. In this regard, the paper [104] concludes that the standard powder flow determination is only a simple comparative test that does not provide a quantitative flow estimate. To confirm this conclusion, we can state that ground ferrosilicon powders with an average particle size of 180, 130, and 81 μm had 12.81 ± 0.08, 13.16 ± 0.14, and 19.29 ± 0.71 sec/50 g flowability [105], but their suitability for the PBF process cannot be determined. Moreover, the flow time of particles increases with decrease in their average size; i.e., their flowability reduces. The powders with particles smaller than 10 μm are not capable of flowing at all [73]. The dynamic properties of such powders are characterized with an angle of fall, which can be accurately calculated with an upgraded device for determining the angle of repose [106]. In general, the powder flow time is not suitable for comparing powders with significantly different apparent densities and particle densities because their flow rate will be different at the same flow time.

The powder flowability was studied in different conditions of depositing a powder layer [107, 108]. The closer the testing method to the actual process, the more accurate is the result. In depositing a layer of specific thickness, the powder behavior was simulated with different models [65, 66]. The methods for determining the flowability and establishing its connection with the quality of parts produced by PBF are described in greater detail in [15]. A number of researchers [109, 110] apply the flowability classification for pharmaceuticals to metal powders. According to this classification, excellent flowability is shown by powders with an angle of repose ranging from 25 to 30°, Hausner ratio from 1.00 to 1.11, and Carr index from 5 to 15%. However, this direct correlation may turn to be insufficiently correct.

POWDER SPREADABILITY

Depositing a thin homogeneous powder layer of the same thickness (from 1 to 3 maximum particle diameters) with high particle packing density (as is the case for the PBF, DED, and BJ processes) at a relatively high speed (80–100 mm/sec) onto a large area is a new problem that is still to be solved by powder metallurgy and requires much effort and knowledge.

In 2018, the notion of powder spreadability was proposed by Snow [111] to be introduced and started to be used extensively [112–117]. This notion was intended to characterize the powder dynamic behavior. Snow developed a spreadability tester to simulate the deposition of a powder layer in the PBF process and determine seven metrics with change in the layer height, recoating speed, recoater blade material, and powder quality. The statistical angle of repose was chosen as the powder quality indicator. The statistical significance of each metric was found with the analysis of variance (ANOVA). Two samples of gas-atomized Al–Si–Mg alloy powder applied in the PBF process were used. Two spreadability metrics were finally selected: the percentage of the build plate covered by the spread powder and the average avalanching angle of the powder (dynamic angle of repose). Nevertheless, the paper [118] established that the average avalanching angle showed poor agreement with the experimental data.

The developers of the GranuDrum[11] instrument [119], conversely, propose the avalanching angle for assessing the powder suitability for the PBF process, relying on the findings reported in [120]. The GranuDrum

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[11] The GranuDrum instrument has the following main components: a horizontal cylinder (drum) with transparent sidewalls, a drive mechanism for controlling the drum velocity, a video camera, and a computer for image processing. The drum rotates at an angular velocity ranging from 2 to 60 rpm. The cylinder inner space is 85 mmm in diameter and 20 mm in height. This space is half-filled with the powder (by approximately 50 ml). When the drum rotates, a camera installed opposite the transparent sidewall takes snapshots (30 to 100 images separated by 1 sec) to be computer-processed to calculate the average flowing angle and deviation from the average value. The drum test was known earlier

instrument allows determining the avalanching angle and its scatter as a function of the drum velocity and determining the average avalanching angle and its scatter (associated with the dynamic cohesion index). Konala also proposes [115] that the avalanching angle is accepted as the spreadability indicator and points out that powder spreadability depends on the powder chemical nature, testing conditions, particle shape, and other factors. Dependences were established between the avalanching angle and moisture content, particle-size distribution, and oxygen content. The average avalanching angle still has not found wide application as the spreadability indicator. There are good reasons for this. Increase in the drum velocity commonly leads to greater avalanching angles [121] (under the centrifugal force that is directly proportional to the squared rotation velocity) and dynamic cohesion indicator. Hence, it is not clear what drum velocity is to be used to determine the avalanching angle. The avalanching angle is influenced by not only the rotation velocity but also by the coefficient of powder friction against the inner drum walls and transparent sidewalls. The results obtained for different drums can thus differ. The avalanching angle determination has not been standardized.

In the paper [116], focusing on the processing of titanium powder to improve its properties, the powder spreadability was assessed with the powder flowability and Hausner ratio.

To reduce the time and cost for spreadability research, discrete element method (DEM) models [70] were used: in particular, the discrete particle model [100] and rainfall model [101]. More specific results were obtained by simulating the powder recoating process. Requirements for powders in the models emerge from conditions (parameters) of the PBF process [71, 75, 122–128]. The main requirement for the powder is to deposit a high-quality powder layer 20–40 μm thick. The packing density in cohesive powders is known to reduce with increasing cohesion forces between the particles that prevent their gravitational settling [75]. Moreover, the packing density increases with greater layer thickness since the particles can be arranged more optimally and the relative influence of (negative) boundary effects in the lower and upper parts of the powder layer weakens. The powder layer thickness in the range close to the maximum particle diameter ($t_0 = d_{\max}$) leads to less dense particle distribution rather than to continuous and uniform powder layers. Similar observations were made in [123]. The powder layer becomes more continuous with increase in the nominal thickness t_0. This is achieved at $t_0 = 2d_{\max}$ for less cohesive powders, while $t_0 = 3d_{\max}$ is required for more cohesive powders for promoting a continuous powder layer. Analysis of the powder packing density indicates that the powder layer thickness should constitute at least two particles, considering that finer particles should be located between them [129]. The spherical particle shape is not always advisable because it increases the powder flow rate, in turn decreasing the packing quality of a single layer in use of a roller recoater. It is currently unknown to what flowability the critical spreading rate corresponds.

The most important layer characteristics are homogeneity, high packing density, and low roughness [130, 131]. The surface quality and packing density of the powder layer decrease with increasing spreading rate (spherical particles spread in the best way, other conditions being equal). In addition, the sensitivity of the powder layer surface roughness increases with greater spreading rate: i.e., the higher the spreading rate, the greater the surface quality deteriorates.

To study the effect of moisture on spreadability [132], the coating area and layer roughness were analyzed for the dry and wet (75% relative humidity) IN718 nickel alloy powder ($d_{\min} = 15$, $d_{50} = 30.4$, and $d_{\max} = 55$ μm). The presence of moisture was found to substantially worsen both indicators. It was also established that moisture had the greatest effect on the behavior of the aluminum alloy powder (AlSi10Mg). There are also difficulties with direct measurement of moisture content [133].

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[73] since it was used to determine the angle of fall. The test was performed as follows: the powder was loaded into the same drum by one-third volume, the drum was rotated very slowly to record the maximum angle, and then, after the powder avalanche descended, the drum was stopped to measure the angle of bank. This method was used to determine two boundary values of the avalanching angle: static and dynamic. With these values, the static and dynamic coefficients of powder internal friction and the dynamic angle, which will be close to the angle of repose, can be calculated.

To improve the spreadability of powders, including water-atomized ones, vibration is proposed to be used [134]. As was shown previously, small vibrations applied to the granular flow significantly reduce the avalanching angle [135].

GENERAL CONSIDERATIONS

Some comments on the PBF process, otherwise called the powder bed deposition (PBD) process, should be added. In the PBD process, some parameters cannot be constant *a priori*. Thus, thermal conductivity of the powder cannot be the same as that of the compact metal located in fusion areas. The heat dissipation of the powder and compact metal under a new powder layer will be different; moreover, it will be determined by the compact weight of the future part [136]. The different temperature of the lower layer, whether it is powder or compact metal, can be accounted for only through the introduction of changes (corrections) to the software that controls the exposure time and laser power and speed.

The features of sintering established in powder metallurgy cannot be extended to the PBD process. Thus, while the properties of a part in powder metallurgy are determined by the average sintering temperature, sintering time, atmosphere, and cooldown conditions and are averaged over the sample, incomplete fusion at one point (and there can be millions of points) in additive manufacturing will cause a defect that may influence the mechanical and service properties of the entire part (product). The paper [136] shows that defects formed on the surface of a part are most dangerous. To eliminate such defects, parts produced by PBF and other related processes are subjected to additional heat treatment—hot isostatic pressing (HIP) [137].

Equal packing densities and melting rates can be promoted only by equal particle sizes and shapes, provided that the same (identical) chemical composition is ensured at all melting points. This has not yet been achieved.

This paper does not address parameters of the machines themselves, such as tolerances for powder layer uniformity, stable energy transfer per time unit, and laser movement accuracy. All these tolerances are considered to be zero, but this is not always the case.

All the factors considered influence, to a certain extent, the quality of parts produced from metal powders. Hence, it is realized that specific properties imparted to and retained by the powders are only a necessary but not sufficient condition for manufacturing parts of high quality.

CONCLUSIONS

The particle size and flowability are most important parameters of powders. The average particle size alone is not sufficient since the upper and lower particle size limits are to be strictly controlled: the upper limit must not be greater than thickness of a single powder layer and the lower limit must prevent the evaporation of particles.

The powder flowability is more important for 3D printers equipped with a blade for powder recoating (recoater) than for 3D printers with a roller recoater rotating counterclockwise. In the latter case, the flowability is not so important, and nonspherical powders (in particular, those produced by grinding) can be used.

There is a correlation between the powder properties and the blade speed and recoater design. Requirements for the recoater are associated with requirements for the powder. Hence, to reduce the effect of lower powder flowability, a roller recoater and even double smoothing are used. The particle size is also associated with the powder recoater. A hard blade and particles coarser than the powder layer thickness cause jamming, which deteriorates the layer quality.

There is no single view on what characteristics should describe the powder flowability and what values of these characteristics can be accepted for a specific 3D printing process. The flow test (standard test) that characterizes the powder dynamic properties was developed for iron powders and does not account for particle density (thus, nickel density is more than three times greater than aluminum density, and 3D printing powders are manufactured from both of these metals). The result presented as the flow time for 50 g of powder does not consider its apparent density either.

For spherical powders, the flowability issues occur with particles smaller than 10–20 μm (depending on particle density) when cohesion forces become close to the gravitational force acting on a particle. Powders of exactly these sizes are applied in the PBF, DED, and BJ processes. The flowability of metal powders can be improved by drying, vacuuming, and applying vibrations of appropriate amplitude and frequency.

In additive manufacturing processes, a new characteristic of the powder dynamic behavior—spreadability—has been introduced. It characterizes the capability of powders to form a thin flat uniform layer with maximum density and without defects on a large area. The percentage of the build plate covered by the spread powder and the avalanching angle of the powder were proposed as spreadability criterion, but the entire plate should be covered by the powder in actual conditions, or otherwise defects can occur. The avalanching angle is not constant and can characterize the spreadability only approximately. To date, there is no accepted spreadability test, nor is there an agreed indicator to characterize the spreadability. There is only an understanding that the research method should reproduce the powder behavior in a 3D printer in the best possible manner. Methods such as powder drum rotation (GranuDrum instrument) or long-developed flowability classification of pharmaceuticals are involved. According to the classification, excellent flowability is inherent in powders having an angle of repose ranging from 25 to 30 deg, Hausner ratio from 1.00 to 1.11, and Carr index from 5 to 15%. The application of these characteristics to metal powders is not quite correct, and this classification needs to be revised and adjusted.

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