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METAL POWDER PRODUCTION ROUTES FOR ADDITIVE MANUFACTURING AND THEIR IMPACT ON PROCESS AND QUALITY: A REVIEW

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Abstract. *Many metal additive manufacturing (AM) technologies use powder as raw material, whose characteristics are decisive to the quality of manufactured components. Therefore, understanding powder production for AM and their impact on processing is of vital importance. Metallic powders are mainly produced by gas atomization, which yield spherical particles with optimum properties; on the downside, it is an expensive technique. Water atomization, as well as mechanical methods (crushing, grinding) are cheaper, but produce irregular powders, leading to subsequent processing challenges. Nonetheless, they can be additively manufactured, and validating their suitability is important to enable cost reduction. Such cost reduction might also come as result of the possibility of utilizing scrap as feedstock. Moreover, metallic powders for AM can be either pre-alloyed, master alloys or elemental. Elemental or master alloy powders can be advantageous for compositional adjustment, in situ alloying and manufacturing of functionally graded components (FGCs). The present work provides a summarized review of the main developments of metallic AM powders. Firstly, a glance of atomized powders is given, followed by insights on mechanical methods and the use of scrap as feedstock. In sequence, in situ alloying studies are presented. Finally, a brief overview on spheroidization of irregular powders is given.*

Keywords: *additive manufacturing, atomization, metal powder, alloying, morphology.*

1. INTRODUCTION

Additive manufacturing (AM) is a continuously growing manufacturing technology, especially promising for the fabrication of complex metallic components. Currently, one of its main challenges is to achieve qualification and certification of materials, processes and components for advanced applications at industrial level. Several metal AM technologies are powder-based; therefore, understanding aspects related to power production and development, their correlation with processing and their impact on the quality of final parts is of utmost importance.

To be processed by AM, metallic powders must, in general, be weldable. Apart from that, other features that define their suitability for AM are flowability, wettability, optical properties (e.g. absorption), thermal properties (e.g. conductivity, diffusivity) and mechanical properties (e.g. hardness, strength) (Doñate-Buendía et al., 2021). The adequate choice of powder production route for additive manufacturing is critical, since powder characteristics exert direct influence on the quality of the manufactured part. Moreover, specific powder characteristics, especially particle size distribution (PSD), are required for each metal AM technology, as shown in Figure 1. For an AISI 316L stainless steel processed by laser-based powder bed fusion (LB-PBF), for instance, Nguejio et al. (2023) showed that powder flowability and porosity level of the built part are negatively affected, albeit slightly, by a wider distribution, whereas microstructural features, such as phases, grain size and texture, are not influenced by PSD. Regarding mechanical behavior, monotonic tensile properties are not affected by size distribution, but fatigue life is also slightly affected due to the porosity effect.

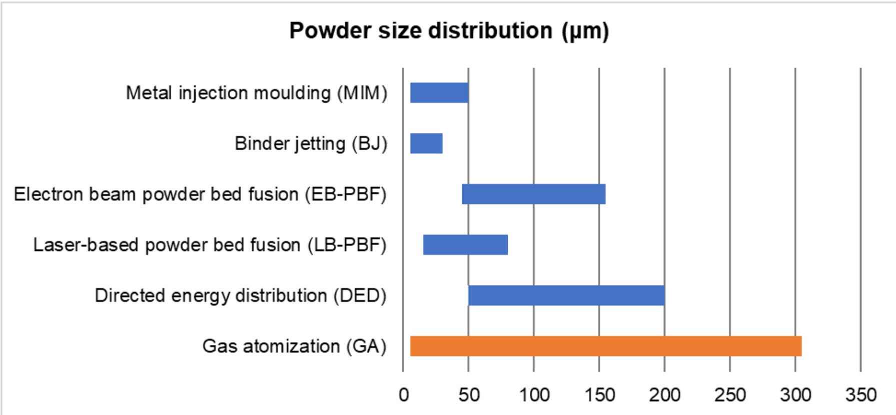

Figure 1. Typical powder size distribution (PSD) ranges obtained by gas atomization and used in different metal additive manufacturing technologies. Based on Dobrzański et al. (2020); Nandwana & Gordo (2022).

Metal powders can be produced by mechanical or physicochemical comminution, or chemical/physicochemical reactions. Particle shape, size and composition, especially contamination and oxygen sensitivity, depend mainly on the production route and the raw material, and are decisive to powder properties and cost. Atomization and chemical reduction are the most common routes for high-volume batches, while mechanical crushing and electrolysis can be used for small quantities. Mechanical methods rely mostly on grinding or milling solid raw materials in ball, vibration or vortex mills, but are not efficient and usually lead to contamination. Moreover, grinding work-hardens the material, resulting in poorer compaction during subsequent processing, which usually makes necessary and annealing treatment prior to use. Another approach is to produce chips by machining – turning or milling – but it is known to be relatively inefficient and prone to contamination (Dobrzański et al., 2020).

The present review will focus on atomization and mechanical routes for powder production. With atomization, the main technologies – gas, plasma and water – will be addressed, and the newly developed ultrasonic atomization will also be presented. Later on, approaches based on mechanical routes will be discussed, in addition to the use of scrap resulting from other processes as feedstock for AM, meaning that emphasis will be given to irregularly shaped powders. The following section will be dedicated to the practice of in situ alloying, in which master alloy or elemental (pure) powders are used as feedstock, as opposed to pre-alloyed powders. A final section will introduce, in a brief manner, existing techniques to transform the morphology of irregular powders in spherical. All alloy compositions are provided in weight percent, unless otherwise stated.

2. METHODOLOGY

This summarized review encompasses mostly scientific papers found upon searches carried out in the main databases – Google Scholar, Science Direct, Scopus and Web of Science – using two terms:

1. "laser powder bed fusion" OR "laser beam powder bed fusion" OR "selective laser melting" OR "LPBF" OR "LB-PBF" OR "SLM";
2. "directed energy deposition" OR "DED" OR “laser metal deposition” OR “LMD”.

Whenever applicable, the search of these terms was limited to title, abstract and keywords. The search was not limited to powders because different additive manufacturing aspects were the focus of different studies developed in parallel. For this review, papers published during the last ten years were considered. Other works, such as books and websites, were also used to provide supporting information. Due to the limited extent of the work, the most relevant studies able to cover the majority of the main features related to metallic powder technology for AM were selected.

3. ATOMIZED POWDERS

Atomization is a liquid-based technique that relies on producing fine droplets atomized by fluid impingement, usually gas (GA), plasma (PA) or water (WA), or mechanical dispersion, such as vibration or rotation. Regardless of the spraying route, all processes come down to melting of the raw material, usually by vacuum induction or plasma heating, droplet atomization and solidification upon rapid cooling. Gas atomization is the most common and efficient technique, since water, despite having some advantages, e.g. higher viscosity, density and cooling capacity, tends to oxidize reactive materials, and its final particle shape is not as spherical and regular as the one attained by GA. Gas atomization usually employs nitrogen or argon – the latter, although inert, can increase powder porosity (Dobrzański et al., 2020; Nandwana & Gordo, 2022). Argon also produces more spherical powders (Gao et al., 2021). Air is basically used only for atomization of aluminum powders, whereas helium is seldom employed due to its high cost and leakage, although it can provide the finest powders (Dobrzański et al., 2020; Nandwana & Gordo, 2022).

Conventional gas atomization can be of the close-coupled (CCGA) or free-fall (FFGA) type, depending on the nozzle, and the main difference between them is that the close-coupled type produces finer powders (Nandwana & Gordo, 2022). Variations of the gas atomization process include VIGA (vacuum induction-melt inert gas atomization), which uses vacuum induction melting and yields fine powders with virtually no contamination and sizes typically ranging from 10 to 40 µm, and EIGA (electrode induction gas atomization), also often used to produce high-purity powders and for processing of reactive and refractory metals. Plasma atomization uses plasma torches to melt and atomize the material, whose feedstock should consist in wires, being this one its main disadvantage. Its main variation is the plasma rotating electrode process (PREP), in which bars are used as feedstock and spun around their axis while a high-powder direct-current plasma arc melts the material (Dobrzański et al., 2020; Nandwana & Gordo, 2022). As compared with GA, the PREP has already shown to mitigate particle porosity issues, which usually arise in gas atomization due to entrapped argon, but has little effect on final mechanical properties (Sames et al., 2014). Table 1 provides a comparison between the main types of gas and plasma atomization.

Table 1. Summary of properties of the main gas and plasma powder atomization techniques: particle size distribution (PSD), median diameter (D50) and melt flow rate. Based on Dobrzański et al. (2020); Nandwana & Gordo, (2022).

Powder production route	PSD (µm)	D50 (µm)	Melt flow rate (kg/min)	Feedstock
Free-fall gas atomization (FFGA)	5-300	100	6-80	Elemental, ingot, bar
Close-coupled gas atomization (CCGA)	5-200	50	2-40	Elemental, ingot, bar
Plasma atomization (PA)	0-150	40	<1	Wire
Plasma rotating electrode process (PREP)	50-300	150	1-4	Bar
Electrode induction-melt inert gas atomization (EIGA)	10-500	90-140	<2	Bar

A novel type of atomization is the ultrasonic atomization (UA). Its concept is already well-established. However, until recently, the technology was mostly applied to organic materials, chemicals and pharmaceuticals. For metallic powder production, it relies on the generation of a plasma arc by a tungsten electrode, which then melts the feedstock – usually in the form of wire or rod – onto a sonotrode, whose ultrasonic vibration generates droplets. These droplets are cooled and turned into particles by an inert gas flow (Hinrichs et al., 2021). UA produces almost perfectly spherical particles, and the vibration frequency can be tuned to yield different PSDs, meaning that it can fabricate powders for different AM techniques, namely LB-PBF and DED. Currently, manufacturers of "benchtop" ultrasonic atomizers exist, which is advantageous for the development of research aimed at powder development for additive manufacturing (3D LAB, 2023; AMAZEMET, 2023). Figure 3 presents a schematic representation of the ultrasonic atomization process, as well as an image of powder particles of a refractory molybdenum (Mo)-based alloy produced by UA.

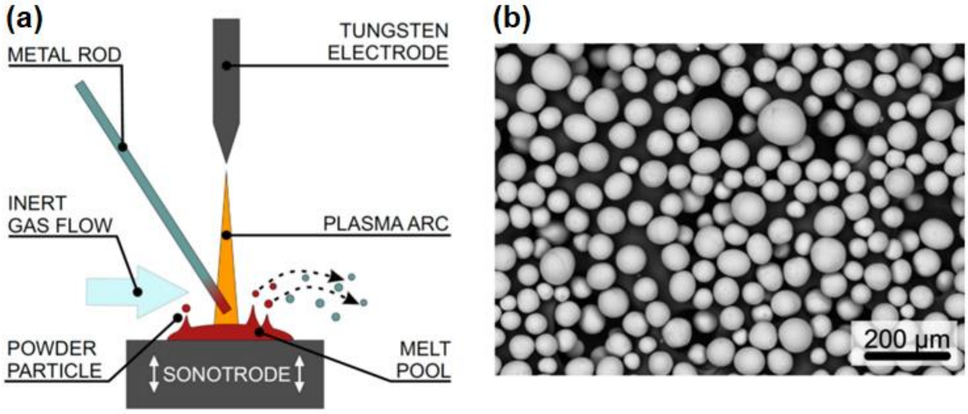

Figure 2. Schematic representation of the UA process (a); powder particles of a Mo-Si-Ti alloy produced by UA (b) (Hinrichs et al., 2021).

There is still much prejudice against irregularly-shaped powders for AM, such as those produced by water atomization, because gas-atomized powder are considered ideal due to their sphericity and smooth surface. Plasma-atomized materials are even more spherical (Figure 3), but the process is much more expensive; therefore, GA becomes preferable (Wallner, 2019). The higher the sphericity, the higher the apparent density of the powder and the flowability (Tan et al., 2017). The irregular shape also leads to higher absorbance by WA powders, leading to higher energy inputs needed for adequate processing. In an AISI 431 stainless steel, for example, such difference led to significant microstructural variations, with the GA powder yielding a fully martensitic microstructure and the WA powder yielding a duplex martenitic/ferritic microstructure, with impacts in wear and corrosion properties (Hatem et al., 2021). However, Hoeges et al. (2017), for instance, showed that additively manufactured parts made of either GA or WA AISI 316L stainless steel presented equivalent mechanical behaviors, which shows that, using the right set of processing parameters, water- and gas-atomized powders can yield components with comparable qualities. They point out that surface finishing of parts fabricated using WA parts is inferior, but this is not critical, since post-processing for surface improvement is a common practice in additive manufacturing. Furthermore, direct comparison between behaviors of alloys fabricated by different routes is not so straightforward, since differences in composition (mainly oxygen content) and size distribution usually exist. All in all, water atomization is the technology of choice to fabricate large batches of alloy steels, stainless steels and copper alloys (Dobrzański et al., 2020).

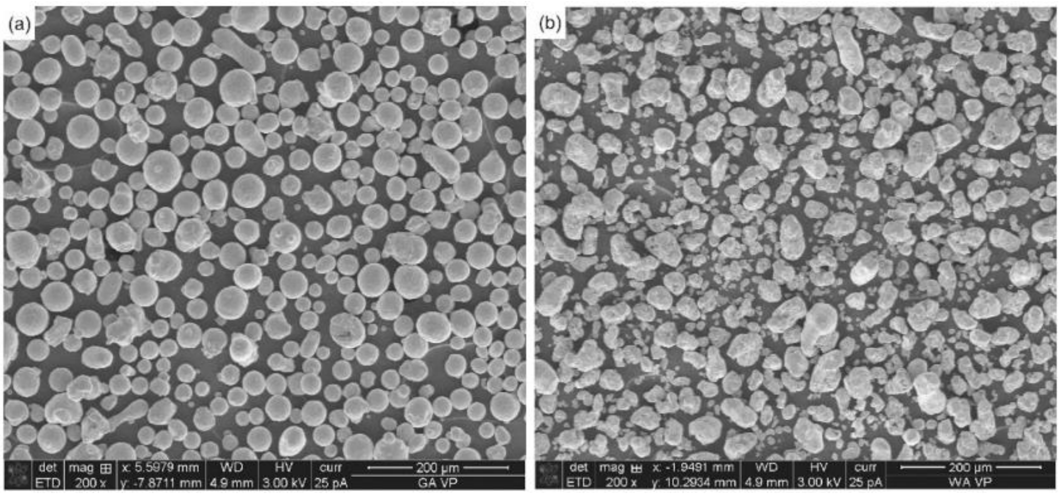

Figure 3. Gas-atomized (a) and water-atomized (b) powders of an AISI 4130 low-alloy steel (Fedina et al., 2020).

Fedina et al. (2020) compared the processability of GA and WA powders of an AISI 4130 low-alloy steel by LB-PBF and concluded that water-atomized material, which is cheaper and has a higher production yield, can be adequately processed upon optimum parameters selection, provided that shape irregularity shown by the WA powder leads to poorer compaction, while the higher amount of oxygen results in higher spatter tendency. Both they and Garg et al. (2023) observed that irregular powders lead to smaller track heights during LB-PBF and DED, respectively, due to limited compaction. With respect to spatial resolution, a minimum circular channel nominal size of 400 µm was achieved using

WA iron powder processed with optimized parameters; however, the elevated roughness (42 ± 3 μm) must be taken into account (Rogalsky et al., 2018).

The presence of satellite particles, which consist in smaller particles attached to the surface of larger ones, negatively affects the flowability and spreadability of powders due to an interlocking effect (Mussatto et al., 2021). The larger the average particle size, the higher the tendency of satellite formation, as shown in Figure 4. Moreover, larger particles demand higher laser powder to promote full melting, thus avoiding lack-of-fusion porosity, and ensure sufficient bonding between the track and the substrate (Chu et al., 2023). Beckers et al. (2020) studied the effect of gas flow on the characteristics of atomized particles and observed that the use of coaxial gas flow (flow tangential to the main one, creating a resulting circular flow) does not affect the amount of satellite particles, regardless of particle size. It does, however, affect particle shape, leading to higher sphericity.

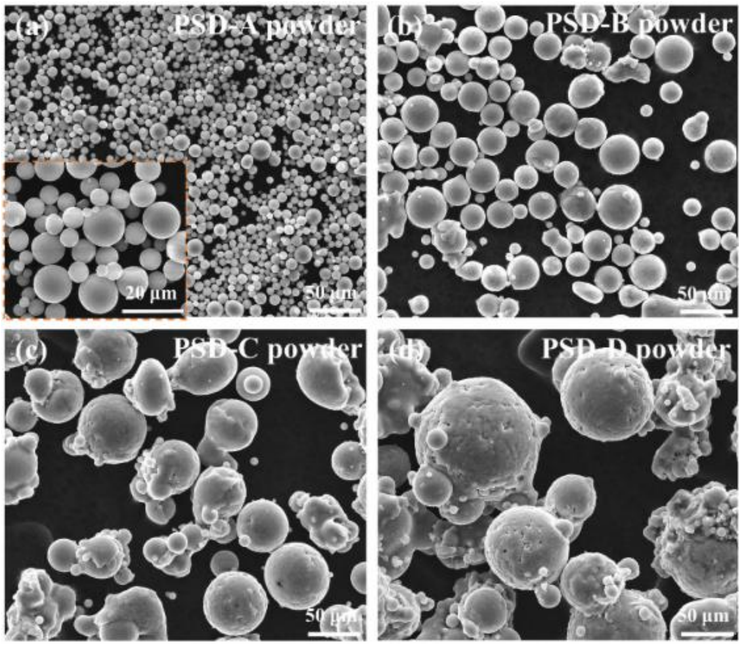

Figure 4. Presence of satellites in Al10SiMg alloy particles with D50 sizes of 16.4 μm (a), 41.0 μm (b), 56.2 μm (c) and 76.3 μm (d) (Chu et al., 2023).

4. MECHANICAL ROUTES AND SCRAP RECYCLING

Aiming at both waste reduction and cost reduction of AM feedstock, a trending approach has been turning metal scrap, such as metallic machining chips remaining from turning, milling, drilling, cutting and grinding operations, into powder. Dhiman et al. (2021) (Dhiman et al., 2021) proposed a framework for such conversion using ball milling. Intermediate steps included shredding for comminution, cleaning and drying prior to milling. They state that milling time and ball diameter are the main parameters to influence size and morphology of the fabricated powder, as well as hardness due to work hardening. The approach is much more sustainable, consuming 20-30% less energy, and the processability of the ball-milled powder by laser AM was comparable to that of GA powders. Figure 5 shows a comparative chart of main costs associated with gas atomization and ball milling. Dhami et al. (2022) were able to obtain spherical particles from conventional grinding of steel just by adjustment of grinding speed and depth, using a rotating alumina wheel. Resulting chips included both spherical particles and elongated strings, which were separated by sieving. The final step involved moisture removal upon heating at 75°C. Preliminary single-track trials by DED showed that the ground material produced consolidated microstructures and hardness comparable to those achieved by gas-atomized powders.

Sun et al. (2016) developed the so-called GSD (granulation-sintering-deoxidation) process, which involves ball-milling with solvent for cleaning and binding to form granules prior to sintering, to produce spherical Ti-6Al-4V powder from scrap suitable for AM. In this case, the feedstock consisted of fine hydride Ti64 powder fabricated from scrap through cleaning, hydrogenating, milling and sizing. Powders are spherical and, although their surface is not as smooth as that obtained by atomization, it is comparable to the surface of atomized powders after reuse, which is common practice in additive manufacturing. The same alloy powder (Asherloo et al., 2023) and powder of a Ti-53Nb alloy (Guzmán et al., 2021), both produced by hydride-dehydride process, which creates irregular and non-spherical powders, were successfully processed by LB-PBF as well. Still regarding titanium, a process using fluidized bed with argon flow to grind particles was also proposed (Ding et al., 2019). By this approach, particle collision leads to removal of particles' sharp edges with consequent improvement of flowability. The main drawback of this technique is the need for high temperatures, meaning that considerable oxygen pick-up takes place.

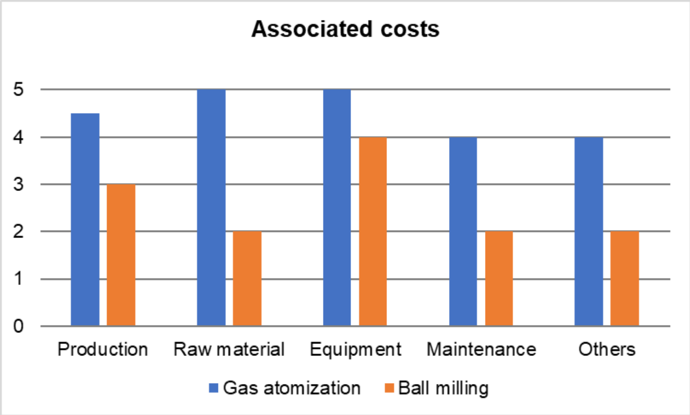

Figure 5. Comparison of costs associated with GA and ball milling (5: higher cost). Based on Dhiman et al. (2021).

A series of works developed by researchers of the Ruhr-Universität Bochum, the Bergische Universität Wuppertal and the IWT Bremen were focused on the AM of tool steel using different starting powders (2021-2022). They used both pre-alloyed, gas-atomized spherical powders and mixtures of gas-atomized pure iron with mechanically crushed pure elements and ferroalloys. The steel processed using the powder mixture presented mostly cracks and lack-of-fusion defects, while gas pores were the main defect in the one fabricated using pre-alloyed powder; however, relative density, mechanical properties and residual stresses of both materials were comparable or even with a better performance of the material consisting of the powder mixture; in this case, higher stiffness and hardness were observed, mainly due to the presence of trapped unmolten ferroalloy particles (Chehreh et al., 2021). Although the flowability of the mixture is poorer, as well as its packing density and permeability, high-density specimens were successfully fabricated. It was also shown that the laser reflectance of the powder mixture is lower than that of the gas-atomized powder (Hantke et al., 2022). They showed that hot isostatic pressing post-treatment, although effective to improve density, does not exert any influence on the steel's chemical heterogeneity; on the other hand, a *supersolidus* liquid phase heat treatment led to both improved density and chemical homogeneity (Großwendt et al., 2021).

5. IN SITU ALLOYING

Apart from the powder manufacturing technologies in terms of particle yielding itself, another relevant aspect is powder composition. The main approach in this regard is to produce powders from pre-alloyed feedstock, so that the composition of the powder is as close as possible to the target composition of the final alloy. However, it is also possible to produce elemental (pure) or master alloy powders for in-situ alloying or blending during AM. While pre-alloyed powders reduce the risk of heterogeneity in the manufactured part, elemental powders can be useful for fine compositional adjustments, rapid alloy design and to fabricate functionally graded components (FGCs), for example (Garrard et al., 2022; Sing et al., 2021). One of the main issues regarding elemental powders is the difference between elements' properties, such as reflectivity, thermal expansion and conductivity, and melting points, which might lead to issues such as increased porosity (Garrard et al., 2022). Strauch et al. (2021) further reported differences in dissolution behavior of individual powders, which also leads to chemical, microstructural and hardness heterogeneity.

The work by researchers of the Ruhr-Universität Bochum, the Bergische Universität Wuppertal and the IWT Bremen cited in the previous section are also examples of in situ alloying. Garrard et al. (2022) managed to fabricate Al-Si alloy with different Al/Si ratios using both pre-alloyed and pure elemental powders of aluminum and silicon. They showed that in situ alloying is useful for reliable alloy design, since both pre-alloyed and elemental powder yielded similar mechanical properties. A review by Ghanavati & Naffakh-Moosavy (2021) provides a robust summary of metallic FGCs with graded compositions fabricated by additive manufacturing. Most studies are focused on titanium and steel-based materials, fabricated mainly by DED technologies. However, studies focused on Al-, Co- and Ni-based FGCs also exist. An interesting approach to fabricate FGCs is the one by Duan et al. (2021), which relied not only on in situ alloying – using titanium (Ti) and molybdenum (Mo) powders, in this case – but also on the variation of the volumetric energy density (VED) input to the system. By this approach, VED variations in the build direction led to different concentrations of unmelted Mo particles (Figure 6) which, in its turn, led to different microstructures. Micropillar compression tests showed that the Ti-Mo FGCs presented an expressive yield strength with higher levels of strain hardening at the early stages of plastic deformation.

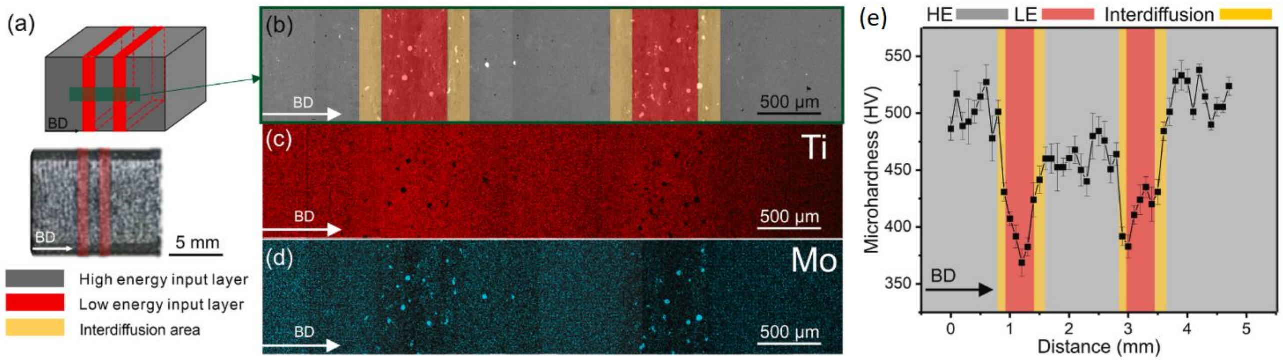

Figure 6. Schematic of the FGC made of Ti-12Mo alloy with alternate energy input layers (a); micrograph (b) and respective elemental distribution maps of Ti (c) and Mo (d); microhardness profile across layers (e) (Duan et al., 2021).

6. SPHEROIDIZATION TREATMENTS

Even though it has been shown that powder with irregular morphology can be processed by additive manufacturing upon careful optimization of the set of parameters used, there are methods to turn them into spherical powders with enhanced processability. Inductively-coupled plasma (ICP) spheroidization is a well-suited and commonly used process, which involves high-temperature plasma produced by induction coils. A carrier gas brings powder particles into the system, where they are subjected to the plasma and, depending on particle size, melting point and exposure time, they can completely or partially melt. Finally, free-falling particles solidify into spheres (Sehhat et al., 2022). The result is an almost perfectly spherical particle, as shown in Figure 7. Another example is the thermomechanical spheroidization treatment (TMST), which employs annealing in a hydrogen atmosphere followed by low-energy ball milling (Mutel et al., 2023). Although it does not lead to full spheroidization of the powder, it does lead to a less irregular morphology, in addition to reducing particle's surface roughness.

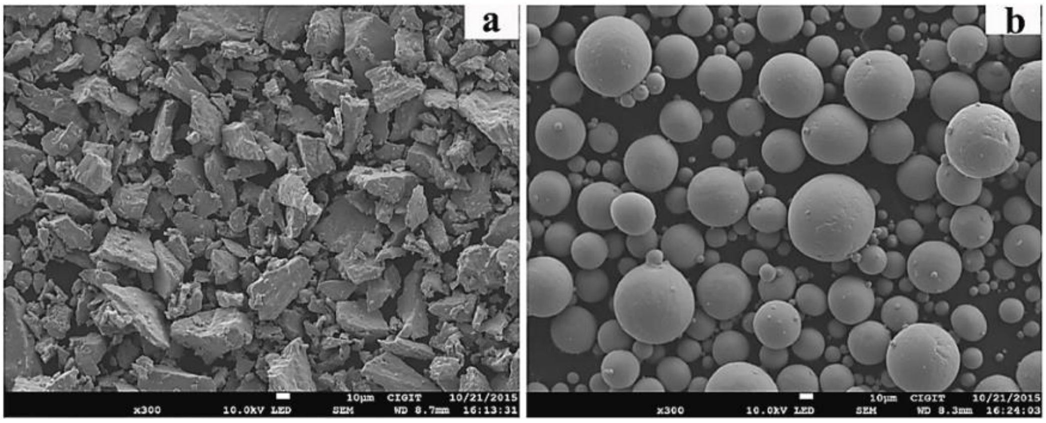

Figure 7. Morphologies of a Ti-6Al-4V alloy powder before (a) and after (b) ICP spheroidization (Wei et al., 2017).

Lee et al. (2023) designed a process for powder spheroidization called SMART (surface modification and reinforcement transplantation). This process is not only suited for spheroidization, but also to produce core-shell particles consisting of powder plus a reinforcement surface layer, or plus another element layer, generating multi-material particles. The process is based on the collision of larger base particles and smaller supplementary particles, and pressure welding takes place upon the collision of base and supplementary powder particles as result of the movement generated by rotary blades in an attrition milling equipment. Collision with blades removes satellite particles and creates a smooth surface. Figure 8 shows an example of application of the SMART process to fabricate core-shell Mo/Inconel 625 composite particles.

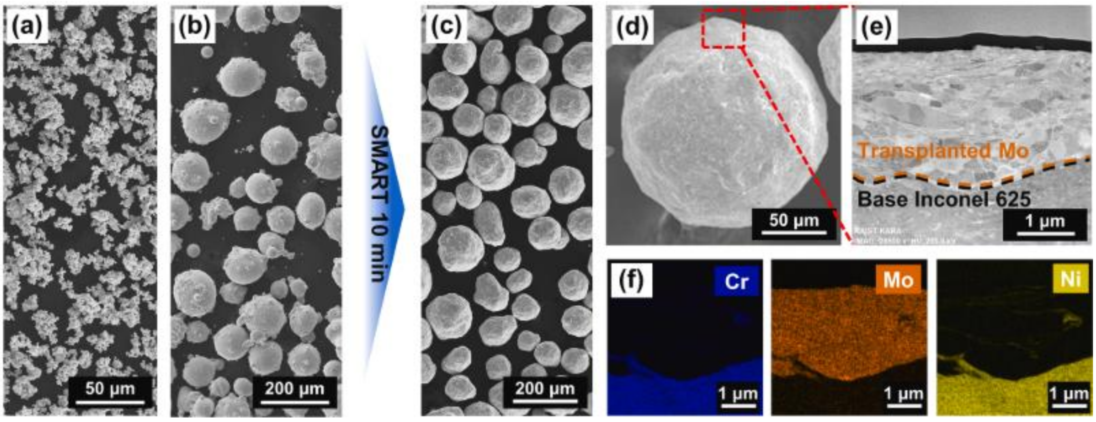

Figure 8. Images of supplementary Mo powder (a) and base Inconel 625 powder (b), and of the Mo/Inconel 625 composite particles produced by the SMART process (c), with details of particle morphology (d) and particle surface (e), and elemental distributions on the surface (f) (Lee et al., 2023).

7. CONCLUDING REMARKS

This work provided a summarized overview of some relevant aspects related to metallic powders for additive manufacturing. Studies aimed at powder development are essential for the continuous improvement of metallic AM, since powder quality and properties directly impact the quality of manufactured components. Understanding powder production in an ever deepening manner enables assertive development of alloys and can also help turning additive manufacturing into an increasingly cost-effective technology, since optimizing feedstock production can result in feedstock cost reduction and more effective usage, leading to waste reduction.

Although gas-atomized powders are preferred due to their sphericity, which, at least theoretically, leads to enhanced processability, literature shows that irregularly-shaped powder, such as water-atomized or mechanically produced, can also be consolidated into components with comparable properties and low density levels upon definition of adequate processing windows. Moreover, not even spherical powders are completely free of defects, since they can suffer from issues such as inner porosity, especially upon atomization with argon, and satellite particles, which hinder powder packing and flowability. The main advantages of irregular powders are the lower cost, and the possibility of using scrap arising from other processes as feedstock. Nevertheless, development of post-processing technologies aiming at spheroidization of irregular powders also exist and can be employed in cases where powder sphericity, for any reason, makes itself necessary. However, more information about process efficiency and costs is necessary to understand their suitability for industrial upscaling.

Regarding composition, using elemental or master alloy powders, as opposed to pre-alloyed powders, can be advantageous for allowing minor adjustments and material tailoring, in terms of either alloy development or graded functionalization to improve performance. Additional advantages include lower cost, generally speaking, while the main drawback regards differences in optical and thermal properties of individual powders, leading to issues such as lack of fusion and chemical heterogeneity.

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