



Powder based additive manufacturing for biomedical application of titanium and its alloys: a review

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Abstract

Powder based additive manufacturing (AM) technology of Ti and its alloys has received great attention in biomedical applications owing to its advantages such as customized fabrication, potential to be cost-, time-, and resource-saving. The performance of additive manufactured implants or scaffolds strongly depends on various kinds of AM technique and the quality of Ti and its alloy powders. This paper has specifically covered the process of commonly used powder-based AM technique and the powder production of Ti and its alloy. The selected techniques include laser-based powder bed fusion of metals (PBF-LB/M), electron beam powder bed fusion of metals (PBF-EB/M), and directed energy deposition utilized in the production of the biomaterials are discussed as well as the powder fed system of binder jetting. Moreover, titanium based powder production methods such as gas atomization, plasma atomization, and plasma rotating electrode process are also discussed.

Keywords Additive manufacturing · Titanium (Ti) and its alloy powder · Biomaterials · 3D printing

1 Introduction

Additive manufacturing (AM) is a powerful tool to fabricate complex geometries layer-by-layer with computer-aided design (CAD) [1–5]. In the recent decades, it has received a great attention globally among diverse fields and has the field has developed rapidly with its advantages. It is capable of fabricating complex parts that are not possible through

other methods, and its potential to be cost-effective, time-saving, and resource-saving has attracted both the academic and industry researchers [6–8]. It is widely used in biomedical applications such as orthopedic implants, dental implants, and cardiovascular systems (Fig. 1) [9–13].

Metallic implants are widely used in load-bearing orthopedic field [14–21]. Some of the commonly used metals include titanium, stainless steels, and cobalt-chromium alloys. Ti6Al4V alloy, also known as Ti64, has been one of the most widely accepted as a reliable material in the biomedical field with its unique combination of mechanical properties, corrosion resistance and biocompatibility [22–25]. Initially, the alloy was designed for use in the aerospace industries back in the 1950s [26]. Ti64 has an α - β structure with high strength, low density, and corrosion resistance thus it was well suited for its use as jet engines, gas turbines and other aircraft structural parts [27–30]. Furthermore, its' corrosion resistance to most corrosive acids and alkalis further spanned its uses in other fields [31, 32]. Today, Ti64 is still extensively used in aerospace applications but also used in numerous non aerospace fields such as marine, automobile, energy, chemical, and biomedical industries [33, 34]. The Ti6Al4V alloy takes about a half of the market share of Ti uses globally, being the most widely used Ti alloy. In the medical field, the material properties of

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Fig. 1 Schematic diagram of the biomedical application of additive manufacturing of biometals includes cranial prosthesis, dental implants, acetabular cup, interbody fusion cage, hip prosthesis and knee prosthesis



Ti64 such as its high strength and fracture toughness, low density, and excellent corrosion resistance and biocompatibility made it an attractive biomaterial candidate for uses in dental and orthopedic applications [35–37].

Modern manufacturing of medical implants mostly used austenitic stainless steel, cobalt-chromium-based and titanium-based materials. In order to obtain customized properties that meets the structural requirements, advanced fabricating techniques such as additive manufacturing (metal-AM) are used [38]. The AM methods can be categorized into powder bed and powder-fed systems. In this review of some common powder bed systems which include laser-based powder bed fusion of metals (PBF-LB/M), electron beam powder bed fusion of metals (PBF-EB/M), and binder jetting (BJ) are discussed as well as the powder fed system of directed energy deposition (DED) [39–41]. These technologies have become a powerful tool in a myriad of applications in orthopedics, dental, and cardiovascular fields. For example, to fabricate an implant that would replace a bone defect, it should replicate the intricate structure and provide mechanical support to mimic the function of the defect site [42]. With the use of X-ray computed tomography or

magnetic resonance imaging data of individual patients, 3D modeling can be done with CAD (computer-aided design) to define the specific geometry. The metal-AM techniques have been selected for use in additive manufactured implants and other biomedical applications with varying material and structural properties with advantages in terms of cost, manufacturing time, and repeatability [43].

The performance of these additive manufactured implants strongly depends on the quality of Ti and its alloy powders. Before the development of AM technology, powder metallurgy (PM) process such as metal injection molding and hot isostatic pressing also used Ti and its alloy powders to fabricate near-net-shape bulk products. The powders for AM have stringent physical and chemical demands. Generally, the powders in powder bed and powder-fed systems must have good flowability, and excellent tap density for uniformity of additive manufactured products [44–46]. Consequently, homogeneous spherical shaped powders are preferred. Furthermore, low oxygen contents in Ti and its alloy powders are needed to ensure sufficient ductility and fracture toughness [6, 47]. Gas atomization (GA), plasma atomization (PA) and plasma rotating electrode process (PREP) are

conventional method to produce spherical powders with high purity for various PM processes [48, 49]. And as the portion of AM increases, the importance of powder production processes also increases.

This paper provides a general review of powder processing of Ti and its alloy for biomedical application using four metal-AM methods (PBF-LB/M, PBF-EB/M, BJ, and DED) and also covers the production process (GA, PA, and PREP) of Ti and its alloys for AM.

2 Powder-based additive manufacturing processes

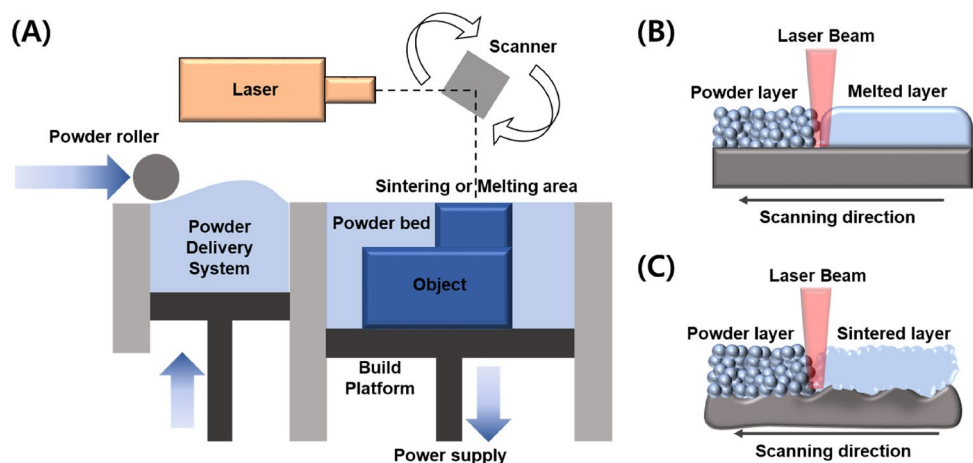
2.1 Laser-based powder bed fusion of metals (PBF-LB/M)

Laser-based powder bed fusion methods can be divided into complete melting and partially sintering of powder. As shown in Fig. 2a, b, high-power lasers that completely melt the powder layer-by-layer is the method that involves melting of the powder. It was first introduced in mid-1990s [50] which was built on the concept of selectively laser sintering powder that was developed in the late 1980s which only partially melts the powder while retaining its solid core as shown in Fig. 2a, c [51]. Powder-based metallic manufacturing involves polymer serving as a binding agent to hold the metallic powder together at relatively low temperature, and it is later removed through a burn-out process which results in a porous structure. As the schematic shows, the two method generally have a powder dispensing platform with a roller and a build platform both on supporting pistons. Roller layers a powder evenly and laser beam is used to either melts or selectively sinters powder according to a CAD model. Once the layer is complete the working piston is lowered and the sequential layers are repeatedly fabricated layer-by-layer. Previous works include working on decreasing the porosity

as well as adding a post-processing step following selective laser sintering to produce fuller density products for use as biomaterials [52, 53]. Studies on porous materials have extensively focused on tuning the geometry of porous structure and biocompatibility to meet the needs for use in orthopedic implants that can interlock the implant in defect site and simultaneously allow better cell-to-implant interaction [54].

The starting material for PBF-LB/M can be a single or multi-component and PBF-LB/M is performed in vacuum or inert-gas protected state to prevent the metallic powders from reacting with other gases [55]. In particular, Ti and its alloys are easily oxidized thus it is crucial to use inert gas or use in vacuum state to minimize the oxygen exposure. Depending on the starting powder, the consolidation process has two binding mechanisms [56]. First mechanism involves liquid phase sintering in which polymer is used to act as a binder for metal particles with a laser beam energy density of 1 J mm^{-3} , and additional thermal treatment is needed to burn out the binder and sinter the green body followed by an infiltration method to obtain higher density product. The other mechanism is of direct melting of metallic powders (e.g. Ti6Al4V and CoCr alloy) with laser beam energy density of 200 J mm^{-3} and no further process is required [57]. The advantages of using PBF-LB/M include near-net-shape fabrication of complex shapes, high mechanical properties, high dimensional accuracy, better surface finish, and high material recycling rate [43]. Generally, maximum beam power, beam spot size, scan speed, and layer thickness are 120 W, 30–250 μm , 0.3–1 m/s, and 20–100 μm , respectively [26]. With successful advances in machine development and efforts to make it more productive and cost-effective, it is the one of the popular powder bed technology today in fabricating metallic materials for biomedical application using AM technique. The involvement of globally leading AM companies in the PBF-LB/M technology development speeded up the advancement in machines such as enlarging building

Fig. 2 Schematic diagram of the **a** PBF-LB/M, **b** the working principles of complete melting of powder layer and **c** selective sintering of powder, respectively



envelope size up to $500 \times 280 \times 365 \text{ mm}^3$, changing the laser type to Quad IPG laser, adjusting the substrate plate, and altering the powder removal process [41]. Current usage of PBF-LB/M includes printing implants with complex-geometry for orthopedic and dental uses. Complex structures such as gyroid scaffolds have been fabricated and studied for suitability as biomaterials [58, 59]. Moreover, studies on post processing of the additively manufactured samples to enhance the mechanical and biological properties for use as biomaterials are ongoing such as HIP, machining and polishing [60–62]. Studies have further studied surface modification of the products for enhancing biocompatibility and reducing side effects [63]. The limitations of the techniques include time consuming process and acute size restriction difficulty in scaling-up and high residual stress [64].

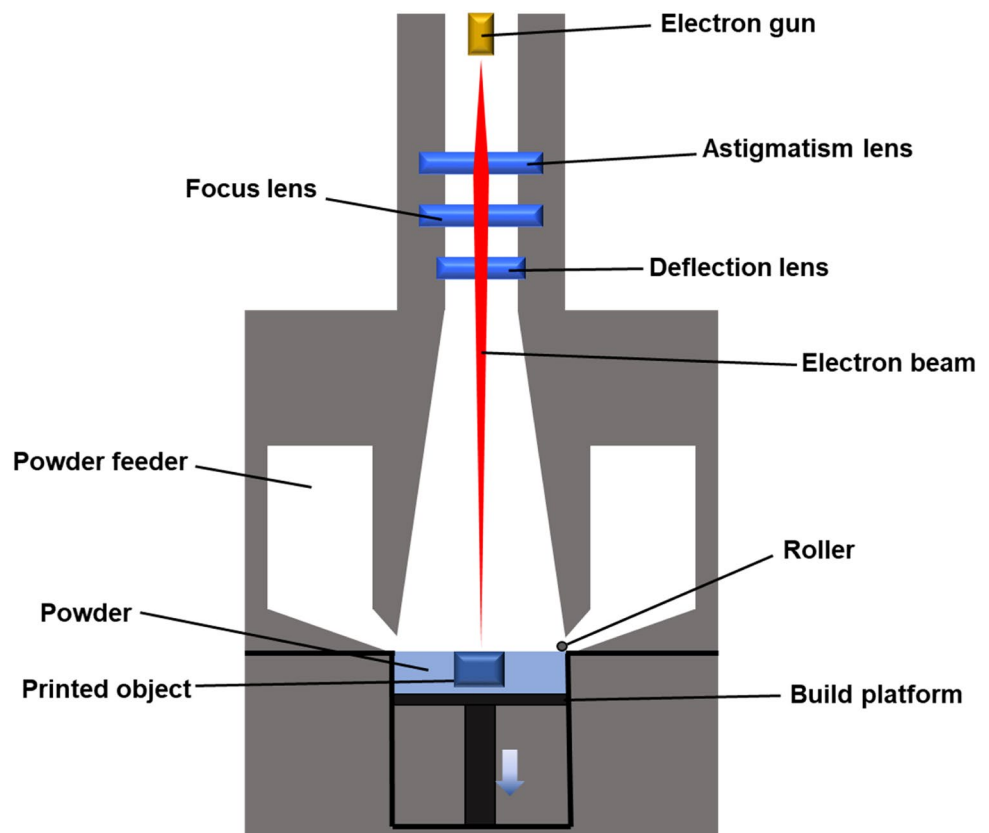
2.2 Electron beam powder bed fusion of metals (PBF-EB/M)

Electron beam powder bed fusion of metals (PBF-EB/M) technique, developed in mid-2010 by Arcam AB, is a powder fed fusion AM method for metals that uses high-speed electron beam energy under high vacuum (10^{-4} – 10^{-5} mbar) [65, 66]. The metal powders are selectively bombarded and melted to fabricate samples as shown in Fig. 3. Today, metal-AM for use as biomaterials mainly use laser and electron

beam sources. As Fig. 3 shows, the components that make up the PBF-EB/M technique are very similar to PBF-LB/M technique in that rake, build platform, powder hopper, and energy source are involved [66]. Powder bed is first prepared on the build platform then electron beam is selectively aligned based on the CAD model and melts the powder. The electromagnetic lens focuses the accelerated electrons into energy beam [67]. The process is repeated layer-by-layer building onto previously fabricated layer. When the manufacturing process is over the residual powder is removed [68]. Generally, the maximum beam power, beam spot size, scan speed, and layer thickness are approximately 3500 W, 200–1000 μm , $> 1000 \text{ m/s}$, 50–200 μm , respectively [26]. The energy density of electron beam above 100 kW cm^{-2} can be obtained at a power of 60 kW [69].

The manipulation of the electron beam is capable by varying the parameters of the magnetic field. The chamber atmosphere along with manufacturing parameters such as the electron and focusing current, action time, powder properties, acceleration voltage, and scanning mode are important factors in PBF-EB/M technique [4, 70]. These can have effects on the size, roughness and porosity [71]. The processing and the final product quality depends largely on the powder properties. It is important for powder used in PBF-EB/M to have high flowability. Generally, sphere powder fabricated through gas atomization with a target size

Fig. 3 Schematic diagram of the electron beam powder bed fusion of metals (PBF-EB/M)



of 40–105 μm is used. However, working with larger mean diameter powder is easier in terms of process stability for which powder produced by plasma rotation electrode process (PREP) is preferred with target size of 50–150 μm [72]. Another important powder properties to take into consideration for EBM is repulsion of charges on powders which can result in smoke events [73]. Pre-treatment such as heating the plate prior to processing to slightly sinter the powder is used which also enhances the conductivity that can help processing stability. The powder used for PBF-EB/M can be reused with sieving [44].

The products fabricated through PBF-EB/M technique in comparison to PBF-LB/M show decreased residual stress but results in a rougher surface since the PBF-LB/M technique can work with smaller particles [74, 75]. In addition it has a higher energy utilization rate, higher material absorption rate, and lower operating cost. PBF-LB/M and PBF-EB/M of metallic materials have great advantages over other AM of metallic materials with better quality of the finished products in terms of resolution and dimensional accuracy thus have shown promising results in orthopedic applications [42, 76, 77]. PBF-EB/M has been used with femoral knee implants with Co-29Cr-6Mo [78]. Complex structures produced by PBF-EB/M [79, 80], corrosion resistance [81], and biocompatibility [82, 83] include some of recent research topics with PBF-EB/M-fabricated materials.

2.3 Directed energy deposition (DED)

DED technology is an additive manufacturing technique first developed by Sandia National Laboratory in the 1990s [84]. The technology is one of the widely studied technology which uses injected powder, instead of a powder bed, along with laser to melt and build on substrate as the schematic shows in Fig. 4. Its advantages include capability to produce complex shapes with near net-shape parts, capability to produce parts with gradients of pores or different metals (such as Ti alloys, stainless steel, and shape memory alloys) and also produce parts with a mixture of different metal powders to create alloys [85]. Moreover, the big differences from powder bed fusion techniques is that it has better fabrication efficiency and cooling effect and re-fabricating capability [86]. The applications of the technology not only include making near net-shaped parts but also used in repairing, modifying the surface, and adding features to parts [43]. The components of DED technology include laser beam, powder nozzle, and a build platform. The DED AM process starts with the delivery of powder (or mixture of powders) with argon-pressurized nozzles to the point on which the laser beam is focused on. Then melting occurs and forms a melt pool while the platform is moving according to the designed CAD model [86, 87]. The melted regions solidifies and forms a layer and the successive layer is repeated

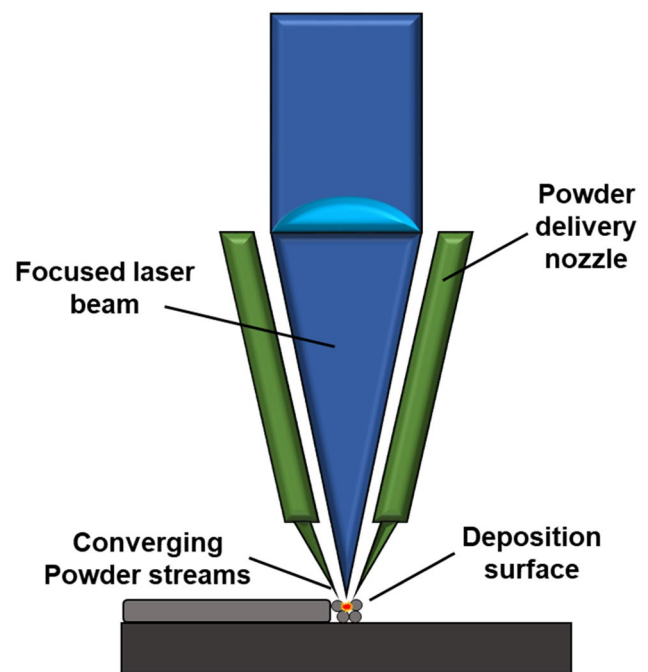


Fig. 4 Schematic diagram of the directed energy deposition (DED)

after raising the laser and the powder nozzle. This all takes place in an enclosed and oxygen-controlled (< 10 ppm) environment to minimize oxidation of the fabricated parts [88]. Significant amount of recent research trend of DED technology is geared toward biomedical load-bearing applications [89, 90]. Functionally structured materials fabricated with DED technology were reported such as porous Ti-based implant designed with the mechanical properties to mimic the human bone more closely compared to dense Ti material [91]. Moreover, the microstructural and corrosion-related effects were studied in terms of the processing parameters of NiTi alloys using DED technology for bone implants [89]. Some recent applications of the technology include developing implants that are antimicrobial and biocompatible with Tanium-silver alloys [92], studying the surface properties [93, 94], and load-bearing properties [91, 95] of Ti–6Al–4V alloys.

2.4 Binder jetting (BJ)

Binder jetting, also known as 3D printing (3DP), is an additive manufacturing technology which uses powder and liquid binding agent and it was developed in the early 1990s at Massachusetts Institute of Technology [68, 96, 97]. Figure 5 shows the components that make up binder jetting include powder spreader, print-head, and build platform [98]. The binding agent is selectively deposited onto a bed of metallic powder followed by lowering of the powder bed and repeating the process layer-by-layer to obtain a “green” body

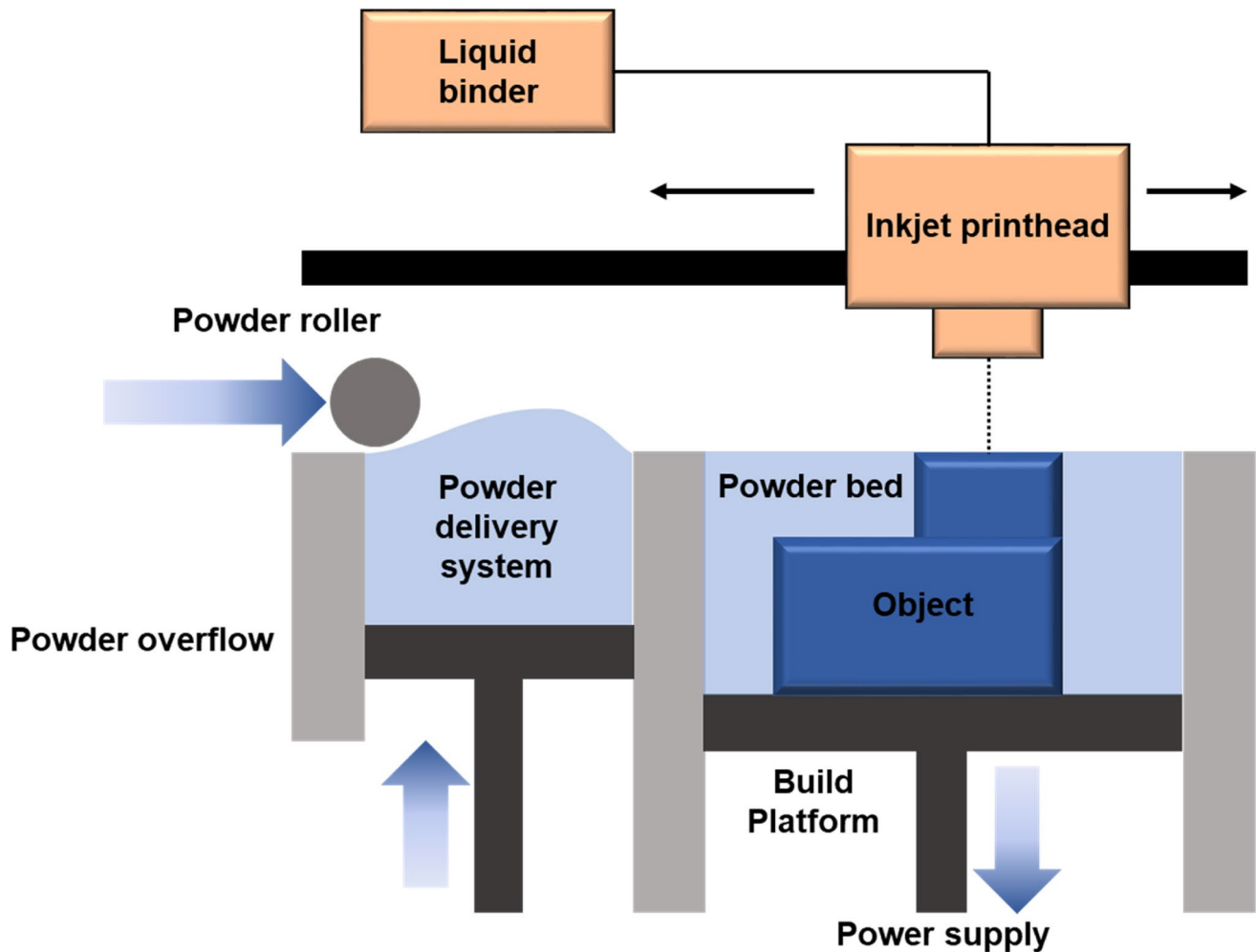


Fig. 5 Schematic diagram of the binder jetting (BJ)

using a predesigned CAD model. The fabricated green body is cured to enhance mechanical strength and is sintered. The sintering process (1100 °C, 24–36 h) burns off binding agent and sinters the green body. Additionally, to liquid state infiltrates the porous scaffold through capillary action as the driving force [68]. The advantages of the manufacturing technique are that it is cost-effective with high efficiency and that no residual stress is involved whereas the disadvantages include shrinkage during sintering, poor quality and low mechanical properties [68].

3 Powder production processes

3.1 Gas atomization (GA)

With a great demand of spherical Ti powder for AM technologies, many studies have focused on developing alternative methods to reduce the cost of traditional wrought method

and improve the quality of powder. Powder metallurgy technology which allows near-net-shape manufacturing, includes processes such as powder production, compaction, sintering, and post-sintering processing [99]. The quality of the parts created using AM will depend heavily on the powder deposition and the manufacturing process of powder which determines the powder properties such as morphology, size, size distribution, tap density, and flowability. The Hausner ratio is the ratio of the tap to apparent densities and serves as a simple way to describe the flowability of powders.

Commonly used methods to fabricate spherical Ti powder today include gas atomization, plasma atomization, and plasma rotating electrode process [99, 100]. Gas atomization is a method that was introduced 1980s by Crucible Materials Corporation [100–102]. As shown in the simplified schematic of GA in Fig. 6, starting material (raw or pre-alloyed materials) in the shape of ingots or bars are induction skull melted in vacuum into a homogeneously melted state which is contained in a thin skull of the same composition

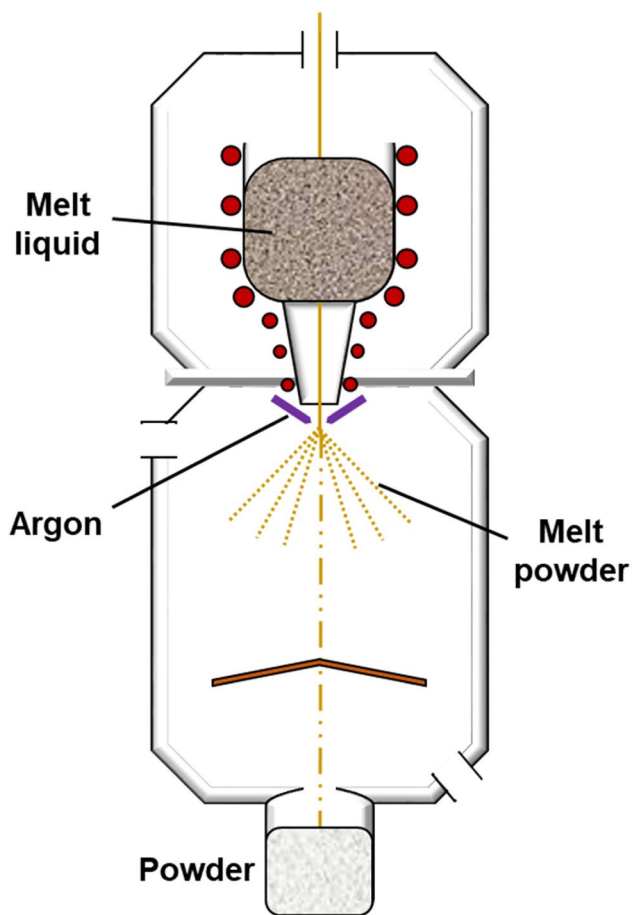


Fig. 6 Schematic diagram of the GA process for AM

which forms in the inner wall of the crucible. When the melt is ready for atomization, a localized melting occurs with a smaller induction coil at the bottom of the skull and it is poured into an induction heated nozzle and atomized with high pressure gas. The droplets then undergoes cooling while in free fall state which is called free-fall gas atomization (FFGA) [100, 103]. The particles generally have a wide size range less than $500\ \mu\text{m}$ [103].

There are several issues which are addressed with the GA technology. First, particles produced with GA tends to have satellite particles which play a negative role on the free-flow of particles. The reason for production of satellite particles is that fine particle interact with droplets. Second, ceramic powder contamination from erosion of the nozzle by the melted liquid is possible. Lastly, argon gas may be trapped within the melted state resulting in porous powder particles which is not removable even with hot isostatic pressing and have detrimental results on the mechanical properties [99, 100].

A critical factor in determining the particle size distribution of GA powder is the gas-to-metal flow ratio [104]. A higher ratio would result in a higher yield of fine particles

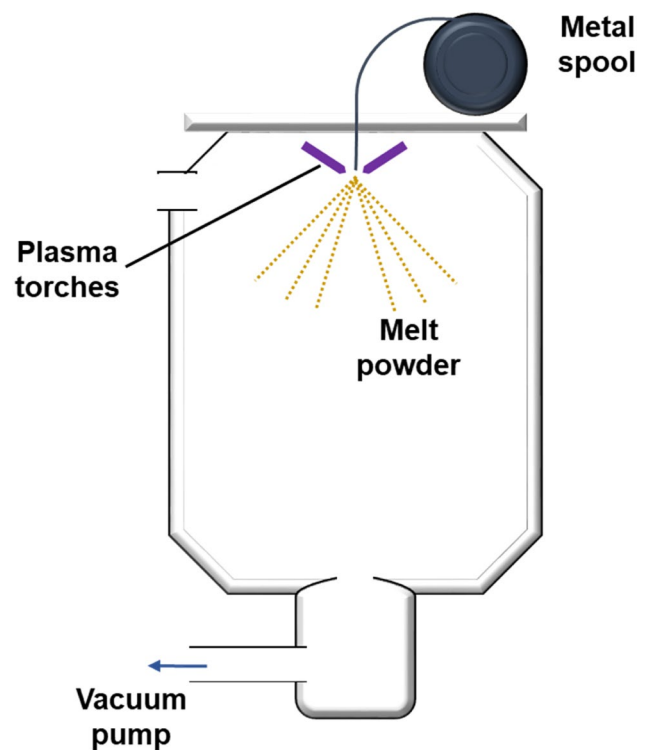


Fig. 7 Schematic diagram of the PA process for AM

which is approximately 15% for FFGA. Close coupled gas atomization (CCGA) is the further developed technique which is used to target the fine particles with a size of less than $45\ \mu\text{m}$ and to increase the production yield and to enhance the atomization efficiency [105, 106]. Y_2O_3 -W-YSZ composite tubes were used to Ti powder with CCGA. However, to knowledge no studies have yet reported the powder yield of fine Ti powder [99].

In addition, with high reactivity of titanium with ceramics and metals, electrode induction gas atomization (EIGA) technology was developed to make ceramic contamination-free powder by ensuring that the melt does not come into contact with any metal or ceramic parts [107]. A rod is melted in cone-shaped induction coil while in slow rotation which is later atomized into small droplets. The diameter of the rod can be increased from the initial 25–70 mm up to 90–120 mm to increase productivity [100]. The atomization apparatus coated with Ti in the inner walls and other walls of the flow path was developed to minimize contamination during atomization [108].

3.2 Plasma atomization (PA)

Plasma atomization was first developed in 1990s to make fine, spherical powder [109, 110]. As shown in Fig. 7, it uses pre-alloyed wire as a feed material which is melted using high velocity plasma torches and broken into droplets

simultaneously. It is capable of producing high purity powder since the melted droplets do not come into contact with any metal or other materials prior to solidifying unlike the gas atomization process. The cooling rate is typically around $100\text{--}1000\text{ }^{\circ}\text{C s}^{-1}$ with the particle size range of $25\text{--}250\text{ }\mu\text{m}$ [99]. Generally, compared to the powder produced with gas atomization, the yield of fine powder and purity are higher [99, 111].

The morphology of plasma atomized particles are high quality spheres, and they tend to have fewer satellite particles than gas atomized particles. However, the issues of potential residual gas pores within the produced particle and existence of satellites are still of concern. The restriction in the type of feedstock usable for the process pose limitations to plasma atomization studies. It increases the manufacturing cost since wire forms are generally more expensive and also limits the experimentation of materials that do not exist in wire form such as Ti_3Al [100]. Factors that can be adjusted to alter the yield of fine particles and the capacity include the following: diameter of wire, feed rate of wire, gas pressure, and the angle and distance between the wire and plasma outlet [100, 111]. A study also reported that an induction coil added plasma apparatus can improve the production rate [111].

3.3 Plasma rotating electrode process (PREP)

The plasma rotating electrode process was developed by modifying the rotating electrode process (REP) developed in the 1960s by Nuclear Metals/Starmet [63, 111] by replacing the heat source with a transferred arc plasma torch to avoid tungsten contamination. The PREP is carried out in stainless steel chamber. A rapidly rotating metal electrode rod is melted in helium environment with helium plasma torch and with the centrifugal force from the rotation. Helium is chosen for its heat transfer and electric arc properties [111]. The rotation speed is generally between 3000 and 15,000 rpm. The melted liquid spins off and forms droplets which solidifies in flight as shown in Fig. 8 [77, 103]. The particle size of the produced Ti–6Al–4V powder is approximately $100\text{--}300\text{ }\mu\text{m}$ [103].

The process is one of the common methods to make spherical Ti alloy powders which possess good packing and flow properties. The apparatus design allows for the final product to have high purity from avoiding contact with other materials prior to solidifying. In addition, interstitial impurities such as oxygen and nitrogen is minimal during the manufacturing process due to its relatively large size and low specific surface area [111]. Since the process does not involve high-pressure gas, there are close to no gas pores in the powder particles. In addition, the droplets formed fly away from the melted surface without back flow of fine particles. Therefore, the possibility of fine particles from colliding

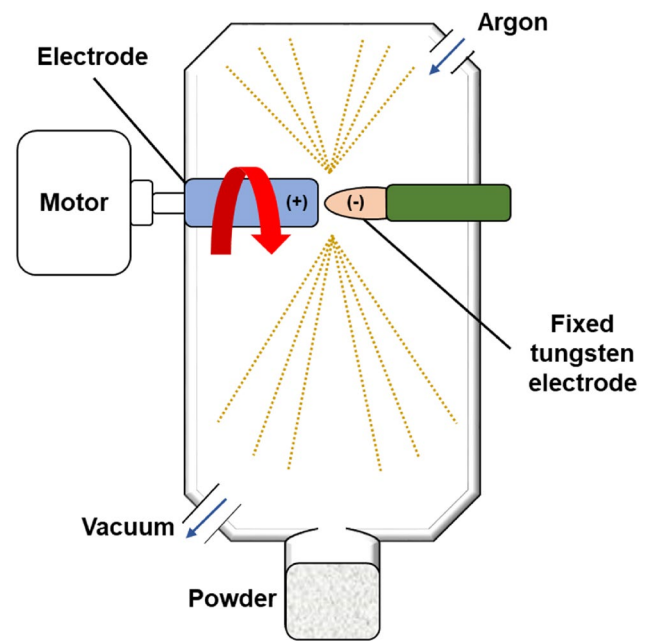


Fig. 8 Schematic diagram of the PREP process for AM

with other particles is very low, consequently lowering the possibility of having satellite particles [112]. Moreover, the yield of finer powder can be increased by increasing the rotation speed and the diameter of the electrode [113]. Yet, one of the drawbacks for PREP is that the size of the produced particles is suitable for powder metallurgy involving hot isostatic pressing but it is not suitable for powder-bed-based AM.

4 Conclusion

The development and progress in additive manufacturing techniques of metals have played an important role in manufacturing customized near-net-shape orthopedic and dental implants that are not possible through other fabrication techniques. In this review, commonly used powder-based additive manufacturing processes which include PBF-LB/M, PBF-EB/M, DED, and binder jetting are discussed. In addition, some of the common powder production techniques to make spherical titanium-based powder which are preferable for metal-AM are presented. These include GA, PA, and PREP.

Although significant progress has been made in the metal AM technology allowing fabrication of complex structures, each technique has its own advantages and disadvantages in terms of cost, accuracy, quality, and speed. In order for metal AM technology to be more widely used in medical applications, an optimized processing method that is capable of printing with high accuracy, high quality, high output,

low defect, and low cost is needed. In addition, further study on the effects of processing parameters on the structure and mechanical stability is needed to predict and optimize manufacturing strategies. Moreover, the performance of the parts also depend on the quality of the starting powder in which spherical powder is preferred. The above mentioned methods which are used to make spherical powder all have advantages and disadvantages as well. There is still room for improvement to reduce the cost and to minimize possible pores, contamination, and satellite particles.

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Compliance with ethical standards

Conflict of interest The authors declare that they do not have any conflicts of interest.

Ethical Approval This article does not contain any studies with human participants or animals performed by the author.

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