1

Overview of Atomization Techniques

1.1 History of Metallic Powder and Atomization Techniques

1.1.1 Metal Powders

The high surface-to-volume ratio (i.e. volume per surface area) of a sphere renders it an ideal geometry for handling, pressing, forming, and/or reacting particles into a bulk form. It is hence not surprising that **metal powders**, in their most elementary form, emerged as useful "engineering" materials, even in ancient times. **Metal powders** can be produced using either chemical/**mechanical methods** or **fluid atomization**. Mechanical methods involve the physical breakdown of a large particle into a smaller one, whereas **chemical methods** typically involve a solid-state process in which a metal oxide is reduced into a metal. For example, the reduction of iron, also called sponge iron, occurs when iron ore is exposed to a reducing gas. Powders produced using chemical reduction methods are not widely used in **additive manufacturing (AM)** and hence are not covered in this chapter. There is some preliminary research on the use of powders produced by mechanical methods in AM, and these will be discussed in a subsequent chapter.

Powder metallurgy, as the practice of working with metal powders is widely known, has a rich history, from the Incas using powder techniques to work precious metals to ancient Egypt, Africa, and India (see Table 1.1). In fact, the use of elevated temperature metals can be traced back about 7 000 years, long before furnaces capable of reaching the temperature required for melting them. Hence, it has been suggested that objects made of iron about 5 000 years ago were likely fabricated using powder metals. The first application of a powder metal product was a bronze ball bearing, capable of self-lubrication and used for automotive applications in 1927 [1].

The Oxford dictionary defines the word ***atomize*** as to reduce something into atoms or into very small pieces. Clearly, based on this definition, one can hypothesize that the term atomization has been loosely applied to the disintegration of any bulk material into smaller components. One can envision the disintegration of water as it emerges from a nozzle or waterfall into droplets or similarly the disintegration of rock by mechanical means into very small pieces. Fluid atomization, however, is generally described as a process where a liquid metal is disrupted by a high-velocity fluid such as air, nitrogen, argon, helium, or water in some cases. The actual process of atomization occurs when there is a transfer of **kinetic energy** from the atomizing medium to the metal being disintegrated. There are different forces in action during atomization that depend on the fluid being used for the energy transfer. In the case of water, for example, it is typically the pressure of the medium that dictates the efficiency of the atomization process and thereby the resultant distribution of droplets that emerges during disintegration. In the case of **gas atomization (GA)**, however, it is typically found that the gas-to-metal ratio dominates the resulting distribution of droplets, and various mathematical relationships have been developed to capture this relationship. In the case of gases, for example, increases in pressure that exceed 0.1 MPa (this is the pressure at which a **sonic velocity** is reached) result in only very small increments in gas velocity. In comparison, to reach sonic velocity with a water jet, for example, a pressure of nearly 40 MPa is needed in the case of water, and velocity continues to increase as the square root of the pressure.

There are various methods that can be effectively used to produce metal powders from valuable metals and alloys such as **nickel**, **cobalt**, copper, titanium, aluminum, and stainless steel base alloys. However, GA is generally viewed as the method of choice due to its capacity for large production quantities, the ability to control chemistry, and the geometrical properties of the powder particles that can be produced. These characteristics render atomization as the preferred technique to manufacture powders for AM, although, as we will discuss in subsequent chapters, alternative methods for powder preparation are being explored.

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Table 1.1 Major Historical Developments in Powder Metallurgy.

Date	Development	Origin
3000 BCE	"Sponge iron" for making tools	Egypt, Africa, India
1200 CE	Cementing platinum grains	South America (Incas)
1781	Fusible platinum-arsenic alloy	France, Germany
1790	Production of platinum-arsenic chemical vessels commercially	France
1822	Platinum powder formed into a solid ingot	France
1826	High-temperature sintering of platinum powder compacts on a commercial basis	Russia
1829	Wollaston method of producing compact platinum from platinum sponge (basis for the modern PM technique)	England
1830	Sintering compacts of various metals	Europe
1859	Platinum fusion process	
1870	Patent for bearing materials made from metal powders (the forerunner of self-lubricating bearings)	United States
1878–1900	Incandescent lamp filaments	United States
1915–1930	Cemented carbides	Germany
Early 1900s	Composite metals	United States
	Porous metals and metallic filters	United States
1920s	Self-lubricating bearings (used commercially)	United States
1940s	Iron powder technology	Europe
1950s and 1960s	Powder metallurgy wrought and dispersion-strengthened products, including powder forgings	United States
1970s	**Hot isostatic pressing**, PM tool steels, and superplastic superalloys	United States
1980s	Rapid solidification, powder injection molding technology, and binder-treated ferrous premixes	United States and Europe
1990s	Intermetallics, metal–matrix composites, spray forming, nanoscale powders, water-atomized pre-alloyed ferrous powders with **molybdenum** as the principal alloying element, and warm compaction	United States and United Kingdom
2000s	Warm-die compaction, additive manufacturing (3D printing) on a commercial basis	United States and Europe

Source: Samal and Newkirk [1] / ASM International.

1.1.2 Atomizer Designs

A critical aspect of atomization technology involves **atomizer design**. The atomizer is used to disintegrate the molten materials into a dispersion of droplets, which eventually solidify into powders, and therefore different designs result in various distributions of droplet (and hence powder) sizes. Since size distribution is an important variable that governs the solidification behavior of droplets, atomizer design directly influences the resultant microstructure of the powder, and hence the final properties of the consolidated material. Historically, a variety of atomizer designs have been developed to various degrees of success. There is, however, no general agreement among the scientific community as to the optimal atomizer design; more likely, the design used depends on the desired powder characteristics and the ultimate intended application of the powder. Moreover, and as will be addressed in a later chapter, optimal processing parameters and atomizer design are often material-specific (these can change even for slight compositional variations).

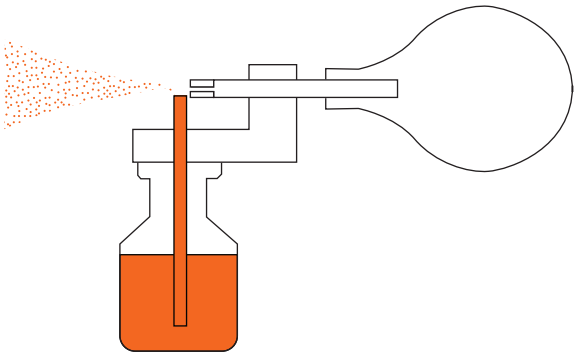


Figure 1.1 Schematic diagram showing design of first "atomizer". Source: De Vilbiss [2] / United States Patent and Trademark office / Public Domain.

The design of an atomizer is based on airflow dynamics; as horizontal air travels over a vertical tube, it creates a suction effect and aspirates the air and liquid in the tube. In essence, an atomizer uses Bernoulli's principle to disintegrate a liquid into a mist of fine droplets. The first atomizer nozzle is described as an **aspirator nozzle** and is credited to Dr. Thomas DeVilbliss of Toledo, Ohio, in the late nineteenth century. He invented this device to spray medicine in the back of patients' throats [2, 3], as shown in Figure 1.1. Not long after Dr. DeVilbliss used the device shown schematically in Figure 1.1 for medical purposes, the first **perfume atomizers** emerged in 1907, and a new industry was born.

In the case of metals, the first atomizer design is credited to Hall, who used it to atomize aluminum powders in the late 1920s [4]. *In the design proposed by Hall, compressed air is used as the atomization media,* and it is delivered via an **annular orifice** at a predetermined **convergent angle** (see Figure 1.2). As the gas flows through the **annular orifice**, it creates an aspiration effect that suctions the molten metal into the nozzle cavity. The amount of gas can be changed by changing the size of the air gap or the temperature and pressure of the gas.

The **Hall nozzle** design falls into a category of nozzle designs known as confined nozzles or close-coupled nozzles. This nozzle design involves closely coupling the liquid metal stream with the gas jets to maximize the transfer of energy from the atomization medium (gas) to the metal being disintegrated. A typical **closed-type atomizer** is illustrated schematically in Figure 1.3 [5]. It normally contains 10–18 gas jets symmetrically arranged around a cavity designed to contain the metal delivery tube. Typically, the gas nozzles are positioned at 1.0–1.5 diameters apart, depending on the desired powder characteristics. The geometry of the gas nozzles is designed to provide maximum

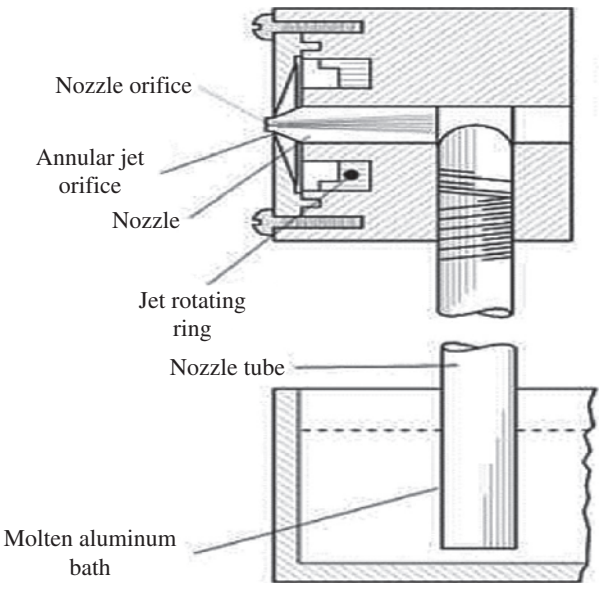


Figure 1.2 Aspirating nozzle design developed by E.J. Hall in 1920s. Source: Hall [4] / United States Patent and Trademark office / Public Domain.

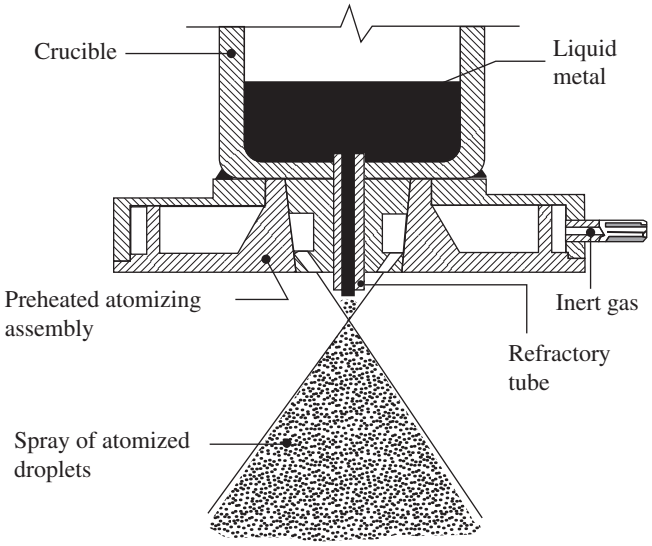


Figure 1.3 Closed-type atomizer used for spray deposition processing. Source: Singer [5].

kinetic energy to the atomization gas, thereby leading to an "efficient" atomization. As shown in Figure 1.3, the primary advantage of a closed-type atomizer is that the molten metal is delivered directly to the atomization region, thereby avoiding instability of the metal stream and producing a stable atomized droplet spray. In confined designs, the molten metal travels a very short distance before being impinged upon by the high-energy atomization gas jets. The major problem that is associated with this type of atomizer is that it is very sensitive to metal freeze-up, a condition that occurs when the liquid metal at the end of the delivery tube solidifies prematurely because of the rapid heat extraction that occurs in this region. Accordingly, careful control of atomization parameters must be exercised when this type of atomizer is utilized [6]. During atomization with confined nozzles, it is frequently reported that the metal travels on the surface of the metal delivery tube, such that when it interacts with the atomization gas, it is effectively a thin film. This filming effect is beneficial for the efficient breakup of the liquid, and its formation is related to the gas flow dynamics, as illustrated in Figure 1.4.

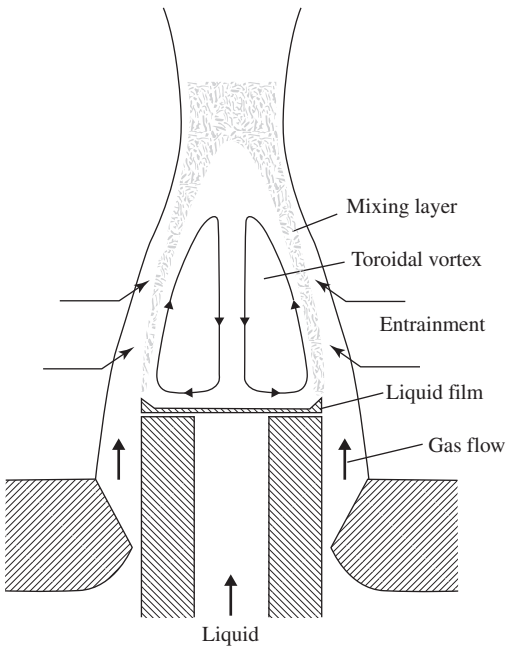


Figure 1.4 **Fluid flow pattern** at the tip of a confined nozzle during atomization. Source: Unal [7] / Taylor & Francis.

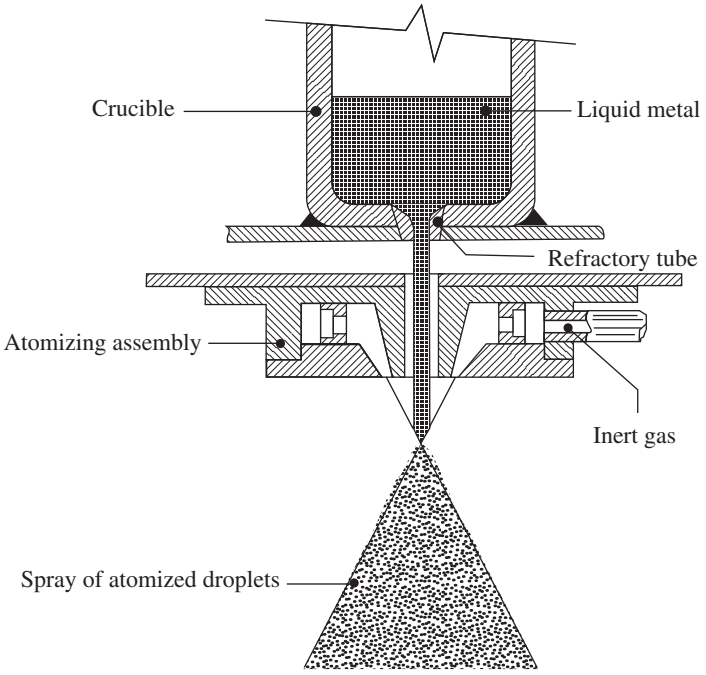


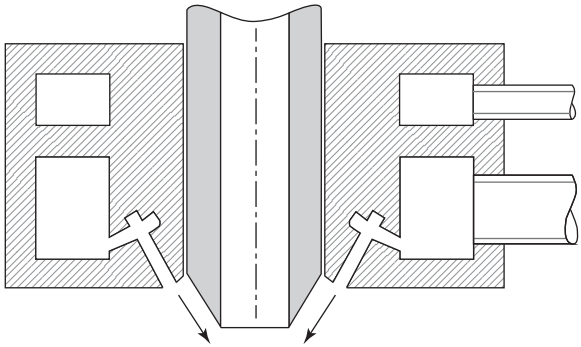
Figure 1.5 Open-type atomizer used for spray deposition processing. Source: Singer [5].

The toroidal recirculation vortexes that form in confined nozzles originate from fluid entrainment that occurs on the inner boundaries of an annular jet [7, 8]. As a jet exits an annular cavity, there is a mixing zone that forms as the gas jet "shears" the molten metal; there, the jet pulls fluid from the mainstream jet. An important consequence of this vortex is the formation of a low-pressure zone at the tip of the nozzle; this can aspirate metal during atomization, thereby leading to changes in the gas-to-metal flow ratio.

In view of the difficulties associated with the **metal freeze-up phenomenon**, many researchers use an open-type atomizer [5, 9, 10]. A schematic diagram of such an atomizer is illustrated in Figure 1.5. An open-type atomizer is also referred to in the literature as having a free-fall design. In the open-type atomizer, liquid metal is allowed to fall freely to the atomization point (i.e. the focal point of the atomization gas jets). Since the metal delivery nozzle tube is not in direct contact with the atomization gas jets, nozzle freeze-up is avoided. The principal problem that is associated with open-type atomizers, however, is the instability of the molten metal stream, which leads to oscillation and asymmetry of the atomization cone. This instability occurs because the free-falling molten metal stream is easily deflected by the turbulence in the surrounding gas envelope.

In the 1980s, researchers used a so-called **ultrasonic atomizer** [11, 12] for GA. The **USGA nozzle** makes use of the theory that a high-frequency (>100 Hz) pulsed gas stream with high-amplitude (primary and secondary) **shock waves** will promote the efficient atomization of molten metals [13, 14] (see Figure 1.6). Accordingly, a narrow distribution of droplets may be produced with relatively small average droplet size. The design of an ultrasonic GA

Figure 1.6 Schematic of USGA nozzle design. Source: Gerking [15].



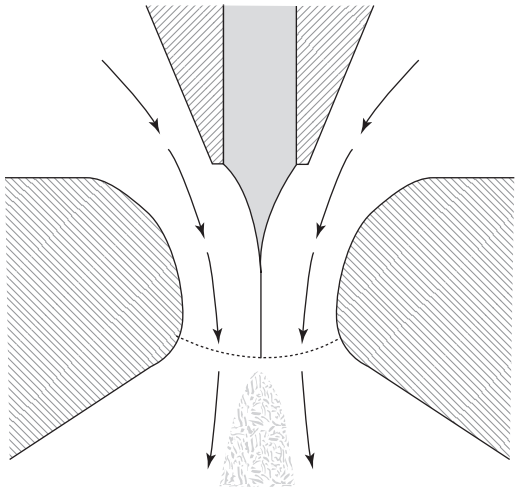


Figure 1.7 Schematic of Nanoval nozzle design. Source: Miller et al. [20].

nozzle incorporates a **Hartmann resonant chamber** to generate high-frequency and high-velocity pulsed gas jets [13]. When operating under proper atomization conditions, the USGA atomizer can generate gas jets of supersonic velocity superimposed with ultrasonic pulses [14]. Hence, it has been argued, albeit not without controversy, that the atomization efficiency that may be achieved with this atomizer design exceeds that of conventional atomizers, thereby allowing close control over the **droplet** size distribution [14, 16, 17].

Other atomizer designs include the National Institute of Science and Technology (NIST) design, which is like the USGA nozzle but does not include the Hartmann cavities [18, 19], and the **Nanoval design** (see Figure 1.7), in which molten metal is delivered directly to the throat of a converging–diverging nozzle in the form of a thin stream [15].

1.2 Melt Atomization

Melt atomization is perhaps the most promising and economic technique for the fabrication of fine and spherical metallic powders used for conventional powder metallurgy and metal AM. The mechanism for melt atomization involves the disintegration of a melt film or a stream into droplets by the application of energy with high-velocity fluids, such as gases (air, **nitrogen**, **argon**, and helium), water, and synthetic oils, and even combinations of two fluids have been used to fabricate fiber powders. Inert gas atomization (IGA), also referred to as GA, has become extremely popular and is increasingly being used to produce fine, high-purity spherical powders in large quantities for the **stock powder** used in AM.

There are other atomization techniques that are not based on the principle of melt disintegration by impingement with a second fluid, such as the use of **centrifugal forces** in **centrifugal shot casting**, **vibrational energy**, **electrical fields**, **melt extraction**, **rotating consumable electrode**, plasma micro-atomization, **melt saturation** and **explosion**, and capillary and **standing wave atomization** [21]. These processes are generally being used to produce special powders for refractory or reactive metals used in advanced materials.

The primary advantage of the melt atomization process lies in its flexibility in terms of producing the desired alloy chemistry and particle size. The process can produce small quantities of experimental powders and can also be scaled up to produce large quantities of powder for commercial applications. The average particle size (ranging 5–250 μm) and cooling rates (ranging 10^2–10^6 K s^{-1}) can be controlled depending on the processing parameters for tailoring the microstructure of produced powder.

Atomization is suitable for producing powders of metals and alloys where the melting point of the material is not too high. High-temperature materials like **tungsten**, **hafnium**, **molybdenum**, and **ceramic** are not usually produced by melt atomization, except by specialized processes such as **plasma atomization**.

Contamination may occur due to the crucible (usually a graphite or ceramic crucible) in which the atomization melt is contained. This problem becomes extremely acute for processing high-temperature and high-performance materials, such as Ti alloys. One way to avoid that problem is to produce a thin "skull" of the same material that will be atomized within the crucible. The "skull" can effectively insulate the molten materials from the crucible and thus

reduce contamination to a great extent. Since the crucibles, in which the "skull" is formed, need to be cooled, they are made of highly thermally conductive metals, like **copper**. Another technique is to have no crucible at all and use the materials to be atomized as an electrode that is gradually melted and atomized. In addition, another alternative is to use ceramic filters to filter out large ceramic particles from the melt before atomization.

The basic concept in the common atomization processes is to deliver energy to the liquid melt via the impinging fluid. Variations in the fluid focusing system and the type of fluid used determine the final powder characteristics. Subsequent sections of this chapter will briefly describe various melt atomization processes that are presently in use and some of the new processes that are being developed to produce finer and spherical powders. The requirements of the powder used for AM are also providing a large impetus to the melt atomization processes to provide fine and spherical powders that are suitable for AM process.

1.3 Gas Atomization (GA)

Melt atomization is the dominant method used commercially to produce metal and alloy powders from Al, Cu, Fe, Ti, Ni, and other alloys, because high production rates favor economies of scale. Atomization, as the name implies, involves the energetic disintegration of a liquid into micrometer-sized droplets, as shown in Figure 1.1. The principle of melt atomization is ancient and formed the basis for a British patent in 1872 to produce **lead powders** by drawing off and spraying molten lead using a stream injector [21]. Since the first large-scale production of atomized iron powder during World War II, melt atomization technology has been steadily implemented and improved, partly due to the widespread application of pre-alloyed powders.

Air atomization is where both atomization and melting are carried out in the air. Compressed air jets are used to break up the molten metal, and often cooling is achieved by sucking large volumes of cooling air through the equipment. This method has been widely used since ~1900 to make tin, lead, **aluminum**, and zinc powders, but also for some copper and copper alloys. It was used for processing Fe–C alloys in the middle of the twentieth century but is now obsolete. As would be expected, oxygen contents of air-atomized powders are relatively high, typically in the range of 1000 ppm to 1%. Particle shapes range from irregular for Al and Zn to spheroidal for copper, gold, and silver. Particle sizes for Ni and Fe alloys are ~50–200 μm and for Sn and Pb are ~10–30 μm.

IGA is where the melt is broken up by inert gas, normally nitrogen or **argon**, and thus protected from oxidation. This was developed in the post-World War II period with melting in air and used for a very wide range of alloys, ranging from Sn and Pb to Au, Ag, Cu, Co, Ni, and Fe alloys. The major fields of application for this technique have been powders for **thermal spraying**, **hard facing**, **brazing**, **dentistry**, and more recently, **hot isostatic pressing (HIP)**, **metal injection molding (MIM)**, and AM. Oxygen contents can be far lower than in air-atomized powders, typically in the range of 100–500 ppm, but highly alloy-dependent. Particle morphologies are typically spherical, but agglomeration and satellite formation can occur. Median particle sizes are typically 30–200 μm for Fe, Ni, and Co alloys with the upper limit dictated by the large vessel sizes needed to avoid splatting of still-molten larger droplets on the walls.

As a type of melt atomization, GA is the process where the molten metal is disrupted by a high-velocity gas. Subsonic, supersonic, and ultrasonic GA utilize different gas velocity jets, as implied by the names used to describe the techniques. During GA, a high-pressure atomization gas, such as air, nitrogen, argon, or helium, is discharged from the pressure reservoir of the atomizer into a chamber, which is typically maintained at a low environmental pressure. The compressible atomization gas is accelerated to a high velocity as it expands from the high reservoir pressure into a low-pressure chamber. The energetic impingement of a high-velocity gas jet causes the molten metal steam to deform and disintegrate into fine droplets. The **gas-to-metal ratio** is an important factor that governs particle size for GA, rather than being dominated by the pressure of the medium like in the case of water atomization.

GA involves the disintegration of an unconfined stream of liquid metal into fine particles by a focused jet or jets of atomizing gas. The design of the nozzle and the liquid-metal feed mechanism often varies. The gas jet can be directed through several discrete openings or through an **annular-ring** type of opening. There are two types of atomization nozzle design – "free-fall" and "confined." A "free-fall" configuration is often used in the conventional GA technique. In this technique, the gas jet(s) strikes the metal stream at a point that is some distance away from the point where the liquid metal leaves the crucible (i.e. flow becomes unconfined). Problems arise from the stream instability when very high gas velocities and sharp jet angles are used to try to produce finer particles. This conventional type of GA can produce powders with a mean particle size usually greater than 50 μm.

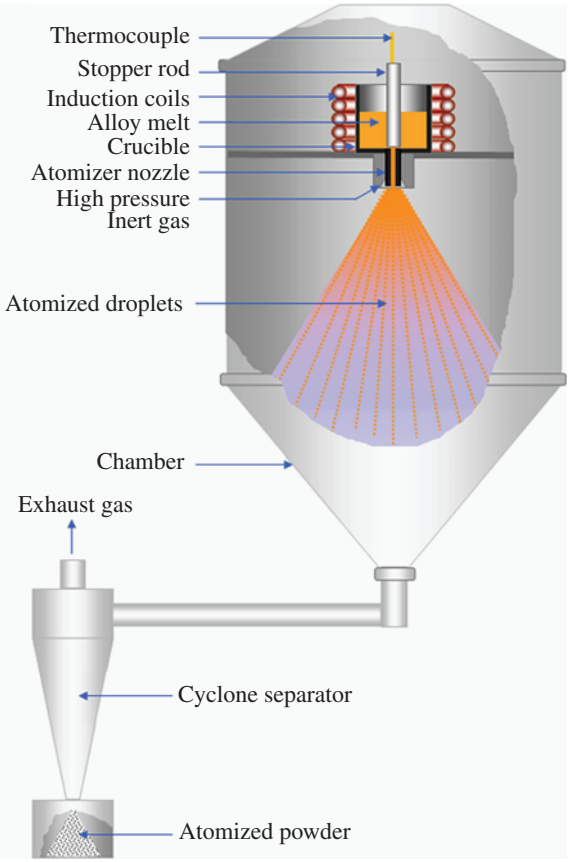


Figure 1.8 Schematic diagram of gas atomization structure and processing. Source: Adapted from Zheng et al. [22].

The liquid stream is disintegrated in a horizontal or vertical direction during GA processing. Most GA processes rely on the vertical direction of liquid metal flow. The horizontal gas atomizers can normally be used for low-temperature metals and alloys. The molten metal is siphoned up through a feed tube and disintegrated by high-velocity gas. Vertical GA is normally used for high-temperature materials, with inert gases being used as the atomization fluid. A schematic of the conventional vertical type of gas atomizer is shown in Figure 1.8.

The chamber size should be large enough so that the particles produced can have free flight through the chamber and solidify prior to reaching the collection chamber wall. In this process, the goal is to generate fine particle sizes (around the 10 μm range). Finer particles can be generated by higher gas flow rates, higher melt temperatures, and steep angles between the gas jet and the melt stream. The **thermal conductivity** of the atomizing gas, the metal surface energy, and the atomizer nozzle design all play important roles in determining the final powder characteristics.

The commonly accepted mechanism for conventional GA involves the formation of a stable sheet that later becomes wavy in nature, although as will be discussed in the next chapter, many questions remain. Cylindrical ligaments are torn off from the sheet, and these in turn break up into number of small, elongated droplets that then become spherical. In fact, the process of GA is very complex and far from being completely understood. Nevertheless, it is well known that the powder particle size distribution depends on a variety of different parameters. One set of parameters is related to the physical properties of the melt, e.g. density and temperature, which have an influence on viscosity and surface tension. Another set of parameters is related to the gas flow field, which depends, e.g. on the geometry of the gas nozzle, the type of atomization gas, and the gas pressure.

To achieve smaller average particle sizes and rapid solidification (RS) rates, close-coupled (also known as confined or direct jet) GA is the preferred route [23]. In this case, the gas jet impinges on the stream of molten metal at the point where it emerges from the melt guide tube, atomization nozzle. This results in the most efficient transfer of the gas kinetic energy to the liquid melt, which is the key to the formation of fine powders. Little of the gas energy is lost in turbulence as in the case of an open-die or **free-fall arrangement**. Also, a higher angle of impingement is possible, thus improving the fine particle yield. The major challenges are due to the proximity of the gas jet to the molten metal guide tube.

High-pressure gas atomization (HPGA) was a natural evolution of the normal GA process. The necessity to develop finer powders and produce fast cooling rates resulted in the development of the HPGA process. It was reported that using 573 κ (300 °C) superheat, 4.5 MPa argon atomizing pressure, and a 2.5-mm-diameter pour tube was optimum for atomizing copper [24]. HPGA of copper, amalgam, and a lead–tin alloy exhibited mean particle sizes of 25, 20, and 15 μm, respectively. From the point of view of the large demand for finer powders, this is also a very important development.

With increasing demand for finer powders, the powder producers are adopting new techniques that could yield a higher percentage of finer powders. Research and development effort in this area has therefore increased tremendously. The need for producing fine particle size with a narrow size distribution has resulted in the development of ultrasonic GA. This form of atomization uses high-frequency, supersonic gas jets as the atomizing medium. The production of the ultrasonic waves is accomplished by accelerating high-pressure gas through a resonance cavity. The passage of the highly pressurized gas such as nitrogen, argon, or **helium** through the resonance cavities results in the attainment of ultrasonic frequencies of 80–100 kHz and supersonic gas velocities often reaching Mach 1.7 – 2.5 [25, 26]. A typical ultrasonic gas atomizer uses around 16–20 resonating cavities that direct the jets to a circle of small radius surrounding the liquid metal stream. Various investigators have used the ultrasonic GA process to produce rapidly solidified powders of different metals and alloys.

Given the high velocity of the gas pulse, the mechanism for this form of atomization does not follow the usual ligament formation mechanism that has been proposed for conventional GA. Instead, the liquid metal stream behaves like an impacted low-shear-strength solid. Despite some progress, it is apparent that more research is needed to complete our understanding of ultrasonic GA.

Among the methods of powder production, the proportion of powder produced by GA has accounted for 80% of the total. The GA method can be divided into the following types according to the different heating elements of the equipment: **vacuum induction melting inert gas atomization (VIGA)**, **plasma melting induction gas atomization (PIGA**), **plasma torch atomization (PA)**, and **electrode induction melting gas atomization (EIMGA**/EIGA).

In the VIGA process, the melting materials may easily be contaminated by the crucible, which is particularly challenging for high-purity reactive metals such as Ti. EIGA can avoid the contamination from the crucible by using ceramic-free electrode induction, but it is limited to small-size feedstocks and low-melt flow rates. PIGA can be used to prepare Ti and Ti alloys. However, considerable electric powder is required in this technique; thus, complex safety precautions are required due to much more liquid volume involved as compared to EIGA. The plasma rotational electrode process (PREP) technique can also be used to prepare the spherical Ti powder, but the powders generally have a wide PSD [26]. Generally, the above methods can be used to prepare Ti powders with good sphericity. However, it remains challenging for these methods to obtain fine Ti powders with a narrow particle size distribution (PSD) in a single batch.

1.4 Vacuum Induction Gas Atomization (VIGA)

Vacuum inert gas atomization (VIGA) involves the melting and pouring of an alloy prior to atomization inside of a vacuum chamber to allow the production of the most oxidation-sensitive and reactive alloys, such as Fe-, Ni-, and Co-based alloys containing Al, Ti, and rare earths. This includes superalloys, such as **IN718**, **maraging steels**, and **M-Cr-Al-Y alloys**. This technique was developed during the 1950s and 1960s when there was a push to explore the potential benefits of RS to allow the production of more highly alloyed superalloys for aerospace and defense applications [27]. This proved to be a very challenging field of application, but, after several decades of development, there are now many thousands of tons per year of VIGA-produced superalloy powders. This intensive development has meant that the technology lends itself well to producing powders for HIP, MIM, and AM. Oxygen contents in the 50–200 ppm range are achievable. Particle morphology is generally spherical, and the size distribution is comparable to that obtained by IGA.

The VIGA atomization system generally consists of three production stages: (i) melting in vacuum induction melting furnace; (ii) transfer of the molten metal or alloy to a crucible; and (iii) powder atomization by pressurized gas resulting in a fine dispersion of powders. In the VIGA system, a vacuum induction melting unit is integrated with an IGA unit. The starting raw materials are heated and melted by frequency induction furnaces using electromagnetic induction, which couples electrical power into the crucible/material under vacuum atmosphere. This design

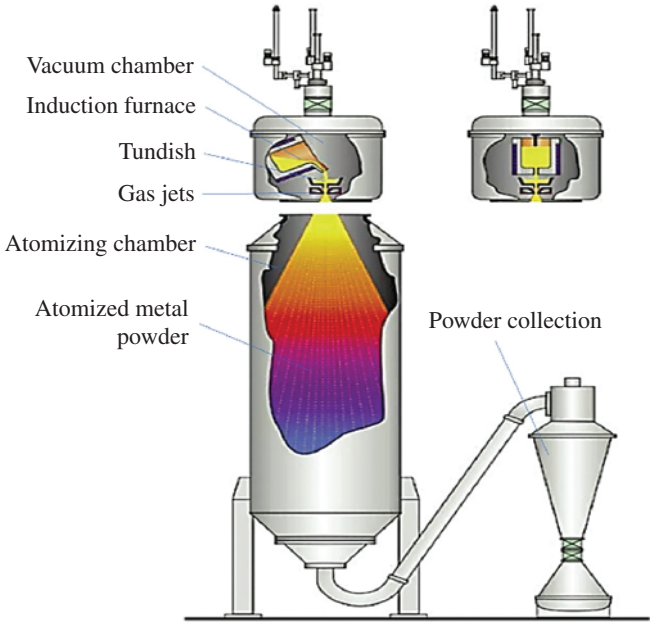


Figure 1.9 Schematic diagram of vacuum induction gas atomization (VIGA) processing. Source: ALD Vacuum Technologies AG [28].

allows the metal alloys to attain the lowest oxygen content possible. Once the desired melt homogeneity and chemical composition have been achieved, the material is poured into a tundish by crucible tilting, as shown in Figure 1.9. The equipment directs the liquid metal to focus the gas jet. The fine metal stream, flowing from the tundish orifice into the atomization nozzle system, is subjected to a high-pressure, inert gas jet and then atomized. The combination of molten metal and gas jet impacts, atomizes, breaks up molten metal into small droplets, and creates a spray of micron-sized droplets that solidify in the atomization chamber located directly underneath the atomization nozzle, and the droplets eventually solidify into spherical morphology. The VIGA process provides excellent control over alloy powder composition and oxygen content.

Two main atomization stages, including primary atomization and secondary atomization, are generally involved in vacuum induction melting GA. The primary atomization refers to the cylindrical flow of high-temperature molten alloy flowing out of the delivery tube, which interacts with the high-speed gas flow field, disperses, forms a liquid film, and then breaks into an initial droplet dispersion. Secondary atomization, also known as the second crushing, refers to the process that occurs as the droplets that are initially formed are entrained by the airflow into the turbulent layer, that is, the process of secondary crushing and additional crushing into smaller droplets.

Due to the limitations of the hardware equipment and crucible, the heating temperature of vacuum induction melting is limited to the range of 1500–1600 °C. And due to the influence of the ceramic crucible and the guide nozzle, impurities will end up in the alloy melt, which will affect the purity of the prepared metal powder. Powders produced using VIGA typically fall in the 0–150 μm size range, and the gases that are used in the nozzle system are N_2, Ar, or He.

VIGA is one of the leading powder-making processes for the production of large volumes of a variety of high-performance spherical, high-grade metal powders, and essential for high-quality manufacturing of Ni-based superalloys, Fe-, Al-, Co-, Cr-based and other special alloy powders, as well as high-purity metal powders with low oxygen and low or adjusted nitrogen concentrations for use in AM, laser cladding, thermal spray, powder metallurgy, HIP, and other advanced manufacturing fields.

1.5 Electrode Induction Melting Gas Atomization (EIMGA)

High-quality metallic powders are in high demand to produce advanced materials that are required by many industries, including the automotive, medical, and aerospace industries, for example. In some cases, performance requirements include behavior at elevated temperatures, and reactive and refractory metals, intermetallic

compounds, and superalloys are examples of such high-temperature, high-performance materials that demand super-clean, low-oxygen-containing, rapidly solidified powders. IGA is well established as the most efficient and cost-effective approach for meeting the stringent requirements of these powders and producing them in the required production quantities. There are, however, some challenges associated with the production of atomized powders, including: ceramic impurity pickup due to the ceramic-lined crucible in which the material is melted prior to IGA and powder yield for a particular size requirement. Ceramic inclusions in these high-performance materials are a major cause of their failure during operating conditions and hence represent an important challenge. The production of reactive, high-melting-temperature metal powders requires a contactless technique avoiding direct contact with refractory crucible walls. The industry has adopted various ingenious approaches to address these challenges. A straightforward approach to address the ceramic inclusion problem is to eradicate the cause of the problem, which is the ceramic crucible. This approach leads to the concept of ceramic-crucible-free or even crucible-free melting coupled with IGA. Direct melting of an electrode by induction heating is a contactless technique suitable for **reactive high melting temperature alloys**, such as Ti, Zr, and Nb. The technique has found application in the production of Ti alloy powders by GA and is widely described as EIMGA [29, 30]. The EIMGA involves the advantageous features of induction melting and atomization of the master alloy without the use of melting crucible.

The EIMGA process can be described as essentially an induction drip-melting atomization process that utilizes a rotating electrode that gradually moves down through a specifically designed induction melting coil. The EIMGA process is a versatile method for converting a wide variety of metals and special alloy electrodes into metal powders. Notably, all metals and alloys with melting points up to 2500 °C can be converted into powders with the EIMGA process. Electrodes with up to 150 mm diameter and up to 1000 mm length with charge weights from 5 to 2000 kg can be used as feedstock.

The EIMGA technique requires a pre-alloyed rod. The desired material is first formed into an electrode and does not require a ceramic crucible. The lower end of the rod is dropped into a conical induction coil, and continuous melting occurs by melting from the surface to the center of the electrode. The end of the rod is heated up, and the melt drops into the center of a gas nozzle, where it is atomized by Ar gas. An electric drive controls the movement of the consuming electrode into the induction coil. The melt flow rate can be precisely controlled by the induction power applied and the downward velocity of the rod. The melt flow rates achievable for y-TiAl alloys can reach to around 50 kg h^{-1} [31]. To guarantee a symmetrical melting of the tip, the rod rotates at a low frequency of 5 rpm. In contrast to rotating electrode processes (REP) [32, 33], where a high rotational speed is used, for EIMGA the rods need not to be balanced.

The electrode bottom is immersed in an induction coil, where it melts and produces dripping or streaming liquid metal, as shown in Figure 1.10a,b. The dynamics of the melting process are complex, involving the dynamic adjustment of the electromagnetic field due to propagation of the melt front and motion relative to the coil. The melt flow is controlled by the melting rate, the gravity force in the liquid film, and the electromagnetic forces. The high-frequency coil current (100–200 kHz) attains the desired energy penetration on the surface of the electrode and creates the induced current layer in the electrode, which is spatially nonuniform along the surface. The arrangement of the induction coil is also of great importance. The special arrangement provides the desired superheat to the melt, which flows down the front-end section and is fed directly into the atomizing nozzle. The current flows in a thin skin and concentrates on the inner surfaces of the coil due to confinement of magnetic flux. The liquid film is confined by an electromagnetic "pinching" force, but the nonuniformity of this force along the surface creates a rather strong electromagnetically driven flow. This type of flow can, in some cases, oppose the dripping of the film flow due to gravity and lead to an interruption of the melt stream. A quasi-steady state is desirable, avoiding unstable dripping and maintaining a conically symmetrical shape at the electrode bottom. To stabilize the circumferential uniformity, the ingot is rotated at low angular velocities of 5–12 turns per minute. This means that angular momentum transport in the conical melt film flow must be included in the model.

The EIMGA system design is generally based on a vertically oriented, rotating electrode that is continuously melted at one end in an inert gas atmosphere without a ceramic crucible using controlled motion into a specially designed conical induction coil. The design of the induction coil and the frequency of the generator are optimized to achieve the necessary heat, causing the melt to drop from one end of the electrode. With proper controls, a continuous stream of molten material falls directly into the center of an annular slit. Then, the molten metal is transformed with an inert gas nozzle using a high-velocity gas stream to **atomize** the melt, as shown in Figure 1.10c. The atomization is carried

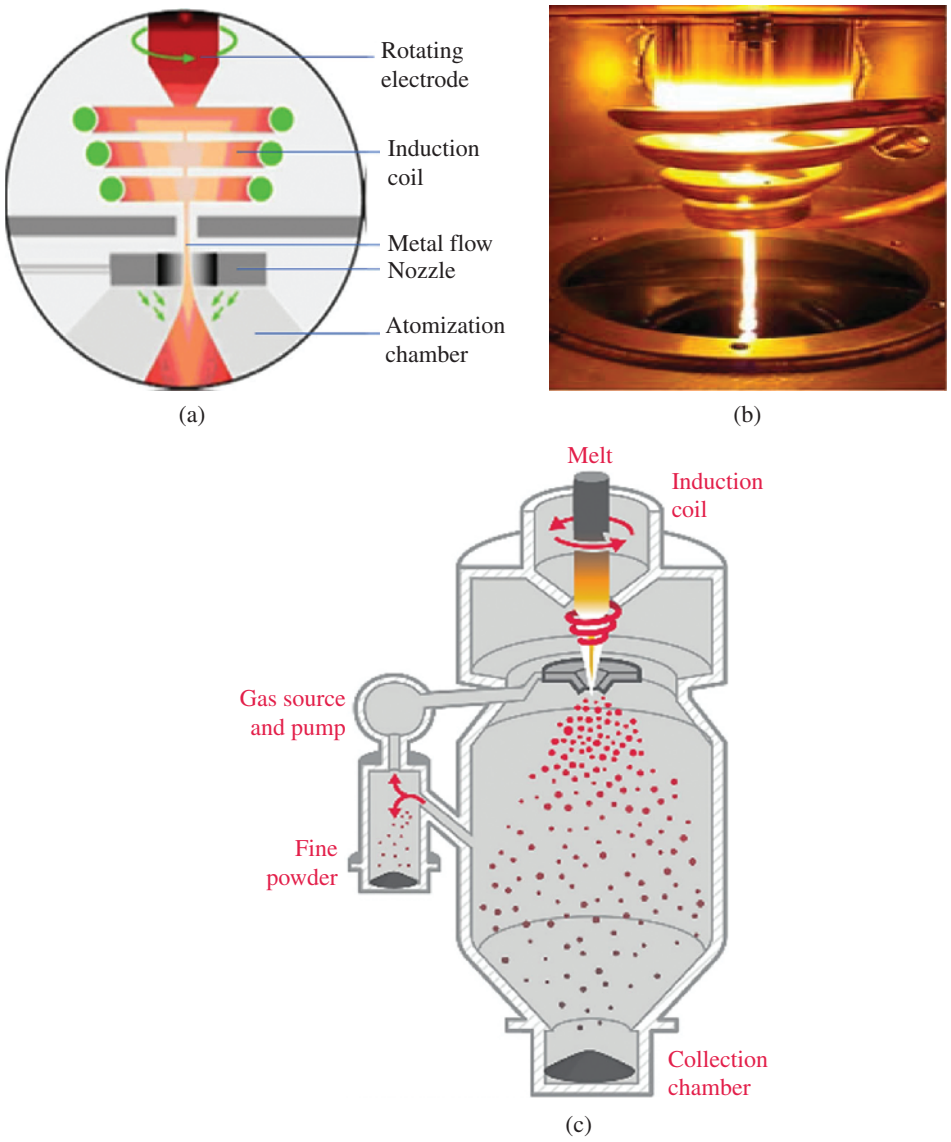


Figure 1.10 (a) Schematic of the electrode induction melting gas atomization (EIMGA) process for the production of metal powders. Source: LPW Technology, UK [34]. (b) Picture of melt liquid in an EIMGA process before atomization. Source: Ref. [35] / Arcast. (c) Schematic diagram of the EIMGA. Source: Lalwani [36].

out by a high-velocity inert gas stream. The micro-droplets spray solidifies while traveling down the atomization tower and forms spherically shaped fine powders which are collected in a vacuum-tight powder container.

Atomization of the molten metal is realized by the spraying action, resulting from injection of inert gas. Unlike other technologies, the EIMGA processing route is devoid of the use of ceramic crucible during melting of alloy, in addition to other advantages such as lower power consumption and simpler equipment setup. The superalloy powders prepared using such a crucible-free technology can prevent the introduction of Al_2O_3, SiO_2, and other ceramic impurities into the powder preparation process. It is also expected to reduce the quantity of inclusions in the powders, thereby improving the high-temperature performances of subsequently formed powder metallurgy superalloys. A further advantage of this technique is the small electric power required, since the liquid volumes are very small. This is also the reason why this technique is considered as very safe. A disadvantage is seen in the use of pre-alloyed rods.

The disadvantage associated with this process is the need to prefabricate electrodes from the desired materials. The cost of alloy prefabricated bars is higher than that of alloy ingots, and the uniformity of the bar alloy has a greater impact on the chemical composition of the powder after atomization. Segregation in the length of the rod can result

in chemically heterogeneous alloy powder. It is difficult to control the melting rate of metal bars. Be sure that it is easy to cause the flow of liquid to be cut off, and the "broken rod" will block the draft tube.

Powders produced with EIMGA systems typically have the following characteristics: spherical shape, good rheological flow characteristics, particle sizes ranging from $40\,\mu m \leq d50 \leq 100\,\mu m$, high purity, high flow ability, low O, N, and N concentrations, and rapidly solidified and homogeneous microstructure. The ceramic-free material processing and the outstanding process simplicity and robustness are the inherent advantages of the EIMGA process. The application-specific combination of melting and atomization techniques can enable high-quality manufacturing of a wide range of super-clean metal powders, including superalloys, precious metals, refractory and reactive metal powders, including Ti and Ti-alloys (e.g. CP-Ti, $TiAl_6V_4$, TiAl), Zr-, Nb-, Ta-base alloys, precious metals and their alloys, ultrapure reactive copper and aluminum alloys, and other specialty metals and alloys. Powders produced by the EIMGA process can be used in numerous applications. The main materials and application areas of powders produced with EIMGA systems are as follows: feedstock for AM technologies, aviation industry, power engineering, sputter targets, feedstock for MIM and HIP, electronics and chemical industries, biomedical engineering, and precious metal industry.

1.6 Plasma Rotating Electrode Process (PREP)

In this class of atomization technique, **centrifugal atomization** is perhaps the most notable. In centrifugal atomization, the breakup of the liquid occurs by thinning the metal into a film using centrifugal forces. This stands in contrast to conventional methods, where disintegration of a molten stream occurs via direct gas impingement. Centrifugal atomization, also sometimes described as **rotary atomization**, involves the delivery of metal onto a rapidly spinning disk that rotates at very high velocities. This technique has been widely used to prepare pre-alloyed powders of aluminum, zirconium, **titanium**, and iron, and various superalloys have been produced on a commercial scale.

PREP is also a centrifugal atomization process where a consumable metal bar is melted while being rotated rapidly about its longitudinal axis. As a first step, material of the target powder composition is fabricated into the geometry of a rod electrode that serves as an anode. This consumable rod electrode is then rotated at velocities that range in the 100's of revolutions per second and gradually moved in as the electrode is consumed. This displacement of the anode is carefully governed by external drives to ensure that the rapidly moving rod electrode remains spinning concentrically. As the metal is ejected from the rapidly spinning electrode, it travels in an inert atmosphere as it spheroidizes under the action of surface tension and hydrodynamic forces. The consumable electrode can be melted using a variety of energy sources. For example, in the case of titanium-alloyed powders in the United States, helium plasma is generally used [37] to melt the end of a rapidly rotating bar, and molten droplets are spun off and solidify in flight in a helium atmosphere. The melting and subsequent atomization are conducted in a circular stainless steel tank (such as 2.5 m in diameter) maintained at a positive pressure of helium. A plasma arc is used to melt the end of a rapidly rotating rod of metal material inside a chamber. Because the melting and solidification processes occur within an inert gas environment, they produce uniform metal particles that contain almost no impurities such as oxygen.

The high-speed rotating electrode (raw material) can also be melted using a plasma arc under the protection of the high-purity inert atmosphere. As the molten metal is ejected and travels in the inert gas environment, sometimes it impinges on the cold wall chamber prior to full spheroidization. The melting of the electrode at one end can also be accomplished using a stationary tungsten electrode. This, however, can result in small amounts of tungsten contamination in the powders.

During PREP processing, the metal or alloy is first manufactured into the self-consuming electrode bars. Then, a plasma arc is used to melt the end of the rapidly rotating bar, and molten droplets are spun off and solidify in flight in an insert atmosphere. The electrodes are pre-alloyed bars nominally of Φ60–65 mm in diameter and are rotated at speeds of up to 15 000 rpm. Particle size depends on the alloy composition, the electrode diameter, and the rotation speed. The typical sizes for Ti-6Al-4V powders are between 100 and 300 μm with a median diameter of about 175 μm. The PREP powder is spherical while having good flow characteristics and a tap density of about 65% solid substance. Various process modifications have been studied and implemented to produce finer powder [38]. In particular, the REP has been combined with GA.

The REP is also used by similar technologies that are available in Russia, China, and Japan. The PREP and REP powders are used to produce PM components for aerospace, gas turbine engineering, environment control, and

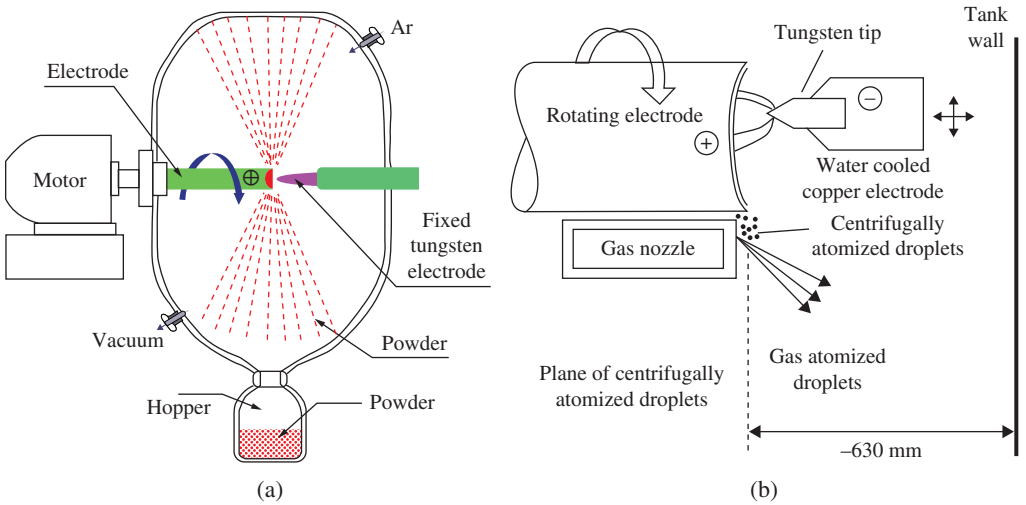


Figure 1.11 Conceptual schematic of (a) PREP principle. Source: Chen et al. [40] / with permission of Elsevier and (b) gas-assisted, rotating electrode process. Source: Nachtrab et al. [38].

biomedical applications. PREP has been developed to produce Ni-based alloy powders, Ti alloy powders, stainless steel alloy powders, and other refractory metal powders. The high-quality metal powders produced by PREP are widely used for electron-selective melting, laser melting, cladding, coating, HIP, etc.

Powders produced by the REP are generally spherical in nature. Compared to gas-atomized powder, PREP powder is relatively coarse. Various process modifications have been investigated to produce finer powder. A typical average size of the powder particle produced by the REP is 150–250 μm, and the particle size distribution is usually narrow [39]. It is interesting to note that the use of a large-diameter disk electrode, rather than a bar electrode, has been reported to decrease the mean particle size by more than 40% and to reduce the cost of input material. In another process modification, the metal droplets can be quenched into liquid argon. It was reported that the results of this approach were a major reduction in powder particle size [38]. In another modification, GA was coupled with the REP, as shown in Figure 1.11a. The REP powder process is similar to PREP, except that a tungsten arc is used instead of a plasma to melt the end of the rotating electrode [41]. As shown in Figure 1.11b, the yield of smaller than 44 μm titanium powder increased from less than 1% with regular REP to greater than 20% with GA assisted REP.

The characteristics of metal powders produced by PREP include low oxygen and other impurities (high purity), perfectly spherical and essentially satellite-free, narrow particle size distribution, high flowability and high apparent/packing density, and low internal porosity. Importantly, this process has been used extensively to produce powders of reactive and refractory materials.

The metal or alloy is made into self-consuming electrode bar stock, and the tip/end face of the electrode rotating at high speed is melted by argon or nitrogen plasma arc, and the centrifugal force generated by the electrode rotating at high speed throws out the melted metal liquid to form small droplets due to the high centrifugal force of the rotation of the rod. The droplets are cooled at high speed in the inert gas and solidified into spherical powder particles.

An inherent limitation of PREP, however, is that it is difficult to obtain finer powders because there are limitations to the maximum rotational speed that can be achieved in the electrode rod. In addition, a small quantity of coarse residual inclusions is commonly found in PREP powders [42]. To further reduce the occurrence of inclusions, research into the development of new preparation technologies for ultra-pure fine powders is essential.

1.7 Spark Plasma Discharge Spheroidization (SPDS)

Spark plasma discharge spheroidization (SPDS) is a metal powder preparation method that was originally developed to reduce the inclusions and refine the particle size of the powder. The **spark erosion technique** was proposed by Svedberg in 1924 [43] and was used to produce colloidal suspensions in the early years. Nowadays, it is

widely used in various industries for the fabrication of hard or tough materials, wire-cutting, and powder production. American materials and electrochemical research (MER) has used the plasma discharge spark process to produce ultrafine Ti and tool steel powders with controlled size distribution ranging from 1 to 10 μm [44]. In addition, Ti–Ni–Zr and Ti–Ni–Hf powders have been obtained via spark erosion in liquid argon from melted shape memory master alloys [45].

SPDS process primarily takes place with the spark discharge between the pre-alloyed charges that are immersed in a dielectric fluid. The molten and subsequently evaporated materials are ejected from the electrodes and quenched in the dielectric medium after a complex plasma creation and breakdown. These materials ultimately condense as powder in the dielectric medium. The powder is quenched in the dielectric fluid and experiences extremely rapid cooling. Compared with the atomization process, like GA or PREP, this process can produce superfine powder at a much higher cooling rate up to $\sim 10^7\ \mathrm{K\,s^{-1}}$.

The major steps of SPDS processing include the dielectric breakdown and the formation of a discharge channel, followed by the energy distribution and heat transmission, RS, and ejection and transfer of powder. A schematic diagram of the SPDS used in the preparation of superalloy powders out of the plates/bars is shown in Figure 1.12.

During SPDS processing, a repetitively pulsed spark discharge between the electrodes immersed in dielectric fluid is maintained. The forming mechanism of particles can be divided into four steps: (i) when the gap between the electrodes becomes narrow enough, a plasma channel is generated between the electrodes, that are immersed in the dielectric fluid (such as water, kerosene, or liquid inert gas) and a pulsed power source, while the plasma channel is surrounded by a sheath of vaporized dielectric; (ii) due to the repetitive spark discharge, a super-high temperature, as high as 10,000 K, develops at local of the electrode, which leads to localized melting or vaporization of the electrode; (iii) the molten metal droplets and vapors are inserted into an insulating medium; (iv) finally, the powders can be obtained by cooling of the molten droplets or condensation and cooling of the vapors in the dielectric liquid. SPDS can avoid the introduction of ceramic during the powder preparation process.

As an example, fine spherical particles of PM Superalloy FGH4096 with sizes less than 40 μm were obtained via the SPDS process [46]. The sphericities and particle sizes of the powders for three different pilot dielectric liquids of kerosene, alcohol with liquid argon, and pure alcohol were reported to be different. FGH4096 powders show higher sphericity and finer particle sizes with alcohol/liquid argon used as the dielectric medium, as shown in Figure 1.13. Experiments on a spark erosion facility showed that spherical powders with particle sizes tuned from several nm to 40 μm can be fabricated. The smooth surface and uniformity in particle size were also obtained by appropriately adjusting the processing parameters.

Interestingly, the solidification microstructure at the surface of the SPDS powder shows no dendritic and cellular morphologies and instead reveals a solidification cell structure. The main causes for the resultant microstructure included (i) the particle sizes were much smaller than those obtained by the traditional processes (GA and PREP), and (ii) the solidification speed and cooling rate of the powder were also relatively higher than those obtained with

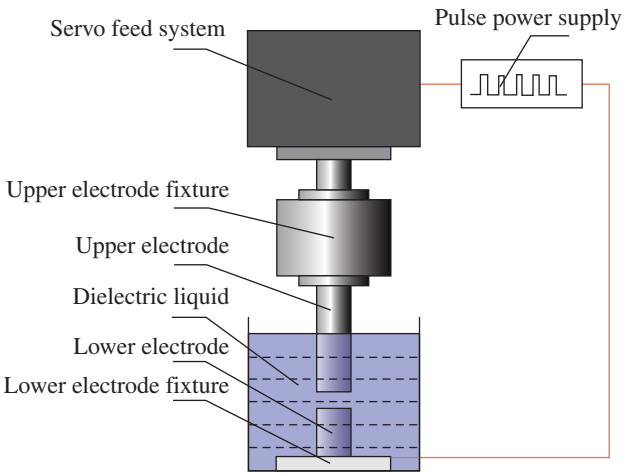


Figure 1.12 Schematic diagram of SPDS apparatus used for the preparation of powders. Source: Jia et al. [46] / IOP Publishing.

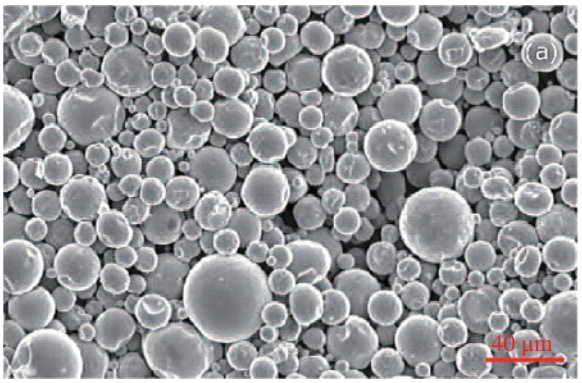


Figure 1.13 Morphology images of the SPDS powders prepared in alcohol with liquid argon. Source: Jia et al. [46] / IOP Publishing.

traditional processes (about 1 order of magnitude higher). Therefore, the heat transfer conditions limit the formation of dendrites, and a cell structure evolves in the powder.

The SPDS powder products have a broad potential market. Besides the preparation of PM superalloys for making turbine disks, the method can also be used for injection molding, rapid laser prototyping technologies, and AM. In short, superalloy powders prepared by SPDS processing have the advantages of high sphericity, small particle size, uniform microstructure, and less inclusion contents.

1.8 Plasma Induction Gas Atomization (PIGA)

The production of high-temperature and reactive materials, such γ-TiAl-based alloy powder, is driven by the need for these materials in many engineering applications. Ideally, the process should be ceramic-free to avoid possible contamination at the elevated temperatures that are required for reactive metals (around 1450 °C). In the molten state, these materials aggressively react with nearly any crucible materials. In addition, the atomized droplets and the hot powder particles are susceptible to reactions with oxygen and nitrogen as well as carbon pickup. For these reasons, only very specialized melting techniques that are crucible-free [32, 47] or that involve cold crucible melting [48, 49] can be employed. An example of a crucible-free technique is the EIMGA technique, as shown in Section 1.3. In addition, the leakage rate of the respective atomization facility must be very low, such that fully inert conditions can be guaranteed throughout the entire process. For subsequent powder handling, it is required that the powder tank can be demounted and transferred into a system of glove boxes operated under argon gas without any breach of the inert gas line.

An example of a cold crucible technique is the plasma melting induction PIGA technique [50]. The main features of the PIGA unit are illustrated schematically in Figure 1.14 [41, 51]. Plasma has been used extensively in fabricating various metal powders. The plasma process may be divided into two categories. One is that the plasma is used to form a melt pool that is then atomized by some conventional process, as applied during PIGA processing. In this case, the plasma essentially serves as the source of thermal energy. In the second case, the plasma is used to form the molten particles from either agglomerates or other starting powders and can be atomized by impacting the molten droplets traveling at very high velocities against a moving or stationary substrate material, as described in Section 1.4, PREP.

This technique has been especially developed for Ti and Ti alloys and is currently operated as a pilot plant at the GKSS Research Center, Geesthacht, Germany. In this technology, the use of ceramic materials is completely avoided. By means of a He-operated plasma torch, a melt bath is generated in a water-cooled copper crucible. In this process, a thin skull of the desired material is formed and prevents the contamination of the actual material in the crucible. At the tap hole of the crucible, an induction heated water-cooled copper funnel is mounted. It forms a thin melt stream and guides it into the center of a gas nozzle, where the melt is atomized by Ar gas. The new guiding system provides additional heating of the molten material if desired. The melt flow rate for TiAl alloys can be about 250 kg h^{-1}. Because of the melt bath formation and the induction guiding, considerable electric power is required. Since the liquid volume is much higher if compared to the EIGA technique, complex safety precautions are required. An advantage of this technique is seen in the chemically homogeneous melt bath, which serves as the source for the melt stream.

The PIGA method uses a plasma heat source to improve the stability and efficiency of the heat source, especially for high-temperature metals. Because the PIGA method uses a water-cooled copper crucible, when the metal stream is in

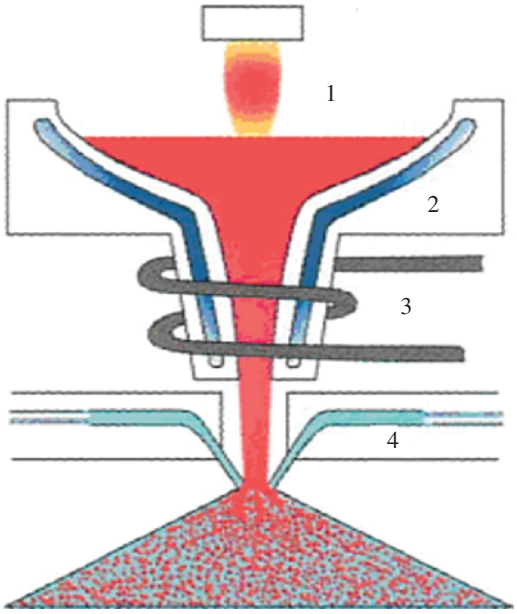


Figure 1.14 Schematic illustration of the plasma melting induction guiding gas atomization (PIGA) technique. (1) Plasma torch, (2) cold copper crucible, (3) induction-heated cold copper funnel, and (4) gas nozzle. Source: Gerling et al. [41] / John Wiley & Sons.

contact with the water-cooled copper crucible, a layer of parent metal is formed on the surface of the crucible, which isolates the subsequent metal stream from direct contact with the copper crucible wall and improves the preparation of metal powder's purity. PIGA is generally suitable for manufacturing reactive metals and high-temperature alloys, such as Ti, TiAl, FeGd, FeTb, Zr, and Cr, which cannot be processed using a ceramic crucible. It is also suitable for manufacturing high-purity metals, oxides, carbides, and nitrides, as well as ultrafine nanopowders.

The process has the advantage of utilizing scrap, granules, bars, etc., as feedstock material, and additional alloying can be accomplished during melting. The new high-power inert gas plasma torch is also suitable for the handling of reactive metal alloys containing volatile constituents. The disadvantages include the use of very high-power plasma torches exhibiting a high degree of erosion of the electrode, which can contaminate the metal powder.

1.9 Plasma-Atomized Wire (PAW)

The fabrication of metal powders using plasma technology is known as plasma atomization. In the **plasma-atomized wire (PAW) process**, plasma torches are used to melt a wire and produce the powder. The droplets are cooled and collected in a manner like that in GA. PAW powders are very clean, since the chemistry comes from a solid metal wire and the powder is spherical with excellent flow rate.

The plasma atomization technology initially went into commercial production in 1998 to enable the production of a fine particle distribution of high-purity reactive metal powders such as titanium. Plasma atomization technology can produce premium-quality spherical powders of reactive and high melting point materials, such as titanium, **nickel**, zirconium, **molybdenum**, niobium, tantalum, tungsten, and their alloys. The PAW process is primarily used for titanium alloys due to the challenges of handling molten titanium in GA. Plasma atomization has been widely developed, resulting in a high-purity spherical titanium powder in a wide variety of sizes. Atomization of plasma has the capacity of producing various sizes of titanium powder from very fine (−25 μm) to coarse (+125 μm) [51].

Powders with a spherical morphology have the benefit of having the properties of good flowability and hence capable of producing high density. In powder manufacturing processes, powders with good flowability require fewer additives than powders with low flowability. Moreover, powders with good flowability will also sinter to a high density when consolidated using HIP, for example.

Figure 1.15 shows a plasma atomization process scheme using a three-plasma arc, with titanium wire as a material feed [52]. The plasma arc is utilized as a high-enthalpy heat source that will melt and spray molten titanium wire, resulting in a titanium powder that has a very high-purity round shape.

Plasma atomization uses wire as a feedstock, which is ideal for traceability from the ingot to the final powder product. Tight quality control of the wire chemistry ensures batch-to-batch consistency. As opposed to conventional

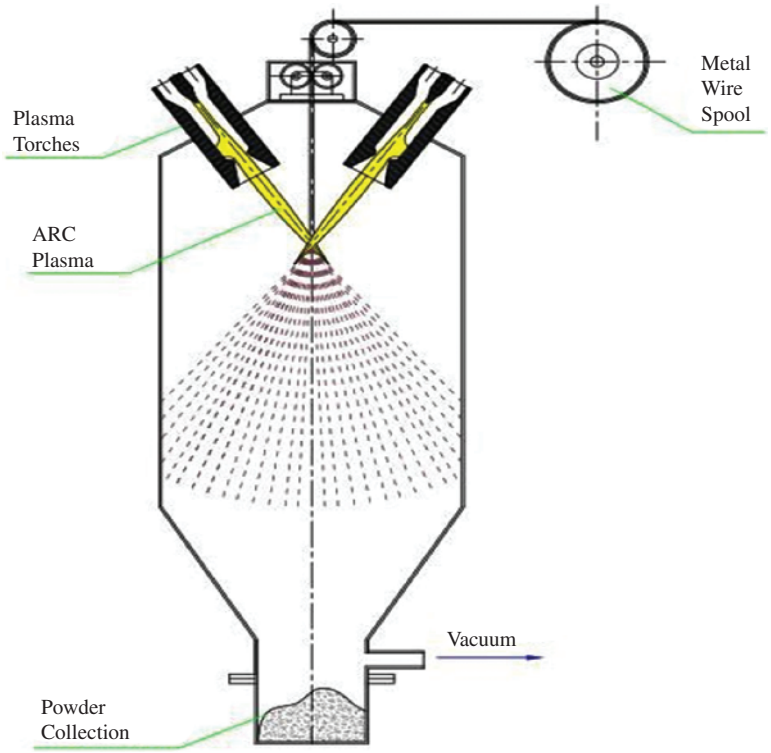


Figure 1.15 Schematic representation of the plasma atomization process to produce Ti powders. Source: Grenier [52].

GA, plasma atomization uses three plasma torches to instantaneously melt and atomize the wire in a single step. The intense heat of the plasma maintains the droplets well above their melting point throughout multiple phases of breakup, which increases the yield of very fine particles.

Once droplets are formed, they require some time to assume the most stable shape, a sphere. The cooling tower provides a low-velocity and low-turbulence environment that allows the particle to spheroidize under ideal conditions.

PAW provides a unique opportunity for producing fine, spherical, rapidly solidified powders of practically any material. The process offers the advantage of container-less melting and thus reduces the chances of contamination from the crucible. PAW process offers the highest purity possible with highly spherical particles and minimal satellite content. PAW powder thus exhibits exceptional flowability and packing properties, is well renowned and extensively used in AM, MIM, hot and **cold isostatic pressing** (HIP and CIP) and thermal and cold **spray coatings**. The main challenges with the PAW process are the high manufacturing costs, due to low production rates, and limited alloy flexibility, as the starting material must be available as wire.

Another related PAW method is PA. The principle of the PA method is that the metal wire is melted by adding a plasma heat source through a straightening machine at a certain rate, and then the molten metal droplets are atomized by high-pressure gas. The powder prepared by the PA method also has the characteristics of high sphericity, high purity, and low oxygen content. However, because the raw material is wire, the cost of raw materials is increased, and the types of metal powders prepared with it are limited.

1.10 Water Atomization (WA)

The process of producing metal powders by the disintegration of the molten metal stream using water is commonly referred to as **water atomization (WA)**. This is one of the most popular techniques for producing nonreactive metal and alloy powders. The WA technique presents a cost-effective and efficient approach to producing metallic powders and is currently used to produce low-alloy iron and steel powders for pressing and sintering.

WA is being broadly used since the middle twentieth century. The use of an atomizing chamber with inert gas has allowed the production of large tonnages of Fe powder and of a wide range of alloy steels, including stainless and tool steels. It is also widely used for Au, Ag, Zn, and Cu alloys and for some types of Ni and Co alloys. Oxygen contents are highly alloy-dependent, ranging from 500 ppm for some self-fluxing Ni-Cr-B-Si alloys and Cu alloys to 1% for high manganese steels. The morphology of WA powders is generally somewhat irregular but is greatly affected by alloy composition and atomizing conditions such that apparent densities can range from as low as 20% to ~50% of solid. Particle sizes for steels range from ~30 to 1000 μm [53], as the rapid quenching of the water jets allows larger particles to freeze quickly.

The rate of heat extraction during the WA process is quite high. The apparatus consists of water jets (usually coming through multiple jets or an **annular ring**) directed onto a molten stream of metal or alloy. The key variables that influence the process are the melt superheat and water pressure, which controls the water velocity. High water pressure results in finer powder particle sizes. Other important parameters are the melt stream diameter, metal flow rate, and water jet apex angle. The nozzle-to-melt distance is not as critical as GA since water has lower compressibility and higher density than gases and thus its energy does not diminish as rapidly. However, this distance should be minimized to maintain the melt superheat.

Due to the rapid extraction of heat, the final particle shape is quite irregular, as the particles have less time to spheroidize compared to the GA process. Thus, it is often necessary to have large superheat values for control of the particle morphology. The nature of the environment results in the final powder being slightly contaminated by a surface oxide layer, which under certain circumstances can be reduced by a subsequent hydrogen reduction step.

A schematic view of the WA process is shown in Figure 1.16 [54]. Water-atomized powders have excellent compressibility, but their apparent density is normally lower than that of gas-atomized powders due to the particle morphology. The water pressure has been reported to be the prime controlling factor for the WA process. It was reported that water-atomized steel powder using a water pressure of 1.7 MPa resulted in powders with a mean particle size of 117 μm compared to a mean particle size of 42 μm when the water pressure was 13.8 MPa [55].

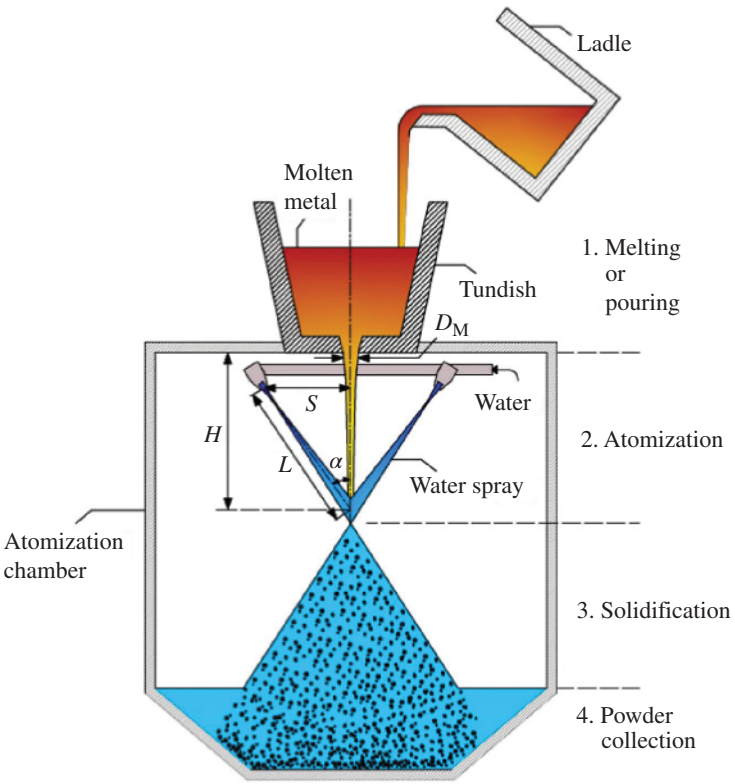


Figure 1.16 Schematic view of the water atomization process. Source: Asgarian et al. [54].

The need for finer powders (in the 10 μm range) has resulted in the high-pressure water atomization (HPWA) process in which water pressures up to 70 MPa have been used to produce powders with an average particle size of around 10 μm [56]. In the same report, it has been shown that the water jet nozzle configuration also influences the powder particle size. The authors have reported that a V-jet nozzle configuration produces finer powders than the commonly used cone nozzle configuration using similar atomizing pressures.

The process of high-pressure WA is an important development in the direction of producing atomized powders in the 10 μm range. The demand for these finer powders is increasing, especially due to the emergence of powder injection molding as an important fabrication route. Thus, it is expected that high-pressure WA could partially fill the gap between the supply and demand for reasonably inexpensive powders in the 10 μm particle size range. However, the powder morphology could be a problem for these powders.

High-pressure WA can provide fine powders, as discussed above. The process also can produce a wide range of powder particle size, size distribution, and powder apparent density. With increasing atomization water pressure, the particle size tends to become finer, and the particle size distribution also tends to become broader. The atomizing water pressure, which varied from 10 to 150 MPa, was found to yield finer average particle sizes with increasing water pressure with water-atomized 316-L stainless steel powders [57].

Ultra-high-pressure water atomization (UHPWA) involves the use of very high water pressures, typically in the range of 100–200 MPa. The water jets are generally supersonic, moving at velocities of 400–500 m s^{-1}, and atomization is partly due to the shockwaves around the water jets at such pressures. It is normally carried out in an inert gas-purged atomizing chamber and allows the production of very fine metallic powders with median particle sizes less than 10 μm. Due to the high surface area of such fine powders and the oxidation potential of the water, the oxygen contents are typically in the range of 2000–5000 ppm. The particle shape can range from somewhat irregular to spherical, allowing high tap densities of ~4.5 g ml^{-1} for MIM grades.

Few studies have been carried out on the mechanisms that govern the WA process. It has been suggested that the water jet is usually broken up into minute fragments before it impinges on the metal stream. Thus, very small water droplets impact the molten liquid stream, causing small appendages to appear on the molten liquid surface (like small waves) from which a metal droplet is ejected. The water droplet after striking the liquid film immediately evaporates. This is termed the "scrape" mechanism that operates during WA events.

In general, WA can produce powders that have an average particle size of around 200 μm. The particle morphology is generally irregular, and the powders usually have a wide size distribution. However, progress in high-pressure WA has led to the successful production of large tonnages of fine powders.

1.11 Summary

The material summarized in this first chapter reveals that powder metallurgy, as the practice of working with metal powders, has a deep and rich history. In some ways, the use of powder metals can be traced back several thousand years. Moreover, it is also evident that powder metals find applications in numerous areas of technology and engineering. A review of early **atomizer designs** reveals that the fundamental idea of breaking a liquid into a dispersion of fine droplets can be accomplished with very simple atomizer designs, such as the first atomizer design created by Hall, who used it for aluminum powder, to much more complex atomizer designs that involve intricate use of fluid flow phenomena to enhance the efficiency of atomization. In this chapter, we also reviewed the various techniques that are being used to produce many types of metal powders. In general, the breakup of metal powders can be accomplished using energetic gas jets, and, depending on the chemistry of the metal being atomized, gas chemistry is selected and used under various pressure conditions. It is also apparent that in many cases, it is imperative to control the environment in the chamber where the metal is being prepared to avoid deleterious surface reactions. Modern atomizer designs include sophisticated melting approaches that allow the handling and processing of reactive metals such as titanium. In general, desired powder morphology and particle size can be achieved through the identification of optimal atomization parameters. Furthermore, powder qualities are known to affect powder flowability, which can improve the injection-ability and spread-ability of powder particles for AM process. Generally, good injection-ability and spread-ability are associated with good powder feeding and packing fractions, which can lead to high-quality AM components. Therefore, in the following chapters, the parametric information for the GA is presented.

Nomenclature

AM	additive manufacturing
CIP	cold isostatic pressing
EIGA	electrode induction melting gas atomization
GA	gas atomization
HIP	hot isostatic pressing
HPGA	high-pressure gas atomization
HPWA	high-pressure water atomization
IGA	inert gas atomization
MIM	metal injection molding
PIGA	plasma melting induction gas atomization
PA	plasma torch atomization
PAW	plasma atomized wire
PIGA	plasma induction gas atomization
PREP	plasma rotational electrode process
RS	rapid solidification
REP	rotating electrode processes
SPDS	spark plasma discharge spheroidization
VIGA/VIMGA	vacuum induction melting inert gas atomization
UHPWA	ultra-high-pressure water atomization
WA	water atomization

References

1 Samal, P.K. and Newkirk, J.W. (2015). *ASM Handbook, Volume 07 – Powder Metallurgy*, 3. ASM International.
2 DeVilbiss, T.A. (1909). Atomizer, vol U.S. Patent #938,648.
3 Harrington, E.L. (1927). *J. Opt. Soc. Am.* 14 (1): 87–97. https://doi.org/10.1364/JOSA.14.000087.
4 Hall, E.J. (1928). Process for Disintegrating Metal, U.S. Patent #1,659,291.
5 Singer, A.R.E. (1970). *Met. Mater.* 426.
6 Lavernia, E.J., Srivatsan, T.S., and Rangel, R.H. (1992). *Atomiz. Spr.* 2: 253.
7 Unal, A. (1987). *Mater. Sci. Technol.* 3: 1029–1039.
8 Unal, A. (1988). *Mater. Sci. Technol.* 4: 909–915.
9 Singer, A.R.E. (1972). *J. Inst. Met.* 100: 185.
10 Brooks, R.G., Leatham, A.G., Coombs, J.S., and More, C. (1977). *Metall. Met. Forming* 9: 157.
11 Lavernia, E.J. 1984. Liquid dynamic compaction of a rapidly solidified 7075 aluminum alloy modified with 1% Ni and 0.8% Zr, thesis, Massachusetts Institute of Technology, Cambridge, MA.
12 Lavernia, E.J. and Grant, N.J. (1988). *J. Mater. Sci. Eng.* 98: 381–394.
13 Hueter, T.F. and Bolt, R.H. (1962). *Sonics*. New York: John Wiley & Sons.
14 Lavernia, E.J. and Grant, N.J. (1986). *Met. Powder Rep.* 41: 255.
15 Gerking, L. (1992). *Powder Met. Int.* 25: 59–65.
16 Mir, J.M. (1980). *J. Acoust. Soc. Am.* 67: 201.
17 Clyne, T.W., Ricks, R.A., and Goodhew, P.J. (1984). *Proceedings, 5th Conference on Rapidly Quenched Metals (R.Q.5)* (ed. S. Steeb and H. Warliment), 903. Wurzburg, Germany: North Holland Physics Publishing Amsterdam.
18 Ridder, S.D., Osella, S.A., Espina, P.I., and Biancaniello, F.S. (1992). *Int. J. Powder Metall.* 28: 133–147.
19 Ridder, S.D. and Biancaniello, F.S. (1989). *Mater. Sci. Eng., A* 98: 47–51.
20 Miller, S.A., Wojcik, L.A., Miller, R.S. et al. (1995). *Advances in Powder Metallurgy and Particulate Materials*, vol. 1 (ed. M. Phillips and J. Porter), 31–40. Metal Powder Industries Federation/APMI.
21 Marriott, W. (1872). vol patent 3322 British.
22 Zheng, B., Lin, Y., Zhou, Y., and Lavernia, E.J. (2009). *Metall. Mater. Trans. B* 40B: 768–778.
23 Lavernia, E.J., Srivatsan, T.S., and Rangel, R.H. (1992). *J. Atomiz. Spr.* 2: 253–274.

24 Lin, C.S., Jih, G.J., Chang, C.S., and Chiou, S.Y. (1989). *Advances in Powder Metallurgy*, vol. 2 (ed. T.G.G.a.W.F. Jandeska), 57. Princeton, NJ: Metal Powder Industries Federation.

25 Veistinen, M.K., Lavernia, E.J., Baram, J.C., and Grant, N.J. (1989). *Int. J. Powder Metall* 25: 89.

26 Mohanty, T., Tripathi, B., Mahata, T., and Sinha, P. (2014). Arc plasma assisted rotating electrode process for preparation of metal pebbles. *International Symposium on Discharges and Electrical Insulation in Vacuum (ISDEIV)* 741–744.

27 Samal, P.K. and Newkirk, J.W. (ed.) (2015). *ASM Handbook: Powder Metallurgy, ASM International*, 907. USA: Materials Park.

28 ALD Vacuum Technologies AG, Metal Powder Technology, http://www.metamak.com.tr/UserFiles/File/demir%20disi/vakum%20metalurjisi/powder%20metallurgy/Metal_powder.pdf.

29 Gerling, R., Schimansky, F.P., Wegmann, G., and Zhang, J.X. (2002). *Materials Sci. Eng., A* 326: 73–78.

30 Franz, H., Plochl, L., and Schimansky, F.P. (2008). Recent advances of titanium alloy powder production by ceramic-free inert gas atomization. *Proc. Titanium 2008, International Titanium Association* 1–4.

31 Gerling, R., Schimansky, F.P., and Clemens, H. (2003). *Gamma Ti- tanium Aluminides* (ed. Y.-W. Kim, H. Clemens, and A. Rosenberger). Warrendale, PA: TMS.

32 Preauchat, B., Popoff, F., and Thomas, M. (2002). *Adv. Eng. Mater.* 4: 133.

33 Grewe, H., Potschke, J., and Weilin, W.G. (1986). *ZwF* 81: 413.

34 Dawes, J., Bowerman, R., and Trepleton, R. (2015). *Johnson Matthey Technol. Rev.* 59: 243–256.

35 http://www.arcastinc.com/news.htm, 2021.

36 Lalwani, G. (2020). https://bonezonepub.com/2020/06/09/titanium-powder-quality-key-to-additive-manufacturing-success/.

37 Froes, F.H. and Eylon, D. (1990). *Int. Mater. Rev.* 35: 162–182.

38 Nachtrab, W.T., Roberts, P.R., and Newborn, H.A. (1993). *Key Eng. Mater.* 77–78: 115–140.

39 Bose, A. (1995). *Advances in Particulate Materials*. Elsevier.

40 Chen, G., Zhao, S.-y., Tan, P. et al. (2017). *Trans. Nonferrous Met. Soc. China* 27: 2647–2655.

41 Gerling, R., Clemens, H., and Schimansky, F.P. (2004). *Adv. Eng. Mater.* 6 (1-2): 23–38. https://doi.org/10.1002/adem.200310559.

42 Zhang, M.J., Li, F.G., Wang, S.Y., and Liu, C.Y. (2011). *Mater. Sci. Eng., A* 528: 4030–4039.

43 Svedberg, T. (1924). *Colloid Chemistry, Wisconsin Lectures*. New York: Chemical Catalog Co.).

44 Newell, K. (1999). *Met. Powder Rep.* 54: 22.

45 Monastyrsky, G., Ochin, P., Wang, G.Y. et al. (2011). *Chem. Metals Alloys* 4: 188.

46 Jia, C., Ge, C., and Yan, Q. (2016). *Chin. Phys. B* 25: 026103.

47 Hohmann, M., Diemar, W., and Ludwig, N. (1992). *Advances in Powder Metallurgy & Particulate Materials*, vol. 1 (ed. J.M. Capus and R.M. German), 27. Princrton, NJ: MPIF.

48 Gerling, R., Schimansky, F.P., and Wagner, R. (1992). *Advances in Powder Metallurgy & Particulate Materials*, vol. 1 (ed. J.M. Capus and R.M. German), 215. Princeton, NJ: MPIF.

49 Yolton, C.F. and Beckman, J.P. (1995). *Mater. Sci. Eng., A* A192: 597.

50 Stenzel, O.W., Sick, G., and Hohmann, M. (1990). DE4011 392A1, German Patent.

51 Alagheband, A. and Brown, C. (1998). *Met. Powder Rep.* 53: 26–28.

52 Grenier (1997). *Met. Powder Rep.* 52: 34–37.

53 Dunkley, J.J. (2019). *Johnson Matthey Technol. Rev.* 63: 226–232.

54 Asgarian, A., Tang, Z., Bussmann, M., and Chattopadhyay, K. (2020). *Powder Metall.* 63: 288–299.

55 German, R.M. (1984). *Powder Metallurgy Science*. Princeton, NJ: Metal Powder Industries Federation.

56 Seki, Y., Okamoto, S., Takigawa, H., and Kawai, N. (1990). *Met. Powder Rep.* 45: 38.

57 Kimura, A., Nakabayashi, K., and Shimura, T. (1990). *Met. Powder Rep.* 45: 106.