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Titanium MIM moves into the mainstream with plasma atomised powders from AP&C

When it was announced earlier this year that Canada's AP&C, a GE Additive Company, had received an order for 30 tons of plasma atomised titanium powder for a Metal Injection Moulding application, it became clear that titanium MIM had reached the mainstream. In the following article AP&C explains the attraction of MIM for titanium components, outlines the company's plasma atomisation process and presents its growing range of products specifically suited to the MIM process.

Product designers in a wide range of industries are carefully eyeing titanium due to its unusual combination of high strength, low weight and excellent corrosion resistance. The primary factor limiting the more widespread use of titanium and its alloys is the high cost of raw materials and processing relative to competitive materials. These costs are particularly high for geometrically complex parts. Metal Injection Moulding (MIM) provides an attractive method of producing small, complex titanium components at a lower cost using processing techniques that are similar to those used in plastic injection moulding.

Expanding the usage of MIM to new applications requires a ramping up of the supply of titanium powders with the required tailored size distributions and consistently high quality. The plasma atomisation process used at AP&C has demonstrated the ability to provide the precise control of powder properties necessary to satisfy the demands of a broad range of current and potential MIM applications. This article will survey the continuing evolution of the plasma atomisation process as it evolves to deliver the large quantities of high quality fine powders required to fully develop the potential of MIM technology.

The attraction of titanium

Titanium is one of the strongest and most durable materials on the planet and has the highest strength to density ratio of any metallic

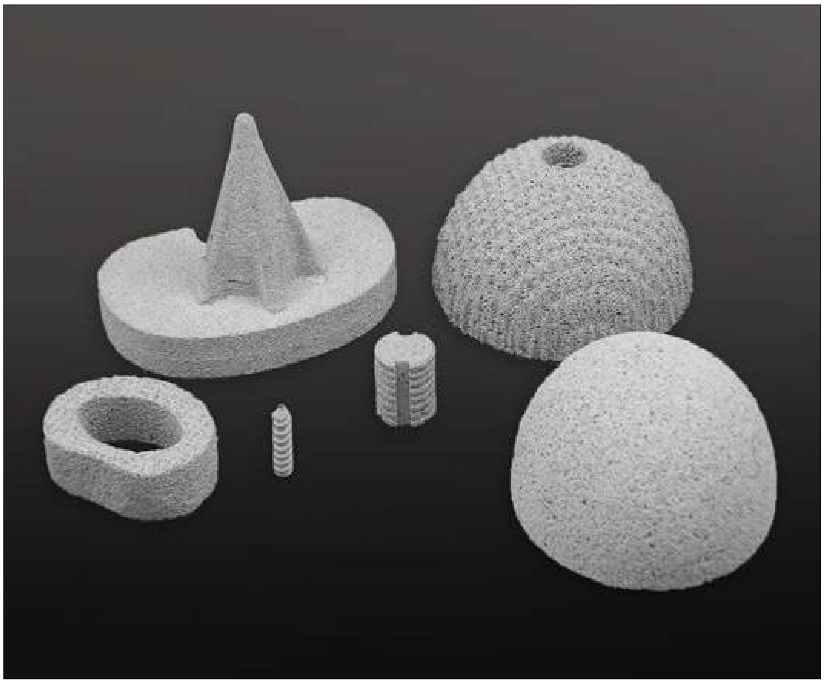

Fig. 1 Titanium hip joint replacement parts made by MIM

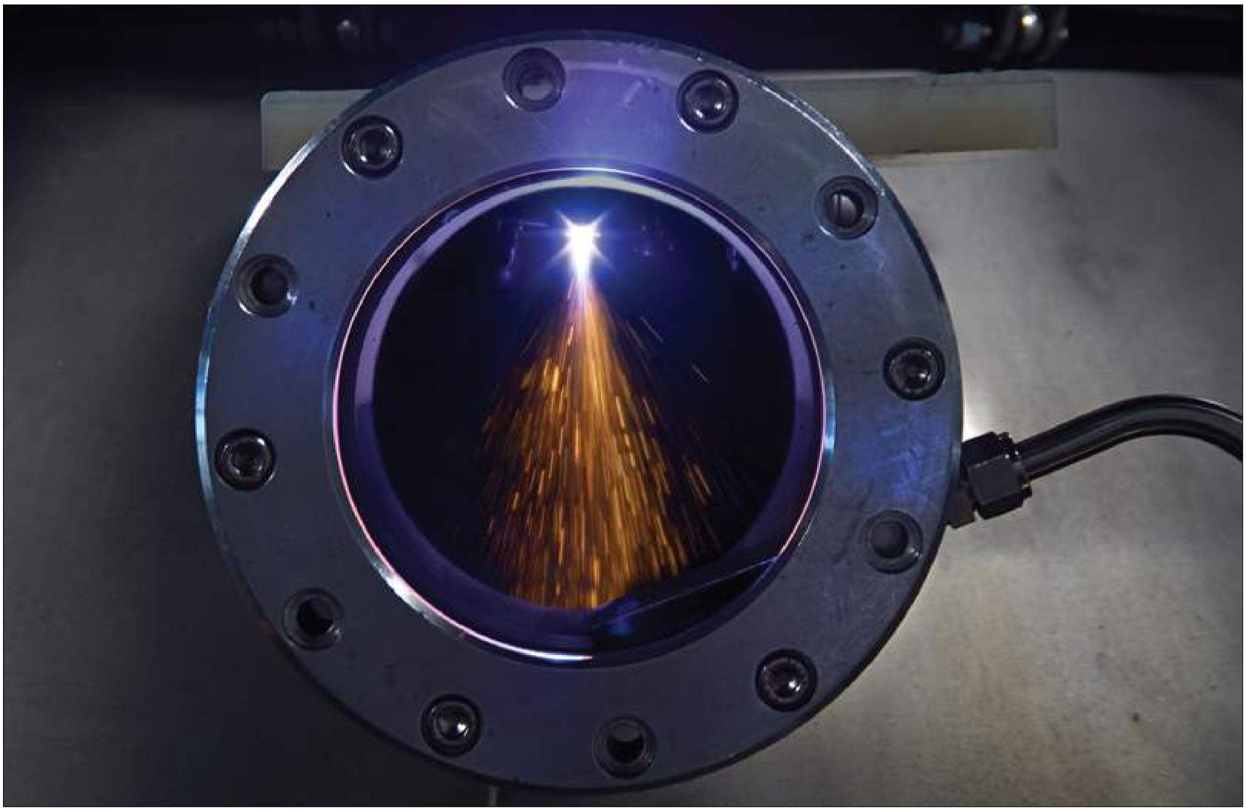

Fig. 2 Plasma torches during an atomisation run at AP&C

element. Bulk titanium is also relatively inert, which means it can survive exposure to unfavourable conditions far better than other materials. Titanium's biocompatibility, which means it is not toxic and not rejected by the body, has led to many applications in the medical industry such as artificial knees and hips, surgical instruments, body piercings and even dental implants (Fig. 1). Titanium's high strength-to-weight ratio means it can also be used to reduce the weight of an aircraft while maintaining structural integrity. The result is a substantial reduction in fuel consumption that can provide enormous savings over the lifetime of the aircraft.

The primary limiting factor in the proliferation of conventional titanium parts is their relatively high cost, which results from their raw material costs, fabricating costs and metal removal costs. The largest expense is typically the machining processes required to bring the part to its final shape. Titanium is recognised as a difficult to machine material, which often raises the costs of cutting tools, fixtures and machine tools compared to conventional materials. However, an even greater factor in most cases is the large proportion of the initial material that ends up as chips on the machine shop floor. It is not uncommon for the weight of the material that is removed from the workpiece during machining to exceed the final part by a factor of 20 to 1; this waste material is generally sold as scrap for a small fraction of the original material cost.

The titanium MIM process

The titanium Metal Injection Moulding (Ti-MIM) process has the potential to substantially reduce the cost of producing many titanium parts at high levels of quality. A key advantage of the Ti-MIM process is that it combines Powder Metallurgy and plastic injection moulding methods to produce parts to net shape without any material waste. High purity spherical titanium fine powder is used in Ti-MIM and this is mixed with a thermoplastic binder and heated to melt the binder and achieve the right level of viscosity for injection moulding. After moulding, the binder is removed either by heating and/or chemical reaction and the part is then sintered at a high temperature to fuse the metal particles into a dense solid.

MIM has the advantage of being able to produce intricate shapes at a relatively low cost because it eliminates material waste and machining expenses. It is considerably faster and can achieve much closer tolerances than investment casting. It is, however, usually limited to small parts below 200 g. The MIM process is also used to manufacture parts made of many other materials, including various types of specialty steels and non-ferrous alloys.

A major challenge in scaling up the production of titanium MIM parts is producing the needed quantities of fine titanium powder that provide the specific characteristics required by the MIM process. One of the most critical requirements is the particle

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size distribution (PSD). Another important requirement is the particle shape – spherical particles are preferred for most MIM processes because they provide high loading which minimises the shrinkage and ensures consistent mould filling and reduced tool wear. Finer PSD provides a better surface finish and sinters at lower temperature, reducing the grain growth during the heating stage. Purity is also important, with the major concern usually being interstitial elements - impurities found in nominally pure metals that are small enough to fit between normal crystalline lattice locations.

Oxygen is typically the major concern in titanium, so common practice involves specifying the maximum oxygen impurity level, with each impurity counting against this limit based on its effect on the properties of the finished part relative to oxygen. Oxygen content is even more critical with fine PSD powder because of its high specific area and native oxide layer. Porosity, which refers to the presence of entrapped gas pockets, must also be held to very low levels to achieve good mechanical properties, but the use of fine PSD minimises such risk.

The plasma atomisation process

Several different methods are used to produce powder for Ti-MIM including inert gas atomisation, plasma spheroidisation, Hydride-Dehydride (HDH) and other chemical reduction processes. Plasma atomisation is a relatively new process that has gained Ti powder market share rapidly because of its ability to produce highly spherically shaped particles in accurate size distributions with low oxygen content and low internal porosity (Fig. 2, 3). In this process, titanium wire (Fig. 4) is fed into a plasma torch which converts the wire into droplets that subsequently solidify to powder form.

Plasma atomisation production has been evolving ever since its initial conception in the 1990s. At that time,

Fig. 3 A plasma atomisation production unit

Fig. 4 Titanium wire feedstock used in the plasma atomisation process

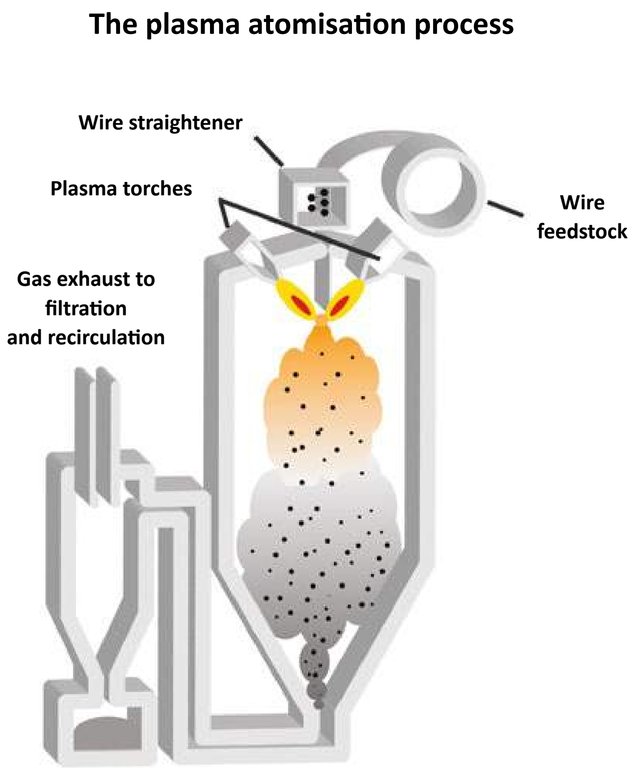

Fig. 5 Schematic of plasma atomisation process

problems were experienced with throughput and poor yield of fine PSD which resulted in high production costs. Over the last few decades, these problems have been overcome, resulting in the development of large capacity production units that consistently deliver high-purity spherical metal powders. AP&C has been at the forefront of the technology's development for more than ten years and has become the world leader in the industrialisation and optimisation of the plasma atomisation process.

The first production step is to straighten the feed wire to ensure its optimal positioning at the apex of one or several plasma torches. The speed of the wire also needs to be monitored to control and adjust the resulting particle size distribution. In a typical case, three DC plasma torches, each delivering about 30 kW, are placed so that their jets converge on the metal wire. The supersonic nozzles installed at the exit of the torches ensure a maximum gas velocity to successfully atomise the metal wire. A schematic of the process is shown in Fig. 5.

The argon gas used as the atomising medium is heated to a high temperature to prevent the metal particles from rapidly freezing together into irregular shapes. Heated argon gas has a higher velocity and thus applies a stronger atomisation force at a lower temperature without increasing the gas flow rate. The plasma superheats the melt and subsequent cooling ensures complete spheroidisation. A low concentration of suspended atomised particles prevents the formation of satellites that reduce flowability.

The importance of minimising agglomerates in powder collection and sieving

Breaking up agglomerates is critical to maintaining the flowability of the feedstock, which helps ensure the right moulding behaviour. Agglomeration is also detrimental to the feedstock preparation because of its impact on the compounding with the polymeric binder. Very small PSD powder has high interfacial adhesion energy, making it difficult to break up agglomerates during the high shear mixing process used in compounding. AP&C has developed

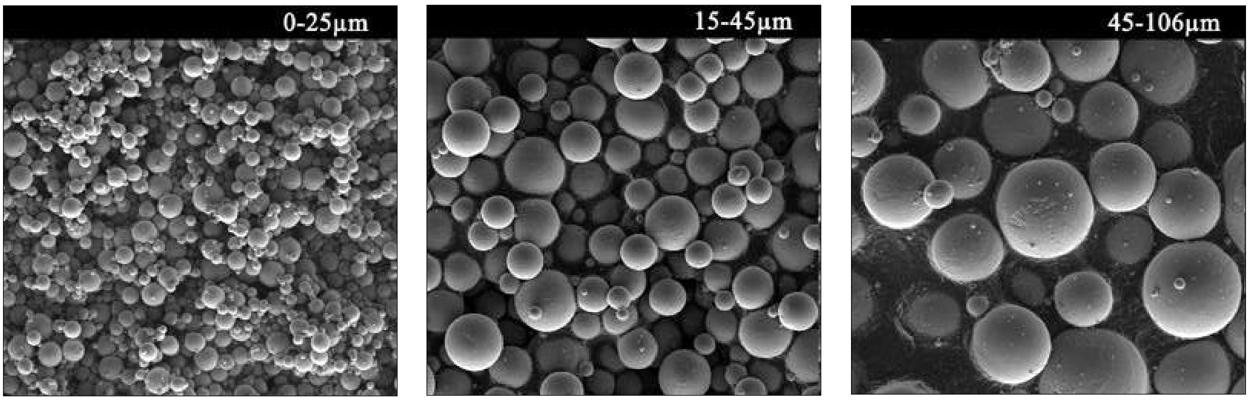

Fig. 6 Various sizes of highly spherical titanium powder produced by AP&C using the plasma atomisation process.

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Fig. 7 Alain Dupont, President of AP&C, at the construction site of AP&C's new state-of-the-art titanium powder factory in St. Eustache, Montreal, Canada, in February

methods to reduce the formation of agglomerates in the powder production process, improving the flowability of the powder.

Powder collection is done via a typical cyclonic device and the powder is carefully passivated to enable safe manipulation in open air and reduce oxygen content. After the passivation stage, the powder is sieved with an ultrasonic resonator according to the final customer's size distribution requirement. The plasma atomisation process typically delivers powders with sizes up to 150 µm. The most common grades are 0-25 µm, 0-45 µm, 15-45 µm and 45-106 µm (Fig. 6). For cut sizes smaller than 25 µm, a gas separation apparatus, based on saltation velocity theory, is used to ensure the effective removal of the smaller particles.

The melt source can be chosen from unalloyed titanium grades (for example grades 1, 2, 3 or 4); titanium alloys modified with palladium or ruthenium (for example grades 7, 11, 16, 17, 26 or 27); alpha and near-alpha titanium alloys (for example grades 6, 9, 12, 18, 28); alpha-beta titanium alloys (for example grades 5, 23 or 29); near-beta and beta titanium alloys (for example grades 19 or 20). The primary titanium powders used in the MIM process are commercially pure titanium (CpTi) and titanium alloys including Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo and Ti-5Al-5V-5Mo-3Cr.

Almost any metal can be atomised with the plasma atomisation process, although it is best suited to reactive materials and materials with high melting points that cannot easily be processed into spherical powders by other methods. Materials that have been successfully atomised with the plasma atomisation process include niobium (Nb), molybdenum (Mo) and nickel-titanium alloy (nitinol). This last material is a super elastic memory alloy that is extremely difficult to process given its sensitivity to any crystallographic modification. Customers can provide the metal wire feedstock for atomisation in quantities as small as 100 kg.

The particle size distributions of different Ti-6Al-4V ELI (Extra Low Interstitial) powders produced by plasma atomisation were measured by laser diffraction. The apparent densities of these powders were about the same at somewhat over 2.50 g/cm^3 but their flowability was very different. The 0-25 µm powder did not flow at all. It is primarily used in cold spray or surface finish applications. The 0-45 µm powder did not flow under normal conditions of temperature and humidity but in a controlled low-humidity, low-static environment it flows well. The 15-45 µm powder was specially engineered to flow in under 35 s in the Hall flowmeter according to ASTM-B213. Finally, the 45-106 µm powder flowed in less than 25 s in the Hall flowmeter according to ASTM-B213. The powder demonstrated high sphericity and very few satellites were present in every size distribution.

Fig. 8 AP&C's management team on the site of AP&C's new titanium powder factory

Sintering Temperature (C)	Part Density (g/cm³)	% of Bulk Density (4.408 g/cm³)
1100	4.36	98.9%
1200	4.38	99.4%

Table 1 Effects of MIM sintering temperature on part density

A typical atomisation run

A typical example of a plasma atomisation run is analysed in which 0.125" diameter Ti-6Al-4V (grade 23) wire is used as the raw material, with three converging plasma jets oriented at about 30° with respect to the vertical axis. The plasma contacts the metal wire less than 2.5 cm from the plasma torch nozzle outlet. Each plasma torch operates at a power of 30 kW with a 150 standard litre per minute (SLM) argon gas flow. A background sheath gas was used to ensure the proper transport of metal droplets. The sheath gas flow is in the range of 550 SLM. An electric current of about 180 A at 45 V DC was used to preheat the wire and this is fed at 13 kg/h through a gas-cooled, adjustable guide to maintain the wire position at the apex of the plasma torch jets. Wire is typically run in batches of approximately 100 kg. The gas to metal ratio in this production run was 8.7. The resulting PSD obtained was determined according to ASTM B214 'Standard Test Method for Sieve Analysis of Metal Powders'. Despite the low gas-to-metal ratio, this application yielded over 90% of 0-106 µm and almost 60% of 0-45 µm powder.

Investing for the future

AP&C has the world's largest production capacity for spherical titanium powder. The company has production capacity of 500 tons per year in its first plant in Boisbriand, Canada. The company's second plant will start production in the third quarter of 2017 at St. Eustache, Montreal, Canada, providing an additional 250 tons per year of production capacity (Fig. 7, 8). AP&C plans to invest up to US $25 million in the new facility. The production capacity of the second plant can rapidly be increased to 750 tons per year. AP&C is investigating the possibility of adding further production capacity at different locations to locally serve strategic markets.

AP&C has developed a new grade of powder that combines very fine PSDs (0-20 µm), low oxygen content and minimal agglomeration. This new powder grade enables low MIM sintering temperatures ranging from 1100°C to 1200°C, high part density and excellent surface finish (Table 1). AP&C recently received a 30-ton order for MIM powder to be delivered in 2017 that is believed to be the largest titanium powder for MIM contract ever. AP&C has worked with the National Research Council (NRC), the Canadian government's premier research organization, to develop a new MIM feedstock containing 1% carbon. This formulation minimises grain growth which improves mechanical properties. The new feedstock's finer microstructure and low porosity translates into better fatigue resistance and fracture toughness.

Conclusion

Titanium powders are now used across many industrial sectors, especially biomedical and aerospace, to manufacture complex structures with properties such as higher strength, high biocompatibility, high resistance to metal fatigue and desired surface morphologies. Precise control of powder properties is necessary to satisfy the demands of individual applications. The plasma atomisation process enables the production of high purity spherical titanium metal powders that meet the requirements of MIM and many other applications. Powder manufacturers are continuing to push the limits to deliver larger quantities of higher quality titanium powder to enable net shape forming of ever more complex products at higher levels of quality and lower cost than conventional manufacturing methods.

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