Induction plasma technology applied to powder manufacturing: example of titanium-based materials

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Abstract: Additive manufacturing technologies require specific powders to ensure a good quality to the manufactured parts. The critical properties are; the powder chemistry, flow ability, packing density, and the absence of porosity. This review highlights the capability of Tekna's Inductively Coupled Plasma (ICP) technology for the production of high quality powders for the additive manufacturing industry.

Keywords: induction plasma, spherical powder, titanium, plasma process

1. Tekna's plasma process for the production of powders dedicated to Additive Manufacturing

Metal-based Additive Manufacturing (AM) technologies requires satellites-free powders having a spherical shape ensuring the highest packing density achievable, a specific particles size distribution, high flow ability as well as an internal particle structure which is free of pores. Despite the various advantages that commercially available powders can offer in terms of affordability and/or ease of availability, they rarely meet all the requirements listed above.

2. Tekna's spheroidization process

Converting a commercial powder (regardless of its original manufacturing process) into a high-performance powder can be achieved using the Inductively-Coupled Plasma (ICP) process developed by Tekna Plasma Systems Inc. The ICP process represent essentially a finishing step which is based on the in-flight heating and melting of the individual particles of the feed material, followed by their gradual cooling and solidification before reaching the bottom of the powder processing chamber. The technology allows for operation using a wide range of plasma gas mixtures at atmosphere pressure or soft vacuum. The plasma can be used as a chemical reactor as well as an enthalpy source. Two different case studies are presented regarding the manufacturing of titanium-based alloy powders.

A flow diagram of the Tekna plasma spheroidization process is shown in Fig. 1. Such a system allows for the industrial scale manufacturing of spherical powders which exhibit controlled chemistry, controlled oxygen level, high flow ability and high density. The system consists essentially of a standard Tekna inductively coupled plasma torch (Model PN-50 or PN-70) placed on the top of a water-cooled stainless steel chamber. The feed material is injected axially into the centre of the discharge using a water-cooled injection probe with an appropriate amount of carrier /diluent gas. As the individual particles of the feed material is heated and melted in the plasma they form perfectly spherical dense molten metal droplets which cool and solidify as they drop by gravity to the bottom of the processing chamber. Processed powders are recovered at the bottom of this chamber, while the plasma gases and any vapours or ultrafine powders emerge from a side port and are directed towards a sintered metal filter. From the filter, the gases are directed towards the vacuum pumping and exhaust system.

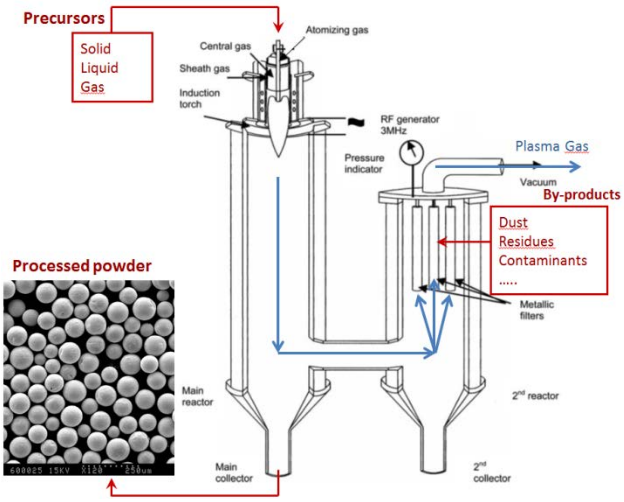


Fig. 1. Tekna's powder spheroidization process.

2.1. Titanium powder

The Hydride-Dehydride (HDH) process is one of the most widely used techniques for the manufacture of titanium powders from ingots. Unfortunately since the process involves the crushing of the hydride of the titanium prior to its dehydriding, the powders produced tends to be highly porous (sponge-like) with angular shape which makes them not suitable for additive manufacturing applications.

Tekna's technology can be used to treat such sponge like titanium powders in order to densify, spheroidize and purify the powder. The spherical powders produced meet all the requirements for powders dedicated to additive manufacturing. Typical results obtained with pure titanium and titanium alloy grade 23 manufacturing (Ti-6Al-4V powder with an oxygen level below 1000 ppm) are presented.

2.2. Titanium matrix composite

The plasma system can be used as a chemical reactor in order to control the chemical reaction between the plasma and the materials in a molten state. The approach can be used to functionalize the surface of the particles by acting like a chemical vapor process (deposit of a material) or by controlling the chemical reaction only on the surface of the materials (oxidation, reduction, carburation …).

In present review, the possibility to manufacture metal matrix composite powder such as Ti/TiC or Ti/TiN using Tekna's ICP technology will be also discussed. This opens more unique opportunities to Additive Manufacturing applications. In fact, it is not possible, or at least very difficult, to produce parts reinforce with different materials directly into the Additive Manufacturing machine. The development of such materials offers the possibility to develop new parts with specific structure or mechanical properties and could allow more important reduction of the cost of manufactured parts.

3. Results

3.1. Titanium powder

Typical results of the plasma treatment of HDH Ti powders using ICP technology are given in Fig. 2. Electron micrographs of the feed material are given in Fig. 2a, while the as-treated powder is shown in Fig. 2b. The latte show in addition to powder spheoridization though plasma treatment the deposition of fine lose debris "satellites" on the surface of the particles. This is due to the partial vaporization of the particles during its plasma heating and melting, followed by the condensation of the formed vapours on the surface of the particles in the cooling phase of their trajectory. These soot particles are softly deposit on the surface of the larger particles, and can be easily removed by a proprietary online classification process developed by Tekna as demonstrated by the quality of the powders shown in Fig. 2c which were obtained after post-treatment and classification of the plasma treated powder (Fig. 2b).

It should be pointed out that the vaporization of a fraction of the materials during the plasma treatment can also allow the purifying of the treated materials. The process is essentially one of distillation of the light weight elements or compounds from the particles. Typical results for two cases of CP-Ti powder purification are given in Table 1 where a significant decrease of calcium, and chlorine content is observed after each treatment.

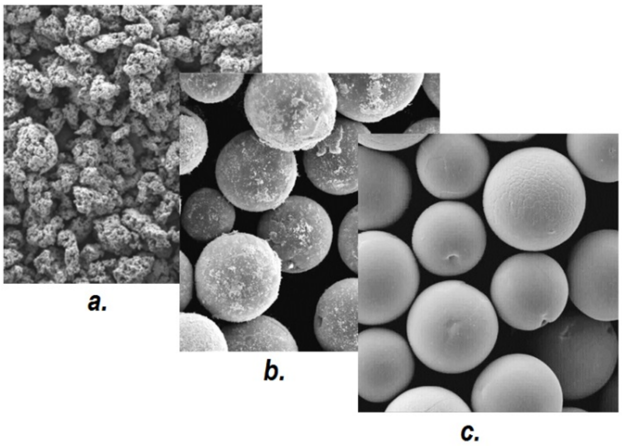


Fig. 2. Electron micrographs of Titanium powders (a) before treatment (b), after plasma treatment and (c) after post-treatment and classification.

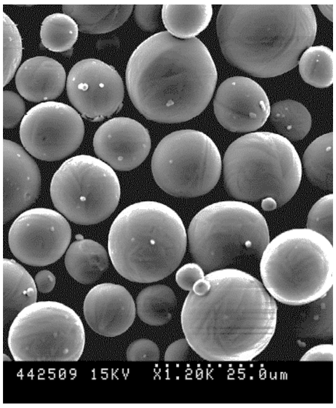
Table 1**.** Effect of the plasma treatment on titanium powders.

Properties	Case1		Case2	
	Before treatment	After treatment	Before treatment	After treatment
Particles size distribution	40-120μm	40-120μm	90-340μm	70-200μm
Flow ability	No Flow	37 s/50g	50 s/50g	23 s/50g
Apparent density	0.8 g/cc	2.4 g/cc	1.89 g/cc	2.74 g/cc
Tap density	1.0 g/cc	2.6 g/cc	2.32 g/cc	2.95 g/cc
Purification (ppm)	**C** = 160 **H** = 166 **N** = 90 **Cl** = 1200 **Al** = 80	**C** = 110 **H** = 112 **N** = 90 **Cl** = 360 **Al** = 52	**O** = 3017 **Ca** = 4600 **Cl** = 7900	**O** = 2100 **Ca** = 250 **Cl** = 700

Finally, the Tekna's plasma process not only allows to produce pure titanium, but it is also possible to pre-alloyed (pre-alloying or agglomeration of different materials) the raw materials in in order to produce powders with a controlled chemistry and oxygen level such as the titanium alloy grade 23 (Ti-6Al-4V). The following micrograph (Fig. 3) highlights the possibility to produce highly spherical titanium alloy powder with exceptional properties.

3.2. Titanium matrix composite

As mentioned earlier, the plasma can be used a chemical reactor. During this experiments, some agglomerated powder (spray-dried and/or freeze dried powders) of different elements (Al, V, Ti, C) were used (see Fig. 4). During their dwelling time into the plasma the agglomerates were melted and the chemical reaction occurs. These chemical reactions are controlled by the operating plasma conditions (power, chemistry) and the



Size = 15-45µm

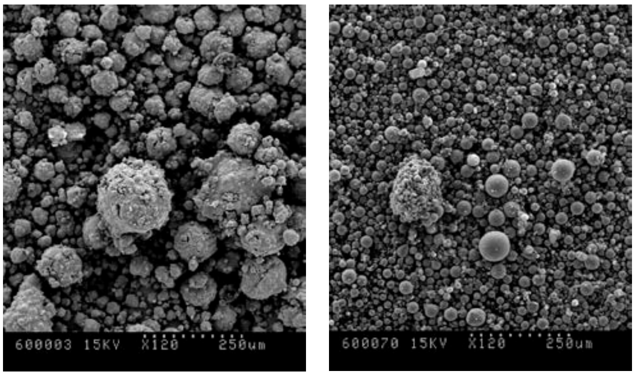
Apparent density = g/cc
Tap density = g/cc

Flow ability = 27.5 s/50g

Chemical analysis (by weight):
Al = 6.18%
V = 4.11 %.
O = 870 ppm
C = 60 ppm
H = 21 ppm
N = 76 ppm

Fig. 3. Ti-6Al-4V grade 23 powder produced by using Tekna's ICP technology.

residence time in high temperature zone. The addition of a hot section or a quench section into the reactor, would allow a closer control of the temperature history of the particles and accordingly the chemistry of the treated powder.



a. Feedstock = Ti+Al+V+C ***b. Processed powder***

Fig. 4. Electron micrographs of Titanium allow powders (a)Feedstock used (b), plasma processed powder.

The adjustment of all the operating parameters allows for the production of Ti-Al-V materials with TiC or TiN reinforcement directly through the chemical reaction between the elements introduced into the plasma as shown by the X-ray patterns of the treated powders given in Fig. 5.

The development of metallic matrix composite materials opens vast opportunities to produce new materials with high mechanical properties which have never been obtained in the past. Tekna's plasma technology offer a unique process which allow to control in the same time the morphology, the size and the chemistry of the powder produced.

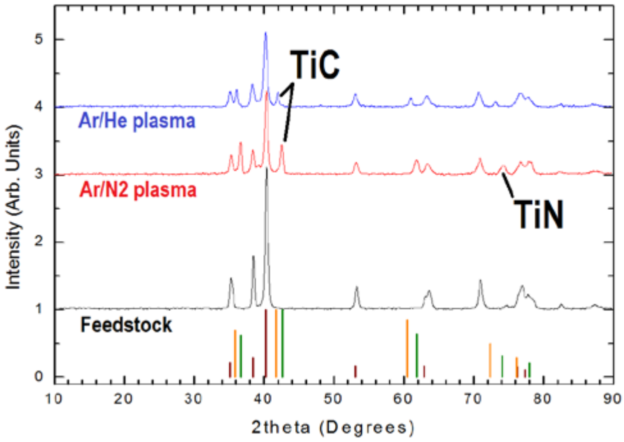


Fig. 5. X-ray diffraction patterns of the processed powders depending on the plasma operating conditions.

4. Conclusion

As demonstrated in this review, Tekna's ICP technology can be used for production of powders with tailored morphological and chemical properties. These include though not limited to;

- Spherical shape;
- High pack density;
- High Flow ability;
- High purity;
- Controlled chemistry;
- Controlled oxygen content.

The availability of such high quality powders in the particle size range of -45+20 um, makes them particularly compatible with the stringent requirment of the rapidly growing Additive Manufacturing industry.