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REVIEW

Toward high performance in Powder Metallurgy

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ABSTRACT: Powder Metallurgy (PM) is technology well known for mass production of parts at low cost but usually with worse mechanical properties than same parts obtained by alternative routes. But using this technology, high performance materials can be obtained, depending of the processing route and the type and amount of porosity. In this paper, a brief review of the capabilities of powder technology is made with the objective of attaining the highest level of mechanical and physical properties. For this purpose, different strategies over the processing can be chosen: to act over the density/porosity level and properties of the pores, to act over strengthening mechanisms apart from the density of the material (the alloying system, the microstructure, the grain size,..), to improve the sintering activity by different routes and to use techniques that avoid the grain growth during sintering.

KEYWORDS: Advanced materials; Powder metalurgy

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RESUMEN: *Hacia las altas prestaciones en Pulvimetalurgia.* La Pulvimetalurgia es una tecnología bien conocida por su faceta de producir piezas de forma masiva a bajo coste, pero habitualmente con una pérdida de propiedades mecánicas si se la compara con tecnologías alternativas para obtener las mismas piezas. Sin embargo, mediante esta tecnología, también se pueden obtener piezas de altas prestaciones, dependiendo de la ruta de procesado y del nivel de porosidad. En este trabajo, se realiza una sucinta revisión de las posibilidades de la tecnología de polvos que permitirían obtener los mayores niveles de prestaciones en cuanto a propiedades mecánicas y físicas. Se pueden elegir distintas estrategias en el procesado: actuar sobre el nivel de densidad/porosidad y las propiedades de los poros, actuar sobre mecanismos de endurecimiento distintos a la densidad (el sistema de aleación, la microestructura, el tamaño de grano,…), mejorar la activación durante la sinterización y utilizar técnicas que inhiban el tamaño de grano durante la sinterización.

PALABRAS CLAVE: Materiales avanzados; Pulvimetalurgia

1. INTRODUCTION. POWDER METALLURGY, A WAY TO PRODUCE HIGH PERFORMANCE MATERIALS?

Powder Metallurgy (PM) is a well-known technology to produce parts of small size and complex shapes at low cost. This could be one of the most important niches of applications for this technology, where an easy manufacturing route of one simple press step followed by one single sintering step is produced. This manufacturing route could be called the mass production PM method, where cost is the main parameter to take into account and properties, always under the engineering requirements, are in a second level of request.

But PM can fulfil further fields of applications. The first one, which is maybe the most known, covers a wide range of applications where price is the driving force in the manufacturing process. In general, these are mass production processes, where PM can supply parts of small size and complex shapes at low cost when compared with alternative technologies. One typical example of this niche is the synchronizing hub for manual transmission of cars (García *et al.*, 2013). This is due to the energy involved in the PM process being much lower than that of alternative technologies, and machining is highly avoided. A typical PM process for this field of application involves uniaxial press and sintering in two different steps.

When pressure and temperature (sintering) is applied in two different steps, the final density of the part never reaches the theoretical density of the material, and as a consequence, it is obtained a part with some remaining porosity that can affect the final properties. If it is compared the properties of these materials against a wrought material we have to consider the microstructural features of each family of materials (PM against wrought). In a wrought material we have a full density material, but usually with a textured microstructure. On the contrary, in a conventional PM materials (made by a mass production method) what it can be expected is a non-textured material, but with a certain amount of porosity. Despite sintering is a relatively high temperature treatment, in PM materials the grain size is usually lower than the expected, because porosity has some anchor effect in grain growth. Despite the resulting properties tend to be worse than through alternative processing routes, there is a better relationship between price and performance with PM processing.

One of the main advantages of the PM process, even more than the price, is the dimensional tolerances that can be reached. In Figure 1, some of the competitive advantages of PM can be observed relative to other forming techniques in terms of their dimensional performance (Tengzelius, 2005), and as shown in Figure 2 (European Powder Metallurgy Association, 2014), in terms of their energy consumption and materials waste.

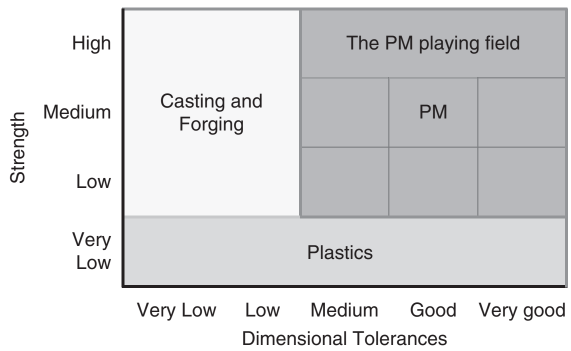

FIGURE 1. Definition of the "Powder Metallurgy Playing Field" related to mechanical strength and dimensional tolerances.

When PM is used as a mass production technique, and a certain level of porosity is assumed in the resulting part, the final properties of the material will be different than the equivalent material obtained through any technology that allows the part to be formed with the theoretical density of the materials. How porosity affects the mechanical properties in sintered materials can be observed in Figure 3 (Höganäs AB, 2002). Low amount of porosity reduced dramatically the properties associated with the ductility of the material, and thus, in applications where high ductility features (such as elongation or impact strength) are required, it is necessary to decrease the porosity level or at least to increase the quality of the pores (roundness, distribution, …).

Taking into account that the improvement of the properties in a PM material occurs through improvement of the density level, we have to consider another two important niches of applications for PM. The second one field of application is related to those materials that only can be obtained by a PM route. There are some important examples for this group of applications, such as the cemented carbides or the most of refractory metals. In these cases, the powder route is the unique route suitable to obtain the desired features in the material. Another example are the self-lubricating bearings, a porous material with special features impossible to obtain by any other alternative processing route.

Finally, there is a third field of applications for the PM materials, and is directly linked with the need of having densities near the theoretical density of the materials. As much as a PM material reach a density near the theoretical one for the specified material, we take all the advantages of using the powder route but without the usual and inherent defects related with the porosity. There are methods to obtain full density materials from powders allowing to have non-textured microstructures, or even with very small grain sizes with the subsequent

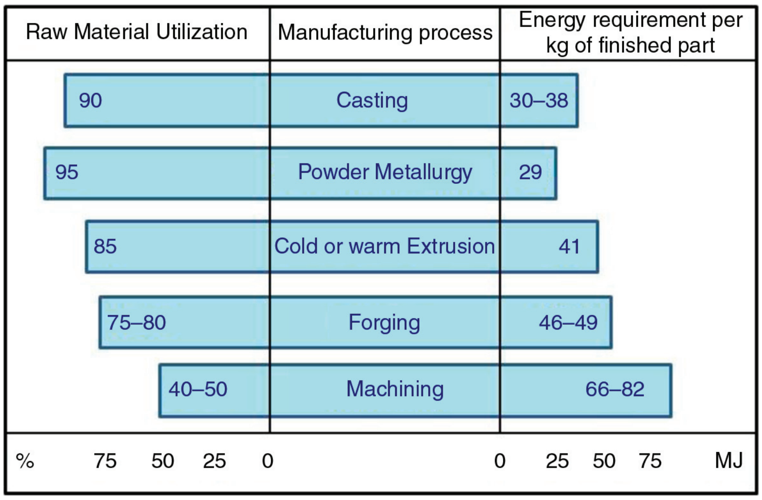

FIGURE 2. Energy and raw material utilization for different manufacturing processes.

positive consequences in the mechanical properties. It is perfectly possible in terms of technology to produce from powders fully dense materials, even with very small grain size, being just a matter of prize. This third niche of applications means levels of properties and performance much higher than the same material obtained by any other manufacturing route. That means that PM is the main way to obtain many of the so call "advanced materials".

This paper tries to explore the way to achieve the high performance in PM, that most of the times run in parallel with the way to obtain full density, but not only (Campos *et. al.*, 2008).

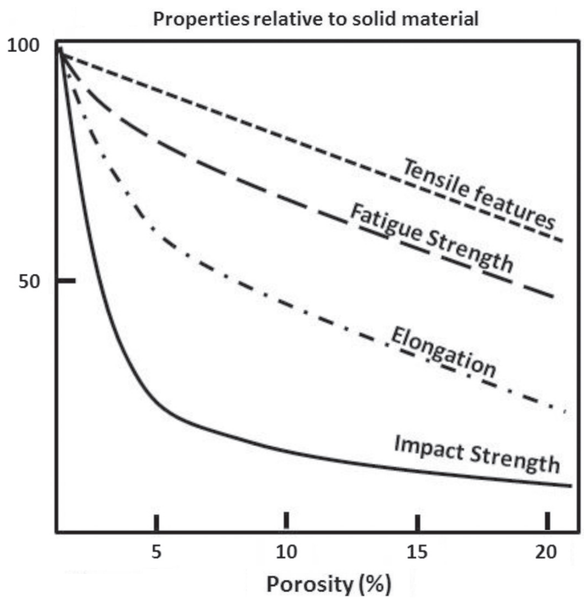

FIGURE 3. Mechanical properties effects due to the level of porosity in PM materials.

2. IMPROVING THE PROPERTIES WITHOUT INCREASING THE DENSITY LEVEL

2.1. Acting over the alloying system

The properties of metals and alloys can be improved by acting on the alloying system. The clearest example is the evolution of PM low alloyed steels since their introduction in the market in the 1930s. In the evolution of this family of steels, the improvements in properties have been attributed to an increase in the density (from values near to 6.5 g cm^{-3} to 7.3 g cm^{-3}). However, the primary changes in the evolution of properties have been obtained through changes in the alloying system.

We have followed the evolution of materials with poor mechanical properties and little uniformity (in the sense of reproducibility) to materials that today are produced with high reliability and performance from when low alloyed sintered steels were introduced to the market for structural parts. The working efficiencies of these steels can be equivalent in many cases to the best-wrought steels while maintaining a good margin in terms of cost and market competitiveness. In this paper, a complete review of the topic is presented, from the early stages when the first parts were made by plain iron or iron-carbon, through the different alloying systems: Fe-Cu, Fe-P, Fe-Cu-Ni-Mo, and more recently Fe-Cr-Mo and Fe-Mn. The development of processing routes is also examined. The main milestones in the field of new alloying systems have been: 1) the introduction of Cu in 1960–1970s, 2) the new complex systems with Cu-Ni-Mo in the 1980s and 3) the introduction of alloying elements with high oxygen affinities (in the late 1990s) (Torralba *et al.*, 2011). In Figure 4 (Oro, 2012), we can observe this evolution as a function of the amount of alloying elements

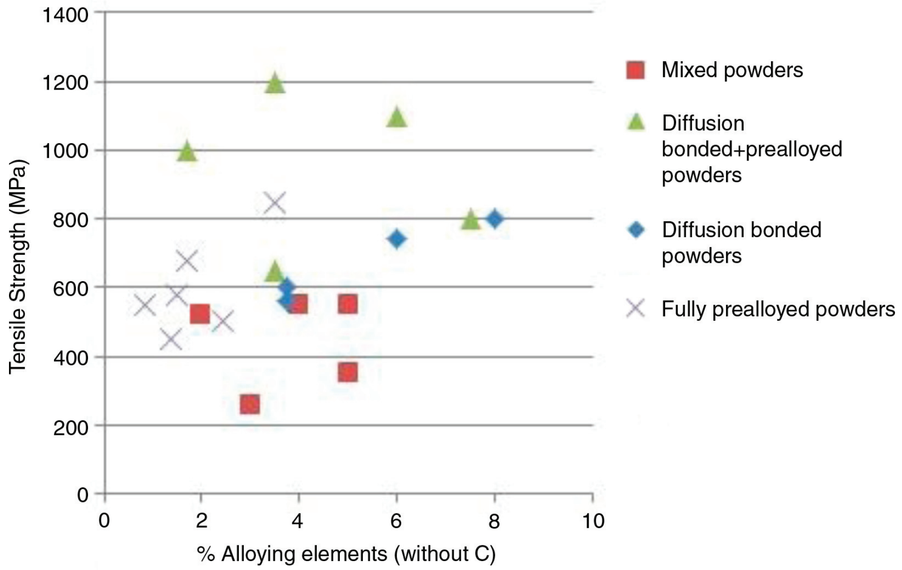

FIGURE 4. Tensile strength for different PM steels (with 0.5% C) manufactured from different kinds of powders and different amounts of alloying elements.

under similar manufacturing conditions. Here, we can clearly observe how the properties acting on the alloying system can be tailored, from the alloying elements to the alloy production (through mixing powders or diffusion bonding or fully prealloying the powders). High strength values can be obtained with small amounts of alloying elements by controlling the type of powders used as raw materials (Oro *et. al*., 2012).

2.2. Acting over the microstructure development

There are many ways to act on the microstructure development without modification of the density of a material. The most prominent are through heat treatments. By heat-treating any alloy, its mechanical properties can be improved (Stoyanova, 2005). In the case of PM materials, heat treatments are similar to those of wrought alloys, when several considerations are taken into account. The primary consideration is the porosity of the material, which tends to reduce the thermal conductivity and cooling rate, and in case of steel, it reduces the hardenability (German, 1994). The presence of porosity is extremely important for thermochemical treatments, especially when the active agent for introducing the change in chemical composition is a gas. Parts with open porosity cannot be efficiently gas surface-treated because any surface hardening is transformed into a full-hardening treatment (Molinari *et. al*., 2001). The composition and alloying method determine hardenability, it can be assumed that prealloyed grades are generally more hardenable than admixed, diffusion alloyed or hybrid alloys, and less hardenable materials need faster quenching rates. The greater the hardenability, the slower the cooling rate needed to produce a martensitic microstructure, and usually, reduced cooling rates result in less distortion. It is necessary to consider that geometrical stability is one of the key aspects for choosing PM technology. Another way to strongly influence the properties is to change the morphology of the pores. With the same amount of porosity, but with different-shaped pores, the mechanical properties can be affected, especially those related to the ductility and the dynamic properties of the material, as described previously. The mechanical properties of PM products depend strongly on porosity features (Stoyanova *et. al*., 2004). Pores reduce strength and cause local stress accumulation such that they act as sites for crack nucleation (Molinari *et. al*., 2011). The effect of porosity on the mechanical properties depends mainly on the following factors: the volumetric fraction of the pores and their interconnection, size, morphology and distribution.

The most important parameters are the total porosity and the shape of the pores or sintering contacts (Blanco *et. al*., 2005). The total porosity is defined by the compaction pressure and the sintering conditions. Accordingly, the sintering contacts can be isolated or interconnected (Danninger and Weiss, 2002). Thus, the porosity results in an out-of-properties decrease in the effective load-bearing cross-section, and it has been shown (Danninger *et al*.,1997) that this parameter is directly related to an alloy's mechanical properties. Nevertheless, not only does the total amount of pores determine the behavior of a sintered part in service, but there are also other singularities of extremely high relevance, such as slag inclusions and pore clusters (German, 1994).

Not only the percentage of pores but also the size of those pores and their shape, along with their distribution within the structure and the contiguity between them, indirectly determine the final behaviour of a material. These factors can be controlled by careful adjustment of the processing parameters (Maroli *et. al*., 2003). Another advantage of powder metallurgy is that porosity can be manage to be of different sizes inside the part to fulfill some specific requirements, like i.e. in biomaterials (Tojal *et al*., 2013).

2.3. Acting over the grain size

The last way in which the microstructural system can be affected is by reducing the grain size. In conventional alloys, grain size reductions are mainly obtained through plastic deformation operations. In PM, grain size reductions can be accomplished in two ways: using precursor powders with small grain sizes (if the original grain size is small, the final one can be more controlled) or by inhibiting grain growth during the sintering step (this second route will be treated later). There are different ways in PM to produce particles with very small grain sizes at the microstructural level. The most important methods are mechanical alloying technologies and rapid solidification technologies.

Mechanical alloying was developed in the 1960s by John S. Benjamin (1970) in the labs of INCO while attempting to develop a way to introduce dispersed oxides in a nickel-based superalloy. The method has been described as being similar to dry milling, and when used to synthetize ceramics is called mechanosynthesis. In all cases, it is considered a high-energy milling method. The main advantage of this process is that chemical compositions that are impossible or difficult to obtain by traditional casting methods can be made, including powders with very poor crystallinity (nanostructured) or even those in an amorphous state. In Figure 5 (da Costa, 1998), one example is shown; on the upper-left, the starting powders in the manufacturing process of a Ni_3Al powder are shown, and on the upper-right, the powder developed after the mechanical alloying procedure can be observed. Metal matrix composite material powders can also be obtained by mechanical alloying. Also in Figure 5, composite powders from an iron-based system (Gordo *et. al*., 2004) and from an aluminium-based system (Fogagnolo *et al*., 2006) can be observed. New families of PM steels have been developed for high performance applications using high energy ball milling (Torralba *et. al*., 2013).

By rapid solidification (Das and Davis, 1988; Lavernia and Srivatsan, 2010), cooling rates from 10^4 to 10^7 K s^{-1} can be understood. At these cooling rates, normal solidification processes occur without development of crystalline-ordered structures. This implies that amorphous or quasi amorphous materials are obtained, or in a worst-case scenario, materials with a nanoscale grain size. As in many other fields related to advanced materials, the best way to obtain materials with these characteristics is in a powder form. One way to obtain these powders is through gas atomizing. If gas is used as an atomizing agent at very low temperatures, cooling rates can be reached for the solidification of droplets via rapid solidification, where the material may be directly obtained in powder form. Other methods to obtain rapidly solidified materials involve the use of "cool wheels". During the "melt spinning" or "melt extraction" method, a wheel (cooled by circulating liquid, Ar, or N inside) extracts pins of rapidly solidified metal from a melt bath. In the "cool wheel" method the molten metal makes contact with the cool wheel and produces small sheets than can be milled into powder form. These methods are widely explained in Jacobson and McKittrick (1994). All these powders must be shaped using special consolidation methods to avoid losing their nanostructured features.

3. IMPROVING PROPERTIES BY INCREASING THE DENSITY FROM HIGH GREEN DENSITY LEVELS

The conventional PM route involves two main steps during the densification of a part: pressing and sintering, and these can be combined to work together. An increase in the final density can be realized by acting on both the pressing step and the sintering step. There are two main technologies used for obtaining green parts (in PM, the 'as pressed' part is the green part) with high-density levels, sometimes resulting in parts near the maximum density.

The first technology is warm compaction. The adjective "warm" in this field refers to a slight increase in temperature, not far from room temperature (and below the recrystallization temperature in cold worked metals). Increasing the temperature of powders and dies can produce two positive and important effects. The first one is a reduction in the yield strength of the powders, and as a consequence, a much higher deformation and green density can be obtained under the same load. In the case of iron-based powders, temperatures in the range of 130–150 °C can reduce the yield strength of the powder by more than 40%. The second positive effect is the reduction of the elastic release (spring back) when the part is extracted from the mould. According to the Long's model (Long, 1960), a reduction in the yield strength of the powder results in the behaviour of the powder as being similar to that of a Newtonian fluid, and consequently, there is a reduction in the radial stress of the die, less opposition to extraction, and less spring back. Another

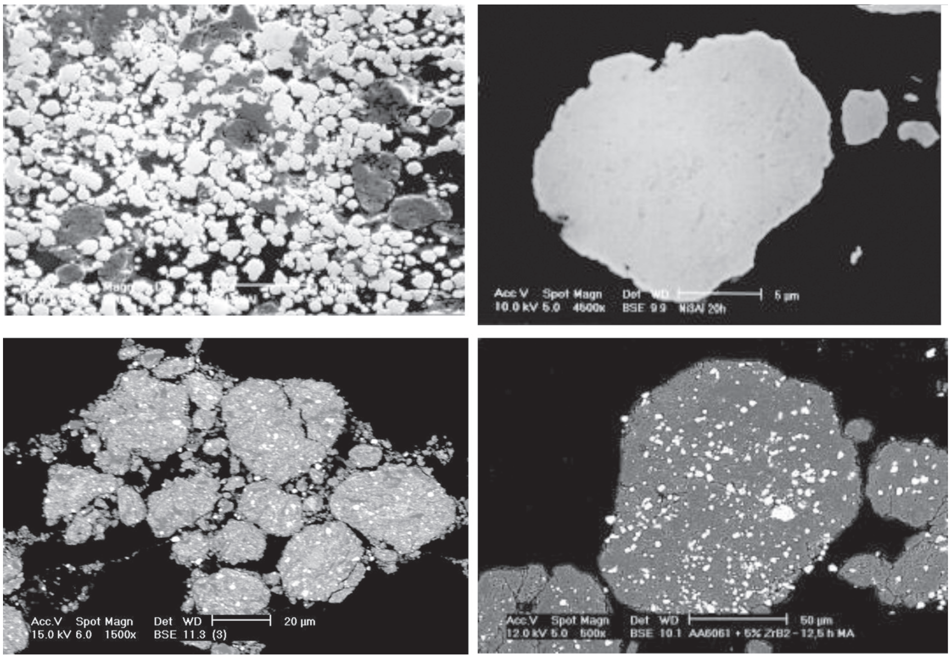

FIGURE 5. Upper-left: mix of aluminum powders (dark) with nickel powders (light). Upper-right: Ni_3Al intermetallic powder obtained after 20 hours by mechanical alloying. Down-left: composite powders obtained by mechanical alloying: iron reinforced with 25% (vol.) TaC-25 (vol.) NbC. Down-rigth: 6061 Al alloy reinforced with 5% (vol.) ZrB_2.

indirect benefit of this temperature effect is a much higher dimensional control. One negative aspect of heating powders is the need for specific lubricants and the trend of agglomeration of the powders. For this reason, the temperature should be lower than 150 °C because from this temperature, the tendency to form agglomerates increases strongly, and the flow behaviour decreases as well (Höganäs, 1998). Green compacts from iron-based powders with densities over 7.3 g cm^{-3} can be obtained through warm compaction and under some conditions, green parts can be green machined. In the last years it was introduced a modification in the process: it is much more economical to warm only the die than the die and the powders and the final results, being slightly lower, are still much more efficient than cold compaction (Li *et. al*., 2002). Using warm die compaction we can obtain also green densities near 7.3 g cm^{-3} with a good optimization of the combination lubricant/pressing design (Warzel *et al*., 2012).

The second technology to be related is High-Velocity Compaction (HVC) or compaction by impact waves. This type of compaction method is based in the production of high deformation in particles by the high-speed application of a large load. At high velocity, a cold welding of the particles is produced, and the consequence is a green compact with high green density and strength. The load in HVC is applied by a hammer with a high mass that hits at high velocity on the upper punch of the compaction system. The punch hits the load to the powder, which is densified depending on the involved energy on the punch impact which is the clue of the technique. In principle, a large energy pulse (controlled by the kinetic energy associated to the stroke length of the hammer, which can be adjusted it) s used to densify the powder. This process was developed commercially in 2001 by Hydropulsor AB (Sweden) (Dore *et al*., 2007). With this technology, PM parts greater than 5 kg can be obtained with small dimensional changes in the process at producing rates of 8–10 parts per minute and with low spring back. In an iron-based powder, densities of 7.5 g cm^{-3} can be achieved, and these densities can be even higher if double cycles are applied. The main limitation of this technology is the complexity of the part, which is limited to simple shapes.

High-velocity compaction principles are based on explosive compaction where a combination of high pressures developed in a very short time in combination with temperature can result in densities near the maximum density. Pressures of 900 GPa applied in microseconds can be generated by the energy released by an explosive charge (Baird and Williams, 1984). This high pressure produces a high level of deformation in the particles, reaching near maximum densities, even in the green state. After sintering, the microstructures and properties obtained as a result of HVC can compete with those obtained by hot isostatic pressing (Cambronero *et. al*., 1996).

4. IMPROVING PROPERTIES BY SINTERING ACTIVATION

Sintering used to be the most critical stage of processing for achieving the final desired properties in a sintered material. If the sintering activity is improved in different ways, the final density of the material may be improved and, consequently, the final properties. There are different ways to activate the sintering process. In this chapter, we will comment four methods, but others exist. Sometimes more than one, and even all of these methods, can be used simultaneously. For example, in Metal Injection Moulding (MIM), small particle sizes are often used with liquid phase sintering. Additionally, in most processes where pressure and temperature are applied at the same time, small particle sizes are used in conjunction with liquid phase sintering.

4.1. By reduction of particle size

The main driving force in sintering is the reduction of the Gibbs free energy, and in a process where particles are used, the main storage site of the system is the surface of the particles. There is a greater potential to lose energy with the high surface area associated with the powders, and as a consequence, the process is accelerated. The surface area can be increased by decreasing the size of the particles and producing particles with a low shape factor (far from the spherical shape, which is the shape with the lowest surface area for a specific volume). It is well known that reducing the particle size we can allow much higher densities at lower sintering temperatures to be obtained. Some PM technologies, such as MIM, use powders of sizes below 25 μm and take advantage of these circumstances to produce high density parts relative to those obtained by conventional press and sinter processes, where the usual particle size is on the order of 80–100 μm. In Figure 6, the effect of the particle size on the densification curves for one specific material is described. Using small particle sizes, we can nearly reach the maximum density at low temperatures.

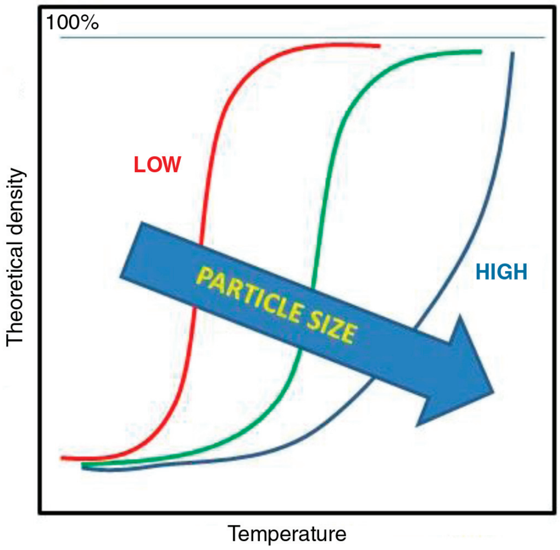

FIGURE 6. Influence of particle size on densification in a sintering process.

4.2. Liquid phase sintering

During sintering, one liquid phase can be formed. This liquid phase normally comes from one minor component of the alloying system; if it was derived from a major component, a loss of shape would be observed. The liquid phase can be permanent if it exists over the duration that the material is at the sintering temperature, or transient if the liquid phase disappears at or below the sintering temperature, due to a total dissolution of the liquid in the solid. The formation of a liquid phase can evolve under different densification situations because a liquid formed in between solid particles with different levels of porosity will behave in different ways. This behaviour depends of the wetting characteristics of the liquid on the solid (usually poor wetting will result in swelling), on the inter-solubility between the solid and the liquid (good solubility of the solid into the liquid tends to improve the densification), and other characteristics. As a result of these parameters, the use of a liquid phase in a sintering process should be carefully studied to assure the correct approach to the densification objective. An extensive review is presented concerning liquid phase sintering, and all methods are clearly studied (German, *et al.*, 2009; Oro *et al.*, 2012a; Oro *et al.*, 2012b; Oro *et. al.*, 2013).

Aside from physical phenomena associated with the formation of a liquid phase during sintering, it is clear that the movement of atoms in the liquid state has much more freedom than in the solid state, and the diffusion phenomena are clearly enhanced. Because of this, LPS is considered a method for enhancing sintering and a common method for improving densification. Occasionally, LPS is so active in enhancing the process evolution that only a few degrees can result in an over-sintering phenomenon.

4.3. Applying pressure and temperature simultaneously

One method for transferring matter during a sintering process is through plastic flow. In a pressureless sintering process, plastic flow can take place due to the movement of dislocations and is usually promoted by the force of gravity. With the simultaneous application of pressure and temperature, plastic flow can be enhanced, as can the densification process, allowing the material to reach its maximum or nearly maximum density.

There are different technological approaches to achieve high densities via plastic deformation, but the two primary methods are hot pressing and hot isostatic pressing.

4.3.1. Hot pressing

In hot pressing, the pressure is applied uniaxially at high temperatures. The term "high temperature" refers to a temperature where the main constituent of the alloying system is in the plastic deformation regime. It can be considered as two types of hot pressing processes. In the conventional hot pressing process, only pressure and temperature (applied by heaters) are applied. If the heat source is an induction furnace, the process is called Inductive Hot Pressing. Another way to heat the sample is with the assistance of an applied electric field. In this case, the heating rate is improved, as is the efficiency of the process. This later process can be considered as the predecessor of Spark Plasma Sintering (SPS). In fact, if the electrical field is applied in a way where high temperature is reached at high rate, the process use to be called "field assisted hot pressing" and similar results (in terms of properties/microstructure) than with SPS can be achieved (Muñoz-Moreno *et. al*., 2013). When this processing system is employed, both pressure and temperature are lower than with the conventional PM process (first pressing and then sintering). This allows the maximum density to be achieved at lower temperatures, resulting in smaller grain sizes than the expected and positive consequences on the properties.

4.3.2. Hot Isostatic Pressing

In Hot Isostatic Pressing (HIP), pressure and temperature are also applied simultaneously, but the pressure is applied through an isostatic media and, as a result, is transmitted to the material with the same load in all directions. The pressure is applied at high temperature, normally with Ar as a fluid, which is a noble gas heavy enough to transmit the pressure even at temperatures higher than 2000 °C. Semi-finished products can be produced directly from powders, or pre-sintered parts with close porosity can be used. HIP can also be used to upgrade castings or to weld dissimilar materials. There is much information related to HIP (Atkinson and Davis, 2000) because it is involved in the development of many "advanced materials", including ceramics, magnetic materials, composites and advanced metals and intermetallics. When HIP is used for processing materials, there are several different advantages: 1) as the pressure is isostatically applied, the part is fully isotropic in all it properties, and this is required for some special applications; 2) as the combined effect of pressure and temperature improves the sintering efficiency, the temperature of the process can be much lower than conventional sintering temperatures, resulting in smaller grain sizes and 3) the maximum density can be nearly achieved, as the residual porosity so small and rounded that it is not detrimental to the mechanical properties of the material. This means that using HIP, we can obtain the "perfect" material, with a high control of the chemical composition, maximum density, isotropic properties, and good control of the microstructure with small grains. This has many of the advantages of using powders as raw materials but without the disadvantage of the inherent porosity obtained with conventional PM processes. Today, HIP can be scaled up to obtain large ingots (on the order of tons) to reduce the cost of the material.

The HIP technology is not only used to consolidate powders to produce large PM semi products, but for other interesting applications, such as the elimination of porosity from manufactured components from other processing routes (castings, presintered PM materials, infiltration techniques, etc.), to produce a functionally gradient or bimetallic materials where a metal or alloy powder is diffusion clad onto a substrate of similar or dissimilar metals to produce internal or external coatings for improved wear, corrosion or heat resistance, and also in the case of very expensive parts and the rejuvenation of service-affected components. May be HIP technology is the materials forming technology with higher relationship with the so call advanced materials, and materials involved in the development of cutting edge technologies such as defence or aerospace (Rao *et al*., 2012).

5. INHIBITING THE GRAIN GROWTH DURING SINTERING

There are different processing methods that can reduce the risk of grain coarsening during sintering. Two of them are examined below.

5.1. Microwave sintering

When microwaves are used to heat a green compact, both electric and magnetic fields act to develop the sintering process. This means that heating takes place quickly, and higher densities can be achieved than with conventional sintering. It is unclear how to evaluate the effect on the properties that the method used to introduce heat on the green part. And the consequence is that the effect of the high densification that takes place with this variety of sintering is not well understood. What is clear is that desirable properties are reached due to the shorter sintering times and lower sintering temperatures. Microwave sintering was first developed for ceramics (Sutton,

1989) and cemented carbides and, more recently, for metals (Roy *et. al.*, 1999). Microwave chambers are capable of processing a wide range of different shapes and sizes and using temperatures from room temperature up to 2000 °C.

5.2. Spark Plasma Sintering

Spark plasma sintering can be viewed as an evolution of other methods, such as hot pressing or electrical resistance sintering, or a combination of many methods. The use of an electrical current as a means of activating the sintering process was recognized in 1933 (Taylor, 1933). This method, as it is known today, has been developed in the last few decades under different names, such as "Plasma Assisted Sintering – PAS" (Shon and Munir, 1995), "Electric Pulse Consolidation – EPAC" (Mishra and Mukherjee, 2000), "Pulse Electric Current Sintering – PECS" (Xie *et. al.*, 2005), and the most widely used "Spark Plasma Sintering – SPS" (Tokita, 1999). The name SPS has been most widely used in the literature, even taking into consideration that the name PECS is probably more accurate because the formation of plasma during sintering has not been well demonstrated (Munir *et al.*, 2006). The SPS method can be viewed as an evolution of the field assisted hot pressing method. A high DC current is used along with uniaxial pressure. The main difference between SPS and conventional hot pressing methods is that in SPS, the current is applied as many pulses over very short times (less than mili seconds) instead one unique pulse for a longer time. As with most methods where pressure and temperature are applied simultaneously, both pressure and temperature are lower than would be required if applying them in subsequent steps. This means that lower sintering temperatures and shorter holding times are required, and as a result, less grain growth and better properties can be obtained.

The densification mechanisms that take place during this process are not well understood. In conductive materials (metals), it is clear that there is a Joule heating effect when the current is applied, but in the case of nonconductive materials (ceramics), it is palpable that the current goes through the punches and the die (usually graphite). Even the formation of plasma is not clear in the literature. However, what is patent is that SPS allows the processing of materials with maximum density and with the lowest possible grain coarsening; this is a real advantage when nanostructures or nano-powders are used, and the benefits of the nano-features should be maintained during the consolidation of the powders (Mamaedow, 2002; Anselmi-Tamburini *et. al.*, 2005; Chen *et. al.*, 2005; Pimentel *et. al.*, 2012).

Today, SPS is emerging as a possible method to obtain advanced materials instead of the HIP process. Figure 7 can be used to compare the microstructure of an intermetallic alloy, γTiAl, used for aeronautical applications made by two different routes (hot isostatic pressing and spark plasma sintering (Muñoz-Moreno *et. al.*, 2013; Muñoz-Moreno *et. al.*, 2014), in an attempt to obtain similar microstructures. This is an example of the adaptability of PM for obtaining advanced materials with different processing solutions.

Another example is using alternative ways to enhance the sintering process, such as HIP (where pressure and temperature are applied simultaneously) and MIM (where the small size of the particles and liquid phase sintering are taken advantage of), where similar microstructures can be obtained in the end. This is shown in Figure 8 (Torralba *et. al.*, 2000), where almost the same material with similar features can be obtained by HIP and MIM. SPS has also been successfully used to develop ODS steels (Torralba *et. al.*, 2013).

In Figure 9 (Alvaredo *et. al.*, 2012) it is possible to analyze the multiple effect on microstructure and on properties of all these methods, which takes profit of the synergies of pressure and temperature. In materials processed by SPS, the bright carbides

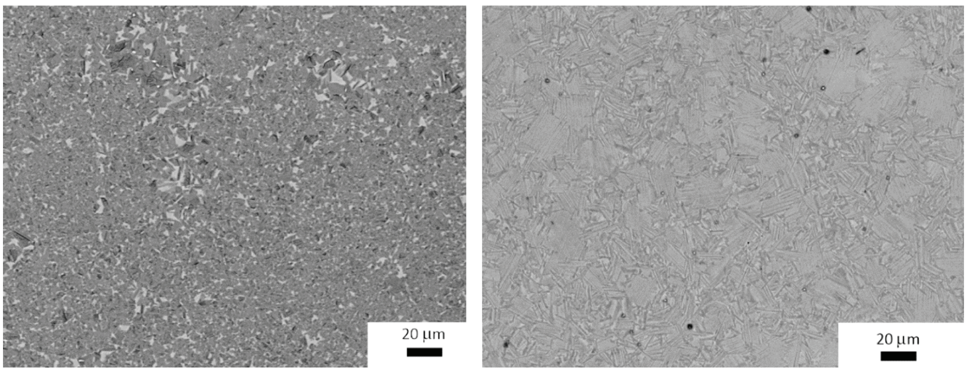

FIGURE 7. γTiAl powder processed by HIP (left) and SPS (right).

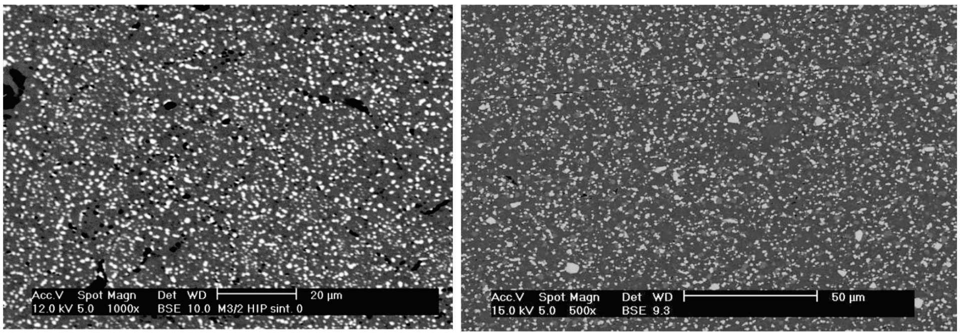

FIGURE 8. High-speed steel manufactured by HIP (left) and metal injection molding (right).

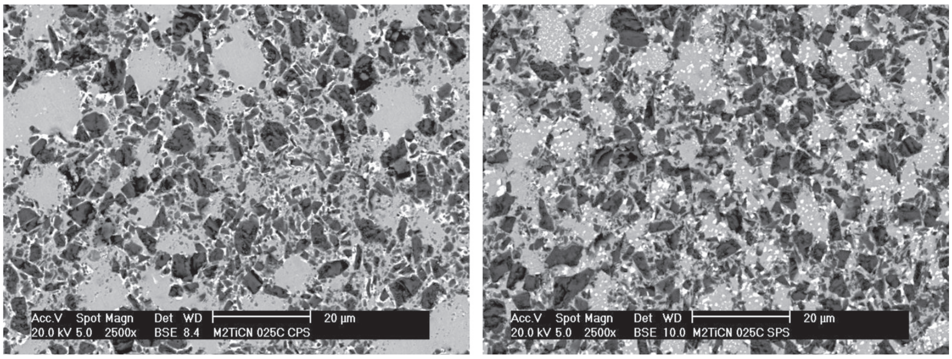

FIGURE 9. SEM microstructures of [M2 + 0.25 wt% C]/50 vol % TiCN processed (left) pressing at 700 MPa and sintering in vacuum at 1400 °C, 60 minutes, and (rigth) Spark Plasma Sintering (SPS) at 1100 °C and 60 MPa during 10 minutes under vacuum.

are precipitated inside the original steel particles. In the case of the press and sintered material, the carbides are precipitated in the interphase steel-TiCN, due to the different sintering mechanism. Thanks to this, the hardness of the SPS material is much higher than the press and sintered one , and hardness is the key property in this composite material.

6. ADDITIVE TECHNOLOGIES

All additive technologies are based in constructing parts layer by layer, taking advantage of the powder route and of the CAD/CAM systems. In the field of metals, there are several terms used to describe additive technologies, and most of these technologies are fully linked with a machine manufacturer. Some of these technologies include Laser Melting (SLM), Direct Metal Laser Sintering (DMLS), and Laser Powder Bed (LPB or LaserCusing). The process usually involves the selective melting of a powder layer using a laser or an electron beam. As soon as the powder has been melted, the bed is moved down to allow a new layer of powder to be deposited and then melted again. All of these processes can produce a full 3D part with high precision and maximum density. To manufacture parts by these methods, the powders used are gas atomized, and are of similar sizes and grades to those used for metal injection molding or hot isostatic pressing (sizes under 25–30 µm). These methods are highly recommended for a low size production series of parts with high added values.

7. CONSIDERATIONS

Several methods to improve the physical and mechanical properties in PM products have been described. All methods show the ability to reach near the maximum density and, as a consequence, to take advantage of all the benefits of working with raw materials in powder form (chemical control, no segregation, grain size, etc.), diminishing the negative and the more detrimental aspect of porosity. Any of these methods can be selected to obtain an advanced material, and many are complementary and can result in a much greater improvement in the properties of the resulting component. This is the primary challenge in the methods using powders: how to reduce the grain size without losing desirable properties. Overall, advanced PM allows the development of new and emerging materials with outstanding properties. In some previous

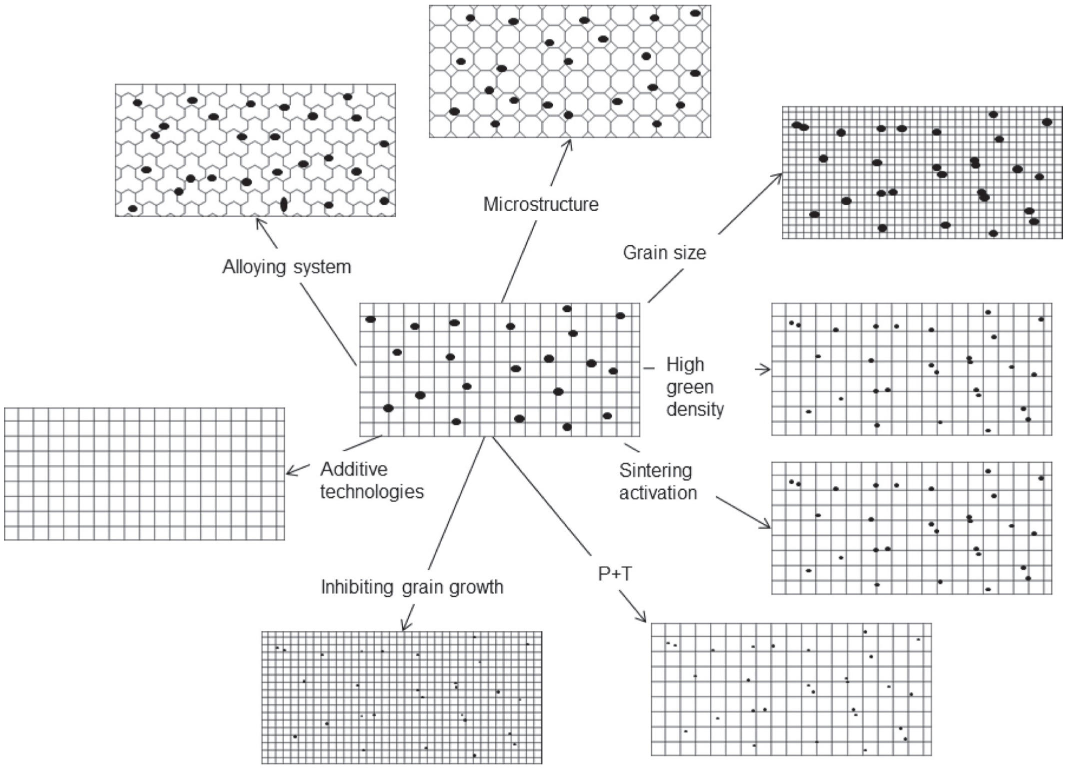

FIGURE 10. Tailoring the microstructure from different PM routes.

figures (7, 8 and 9) it can be seen these versatility of PM as technology that, starting from powder, can follow several manufacturing routes, to achieve a determined microstructure/performance in a component, and this is not only possible in the metallic field, but also with ceramic materials (Delaizir *et. al*., 2012).

As a final summary, in Figure 10 can be seen the flexibility of PM to tailor the microstructure from different processing routes. Departing from a conventional PM porous material (in the centre of the figure) it is possible to move to different situations in terms of density (porosity) and grain size, giving to the materials designer multiple possibilities of performance without change the alloy.

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