

REVIEW

Permanent magnets and its production by powder metallurgy

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ABSTRACT: In this work, the historical relationship between permanent magnets and powder metallurgy is reviewed. Powder metallurgy is a manufacturing technique based on the compaction of powders that are sintered to create a solid product. This technique was used in the production of permanent magnets for the first time in the 18th century and, nowadays, most permanent magnetic materials are manufacturing by this mean. Magnetic properties are highly dependent on the microstructure of the final product, the magnetic alignment of domains and presence of porosity, to mention a few, and powder metallurgy enables fine control of these factors.

KEYWORDS: Magnetic materials; Permanent magnets; Powder Metallurgy; Sintering

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RESUMEN: *Imanes Permanentes y su Producción por Pulvimetalurgia.* En este estudio se ha revisado la relación histórica entre la pulvimetalurgia y los imanes permanentes. La pulvimetalurgia es una técnica de fabricación basada en la compactación de polvos que son sinterizados para crear un producto sólido. Esta técnica fue utilizada en la producción de imanes permanentes por primera vez en el siglo XVIII y, hoy en día, la mayoría de materiales magnéticos permanentes se fabrican de esta forma. Las propiedades magnéticas dependen de la microestructura del producto final, de la alineación de los dominios magnéticos y de la presencia de porosidad; y la pulvimetalurgia permite un control adecuado de estos factores.

PALABRAS CLAVE: Imanes permanentes; Materiales magnéticos; Pulvimetalurgia; Sinterización

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1. POWDER METALLURGY

Powder metallurgy is a production technology that encompasses the manufacture of products from powders by sintering. Powder metallurgy processing involves (i) mixing together the different powders that are part of the alloy, then (ii) compacting

the powder and, finally, (iii) heating the compact, usually under a protective atmosphere, to a temperature below the melting point of the main constituent. As each of these steps comprises a number of factors, they *should* be studied individually focusing on their specific application in permanent magnets.

1.1. Alloy preparation

Most powder metallurgy *processes* start by mixing the elements in their pure forms or as compounds. In this case, the elements are added in the required percentages to reach the defined composition. This is the case for aluminium-nickel-cobalt (Al-Ni-Co) magnets and ceramic magnets.

However, both rare earth magnets samarium-cobalt (Sm-Co) and neodymium-iron-boron (Nd-Fe-B) cannot be manufactured from pure elements due to the high reactivity of some of them. Therefore, casting of an ingot is required, followed by either mechanical breaking (jaw crushing, hammer crushing) or hydrogen decrepitation (HD); and then milling as a mean to pulverize and decrease the particle size of the resultant powder.

Hydrogen decrepitation was first proposed in 1979 to process Sm-Co magnets overcoming the difficulties encountered with mechanical breaking (Harris *et al.*, 1979). Mechanical breaking of cast ingots is difficult as a consequence of the presence of iron-based precipitates within the microstructure formed during solidification due to a peritectic reaction. This hydrogen-based technique was later applied to the production of Nd-Fe-B magnets (Oesterreicher, 1984; Harris *et al.*, 1985; Wiesinger *et al.*, 1987).

The two principal conventional casting methods are induction-melting and co-reduction of oxides. In the latter, the rare earths are introduced as oxides and the transition metals as metal powders as well as calcium granulate as a reducing agent. The rare earth metal diffuses into the transition metal powder resulting in an alloy mixed with calcium oxides; which are lately leached by aqueous solutions (De Castro *et al.*, 2014). This calciothermic reduction process is used in Sm-Co, but with Nd-Fe-B there are several problems (Strnat, 1990). The induction-melting method uses the metallic components in either pure form or master alloys, although in industry the latter is preferred over the former; which are melted together under argon and then poured and cast into thin moulds. This process is preferred over the co-reduction for the production of Nd-Fe-B alloys.

The oxygen content using this technique is below 200 ppm (Corfield, 2003). In induction-melting casting, α -Fe dendrites appear due to the peritectic reaction of the $\text{Nd}_2\text{Fe}_{14}\text{B}$, which enhances the difficulty of making powder and hinders grain alignment in the subsequent processing (Fidler and Knoch, 1989). Furthermore, large Nd-rich areas, which are sensitive to oxidation, are present in the ingot. Therefore controlling the amount of α -Fe and the Nd-rich phase presence is an important issue.

For Nd-Fe-B production the strip casting process can also be used. It entails pouring the molten alloy, through a series of ceramic tundishes, onto a water-cooled copper wheel, which cools the material at $10^4\text{ }^\circ\text{C s}^{-1}$ rates, enabling the continuous formation of few millimetres thickness flake-shape ingots.

Strip casting can enhance the cooling rate, thus suppressing the formation of α -Fe dendrites and improving the distribution of the Nd-rich phase. The strip casting technique is employed to obtain a favourable microstructure during casting and the magnetic properties of the sintered Nd-Fe-B can be expected to improve (Pei *et al.*, 2002).

The HD process, shown schematically in Fig. 1, relies on the hydrogen absorption of the alloy. The hydrogen absorption consists of two stages. First hydrogen is preferentially absorbed at the grain boundary phase which causes a volume expansion in the intergranular regions leading to intergranular cracking. The heat generated by this reaction causes hydrogen absorption into the matrix phase forming a hydride, which results in transgranular cracking. The hydrogen absorption is exothermic and is accompanied by a volume expansion. The resulting powder needs to be handled in an inert protective atmosphere due to its high reactivity with oxygen.

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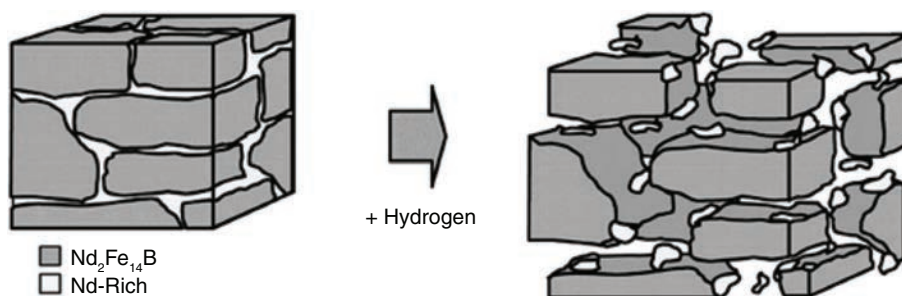


FIGURE 1. Schematic of the hydrogen decrepitation process of Nd-Fe-B magnets (Williams, 1994).

is accompanied by a volume expansion generating internal stresses that cause transgranular cracking and final breakup of the alloy into a coarse powder. The resulting powder needs to be handled in an inert protective atmosphere due to its high reactivity with oxygen.

The main objective of milling the alloys is to produce a narrow particle size distribution. The two more common milling techniques are mechanical milling and jet milling. Mechanical milling is performed utilising hardened steel balls in an organic liquid media which is used to aid dispersal of the material to ensure good particle size distribution, and to act as a coolant and to limit oxidation. Due to the introduction by the organic liquid of carbon, oxygen and water, this technique is widely employed in small scale laboratory research and not in the commercial route, where jet milling is more common.

In jet milling the powder is subjected to high pressures gas jets, between 5 and 10 bar, resulting in a fine powder by colliding the particles at each other. This milling technique presents a sharper particle size distribution and lower oxygen content when compared to mechanically milled powder (Corfield, 2003).

1.2. Pressing and aligning

For the purpose of achieving good magnetic properties, the easy axis of all the powder particles must be aligned in the direction of the final magnetisation. This is done by magnetically aligning the powder in an applied field which is followed by a pressing stage to keep the orientation in the pressed compacts. These pressed compacts, which are also known as green compacts (EPMA, 2008), have typically a density of around 60 % of the theoretical density of the alloy. At this stage, the compacts maintain their shape by virtue of cold-welding of the powder grains within the mass. The compacts must be sufficiently strong to be handled safely and avoid its breakage. This is a critical operation in the process, since the final shape and several properties are essentially determined by the level and uniformity of the as-pressed density.

There are three main pressing and aligning techniques, as can be seen in Fig. 2: (a) axial pressing, in which the compact is pressed in the presence of

a magnetic field parallel to the pressing axis; (b) transverse pressing, in which the compact is pressed in the presence of a magnetic field perpendicular to the pressing axis; (c) isostatic pressing, in which the magnetic field is applied to the compact and is then isostatically pressed. The degree of alignment of isostatically pressed compacts is higher than that of axial or transverse pressed compacts, hence leading to higher magnetic properties. In addition to press-based techniques, mixed powders can also be pressed to shape using an injection system.

1.3. Sintering and heat treatment

Sintering is a heat treatment applied to a powder compact by which particles are fused together to form a solid, dense material at a temperature below the melting point of the major constituent. Sintering occurs by solid state diffusion which implies transportation of mass in a number of different ways, as is shown in Fig. 3.

The sintering of permanent magnets is carried out in an inert gas atmosphere (Ar, H₂) or under vacuum and a constant and well-defined sintering temperature is necessary in order to ensure good final density of the magnet. A final density above 95% of the theoretical should be achieved to minimise porosity, which could lead to oxidation and ageing

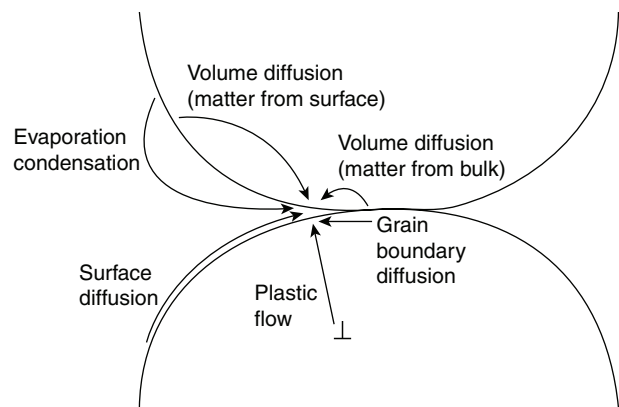


FIGURE 3. Schematic of transport mechanisms in solid state diffusion between two particles in contact (Blackford *et al.*, 2012).

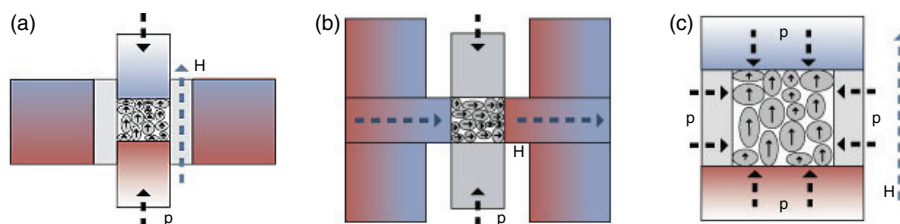


FIGURE 2. Schematic of (a) axial pressing, (b) transverse pressing and (c) isostatic pressing; where the pressure is denoted as p and dashed in black; and the applied field is denoted as H and dashed in blue (Vacuumschmelze, 2012).

during use. The sintering process and the final density are enhanced by the presence of a liquid phase.

Liquid phase sintering involves the presence of a low melting point element that melts and acts as a sintering aid. As a consequence, densification can occur without significant grain growth.

The driving force for densification during sintering is the capillary pressure and the surface tension, although both grain boundary diffusion and volume diffusion also play a relevant role during densification. When surface energies are dominant, liquid-phase densification occurs in stages as shown in Fig. 4 (Thümmeler and Oberacker, 1993; German, 1996; German *et al.*, 2009).

These stages occur sequentially during heating with certain overlap after the liquid forms. The first stage is rearrangement, in which, as the liquid melts, it wets the surface of the particles and the capillary action forces the liquid to enter inside the pores and cause grains to rearrange into a more favourable packing arrangement.

The degree of wetting, also known as wettability, is characterised by the contact angle, θ , which is the included angle in the liquid as shown in Fig. 5.

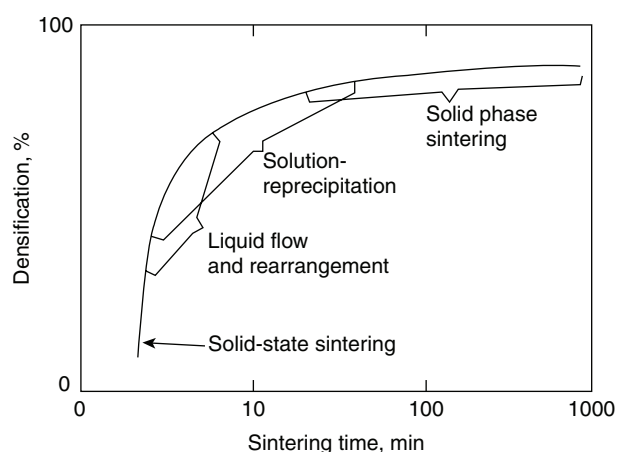


FIGURE 4. Schematic of the liquid phase sintering stages (German *et al.*, 2009).

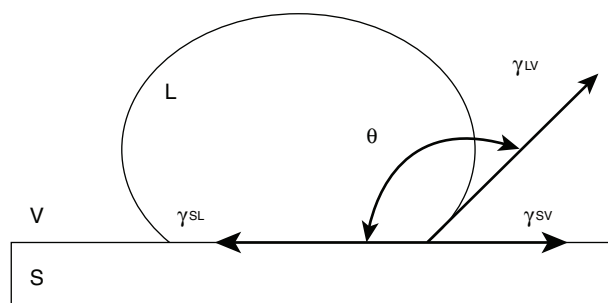


FIGURE 5. Schematic of the solid-liquid-vapour equilibrium where θ is the contact angle, and γ^{SL} , γ^{LV} , and γ^{SV} are, respectively, the interfacial energies at between the solid-liquid, liquid-vapour and solid-vapour interfaces (Vicente *et al.*, 2012).

When a liquid forms, the microstructure consists of solid, liquid and vapour phases. The contact angle represents a balance of the three involved interfacial energies; and the lower the value, the better the wettability. A low contact angle increases the capillary force and the amount of rearrangement, whereas with a large contact angle no rearrangement is possible and the liquid causes separation of the particles.

The second stage is solution-reprecipitation where solution refers to solid dissolution into the liquid in areas where capillary pressures are high and reprecipitation refers to solid leaving the liquid by precipitation on existing grains. Solution-reprecipitation contributes to grain coarsening and also to densification via grain shape accommodation which in addition helps to eliminate porosity.

Densification occurs, as shown in Fig. 6 by the elimination of smaller grains and their preferential reprecipitation on the larger grains, which allows the release of liquid into any remaining pores. A wetting liquid induces particle contact due to an attractive capillary force. The coalescence of small grains with contacting large grains also contributes to grain coarsening and shape accommodation.

The third and last stage is solid phase sintering and corresponds to a microstructure of connected solid grains with liquid occupying the space in between. In this rigid system grain growth continues and there is liquid movement from efficiently packed regions into pores to increase densification.

Post-sintering heat treatment can increase the magnetic properties of sintered permanent magnets, i.e. the coercivity of Nd-Fe-B magnets can increase because of the recovery of defects at the grain boundary phase and the formation of more continuous layers of the Nd-rich intergranular phase (Kaneko *et al.*, 2006).

2. ADVANTAGES OF POWDER METALLURGY IN PERMANENT MAGNETS PRODUCTION

Powder metallurgy is a technology which involves considerable time and energy to convert the starting material to the powder form and shape required and then even more in compacting and producing a solid product. When described in these terms, it may be observed as unreasonable to use this technique to manufacture any product. Nevertheless there are two main reasons for using this technique and, more specifically, using it in the production of permanent magnets. These reasons are cost effectiveness and uniqueness.

2.1. Cost effectiveness

Powder metallurgy is a near net shape processing technique. This implies that the dimensional tolerances of final product can be tightly controlled thus

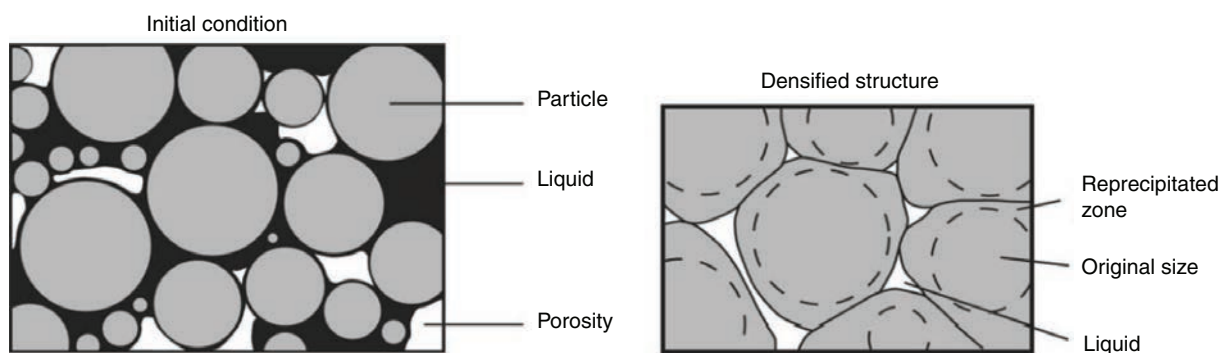


FIGURE 6. Schematic of the solution-recipitation densification where both grain growth and grain shape accommodation act to release liquid to fill pores (German *et al.*, 2009).

minimizing or, in some cases, even suppressing the need of machining. This, added to the fact that very complex shapes can be achieved, results in high material utilization using this technique. Nevertheless, it should be noted that in some cases, e.g. small sintered magnets, material utilization is relatively low due to machining operations. The European Powder Metallurgy Association claims that powder metallurgy has the lowest energy requirement per kilogram of finished part and that the number of manufacturing steps in most cases can be reduced greatly (EPMA, 2017).

Therefore, high material utilisation, low energy consumption and low number of steps are the key cost effectiveness features of powder metallurgy.

2.2. Uniqueness

Some characteristics of the final product can be only achieved when processing and sometimes impossible to yield in conventional processing.

Porosity control and, consequently, density can be carefully controlled by using powder metallurgy. Presence of pores within the microstructure of a permanent magnet is detrimental to the final properties as the pore is an empty volume that does not contribute to the remanence (Zakotnik *et al.*, 2006; Zakotnik *et al.*, 2008; Zakotnik *et al.*, 2009; Degri, 2014; Li *et al.*, 2015). In addition, it may act as a nucleation site which may cause a decrease in intrinsic coercivity by reversing magnetic domains.

Particle size distribution of the feedstock powder is key in the magnetic properties of the final product. It has been demonstrated that using a starting feedstock with a small particle size is beneficial to yield better magnetic properties (Ramesh *et al.*, 1988; Mazda, 1989; Liu and Kim, 1990; Nothnagel *et al.*, 1991; Li *et al.*, 2009). This arises from the lower probability for strong surface defects which could act as a nucleation site, leading to higher magnetic properties (Mazda, 1989). However, decreasing

the particle size of the feedstock powder may result in an increase its reactivity as a result of the raise in specific surface.

Microstructural control is also desired and is achievable by using powder metallurgy. This is especially important in permanent magnets as improvements in magnetic properties lie in a clear understanding and control of the microstructure.

3. EARLY ALLOY

The first magnet known to be made by powder metallurgy was manufactured by Gowin Knight, an English entrepreneur. Knight gained fame by the strength of the magnets he produced, able to lift 28 times its own weight (Uestuener *et al.*, 2006). As he made a fortune by manufacturing such magnets, he did not publish his methods whilst alive. Knight died in 1772 and his method to produce magnets was made public by Benjamin Wilson in 1779 by publishing it in the Philosophical Transactions of the Royal Society of London (Wilson, 1779).

The manufacturing process consisted of adding iron fillings in a tub of water which was stirred for some time until a suspension of very finely divided iron oxide was obtained. The water was poured off and the particles settled forming sludge. This was mixed with linseed oil to create a stiff paste that was moulded into shape and baked before a fire. The resulting block was then magnetised. Therefore, the first time powder metallurgy was applied to the production of permanent magnets was over 200 years ago.

During the 18th century brass was found to be magnetic by hammering and magnetising. In addition, hardened steels were found to be harder to demagnetise and offered a larger strength as a consequence of the domain wall motion restriction due to carbides or a martensitic structure that cause internal strains (Overshott, 1991). Therefore, casting and hardening of steel were the dominant permanent magnet manufacturing method.

4. AL-Ni-CO MAGNETS

In 1930, the first precipitation hardenable magnet alloy based on aluminium, nickel and iron (Al-Ni-Fe) was developed by Mishima in Japan (Mishima, 1931). After this discovery, a new generation of permanent magnets was developed based on Al-Ni-Fe with additions of copper, cobalt, niobium and titanium. These materials were given the family name of the Al-Ni-Co magnets. The coercivity of Al-Ni-Co alloys arises from the shape anisotropy of small ferromagnetic particles of approximately 20 nm in diameter.

Al-Ni-Co magnets can be produced both by conventional casting techniques and by powder metallurgy. Al-Ni-Co alloys are very hard and brittle thus grinding and post-machining operations are very expensive and, sometimes, may be detrimental to the final properties of the product (Heck, 1974).

Cast Al-Ni-Co magnets have a greater density, hence yielding larger magnetic properties than powder metallurgy processed magnets (Campbell, 1994). Nevertheless, due to Al-Ni-Co brittleness and low shape complexity, casting is usually only used to produce large-dimension magnets. In cast Al-Ni-Co, high anisotropy is achieved by two means: annealing in a magnetic field; or close thermal control of the solidification texture. The combination of both techniques produces the best results, as shown in table 1, in the so-called columnar Al-Ni-Co magnets (Strnat, 1990).

Al-Ni-Co magnets produced by powder metallurgy can obtain better dimensional tolerances and more complex shapes; thus being preferred for small-dimension magnets. Sintered Al-Ni-Co magnets production starts with the preparation of the starting material by mixing raw materials either in pure form or in compounds, with the aim

of obtaining the desired composition. Afterwards, mixing is required to ensure homogeneity within the material. Pressing to the desired shape can be done using a range of techniques, although isostatic pressing, either cold or hot, is preferred (Dillon, 2014; Tang *et al.*, 2015).

Sintering of Al-Ni-Co magnets occurs under a hydrogen protective atmosphere at temperatures between 1200 and 1300 °C, and is commonly followed by quenching (EPMA, 2008; Gupta, 2015). Since the magnetic properties of as-cast and as-sintered Al-Ni-Co are poor, a special three-stage heat treatment is necessary with optimized magnetic properties, including (i) heating to 1820 °C for homogenisation, (ii) slow cooling at 1–5 °C·s⁻¹ with or without the presence of an external magnetic field, whether anisotropy or isotropy respectively are desired, to about 1070–1370 °C, and (iii) tempering-ageing at 1170 °C for several hours to develop the desired microstructure (Kittel *et al.*, 1950; De Vos, 1969; Iwama and Takeuchi, 1974).

5. CERAMIC MAGNETS

Powder metallurgy has been used in the production of ceramic permanent magnets, or ferrites, since their discovery by Philips Company (USA) in the 1950s (Went *et al.*, 1952; Hakker and Weber, 1958; Stuyts *et al.*, 1959). In general, ferrites are based in MFe₁₂O₁₉ where M can be barium, strontium or lead; although the first and the second are the most common.

Ferrites can be described as an oxide which contain iron oxide as the main component. These ferrites are hard magnets as *they* present ferrimagnetism whereas all metal-based magnets present ferromagnetism. They have a hexagonal crystal structure known as hexaferrite and characterised by

TABLE 1. Summary of the permanent magnets and their magnetic properties

Material	Coercivity (kA·m ⁻¹)	Remanence (T)	Maximum Energy Product (kJ·m ⁻³)	Reference
Al-Ni-Fe	32	0.60	10	Mishima (1931)
Al-Ni-Co casting	40	0.75	12.6	Smithells (1976)
Al-Ni-Co columnar	128	1.05	72	McCaig (1977)
Al-Ni-Co sintering	38	0.75	12.6	Smithells (1976)
BaFe ₁₂ O ₁₉ sintering	180	0.38	27	Ślusarek and Zakrzewski (2012)
SrFe ₁₂ O ₁₉ sintering	275	0.39	29.2	Ormerod (1988)
MFe ₁₂ O ₁₉ injection moulding	225	0.26	15.5	Ślusarek and Zakrzewski (2012)
SmCo ₅	493	1.04	209	Strnat <i>et al.</i> (1967)
Sm ₂ Co ₁₇	533	1.12	240	Strnat (1978)
Nd-Fe-B sintering	960	1.23	290	Sagawa <i>et al.</i> (1984b)
Nd-Fe-B melt spinning	1194	0.82	114	Croat <i>et al.</i> (1984)
Nd-Fe-B hot pressing	1500	0.8	125	Doser <i>et al.</i> (1991)

a high magnetocrystalline anisotropy which results in higher coercivities than Al-Ni-Co magnets.

They decided to choose powder metallurgy as the production technique due to the large magnetocrystalline anisotropy of the crystal structure. This decision was helped by the massive development of powder metallurgy technology as a consequence of the manufacture of tungsten filaments for bulbs as well as the development of cement carbides in 1909 and 1922 respectively (Ramakrishnan, 1983).

Hard ferrite magnets are manufactured from iron oxide (α -Fe₂O₃) and either barium carbonate or strontium carbonate. The raw materials are mixed in the correct proportions as this is critical in achieving high remanence and coercivity. If the carbonate is in excess then lower remanence would be achieved whereas if the carbonate quantity is insufficient then the coercivity would decrease (Verma *et al.*, 2000). The mixed powder is then pelletized to enhance calcination, which takes place at 1000–1300 °C (Heck, 1974) and facilitates solid state diffusion of the iron oxide and the carbonate.

When calcined, the pellets are ball milled to decrease and narrow the particle size distribution to 0.7–0.9 µm. The resultant powder, wet or dry, is pressed in the presence or absence of an external applied magnetic field in order to produce, respectively, anisotropic or isotropic magnets. For wet pressing, the powder is mixed with a binder as polyvinyl alcohol or camphor, which are then removed during sintering (Reed, 1995). Sintering is carried out at 1200–1400 °C in air and the resultant magnet is magnetized.

Even though ferrites are mainly manufactured by sintering, other powder metallurgy techniques, such as metal injection moulding, have been reported (Zlatkov *et al.*, 2009). These magnets exhibit lower magnetic properties in comparison with their sintered counterparts as shown in Table 1 (Slusarek and Zakrzewski, 2012).

6. RARE-EARTH MAGNETS

6.1. SmCo₅

In 1967 the first generation of rare earth magnets appeared. It is based on SmCo₅ after investigations by Strnat *et al.*, who produced RCo₅ (R=Y, Ce, Pr, Sm) by powder metallurgy in the form of resin bonded magnets (Strnat *et al.*, 1967). However, the magnetic properties of the permanent magnet were found to decrease with corrosion or with time at elevated temperatures due to the high reactivity of rare-earth elements. The stability problems of SmCo₅ were solved by producing full density magnets through liquid phase sintering techniques.

Powder metallurgy production of SmCo₅ starts with the induction melting of the alloy at 1350–1450 °C in vacuum, argon or helium. Additional samarium

is commonly added to cover the loss of Sm that occur as a consequence of vaporization during melting. Afterwards, the ingot is put through subsequent homogenization at 1150–1250 °C.

The as-cast alloy is crushed into small particles by hammer or jaw crushing. Alternatively to crushing, the alloy can be decreased in size by means of using the hydrogen decrepitation process. Further pulverization by jet milling in an inert atmosphere, usually nitrogen, is required to decrease the particle size of the alloy to 3–10 µm. SmCo₅ powder is then *uniaxially pressed in the presence* of a magnetic field parallel to the pressure direction. Magnetic alignment followed by isostatic pressing is also possible.

Sintering occurs at 1100–1200 °C for 0.5–1 hour under an atmosphere of argon, helium or vacuum. A typical heat treatment includes slow cooling at 1–2 °C per minute from the sintering temperature to 850–900 °C with a holding time of 1–4 hours in order to refine the grain boundary and hence increase the coercivity of the magnet. Afterwards, cooling to room temperature at 200 °C per minute (Campos *et al.*, 2004).

After the development of SmCo₅ permanent magnets, alloys with some quantity of copper emerged. These alloys became known as the precipitation hardened family of R(Cu,Co)₅. Less expensive alternatives to SmCo₅ have also been investigated, including partial substitution of Sm by Pr, or total substitution using Ce or La (Nesbitt and Wernick, 1973; Wallace *et al.*, 1984).

6.2. Sm₂Co₁₇

A second generation of rare-earth-based magnets emerged in the early 1970's based on the Sm₂Co₁₇ alloy (Tawara and Senno, 1972; Strnat, 1978). These magnets evolved by the addition of iron, to partially substitute cobalt, to become Sm₂(Co,Fe)₁₇, which opened a door to the addition of more alloying elements such as zirconium or niobium. The most widely used of the Sm₂Co₁₇ alloys are of the type Sm(Co,Cu,Fe,M)₇₋₈ where M = Zr, Ti or Hf (Liu *et al.*, 1998; Liu *et al.*, 1999a; Liu *et al.*, 1999b; Liu *et al.*, 1999c).

These *types* of magnets are well known pinning-controlled magnets *which* implies that the coercivity of Sm₂Co₁₇ magnets is determined by a domain-wall pinning process. Therefore, in order to form suitable pinning centres a sophisticated heat-treatment process has to be introduced. Besides this, the number of alloying elements used generate a multiphase microstructure which plays a vital role in the magnetic properties and that has to be carefully controlled (Strnat, 1978).

Powder metallurgy process of Sm₂Co₁₇ magnets is very similar to that of SmCo₅ magnets with differences in the sintering and heat treatment stage. Sintering occurs at the same temperature,

1100–1200 °C, but it is held longer, in the range 4–10 hours, followed by quenching. A typical $\text{Sm}_2\text{Co}_{17}$ heat treatment is more complicated than those for SmCo_5 magnets. It includes an isothermal ageing at 750–850 °C for 12–24 hours followed by slow cooling at 0.7 °C per minute until 400 °C, when the temperature is held for 1 hour and finally quenching (Gutfleisch *et al.*, 2006).

6.3. Nd-Fe-B

In 1983, a new material was discovered based upon a Nd-Fe-B ternary alloy, the third generation of rare earth permanent magnets. Uncertainties in world cobalt supply in the 1970s led to huge fluctuations in the price of the raw material, therefore increasing the need for a new permanent magnet material to replace Sm-Co.

This ternary alloy system was discovered simultaneously by General Motors Corp. in the United States and Sumitomo Special Metals Company Ltd in Japan (Croat *et al.*, 1984; Sagawa *et al.*, 1984a; Sagawa *et al.*, 1984b) using two totally different processing routes although both used powder metallurgy techniques. Magnets produced by Sumitomo were made by sintering aligned and compacted powder whereas magnets manufactured by General Motors were produced by mechanical compaction of ribbons from melt spinning. Magnetic properties of magnets produced from both routes are shown in Table 1.

Conventional powder metallurgy processing of Nd-Fe-B magnets includes conventional casting of ingots which are then hydrogen decrepitated into broken friable powder. Such powder is afterwards aligned and pressed, mostly at the same time, prior to sintering at 1000–1100 °C for 0.5–2 hours and then rapidly cooled (Liu and Kim, 1990; Zakotnik *et al.*, 2006; Zakotnik *et al.*, 2008; Zakotnik *et al.*, 2009; Degri, 2014). *The sintering process is aided by liquid phase from Nd-rich phase melt that enables good densification and, consequently, good magnetic properties.* A typical heat treatment involves annealing at temperatures between 480–650 °C for 1 hour under an atmosphere of argon, helium or vacuum, followed by quenching (Hsu *et al.*, 1987; McGuinness *et al.*, 1989; Yang *et al.*, 1993).

Although being the most common *process*, the above-mentioned production route is not the only one. A variation over the former process is *hammer crushing* the as-cast ingot instead of using hydrogen decrepitation. Another possibility would be using strip casting instead of conventional casting. Sintered magnets using strip casting were found to achieve better magnetic properties (+2.5% in remanence, +11.6% in coercivity and +6.4% in maximum energy product) than by using conventional casting. This was attributed to a small mean particle size of approximately 3 µm, which resulted in

a smaller grain size, and homogeneous microstructure (Pei *et al.*, 2002).

A totally different production route involves using strip casting followed by hot pressing at 650–770 °C under vacuum, and then the compacts are hot deformed to induce grain alignment by plastic flow (Lee, 1985; Gutfleisch *et al.*, 1998). As a consequence of the hot deformation process, internal cracks may appear; therefore, parameters processing must be carefully adjusted in order to avoid such situation. These magnets are anisotropic but not as much as sintered magnets, what results in lower magnetic properties when compared, as shown in Table 1. However, an increase in the degree of deformation may lead to a larger anisotropy and hence higher magnetic properties.

7. CONCLUSIONS

It has been shown the significance of using powder metallurgy techniques in the production of permanent *magnets*. Permanent magnet materials are required to offer great magnetic properties and these are dependent on the chemical composition, particle size of the powder, magnetic alignment, pressing technique, sintering conditions and heat treatment, amongst others. Many of these parameters can be controlled by powder metallurgy, thus improving the magnetic properties of the final magnet. Besides this, powder metallurgy is a near net shape technique that minimises or suppresses the need of machining, hence making of powder metallurgy the preferred route to manufacture a permanent magnet.

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