

Review Article

New Technology of Making NdFeB by Sintering

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ABSTRACT

Sintered NdFeB as a third-generation rare earth permanent magnet material, has been in rapid development since 1980's due to its broad industrial applications and high electromagnetic performance. Sintered NdFeB metal materials can easily be oxidized, so future efforts to improve their antioxidative stability are required and the study of new manufacturing technology and new technology is necessary. At present, the traditional technology of sintering NdFeB is discussed, together with the processing technology of powder preparation, hydroforming and vacuum sintering. The article focuses on the milling and forming process, including the traditional ball mill to hydrogen explosion, air grinding powder, and dry magnetic field molding to wet pressure magnetic field oriented molding, to prevent the production process of oxidation, thereby enhancing the electromagnetic properties and their anti-corrosion antioxidant capacity. During the NdFeB sintering process, content of oxygen is regulated to control the phase change. As a result, electromagnetic properties of sintered NdFeB obtained a qualitative leap.

KEYWORDS: NdFeB, Permanent magnetic material, Rare earth element, Sintering

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Introduction

Since the advent of rare earth permanent magnet materials in the late 1960s, rare earth permanent magnetic materials have been in rapid development from the first generation (samarium cobalt) to the third generation (NdFeB). NdFeB permanent magnet materials are significantly improved at the electromagnetic properties than the first and second generations but the metal elements within can be easily oxidized when encountered humid air. To obtain high electromagnetic NdFeB, we need to control not only the content of rare earth elements and compound addition, but also the entire synthesis process.

After 20 years' development, the traditional process of sintered NdFeB is gradually replaced by new technology. The traditional process of melting, ball milling, dry magnetic field orientation molding, vacuum sintering and heat treatment lacks control of oxygen content. Ball milling and dry magnetic field orientation molding are the most important manufacturing processes. The traditional ball milling required gasoline blending, which led to more impurities in the sintered NdFeB. Therefore, the NdFeB sintering process involving hydrogen explosion and air grinding powder cannot effectively prevent the introduction of impurities, but also hamper the separation of organic solvents when sintering. NdFeB produced by the dry pressure magnetic field orientation molding has low density, orientation degree, powder flow and the contact area. To obtain high-performance sintered NdFeB material, the traditional dry magnetic field orientation molding was replaced by the wet pressure magnetic field orientation molding, which strengthens the molding density and orientation, reduces the time of powder exposure to the air, and reduces the degree of powder oxidation.

The new NdFeB sintering technology succeeded the traditional ball milling and dry magnetic field orientation molding with wet magnetic field-oriented molding. Domestic metal permanent magnet material molding is currently using dry pressure magnetic field oriented molding, therefore the transition from dry pressure to wet pressure molding remains challenging in China.

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1. Basic principle of sintering NdFeB manufacturing

1.1. Sintered NdFeB traditional manufacturing process

The basic manufacturing process of NdFeB sintering include melting, milling, molding, sintering and aging treatment, in which the ball mill joins the organic additives to complete the milling. Domestic production of NdFeB still employs the traditional dry magnetic field orientation molding.

1.2. Composition of sintered NdFeB alloy

NdFeB is based on the $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound (a single cell crystal) and is rich in Nd and B. The unit cell consists of four $\text{Nd}_2\text{Fe}_{14}\text{B}$ molecules and 68 atoms: 8 Nd atoms, 56 Fe atoms and 4 B atoms. They form a tetragonal structure (tetragonal system). The addition of elements such as metal B plays a decisive role in the formation of tetragonal $\text{Nd}_2\text{Fe}_{14}\text{B}$.

The effect of Fe content on the magnetic properties of the alloy is shown in Figure 1. It can be seen that the Fe content is proportional to the magnetic properties of the alloy and the composition is as close as possible to the $\text{Nd}_2\text{Fe}_{14}\text{B}$ tetragonal phase to obtain a high energy alloy. For example, the magnetic properties of $\text{Nd}_{12.4}\text{Fe}_{81.6}\text{B}_6$ alloy can reach: $B_r = 1.48\text{T}$ (14.8 kGs), $\text{HCB} = 684.6\text{ kA/m}$ (8.6 kOe), and $(\text{BH})_m = 407.56\text{ kJ/m}^3$ (51.2 MGOe). The contents are 0.64 at% Nd and 0.02 at% B respectively higher than those of the tetragonal phase. The manufacture of such a high-energy product requires not only high-purity raw materials, but also the final composition as close as possible to $\text{Nd}_2\text{Fe}_{14}\text{B}$ tetragonal phase. Using a low oxygen process, the oxygen content is always below 1500 PPM, while the non-magnetic phase volume control is in 1.0% or less to obtain density and degree of orientation as high as possible.

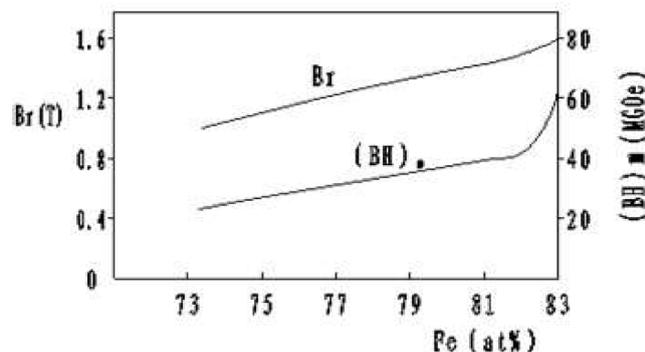


Figure 1. Effect of Fe content on the properties of sintered NdFeB permanent magnets

The formation of the main phase should be controlled before the α -Fe precipitation, which captures the main phase iron and reduces the main phase and the magnet performance. $\text{Nd}_2\text{Fe}_{14}\text{B}$ is the product of the peritectic reaction, which occurs when liquid begins to solidify. So the first solidification from the liquid phase is bound to α -Fe. To eliminate α -Fe, the most effective way is to accelerate the solidification, that is, the use of quick-drying process to eliminate α -Fe.

The composition of NdFeB permanent magnetic alloy is usually located in the triangle near the $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound, and is composed of $\text{Nd}_2\text{Fe}_{14}\text{B}$, a small amount of Nd-rich phase and boron-rich phase at room temperature. Sintered NdFeB permanent magnet alloy microstructure has (1) a boron-rich phase exists in isolated state or granular form, (2) a neodymium-rich phase along the grain boundary or grain boundary cross section of the step, (3) some boron-rich particles, (4) Nd_2O_3 , and (5) α -Fe phase. Foreign dopants devoid of neodymium can also be observed. In the sintered NdFeB permanent magnet material, the $\text{Nd}_2\text{Fe}_{14}\text{B}$ matrix (T1 phase) is the only ferromagnetic phase, whose volume fraction determines the B_r and $(\text{BH})_m$ of the alloy. Usually the number of boron-rich phase is between 0 and 0.8%, which dilutes NdFeB. The neodymium also plays an important role in hardening the sintered NdFeB alloy.

1.3. Vacuum induction melting

Vacuum induction melting furnace uses electromagnetic induction in the metal charge to generate eddy current, so that the furnace can be heated to a high enough temperature. A variety of metal raw materials will melt in the furnace and then fuse together in the molten state through the atomic diffusion to eventually become an alloy. As the vacuum induction melting furnace is easy to control, so that the purity, composition, quality and stability of the alloy can be guaranteed. As the basic means of alloying, this technology cannot be replaced by other technologies.

Due to the low-pressure surface of the molten pool and the electromagnetic stirring effect, some non-metallic inclusions may float on the surface of the molten pool to form an oxide film. If this film is mixed into the alloy, it will inevitably affect the product quality. This is the biggest problem of vacuum melting. The removal of inclusions in the alloy is mainly achieved by the decomposition of inclusions, the volatilization of low-valent oxides and the combination of carbon and oxygen (generation of CO). At the melting temperature, the partial pressure of O₂ and N₂ in the system is about 13.3 Pa, which is too high for the decomposition of the inclusions to occur. So the inclusions in the alloy can only be removed in other processes.

1.4. Ball mill powder

Alloy ingot should be removed of the surface oxide before coarse crushing or milling. To make fine alloy powders, ingots are subject to rough crushing and milling. There are two ways of crushing: hydrogen or mechanical crushing, to break the ingots into 175-246 μm (60 to 80 mesh) pieces. There are two ways of milling as well, ball milling and airflow grinding, to make alloy pieces into 3 to 4 μm fine powder. Ball milling uses steel (or other) balls, including rolling ball mill, vibration ball mill and high-energy ball mill and other methods. These balls are irregular in shape and are not conducive to the magnetic field orientation. High-energy ball milling produces powder particles whose sizes are too dispersed, so it will not improve the performance of the magnet. Rolling mill can better avoid these defects. Ball milling usually uses #120 aviation gasoline in a stainless steel can. There are four diameters of the cans, respectively, φ2mm, φ5mm, φ8mm and φ10mm, in consistent with the weight of each ball. When the ingot is broken for about 20 mm or so, the method of hydrogen breakage is used to improve the breakability and to make the ball milling easy to carry out. When the airflow grinding is further refined by the collision between the powder particles passing through the supersonic airflow, the particles are ground to 3 to 4 μm fine powder, and the majority of the magnetic powder was a single crystal. Air grinding exerts air to make a supersonic grinding. The current production of smaller manufacturers prefers rolling ball mill, whereas most of the NdFeB production plant uses air grinding.

1.5. Dry pressure magnetic field orientation molding

The magnetic properties of sintered NdFeB permanent magnets are mainly derived from Nd₂Fe₁₄B matrix with tetragonal structure, which is uniaxial anisotropic crystal: C-axis is the easy axis and A-axis is the hard axis. For a single crystal, the maximum remanence $B_r = \mu M_s$ when magnetized along its easy axis. If the c-axis of each powder particle of the sintered permanent magnet is oriented in chaos, an isotropic magnet is obtained: $B_r = \mu M_s/2 = J_s/2$, which is the lowest. C-axis being oriented in the same direction produces an anisotropic magnet, who has a maximum remanence. The powder particles of 3 to 5 μm obtained in the milling stage are generally single crystals, but not single domains, so the orientation is completed in two stages. In the first stage, each powder particle becomes a single domain and in the second stage, the magnetic moment is defined in the magnetic field rotation process.

The degree of orientation is affected by a number of factors: the composition, the particle size distribution of the powder, the orientation of the magnetic field within the mold, the forming pressure and the grain growth caused by sintering. The fact that the magnetic powder cannot be completely oriented is because the magnetic coagulation of the magnetic powder hinders the rotation of the magnetic powder. Adequate degree of orientation can be obtained with the appropriate variety and quantity of lubricant. However, if the magnetic agglomeration between the magnetic particles is too small, the magnetic powder cannot be molded without friction. Therefore, the magnetic agglomeration should be controlled within an honest range. The orientation magnetic field is a dynamic force, and the degree of orientation increases with the increasing orientation magnetic field. However, when the orientation field reaches 796 ka/m or more, the degree of orientation is difficult to increase any more, and therefore, it is not necessary to raise the orientation field too high. Molding pressure is the resistance of the magnetic powder orientation, forming a pressure below 4.9×10^7 Pa. The degree of orientation has a great change: the greater the pressure, the lower the degree of orientation. Therefore, in order to improve the degree of orientation, molding should be carried out at a minimum pressure in which a molded body can be obtained. There are two opposing tendencies in the sintering process, i.e., the degree of orientation caused by the growth of the grains and the degree of orientation resulting from sintering shrinkage. In the sponge-like green body molded at a low pressure (1.67×10^7 Pa), the sintering shrinkage is large and the degree of orientation is low. However, since the initial orientation of the green body is high, the final degree of orientation after sintering is still higher than that of the high pressure (19.6×10^7 Pa).

1.6. Sintering and heat treatment

To further improve (i) the performance and usability of magnets, (ii) the contact properties between the powder particles, and (iii) the strength of the magnet with high performance characteristics of the microstructure, it is necessary to heat the base of the powder below the melting point and insulation for some time in the NdFeB sintering process.

All the manufacturers and the vast majority of researchers extremely value the sintering process. NdFeB powder compact density is generally 50% to 70%, porosity is generally 30% to 50%, the combination of particles is all mechanical combination of very low strength. A too high molding pressure can lead to that (i) the particles in contact with each will have some elastic or plastic deformation, (ii) the sample is easier to crack, and (iii) its microstructure fails to produce high enough magnetic properties.

A series of physical and chemical changes will occur to the green body during the sintering. First, the adsorbed gas (including water vapor) on the surface of the powder particles excludes the evaporation and volatilization of the organic matter (such as the oil that may be stained in isostatic or the added antioxidants and lubricants), the elimination of the surface stress, and the reduction of the oxide. Second, the atomic diffusion, material migration, the contact between the particles by the mechanical contact to physical contact, the formation of metal bonds and covalent bond combination. Finally, the contact surface between the powder to expand, the sintering neck, followed by sintering neck growth, increased density, grain growth and so on. Powder green porosity and surface area are also large, so the surface energy is also large. But still it has a lattice distortion energy so that the overall green powder is in a high energy state. From the energy point of view, this is unstable, with spontaneous sintering and bonding into a dense body of the tendency and driving force. Thus, under conditions of a certain temperature, i.e., kinetic conditions permitting, the contact between the powder particles will be directed to the surface in order to reduce the surface area and surface energy. With the expansion of the interface between the particles, the green body began to shrink and density, and finally become a sintered body. In short, sintering is the process of turning the powder into a rough body.

Heat treatment refers to the alloy in the solid range of heating, insulation and cooling to change its organization and to obtain the required performance. Sintered NdFeB production is a two-stage tempering treatment. After tempering its coercivity, (BH) max, has been greatly improved. A high coercivity of microstructures can be obtained by two-stage tempering treatment. It is important to control the preparation process to obtain the desired microstructure. An important process for adjusting the microstructure of sintered NdFeB alloy is the tempering treatment after sintering.

The organization is closely related to the change in tempering temperature. When the primary tempering temperature is lower than 910 °C, the magnetic energy product (BH) max, the coercivity (H_{cj}) and remanence Br increases with increasing temperatures; when higher than 930 °C, the magnetic properties are significantly compromised; a tempering temperature between 910-930 °C makes the optimal magnetic properties:

$$Br = 1.125 \text{ T}, (BH) \text{ max} = 270.972 \text{ kJ/m}^3, H_{cj} = 1677.644 \text{ kA/m}$$

In the sintering process, the rich Nd phase at the grain boundary and the small amount of the main phase are molten into the liquid phase. After the sintering, the cooling rate is faster, the eutectic reaction is suppressed and the main phase cannot be completely precipitated, so the number of main phase decreases, cooling speed, and rich Nd-phase distribution is uneven, resulting in a large number of particles agglomeration. The reduction of the number of the main phase and the magnetic decoupling effect of the Nd-rich phase cannot be completely eliminated along the main grain boundary, so that the magnetic properties of the sintered magnet are poor. Using a two-stage tempering process, a tempering temperature of 900 °C, 2h heat insulation after the natural cooling to 600 °C; insulation for 1h and then cooling to room temperature can partially release this problem.

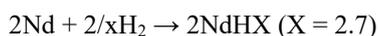
2. Sintering NdFeB manufacturing new technology

2.1. Principle of hydrogen explosion treatment

HD is essentially a physicochemical method that is distinct from a crushing material that is mechanically broken, and is particularly suitable for rare earth-containing alloys or intermetallic compounds because such materials have hydrogen absorption characteristics. For example, NdFeB hydrogenation after the formation of hydride, due to hydride generation lattice expansion, the enormous stress generated by NdFeB crystal to produce many micro-cracks, the material becomes loose and even become coarse powder, followed by heating dehydrogenation, large part of the main phase hydride back to the original Nd₂Fe₁₄B coarse powder, and a part of the Nd-rich hydride remains in the material. It is important to note that the coarse powder produced by HD is not only the coarse powder from the original ingot or quick cast slab (SC), which still retains some hydrides, whose chemical composition and magnetic properties are changed. This must be fully considered in the subsequent steps of NdFeB magnet production and dealt with accordingly. HD crushing method is only suitable for hydrogenation of metal or alloy that can be broken and in the feed size of 0.1-100 mm. The powder size is about 10-1000 μm, for hydrogen storage alloy or Ni. HM battery material is required for the powder, the particle size has to meet the practical requirements, but for the preparation of NdFeB magnets, the powder size should be 0.7-7 μm, that is, the average particle size is in the range of 3 - 4 μm. One must further fine grinding to meet the requirements.

2.2. Hydrogen explosion process

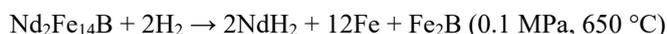
Under some conditions, hydrogenation of metal or alloy is reversible. The condition of the first formation of hydride is certain that hydrogen ions rather than hydrogen molecules can penetrate the intermetallic compound, and the dissociation of hydrogen molecules requires activation energy. In addition, the intermetallic compound (alloy) interface should be a clean surface, meaning that the oxide layer and other impurities that do not react with hydrogen will hinder the progress, for which the need for 'activation' from hydrogen until the hydrogen absorption reaction began, this time is called breeding period. Hydrogen absorption is an exothermic reaction, accompanied by heat (hydride generation heat) until the hydrogen absorption is saturated, that is, the hydride generation process. NdFeB hydrogen absorption can be divided into two steps. First, hydrogen absorption is exposed to the surface of the Nd-rich phase, reaction as follows:



Followed by the main phase $\text{Nd}_2\text{Fe}_{14}\text{B}$ react with H_2 :



When the hydride $2\text{NdH}_{2.7}$ transforms from the face of the cubic cube to hexagonal crystal, the character constant becomes larger and the volume expands for 20%. Hydride $\text{Nd}_2\text{Fe}_{14}\text{BH}_5$ lattice is also larger, with a corresponding volume expansion of 4.5-5.0%. The formation of the main phase hydride is accompanied by exothermic reaction, and the formation of heat is $\Delta H = -57.2 \text{ kJ/mol}$, the total heat makes the reaction temperature rise from room temperature to $\sim 300 \text{ }^\circ\text{C}$. The saturation magnetization of $\text{Nd}_2\text{Fe}_{14}\text{BH}_5$ is $M_s = 152\text{A} \cdot \text{m}^2/\text{kg}$ (300K), while Br and Hcj decrease sharply and become soft magnetic phase.



This hydride and Fe_2B completely return to $\text{Nd}_2\text{Fe}_{14}\text{B}$ main phase. Only in high temperature dehydrogenation can be achieved:



H_2 can be completely discharged only when the system is heated to $1040 \text{ }^\circ\text{C}$. When the temperature is raised to $650 \text{ }^\circ\text{C}$, the Nd-rich phase becomes soft and the inter-granular embrittlement may change. If the temperature continues to increase, the HDDR reaction will occur, apparently embarked on another direction of hydrogenation reaction.

Based on the above characteristics, the optimum dehydrogenation temperature now used is $500 \text{ }^\circ\text{C}$. Under these conditions, the hydrogen content of the main phase hydride is substantially released, and the neodymium phase hydride NdH_3 is partially hydrogenated to become NdH_2 . Dehydrogenation of this part of the hydride should be carried out during vacuum sintering.

HD milling can effectively reduce the oxidation of the grinding section and the powder oxygen content. Sintering process in the presence of hydrogen can restore the neodymium oxide, clean grain boundaries, and promote the densification, to achieve part of the activation sintering. HD crushing is preferentially carried out along the Nd-rich phase boundary or the Nd-rich ultra-structured layer in the main phase $\text{Nd}_2\text{Fe}_{14}\text{B}$. The HD + JM particles are monocrystalline grains whose size is close to the optimal particle size of $2\text{-}3 \mu\text{m}$. More importantly, for the edge of each grain in rich neodymium phase, coercivity can be increased by $40\text{-}400 \text{ kA/m}$ ($500\text{-}5000 \text{ Oe}$).

After the HD treatment is completed, the reaction tank will be placed vertically and all the alloy material in the tank will be injected into the aggregate tank through the funnel and the valve door (Figure 2). This will be safe and free from any leakage. It is clear that this apparatus is not only easy to achieve the requirements, but also more secure and effective.

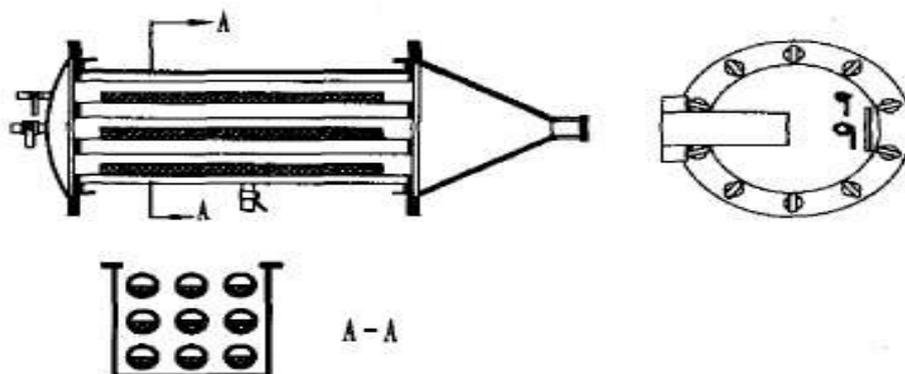


Figure 2. Hydrogen Bursting Furnace Reaction Tank Structure

2.3. Air grinding principle

NdFeB permanent magnet alloy is commonly made of air-grinding powder. The material in the feed mill uses the air impingement energy of several nozzles arranged in three dimensions and the collision of the airflow is in the fluidized bed. The friction is pulverized and driven by the rising air flow through the top turbine grading device. Fine powder is discharged outside the separation and recovery. Coarse powder falls back to the crushing area because of gravity to be continuously crushed.

It is assumed that two spherical particles of mass are accelerated by high-speed airflow and move in the opposite direction of the same straight line, such as the speed at which two balls collide with $V = 1.5V_c$. The two balls have the same energy. $E = 1$, D = the diameter of the original powder particles; r -surface energy density; V_c -sonic velocity, 16 °C in air, $V_c = 340$ m/s, which can be estimated from the mass of the original powder particles; d = the diameter of the original powder particles of the NdFeB particle collision before and after the energy changes.

The surface energy density of a metal is between 1.1 and 2.8 J/m², which depends on the hygienic properties of the atoms in the metal and is related to the melting point of the metal. The two powder particles collide down and the particles are crushed into smaller powders. When the two particles collide, the kinetic energy is only a small part of the surface energy, part of the consumption of particles is in the fragmentation, most of them are converted into sound energy, thermal energy and lattice distortion energy. The airflow mill is combined into a closed-circuit crushing system by a pulverizer and a classifier. The crushing system is a closed circuit due to the recycling of coarse powder. The theoretical analysis shows that it is necessary to reduce the eddy current radius and length to increase the number of blades and to reduce the blade thickness.

2.4. Air grinding process

In addition to the high saturation magnetization (M_s), the key to how to maintain the high coercivity (H_{c_j}) of the high-performance magnets is studied. It is found that the microstructure of the magnet should be controlled so that the sintered magnets of the grain is fine, and the size is uniform, which requires the magnetic powder is very fine, and the particle size distribution cannot be too scattered, must be highly concentrated.

Japan NEOMAX gold Yu governance concluded that: particle size should be 0.7 – 7 μm ; particles smaller than 3 μm should be discarded and then the sintered magnets should be in a uniform size (4.8 – 6.0 μm). The grain size distribution curve should be concentrated and sharp. The results show that the particle size distribution of the magnetic powder was wide (0.7 - 7 μm), but the particle size distribution of sintered magnets was very narrow. In addition to the effective elimination of α -Fe, the use of HD + JM milling makes it easier to obtain fine powder of 3 to 4 μm . Casting alloys and conventional cast alloys are pulverized with airflow. On the other hand, if the cast alloy is used, the particle size of the magnetic powder remains below 3 μm , although the feed rate is increased from 20 to 40 kg/h. If the ingot is used as raw material, the feed should be extremely slow, otherwise the grinding would be too coarse. Air grinding is more suitable for slab, grinding efficiency is extremely high, at high feed speed, still get fine powder.

The air crushing of NdFeB magnetic powder is performed using high-purity N₂ (more than 99.99%) under the pressure of 0.6 - 0.7 MPa, and is cooled by the cooler. The compressed air stream is accelerated into supersonic speed through the nozzle, causing the material to collide at the nozzle interchanges, thereby breaking the powder particles.

2.5. Wet pressure magnetic field oriented molding

The traditional molding method dies magnetic field molding with low orientation degree, different density and long-term exposure to metal power in the air, so that neodymium is prone to oxidation and destruction of the phase structure. Therefore, the traditional molding method will compromise the magnetic properties of sintered NdFeB. The wet magnetic field molding overcomes this problem by controlling the oxygen content and the useful phase structure, thereby improves the magnetic properties. The use of higher performance NdFeB sintered magnets in miniaturized applications is an important way to increase the (BH)_m of NdFeB sintered magnets to minimize the non-magnetic phase (rare earth oxides, boron-rich), to maximize the grain orientation and to optimize the alloying composition. Minimizing the nonmagnetic phase is to improve the magnetic properties and to reduce the oxygen content of the sintered magnets. For this purpose, several different powder metallurgy processes have been developed. It is difficult to use ordinary methods to prevent oxidation of the powder and its compacts which are strong in chemical activity and exposed to the air. The use of an organic solvent such as n-hexane wet process, or a dry process using an oxygen-free glove box, is likely to achieve a low oxygen content of 0.20% in the sintered magnets at the laboratory scale.

We produce a wet process - Hitachi low oxygen process, which uses mineral oil as a solvent so that the oxygen contents are kept below 0.2% during the production of NdFeB sintered magnets. A large-scale wet-forming process was prepared by modeling the Japanese company's low oxygen process. The developed wet process is based on the ordinary powder metallurgy process, which is characterized by grinding from the grinding to the grinding of the powder and the pressure of the blank is stored in the oil. The as-cast alloy is heated to 1100 °C for 6 h and then subjected to hydrogen explosion and ground to less than 500 μm pieces. In the ordinary process, in order to stabilize the surface of fine powder and to prevent rapid oxidation in the air, oxygen is depleted (<1000 ppm) by high-purity nitrogen during the milling.

However, in the wet process, the ground powder is immersed into mineral oil when it is ground so that the coarse powder is anaerobic (oxygen content below the detection limit, i.e., 0 ppm). The slurry is molded and the directional magnetic field is 14 kOe, perpendicular to the pressing direction. This mineral oil has very low evaporation rate when compared with n-hexane and other organic solvents, so the pressure of the green body can be operated in the air. Before sintering, the green body is placed in the temperature range of 100 - 300 °C, and the mineral oil present in the green body is removed by vacuuming for 1 hour. The sintered body is (i) heat treated at 1050 - 1080 °C, (ii) vacuumed at 10⁻⁵ Torr for 5h, (iii) a second heat treated at 900 °C × 2h, (iv) a third heat treated at 460 - 560 °C × 1h, and then the magnetic properties are measured on B-H trace instrument. The magnets are exposed to 120 °C, 100% relative humidity, 2 atmospheres in a high-pressure steamer, and the weight loss of the magnet after this test was measured to determine the corrosion rate.

The carbon content of the magnet produced by the wet process is only about 0.01% higher than the mass fraction of the conventional process, indicating that the oil remaining in the green body has been well removed in the release agent process. The wet process produces three times less material than the common method. The oxygen content of the coarse powder is 0.14%, and the oxygen content in the process of air grinding, pressing, dissolving and sintering is only increased by 0.02%. The oxygen absorption rate of the powder stored in the oil was 0.007% /day, which is much lower than the oxygen absorption rate of 0.016% /day of the powder stored in the organic solvent (n-hexane). The oxidation resistance of the oil and the low evaporation rate make the wet pressure process highly reliable and suitable for large production. As the final oxygen content of the wet process is low, the total rare earth content can be reduced accordingly.

With the mineral oil to prevent the oxidation and control pressure, this wet process has been established. As a result, a product having an oxygen content of below 0.2% and a (BH) max of more than 45 MGOe is obtained. This magnet also has a higher resistance to corrosion and higher mechanical strength, mainly because that the sintered magnets are in the smaller grain size and the total rare earth content is low. This process reduces the final oxygen content in the production, so it is used to produce high-performance sintered NdFeB. Further improvement in grain orientation will result in the production of higher magnetic magnets.

New technology is built on the traditional process. Hydrogen explosion and airflow milling not only produce large output, but also a high degree of automation. Completely using the pipeline operation reduces the power impurities and oxidation that are generated in the traditional process. The wet magnetic field oriented molding improves the overall green density, the degree of orientation of the powder, reduces the degree of oxidation of the green body in the air, and obtains the products of high electromagnetic performance and strong anti-oxidation ability.

3. Conclusion

- (1) Traditional ball milling process of NdFeB sintering has access to high oxygen content, impurities and more coarse powder. Hydrogen explosion and airflow grinding helps get almost pure powder with relatively low oxygen content.
- (2) The dry pressure magnetic field-oriented molding, generates not only green bodies of low density and poor orientation degree, but also severe surface oxidation because the entire molding process is exposed to the air. Oxygen content is not actively controlled. The development of wet pressure magnetic field solves the problem that the introduction, orientation and density of impurities are further improved.
- (3) New technology has enabled sintered NdFeB of high electromagnetic properties. Wet pressure field orientation molding, hydrogen explosion and air milling successfully interpret the perfect combination of low oxygen process.

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