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Metal Injection Molding (MIM) of NdFeB Magnets

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Abstract. Due to the increased and unstable prices for Rare Earth elements there are activities to develop alternative hard magnetic materials. Reducing the amount of material necessary to produce complex sintered NdFeB magnets can also help to reduce some of the supply problem. Metal Injection Molding (MIM) is able to produce near net shape parts and can reduce the amount of finishing to achieve final geometry. Although MIM of NdFeB has been patented and published fairly soon after the development of the NdFeB magnets there has never been an industrial production. This could be due to the fact that MIM was very young at that time and hardly developed. Thus, the feasibility of the process needs to be revaluated. This paper presents results of our work on determining the process parameters influencing the magnetic properties of the sintered magnets as well as the shrinkage during processing. The role of binder and powder loading on the alignment of the particles as well as on the carbon and oxygen contamination was examined.

1 Introduction

Patents covering the MIM of rare earth hard magnets as another powder metallurgical process were published soon after their development. Papers covering the topic are very scarce and do not explain why there is no industrial production, yet. In order to understand the problems involved and the possibilities to be had a feasibility study was started.

For the MIM of NdFeB the literature gives some hints for processing which are sometimes contradictory. While water extraction may be all right [1, 2] the binder should contain no or very few oxygen groups [3, 4]. The removal of the binder can be performed in hydrogen atmosphere, while the sintering has to be done in argon or in vacuum [1]. For debinding a slow heating rate is preferable to fast heating [5]. The orientation of the particles in the magnetic field needs a magnetic field of about 1 T and reaches around 92 %, similar to the shaping by pressing [6]. In the present study the influence of the binder and the MIM processing on the properties of the sintered magnets was investigated and compared to these hints.

2 Experimental

The induction melted alloy with a composition of (Nd,Dy)14.35Fe79.4B6.14 was pulverized by jet-milling under inert atmosphere obtaining a mean particle size of 4.6 µm. The powder was obtained from Magnetfabrik Schramberg. To prepare the feedstock, the powder was mixed at 120 °C in argon atmosphere with wax-polymer binders and at 170 °C with a POM based (Atect, Japan) binder using 60 vol% and 58 vol% solid content. The feedstocks were injection molded using either an Arburg machine without or a Thermo Scientific Minijet with an external magnetic field, the molding in a magnetic field leading to anisotropic magnets. The magnetic field in the mold was created by hard magnets of different quality having higher and lower remanence. The resulting magnetic field inside the 10 x 10 x10 mm3 cavity was calculated using ANSYS software. The injected parts were solvent debinded in hexane; afterwards thermal debinding was carried out in hydrogen up to 500 °C. Subsequently sintering was performed at different maximum temperatures up to 1100 °C for two hours under high vacuum conditions. After sintering, the samples were subjected to a post heat treatment for 1h at 520 °C. In order to obtain a reference, isotropic and anisotropic NdFeB magnets were produced via conventional route using cold isostatic pressing (CIP). Powder was pressed isostatically (1500 bar) without any binder addition and subjected to the same sintering and heat treatment as the MIM parts. The carbon and oxygen impurities were measured by hot gas extraction; the density of the samples using the Archimedes method. To perform the magnetic characterizations, samples were pulse magnetized in a 3.5 T field and demagnetization curves were traced with a Brockhaus hystograph.

In order to quantify the alignment factor, the average angle of misorientation, φ, is calculated by the following equation [7]:

$$\varphi = \tan^{-1}\left({}^{2J_{r\perp}}/_{J_r} \right). \quad (1)$$

Where Jr represents the magnet's remanence in the aligning direction (easy direction) and Jr⊥ represents the remanence perpendicular to the aligning direction (hard direction), both measured in the same magnet. The aligning factor (fφ) is then calculated:

$$f_\varphi = 100\% \cdot \cos\varphi \quad (2)$$

This method allows a quick evaluation of the efficiency of the alignment, if the sample geometry permits the measurement in both easy and hard directions. Commercial, uniaxially pressed Nd-Fe-B magnets have an fφ around 89-90%, while laboratory isostatically pressed magnets could reach fφ = 97-99%.

3 Results and discussion

3.1. Binders

The POM binder was used as Moon et al. postulated that binders which easily decompose into their monomers cause less carbon contamination than other binder constituents [4]. The mixing of the feedstocks was performed in argon atmosphere while the molding was performed in air hoping that the binder protects the powder from the air. In the case of the POM binder, molding proved to be impossible as the feedstock started to decompose with a fairly rapid deterioration at the molding temperature of 170 °C. It was assumed that oxygen containing binder components and comparatively high moulding temperatures should be avoided in the processing of NdFeB. It is not clear though if a reaction of the powder and the binder took place as this was not observed during mixing. The other possibility is that reaction with moisture and oxygen from the air caused a start of the reaction which increased the temperature to levels where also a reaction with the binder started. This could be prevented by also moulding in protective atmosphere which was not possible in the present study. Yamashita [1,2] found that the oxidation of the NdFeB powder much increases above 100 °C and proposed mixing and molding below that temperature.

The wax polymer binders allow processing at temperatures around 100 °C and did not show the incendiary properties of the feedstock based on POM binder. Here it was found that lower percentages in the binder of the backbone polymer which is decomposed thermally prior to sintering results in lower carbon contamination of the sintered magnet. Moon [3] proposed to use only wax as a binder but this was not done in the present study as this will cause problems during molding due to the low viscosity of the binder. For example, separation of powder and binder may happen which will cause warpage and inhomogeneous shrinkage

3.2 Debinding

Samples from a wax based feedstock were solvent extracted to remove all of the wax and only leave the PE based backbone. This was then thermally decomposed in the first stage of the debinding and sintering stage up to 500 °C in hydrogen at various heating rates. The sintering cycle was then concluded by removing the gas and sintering in high vacuum. Analysis of the sintered magnets is presented in figure 1.

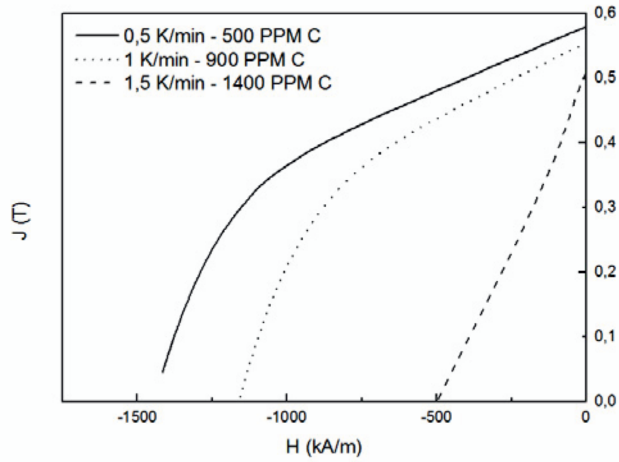

Fig 1. Deterioration of the magnetic properties of samples debinded with varying heating rates which caused the given contamination with carbon in the sintered magnet.

It seems that the reactive and complete removal of binder backbone is more important than the longer time at temperature in a carbon containing atmosphere.

3.3 Shrinkage

The shrinkage of the cubic test samples during sintering varies with the powder loading in the feedstock. A powder loading of 60 % will cause less overall shrinkage than a powder loading of 58 % if the final density of the sintered sample is the same. The shrinkage of isotropic magnets produced from a feedstock with 60 % powder and 40 % binder amounted to 15.8 %. In the case of the anisotropically molded samples the shrinkage was found to be 13.2 % in the hard direction and 24 % in the easy direction. As this inhomogeneity of shrinkage in the anisotropic magnets also depends on the percentage of alignment, several experiments were performed to elucidate the factors influencing the alignment. Obviously, it must be the aim to have a reproducible shrinkage if aiming at a given final geometry and tight tolerances.

3.4 Alignment

Molding of the feedstock with 60 % powder loading was performed in an external field of 0.6 T and of 1 T. The resulting magnetic properties are shown in figure 2, measured in easy direction (along the magnetic field) and in hard direction. The resulting alignment factor was calculated to be fφ = 90.5% for the higher external field. In low field, the remanence fell to 1.03 T and the fφ decreased to 85.6%. This clearly shows that for the given geometry and binder system with its inherent viscosity an alignment similar to what is reached in industrial production can be reached already using a field of 1 T. When designing a mold some reserve may well be advisable as the lower field reduces the alignment fairly quickly. Also, a slightly higher orientation of the particles may well be possible as some CIP samples produced from the same powder and aligned using 3,5 T resulted in even better magnetic properties which may also be attributed to a better alignment.

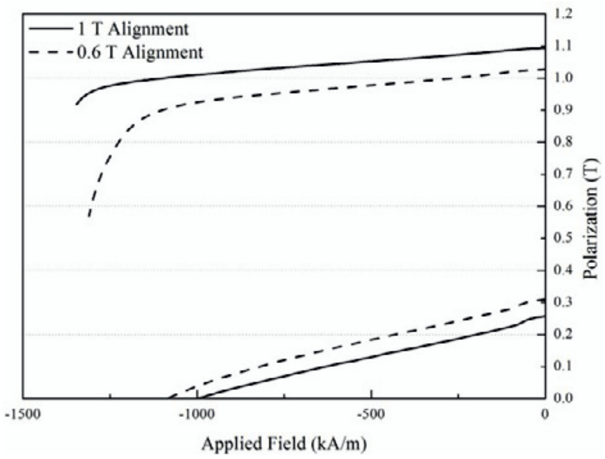

Fig. 2. Demagnetization curves in easy and hard directions for 60% powder loading aligned with high and low field.

Figure 3 shows the alignment along easy and hard direction for samples produced from feedstocks with 58 % and 60 % powder loading and an alignment field of 0.6 T. The f_φ for the 58 % powder loading was calculated with 91.8%, while the sample from 60% powder loading only reached 86.4%.

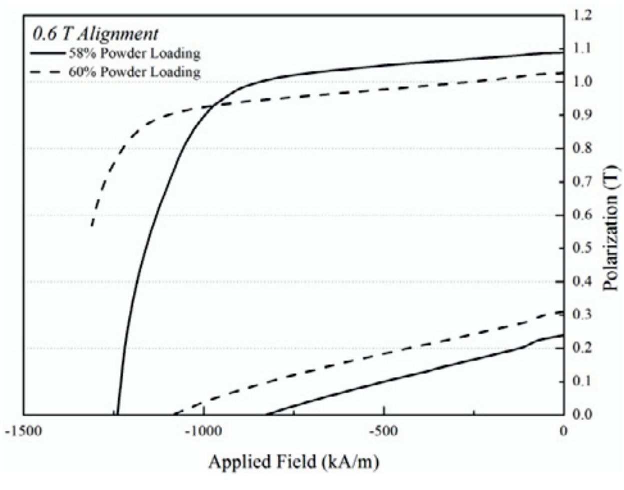

Fig. 3. Demagnetization curves in easy and hard directions for samples made from feedstocks with 58 and 60% powder loading in low field (0.6 T).

Again a stronger alignment field leads to slightly better values as can be seen in figure 4 showing the magnetic properties obtained for samples aligned in 1 T. The alignment factors were calculated with 94.4 % for the feedstock with 58 % loading as compared to the fφ = 90.5 % for 60 % solids loading.

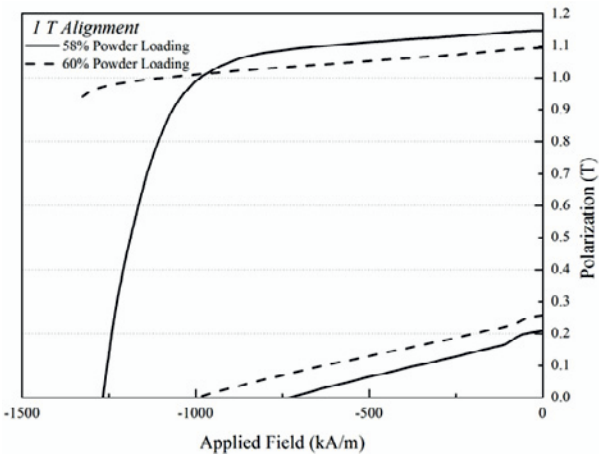

Fig. 4. Demagnetization curves in easy and hard directions for magnets made from 58 and 60% powder loading aligned in high field (1 T).

The idea in MIM is that all powder particles are coated with a thin layer of binder and all of the interspaces are also filled with the binder. This allows the flow similar to a highly filled polymer during molding. If the powder loading becomes to large the viscosity increases so that no molding is possible any more. It seems that for the turning of the irregular NdFeB particles some additional binder is needed. The slightly higher viscosity as well as the slightly stronger particle particle interaction of the feedstock with 60 % loading seems to hinder the alignment of the powder particles and makes a lower powder loading more advisable.

One possible disadvantage of the lower powder loading is that this may have caused the reduced coercivity of the sample as shown in figure 3. An increase in the carbon levels may have been caused by the higher amount of binder in this sample. This was confirmed by carbon quantification in sintered samples, indicating an average of 560 and 730 ppm of carbon with a standard deviation of 50 ppm for 60 % and 58 % binder samples, respectively. In order to decrease the carbon for the 58% binder samples a lower debinding rate is necessary as explained above.

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