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Injection-molded Sm–Fe–N anisotropic magnets using unsaturated polyester resin

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Abstract

New injection-molding technology has been developed using an unsaturated polyester (UP) resin in order to produce bonded Sm–Fe–N anisotropic magnets. Sheet magnets of 50 mm × 20 mm × 0.36 mm are successfully made within a cycle time of 90 s. Magnetic properties obtained are almost the same as those obtained in column magnets of ϕ 10 mm × 7 mm. Typical data of magnetic properties are as follows: $B_r = 0.72 \text{ T}$, $H_{CJ} = 796 \text{ kA/m}$ and (BH)_{max} = 94.7 kJ/m³. The density is 4.79 Mg/m³. The degree of orientation of (0 0 6) is 5.31 calculated using the Wilson formula from X-ray diffraction result. Ring magnets of 7.4 mm × 3.2 mm × 0.3 mm are easily made from sheet magnets. © 2005 Elsevier B.V. All rights reserved.

Keywords: Injection-molding; Anisotropic; Sm-Fe-N; Unsaturated polyester

1. Introduction

Mobile technologies of electronic devices have been progressing and thin magnets have been strongly required for small motors. In mobile devices, such as cell phones and PDA dwarf, downsizing the motor in the drive part is more demanded. Stamp size HDD and the extraordinarily small CPU cooling unit need high magnetic flux density with thin magnets.

There are several approaches using thin film technology. It was reported that Nd–Fe–B thick film with 200 μ m in thickness was obtained by a pulsed laser deposition method at a deposition rate of 0.3–1.2 μ m/min [1]. It was also reported that Sm–Fe–N thick film with 40 μ m in thickness was obtained by an aerosol deposition method at a deposition rate of 5 μ m/min [2]. Although the rate of deposition is improved compared with the conventional deposition method, it still needs long time to make thick magnets.

It is of course possible to make thin magnets by cutting and polishing sintered magnets or to make them by conventional bonded magnet technologies, such as an injection-molding using a polyamide resin or a compaction molding using an

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epoxy resin. However, when the thickness of the magnets required becomes thinner than $500 \,\mu\text{m}$, it becomes very difficult to make the magnets commercially.

In the case of conventional injection-molding, melted compound is injected into a mold, the temperature of which should be lower than that of a cylinder. Therefore, the viscosity increases quickly in the mold and in the case of making thin magnets, sometimes compound does not permeate through the mold. It becomes more difficult because the orientation of crystal axis is needed when making anisotropic thin magnets. The more the viscosity decreases, the easier the orientation of the crystal axis becomes.

In this paper, a new injection-molding technology that uses unsaturated polyester (UP) resin is reported. It is then compared with the conventional injection-molding technology that uses a polyamide (PA) resin.

2. Experimental procedures

A new injection-molding technology has been developed using the UP resin in order to produce bonded Sm–Fe–N anisotropic magnets. It was expected that heat damage to the magnet powder would be small because an injection-molding using the UP resin should be done at lower temperature than

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that using the PA resin. It was also expected that the orientation of the crystal axis by a magnetic field was easy compared with that using the PA resin because the viscosity of the compound decreases in the warmed mold.

These compounds contain approximately 58 vol.% of Sm-Fe-N magnet powder and approximately 42 vol.% of UP resin that includes additives and hardener. Sm-Fe-N magnet powder was made as follows: coarse Sm-Fe-N powder of about 30 µm is manufactured by the nitrogenation of Sm-Fe alloy powder made by a reduction and diffusion method. The anisotropic magnet powder was obtained by ball milling the powder into a size as small as 2 µm in Fisher sub-sieve size [3]. It is well known that additives in UP resin can control the rate of shrinkage. Two kinds of UP compound (standard and low shrinkage) were prepared. The curing of UP resin was performed via a free radical mechanism. Styrene was used as a monomer for linking the polyester chains and also adjusting the viscosity of compounds. Organic peroxide was used as the source of free radicals. Sm-Fe-N powder also acts as another source of free radicals. An inhibiter was added in order to control the "pot life" of the compound. The UP compound was kneaded using a dispersion kneader, the kettle of this dispersion kneader was water-cooled. Compound of 5 kg was kneaded at the rotational speed of the screw of approximately 50 rpm. The kneading time was half an hour.

An injection-molding machine used was for a thermosetting resin. The screw was designed to shear with as little heat as possible when kneading compounds because compounds easily harden by heating. The resulting clay-like compound was set into the screw using a piston-stuffing unit. The cylinder temperature was set at 30-50 °C and the mold temperature was set at 110-150 °C. The cycle time was 20-180 s. Several kinds of lubricant were used to release magnets from the mold.

The magnetic properties were measured using a Cioffi type recording flux meter. Samples were prepared by stacking magnet pieces in the case of thin magnets. Degree of orientation was calculated using the Wilson formula from the X-ray diffraction result.

3. Results and discussion

Fig. 1 shows the dependence of remanence B_r of column magnets on a magnetic field applied parallel to the axis at the injection-molding. The size of column is $\phi 10 \text{ mm} \times 7 \text{ mm}$. It is usually used as a standard magnet to evaluate magnetic properties and aging properties. The compounds used were two kinds of UP resin and conventional PA resin. One kind of UP resin was a standard type and another was a low shrinkage type. Since B_r at 500 kA/m is almost the same in all magnets, the degree of orientation of magnet powder might be the same.

Table 1 shows the magnetic properties of these magnets. The coercive force H_{CJ} of UP magnet was 840 kA/m, which was higher than that of PA magnet of 680 kA/m. The magnetic field related to squareness H_k for UP magnets was also



Fig. 1. Dependence of remanence B_r of column magnets on a magnetic field applied parallel to the axis at the injection-molding.

higher than that for PA magnet. Here, the H_k is a field at which the remanence B_r becomes 0.9 times. The reason for the high coercive force of UP magnet is attributed to the low fabrication temperature because exposing Sm–Fe–N fine powder at high temperature degrades the coercive force of the powder. In the case of PA magnet, compound was kneaded at 250 °C and injection-molding was done at 210 °C. The temperatures of initial irreversible flux loss of -5% were 151 and 115 °C for UP magnets and PA magnet, respectively. The reason for the higher temperature of UP magnet is also related to the higher value of coercive force.

Fig. 2 shows the dependence of remanence B_r of sheet magnets on a magnetic field applied parallel to the thickness at the injection-molding. The size of sheet was 40 mm × 10 mm × 1.5 mm. It was used in order to evaluate the moldability of compounds. In the case of UP magnets, remanence B_r at 500 kA/m was almost the same as that shown in Fig. 1. On the other hand, in the case of PA magnet, it was lower than that shown in Fig. 1.

Table 2 shows magnetic properties of these magnets. While remanence B_r decreased 5% compared with the column magnet in the case of UP magnet, it decreased 12% for the PA magnet.

Table 1

Magnetic properties of standard and low shrinkage UP magnets and of PA magnet, the size of which is $\phi 10 \text{ mm} \times 7 \text{ mm}$ (magnetic field applied was approximately 950 kA/m)

	UP (S)	UP (L)	PA
$\overline{B_{\rm r}({\rm T})}$	0.74	0.73	0.73
$H_{\rm CJ}$ (kA/m)	840	840	680
$H_{\rm k}$ (kA/m)	472	480	384
(BH) _{max} (kJ/m ³)	102	98.4	96
Density (Mg/m ³)	4.79	4.67	4.71
Temperature* (°C)	151	151	115

Permeance is -2 evaluated from the magnet size, Temp.* means the temperature of initial irreversible flux loss of -5%, UP (S) and UP (L) mean standard and low shrinkage types, respectively.



Fig. 2. Dependence of remanence B_r of sheet magnets on a magnetic field applied parallel to the thickness at the injection-molding.

Table 2

Magnetic properties of UP and PA magnets, the size of which is $40\,mm\times10\,mm\times1.5\,mm$ (magnetic field applied was approximately $1540\,kA/m)$

	UP	PA
$\overline{B_{\rm r}({\rm T})}$	0.70	0.64
$H_{\rm CJ}$ (kA/m)	844	692
$H_{\rm k}$ (kA/m)	478	350
(BH) _{max} (kJ/m ³)	90.3	72.2

Fig. 3 shows an example of sheet magnet made using PA resin. The size of sheet was $50 \text{ mm} \times 20 \text{ mm} \times 0.5 \text{ mm}$. Because the temperature of the injected compounds decreased and the viscosity quickly increased, the compound did not permeate through the mold. It might be very difficult to manufacture thin sheet magnets, the thickness of which becomes less than 0.5 mm.

Fig. 4 shows an example of sheet magnet using UP resin. The mold used was the same as shown in Fig. 3. Because the temperature of the injected compound increased, the viscos-



Fig. 3. Example of sheet magnet made using PA resin.



Fig. 4. Example of sheet magnet made using UP resin.

Table 3

Magnetic properties of UP magnet, the size of which is $50 \text{ mm} \times 20 \text{ mm} \times 0.5 \text{ mm}$ (magnetic field applied was approximately 1540 kA/m)

	UP	
$\overline{B_{\rm r}}$ (T)	0.70	
$H_{\rm CJ}$ (kA/m)	836	
$H_{\rm k}$ (kA/m)	485	
(BH) _{max} (kJ/m ³)	89.5	

ity of it quickly decreased and compound permeated through the mold.

Table 3 shows magnetic properties of the UP sheet magnet. The properties obtained were almost the same as that obtained in the sheet magnet of 1.5 mm in thickness.

Table 4 shows magnetic properties of the UP sheet magnet, the size of which is $50 \text{ mm} \times 20 \text{ mm} \times 0.36 \text{ mm}$. The size of the mold was $50 \text{ mm} \times 20 \text{ mm} \times 0.3 \text{ mm}$ and the cycle time was 90 s. The coercive force is a little bit smaller than that shown in Table 3 because of the different lot, but it was confirmed that the magnetic properties of three areas (gate, center and anti-gate) were almost the same. The degree of orientation of (006) was calculated using the Wilson formula (1) from X-ray diffraction result. Generally speaking, it is thought that degree of orientation is very high when the value is larger than 2.

$$K_{S1} = \frac{\frac{I_{S1}}{I_{S1} + I_{S2} + I_{S3} + \dots}}{\frac{I_{S1}}{I_{D1} + I_{D2} + I_{D3} + \dots}}$$
(1)

Table 4

Magnetic properties of UP magnet, the size of which is $50\,mm\times20\,mm\times0.36\,mm$ (magnetic field applied was approximately $1540\,kA/m)$

	Gate	Center	Anti-gate
$\overline{B_{\rm r}({\rm T})}$	0.72	0.71	0.71
$H_{\rm CJ}$ (kA/m)	796	788	788
$H_{\rm k}$ (kA/m)	438	430	430
$(BH)_{max} (kJ/m^3)$	94.7	92.3	92.3
Degree of orientation of $(006)^*$	5.31	5.25	5.27

^{*} Degree of orientation of (006) is calculated using Wilson formula from X-ray diffraction result.



Fig. 5. Ring magnet punched from sheet magnet.

where K_{S1} is the degree of orientation of index S1 of a sample, I_{S1} , I_{S2} , I_{S3} ... the intensity of each index of a sample and I_{D1} , I_{D2} , I_{D3} ... are the intensity of each index of a standard sample.

Fig. 5 shows a ring magnet punched from sheet magnet. The size of it is ϕ 7.4 mm × ϕ 3.2 mm × 0.3 mm. It was easily made from sheet magnets.

4. Conclusions

New injection-molding technology has been developed using unsaturated polyester resin in order to produce bonded Sm–Fe–N anisotropic magnets commercially.

Sheet magnets of $50 \text{ mm} \times 20 \text{ mm} \times 0.36 \text{ mm}$ were successfully made within a cycle time of 90 s. Magnetic properties obtained were almost the same as those obtained in column magnets of $\phi 10 \text{ mm} \times 7 \text{ mm}$.

Typical magnetic properties of the sheet magnets at the density of 4.79 Mg/m^3 were as follows: $B_r = 0.72 \text{ T}$, $H_{CJ} = 796 \text{ kA/m}$ and $(BH)_{max} = 94.7 \text{ kJ/m}^3$. The degree of orientation of (006) was 5.31 calculated using the Wilson formula from X-ray diffraction result.

Ring magnets of 7.4 mm \times 3.2 mm \times 0.3 mm were easily made from sheet magnets.

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