Nd$_2$Fe$_{14}$B Permanent Polymeric Composite Magnets

Mehrdad Kokabi[*], Fatemeh Arabgol[1], and Mehrdad Manteghian[2]

(1) Polymer Engineering Group and (2) Chemical Engineering Group
Tarbiat Modarres University, P.O. Box: 14115-143, Tehran, I.R. Iran

A B S T R A C T

Manufacture of polymeric composite magnets using isotropic NdFeB powder and different polymeric matrices; polyethylene glycol (PEG) as thermoplastic, epoxy as thermoset and styrene butadiene rubber (SBR) as elastomeric matrices have been studied in this work. Magnet powder was crushed mechanically and sorted in the particle sizes of 74-100, 2-10, and ≤1 microns. PEG Nano-powder was also prepared via solvent/non-solvent method. The effects of particle size of the magnetic powder, polymer content, type of polymeric matrices and processing conditions on the magnetic properties of the product, i.e. residual magnetic induction (B$_r$), coercive field strength (H$_c$), maximum energy product (BH)$_{max}$ and density were investigated. NdFeB-PEG permanent composite magnet made of magnetic powders with a particle size of 74-100 μm and 5 php (the ratio of polymer loading per 100 g magnetic powder) of PEG nano-powder was found to have the best magnetic properties. The measured properties of this product were as follows: final density = 5.444 g/mL; B$_r$= 3.82 kG, H$_c$ = 8.415 and kOe; (BH)$_{max}$= 14.454 MGOe.

(∗)To whom correspondence should be addressed.
E-mail: Mehrir@Modares.ac.ir

INTRODUCTION

It has been more than half a century since bonded magnets were first developed by Baermann in 1934[1]. He made his polymer-bonded magnet by mixing isotropic Alnico powders and phenolic resins. At present, polymer-bonded magnets with dif-ferent properties are used in various applications. Type of polymers, polymer/magnetic powder proportion, particle size and shape of both magnetic powder and polymer and processing conditions are the most important parameters ruling the

properties of the magnetic composite product [2].

There has been a great interest in using Neodymium-Iron-Boron (Nd-Fe-B) material for fabrication of permanent polymeric composite magnets. Low density, besides favourable properties such as high corrosion resistance, ease of processing and simplicity of mass production can be counted as some advantages.

Croat, Sagawa and Herbst first reported high-energy and new magnets based on triple alloy of rare earth Nd-Fe-B in late 1983 [3,4]. Polymer-bonded NdFeB permanent magnets are mainly composed of NdFeB permanent magnetic powder and polymeric binders such as thermoplastic polyolefin; polyphenylene sulphide (PPS), polyvinyl chloride (PVC), polypropylene (PP), polyethylene (PE), high density polyethylene (HDPE), polyamide or thermosetting epoxy resin [5,6], nitrile rubber or other elastomers. Conventional polymer processing methods may be used in production of bonded permanent magnets.

The disadvantages to mention about NdFeB alloy are weak thermal properties, and poor corrosion resistance in a long term [7,8].

The most important advantages of polymer bonded magnets are as follows; low weight, corrosion resistance, easy machining ability, forming and handling, high production rate, insensitiveness to crack or break. Bonded magnets provide an almost infinite variety of combinations of mechanical, physical, chemical, thermal and magnetic properties due to various kinds of polymeric matrices [9,10].

These materials are of great importance for the electronic, communication and microelectro-mechanical (MEMS) applications [11,12].

Low energy density and poor hygrothermal resistance properties, however, limit their applications. Their poor heat resistance is due to the intrinsic poor thermal and hygroscopical resistance of magnetic rare-earth alloy particles [13,14]. However, choosing an adaptable and suitable polymer can solve the problem. Using polyphenylene sulphide (PPS), liquid crystal polymer (LCP) or heat-resistant epoxy resins raise the maximum applicable temperature of bonded magnets up to 180ºC [15,16]. For permanent bonded magnets, the particle size of magnetic powder plays an important role in determination of powder to binder ratio, degree of particle alignment and magnetic and mechanical properties [17,18].

In this work three different polymeric matrices; thermoplastic i.e., PEG, a thermoset i.e., epoxy resin and an elastomer i.e., SBR and an isotropic magnetic powder NdFeB with three different particle size ranges of ≤1, 2-10 and 74-100 µm have been used and the effects of magnetic powder particle size, loading level and the type of polymeric matrix and the processing conditions on magnetic properties and density have been studied. The main question in this research is that how a reduction in the magnetic powder particle size to a lower scale can lead to production of permanent polymeric composite magnets with superior properties.

EXPERIMENTAL

Materials
The magnetic powder, Nd (FeCo) B, also brand named as NQP-B used in this study was supplied by Xiamen Yuxiang Magnetic Materials Ind. Co. Ltd, China with a particle size from 74 to 177 m. Table 1 shows some characteristics of the powder. The polymers used were: (1) a PEG supplied by Merck, Germany, with a molecular weight of 35000 melting at 60-65ºC and a T$_g$ of -50ºC; (2) an epoxy resin, Epicoat Shell 246/L with a density of 1.1 g/mL, curing temperature of 150ºC and curing time of 20-30 min and its hardener EPI232, both supplied by Saman Co., I.R. Iran; and (3) SBR compound complying with ASTM 3185 with a density of 0.94 g/mL, curing temperature of 150ºC and curing time of 7 min.

Crushing the Nd$_2$Fe$_{14}$B Magnetic Powder
The magnetic powder was crushed mechanically by a planetary ball mill twice, each time for an hour, to reach a mean particle size of 50 µm. It was then transferred to an electrical steel mortar for an hour to achieve a mean particle size of 44 m and at last to a ceramic ball mill for further crushing in environmental conditions.

For this purpose an alumina jar with a volume of around 0.5 US gal i.e., 1.8927 L, and cylindrical alumina balls with dimensions of 12.5 cm × 12.5 cm (ML× D) were used. Total weight of balls was 1.9 kg (45-55% of the total volume of jar), and 1 kg of magnetic powder in dried conditions (equal to 25% of the total volume of jar) was fed. Finally a fine powder with a mean particle size of 25 µm was obtained which had a broad particle size distribution (PSD) from less than 1 µm to near 100 m.

Table 1. Physical, chemical and magnetic characteristics of NQP-B magnetic powder.

No.	Characteristics	Unit	Value
1	Apparent density	(g/mL)	2.4
2	Theoretical density	(g/mL)	7.4
3	Fluidity	(g/s)	2.1
	Particle mesh size distribution		
	80	(wt%)	4.72
4	100	(wt%)	4.72
	160	(wt%)	51.6
	200	(wt%)	21.66
	200		7.3
	Elemental chemical analysis		
	Nd	(wt%)	25
5	Fe	(wt%)	66
	B	(wt%)	1.4
	Co	(wt%)	7
6	Residual magnetic induction, B$_r$	kG	8.04
7	Coercive field strength, H$_{cb}$	kOe	5.87
8	Maximum energy product (BH)$_{max}$	(MGOe)	12.2
9	Maximum magnetic field, H$_m$	kOe	14.61
10	Temperature coefficient of B$_r$	(%per°C)	-0.1

Sorting the Magnetic Powder

Magnetic powder with a particle size of 74-100 μm was separated by dry sieving (retained between sieves with 140 and 200 mesh), and powder with a particle size of 2-10 μm was separated by wet sieving (mesh 800) in non-aqueous media i.e., 96% ethanol.

It should be noted that the passage of the powder through the aforementioned sieve in dried condition was impossible. Also aqueous media caused severe powder oxidation (the colour of the powder turning to yellow and then to red-brown). A powder with particle size less than 1 μm was separated by sedimentation based on Stokes law. The maximum allowable sedi-

mentation time was less than 60 s.

Preparing PEG Nano-powder

Solvent/non-solvent method was used to prepare PEG nano-powder. In this method, PEG is dissolved in a solvent such as CCl$_4$ to the saturation state, then added to a non-solvent such as (lead-free) benzene, while stirred at a desired rate. PEG Nano-particles are then precipitated, separated from non-solvent and then dried.

Higher mixing levels are applied when ingredients of finer particle size are desired. Nano-powders of the polymer and the magnetic admixture are, therefore, preferred in this respect.

Mixing of Magnetic Powder with the Polymer

Choosing the suitable mixing conditions and the procedure was strongly affected by the nature of polymer matrices. PEG Nano-powder was manually mixed with the magnetic powder for at least 15 min in order to achieve a good (homogeneous) mixture. Liquefied epoxy resin was also properly mixed for 15 min with the magnetic powder before adding its corresponding hardener to avoid higher viscous media causing mixing problems. A two-roll mill as an intensive high shear mixer was used for mixing SBR compound (Table 2) with the magnetic powder.

The mixtures were then moulded in a cylindrical die with a diameter of 13 mm by hot press under a pressure of 100 bars at 65°C for magnets with PEG matrix, 150°C for a duration of 20 min for magnets with epoxy resin matrix, and 150°C for a duration of 7 min for magnets with SBR matrix. The characteristics of samples are detailed in Table 3.

Determination of the Samples' Properties

Density

Density of samples was calculated by dividing their mass to their volume. A balance was used to measure the mass of materials with an accuracy of 0.01g and the dimensions were determined by micrometer with an accuracy of 0.01mm.

Table 2. Components of SBR-based compound.

Material	SBR	ZnO	Stearic acid	Sulphur (s)	MBT[1]	TMTD[2]	Paraffin oil
Function in the compound	Rubber base	Activator	Activator	Curing agent	Accelerator	Accelerator	Plasticizer
Quantity parts by mass	100	3.00	1.00	1.75	0.5	0.5	35.00

Table 3. The characteristics of bonded magnets and magnetic characteristics of the samples.

No.	Sample code	Magnetic particle size (μm)	Polymer content (%wt)	ρ (g/mL)	B$_r$ (kG)	H$_c$ (kOe)	(BH)$_{max}$ (MGOe)	(BH)$_{max}$/ρ (MGOe/g.mL^3)	(BH)$_{max}$/m (MGOe/g)
1	PEG 100-5	74-100	5	5.444	3.82	8.415	14.454	2.655	3.896
2	PEG 100-50	74-100	50	2.487	1.51	8.415	5.770	2.320	3.000
3	PEG 100-80	74-100	80	2.111	1.04	8.415	4.000	1.890	2.28
4	PEG 10-5	2-10	5	5.126	3.70	7.905	11.644	2.27	2.74
5	PEG 10-80	2-10	80	2.233	0.91	7.650	2.375	1.040	1.74
6	PEG 1-5	≤ 1	5	2.127	3.33	7.905	10.555	4.960	7.00
7	PEG 1-80	≤ 1	80	1.750	0.84	7.650	2.503	1.430	2.00
8	FPO 100-5	74-100	5	4.546	3.74	8.343	14.243	3.133	3.13
9	EPO 100-80	74-100	80	3.573	2.52	8.343	9.759	2.730	3.75
10	EPO 10-5	2-10	5	4.518	0.85	7.905	2.804	0.620	0.60
11	EPO 100-80	2-10	80	3.514	0.65	6.757	1.501	0.385	0.54
12	SBR 100-50	74-100	50	3.812	1.85	8.287	6.450	1.690	2.35
13	SBR 100-80	74-100	80	1.806	0.88	8.287	3.224	1.790	1.90
14	SBR 10-50	2-10	50	2.775	1.68	7.395	4.322	1.560	1.63
15	SBR 10-80	2-10	80	1.830	0.77	7.395	1.872	1.040	1.10

Magnetic Properties

Typical remanence (B$_r$), intrinsic coercivity (H$_c$), and maximum energy product (BH)$_{max}$ of the samples were obtained from magnetic hysteresis loop derived by B-H loop tracer in the Physics Department of Isfahan University. Typical magnetic hysteresis loop and magnetic characteristics of sample PEG-100-5 (Table 3) are shown in Figure 1. The samples were held in a magnetic field of 1.3 T which was persuaded by direct current (DC). The B-H hysteresis loop was plotted by magnetizer.

Calibration of H and B axes was required to interpret these quantities. This was carried out by a 1 T standard magnet and a Gauss meter for H and B, respectively. The calculations for sample PEG100-5 are presented in Figure 1 as well.

RESULTS AND DISCUSSION

Effect of Powder Shape, Particle Size and Loading Level on the Density

TEM Micrograph of PEG fine powder, prepared by sol-

vent non-solvent method, is shown in Figure 2. The magnification is 30000. Sphericity of the particles is shown in the micrograph. Scanning electron microscopy (SEM) photographs of magnetic powders which were crushed and separated in various particle size ranges are shown in Figures 3-5. Figure 3 indicates that Nd-Fe-B magnetic powder with a particle size
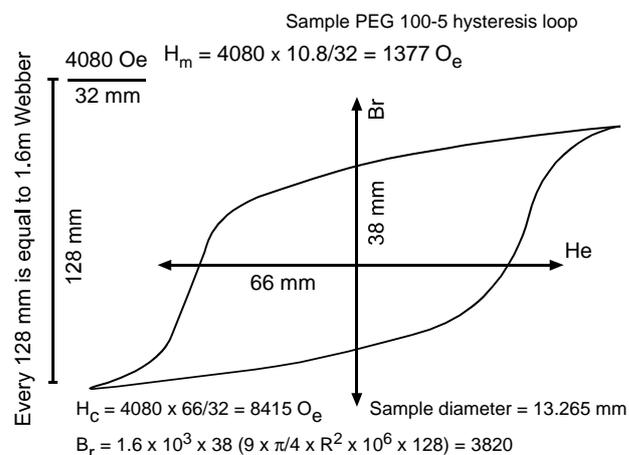
Figure 1. Magnetic hysteresis loop and magnetic characteristics of a typical magnet.
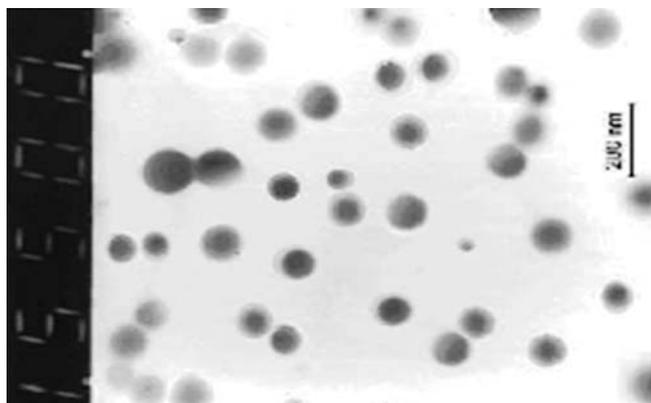
Figure 2. TEM of PEG powder made by solvent/non-solvent method.

Figure 4. SEM of NdFeB powder, with particle size range of 2-10 µm.

range of 74-100 µm has plate-like shapes. Figure 4 shows that powder particles with size range of 2-10 µm have irregular shapes and Figure 5 indicates that particles with a size of ≤1 µm are nearly spherical. It is well known that particle shape and size affect packing density of the powder, so densities of the samples can be affected by these two factors. Plate-like particles would result in more packing density under the same compression force.
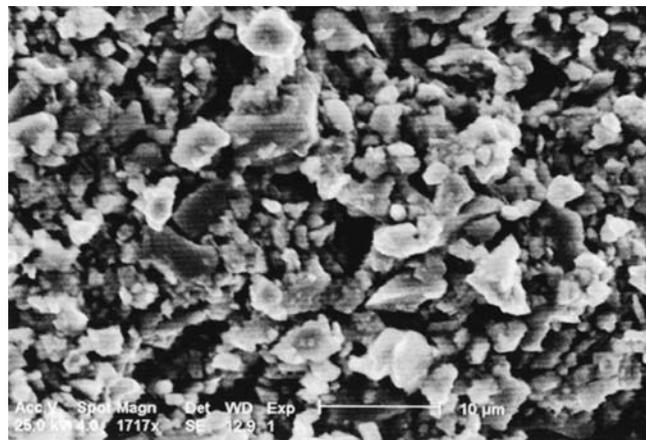
In this case, it was found out that the density decreases with a decrease in the powder particle size. Having in mind that the shape of particles changes from plate-like to irregular and semi-spherical as the particle size decreases (Table 4).

It can be concluded that the shapes of particles have a significant effect on packing density. Further points concluded from Table 4 are as follows:

- The densities of the samples decrease due to particle size reduction of the magnetic powder (for the same loading level) in each polymeric matrix, (for instance, compare the results for samples: A_1, B_1 and C_1 or D_1 and E_1 or F_1 and G_1) in Table 4.

- The coarser particles with plate-like shape show more tendency to compaction than the finer particles with irregular and semi-spherical shapes.

In order to evaluate qualitatively the changes in the density of samples containing magnetic powder of the same size in various polymeric matrices, index A is introduced which is defined as density differences divided by polymer loading level differences (Table 4). The value of index A changes as the matrix and particle size/shape varies.

When coarser magnetic powder with plate-like shape is used, the variation in the index is attributed to the matrix variation in the order of $A_{SBR} > A_{PEG} > A_{Epoxy}$.

This can be explained by different sample prepara-

Figure 3. SEM of NdFeB powder with particle size range of 74-100 µm.
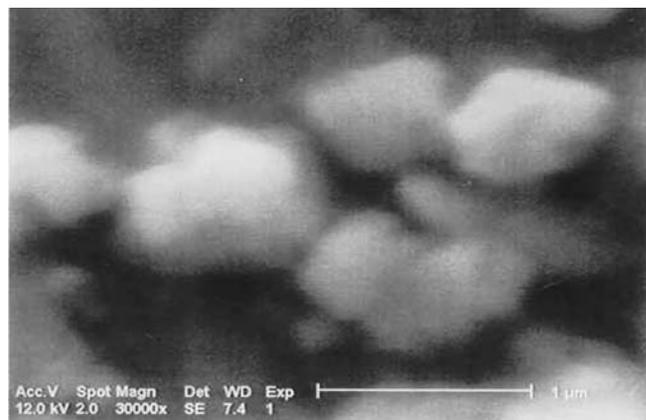
Figure 5. SEM of NdFeB powder with particle size ≤1 µm.

Table 4. The ratio of density differences per polymer loading differences (Index A).

No	Sample code	ρ (g/mL)	Density differences/polymer loading level differences (%) (Index A)
1	PEG 100-5	5.444	4.444
2	PEG 100-80	2.111	
3	PEG 10-5	5.126	3.8573
4	PEG 10-80	2.233	
5	PEG 1-5	2.127	0.5026
6	PEG 1-80	1.75	
7	EPO 100-5	4.546	1.3
8	EPO 100-80	3.573	
9	EPO 10-5	4.518	1.3
10	EPO 10-80	3.514	
11	SBR 100-50	3.812	6.686
12	SBR 100-80	1.806	
13	SBR 10-50	2.775	3.24
14	SBR 10-80	1.803	

tion conditions employed. An intensive high shear mixer, i.e. two-roll mill was used to make SBR compounds while mixing of other matrices was carried out by manual stirring.

The magnet made by SBR was, therefore, much more compact than those made by PEG or epoxy, solely due to the effect of processing conditions on plate-like particles used in these cases.

As the size of the magnetic powder is reduced, its irregular and semi-spherical shape is the main barrier to obtain good packing, i.e. the density is not much affected by application of intensive and high shear mixing.

When the finer particles of irregular (semi-spherical) shape have been used, the order of the changes in index (A) in the case of different matrices has been as follows; $A_{PEG} > A_{SBR} > A_{Epoxy}$.

The method of processing seems to have no effective role on compaction of the magnets. This may be due to the resistance of these irregular (semi-spherical) particles toward compacting. The order of index changes in this case could be due to the variations in the chemical structures of the polymeric matrices.

Compared to cross-linked SBR and cured epoxy resin, PEG has a simple chemical structure with a lower T_g. It, therefore, provides more free volume resulting to higher polymer chain flexibility.

Effect of Particle Size, Loading Level of the Magnetic Powder and the Type of Polymer on B$_r$, H$_c$ and (BH)$_{max}$

The remanence of a magnet is proportional to its magnetic powder loading level and can be improved by increasing the density of the magnet and/or decreasing the proportion of the binder loading, as seen in Figure 6.

B_r is not only affected by the magnetic powder, it also depends on the type of the polymer used. Highest values of B_r are obtained when PEG is used as the binder. This could probably be attributed to higher level of polarization in this polymer.

Figures 7-9 indicate that the permanent magnetic properties of the composite are deteriorated with a decrease in the size of the NdFeB particles. This could be due to the higher level of oxidation of these particles, owing to more intensified crushing applied to them and their higher surface area.
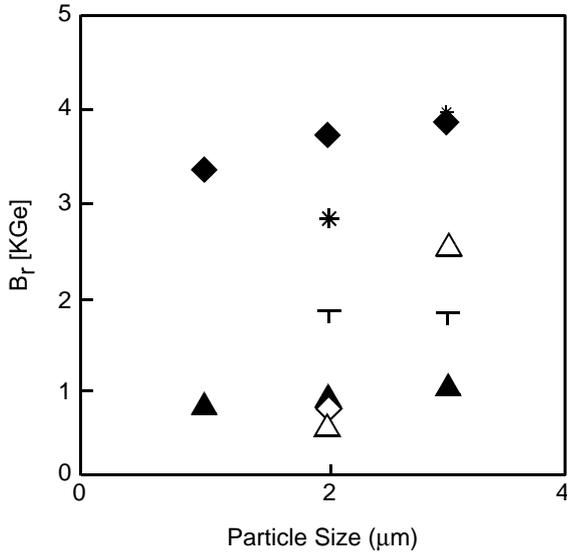
Figure 10 indicates that H_c does not change with the loading level of the magnetic powder. This is because H_c does not depend on density.

The value of (BH)$_{max}$ is ruled by the values of B_r and H_c, so it is affected by compositional changes of the magnets. Since (BH)$_{max}$ is proportional to the
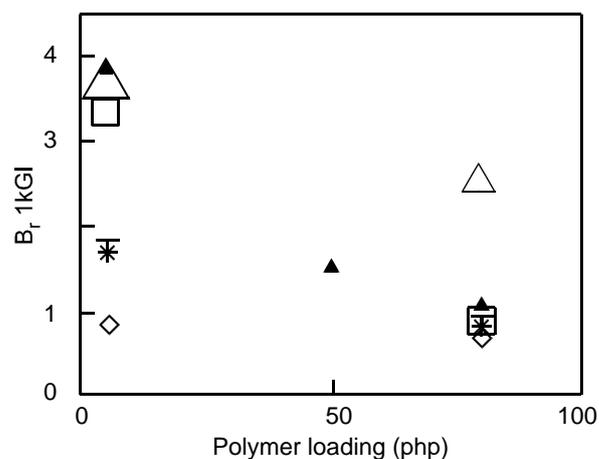
($\square$) PEG-magnetic powder particle size: 1 μm;

($\blacktriangle$) PEG-magnetic powder particle size: 74-100 μm;

($\diamond$) Epoxy-magnetic powder particle size: 2-10 μm;

($\triangle$) Epoxy-magnetic powder particle size: 74-100 μm;

($\ast$) SBR-magnetic powder particle size: 2-10 μm;

($\top$) SBR-magnetic powder particle size: 74-100 μm.

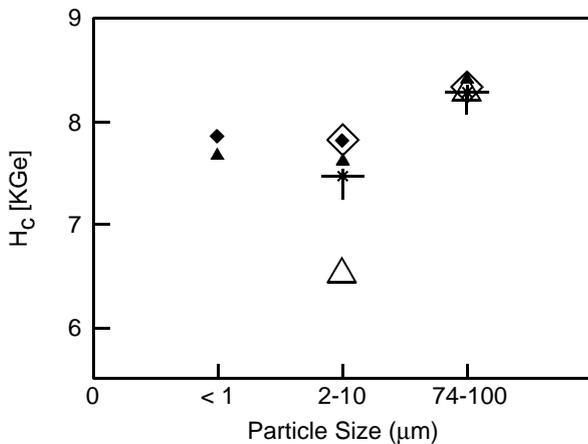**Figure 6.** The effect of polymer loading level on B$_r$.

(◆) PEG - 5 php; (▲) PEG -80 php; (◇) epoxy -5 php; (△) epoxy - 80 php; (✳) SBR - 50 php; (⊤) SBR - 80 php.

Figure 7. The effect of magnetic particle size on B$_r$.

(◆) PEG-magnetic powder particle Size: 1 µm;
(▲) PEG-magnetic powder particle Size: 74-100 µm;
(◇) Epoxy-magnetic powder particle Size: 2-10 µm;
(△) Epoxy-magnetic powder particle Size: 74-100 µm;
(✳) SBR-magnetic powder particle Size: 2-10 µm;
(⊤) SBR-magnetic powder particle Size: 74-100 µm.

Figure 9. The effect of magnetic particle size on (BH)$_{max}$.

square of the remanence, so factors that affect remanence have a larger effect on (BH)$_{max}$. Maximum (BH)$_{max}$ belongs to the PEG magnet with 5 php and the magnetic particle size in the range of 74-100 µm, having plate-like shape.
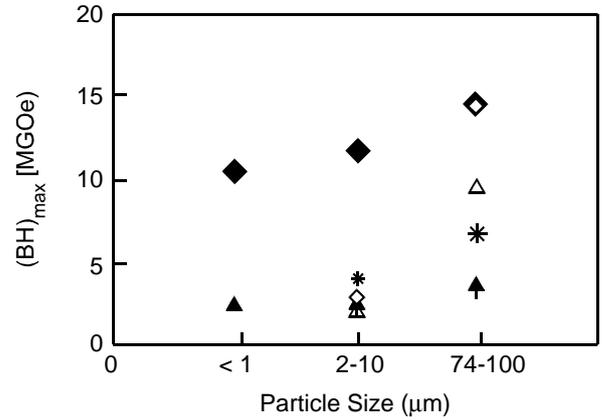
Increasing the amount of polymeric binder has a direct effect on magnetic properties such as B$_r$ and (BH)$_{max}$ (Figure 11).

Index N is defined as the difference in B$_r$ divided by difference in polymer loading level. It, therefore, represents the sensitivity of B$_r$ to the amount of poly-

mer binder. Index M, likewise, is defined as the difference in (BH)$_{max}$ divided by the difference in the polymer loading level (Table 5).

Table 5 indicates that both M and N change according to the following rule: (M,N)$_{PEG}$> (M,N)$_{SBR}$>

(◆) PEG - 5 php; (▲) PEG -80 php; (◇) epoxy -5 php; (△) epoxy - 80 php; (✳) SBR - 50 php; (⊤) SBR - 80 php.

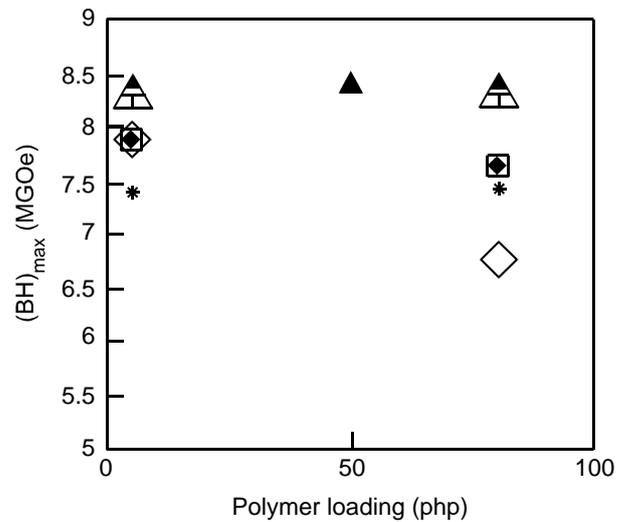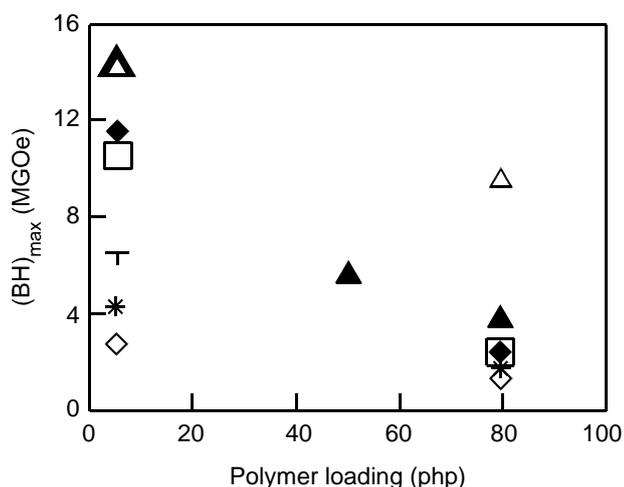**Figure 8.** The effect of magnetic particle size on H$_c$.

(□) PEG-magnetic powder particle size: 1 µm;
(◆) PEG-magnetic powder particle Size: 1 µm;
(▲) PEG-magnetic powder particle size: 74-100 µm;
(◇) Epoxy-magnetic powder particle size: 2-10 µm;
(△) Epoxy-magnetic powder particle size: 74-100 µm;
(✳) SBR-magnetic powder particle size: 2-10 µm;
(⊤) SBR-magnetic powder particle size: 74-100 µm.

Figure 10. The effect of polymer loading level on H$_c$.

(□) PEG-magnetic powder particle size: 1 μm;

(◆) PEG-magnetic powder particle Size: 1 μm;

(▲) PEG-magnetic powder particle size: 74-100 μm;

(◇) Epoxy-magnetic powder particle size: 2-10 μm;

(△) Epoxy-magnetic powder particle size: 74-100 μm;

(✳) SBR-magnetic powder particle size: 2-10 μm;

(⊤) SBR-magnetic powder particle size: 74-100 μm.

Figure 11. The effect of polymer loading level on (BH)$_{max}$.

(M,N)$_{Epoxy}$. This may be explained in terms of processing temperature and the duration of processing for

Table 5. The ratio of magnetic properties differences to differences in polymer loading level (Indices M and N).

No	Sample code	B$_r$ (kG)	(BH)$_{max}$ (MGOe)	Index N	Index M
1	PEG 100-5	3.82	14.454	3.7	13.93
2	PEG 100-80	1.04	4.000		
3	PEG 10-5	3.70	11.644	3.7	12.36
4	PEG 10-80	0.91	2.375		
5	PEG 1-5	3.33	10.555	3.32	10.736
6	PEG 1-80	0.84	2.503		
7	EPO 100-5	3.74	14.243	1.63	6.0
8	EPO 100-80	2.52	9.759		
9	EPO 10-5	0.85	2.804	0.26	1.7
10	EPO 10-80	0.65	1.501		
11	SBR 100-50	1.85	6.450	3.23	10.753
12	SBR 100-80	0.88	3.224		
13	SBR 10-50	1.68	4.322	3.03	8.173
14	SBR 10-80	0.77	1.872		

each magnet.

PEG Magnet was processed at 60°C for less than 5 min. SBR and epoxy magnets were both processed at 150°C, for 7 and 20 min, respectively.

Therefore, higher temperature and pressure and longer preparation time are believed to have caused the decrement in magnetic properties of the samples.

CONCLUSION

The density of the magnet depends on the amount, shape, particle size and the compressibility of the magnetic powder:

- The particles with plate-like shape i.e., the coarser ones, show more tendency to compaction, so have higher densities than the finer particles with round shape.

- Particle size reduction of the magnetic powder leads to a decrease in the densities of samples for the same loading level in each polymeric matrix.

- Sample preparation (processing) conditions affect the density of the magnet. Using intensive high shear mixer has the main effect on the compressibility of magnets containing plate-like shape magnetic powders in SBR matrix.

- Remanence is proportional to the magnetic powder loading level and increases with rising this level.

- Highest values of B$_r$ and H$_c$ are obtained when PEG, a polar polymer, is used. B$_r$ is decreased by particle size reduction, due to higher level of oxidation owing to intensified crushing and higher surface area.

- (BH)$_{max}$ is also influenced by the amount of polymeric binder in a direct manner. All of the indices introduced show a good sensitivity to polymer loading level which varies from one polymer to the other.

- The composite made by 5 php PEG and NdFeB powder with their size in the range of 74-100 μm presented the best overall magnetic properties.

(B$_r$ = 3.82 kG, H$_c$ = 8.415 kOe, (BH)$_{max}$ = 14.454 MGOe).

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