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Application of the Current-applied Pressure-assisted Method for Anisotropic NdFeB Magnets

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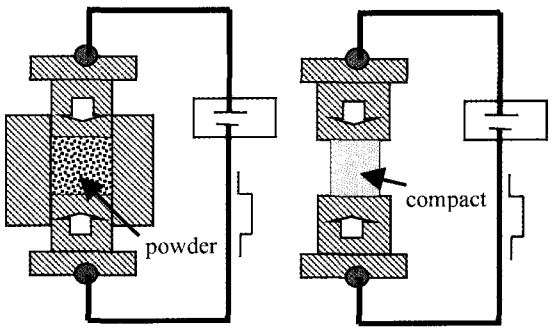
Using the current-applied pressure-assisted (CAPA) process, we could obtain fully dense isotropic and anisotropic NdFeB magnets from rapidly quenched MQP-A powder. The Nd content is found to play an important role during the current applied (CA)-pressing and CA-deformation processes. The $(BH)_{max}$ of CA-pressed and CA-deformed magnets are 131 kJ/m^3 (16.5 MGOe) and 352 kJ/m^3 (44.2 MGOe), respectively. The change in texture of CA-deformed anisotropic NdFeB magnets with thickness reduction was investigated by pole figures and the (006) texture was found to increase with greater thickness reductions. As the thickness reduction increases from 50% to 60% to 80%, W_{50} (the average angle of the contour with 50% intensity) decreases from 76° to 62.5° to 17°.

1. Introduction

NdFeB permanent magnets with high-energy products based on the intermetallic compound $Nd_2Fe_{14}B$ are used for various applications in electric devices [1]. There are two completely different processes for producing NdFeB magnets. One is powder metallurgy [2] and the other is the rapid quenching process (MQ process). To obtain fully dense magnets from rapidly quenched powders, hot pressing [3] and hot deformation [4] of the hot pressed isotropic magnet are needed. The hot working is performed at a high temperature of about 700 °C and a high pressure of about 100 MPa, which lead to grain growth and deterioration of coercivity. Although useful compositions for magnetic alloys from rapidly quenched powders have been identified, there is a need to develop a technology for obtaining fully dense magnets utilizing various melt-spun powders. Recently, several studies [5-9] have been aimed at developing a technique to obtain fully dense magnets from melt-spun powder while avoiding prolonged heating. These methods possess the advantage of short sintering times because explosion, high-velocity impact, and high pulse energy are used. The field-activated and pressure-assisted (FAPA) process [10] is a combined electric field-activated combustion synthesis with the application of mechanical pressure to produce dense compacts of composites or hard metals from their loose powders. Recently, we developed a new method to make fully dense anisotropic NdFeB magnets by modifying the FAPA process. The method, the current-applied pressure-assisted (CAPA) process, utilizes DC electric current and pressure simultaneously. To obtain anisotropic magnets, the magnetic alloy powder was CA-pressed and subsequently CA-deformed. This is similar to the Magnequench process, except for the heating method. In this work, we report the magnetic properties and the texture of specimens obtained by the CAPA process.

2. Experimental Procedure

Commercial NdFeB powders (Magnequench Co.) denoted as MQP-A, MQP-B and MQP-B$^+$ were used as starting materials in the experiments. The powder was placed in a graphite die, between upper and lower graphite punches; then the system was evacuated to 4×10^{-2} Torr and filled with Ar gas. DC current (I_{dc}) in the range 1000~3000 A and pressure (P_a) of 10~90 MPa were applied simultaneously through the upper and lower punches, and the powder was compacted until densification was attained. Finally the sample was cooled down to room temperature, and a fully



(a) CA-press process (b) CA-deformation process

Fig. 1. Schematic diagram of CAPA process.

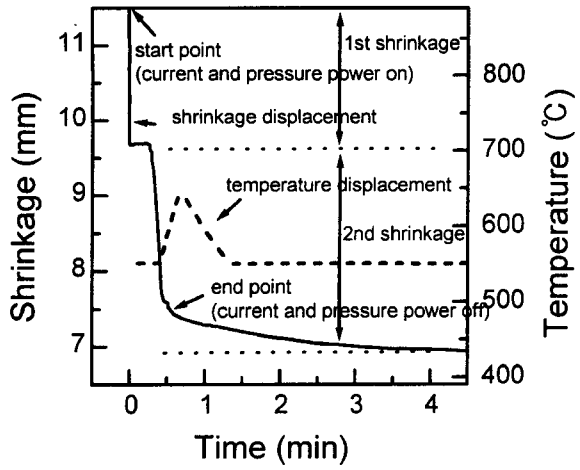


Fig. 2. A practical profile of shrinkgae and temperature displacement.

dense isotropic magnet was obtained. This process is referred to as the current applied press (CA-press) process (See Fig. 1(a)). In order to obtain an anisotropic magnet, the CA-pressed magnet was placed between upper and lower graphite punches without a die and then compressed to deform transversely while current is flowing. This procedure is referred to as the current applied deformation (CA-deformation) process (See Fig. 1(b)). During the CAPA process, the shrinkage of the specimen due to densification or deformation was detected by an LVDT (linear variable differential transformer). Figure 2 shows an experimental profile obtained during the CA-press process. Here, the 1st and the 2nd shrinkage correspond to the compaction of the loose powder and densification due to the Nd-rich liquid phase, respectively. The temperature variation is measured by a pyrometer, which allows measurements only above 550 °C. The time interval between power on and off is approximately 30 seconds. The density of the sample obtained was measured by Archimedes method. After premagnetization at 6400 kA/m (~80 kOe), the magnetic properties were measured by a hysteresisgraph system with a maximum field of 1600 kA/m (~20 kOe).

Pole figures of the (006) reflection were obtained using a Philips X-ray diffractometer and the Schulz reflection method [11]. The detector was set at a fixed angle $2\theta = 44.5°$ relative to the incident beam. The tilt angle (α) was varied from 0° to 80° in steps of 5°. The second rotation axis, the azimuth angle (β), was varied from 0° to 360° in steps of 20°.

3. Results and Discussion

Figure 3 shows the magnetic properties and density of CA-pressed magnets obtained from MQP-A powder at I_{dc} = 3000 A with various P_a. The specimens obtained were disks of 20-mm-diameter and 7-mm-height. As shown in Fig. 3, the remanance and energy product reach their maxima at $P_a = 20$ MPa. The $_iH_c$ increases considerably with increasing P_a. Similar behavior was also observed in specimens fabricated with $I_{dc} = 2000$ A. It is considered that the increase of $_iH_c$ is due to the decrease of exposure time at high temperature. Demagnetization curves of the CA-pressed magnets prepared at $I_d = 3000$ A are shown in Fig. 4. As P_a increases from 10 to 70 MPa, $_iH_c$ increases from 669 kA/m (8 kOe) to 1226 kA/m (16 kOe).

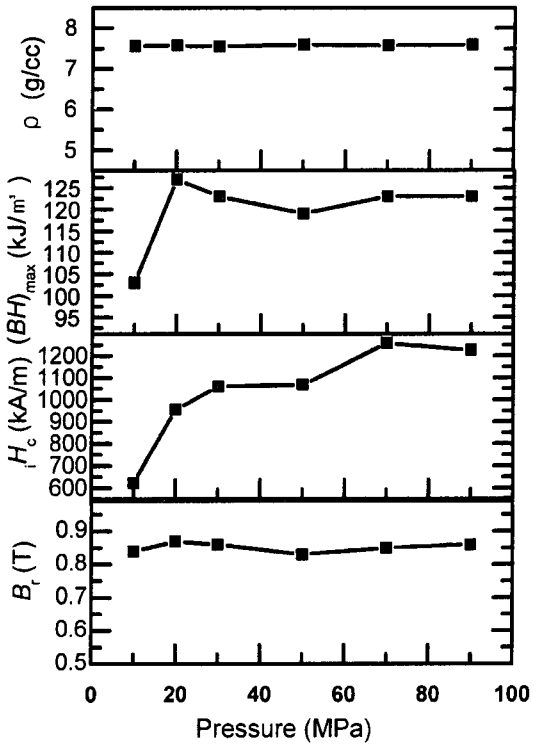


Fig. 3. The magnetic properties and density of CA-pressed magnets at I_{dc} = 3000 A as a function of P_a.

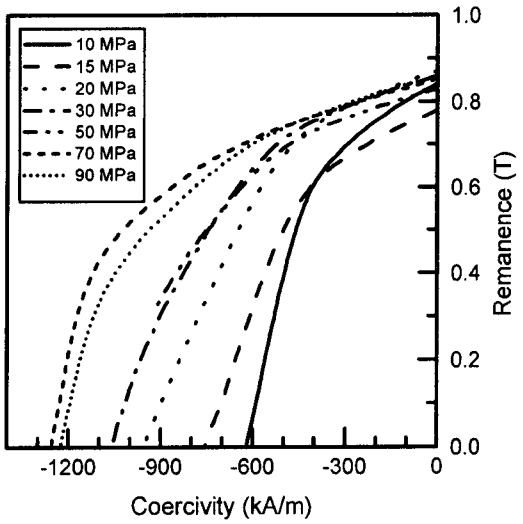


Fig. 4. Demagnetization curves of CA-pressed magnets prepared at I_{dc} = 3000 A with various pressures.

Tables 1 and 2 summarize the relation between the magnetic properties and experimental conditions. There are three kinds of specimens made from MQP-A, MQP-B and MQP-B$^+$ powders, respectively. The CA-pressed isotropic

Table 1. Magnetic properties of isotropic CA-pressed magnets with various I_{dc} and P_a

	Powder	I_d (A)	P_a (MPa)	B_r (T)	$_iH_c$ (kA/m)	$(BH)_{max}$ (kJ/m^3)
ISOA	MQP-A	2000	50	0.87	1345	131
ISOB	MQP-A	2000	70	0.86	1358	127
ISOC	MQP-A	1510	70	0.85	1393	123
ISOD	MQP-A	1510	70	0.85	1393	123
ISOE	MQP-A	1350	70	0.72	1321	88
ISOF	MQP-A	1450	70	0.81	1353	113
ISOG	MQP-B	2000	30	0.74	199	48
ISOH	MQP-B	2000	70	0.81	446	92
ISOI	MQP-B$^+$	2000	50	0.8	287	80

Table 2. Magnetic properties of anisotropic CA-deformed magnets with various thickness reductions TR

	TR (%)	B_r (T)	$_iH_c$ (kA/m)	$(BH)_{max}$ (kJ/m^3)
ANISOA	71	1.28	677	302
ANISOB	77	1.32	804	334
ANISOC	75	1.28	860	308
ANISOD	66	1.2	1059	271
ANISOE	75	1.28	692	295
ANISOF	74	1.34	613	341
ANISOG	84	1.1	374	137
ANISOH	70	10.9	239	111
ANISOI	71	10.8	175	72

magnets and CA-deformed anisotropic magnets are designated as "ISO" (See Table 1) and ANISO" (See Table 2), respectively. The "ANISOA" specimen means the CA-deformed anisotropic magnet obtained from CA-pressed "ISOA" magnet. In CA-pressed magnets obtained from MQP-A powder, although the $_iH_c$ did not differ significantly from the powder precursor, the B_r is somewhat higher than that of powder precursor (B_r = 0.76 T). In the CA-pressed magnets obtained from MQP-B and MQP-B$^+$ powders, $_iH_c$ is much lower than the powder precursors, which results in considerable decrease of $(BH)_{max}$.

The Nd content in MQP-A is much higher than that in MQP-B and MQP-B$^+$, which suggests that the Nd content of the powder significantly affects the magnetic properties of CAPA-magnets. Therefore, high $(BH)_{max}$ anisotropic magnets were not obtained by the CA-deformation process from MQP-B and MQP-B$^+$ isotropic magnets.

Micrographs of polished and etched surfaces parallel to the pressing direction are shown in Fig. 5. It is evident that the particles of a CA-pressed magnet round off and accommodate to the available volume without cracks (Fig. 5(a)). In a CA-deformed magnet, the obvious feature is the substantial reduction in ribbon thickness, which accompanies lateral plastic flow during the CA-deformation process (Fig. 5(b)).

In order to investigate the texture evolution of CA-

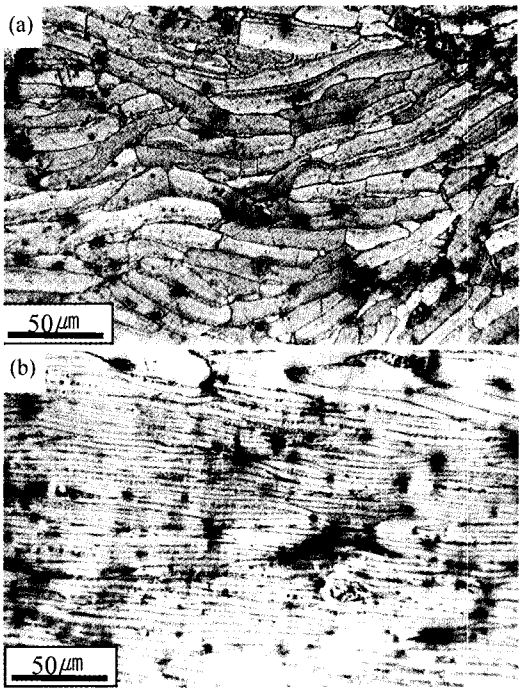


Fig. 5. Micrographs of the surface parallel to the press direction. (a) CA-pressed magnet (b) CA-deformed magnet

deformed magnets prepared with various thickness reductions (TR), pole figures of the (006) reflection were examined. In this work, the TR was defined as the percentage (%) reduction in height of the sample. In Fig. 6, the center of the plot is the specimen normal direction with α=0° and β=0°. The α (radial position) indicates the tilting angle from the specimen normal and β (rotation position) indicates the rotation around the specimen normal. The inner concentric circle is α=45° and the 2nd circle is α=80° (See Fig. 6(d)). The pole density at each point was taken from the integrated intensity of the (006) peak, and the pole figures consist of 7 intensity contours (The highest intensity is marked as number 7 with a bold line). If the specimen is a single crystal or has an ideal grain orientation with the c-axis parallel to the pressing direction, all contours are concentrated at the center point of the stereographic plot. More concentric contours with high intensity at the center imply a higher degree of alignment of the c axes.

Some typical results of the measured (006) pole figures are shown in Fig. 6. By comparing the CA-pressed magnet prepared at I_{dc} = 2500 A and P_a = 70 MPa, the enhancement of (006) texture by increasing the TR from 50% to 70% to 80% is clearly observed in CA-deformed anisotropic magnets. From the demagnetization curves, it was observed that all the CA-deformed magnets had a high remanence and good squareness, and the increase of TR led to an increase of B_r and $(BH)_{max}$.

In order to analyze the effect of deformation on the enhancement of texture, two indexes W_{50} and d are defined as the distribution width of the 50% intensity contour (in Fig. 6, the contour numbered as 4) and the tilting angle of

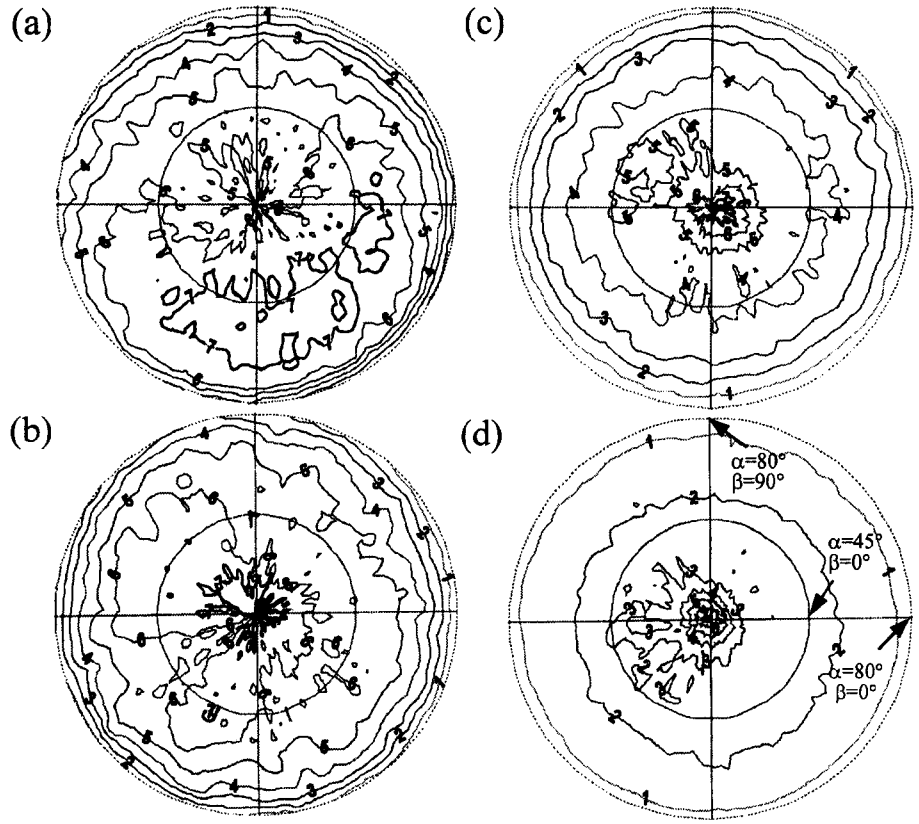


Fig. 6. The (006) pole figures of CA-pressed (a) and 50% (b), 70% (c) and 80% (d) TR CA-deformed magnets.

Table 3. Relation between texture indexes and magnetic properties

TR (%)	0%	50%	70%	80%
W_{50} (deg)	80	76	62.5	17
δ (deg)	50	5	5	0
B_r (T)	8.2	12	13	13.9
$_iH_c$ (kA/m)	1290	1270	820	868
$(BH)_{max}$ (kJ/m^3)	123	299	330	352

the maximum intensity peak (i.e., 100%), respectively. The index W_{50} indicates the width of the pole distribution and d denotes the departure of the texture axis from the pressing axis. The magnetic properties, W_{50} and δ of CA-deformed magnets for various TR are listed in Table 3.

Figure 7(a) shows the relationship between W_{50} and TR. It shows that W_{50} decreases as the TR increases. The variation of B_r, $_iH_c$ and $(BH)_{max}$ with W_{50} are shown in Fig. 7(b), 7(c) and 7(d), respectively. The $(BH)_{max}$ and B_r increase with the decrease of W_{50}, while $_iH_c$ decreases. This trend is similar to that observed by Wang *et al.* [12].

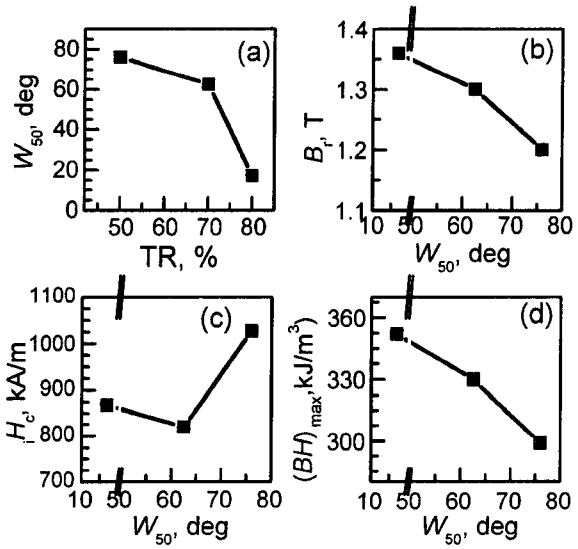


Fig. 7. Variation of W_{50} with TR (a) and variation of B_r (b), $_iH_c$ (c) and $(BH)_{max}$ with W_{50}.

4. Conclusion

Using the CAPA process, we could obtain fully dense isotropic magnets with $(BH)_{max}$ = 131 kJ/m^3 (16.5 MGOe) and anisotropic magnets $(BH)_{max}$ = 352 kJ/m^3 (44.2 MGOe) from MQPA powder. The CAPA-magnets obtained from MQP-B and MQP-B$^+$ powders, however, show poor hard magnetic properties due to their low Nd content. In CA-deformed anisotropic magnets, the (006) texture increased continuously with the increase of thickness reduction.

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