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Application of the current-applied pressure-assisted method for anisotropic NdFeB magnets

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Abstract

By applying Current-applied Pressure-Assisted process, we could obtain full dense isotropic and anisotropic NdFeB magnets from rapidly quenched MQP-A powder. The Nd contents are found to play an important role during the CA-press and CA-deformation process. 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 texture of CA-deformed anisotropic NdFeB magnets with thickness reduction was investigated by pole figure, and the (006) texture was increase with the increase of thickness reduction. With the increment in thickness reduction from 50 %, 60 % to 80%, W_{50} decreases from 76 °, 62.5 ° to 17 °, respectively.

1. Introduction

NdFeB permanent magnets with high-energy products based on $Nd_2Fe_{14}B$ intermetallic compound are promising for various applications of electric device [1]. There are two completely different processes applied producing NdFeB magnets. One is powder metallurgy [2] and the other is rapid quenching process(MQ process). To obtain full dense magnets from rapidly quenched powders, hot pressing [3] and hot deformation [4] of the hot pressed isotropic magnet are needed. The hot workings are performed at high temperature of about 700℃ and high pressure of about 100 ㎫, which led to grain growth and deterioration of coercivity. Although promising composition for magnetic alloys of rapidly quenched powders have been identified, there is a need to develop a technology obtaining full dense magnets utilizing various melt-spun powders. Recently, several studies [5-9] have been focused to develop a technique to obtain full dense magnets from melt-spun powder while avoiding prolonged heating. These methods possess such advantages of high sintering speed

because explosion, high-velocity impact and high pulse energy are used. The field-activated and pressure-assisted(FAPA) process [10] is 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 full dense anisotropic NdFeB magnets by modifying FAPA process. The method, Current-applied pressure-assisted(CAPA) process, utilizes electric DC current and pressure, simultaneously. To obtain anisotropic magnet, the magnetic alloy powder was CA-pressed and subsequently CA-deformed. This is similar to magnequench process, except for heating method. In this work, we report the magnetic properties and the texture of the specimens obtained by the CAPA process.

2. Experimental Procedure

The commercial NdFeB powders of MQP-A, MQP-B and MQP-B$^+$ were used as starting materials in the experiments. The powder were 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_d) of range 1000 ~ 3000 A and pressure(P_a) of 10 ~ 90 MPa were applied simultaneously through upper and lower punches, and the powder was compacted until densification was attained. Finally the sample was cooled down to room temperature, and full 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 anisotropic magnet, the CA-pressed magnet was placed between upper and lower graphite punches without die then compressed to deform transversely while current is flowing. This procedure is referred to as the current applied deformation(CA-

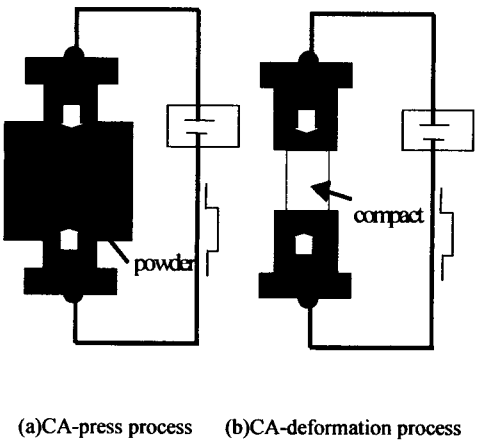

Fig. 1. Schematic diagram of CAPA process

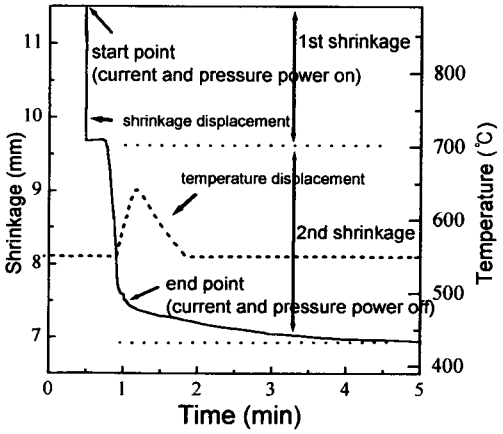

Fig. 2. A practical profile of shrinkage and temperature displacement.

deformation) process(See Fig. 1(b)). During the CAPA process, the shrinkage of specimen due to densification or deformation was detected by LVDT. Figure 2 shows a practical profile obtained during CA-press process. Here, the 1st and the 2nd shrinkage correspond to the compaction of loose powder and densification due to Nd-rich liquid phase, respectively. The duration between power on and off is within 30 seconds, approximately. 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 corresponding to (006) reflection were obtained using a Philips X-ray diffractometer by employing the geometry of 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, 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_d = 3000 A with various P_a. The specimens obtained were disk type of 20-㎜-diameter and 7-㎜-height. As shown in Fig. 3, the remanance and energy product reach to the maximum at P_a = 20 ㎫. The $_iH_c$ increases considerably with increasing P_a. The similar phenomenon was also observed in specimens fabricated by I_d = 2000 A. It is considered that the increase of $_iH_c$ is due to the decrease of exposure time at high temperature because the melting point of Nd-rich phase is lowered at high

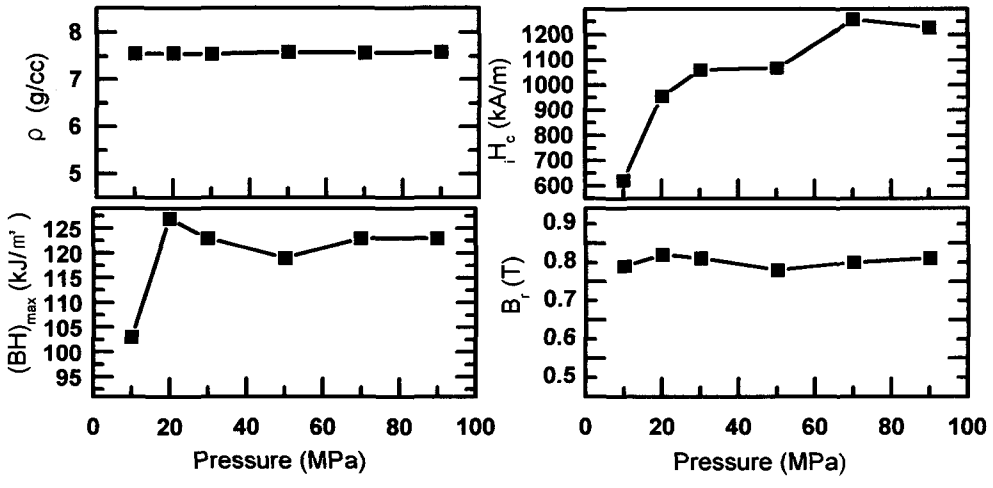

Fig. 3. The magnetic properties and density of CA-pressed magnets as a function applied pressure(I_d = 3000 A).

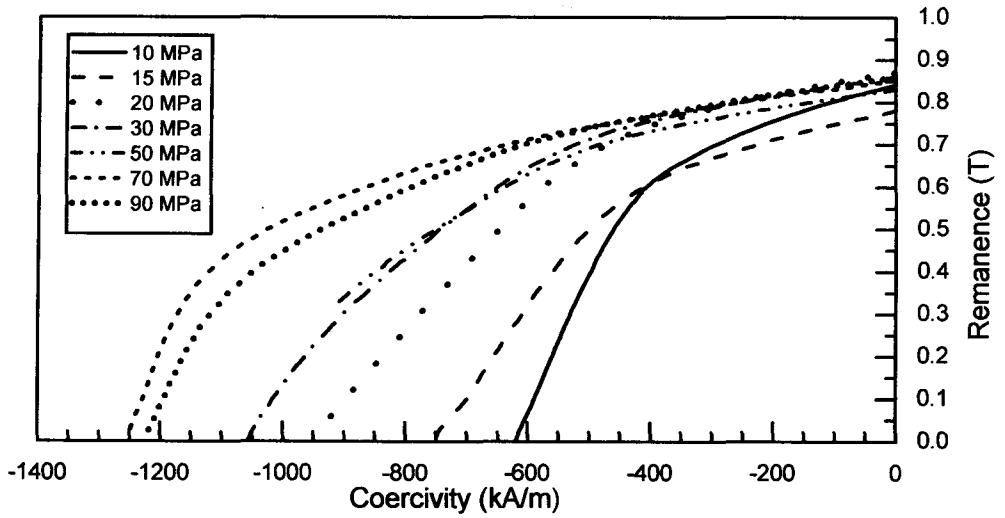

Fig. 4. Demagnetization curves of CA-pressed magnets prepared at 3000 A with various pressure.

applied pressure. Demagnetization curves of the CA-pressed magnets prepared at I_d = 3000 A are shown in Fig. 4. According to the increase of P_a from 10 to 70 MPa, the ${}_iH_c$ increases from 669 kA/m(8 kOe) to 1226 kA/m(16 kOe).

Table 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 magnets and CA-

Table 1. Magnetic properties of CA-pressed magnets with various I_d 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 CA-deformed magnet with TR.

	TR(%)	B_r(T)	${}_iH_c$(kA/m)	$(BH)_{max}$(kJ/m^3)
ANISOA	71	0.128	677	302
ANISOB	77	0.132	804	334
ANISOC	75	0.128	860	308
ANISOD	66	0.12	1059	271
ANISOE	75	0.128	692	295
ANISOF	74	0.134	613	341
ANISOG	84	0.11	374	137
ANISOH	70	0.109	239	111
ANISOI	71	0.108	175	72

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 more or less high than that of powder precursor. In the CA-pressed magnets obtained from MQP-B and MQP-B$^+$ powders, the ${}_iH_c$ is severely deteriorated as compared to 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$^+$. It suggests that Nd content of powder precursor significantly affects on magnetic properties of CAPA-magnets. By this reason, the high $(BH)_{max}$ anisotropic magnets were not obtained by CA-deformation process from MQP-B and MQP-B$^+$ isotropic magnets (ISOG, ISOH and ISOI specimens in Table 1).

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

In order to investigate the texture evolution of CA-deformed magnets prepared with various thickness reduction(TR), the pole figures corresponding to (006) reflection were examined. In this work, the TR was defined as the percentage(%) of reduced height to the original height of sample. In Fig. 6, the center of the plot refers to the specimen normal direction with $\alpha = 0°$ and $\beta = 0°$. The α(radial position) indicates the tilting angle from the specimen normal and β(rotation position) indicates the rotation around the specimen normal. The most inner concentric circle is $\alpha = 45°$ and the 2nd circle is $\alpha = 80°$(See Fig. 6(d)). The pole density at each point was taken from the integrated

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

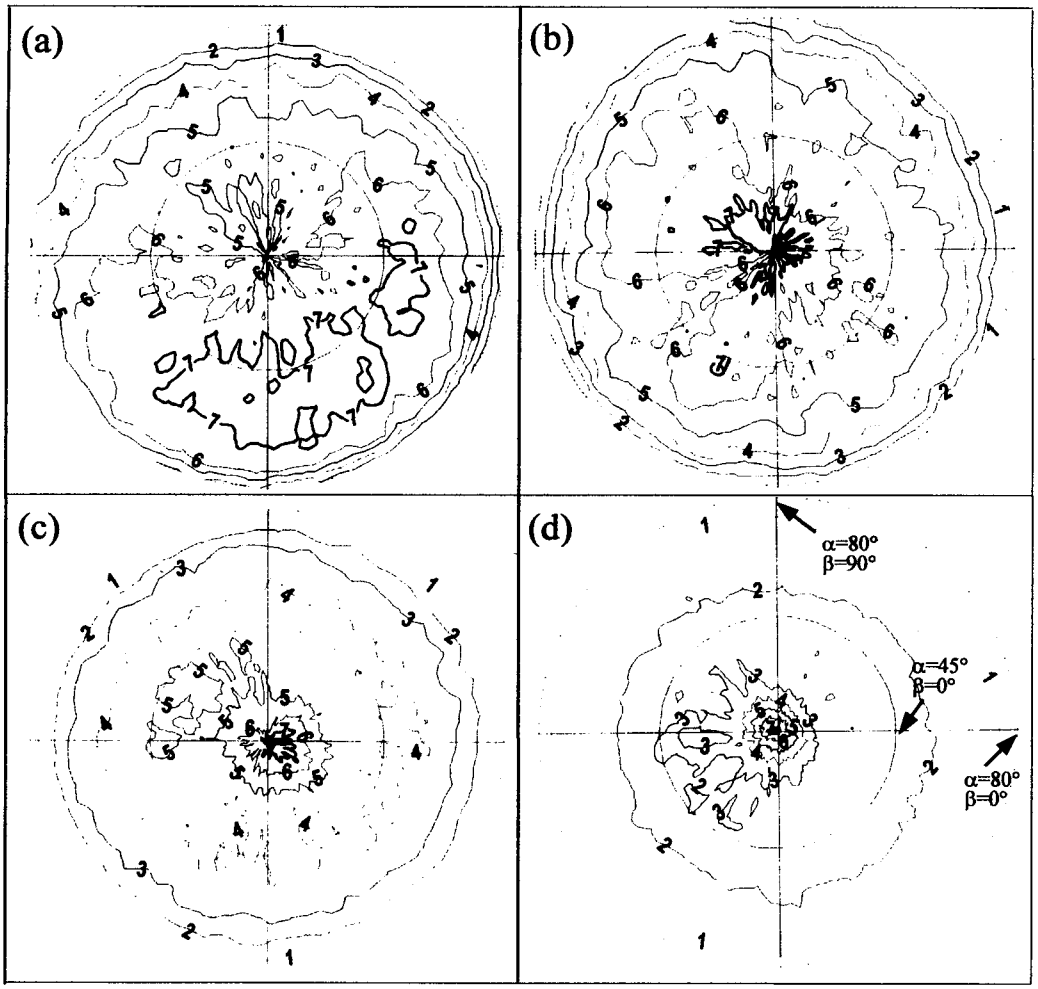

Fig.6. The (006) pole figures of CA-pressed(a) and 50 %(b), 70 %(c) and 80 %(d) TR of CA-deformed magnets, respectively.

intensity of the (006) peak, and the pole figures consist of 7 intensity contours(The highest intensity is marked as number 7 with bold line). If the specimen is single crystal or has an ideal grain orientation with c-axis parallel to the press direction, all contours are focused on the center point of the stereographic plot. The more concentric contours with high intensity at the center imply the more alignment degree to the particular reflection.

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

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

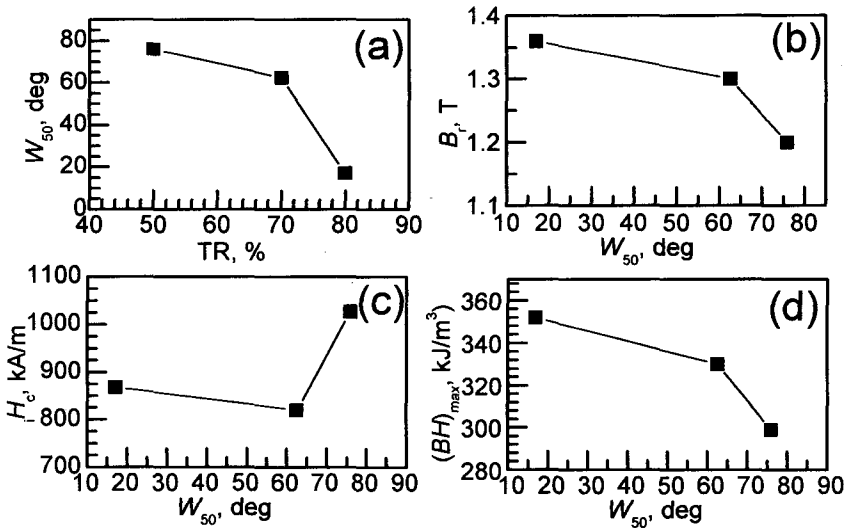

Fig. 7. Variation of W_{50} with TR(a) and variation of B_r(b), ${}_iH_c$(c) and $(BH)_{max}$ with W_{50}.

In order to analyze the effect of deformation on the enhancement of texture, two indexes W_{50} and δ are defined as the distribution width of the 50 % intensity contour(In Fig. 6, the contour numbered as 4) and the tilting angle of the maximum intensity peak(i.e., 100 %), respectively. The index W_{50} indicates the width of the pole distribution and δ denotes the shift of texture from the prealigned 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 TR, while ${}_iH_c$ decreases. This trend is similar to that observed by Wang *et al* [12]. In conclusion, W_{50} can be used as a quantitative index of texture of the anisotropic magnets prepared by CA-deformation process.

4. Conclusion

By applying CAPA process, we could obtain full dense isotropic $(BH)_{max}$ = 131 kJ/m^3

(16.5 MGOe) and anisotropic $(BH)_{max}$ = 352 kJ/m^3(44.2 MGOe). The CAPA-magnets obtained from MQP-B and MQP-B$^+$ powders, however, should poor had magnetic properties due to their low Nd content. In CA-deformed anisotropic magnets, the (006) texture was increased continuously as according to the increase of thickness reduction.

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