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Improving magnetic properties in mischmetal-based sintered composite magnets by regulating element distribution

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ABSTRACT

Mischmetal-based magnets were prepared by mixing 60 wt.% (MM,Nd)14.2-xFe79.8+xB6 (x=0, 0.5, 1, and 1.5) with 40 wt.% Nd15Fe79B6 powders. The coercivity is 9.50 kOe in sintered MM9.8Nd4.4Fe79.8B6/Nd15Fe79B6 magnets, and it decreases to 6.61 kOe in MM9.3Nd3.9Fe80.8B6/Nd15Fe79B6 due to the decrease MM-Nd content of (MM,Nd)-Fe-B alloy and the reduction of the amount of intergranular phase. While for further decreasing MM-Nd content of (MM,Nd)-Fe-B, the effect of Nd diffusing into (MM,Nd)-Fe-B phase to substituting for La-Ce is enhanced in the sintered composite magnets, which would lead to a more increase of magnetocrystalline anisotropy in (MM,Nd)-Fe-B phase. So the coercivity increases to 7.70 kOe in MM8.8Nd3.4Fe81.8B6/Nd15Fe79B6, and the energy product of 33.67 MGOe were obtained in this composite magnets with the 40 wt.% mischmetal in total amount of rare earth. Though the average of magnetocrystalline anisotropy decreases in the sintered composite magnets, the increase of anisotropy in local regions possibly gives rise to the enhancement of coercivity. These investigation shows that the magnetic properties could be optimized by regulating the element distribution in the sintered composite magnets.

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I. INTRODUCTION

Nd-Fe-B magnets are essential components in many technology fields, but their continually growing demand and the wide application over-consume the less abundant rare earth of Nd, and the abundant and inexpensive rare earth elements of La and Ce are overstocked. The mischmetal (MM) is unseparated rare earth, which contains nearly 78 wt.% of La and Ce. Utilizing mischmetal to substitute for Nd in the preparation of permanent magnets could reduce the purification cost and decrease the consumption of Nd. However, the coercivity of MM-Fe-B are much inferior to that of Nd-Fe-B due to the low magnetocrystalline anisotropy of (La,Ce)-Fe-B. Recently, it was found that the coercivity could be improved in sintered (MM,Nd)-Fe-B/Nd-Fe-B prepared by mixing (MM,Nd)-Fe-B powders with Nd-Fe-B powders, since Nd-Fe-B phase bears high magnetocrystalline anisotropy against magnetization reversal in the composite magnets. In high temperature sintering the diffusion of rare earth elements is evident, which would drastically affect the magnetic properties in the sintered composite magnets. Nd substitution for La-Ce should also contribute to the increase of coercivity in the sintered composite magnets. The atomic diffusion is strongly dependent on the atom concentration. In this paper, in order to promote Nd diffusion and substitution for La-Ce at the grain boundary of (MM,Nd)-Fe-B as well as weaken the effect of La-Ce diffusion, MM-Nd content was reduced in (MM,Nd)-Fe-B powders and Nd content was a little higher in Nd-Fe-B powders. It is expected to regulate the atomic diffusion and further improve the magnetic properties in the resource-saving rare earth magnets.
II. EXPERIMENT

The commercial mischmetal, Nd, Fe and Fe-B alloy were mixed according to the nominal composition of \((\text{MM,Nd})_{14.2-x}\text{Fe}_{79.8+x}\text{B}_6\) (x=0, 1, 2, and 2.5). The mischmetal was extracted from Bayan Obo Mine in Baotou, China. It contains La of 28.63 wt.%, Ce of 50.13 wt.%, Pr of 4.81 wt.%, Nd of 16.38 wt.% and other inevitable impurities. The compositions of \((\text{MM,Nd})\)-Fe-B alloys are \(\text{MM}_{9.8}\text{Nd}_{4.4}\text{Fe}_{79.8}\text{B}_6\), \(\text{MM}_{9.3}\text{Nd}_{3.9}\text{Fe}_{80.8}\text{B}_6\), \(\text{MM}_{8.8}\text{Nd}_{4.1}\text{Fe}_{81.8}\text{B}_6\), and \(\text{MM}_{8.6}\text{Nd}_{4.2}\text{Fe}_{82.3}\text{B}_6\), respectively. Nd\(_{15}\text{Fe}_{79}\text{B}_6\) alloys were prepared by induction melting of Nd, Fe and Fe-B alloys. (MM,Nd)-Fe-B and Nd-Fe-B powders with the averaged size of 3 μm were obtained, respectively, by the hydrogen decrepitation and jet-milling. (MM,Nd)-Fe-B powders with 6 grams and Nd-F4 powders with 4 grams were mixed and then blended for 10 minutes to ensure the components uniformity. The mixture was pressed into a cylinder after an alignment under 2 T pulsed magnetic field. The cylinder compacts were putted into a furnace and sintered in vacuum at 1030° C∼1040° C for 2 hours, followed by the annealing at 510° C for 2 hours. The phase constitution was checked by X-ray diffraction measurement (XRD) using Co Kα radiation. Scanning electron microscope (SEM) with backscattered electron (BSE) and energy dispersive spectrometer (EDS) were used, respectively, to observe the microstructure and chemical composition. The demagnetization curves were measured using NIM-200C Loop Tracer at room temperature, and the magnetization curves were measured in the easy and hard magnetization axes, respectively, using Quantum Design Versalab at temperature of 300 K.

III. RESULTS AND DISCUSSION

XRD patterns show that the phase constitutions are nearly the same for these sintered magnets (shown in Fig. 1). Besides the diffraction peaks of main phase of \(\text{R}_2\text{Fe}_{14}\text{B}\), there exist weak diffraction peaks corresponding to the minor phases of \(\text{RFe}_2\text{B}_4\), \(\text{RFe}_2\) and unidentified intergranular phase. The amount of minor phases is a little larger in \(\text{MM}_{9.8}\text{Nd}_{4.4}\text{Fe}_{79.8}\text{B}_6\) due to the stronger intensity of diffraction peaks. Reducing MM-Nd content in (MM,Nd)-Fe-B, the amount of minor phases decreases, however, which increases in \(\text{MM}_{8.6}\text{Nd}_{1.1}\text{Fe}_{62.3}\text{B}_6/\text{Nd}_{15}\text{Fe}_{79}\text{B}_6\) for reducing largely MM-Nd content in (MM,Nd)-Fe-B. Fig. 2 shows the surface micromorphologies of the samples. The brighter regions are the intergranular phase, and the grey regions are the main phase of \(\text{R}_2\text{Fe}_{14}\text{B}\). The darker regions should be the pores, which may result from the detaching in the cutting and polishing for the preparation of SEM sample. The size of most grains of main phase are in the range of 3−7 μm, and the amount of intergranular phases decreases for the reduction of MM-Nd content of the (MM,Nd)-Fe-B alloys in the composite magnets. In order to make clear how the two different types of powders combine in the sintered magnets, the element distribution was observed by EDS. Fig. 3 shows the elemental mappings, the left panel is La, the middle is Ce, and the right is Nd mapping. The color is brighter if the content of the corresponding element is high in the regions, whereas the color is darker. The intergranular phase is much brighter, confirming the rare-earth-rich characteristics of the intergranular phase, and so the element mappings are well consistent with the surface

![FIG. 1. The XRD patterns for the sintered (MM,Nd)-Fe-B/Nd-Fe-B magnets.](image-url)

![FIG. 2. The surface micromorphology for MM\(_{8.6}\text{Nd}_{1.1}\text{Fe}_{62.3}\text{B}_6/\text{Nd}_{15}\text{Fe}_{79}\text{B}_6\) (a), MM\(_{8.6}\text{Nd}_{1.1}\text{Fe}_{62.3}\text{B}_6/\text{Nd}_{15}\text{Fe}_{79}\text{B}_6\) (b) and MM\(_{8.6}\text{Nd}_{1.1}\text{Fe}_{62.3}\text{B}_6/\text{Nd}_{15}\text{Fe}_{79}\text{B}_6\) (c).](image-url)
morphology observed by SEM in Fig. 2. On Nd mapping, Nd-lean regions are labeled by the dotted boxes, and these regions are La-Ce-rich main phase, which correspondingly are brighter in La and Ce mappings. On Ce mapping the darker regions should be Nd-rich main phase. As shown in the Figs. 3(b) and (c), the size of most of La-Ce-rich main phase decreases for reducing MM-Nd content of (MM,Nd)-Fe-B alloys. The change in the size for La-Ce-rich main phase may be ascribed to the atomic diffusion in high temperature sintering.

In order to check the atomic diffusion the element distribution was observed by EDS in these composite magnets. The contents of La, Ce, Nd and Fe are listed in Table I for spots of 1, 2, 3 and 4. The spot 1 are in La-Ce-rich main phase, in which La and Ce contents are larger, and the spot 3 are in the main phase of Nd-Fe-B, in which Nd content is larger. In Fig. 3(a) the atomic percents of La, Ce, Nd-Pr are 2.25 %, 5.65 % and 5.21 %, respectively, for spot 1 of La-Ce-rich main phase. While in Figs. 3(b) and (c) the atomic percents of La and Ce decrease, and that of Nd-Pr increases for spot 1. This result indicates that more atoms of Nd diffuse into La-Ce-rich main phase and substitute for La-Ce. The reduced content of MM in (MM,Nd)-Fe-B alloy and high content of Nd in Nd_{15}Fe_{79}B_{6} should be the origin of more atoms of Nd diffusing into La-Ce-rich main phase. For the spot 2 of the main phase, the content of La-Ce content is 2–4 at.%, verifying that the atomic diffusion is evident in these sintered composite magnets. For spot 4, the atomic ratio of (La,Ce)/Nd is more than the average ratio in the magnets, and La content is much larger in the intergranular phase.\[^{10}\] La_{2}Nd_{12}B phase is relatively unstable in structure,\[^{23}\] and the substitution energy of Nd for La is negative in R_{2}Fe_{14}B phase, which are the thermodynamic origin for La-Ce diffusing out of the main phase.\[^{24,25}\] Though the magnets were prepared under the oxygen-free condition in the powders preparation, pressing and sintering, the oxidation is inevitable due to the easily oxidizable feature of rare-earth, and the oxygen element aggregates mainly in the intergranular rare-earth-rich phase.

| Fig. 3(a)   | 1        | 2        | 3        | 4        |
|-------------|----------|----------|----------|----------|
| O           | 12.33    |          |          |          |
| Fe          | 86.89    | 86.88    | 86.64    | 25.12    |
| La          | 2.25     | 0.35     | 0.22     | 13.39    |
| Ce          | 5.65     | 2.11     | 0.40     | 15.14    |
| Nd-Pr       | 5.21     | 10.66    | 12.74    | 34.02    |

| Fig. 3(b)   | 1        | 2        | 3        | 4        |
|-------------|----------|----------|----------|----------|
| O           | 37.45    |          |          |          |
| Fe          | 85.94    | 86.50    | 86.18    | 10.73    |
| La          | 2.17     | 1.04     | 0.24     | 12.46    |
| Ce          | 4.89     | 2.54     | 0.50     | 7.20     |
| Nd-Pr       | 7.00     | 9.92     | 13.08    | 32.16    |

| Fig. 3(c)   | 1        | 2        | 3        | 4        |
|-------------|----------|----------|----------|----------|
| O           | 39.58    |          |          |          |
| Fe          | 86.84    | 86.98    | 86.08    | 10.33    |
| La          | 1.55     | 0.65     | 0.17     | 12.04    |
| Ce          | 3.83     | 2.33     | 1.51     | 6.55     |
| Nd-Pr       | 7.78     | 10.04    | 12.24    | 31.50    |
Though reduce MM content in (MM,Nd)-Fe-B alloys, the diffusion of La-Ce couldn’t be suppressed in the sintered composite magnets. However, La-Ce prefers to enter the intergranular phase rather than enter into the main phase. The amount of La-Ce diffusing into Nd-Fe-B is limited, and more atoms of Nd diffuse into (MM,Nd)-Fe-B phase and substitute for La-Ce. The distinct atomic diffusion and element distribution would affect the magnetic properties. Fig. 4 shows the demagnetization curves of the sintered (MM,Nd)-Fe-B/Nd-Fe-B composite magnets, and the magnetic properties are listed in Table II. In MM$_{8.8}$Nd$_{1.4}$Fe$_{79.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$, the coercivity is 9.50 kOe, and for reducing the content of MM-Nd the coercivity decreases to 6.61 kOe in MM$_{9.3}$Nd$_{1.8}$Fe$_{80.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$. The decrease of rare-earth content would lead to the decrease in the amount of intergranular rare-earth phase, and so the coercivity decreases due to the weakening of decoupling effect of intergranular phase. While for further reducing MM-Nd content, the coercivity increases to 7.70 kOe in MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$. As shown in Fig. 3(b), La-Ce content decreases and Nd content increases in (MM,Nd)-Fe-B main phase, which would lead to the more increase of the magnetocrystalline anisotropy in (MM,Nd)-Fe-B main phase and should be responsible for the increase of coercivity. In MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ the content of mischmetal in rare-earth is 40 wt.%, and the magnetic properties are better than those of the magnets prepared by mixing 40 wt.% MM$_{15.36}$Fe$_{78.59}$B$_{1.11}$ powders with 60 wt.% (Pr,Nd)$_{13.14}$$(Dy,Fe)_{79.81}$B$_{0.5}$, in which the coercivity is 5.01 kOe and the content of mischmetal in rare-earth is nearly 40 wt.%. For decreasing MM-Nd content to 11.7 at.%, the coercivity decreases to 4.89 kOe in MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ due to the rather low rare-earth content in the magnets. With the reduction of MM-Nd content from 14.2 at.% to 11.7 at.% in (MM,Nd)-Fe-B alloy, the remanence increases monotonously. The increase of remanence should arise from the enhanced saturation magnetization owing to the increase of Fe content and the decrease of rare earth content in the alloys. The energy product of 33.67 MGOe were obtained in MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ magnets, which is higher than that of magnets with the same mischmetal content.

Magnetocrystalline anisotropy is the origin of the high coercivity in R$_{15}$Fe$_{81}$B magnets. Fig. 5 shows the magnetization curves measured along the directions of hard and easy magnetization axes, respectively, for these sintered composite magnets. The average of magnetocrystalline anisotropy could be obtained by the intersection of the extension lines of experiment data, and they are 6.76 T, 6.46 T, 6.38 T, and 6.35 T, respectively, for MM-Nd atomic percent of 14.2%, 13.2%, 12.2%, and 11.7%. With the reduction of MM-Nd content in (MM,Nd)-Fe-B the average of magnetocrystalline anisotropy decreases slightly in the composite magnets (shown in the inset of Fig. 5). So it is believed that the increase of coercivity in MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ magnets should be attributed to the change of anisotropy in local regions due to the distinct element diffusion. Since the more amount of the excess Nd in Nd$_{15}$Fe$_{79}$B$_6$ diffuses into (MM,Nd)-Fe-B phase and substitutes for La and Ce, the magnetocrystalline anisotropy increases more largely in (MM,Nd)-Fe-B phase. Magnetization reversal undergoes the nucleation of reversed domain for decreasing external applied field. Because the reversed domain nucleation occurs in the regions of weak anisotropy, the nucleation field would increase for the more increase of anisotropy in (MM,Nd)-Fe-B phase, leading

![FIG. 4. The demagnetization curves for the sintered (MM,Nd)-Fe-B/Nd-Fe-B magnets.](image)

**TABLE II.** Magnetic properties for the sintered composite magnets at room temperature.

| Composition          | $M_r$ (Gs) | $H_c$ (kOe) | $(BH)_{\text{max}}$ ((MGOe)) |
|----------------------|------------|-------------|-------------------------------|
| MM$_{8.8}$Nd$_{1.4}$Fe$_{79.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ | 11.31      | 9.50        | 28.00                         |
| MM$_{8.2}$Nd$_{3.9}$Fe$_{80.3}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ | 12.09      | 6.61        | 31.61                         |
| MM$_{8.8}$Nd$_{3.4}$Fe$_{81.8}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ | 12.27      | 7.70        | 33.67                         |
| MM$_{8.8}$Nd$_{3.1}$Fe$_{82.3}$B$_6$/Nd$_{15}$Fe$_{79}$B$_6$ | 12.61      | 4.89        | 32.55                         |

![FIG. 5. The magnetization curves in the direction of easy and hard magnetization axes and their linear extension of the experimental data up to intersection, and the inset shows the variation of magnetocrystalline anisotropy with the MM-Nd content in (MM,Nd)-Fe-B alloy.](image)
to the enhancement of coercivity. The amount of La-Ce diffusing into Nd-Fe-B is limited, and so it nearly keeps high magnetocrystalline anisotropy in Nd-Fe-B phase. The magnetization reversal in (MM,Nd)-Fe-B phase could be suppressed by Nd-Fe-B phase, which also contributes to the increase of coercivity. Employing the method of conventional magnets of strip casting and the additions of Cu, Ga and Nb for modifying the microstructure, the further improvement of coercivity and energy product could be anticipated in the resource-saving magnets with dual-main-phase.

IV. CONCLUSION

In summary, the sintered (MM,Nd)-Fe-B/Nd-Fe-B composite magnets have been prepared by varying the content of MM-Nd in (MM,Nd)-Fe-B alloys for regulating the atomic diffusion. La and Ce elements are more probably expelled from the main phase and diffuse into the intergranular phase. For decreasing MM content of (MM,Nd)-Fe-B, Nd content increases in (MM,Nd)-Fe-B phase and substituting for La-Ce, which would lead to the more increase of magnetocrystalline anisotropy in (MM,Nd)-Fe-B phase. So the coercivity increases to 7.70 kOe in MM$_{33}$Nd$_{15}$Fe$_{79}$B$_6$ though the amount of intergranular phase decreases in the sintered magnets. With the reduction of MM-Nd content the remanence increases monotonously, and the energy product of 33.67 MGOe were obtained in this composite magnets with 40 wt.% mischmetal and X. Y. Zhang, Appl. Phys. Lett. 103, 390 (2018).

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