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Effect of Fe-Mn addition on microstructure and magnetic properties of NdFeB magnetic powders

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Abstract. In this paper, the effect of Fe-Mn alloy addition on microstructures and magnetic properties of NdFeB magnetic powders was investigated. Varied Fe-Mn compositions of 1, 5, and 10 wt% were mixed with commercial NdFeB type MQA powders for 15 minutes using shaker mill. The characterizations were performed by powder density, PSA, XRD, SEM, and VSM. The Fe-Mn addition increased the powder density of NdFeB/Fe-Mn powders. On the other side, particle size distribution slightly decreased as the Fe-Mn composition increases. Magnetic properties of NdFeB/Fe-Mn powders changed with the increasing of Fe-Mn content. SEM analysis showed the particle size of NdFeB/Fe-Mn powder was smaller as the Fe-Mn composition increases. It showed that NdFeB/Fe-Mn particles have different size and shape for NdFeB and Fe-Mn particles separately. The optimum magnetic properties of NdFeB/Fe-Mn powder was achieved on the 5 wt% Fe-Mn composition with remanence M_r = 49.45 emu/g, coercivity H_c = 2.201 kOe, and energy product, BH_{max} = 2.15 MGOe.

1. Introduction

Nanocomposite magnets composed by magnetically hard and soft phases have attracted more attention due to its higher remanence, coercivity and energy product for permanent magnet development [1–4]. In order to enhance magnetic properties such as remanence (M_r/M_s > 0.5 for isotropic magnets) and fairly coercivity values, both hard and soft magnetic phases are in nanoscale range so that the adjacent grain are exchange coupled [5]. To optimize the exchange couple magnetization, some soft magnetic phases have been added by researchers for specific elements such as α-Fe, Zr, Mn, and MnBi [6–11].

The work from Li et al. [12] showed that the core-shell structure of nanocomposite system based NdFeB/α-Fe could increase both the remanence and coercivity, which leads to energy product of isotropic magnets exceeding 25 MGOe. The addition of FeB to $Nd_2Fe_{14}B$ structure also enhanced the coercivity field as reported by Mingxiang et al. [13]. Previous studies revealed that the addition of Mn to NdFeB could promote crystallization and enhance hard magnetic properties of materials [5]. On the other hand, the particle size of Mn doped NdFeB could affect the curie temperature, but lower the coercivity values. These effects were contributed by the decreasing of magnetocrystalline anisotropy [8].

In this paper, we reported and discussed the effect of Fe-Mn alloy addition to the NdFeB structure by mechanical mixing method. The magnetic properties from VSM measurements are reported. The microstructure and physical characteristics analyzed by XRD, SEM, PSA and density measurement are also discussed.

2. Experimental method

The commercial powders of NdFeB type MQA from Magnequench Inc. and Fe-Mn were utilized in this experiment. The powders were sieved using standard sieves until they passed #200 meshes. Varied composition of Fe-Mn from 1, 5, and 10 wt% was added to NdFeB powders and mixed with shaker mill for 15 minutes in toluene medium to prevent oxidation from environment. During mixing process, the powder to ball ratio was 1:10. After milling, the mixed slurry were dried in vacuum chamber (P = 10 mbar) at room temperature to obtain dried NdFeB/Fe-Mn powders.

The powder density was measured by pycnometer method using toluene as medium. Particle size distribution was analyzed by CILAS Particle Size Analyzer. SEM images were obtained by SEM Hitachi SU-3500. Magnetic phase analysis was performed by Smartlab Rigaku X-Ray Diffractometer using Cu-Kα radation. Magnetic properties were analyzed using Vibrating Sample Magnetometer type VSM250 from Dexing Magnet Ltd.

3. Results and discussions

The mean particle size and powder density measured by PSA and Pycnometer method are displayed in figure 1. It showed that the average value of particle size decreases as Fe-Mn content increases. This effect was contributed by the brittle characteristics of Fe-Mn alloys [14]. Therefore, the powder density of NdFeB/Fe-Mn is expected to increase as also displayed in figure 1. The results showed that the correlation of mean particle size and powder density is inversely proportional as the Fe-Mn content increases.

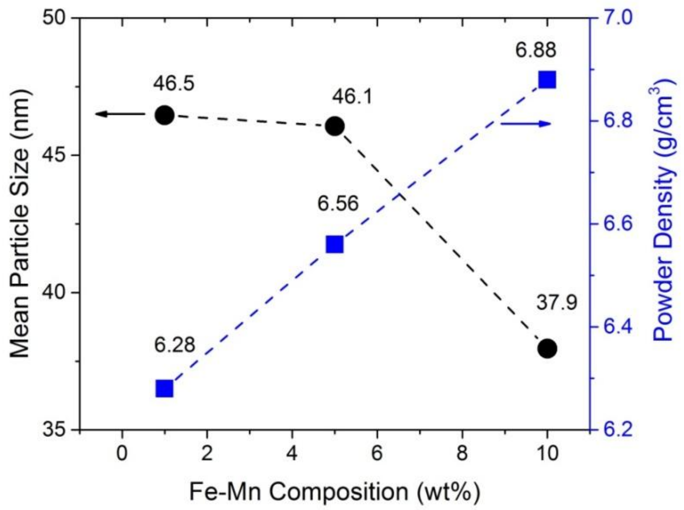
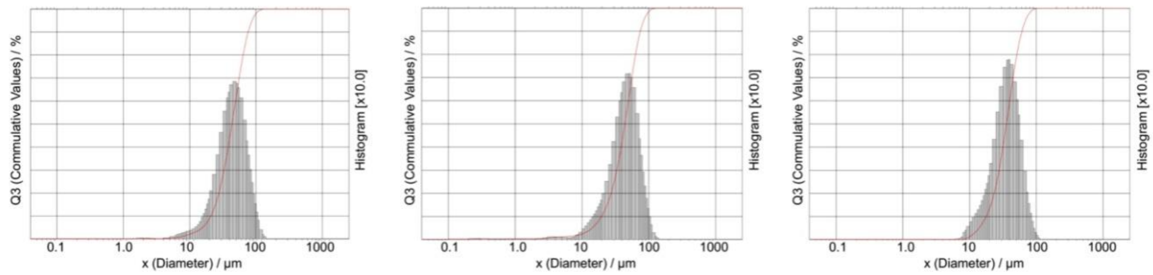


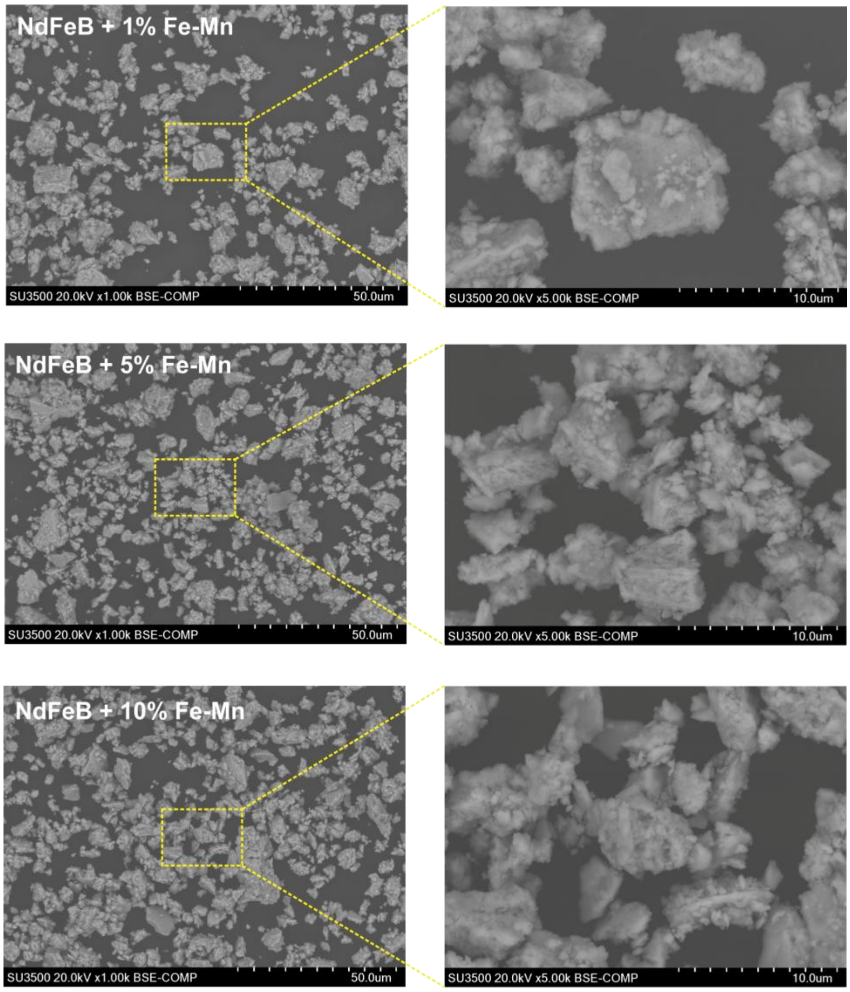
Figure 1. Mean particle size and powder density of NdFeB/Fe-Mn powders as a function of Fe-Mn composition.

The size distribution of NdFeB/Fe-Mn powders as measured by PSA is displayed in figure 2. It showed that the particle size distributions are narrower as the Fe-Mn content is increasing. Compared to the mean particle size values, the 50% cumulative diameter values in figure 2 are shifting to lower values.



(a) (b) (c)

Figure 2. Size distribution of NdFeB/Fe-Mn powders measured by PSA with variation Fe-Mn composition of (a) 1 wt%, (b) 5 wt% and (c) 10 wt%.



(a) (b)

Figure 3. SEM analysis of NdFeB/*x*Fe-Mn (*x* = 1, 5, 10 wt%) powder with different magnification of (a) 1.000X and (b) 5.000X.

The morphology and microstructure analysis was performed by SEM analysis as shown in figure 3. The results showed that the particle size of NdFeB/Fe-Mn powder is confirmed to be smaller as the Fe-Mn composition increases. It showed that the NdFeB/Fe-Mn particles have different size and shape for NdFeB and Fe-Mn particles separately. In addition, the NdFeB has larger size and rounded shape with particle size of 5–10 µm. The Fe-Mn has smaller size and platelet shape with particle size of 1–3 µm. These values are far lower than the PSA analysis average values due to sampling area method in SEM analysis.

The magnetic phases analysis performed by XRD is displayed in figure 4. It showed that the Fe-Mn alloy (sample a) is composed by Fe_3Mn_7, α-Fe, and MnO_2. On the other hand, pure NdFeB powder is composed by single phase $Nd_2Fe_{14}B$ (sample b). The increasing amount of Fe-Mn phases in NdFeB/Fe-Mn powders are shown by the increasing Fe-Mn phases peak intensity. However, the mixing process by shaker mill affects the peak broadening of $Nd_2Fe_{14}B$ structure. It indicates that the crystalline size decreases as the milling process conducted. The work of Chikhalia et al. stated that the milling process could deteriorate the crystalline structure and reduce the particle size [15]. Based on the Scherrer formula, the average crystalline size of the NdFeB/Fe-Mn powders is 22.8 nm. Compared to the unmilled sample, the average $Nd_2Fe_{14}B$ crystalline size is 35.6 nm.

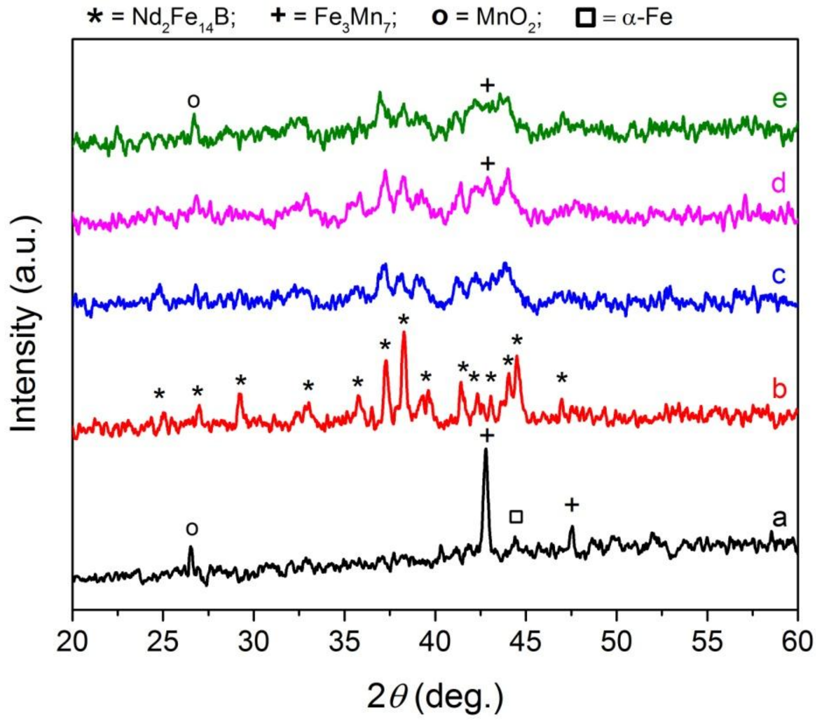


Figure 4. XRD curve of (a) pure Fe-Mn, (b) pure NdFeB and NdFeB/Fe-Mn powder with varied Fe-Mn composition of (c) 1 wt%, (d) 5 wt% and (e) 10 wt%.

The hysteresis curve analyzed by VSM is displayed in figure 5. It showed that the addition of Fe-Mn to NdFeB powders changed the magnetic behavior such as magnetic saturation (M_s), remanence (M_r), coercivity field (H_c), and energy product (BH_{max}). The magnetic remanences slightly decreased as the Fe-Mn content increases. However, the coercivity values have maximum point, which is obtained at 5 wt% Fe-Mn addition with M_r = 49.45 emu/g and H_c = 2.201 kOe. These properties lead to higher energy product, BH_{max} with value of 2.15 MGOe. The detailed magnetic properties data for all samples are presented in table 1.

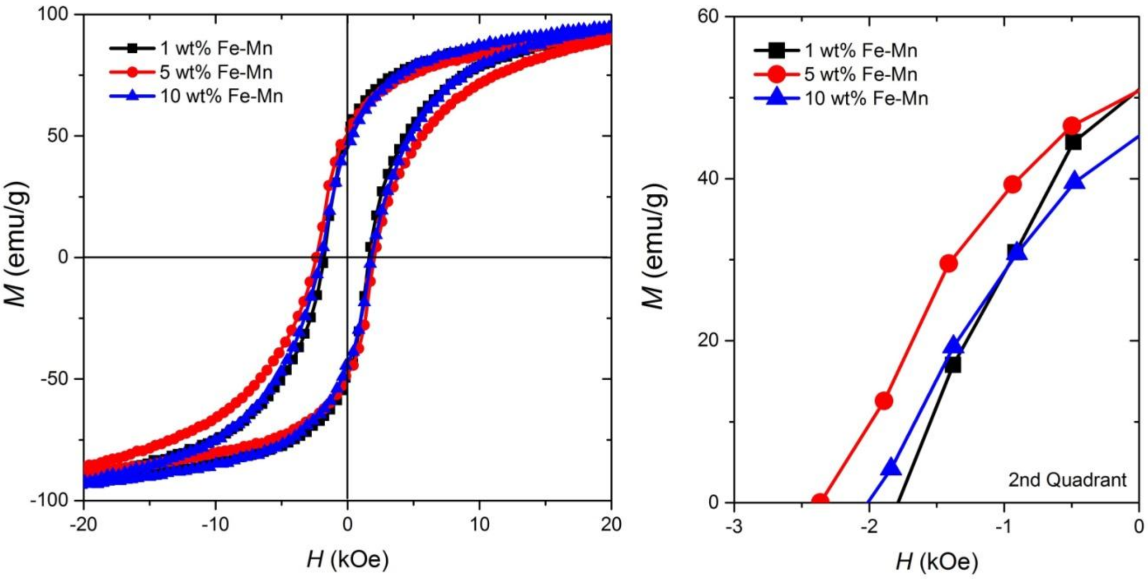


Figure 5. Hysteresis curve and magnification of the 2nd quadrant of NdFeB/Fe-Mn magnetic powders with varied Fe-Mn composition of 1 wt%, 5 wt%, and 10 wt%.

Table 1. Magnetic properties of NdFeB/Fe-Mn powder.

Sample	M_s (emu/g)	M_r (emu/g)	H_c (kOe)	M_r/M_s	BH_{max} (MGOe)
NdFeB/1 wt% Fe-Mn	94.12	49.94	1.706	0.531	1.63
NdFeB/5 wt% Fe-Mn	92.18	49.45	2.201	0.536	2.15
NdFeB/10 wt% Fe-Mn	96.22	44.07	1.911	0.458	1.65

4. Conclusion

In this work, the effect of Fe-Mn addition to NdFeB powders by mechanical mixing method has been investigated. The Fe-Mn addition increased the powder density of NdFeB/Fe-Mn powders. On the other side, the particle size distribution slightly decreased as the Fe-Mn composition increases. The SEM images showed that the particle size of NdFeB/Fe-Mn powder was smaller as the Fe-Mn composition increases. It showed that the NdFeB/Fe-Mn particles have different size and shape for NdFeB and Fe-Mn particles separately. Magnetic properties of NdFeB/Fe-Mn powders change as the increasing of Fe-Mn content. The optimum magnetic properties of NdFeB/Fe-Mn powder was achieved on the 5 wt% Fe-Mn composition with remanence M_r = 49.45 emu/g, coercivity H_c = 2.201 kOe, and energy product, BH_{max} = 2.15 MGOe.

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