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Microstructure and magnetic properties of Fe–79%Ni–4%Mo alloy fabricated by metal injection molding

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ABSTRACT

Fe–79%Ni–4%Mo alloy is well known to be a soft magnetic material widely used for the magnetic head, magnetic shield, and instrumentation components because of high permeability and low coercivity. In order to realize economical mass production of minisize, complex shaped and high performance soft magnetic parts, Fe–79%Ni–4%Mo alloy was produced by metal injection molding, using carbonyl iron, carbonyl nickel and molybdenum powder as raw materials. The effects of sintering temperature and time on the microstructure and magnetic properties of the alloys were investigated. The results indicate that the magnetic properties are dependent on the microstructure. The densification and grain size of the alloys increase with increasing sintering temperature and time, facilitating the enhancement of permeability and saturation induction, as well as the decrease of the coercive force. In the case of the sintering temperature of 1320 °C for 10 h, the relative density of 97.2%, the maximum permeability of 116000, saturation induction of 0.8 T and coercive force of 1.126 A/m were achieved. Further elongation of sintering time did not bring about an increase of densification and grain size.

1. Introduction

For microminiaturization and multifunction of the shape of magnetic devices in recent years, improvement of magnetic properties is being required to soft magnetic components [1–5]. Fe–79%Ni–4%Mo alloy exhibits excellent magnetic performances of high permeability and low coercive force, which is produced for a wide range of applications in areas including industrial engineering, electronics, automotive and electrical industry [6–9]. Conventional soft magnetic alloys were fabricated by the methods of casting and machining. The maximum permeability, saturation induction and coercive force of wrought Fe–79%Ni–4%Mo alloy are usually 100,000–220,000, 0.7–0.8 T and 2.4–0.96 A/m respectively [10,11]. The magnetic properties are good, however, mass production of the miniaturization parts with complex shape is greatly limited due to the long production period, low efficiency and high cost. Generally, soft magnetic properties are dependent on density, grain size and the amount of impurities. Not full dense materials could have their density dependent properties decreased, and also the generated microstructure can be affected, showing large pores, that could act domain-wall barriers reducing the soft magnetic performance [1,12]. The Fe–79%Ni–4%Mo alloy fabricated by powder metallurgy, whose maximum permeability is 77,000, saturation induction is 0.7 T, coercive force is 5.57 A/m and relative density is 90% and the magnetic properties of the alloy are lower than the Fe–79%Ni–4%Mo alloy fabricated by casting method [13]. This is attributed to its low density and too much microstructure defects.

Metal injection molding (MIM) is a near-net shaping technique that is particularly advantageous for the applications where complex shape with high dimensional accuracy and high density are required [14–17]. The advantages including high part density, more intricate shape and superior of the MIM technology give the way for the manufacturing of minisize, complex shaped and high performance soft magnetic parts. The relationships between processing parameters and density, microstructure, contamination and magnetic properties of the Fe–50%Ni alloys produced by MIM were investigated. The results indicated that the magnetic properties of the Fe–50%Ni alloys were greatly dependent on microstructure and the content of interstitial atom such as C, O, N, etc. High magnetic property Fe–50%Ni alloy with the maximum permeability of 89,000, saturation induction of 1.58 T, coercive force of 2.52 A/m was obtained MIM [18–20]. The differences between Fe–50%Ni alloys and Fe–79%Ni–4%Mo alloys are that Fe–50%Ni alloys have the higher saturation magnetic induction but lower maximum permeability while Fe–79%Ni–4%Mo alloys have higher permeability and lower coercive force, which can be used in circuits that are excited by very low currents and require quick response. Some researches concerning Ni–Fe–Mo powders system produced by mechanical alloying were reported and FeNiMo nanocapsules have been studied and prepared by the arc-discharge technique [21,22]. However, few researchers have studied the magnetic properties of Fe–79%Ni–4%Mo fabricated by MIM. In this paper, Fe–79%Ni–4%Mo alloy was produced by metal injection molding and the effect that several important processing parameters had on the magnetic performance of the Fe–79%Ni–4%Mo alloy was reviewed. The factors that influence the magnetic properties were analyzed.

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Table 1
Characteristics of the powders.

Powder	Mean particle size (μm)	ρ_{tap}/g cm^{-3}	Impurity (wt.%)		
			C	O	N
Fe	4.33	3.97	0.63	0.27	<0.001
Ni	4.45	1.95	0.06	0.049	<0.001
Mo	6.30	2.63	0.12	0.13	<0.001

2. Experimental

Carbonyl iron, carbonyl nickel and molybdenum were mixed at the weight ratio of 17:79:4. The characteristics of the powders are given in Table 1. The mean particle sizes of carbonyl iron, carbonyl nickel and molybdenum are 4.33, 4.45 and 6.30 μm, respectively. The morphologies of the three kinds of powder are shown in Fig. 1. Carbonyl iron is mainly regular spherical particles without severe agglomeration, as shown in Fig. 1 (a). From Fig. 1 (b) and (c), it can be seen that the carbonyl nickel has a branched chain and the agglomeration of carbonyl nickel and molybdenum is obvious. Fig. 2 shows the morphologies of the mixing powder. The branched chain of carbonyl nickel is broken and the mixture is homogeneous with a good dispersion.

Metal powder mixture and binder were mixed using a Qunyi PSJ32 type mixer at the temperature range of 140–150 °C for 60–90 min. The wax-based binder was composed of 60 wt.% paraffin, 15 wt.% high density polyethylene, 10 wt.% polypropylene, 10 wt.% polystyrene and 5 wt.% stearic acid. The powder loading of the obtained feedstock was 53 vol.%.

The feedstock was injected into ring-shaped samples with a Zhengde CJ-80E type injection molding machine at 150–160 °C. The injected preforms were subjected to solvent debinding and thermal debinding. Solvent debinding was carried out in trichloroethylene solvent at room temperature for 360 min, and 58 wt.% of the binder was removed. Thermal debinding and pre-sintering was performed under a hydrogen atmosphere with the top temperature of 800 °C with a total cycle of 900 min. Subsequently, the debound samples were sintered at the temperature range of 1240–1360 °C for varied times under the hydrogen atmosphere.

Optical microstructure was observed on a Reicher MeF3A metallurgical microscope. Observation of the morphology of the elemental powder was conducted on a Hitachi S-360 scanning electronic microscope (SEM). The densities of the samples were measured by the Archimedes method. The magnetic properties such as saturation induction (Bs), coercive force (Hc) and maximum permeability (μ_m) were tested on the NMIs NIM-2000S dc soft magnetic properties measuring device.

3. Results and discussion

3.1. Effect of sintering temperature

Density is the key factor that affects the magnetic properties of MIM parts. Higher sintering temperature generates higher sintered density, which improves magnetic properties and microstructure. The effects of sintering temperature on density, microstructure and magnetic properties of Fe–79%Ni–4%Mo alloy were studied in the temperature range of 1240–1360 °C.

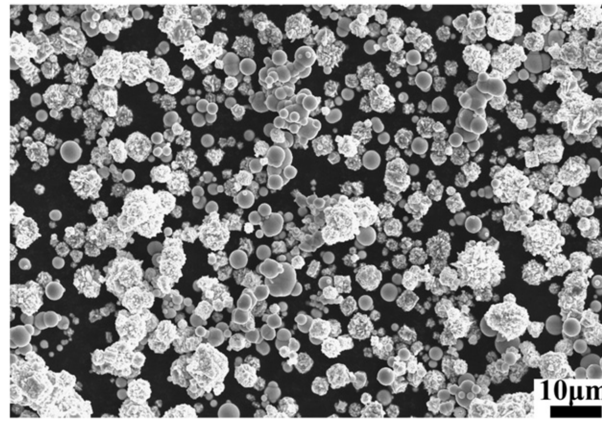

Fig. 2. The morphologies of the mixing powder.

Fig. 3 shows the effect of sintering temperature on the relative density and saturation induction of the specimens sintered at varied temperature for 2 h. The relative densities of the samples increase from 92.1% to 94.4% in the temperature range of 1240–1360 °C, and corresponding saturation induction increases from 0.781 T to 0.795 T. The densification is improved due to the enhancement of solid state diffusion at high temperature. As for soft magnetic, saturation induction belongs to the microstructure insensitive parameters, which depends on the chemical composition and density of the specimens. For a given alloy system, saturation induction is merely related to the density [23,24]. Thus, the saturation induction increases with the improvement of density, as shown in Fig. 3.

Fig. 4 shows the effect of sintering temperature on the microstructures. It can be seen in Fig. 4 (a) that the mean grain size is about 20 μm at the temperature of 1240 °C. The grains grow as the temperature increases. The black dots existing in the figure are porosities. It is obvious that the amount of porosity decreases when the temperature ranged between 1320 and 1360 °C.

Fig. 5 shows the relationship between maximum permeability, coercive force and the sintering temperature. The maximum permeability increases while the coercive force decreases gradually with increasing temperature. The maximum permeability reaches 64,000 and coercive fore is 2.201 A/m at the sintering temperature of 1360 °C. Compared to the magnetic property of saturation induction, the maximum permeability and coercive force are microstructure sensitive parameters, which are influenced by the density, porosity and grain size [25–27]. As indicated in Figs. 3 and 4, higher sintering temperature generally results in higher sintered density as well as larger grain sizes, which improves the magnetic performance of Fe–79%Ni–4%Mo alloy.

These above experiments demonstrate that increasing sintering temperature generally results in higher sintered density, larger grain sizes and more refined pore morphology and as a result, greatly enhanced soft magnetic properties.

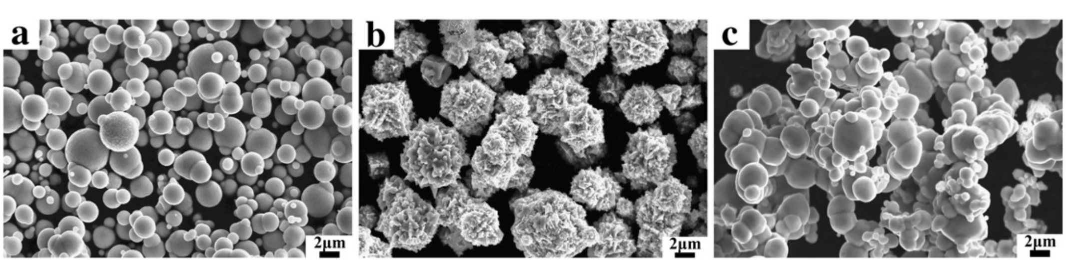

Fig. 1. SEM images of powders: (a) carbonyl iron; (b) carbonyl nickel; (c) molybdenum.