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Effect of ball milling on the rheology and particle characteristics of Fe–50%Ni powder injection molding feedstock

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ABSTRACT

Fe–50%Ni soft magnetic alloys were produced by powder injection molding (PIM) using carbonyl iron and carbonyl nickel as raw materials. The effect of mixing technique of metal powders on the characteristics of mixed powders and PIM process was studied. It was found that the branched chain of carbonyl nickel was broken and the mixture was homogeneous with a good dispersion by ball milling. Due to the improvement of the powder characteristics, the tap density increased from 3.21 g/cm^3 for mixed powders without ball milling to 4.86 g/cm^3 for ball mixed powders. Additionally, the critical powder loading of the feedstock was raised from 52% for the mixed powders without ball mixing to 62% for the ball milled powders, which benefited dimension precision. As the degreased specimen sintered at 1360 °C in a flowing hydrogen gas, the high magnetic property Fe–50%Ni alloy with maximum permeability of 43541, saturation induction of 1.48 T, coercive force of 6.8 A/m and relative density of 97% was obtained.

1. Introduction

Fe–50%Ni combines both high saturation induction and a relatively high permeability, which is widely used in computer, printer, disk drive components, and motors and so on [1–7]. For the miniaturization and complication of the shape of the magnetic devices in recent years, the conventional fabrication methods of casting and machining are very difficult to realize the mass production due to the secondary operations such as: punching, grinding, honing and drilling, etc. [8]. Powder injection molding (PIM) is a near net shape technique by the introduction of plastic injection molding into powder metallurgy area and has the predominance in the economical mass production of minisize, complex shaped soft magnetic parts [9–13]. Fabrication starts by compounding a thermoplastic binder and powder mixture, referred as feedstock, followed by injection molding, binder removal and sintering to achieve the required properties [14–17].

In the PIM process, the powder characteristics, such as morphology of particles, particle size and particle size distribution, have great effect on the powder loading, rheological behaviour of feedstock and sintering shrinkage. The presence of agglomerated particles can affect the rheological behavior of feedstock and consequently impair the quality of injection molded components [18–20]. In order to obtain a uniform and compact structure of sintered component, extremely pure powder with narrow particle size distribution should be used for the preparation of PIM components. Trunec et al. [21] applied stearic acid which was often used with oxidic ceramic particles as dispersive surfactant acting on the principle of steric stabilization to improve the rheological behaviour of Y-TZP ceramics feedstock. Fan et al. [22] used the ball milling and stearic acid to eliminate powder agglomeration and ultimately increased the powder loading of feedstock and improved compatibility of powder binder.

In this paper, Fe–50%Ni soft magnetic alloys were produced by powder injection molding using carbonyl iron and carbonyl nickel as raw materials. The influence of ball milling process on the characteristics of the mixed powders, injection process, the dimension precision and materials performance of samples was studied. The appropriate binder systems were selected to prepare Fe–50%Ni PIM feedstock and the rheological properties of the resulting feedstock were analyzed. The effect of these treatments on magnetic properties of the samples was investigated.

2. Experimental details

2.1. Powder and binder characteristics

The characteristics of the raw powders including carbonyl iron and carbonyl nickel are shown in Table 1. The morphologies of the two kinds of powder are displayed in Fig. 1. From Fig. 1(a), it can be seen that carbonyl iron is mainly regular spherical particles without severe agglomeration. Carbonyl nickel has branched chain and obvious agglomeration is observed, as shown in Fig. 1(b).

Binder is the core of the PIM technology, which is a multicomponent system with two components: a backbone polymer that provides strength, and a low molecular weight component, which provides high flowability to the feedstock [23,24]. In this study, wax-based binder system was chosen: the paraffin (PW) with low melting point and good flowability as main components, the polymer

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Table 1
Characteristics of the powders.

Powder	Mean particle size/μm	ρ_{spp}/g cm^{-3}	ρ_{tap}/g cm^{-3}	Impurity/wt%		
				C	O	N
YTF-01C Fe	4.33	2.52	3.97	0.63	0.27	<0.001
INCO Ni-123	4.45	2.20	3.00	0.06	0.049	<0.001

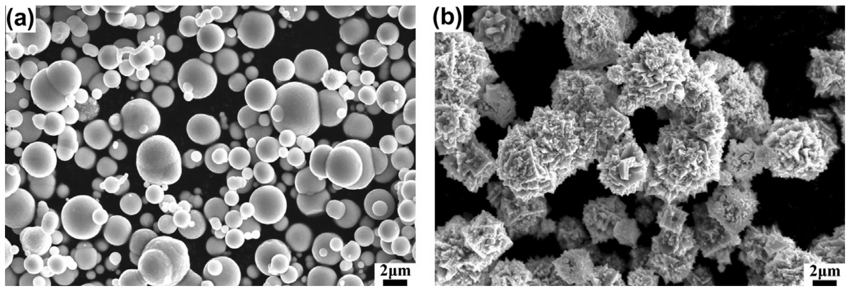

Fig. 1. SEM images of powders: (a) hydroxyl iron; (b) hydroxyl nickel.

polypropylene (PP) and poly styrene (PS) with higher melting point and strong toughness providing sufficient strength as a skeleton material, a small amount of stearic acid (SA) as a surface active agent, playing a bridging effect between the binder and powder particles to prevent the separation of two-phase and ensure the mixing uniformity. The high density polyethylene (HDPE) assures low viscosity of binder but good green strength of pieces due to the crystalline feature of HDPE that confers good mechanical strength. The specific composition is shown in Table 2.

2.2. Processing and experimental techniques

The mixture of carbonyl iron and carbonyl nickel with the weight rate of 1:1 was used as the raw materials. The materials were mixed in a VH-0.2 type mixer under two different conditions: (i) without ball and (ii) the ball-to-powder weight ratio was 2:1. Both of the mixing time was 4 h. The feedstock was prepared by mixing the powders to the binder system using a PSJ32 type mixer at the temperature range of 140–150 °C for 60–90 min. Then the feedstock was injected into ring-shaped samples for magnetic measurements with a CJ-80E type injection molding machine at 150–160 °C. Prior to the sintering process, the binders were removed by solvent debinding and thermal debinding. Subsequently, the debound samples were sintered at the temperature range of 1360 °C for 10 h under hydrogen atmosphere.

An Instron 3211capillary rheometer was used to measure the viscosities of the PIM feedstock. A die with 1.27 mm diameter and 76.20 mm length was used. Observation of the morphology of the starting elemental powder was conducted on S-360 scanning electronic microscope (SEM). Optical microstructure was observed on MeF3A metallurgical microscope. Particle size distribution analysis and specific surface area of mixed powder were studied on laser particle size analyzer (LMS-30). 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 NIM-2000S dc soft magnetic properties measuring device.

Table 2
The components of binder.

PW/wt%	HDPE/wt%	PP/wt%	PS/wt%	SA/wt%
60	15	10	10	5

3. Results and discussion

3.1. Effect of ball milling on particle size distribution

Fig. 2 shows the morphologies of the two mixing ways of powder: powder A and powder B. Using the mixing method without ball (powder A), the morphology of the raw material powder has few changes, as shown in Fig. 2(a). Carbonyl iron and carbonyl nickel still keep their original morphologies respectively and with a high tendency for agglomeration. Using the mixing method with ball (powder B), the branched chain of carbonyl nickel is broken and the mixture is homogeneous with a good dispersion, as displayed in Fig. 2(b). Fig. 3 represent the frequency particle size distributions of powder A and powder B. Comparing to Fig. 3(a), Fig. 3(b) demonstrates a smaller mean particle size and a narrower particle size distribution range. The characteristics of the two mixing powders are given in Table 3. The ball milling process results in disappearance of agglomerated powder and the improvement of dispersity. The values of D10, D50 and D90 changed from 2.40 μm, 6.11 μm and 15.12 μm for the mixed powders without ball milling to 2.02 μm, 4.98 μm and 10.93 μm for the ball mixed powders, which is beneficial for the increase of powder loading.

3.2. Powder loading and dimension precision

Fig. 4 shows the variation of powder loading with feedstock density. For the powder A, the inconsistency between feedstock and theoretical value is 52%, indicating a 52% critical powder loading of powder A. After ball milling, the critical powder loading of powder B is 62%, displaying a 10% improvement than that of powder A.

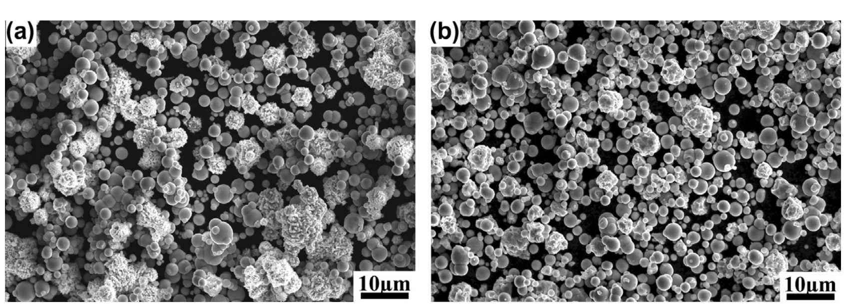

Fig. 2. SEM images of powders: (a) powder A; (b) powder B.