Journal of Magnetism and Magnetic Materials 397 (2016) 240–246

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Effect of Fe_3P addition on magnetic properties and microstructure of injection molded iron

Jidong Ma [a,b], Mingli Qin [a,*], Lusha Tian [a], Lin Zhang [a], Dil Faraz Khan [c], Xiangying Ding [a], Xuanhui Qu [a], Houan Zhang [b]

[a] School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China
[b] School of Materials Science and Engineering, Xiamen University of Technology, Xiamen 361024, China
[c] Department of Physics, University of Science and Technology Bannu, Bannu 28100, Pakistan

ARTICLE INFO

Article history:
Received 22 December 2014
Received in revised form
16 August 2015
Accepted 24 August 2015
Available online 28 August 2015

Keywords:
Fe–P
Powder injection molding
Magnetic properties
Microstructure
Density

ABSTRACT

Phosphorus powder was used to improve the performance of iron based alloy products fabricated by metal injection molding. Seven kinds of Fe–xP soft magnetic alloys were formed using carbonyl iron powder and Phosphorus powder as raw materials where x=0–1.2 wt% with 0.2 wt% increment. Samples were sintered in hydrogen atmosphere at the temperature range of 1100–1450 °C for varied times. The effects of sintering temperature and time on the density, microstructure and magnetic properties like magnetic induction, maximum permeability and coercive force of the alloys were examined. The results demonstrated that better magnetic performances of the injection molded Fe–xP alloy is due to increased density of the sintered compacts because of formation of liquid phase at low temperature. For Fe–0.8%P alloy, optimum density 7.84 g/cm^3 (relative density 99%) and magnetic induction (B_{6000}) 1.77 T, maximum permeability 17,100 were obtained at sintering temperature 1420 °C while the coercive force was 21 A/m respectively.

1. Introduction

Pure wrought iron is an excellent soft magnetic and widely used in magnetic components of electrical instrumentation and dc motor due to its high saturation magnetization; large quantity of ore resources and low environmental impact [1–3]. However, it is expensive and difficult to fabricate parts with complicated shapes because wrought iron has very poor cutting performance. Metal injection molding (MIM) technology is a near-net shaping technique, which has the predominance in the economical mass production of minimize, complex shaped soft magnetic parts [4–8]. The main disadvantage of pure iron fabricated by MIM method is low relative density, which degrades the magnetic properties. Methods such as adding P element to promote the formation of a liquid phase at low temperatures have been employed to provide higher densities to meet the performance as required [1,9,10]. Investigations have been made for the improvement of the magnetic properties and structure of sintered iron materials containing phosphorus addition in the form of Fe_3P [11]. Better magnetic properties are the result of increased density of the sintered compacts due to high temperature sintering in the presence of the liquid phase, the increase in the size of grains and coagulation and spheroidization of pores [12]. However, a variety of research articles based on properties of iron have been published that focus on traditional pressing-sinter technique which use original powders with coarser size. Few researchers have studied the magnetic properties of iron with the addition of P fabricated by MIM.

In this study, the effect of Phosphorus addition to Fe based on the sintering behavior and magnetic properties was investigated by metal injection molding. The aim was to find a correlation between the structure and the magnetic properties of MIM iron sintered compacts admixed with P. The effects of several important processing parameters on the magnetic performance of the Fe with addition of P were reviewed.

2. Experimental details

Carbonyl iron powder having particle size 3–5 μm and Fe_3P powders were used in this research work. Chemical analysis of the Fe_3P powder showed 17.98% P content. Seven kinds of Fe–x%P soft magnetic alloys were formed using carbonyl iron powder and Phosphorus powder as raw materials where x=0–1.2 wt% with 0.2–wt% increment which is shown in Table 1. The powders were mixed in a laboratory through V-type mixer for 4 h. Metal powder

* Corresponding author at: School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 100083, China. Fax: +86 10 62334321.
E-mail address: qinml@mater.ustb.edu.cn (M. Qin).

http://dx.doi.org/10.1016/j.jmmm.2015.08.103
0304-8853/

Table 1
P content in samples.

Designation	*P* additions (%)	*P* content-chemical analysis (%)
P0	0	0.01
P2	0.2	0.21
P4	0.4	0.38
P6	0.6	0.62
P8	0.8	0.81
P10	1.0	1.02
P12	1.2	1.17

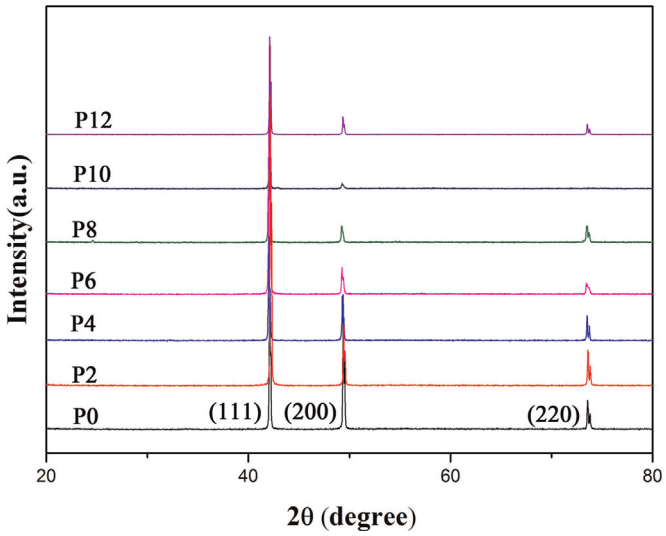

Fig. 1. XDR patterns of different samples.

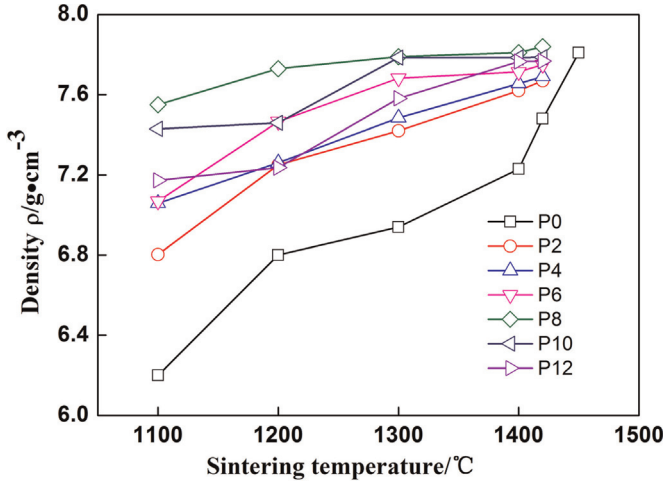

Fig. 2. Effect of sintering temperature on density of sintered samples with different levels of P addition to Fe.

mixture and binder were mixed using a PSJ32 type mixer at the temperature range of 140–150 °C for 60–90 min. The wax-based as binder was composed of 60 wt% paraffin, 15 wt% high density polyethylene, 10 wt% polypropylene, 10 wt% polystyrene and 5 wt% stearic acid. The feedstocks were prepared by mixing the powders with the binder with the powder loading of 58 vol%.

The feedstocks were injected into ring-shaped samples with a 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. Thermal debinding and pre-sintering was performed under hydrogen atmosphere with the top temperature of 800 °C. Subsequently, the debound samples were sintered in hydrogen atmosphere at the temperature range of 1100–1450 °C for varied times.

The compositions of the alloys were determined by a glow discharge spectrometer (Leco, GDS-850A) using sintered bulk specimens. Table 1 shows the seven alloys of different admixtures of P and contents of chemical analysis (%). The phase structure was analyzed by X-ray diffraction (XRD, Rigaku, D/max-RB12) with a Cu–Ka radiation. Fig. 1 shows the XRD patterns of the seven alloys. Only the diffraction peaks of γ–(Fe,P) could be easily identified in these patterns. Optical microstructure was observed on MeF3A metallurgical microscope. The densities of the samples were measured by the Archimedes method. NIM-2000S dc soft magnetic properties measuring device was used to test the magnetic properties such as magnetic induction (B_{6000}), coercive force (Hc) and maximum permeability (μ_m).

3. Results and discussion

3.1. Effect of different P additions on the properties of Fe

Fig. 2 shows the variation of sintered density with sintering temperature for the seven alloys investigated in this study. With the increase of sintering temperature in the range of 1100–1400 °C, the sintered density of the alloys with and without Phosphorus powder increases gradually but the alloys with addition of different wt% P shows better results.

The density of the sintered compacts increases as the P content increases and maximum values are obtained in the range of 0.6–1.2 wt% P. Observing Fig.2, at same sintering temperature 1200 °C the density of alloys with 0.6 wt% P and 0.8 wt% P were achieved 7.47 g/cm^3 (relative density 95%) and 7.73 g/cm^3 (relative density 98%) respectively. The abrupt increase of this density with small amount of P addition at lower sintering temperatures is due to sintering taking place in the presence of liquid phase: the liquid phase is formed at a temperature above the peritectic reaction in the Fe–P equilibrium diagram (1164 °C) [13]. As for soft magnetic, density is the prime factor which influences magnetic induction [14–16]. Thus, the magnetic induction (B_{6000}) increases with the improvement of density. Fig. 3 describes the effect of density on the magnetic induction (B_{6000}) of the sintered specimens with different contents of P, from which, it can be seen the magnetic induction (B_{6000}) of the samples increase as density increased. And as shown in Fig. 4, highest magnetic induction is achieved with 0.8% P content when density is at a maximum. The effect of P content on the maximum permeability and coercive force of the sintered samples are shown in Figs. 5 and 6. The maximum permeability increases while the coercive force decreases gradually with increasing P content. The maximum value of permeability and the minimum value of coercive force are also obtained with 0.8% P content addition. 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, impurities content and grain size [17,18]. Previous studies have shown total solubility of P in a-Fe phase up to 0.8% content. Above this value, P has been found to change in the form of Fe_3P at the grain boundaries and also in the grain [19]. However, except for the patterns of γ–(Fe, P), no distinct diffracted signal arising from the second phase could be observed in XRD patterns, as shown in Fig. 1. In addition, no significant change of lattice was observed, although phosphorus has an atomic radius much smaller than that of iron and the dissolution of phosphorus in iron is substitutional. Fe_3P, as impurity, can pin down or drag grain boundary movement, which contributes further to lower permeability by blocking domain wall motion [20]. Fig.7 shows the microstructures for the alloys with