The preparation and magnetic performance of the iron-based soft magnetic composites with the Fe@Fe₃O₄ powder of in situ surface oxidation

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1. Introduction

Soft magnetic composite (SMC) is a kind of electrical inductance material which is obtained by the easy powder metallurgy (PM) procedure using a ferromagnetic particle coated with a thin electrically insulating layer on the surface [1]. The preparation of SMC contains three steps: 1) preparing the insulating coating of soft magnetic powder; 2) pressing the powder into core; 3) annealing. Compared with the traditional laminated steel core, SMC core possesses unique advantages, such as three-dimensional isotropic magnetic properties, low eddy current loss, low total loss at high frequency and structural freedom for complex designs [2]. Hence, the soft magnetic composites can significantly reduce the mass and volume of the magnetic core [3,4]. However, the addition of coating material reduces the magnetic properties of the SMC such as the permeability and saturation magnetization [5]. Besides, the mechanical properties of the SMC are also insufficient for the high speed vibration in the industry. Therefore, a suitable insulation material with simple preparation process is crucial for SMC to be used in motor to replace the laminated steel. By choosing the appropriate insulation material and optimizing the preparation process, a number of efforts have been made to decrease the core loss of the soft magnetic composite. Insulation coating is divided into two types of organic and inorganic. Organic materials are mostly thermosetting polymers such as silicon epoxy resins [6], acrylics, polyesters, polyurethanes, phenol-formaldehyde resin [7] and so on. These materials have higher resistivity and low eddy current loss, while having the disadvantages of lacking thermal stability, low density and low soft magnetic properties. Inorganic coating is the direction of future development due to the higher thermal stability and enhanced magnetic properties. Phosphates [8–10], MgO [11], SiO₂ [12], and Al₂O₃ [13] are the common inorganic coatings. But these insulating materials introduce many non-magnetic materials during the coating process, which results in a decrease of the saturation magnetization and permeability. To solve the problem of the magnetic dilution, ferromagnetic material such as Mn-Zn ferrites [14], Ni-Zn ferrites [15] and Fe₃O₄ (mainly Fe₂O₃) [16–19] has been regarded as the suitable insulation layer. However, Manganese-zinc ferrite and nickel-zinc ferrite particles are mostly obtained by coprecipitation or mechanical alloying which is difficult to form a uniform coating layer on the iron powder surface. Commonly, the manganese-zinc ferrite and nickel-zinc ferrite are generally applied at very high frequency (MHz, GHz) due to the high electrical resistivity [20]. The SMC is studied to replace
the traditional laminated silicon steel in the frequency of 400 Hz–200 kHz. Hence, magnetite (Fe₃O₄) is the most suitable coated material which serves as a semiconductor.

Heat treatment is another necessary process to both enhance the magnetic properties and improve the binding strength. But heat treatment at high temperature would destroy the coating layer and increase the air gap between the iron particles which would weaken the bonding strength and the magnetic properties. This would result in the low mechanical strength which is a major drawback of the SMC when it is used in a high speed rotor in the high frequency field. In the past studies, the transverse rupture strengths (TRS) were generally used to measure the mechanical properties of the core [21]. Our work explored the hardness and elasticity modulus of the compressed core using a nanoindentation technique at a depth of 200 nm. Therefore, it is important to choose the appropriate heat treatment temperature to avoid decomposition of the Fe₃O₄.

In this paper, we successfully fabricated uniform magnetic insulated coating layer through a simple and controllable method of in situ surface oxidation. Moreover, in order to avoid the negative effects of the organic decomposition in the heat treatment process, no binder is added during the pressing process. A new kind of SMC with very low total core loss and higher mechanical strength has been obtained. Various characterizations were employed to investigate the micro structures and magnetic properties of iron-based soft magnetic composites.

2. Experiments

The reduced iron powder was supplied by Jilin Huaxing Powder Metallurgy Technology Co. Ltd with particle size about 75 µm. 20 g iron powder were weighted into a ceramic crucible, then add 10–20 ml deionized water. The mixture was placed in a muffle furnace for reaction at different temperatures from 150 °C to 300 °C for 10 min. And the zinc stearate (0.5%) served as the lubricants were mixed with the as-prepared powders. The composite powders were compressed at 800–1200 MPa into a ring-like size with an outer diameter of 12.7 mm, inner diameter of 7.6 mm, and height of 3.4 mm. Finally, all the cores were heat treated at different temperatures from 400 °C to 650 °C for 1 h in a N₂ atmosphere. The experimental diagram is shown in Fig. 1.

The phase purity of the synthesized composites was determined by X-ray diffraction (XRD, PANalytical X'Pert PRO) using Cu Kα radiation. The particle morphologies of the powder and the fracture of annealed core were detected by scanning electron microscopy (SEM, Hitachi SU1510). The density and resistivity of the cores were analyzed by the principle of Archimedes and a 4-point method. The initial permeability was measured by LCR meter (Agilent E4980A), while the core loss of the SMC was determined by a B–H analyzer (SY-8218, IWATSU, with less than 0.5% tolerance) at a magnetic excitation level of 0.05 T. The DC magnetic property was recorded using a DC B–H tracer (SK-110, Metron, with less than 0.5% tolerance) under the maximum applied magnetic field of 8000 A m⁻¹. Elastic modulus and hardness were measured by nanoindentation (Nano Indenter G200) at a depth of 200 nm.

3. Results and discussions

3.1. Characterization of the insulating layer

Fig. 2 shows the effects of different oxidation temperature on XRD patterns of the iron powders. The diffraction peaks of the composites at 2θ = 44.582°, 64.939° and 82.258° correspond to the (110), (200), (211) planes of the α-Fe (JCPDS 06-0696). With the increasing of oxidation temperature, we can find additional characteristic peaks at 2θ = 35.300°, 62.585°, which are assigned to the (311), (440) planes of the Fe₃O₄ (JCPDS 03-0863). Besides, there were no other peaks. The slightly increased intensity of the peak corresponding to the Fe₃O₄ phase indicates increased thickness of the coating. Also of importance, our synthesized powders present a obvious color change from silver to black which indicates the Fe₃O₄ coating.

Fig. 3 shows SEM images of irregular Fe powders prepared under different oxidation temperature. With the increasing of temperature, a significant brittle nano layer formed on the surface of the iron powder. Indicating the formation of the Fe₃O₄ insulation layer after surface oxidation according to the XRD patterns above. Moreover, as the temperature gradually increases, the insulation layer becomes thicker.

Fig. 4 shows the fracture surfaces SEM image of the compacted magnetic core. Fig. 4(a) and (b) are the images of the unannealed compacted magnetic core and some flaked materials appears among the particles. Fig. 4(c) and (d) are the fractal surfaces images of the cores annealed at 450 °C. It is obviously that the surface of iron powder core shown in Fig. 4(c) is very smooth. However, the fractal surface of the core compacted by the surface oxidized iron powder at 250 °C still has some brittle nano ceramic among the particles. This is due to the existence of the insulation layer, further confirming the formation of the Fe₃O₄ insulating layer.

Cross-sectional BSE images in Fig. 5 present the interface of the core which is compacted and treated at different temperatures. Compared Fig. 5(b) with (a), the boundary between the iron particles obviously appears at the surface oxidized sample at 250 °C and anneal after 450 °C. These particulates create a well-bonded
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