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Enhanced electromagnetic properties of Fe–Cr–Si alloy powders by sodium silicate treatment

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ABSTRACT

The Fe–Si–Cr alloy powders were successfully modified by using the phosphatizing and sodium silicate coating processes. The optimized surface pre-treatment for Fe–Si–Cr powder was phosphatized in 2 wt.% phosphoric solution and further treated with 4 wt.% sodium silicate solution at 75 °C for 30 min. These treatments increased the high-frequency stability for Fe–Si–Cr alloy powders, and the annealed cores further showed the enhanced performance of permeability, impedance, quality factor and anti-corrosion ability. Comparing with the Fe–Si–Al alloy, the Fe–Si–Cr also showed a superior performance in density, moldability and saturated magnetic induction.

1. Introduction

How to use energy efficiently is always an important issue for our modern life, especially focusing on energy saving and power transforming. For such applications, soft magnetic materials play a key role, and are made into soft-magnetic compound (SMC) cores with a specific isolation gummy. The cores are of a higher resistivity, lower eddy current loss, and applicable in the high-frequency environment, so they are generally applied in our daily life [1,2]. Among various magnetic materials, iron is the most common one of abundant reserve and relatively cheap. Because of its strong magnetism, permeability and saturating flux density, they have already been applied to many industrial facilities, such as motors and convergence transformer [3]. Further, in order to satisfy various requirements, iron alloys with different compositions were proposed [4–9].

Laminated silicon steel and ferrite are two popular core materials. At low frequency, silicon steel owns a high flux density and permeability, but its eddy current loss would reduce seriously as frequency increases. Although ferrite performs as high resistivity and low core loss at high frequency, the low flux density is still its critical problem. In addition, both face the difficulty in the miniaturization of AC facilities. SMC process provides a solution to the product with high permeability and magnetic induction, and of low core loss [10].

In 1931, the researchers began on Fe–Si alloys with different silicon content. A high Si content could result in high resistivity (≈100 μΩ cm), low eddy current loss, good anti-corrosion ability, and high hardness (HV ≈ 500) [11]. Fe–Si–Al alloy, composited of 85% iron, 9% silicon and 6% aluminum, performed the high storage and high-temperature stability, and was also an excellent noise filter due to its high initial permeability [12]. The function of Fe–Si–Cr powder was similar to Fe–Si–Al, and the two stable compounds, CrSi and $CrSi_2$, would form at high temperature, which benefited to high-temperature applications. In this research, a uniform silicon oxide film was benefit, so Fe–Si–Cr alloy was chosen and compared with Fe–Si–Al [11].

Moreover, insulating impedance is an important parameter for electrical appliances, and a higher impedance further presents the better isolation. In order to make sure that electrical devices would work safely, the minimum insulating impedance is regulated for each facility [13]. The Fe–Si–Cr alloy powder is of excellent DC superposition, low power loss, high saturated magnetic induction, high resistivity and high-temperature stability, so it is widely applied to the large current and high power inductances and DC/DC convertor.

In this study, Fe–Si–Cr powders were coated with sodium silicate to form a stable composite material with a good isolating impedance and high permeability. Furthermore, the producing conditions for cores were optimized based on their permeability, quality factor and surface insulating impedance. The physical

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properties of cores were also compared with the Fe–Si–Al alloys to evaluate the improving results.

2. Experimental

The Fe–Si–Cr alloy powders used in this research were provided by Epson Atmix Corp., and cored as PF-20F. The average particle size D50 is approximately 10 μm, and the powder density is 5.18 g/cm^3, and the chemical composition is 92 wt.% Fe–3.5 wt.% Si–4.5 wt.% Cr.

First, the Fe–Si–Cr alloy powders were phosphatized to form a phosphate oxide coating, which would increase the electric resistivity and enhance the quality factor and surface impedance. The phosphatizing treatment was proceeded at 75 °C for 60 min in the phosphoric acid solution of 1, 1.5, and 2 wt.%. Phosphate can protect the Fe–Si–Cr alloy against oxidizing and enhance its adhesion and lubrication [14].

Then, the phosphatized powders were individually mixed with 2, 4 and 6 wt.% sodium silicate solutions at 75 °C for 30 min. Calcium stearate was also added as a release agent to assist the post-treatment. After granulated and forming with the pressure of 460 kg/cm^2 by using a conventional oil hydraulic press, the toroidal cores were obtained. The core size is 13.9 and 8.05 mm for OD and ID. In order to release the inner stress, vacuum annealing treatments were carried out at 450, 550 and 650 °C in a rapid thermal annealing (RTA) oven at 20 °C/min for 30 min.

The morphology was observed by a scanning electron microscope (SEM, JSM-6510) with an energy dispersive spectrometry (EDS, Oxford Inca x-act). The microstructure changes after annealing were verified by X-ray diffraction (XRD, Rigaku DMX-2200). The LCR meter (HP-4285A) was utilized to measure the permeability and quality factor at different frequency. The vibrating sample magnetometer (VSM, DMS1660) was used to examine the magnetic properties of powders or ribbons. The corrosion tests were taken according to ASTM criteria by salt spray tester (TMJ-9701), and the bending tests were examined by a universal test machine (XTJ-STR-A).

Quality factor, so-called "Q value", was used to evaluate the quality of inductance devices, and usually shown as:

$$Q = \frac{1}{\tan\delta} \tag{1}$$

where $\tan\delta$ is the total loss of inductance. The higher Q, the better. The total loss of a working AC inductance can be expressed as [15]:

$$\tan\delta = \tan\delta_h + \tan\delta_e + \tan\delta_r \tag{2}$$

where $\tan\delta_h$ is hysteresis loss, $\tan\delta_e$ is eddy current loss, and $\tan\delta_r$ is residual loss. Eddy current loss is a main loss in the amorphous alloys. The core losses all increase with high frequency.

The inductance and quality factor can be calculated by this formula, and the initial permeability, μ_i, may further be obtained [16]:

$$\mu_i = \frac{L \times (\mathrm{OD} + \mathrm{ID})}{8 \times N^2 \times A} \times 10^3 \tag{3}$$

where L is the inductance of toroidal cores; N is the number of windings; A is the area; OD is the outer diameter of toroidal cores and ID is the inner diameter of toroidal cores.

Table 1
Electromagnetic properties and strength of as-cast Fe–Si–Cr powders.

μ_i (H/m)	Q	Bending strength (kg)	Surface impedance (MΩ)
69.7	14.9	4.6	<1

3. Results and discussion

3.1. Fe–Si–Cr powder characterization

The properties of as-cast Fe–Si–Cr powders were firstly examined. The permeability was 70 H/m; Q value was 14.9; the impedance was lower than 1 MΩ. It showed that the as-cast material was with high permeability. After forming the alloy powders and testing the flexural strength, the bending strength was 4.6 kg at the rate of 5 mm/min. The results were further ordered in Table 1.

Fig. 1 shows the SEM images of Fe–Si–Cr powders for each status, including the phosphatizing and the sodium silicate treatments. In Fig. 1(a), the as-cast powders were glossy, which was unfavorable for the coating of sodium silicate through physical contact. After phosphatized in a 2% phosphoric solution, the powder's appearance became rough and porous, as shown in Fig. 1(b). The phosphate structure could enhance the anti-corrosion ability and improve the adhesion of sodium silicate [17]; additionally, the phosphatized alloy would further reduce its energy loss with a uniform surface coating of sodium silicate. In the other words, a non-uniform coating might result in weak magnetic properties and poor electric isolation. The Fe–Si–Cr powders coated with 2 wt.% sodium silicate solution provided a dense and uniform structure; however, 4 wt.% treatment showed the better result in Fig. 1(c). When the concentration increased to 6 wt.%, the coating structure became rough again (in Fig. 1(d)), which would influence

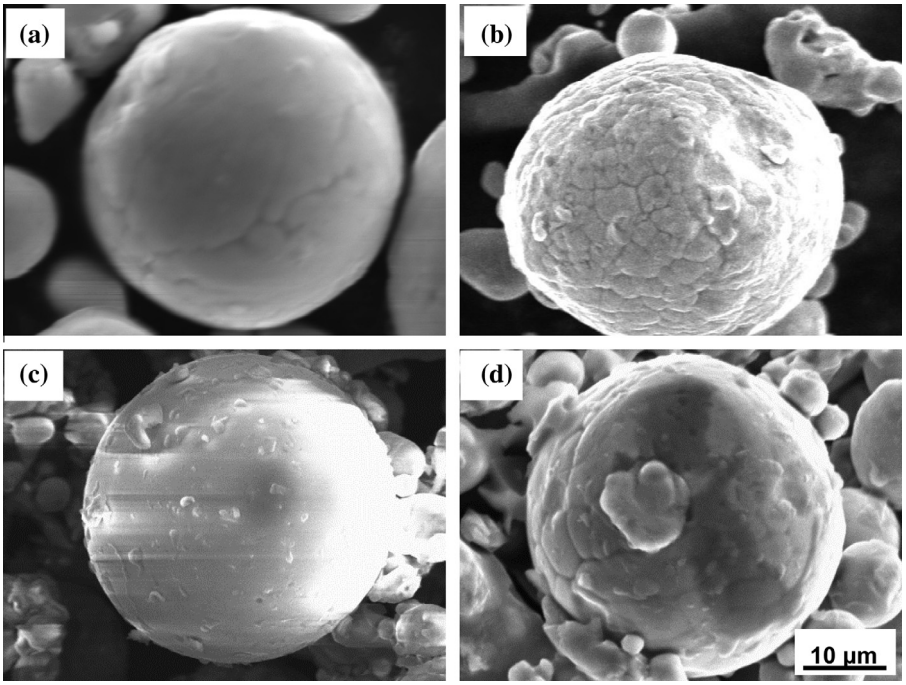

Fig. 1. SEM photos of (a) as-cast and (b) phosphatized Fe–Si–Cr powders, coated with the sodium silicate solutions of (c) 4 wt.% and (d) 6 wt.%.