

## Magnetic Properties of Amorphous FeCrSiBC Alloy Powder Cores Using Phosphate-coated Powders

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(Received 18 August 2006)

The phosphate coating on the  $(\text{Fe}_{0.97}\text{Cr}_{0.03})_{76}(\text{Si}_{0.5}\text{B}_{0.5})_{22}\text{C}_2$  amorphous powders with an average size of 10  $\mu\text{m}$  in diameter has been carried out in aqueous 1.0-2.0 wt%  $\text{H}_3\text{PO}_4$  solutions, and the consolidation behavior and magnetic properties of their compressed powder cores has been investigated. The phosphate coating could provide efficient electrical insulation between amorphous powders and improved consolidation ability at room temperature. Especially when the powders were treated in more concentrated phosphoric acid solution, enhanced phosphate covering and higher frequency/dc-bias stability were achieved. The powder cores phosphate-coated in 2.0 wt%  $\text{H}_3\text{PO}_4$  solution exhibited constant permeability of 21 up to 10 MHz, 110 of the quality factor at 0.9 MHz, 610  $\text{mW}/\text{cm}^3$  core loss at 100 kHz/0.1 T and 89 of percent permeability at 100 kHz.

**Key words :** phosphate coating, high-frequency magnetic properties, Fe-based amorphous alloy powder cores, cold pressing

### 1. Introduction

Powder cores fabricated by compacting the mixture of soft magnetic alloy powders and organic/inorganic electrically insulating materials exhibit low magnetic core loss, unvaried permeability up to high frequencies and high magnetic stability under the superposition of dc-bias field, and are therefore widely used as various chokes, reactors in power factor correction circuits and noise filters etc. [1].

To date, the powder cores have been mostly produced by using iron, permalloy (Ni-Fe) and sendust (Fe-Si-Al) powders [1]. However, with the development of amorphous and nanocrystalline alloys having excellent soft magnetic properties and high electrical resistivity, it has been extensively examined whether more advanced powder-core properties would be obtained or not in these alloys [2-9].

In much earlier study of Hasegawa *et al.* [2], amorphous alloy powder cores were consolidated upon high-temperature pressing with the ribbon-crushed powders (powder size: 180  $\mu\text{m}$ -1.4 mm) insulated by various

oxides. Afterwards Yagi *et al.* [3, 4] have reported mass-production technique for FeCrSiBC amorphous alloy powders and its powder cores insulated with glass binders, where the cores were prepared by cold-pressing and subsequent heating at relatively high temperature below crystallization temperature [3] or hot-pressing to give the adhesion and electrical insulation between amorphous powders (powder size: 50-60  $\mu\text{m}$ ) [4].

Most of the amorphous alloy powders have poor compaction ability at room temperature because of their high strength and lack of plastic deformability. Accordingly hot-pressing method was preferentially used to fabricate amorphous alloy powder cores as was done in previous studies [2, 4, 5], even though cold-pressing at room temperature would be much favorable in economic aspects.

In this study, phosphate coating, in which various phosphates and/or other inorganic materials are chemically deposited in phosphatizing solution on the metallic powder surfaces, has been newly carried out to achieve the enhanced room-temperature consolidation behavior and effective electrical insulation between metallic powders. In addition to that, we have used very small amorphous alloy powders (powder size:  $\sim 10 \mu\text{m}$ ) comparing to those of the previous studies [2-4] to improve the high-

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frequency magnetic properties of the powder cores.

## 2. Experimental Procedure

The gas-atomized  $(\text{Fe}_{0.97}\text{Cr}_{0.03})_{76}(\text{Si}_{0.5}\text{B}_{0.5})_{22}\text{C}_2$  (at%) amorphous alloy powders with an average size of  $10\ \mu\text{m}$  in diameter have been prepared, and then those were immersed in a bath including aqueous solutions with various  $\text{H}_3\text{PO}_4$  concentrations (1.0, 1.5, 2.0 wt%) for phosphate coatings. For activating the reaction between metal surfaces and aqueous solutions, the bath was heated to elevated temperature.

After blending a lubricant of 1 wt% zinc stearine, the powder mixtures were consolidated by cold-pressing at the pressure of 1.8 GPa to form powder cores having inside and outside diameters of 7.2 and 12.6 mm, respectively, and height of about 3.5 mm. Subsequently the compressed powder cores were annealed at  $460\ ^\circ\text{C}$  for 30 min in  $\text{N}_2$  gas atmosphere to relieve the press-induced internal stress.

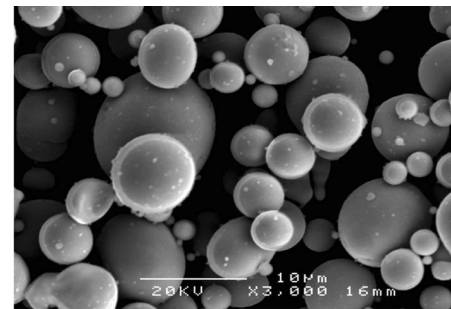
The magnetic properties of the annealed powder cores were measured by using a LCR meter to obtain the data on effective permeability, quality factor and dc-bias properties, and the B-H analyzer was used for core-loss measurement. Further the morphology and microstructure of phosphate-coated amorphous alloy powders were observed and analyzed with scanning electron microscope (SEM) and X-ray diffractometer (XRD).

## 3. Results and Discussion

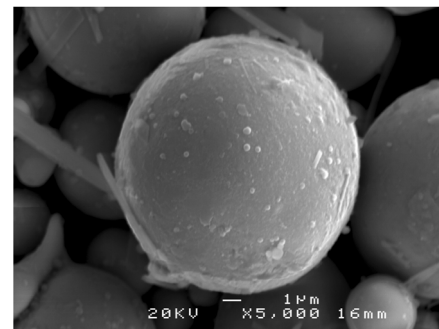
Figure 1 shows the micrographs of the  $(\text{Fe}_{0.97}\text{Cr}_{0.03})_{76}(\text{Si}_{0.5}\text{B}_{0.5})_{22}\text{C}_2$  amorphous alloy powders in as-atomized state (a) and phosphate-coated in aqueous solutions with different  $\text{H}_3\text{PO}_4$  concentrations of 1.0, 1.5 and 2.0 wt% (b-d), respectively. As shown in Fig. 1(b), (c) and (d), a substantially insoluble covering of phosphate crystallization which could provide an electrical insulation were formed on the surface of the amorphous powders after reacting with an acidic solution. Moreover it is notable here that the enhanced phosphate covering was obtained in the solution of higher concentration of  $\text{H}_3\text{PO}_4$ .

In Fig. 2, the X-ray diffraction patterns of the deposits on the metallic powder surfaces are presented. Even though the peak intensity is considerably weak, the surface products are identified to be an iron phosphate  $\text{FePO}_4$  and oxide  $\text{P}_2\text{O}_5$ , and the peak strength was intensified with the increase in  $\text{Fe}_3\text{PO}_4$  contents indicating the enhancement of phosphate covering.

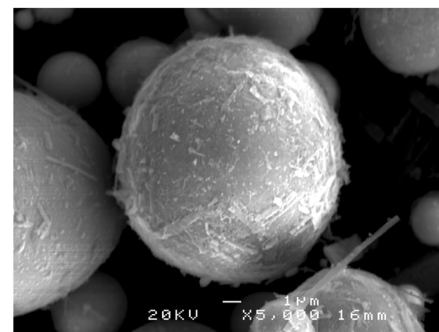
It has been empirically confirmed that the powders coated with an iron phosphate were easily consolidated



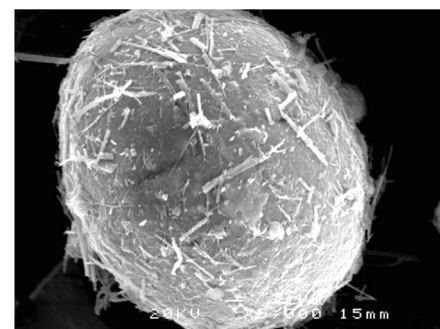
(a)



(b)



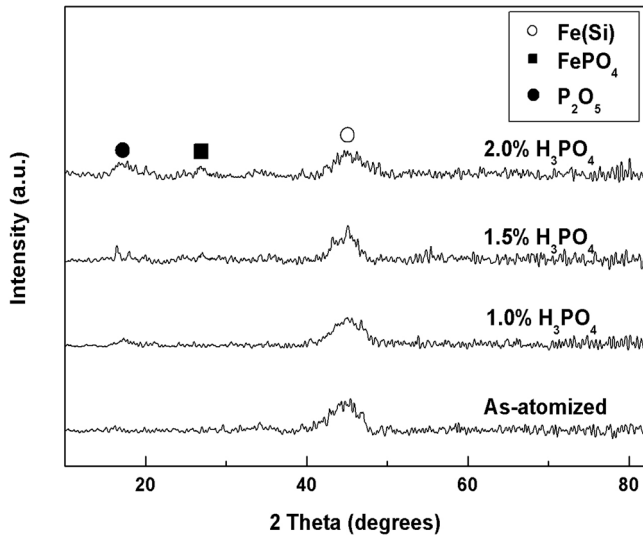
(c)



(d)

**Fig. 1.** SEM micrographs of as-atomized (a) and phosphate-coated  $(\text{Fe}_{0.97}\text{Cr}_{0.03})_{76}(\text{Si}_{0.5}\text{B}_{0.5})_{22}\text{C}_2$  amorphous alloy powders treated in various aqueous solutions with 1.0 wt% (b), 1.5 wt% (c), and 2.0 wt% (d)  $\text{H}_3\text{PO}_4$ .

into magnetic powder cores by pressing at room temperature, as shown in Fig. 3. On the contrary, the room-



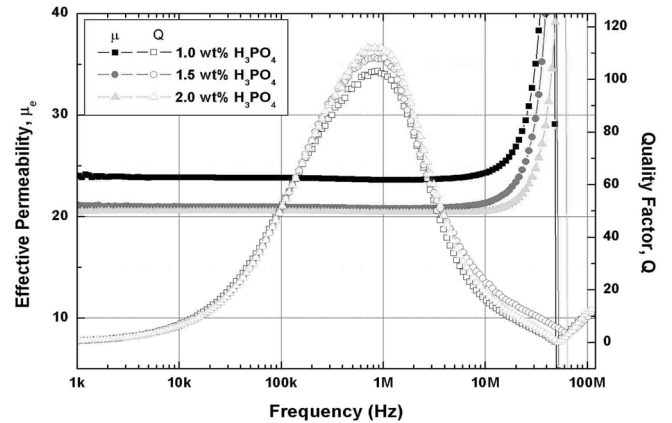
**Fig. 2.** XRD patterns for phosphate-coated amorphous FeCrSiBC alloy powders treated in aqueous solution with 1.0, 1.5 and 2.0 wt% H<sub>3</sub>PO<sub>4</sub>.



**Fig. 3.** The amorphous FeCrSiBC alloy powder cores using phosphate-coated powders prepared by cold-pressing.

temperature consolidation of FeCrSiBC amorphous alloy powders subjected to conventional insulation treatment such as mixing with glass binders was quite unsatisfactory.

Figure 4 shows the frequency dependence of the effective permeability and quality factor for phosphate-coated amorphous alloy powder cores treated in different concentration of phosphoric acid solutions. The powder cores prepared from the concentrated H<sub>3</sub>PO<sub>4</sub> aqueous solution exhibited higher frequency-stability of permeability and larger quality factor. For the powder cores made from 2.0 wt% H<sub>3</sub>PO<sub>4</sub> solution, permeability of 21 persisted up to 10 MHz and 110 of the peak quality factor at 0.9 MHz were attained, whereas the sample coated in 1.0 wt% H<sub>3</sub>PO<sub>4</sub> solution showed permeability of 24 up to 6 MHz and 100 of the quality factor at 0.9 MHz. This persistence of permeability up to very high frequency



**Fig. 4.** The frequency dependence of effective permeability and quality factor for amorphous FeCrSiBC alloy powder cores using phosphate-coated powders treated in various aqueous solutions with 1.0, 1.5 and 2.0 wt% H<sub>3</sub>PO<sub>4</sub>.

could certainly be obtained by the efficient electrical insulation of phosphate covering.

Moreover Yagi *et al.* [3, 4] have previously reported that the amorphous FeCrSiBC amorphous alloy powder cores with an average powder-size of 50-60 μm in diameter had about 110-120 of permeability, and the values were approximately constant in the frequency range up to 1 MHz. Even though the powder cores of this study showed considerably lower permeabilities owing to small size of the powders (~10 μm), the stability of permeability against frequency increase and the quality factor were remarkably enhanced due to the improved skin effect. In this figure, only resonance-type permeability behavior can be observed at the frequency higher than 10 MHz, without any eddy-current induced decrease in permeability.

The lower permeability and the higher cut-off frequency for the powder cores treated in more concentrated phosphoric acid solution were considered to be mainly due to the higher phosphate covering, as shown in Figs. 1-2.

In Fig. 5, the frequency dependence of core loss measured at 0.1 T induction for amorphous alloy powder cores using phosphate-coated powders treated in various aqueous solutions with different phosphoric acid concentration is presented. The core loss was reduced in the entire frequency range with increasing phosphoric acid concentration, and very low value of 610 mW/cm<sup>3</sup> at 100 kHz was obtained from the coating in the 2.0 wt% H<sub>3</sub>PO<sub>4</sub> solution. This low core loss obtained from treatment in higher acidity solution can be definitely ascribed to the more efficient phosphate covering on the whole powder surface than others.

The dc-bias field dependence of percent permeability,

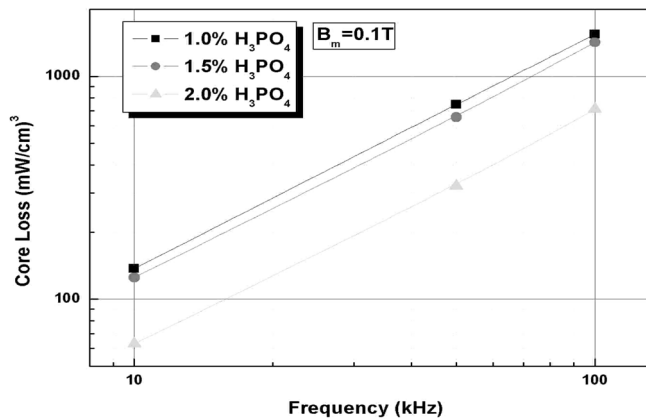


Fig. 5. The frequency dependence of core loss for amorphous FeCrSiBC alloy powder cores using phosphate-coated powders treated in various aqueous solutions with 1.0, 1.5 and 2.0 wt%  $H_3PO_4$ .

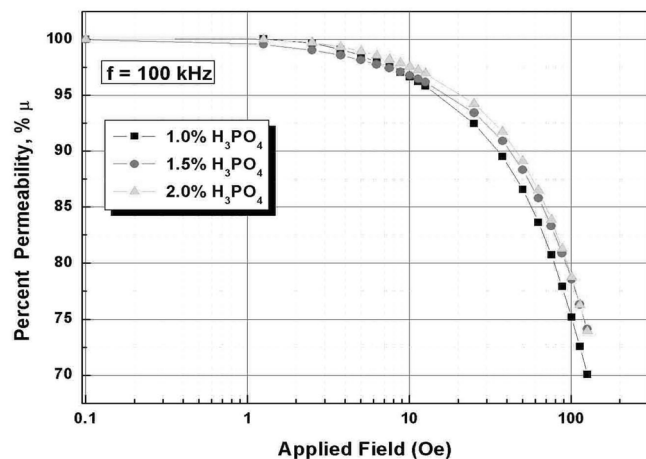


Fig. 6. The variation of percent permeability with dc-bias field for amorphous FeCrSiBC alloy powder cores using phosphate-coated powders treated in various aqueous solutions with 1.0, 1.5 and 2.0 wt%  $H_3PO_4$ .

which is defined by the percentage of the permeability upon dc-bias field to the permeability in free bias field, for amorphous alloy powder cores at 100 kHz is shown in Fig. 6. All the cores have values higher than 86 and 75 at 50 and 100 Oe, respectively, and the largest values of 89 and 78 at 50, 100 Oe were obtained for the powder core using phosphate-coated powders treated in 2.0 wt%  $H_3PO_4$  solution. The higher percent permeabilities of the powder cores coated in more concentrated phosphoric acid solution are also considered to be due to the enhanced phosphate covering, and much larger value of percent

permeability of this study than previous report[3] are certainly attributed to the small size of the used powders.

#### 4. Conclusion

The effects of phosphate coating on the consolidation and magnetic properties of the  $(Fe_{0.97}Cr_{0.03})_{76}(Si_{10.5}B_{0.5})_{22}C_2$  amorphous powder cores with small-sized ( $\sim 10 \mu m$ ) magnetic powders have been investigated.

The phosphate coating could provide efficient electrical insulation between magnetic powders and the phosphate-coated powders were well consolidated into toroidal cores by cold compression. Furthermore, the enhanced phosphate covering was obtained in higher concentration of phosphoric acid solution.

The powder cores phosphate-coated in 2.0 wt%  $H_3PO_4$  aqueous solution exhibited following high-frequency magnetic properties : constant permeability of 21 up to 10 MHz, 110 of the quality factor at 0.9 MHz, 610  $mW/cm^3$  of core loss at 50 kHz/0.1 T and 89 of percent permeability upon 50 Oe at 100 kHz. It has been considered that these results were due to the effective inter-particle insulation by phosphate coating and suppressed eddy current effect of small powders.

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