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Research articles

Fabrication of carbonyl iron powder/SiO_2–reduced iron powder/SiO_2 soft magnetic composites with a high resistivity and low core loss

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ABSTRACT

Carbonyl iron powder (CIP) and reduced iron powder (RIP) were homogeneously coated with an SiO_2 insulating layer by a controlled in-suit chemical deposition procedure and used as raw materials to fabricate CIP/SiO_2-RIP/SiO_2 (C-R) soft magnetic composites (SMCs) by powder metallurgy techniques. Compared with RIP/SiO_2 powders, CIP/SiO_2 powders possess a higher SiO_2 content and more stable Si-O covalent network. The addition of CIP/SiO_2 to the C-R SMCs leads to a significantly increase in the resistivity due to the full electrical isolation of the RIP/SiO_2 particles. Transmission electron microscopy analysis confirms that a SiO_2 amorphous layer approximately 100–400 nm in thickness grows with a high packing density and adheres tightly to the iron grains, resulting in the effective constraint of electron transfer between the Si and O atoms. Annealed C-R SMCs containing 15–20 wt% CIP/SiO_2 have optimum properties with a high resistivity of 4980–6383 μΩ·m and a low core loss of 19.08–23.66 W/kg (50 mT, 100 kHz). The results of this present study provide a significant method to improve resistivity and reduce core loss.

1. Introduction

Recently, soft magnetic composites (SMCs) have been extensively studied as substitutes for laminate steels in view of their attractive properties for electromagnetic applications, such as electrical coils, sensors and stators [1]. SMCs show unique 3D magnetic and electrical properties such as good permeability, high magnetic saturation and high electrical resistance, and the isotropy of the properties removes the design constraints imposed on conventional electrical machines by lamination [2,3]. The high resistivity is a major reason these materials are attractive for low-loss applications, in particular at medial and high frequencies, and is a benefit for the maximum power rating, running costs and environment [1–5].

SMC materials consist of high-purity iron powder surrounded by an electrical insulating layer to prevent the formation of an eddy current between adjacent Fe particles. In the past decades, various insulating materials have been used to coat powder particles and reduce core loss, including organic resins, phosphates, ZrO_2, MgO, Al_2O_3, ferrites, and silica [6–15]. Because of their low thermal resistance, organic resins and phosphates cannot endure high-temperature annealing, leading to destruction of the insulating layer and sharp deterioration in the electrical and magnetic properties [6,7]. An inorganic ZrO_2, MgO and Al_2O_3 nanoparticle layer offers a high thermal resistance, but the intrinsic brittleness and thermal stress generated by the difference in the thermal expansion coefficients of these materials and iron deteriorate the magnetic properties [8–10]. On the other hand, ferrites provide a high electrical resistivity and suitable magnetic properties, but nano-scale ferrite particles cannot form a uniform layer on the surface of iron particles, leading to increased core loss [11,12].

A silica insulating layer has long been considered an optimum insulating material for iron-based SMCs and has been investigated by many researchers [13–18]. SiO_2 has a large, 3D network connected by strong Si–O covalent bonds, and electrons can be effectively constrained between silicon and oxygen atoms, contributing to a high resistivity of approximately 6.0×10^{12} μΩ·m [19]. The resistivity of SiO_2 decreases as the thickness decreases to a thin nanoscale layer [20], while an increase in the thickness would reduce the magnetic flux density due to the increased volume of the non-magnetic phase. The resistivity for Fe/SiO_2 SMCs is reported so far not higher than 500 μΩ·m [13–15,17]. To solve this problem, we propose a promising route to remedy these drawbacks by a rational design of Fe/SiO_2 SMCs and to further upgrade their electrical and magnetic properties. Herein, fine carbonyl iron powder (CIP) and coarse reduced iron powder (RIP) were homogeneously coated with SiO_2 insulating layers with different thicknesses. The thick and stable SiO_2 network on the CIP surface leads to a significantly high resistivity. Then, the as-synthesized CIP/SiO_2 particles were used as intergranular insulating fillers to fabricate RIP/SiO_2-CIP/SiO_2 (C-R) SMCs by powder metallurgy techniques. A high

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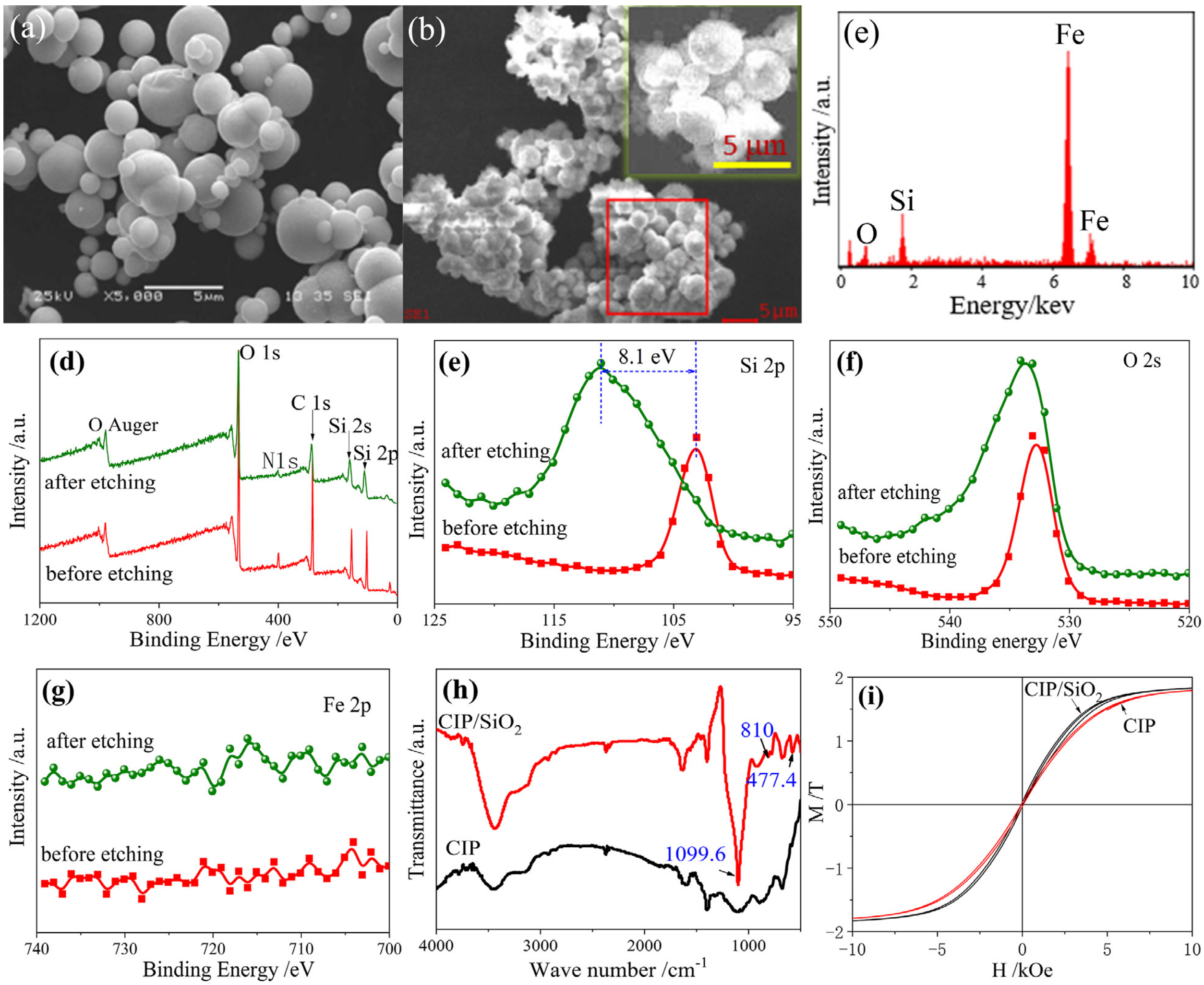

Fig. 1. (a) SEM image of the raw CIP powder; (b) and (c) SEM image and EDS analysis of CIP/SiO_2; XPS survey spectra (d), Si 2p (e), O 1 s (f) and Fe 2p (g) of the CIP/SiO_2 powders before and after 500 s of etching; (h) FTIR analysis; (i) Magnetic hysteresis loops.

resistivity of approximately 6383–105,000 μΩ·m is obtained, and the resulting SMCs exhibit low core losses. We believe that a rational design of SMCs may present a new route for the fabrication of high-performance SMCs.

2. Experimental

2.1. Materials

The raw materials included spherical carbonyl iron powders (CIPs) with an average particle size of 2 μm and reduced iron powders (RIPs) with a size of approximately 100 μm supplied by Hotline Electronics (Shen Zhen) Co. 3-Triethoxysilypropylamine (APTES), tetraethyl orthosilicate (TEOS), ammonia (NH_4OH) and ethanol (C_2H_5OH) were purchased from Tianli Chemical Reagent Co. Deionized (DI) water was prepared in our laboratory by an automatic double pure water distillatory.

2.2. Synthesis of CIP/SiO_2 and RIP/SiO_2 core-shell powders

In a typical chemical coating procedure, 30 g of CIP, 1 ml of APTES and 10 ml of DI water were dispersed in 190 ml of C_2H_5OH by mechanical stirring for 1 h. Then, 45 ml of NH_4OH and 9 ml of TEOS were introduced and vigorously stirred for 4 h at 40 °C until the reaction was complete. After washing with ethanol several times, the as-synthesized CIP/SiO_2 powders were dried at 60 °C for 2 h in air and heated at 500 °C for 1 h in hydrogen to remove residual organic species. The RIP/SiO_2 core-shell powders were synthesized via the same method, but the additions of NH_4OH and TEOS were 15 ml and 5 ml, respectively. The chemical reaction can be expressed by the following formulas [21]:

$$Si(C_2H_5O)_4 + 4H_2O = Si(OH)_4 + C_2H_5OH$$

$$Si(OH)_4 = SiO_2 + H_2O$$

2.3. Fabrication of the SMCs

The RIP/SiO_2 and C-R SMCs were fabricated by powder metallurgy techniques. The SMCs based on RIP/SiO_2 core–shell powders were first moulded into a toroidal-shape compact with a 20.3 mm outer diameter, 12.7 mm inner diameter and 6.35 mm thickness under 600–1000 MPa and annealed at 600–1000 °C for 1 h in a N_2 atmosphere to investigate the optimum manufacturing parameters. After that, the RIP/SiO_2-based C-R SMCs with 5–30 wt% CIP/SiO_2 core-shell powders were pressed under 600 MPa and annealed at 600 °C for 1 h in a N_2 atmosphere. To investigate the effect of the insulating coatings on the electrical and magnetic properties, the raw RIP core without SiO_2 coatings was also prepared under the same conditions as the C-R SMCs.

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