Journal of Alloys and Compounds 691 (2017) 165–170

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Synthesis and magnetic properties of core-shell structured Finemet/Ni–Zn ferrite soft nanocomposites by co-precipitation

Xin Peng, Kun Peng*, Jianping Huang

College of Materials Science and Engineering, Hunan University, Changsha, 410082, China

ARTICLE INFO

Article history:
Received 29 June 2016
Received in revised form
12 August 2016
Accepted 13 August 2016
Available online 18 August 2016

Keywords:
Toroid cores
Co-precipitation
Magnetic properties
Core loss

ABSTRACT

Finemet/Ni–Zn ferrite core-shell particles and toroid core have been synthesized by co-precipitation method combined with following compact pressure and high temperature sintering process. The toroid core were produced from these particles by mold compacting with a compact pressure of 1400 Mpa at room temperature, then sintered at 600 °C for 1 h. The SEM results indicated that a layer of ferrite was uniformly coated on the surface of Finemet particles, with the increase of ferrites thickness, the permeability, saturation magnetization and magnetic loss of toroid core decrease, and the toroid core with 5 wt% ferrites shows excellent soft magnetic properties. It exhibits a high permeability of 56, high saturation magnetization of 127.1 emu/g and lower core loss of about 1.51 w/kg at 100 kHz and 50 mT.

1. Introduction

With the rapid development of microelectronic and magnetic-electronic devices, the development of magnetic core materials with excellent magnetic performance have became one of key issues to be solved in this field. Soft magnetic materials are being generally used in the field of transformers, electrical motors and instrument equipments [1,2]. And among various magnetic materials, metallic materials exhibit high saturation magnetization, high permeability but low electrical resistivity, which causes high eddy-current loss particularly at high frequencies. In contrast, ferrites can be used for higher frequency due to high electrical resistivity, but its saturation magnetization is low. Thus soft magnetic composites (SMCs) [3] consisting of magnetic particles surrounded by an electrical insulating, which have attracted considerable attentions for their good thermal stability, high permeability and low eddy current loss.

Till now researchers have adopted many methods to coat oxide insulating layer on the alloy particles, such as sol gel method [4], thermal oxidation [5], co-precipitation route [6] to improve resistivity and decrease the loss. The insulating layer including SiO_2 [7], Al_2O_3 [8] and MgO [9], NiO [10], these oxides coatings have some drawbacks, low content of insulating layer will insufficient to suppress eddy current loss [11], but a larger amount of insulator will reduce the saturation magnetization owing to the insulating layer are not ferromagnetic. Therefore compatible thickness core-shell structured magnetic particles with relatively higher saturation magnetization and stability in air are greatly expected. Ferrites used as shell material for its high resistivity and ferrimagnetic properties have been coated on metal and alloy powders particles, Schönlein M et al. [12] synthesis Fe/ferrite shell-core composites by conventional ceramic method as insulating layer, suggesting the composites have low core loss less 250 Ω/H than the raw core about 800 Ω/H and have high stability of permeability with increase of ferrite, other scholars [13,14] adopt different methods to coat ferrites on the surface of metal and alloy powders, the results confirmed that the ferrites was coated on core obviously increase electrical resistivity, decrease core loss and improve stability of permeability especially in high frequency. In general, iron powders are the most used ferromagnetic materials for its lower cost and high saturation magnetization, while it still high magnetic loss originating from a broad hysteresis. FeSiBNbCu (Finemet) alloy have low coercivity, high saturation magnetization and high permeability for a given application, but they cannot applied high frequency for its eddy loss. In order to improve comprehensive magnetic properties, Finemet/ferrite shell-core composite maybe obtain excellent magnetic properties. However, the key factors such as the coating amount, thickness of ferrites coats effects are still defective in the above research works. Thus, it is reasonable and necessary to do more systemic investigations about the factors

* Corresponding author. Tel.: +86 731 88822663.
E-mail address: kpeng@hnu.edu.cn (K. Peng).

http://dx.doi.org/10.1016/j.jallcom.2016.08.102
0925-8388/

determining the performances of produced SMCs.

In this paper, a layer of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ with various thickness were coated on the surface of Finemet prepared by chemical co-precipitation method and their structure, magnetic properties were investigated.

2. Experimental method

2.1. Material and reagents

All chemicals used were of analytical grade, used as received without further purification. The commercial atomized FeSiBNbCu alloy powders with average size about 25 μm were provided from Jiayang Electronic Technology Co., Ltd. Analytical grade reagents ($FeCl_3 \cdot 6H_2O$, $ZnCl_2$, $NiCl_2 \cdot 6H_2O$) were purchased from Sinopharm Chemical Reagent Co.,Ltd, Demonized water (H_2O) was prepared by Automatic Double Pure Water Distillatory (SZ-93A, China) in laboratory.

2.2. Preparation of Finemet/$Ni_{0.5}Zn_{0.5}Fe_2O_4$ particles

Finemet powders were put into demonized water and stirred at 200r/min, then the analytical grade $FeCl_3 \cdot 6H_2O + ZnCl_2 + NiCl_2 \cdot 6H_2O$ in different content to Finemet powders were added, respectively. And precipitate agent of NaOH was added at 5 ml/min into the mixtures. During the treatment, the bath was continuously stirred at 200r/min for 60 min to expose each particle in the solution in order to uniformly deposited precipitation on the surface of Finemet, the composites particles were ultrasonic cleaning in ethanol and then heated at 60°Cfor 24 h to dry the particles in drying cabinet.

2.3. Preparation of Finemet/$Ni_{0.5}Zn_{0.5}Fe_2O_4$ toroid core

Finemet/$Ni_{0.5}Zn_{0.5}Fe_2O_4$ core-shell particles were pressed into a toroidal core compact with dimensions of 13.1 mm in outer diameter, 7.8 mm in inner diameter and 5.5 mm in thickness (Φ 13.1 × Φ7.8 × h 5.5 mm) at 1400 Mpa at room temperature, then sintered under 600 °Cfor 1 h. To investigate the effect of insulating coatings on magnetic properties, the raw Finemet core without ferrite coatings were also prepared under the same conditions.

2.4. Characterization

Differential scanning calorimeter (DSC) was used to investigate the phase transformation with heating rate of 10 k/min at air atmosphere. The phase identification and surface analysis of the particles were characterized by X-ray diffraction (XRD, Siemens D5000 using Cu-Ka radiation) and scanning electron microscopy SEM (FEI Quanta 200) coupled to an energy dispersive analyzer (EDS). The hysteresis loop was analyzed by a superconducting quantum interference device (SQUID Quantum Design PPMS EverCool-II) at external magnetic field up to 10 kOe. The permeability was measured by LCR meter and the magnetic losses were measured at 25 KHz–150 KHz by an AC B-H analyzer, all the measurements were performed at room temperature.

3. Results and discussion

3.1. Microstructure of composites particles

Fig. 1 shows the XRD spectra for composites particles before and after annealing at 600 °Cfor 1 h. There is an obvious amorphous diffraction peak corresponding to the diffraction peak of Finemet alloy before annealing, which suggested that the raw Finemet particles are mainly in amorphous state, and the diffraction peaks of α-Fe(Si) phase and NiZn ferrites (JCPDS# 52-0278) can be found in the products after heat treatment. The XRD analysis results indicated that the Finemet alloys have been crystallized and NiZn ferrite have formed after heat treatment.

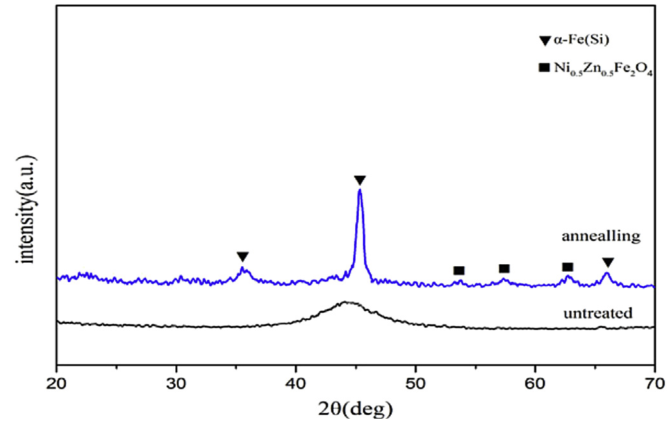

Fig. 1. X-ray diffract gram of Finemet/$Ni_{0.5}Zn_{0.5}Fe_2O_4$ with untreated and annealing at 600 °C.

Fig. 2 shows the SEM images of the raw Finemet particles and the corresponding composite particles after chemical coating process. The raw Finemet particles before chemical coating process are relatively clean and smooth as shown in Fig. 2 (a), while they tend to be rougher after co-precipitation coating process as shown in Fig. 2 (b). The high-magnified image in Fig. 2(c), indicating the particles are uniformly coated by a layer of co-precipitation products. These composites particles were compacted into toroid core, the cross profiles of the core is characteristiced by SEM, Fig. 3 shows the microstructure of the polished surface for the toroid core with 5 wt% ferrite, it can seen that each finemet particle are coated a layer of ferrites.

In order to investigate the thickness of coating layer on the particles, **t**he cross profiles of composite particles are examined, the composites particles mixed with bakelite powder, and then pressed the mixtures to obtain the sheet samples, through polished the samples, the cross profiles have been obtained. The SEM results are shown in Fig. 4, it can be clearly observed that the composites particles have a core-shell structure, Finemet alloy particles are enclosed by a layer of ferrites, and the thickness of the shell increase with adding ferrite content. However, only particles are cut in the center, the correct layer thickness of the composites particles can be obtained by SEM images, otherwise the thickness is always larger than actual thickness [15], due to it is difficult to precisely control the central position in the work, so the observed thickness of layer has some lager than actual thickness. These results indicated that the ferrites are all coated on the surface of Finemet, the EDS results also indicated that shell and core are corresponding to NiZn ferrite (7 wt%) and Finemet alloy powders as shown in Fig. 5, which suggesting core-shell Finemet/Ni–Zn ferrite composite particles have formed. and the formation of shell-core structure can be explained by mechanism of heterogeneous nucleation forming process.

A schematic representation of the experimental layout carried out to obtain the composites is presented. The ferrites were formed through the chemical reacting process and the process of reaction can be presented roughly as follows:

$$Fe^{2+} + M^{2+} + 8ROH \rightarrow M(OH)_2 + Fe(OH)_3 + 8R^+ \quad (1)$$