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Journal of Magnetism and Magnetic Materials 292 (2005) 304–309



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# A study of NiZnCu-ferrite/SiO<sub>2</sub> nanocomposites with different ferrite contents synthesized by sol–gel method

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Received 15 October 2003

Available online 2 December 2004

## Abstract

Ni<sub>0.65</sub>Zn<sub>0.35</sub>Cu<sub>0.1</sub>Fe<sub>1.9</sub>O<sub>4</sub>/SiO<sub>2</sub> nanocomposites with different weight percentages of NiZnCu-ferrite dispersed in silica matrix were successfully fabricated by the sol–gel method using tetraethylorthosilicate (TEOS) as a precursor of silica, and metal nitrates as precursors of NiZnCu ferrite. The thermal decomposition process of the dried gel was studied by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). The obtained Ni<sub>0.65</sub>Zn<sub>0.35</sub>Cu<sub>0.1</sub>Fe<sub>1.9</sub>O<sub>4</sub>/SiO<sub>2</sub> nanocomposites were characterized by X-ray diffraction (XRD), transmission electron microscope (TEM), scanning electron microscope (SEM), Mössbauer spectroscopy and vibrating sample magnetometry (VSM). The formation of stoichiometric NiZnCu-ferrite dispersed in silica matrix is confirmed when the weight percentage of ferrite is not more than 30%. Samples with higher ferrite content have small amount of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The transition from the paramagnetic to the ferromagnetic state is observed as the ferrite content increases from 20 to 90 wt%. The magnetic properties of the nanocomposites are closely related to the ferrite content. The saturation magnetization increases with the ferrite content, while the coercivity reaches a maximum when the ferrite is 80 wt% in the silica matrix.

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PACS: 75.50.Gg; 75.50.Tt

Keywords: Nanocomposites; NiZnCu-ferrite; Sol–gel; Mössbauer spectra; Magnetic properties

## 1. Introduction

Magnetic nanosized particles have been actively investigated due to their significant differences from their equivalent bulk materials in physical, chemical and electromagnetic properties and their

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potential application in recording medium, information storage and magneto-optical devices [1].

Many synthetic approaches [2] such as chemical coprecipitation, ball milling, hydrothermal synthesis and sol–gel technique have been employed to prepare magnetic nanoparticles. However, the nanocrystals obtained usually have a strong tendency to aggregate, which makes it difficult to exploit their unique properties.

To reduce the unwanted crystallite coarsening and particle aggregation, attempts have been made to synthesize nanocomposites by embedding nanoparticles in a suitable matrix. Different systems such as  $\text{NiFe}_2\text{O}_4$  [3],  $\text{ZnFe}_2\text{O}_4$  [4],  $\text{Fe}_2\text{O}_3$  [5],  $\text{CoFe}_2\text{O}_4$  [6],  $\text{NiZn}$ -ferrite [7] dispersed in an insulating matrix like silica have been fabricated by ball milling, sputter deposition and sol–gel methods.

In this work,  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Cu}_{0.1}\text{Fe}_{1.9}\text{O}_4/\text{SiO}_2$  nanocomposites with different contents of  $\text{NiZnCu}$ -ferrite dispersed in silica matrix were first synthesized by sol–gel method. The thermal decomposition behavior of the dried gel was investigated. The structural and magnetic properties of the resultant nanocomposites were investigated, for different ferrite contents.

## 2. Experimental procedure

$\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Cu}_{0.1}\text{Fe}_{1.9}\text{O}_4/\text{SiO}_2$  nanocomposites with different weight percentages of ferrite ranging from 20% to 90% in silica matrix were prepared by sol–gel method. Analytical grade nickel nitrate, zinc nitrate, copper nitrate and iron nitrate were weighted according to the required stoichiometric proportion and dissolved in deionized water. Then tetraethylorthosilicate (TEOS) and ethanol (EtOH) were slowly added into the aqueous solution of mixed metal nitrates. The TEOS/EtOH/ $\text{H}_2\text{O}$  molar ratio was fixed at 1:4:6 while the  $\text{NiZnCu}$ -ferrite/( $\text{NiZnCu}$ -ferrite +  $\text{SiO}_2$ ) weight ratio was controlled by varying the amounts of metal nitrates and TEOS. The precursor solution was stirred for 2 h when hydrolysis of TEOS took place. The sol was left to rest and allowed to gel at 50 °C for 48 h and then heated at 110 °C for 12 h to obtain a dried gel.

The dried gel was annealed at 750 °C for 2 h in the air to obtain nanocrystals of  $\text{NiZnCu}$ -ferrite dispersed in the silica matrix.

The thermal decomposition behavior of the dried gel was examined by means of thermogravimetric analysis (TGA) and differential thermal analysis (DTA) with a heating rate of 10 °C/min in the air on the Perkin–Flamer TGA-7 and DTA-7 instruments, respectively. Phase identification was carried out using Rigaku D/max 2500 PC X-ray diffraction (XRD) with  $\text{Cu-K}\alpha$  radiation. Transmission electron microscope (TEM) examination of the synthesized nanocomposites was performed using JEOL JEM2010 electron microscope. Scanning electron microscope (SEM) was carried out in a FEL XL30 ESEM FEG microscope.  $^{57}\text{Fe}$  Mössbauer spectra were measured at room temperature by an Oxford MS-500 constant source, the velocity was calibrated with an  $\alpha$ -Fe foil. The magnetic measurement for synthesized nanocomposites was taken on a ADE DMS-HF-4 vibrating sample magnetometer (VSM) in an applied field of 16 kOe.

## 3. Results and discussion

### 3.1. Thermal decomposition behavior of dried gel

Fig. 1 shows the typical curves of TGA, differential thermogravimetry (DTG) and DTA

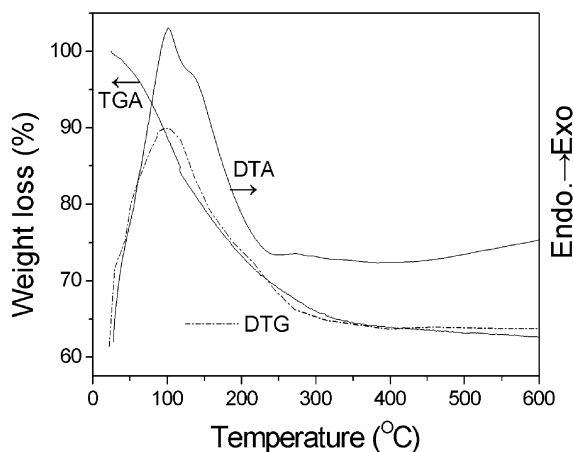


Fig. 1. The DTA/TGA/DTG curves of the dried gel.

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