

Short communication

# Effects of annealing on phase evolution, microstructure and magnetic properties of mechanically synthesized nickel-ferrite

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Received 2 March 2010; received in revised form 6 March 2010; accepted 26 April 2010

Available online 25 June 2010

## Abstract

The influence of milling and subsequent annealing on nickel-ferrite phase formation was investigated by X-ray diffraction (XRD). Microstructure and magnetic properties of  $\text{NiFe}_2\text{O}_4$  were determined by field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and vibration sample magnetometry (VSM). Single phase nanosized nickel-ferrite was obtained by 30 h mechanical alloying (MA) and subsequent annealing at 600 °C for 1 h. Magnetic properties of the milled powder were extensively affected by the annealing temperature. Considerable growth of the particles and necking by sintering resulted from annealing at 1000 °C.

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**Keywords:** A. Powders; solid state reaction; C. Magnetic properties; D. Ferrites; E. Soft magnets

## 1. Introduction

Synthesis of ultrafine nickel-ferrite ( $\text{NiFe}_2\text{O}_4$ ) particles has been extensively investigated during recent years [1–5]. Nickel-ferrite nanoparticles present a wide range of applications requiring low microwave-number and from low to high permeability such as electronic devices [6], ferrofluids [7], magnetic drug delivery [8] and high density information storage [9]. Magnetic properties depend substantially on the synthesis route of nickel-ferrite. Production of  $\text{NiFe}_2\text{O}_4$  nanoparticles has been practiced via several techniques like chemical co-precipitation [10], sol–gel [11], shock wave [12], microwave processing [13] and aerosolization [14].

High-energy ball milling of metallic oxides is a simple solid state technique for the production of nanosized ferrite powder exhibiting unusual properties [15–19]. During MA, some magnetic properties can be improved, while others deteriorate due to stress, strain and defects introduced by milling. Residual

stress/strain elimination by annealing is required to improve the magnetic properties [18,20].

In this study, the effect of annealing temperature on phase evolution, morphology, particle size and magnetic properties of nano-sized nickel-ferrite powders synthesized by mechanical alloying at room temperature have been investigated.

## 2. Materials and experimental procedure

Pure powders of  $\text{Fe}_2\text{O}_3$  (Merck, Gmbh, 99 wt%) and NiO (Sigma, USA, 99.99 wt%) were mixed at a molar ratio of 1:1. The mixture was loaded into a steel vial together with steel balls of 10 and 15 mm diameters. Ball to powder weight ratio (BPR) was 15:1. Milling was continued for up to 30 h with running speed of 300 rpm. The as-milled sample was annealed at different temperatures according to: (1) heating up to various temperatures with a rate of 10 °C/min in air, (2) dwelling for 1 h within the furnace, (3) cooling inside the furnace to a lower temperature (~400 °C) and (4) cooling down to room temperature. Phase characterization of the milled/annealed powder was determined by X-ray powder diffraction analysis (XRD, Philips, PW 3710) with Cu-K $\alpha$  radiation. Morphology and size of the particles were determined by field emission

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