

Original Research

# The effect of neodymium substitution on the structural and magnetic properties of nickel ferrite

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## Abstract

Nickel ferrite  $\text{NiFe}_2\text{O}_4$  is a typical soft magnetic ferrite with high electrical resistivity used as high frequency magnetic material. Neodymium ( $\text{Nd}^{3+}$ ) doped  $\text{NiFe}_2\text{O}_4$  materials were fabricated using solid state reaction. The properties of the obtained material were investigated by X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), Fourier-Transform Infrared Spectroscopy (FT-IR), magnetic measurements on SQUID and Mössbauer spectroscopy. It was found that the material consists of two different phases:  $\text{Nd}^{3+}$  doped  $\text{NiFe}_2\text{O}_4$  and  $\text{NdFeO}_3$ . The  $\text{Nd}^{3+}$  ions occupy cation sites of the  $\text{NiFe}_2\text{O}_4$  inverse spinel structure.  $\text{NdFeO}_3$  phase occurred when the level of  $\text{Nd}^{3+}$  atoms exceed a percolation limit. The presence of both phases was confirmed by SEM observations. The Mössbauer spectra analysis showed two sextets, which can be ascribed to iron atoms in tetrahedral and octahedral positions. From their intensities it is concluded that  $\text{Nd}^{3+}$  occupies octahedral sites in the spinel structure of  $\text{NiFe}_2\text{O}_4$ , which were originally occupied by  $\text{Ni}^{2+}$ .

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## 1. Introduction

The spinel ferrites constitute an important group of magnetic materials both from the fundamental and applied reasons. Generally, spinel ferrites are described by a stoichiometric formula given as  $\text{MeFe}_2\text{O}_4$ , where Me stands for metal or metallic elements with 2+ valence state. These materials crystallize adopting the cubic spinel structure ( $Fd3m$  space group) [1,2]. Within the spinel cubic crystal structure two crystallographic non-equivalent cation sites are recognized, i.e., tetrahedral (T) sites and octahedral (O) sites. In the normal spinel structure, the T sites are occupied by divalent metal

atoms and  $\text{Fe}^{3+}$  locates on the O sites. However, in some cases, divalent metal atoms can replace half of  $\text{Fe}^{3+}$  atoms on the O sites and thus, the T sites are completely filled with  $\text{Fe}^{3+}$  atoms, i.e.,  $(\text{Fe}^{3+})_T(\text{Me}^{2+}\text{Fe}^{3+})_O\text{O}_4$ . Me is most frequently Zn, Cu, Co, Ni etc. and it is well known that Ni, Cu, and Co ferrites are materials with inverse spinel structure, while Zn ferrite is a normal spinel [3–6].

The nickel ferrite  $\text{NiFe}_2\text{O}_4$  is a ferrimagnetic material when T sites and O sites form magnetic sub-lattices [1,7]. In the  $\text{NiFe}_2\text{O}_4$  unit cell,  $\text{Fe}^{3+}$  ions occupy 8 tetrahedral and 8 octahedral crystallographic sites, i.e., it exhibits the inverse spinel structure [8]. It has been shown that the calculated spin moment of Fe ions at T sites is  $3.36 \mu_B$ , while average moment for the O sites is  $2.45 \mu_B$  [9]. Thus, the net moment is oriented in the direction of the T sub-lattice magnetization.

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To synthesize  $\text{NiFe}_2\text{O}_4$ , several preparation routes have been proposed including co-precipitation [3,10], combustion synthesis [11,12], the reverse micelle technique [13], electro-spinning [14], thermal decomposition [15–17], synthesis by thermal plasma [18], high-energy milling [19,20], sol–gel method [21,22], microwave-hydrothermal method [23], and the solid-state method [24]. This broad spectrum of syntheses has several advantages and drawbacks. In particular, if the preparation of nickel ferrite by the solid state reaction or by co-precipitation is chosen, high temperatures (600–800 °C) are required in order to yield good crystallinity powder [3,24]. On the other hand, during the sol–gel method, there is a possibility to control either stoichiometry or size distribution of nanoparticles [21].

It seems very interesting to investigate the effect of Fe substitution by the rare earth ions (RE) on structural, magnetic, and electric properties of  $\text{NiFe}_2\text{O}_4$ . The existence of unusual properties of doped nickel ferrite is consequence of the introduction of the rare earth atoms into the spinel structure, which leads to a change in the spin coupling. The magnetic behavior of such compound is not governed only by Fe–Fe interaction (3d electrons), but also by RE–Fe interaction which originates from 3d–4f spin coupling; this interaction triggers changes in magnetic properties [25,26]. The effect of Y-, Eu- or Gd-for Fe substitution has been studied by Sun et al. [27]. It was found that the substitutions of all rare earth ions (Y, Eu and Gd) greatly increase the electrical resistivity and decrease relative loss factor (RLF). Substitution of  $\text{Nd}^{3+}$  and its influence on magnetic properties was reported by Shinde et al. [28] observing a decrease in the Curie temperature and increase in the saturation magnetization with increasing  $\text{Nd}^{3+}$  content. Harish et al. [29] successfully reported a dramatic change in the photocatalytic activity of the inert  $\text{NiFe}_2\text{O}_4$  and highly solar active Nd substituted  $\text{NiFe}_2\text{O}_4$ . On the other hand, Bharathi et al. [30] reported the measurement of dielectric and magnetic properties of Gd- and Nd-doped nickel ferrites. It was found that the saturation magnetostriction does not change for  $\text{NiOFe}_{1.95}\text{Gd}_{0.05}\text{O}_3$  and  $\text{NiOFe}_{1.95}\text{Nd}_{0.05}\text{O}_3$ ; also, a small amount of  $\text{NdFeO}_3$  phase was identified in this Nd-substituted nickel ferrite. Sometimes, during the synthesis of such ferrites (iron perovskite),  $\text{NdFeO}_3$  can be synthesized as an additional product. The crystal structure of  $\text{NdFeO}_3$  has been described as orthorhombic with the *Pbnm* space group. From the magnetic viewpoint,  $\text{NdFeO}_3$  shows an antiferromagnetic ordering of the  $\text{Fe}^{3+}$  magnetic moments with the Néel temperature  $T_N=760$  K [31].

In this work, we report on changes of structural and magnetic properties of  $\text{NiFe}_2\text{O}_4$  doped with various level of Nd. The substitution-induced changes were monitored by X-ray powder diffraction (XRD), Fourier Transform Infrared spectroscopy (FTIR), magnetization measurements, and  $^{57}\text{Fe}$  Mössbauer spectroscopy. From the experimental data, the position of Nd ions in the spinel structure of  $\text{NiFe}_2\text{O}_4$  was investigated.

## 2. Materials and experimental methods

All materials were prepared from stoichiometric amounts of high-purity NiO,  $\text{Fe}_2\text{O}_3$ , and  $\text{Nd}_2\text{O}_3$  (Sigma-Aldrich > 99.99%), also were synthesized with various dope ranges (0.2, 0.3, 0.4,

0.5, and 5.0% (mol) by solid-state reaction. For undoped material, the  $\text{Fe}_2\text{O}_3$  and NiO were weighed with precision balance, and then mixed in the agate mortar. Finally, all mixed materials were heated in cylindrical oven at 1200 °C for 12 h and then were cooled down to room temperature. For Nd doped material, the same procedure was applied again at 1250 °C per material using  $\text{Nd}_2\text{O}_3$  chemical.

XRD measurements were carried using X'Pert PRO MPD diffractometer (PANalytical) in Bragg-Brentano geometry equipped with a Co X-ray tube (iron filtered  $\text{CoK}\alpha$  radiation:  $\lambda=0.178901$  nm), fast X'Celerator detector and programmable divergence and diffracted beam antiscatter slits. All materials were prepared on zero-background Si slide and measured in  $2\theta$  range 5–105° (resolution 0.017°  $2\theta$ ). Commercial standards SRM640 (Si) and SRM660 (LaB6) from NIST (National Institute of Standards and Technologies) were used for line position and instrumental line broadening evaluation. High Score plus software (PANalytical) in conjunction with PDF-4+ and ICSD databases were used for crystalline phase identification and Rietveld refinement, respectively.

The morphology and grain size of the prepared powders were monitored with a scanning electron microscopy (Philips XL-30 S FEG, SEM) and chemical composition was assessed by Energy Dispersive X-Ray (EDX) analysis.

FTIR spectra of the materials were measured at room temperature using an Agilent Cary 660 FTIR-ATR system. All measurements of the materials were reported in the spectral range from 400 to 1500  $\text{cm}^{-1}$ .

The superconducting quantum interference device (SQUID, MPMS XL-7, Quantum Design, USA) was used for magnetization measurements. Hysteresis loops were recorded at a temperature of 300 K and in externally magnetic fields ranging from – 5 to +5 T.

The  $^{57}\text{Fe}$  Mössbauer spectra of the studied materials were measured at room temperature employing a MS2007 Mössbauer spectrometer based on virtual instrumentation technique [32,33], operating at a constant acceleration mode and equipped with a  $^{57}\text{Co}$ (Rh) source. The acquired Mössbauer spectra were processed (i.e., noise filtering and fitting) using the MossWinn software program [34]. The isomer shift values are referred to  $\alpha$ -Fe foil sample at room temperature.

## 3. Results and discussion

### 3.1. XRD and SEM

In the XRD diffraction patterns, the  $\text{NiFe}_2\text{O}_4$  phase (PDF-4+ number: 01-078-6781) was identified for the materials with all Nd concentrations. For the material with 0.5% Nd concentration in the reaction mixture the additional peaks were observed and the analysis showed that they correspond to  $\text{NdFeO}_3$  (PDF-4+ number: 01-082-2421) (see Fig. 1 and Table 1). From the XRD analysis, it turned out that the Nd atoms are substituted into the spinel structure of  $\text{NiFe}_2\text{O}_4$ , and  $\text{NdFeO}_3$  phase formed when the Nd content exceeded a percolation limit, i.e., limit when the crystal structure of  $\text{NiFe}_2\text{O}_4$  cannot accommodate more Nd atoms.

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