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RESEARCH ARTICLE

STRUCTURAL, ELECTRICAL AND MAGNETIC STUDIES OF NEODYMIUM SUBSTITUTED NICKEL FERRITE NANOPARTICLES

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ABSTRACT

Nanoparticles $\text{NiFe}_{2-x}\text{Nd}_x\text{O}_4$ ($x=0.0, 0.05, 0.1, 0.15$) ferrite was prepared by sol-gel combustion method. The samples were characterized with X-ray diffraction and SEM measurements. The effect of Nd^{3+} cations substitution on structure of prepared nano particles was investigated. From the analysis, the system was found to be inverse spinel cubic structure. The lattice parameter (a) increases with Nd doping content. Room temperature DC electrical resistivity decreases. Magnetic studies by Vibrating Sample Magnetometer (VSM) shows that magnetization (M_s) decreases with increase in Nd^{3+} concentration. Coercivity (H_c) first decreases for $x=0.05$, after which it increases up to $x=0.15$.

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INTRODUCTION

Nano particles having different properties and applications are appeared as compared to their bulk counterparts. The magnetic properties of nano materials are used in medical, electronic, and recording industries that depend on the size, shape, purity and magnetic stability of these materials. In biomedical application, one can use nano magnetic materials as drug carriers inside body where the conventional drug may not work. For this purpose, the nano size particles should be in the super paramagnetic form with a low blocking temperature. Ferrite nano materials are object of intense research because of their proper magnetic properties. It has been reported that when the size of particles reduced to small size or in range of nano materials, some of their fundamental properties are affected. nano ferrites are simultaneously good magnetic and dielectric materials. These properties of the nano ferrites are affected by the preparation conditions, chemical composition, sintering temperature and the method of preparation. Several chemical and physical methods such as spray pyrolysis, sol-gel, co-precipitation, combustion technique, high energy milling etc. have been used for the fabrication of stoichiometric and chemically pure nano ferrite materials.

Among the available chemical methods, the sol-gel technique is an excellent method to synthesize rare earth substituted nano particles with maximum purity. In spite of the development of a variety of synthesis routes, the production of nickel ferrite nano particles with desirable size and magnetic properties is still a challenge. This would justify any effort to produce size tuned nickel ferrite nano particles with rare earth substitution. In the present paper, the structural and magnetic properties of neodymium substituted nickel ferrite and XRD, SEM, TEM and electrical properties were investigated.

Experimental

Synthesis

Nano particles of neodymium substituted nickel ferrite were synthesized by the sol-gel combustion method. A stoichiometric ratio of $\text{NiFe}_{2-x}\text{Nd}_x\text{O}_4$ ($x=0.0, 0.05, 0.1, 0.15$), were dissolved in ethylene glycol using a magnetic stirrer. The five sample solutions was then heated at 60 °C for 2 hours until a wet gel of the metal nitrates was obtained. The gel was then dried at 120 °C. This resulted in the self ignition of the gel producing a highly voluminous and fluffy product. The combustion can be considered as a thermally induced redox reaction of the gel wherein ethylene glycol acts as the reducing agent and the nitrate ion acts as an oxidant. The nitrate ion provides an oxidizing environment for the decomposition of the organic component. The obtained powder of different samples

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$\text{NiFe}_{2-x}\text{Nd}_x\text{O}_4$ ($x=0.0, 0.05, 0.1, 0.15$) was ground well collected in different packets for the measurements.

Characterization

The nickel ferrite samples were characterized by an X-ray powder diffract meter (XRD, Bruker AXS D8 Advance) using radiation ($wavelength= 1.5406 \text{ \AA}$) at 40 kV and 35 mA. Lattice parameter was calculated. The crystal structure, crystallite size and X-ray density were determined. The particle size was determined using scanning electron microscopy (TESCAN VEGA3SBH). Electrical resistivity was measured using two probe methods at room temperature. Magnetic parameters were calculated from the standard relations, and measurements were done at room temperature using a vibrating sample magnetometer (VSM) under an applied field of 10 kOe.

RESULTS AND DISCUSSION

Structural Analysis

The XRD patterns of nano particles are depicted in Fig. 2 and are typical of spinal structure. Comparing the XRD pattern with the standard data, the sample with Neodymium concentration zero shows highest peak and concentration 0.15 shows lowest.

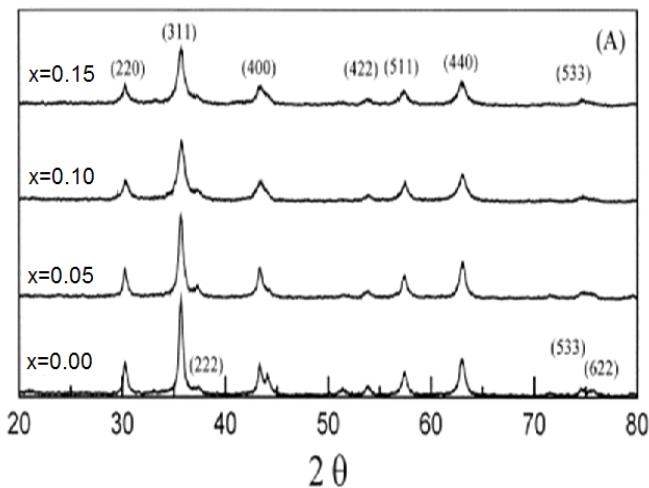


Fig. 1. XRD patterns of $\text{NiFe}_{2-x}\text{Nd}_x\text{O}_4$ ($x=0.0, 0.05, 0.1, 0.15$)

The diffraction peaks are broad because of the nanometer size of the crystallite. The crystallite size 'D' of the samples has been estimated from the broadening of XRD peaks using the Scherrer equation.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Lattice parameter 'a' for all the samples has been calculated by inter planar spacing (d_{hkl}) and 2-theta values using the standard relation,

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2}$$

Value of lattice constant for $x=0.0$ comes out to be 8.3865 \AA , well in agreement with reported value. Lattice constant has

increased monotonically with increment in Nd^{3+} concentration. This increase can be easily explained due to substitution of large ionic radii of Nd^{3+} (0.94 \AA) in place of smaller Fe^{3+} (0.67 \AA) ions. Also rare earth ions are usually present at grain boundaries that cause hindrance in the grain growth, therefore crystal size and unit cell parameters increases. The crystallite size was observed to increase with the increase of gadolinium concentration. It has been reported that the doping process generally decreases lattice defects and strain, but this technique can cause the coalescence of smaller grains, resulting in an increased average grain size for the nano particles. Calculated values of lattice parameter of neodymium substituted nickel ferrite samples were in close agreement with standard data.

SEM Analysis

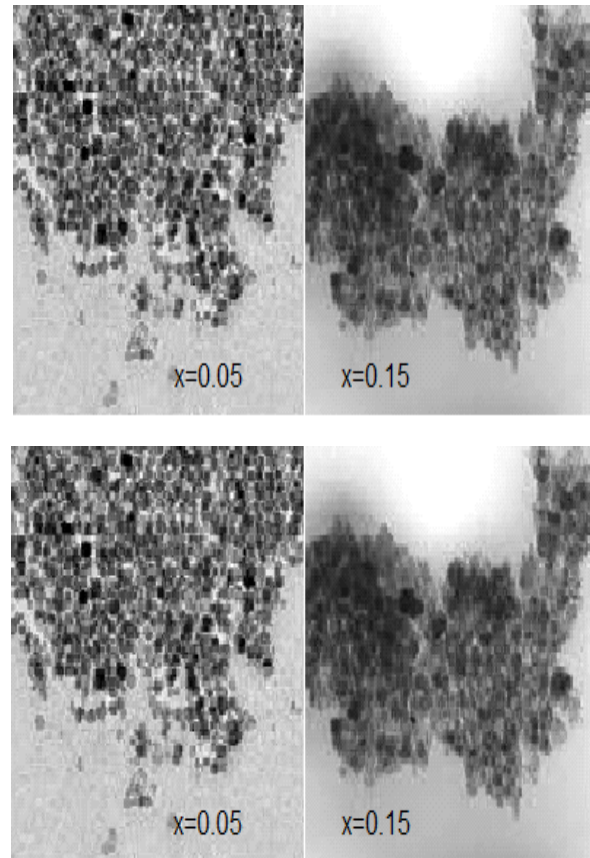


Fig. 2. SEM Image of $\text{NiFe}_{2-x}\text{Nd}_x\text{O}_4$ at $x= (0.00, 0.15)$

The SEM system that has been used for morphology of sample was CAMSCAN MV2300 model with 15 KV applied voltage. Photograph has been taken from the samples which were doped with Neodymium concentration 0.0 and 0.15. Particle size is obtained around 23.0 nm with mono dispersed nano particles as one can see from the photograph. It Shows the surface morphology and grain size for the prepared sample $x=0.0$ and $x=0.15$. It is clear from the image that uniformly distributed; less agglomerated and homogenous spherical particles have been formed in a controlled environment by sol-gel combustion technique. Micrographs also confirm the increase in grain size with Nd^{3+} doping in the parent crystal structure. The homogeneity of shape and grain size largely affects the electrical and magnetic properties of ferrites.

Dielectric study

Dielectric behaviour of nano spinel ferrites mainly depends upon the nature and distribution of metal cations on A-sites and B-sites in the spinel lattice. Spinel nickel ferrites are considered good dielectric materials and the high frequency dielectric behaviour is mainly dependent upon the particle size and method of synthesis of nano particles. Different studies have been provided relating the dielectric parameters of Nd³⁺ doped ferrites. Dielectric parameters (real and imaginary parts of relative permittivity, dielectric loss tangent) for the prepared series of NiNd_xFe_{2-x}O₄ (x=0.0 to x=0.15) have been studied in the frequency range 1 MHz to 1GHz at room temperature. Figs.3 shows the variation of relative permittivity with frequency at room temperature. It can be observed from the figure that relative permittivity for all the samples decreases with increase in frequency and ultimately becomes constant at higher frequencies. This decrease in permittivity is more rapid in the low frequency region and becomes sluggish as the applied frequency increases. This behaviour is subjected to dielectric polarization under the application of AC field.

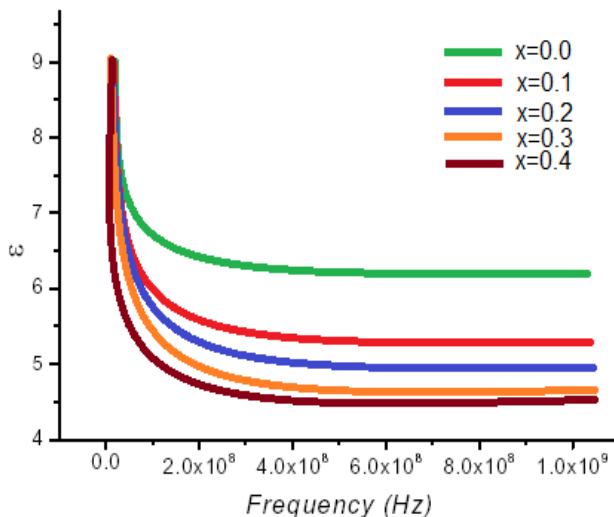


Fig. 3. Variation of permittivity as a function of frequency

It can be seen that dielectric loss tangent has the same trend as permittivity losses. It decreases with increase in frequency and becomes constant up to 1GHz due to decreased polarization at high AC fields. At x=0.15 shows a low loss dielectric behaviour which allows its use in high frequency data reading/writing in electronic structures.

Variation of DC electrical resistivity

Fig.5 shows the DC electrical resistivity of NiFe_{2-x}Nd_xO₄ (x=0.0, 0.05, 0.1, 0.15) Calculated at room temperature. Nickel Ferrite is a highly resistive material with high activation energy. Method of synthesis, type and substitution of doped cations at A and B site, particle size and morphology and sintering temperatures largely affects the electrical properties of spinel ferrites.

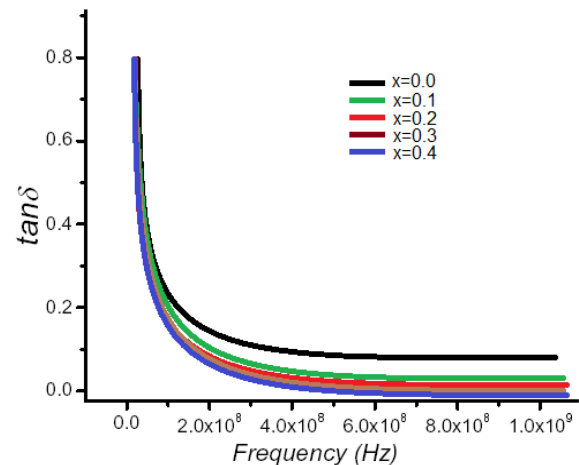


Fig. 4. Variation of dielectric loss as a function of frequency

This can be observed from the graph that DC electrical resistivity decreases up to a concentration of x=0.05 but then increases with increase in Nd³⁺ concentration. There is a significant increase in the DC electrical resistivity with Nd³⁺ substitution (x=0.15). Resistivity variations with Nd³⁺ substitution can be explained as for x=0.05, Nd³⁺ ions occupy A site replacing Fe³⁺ due to which number of Fe³⁺ ions at B-site increases and thus Fe²⁺. This may happen due to lattice distortion caused by doping a concentration of x=0.05. A low concentration of Nd³⁺ causes a severe change in Centrosymmetric FCC structure of parent crystal that causes an increase in hopping of electron and a net electric polarization. This reason is reflected in the high dielectric parameters and a sharp decrease in saturation magnetization for x=0.05.

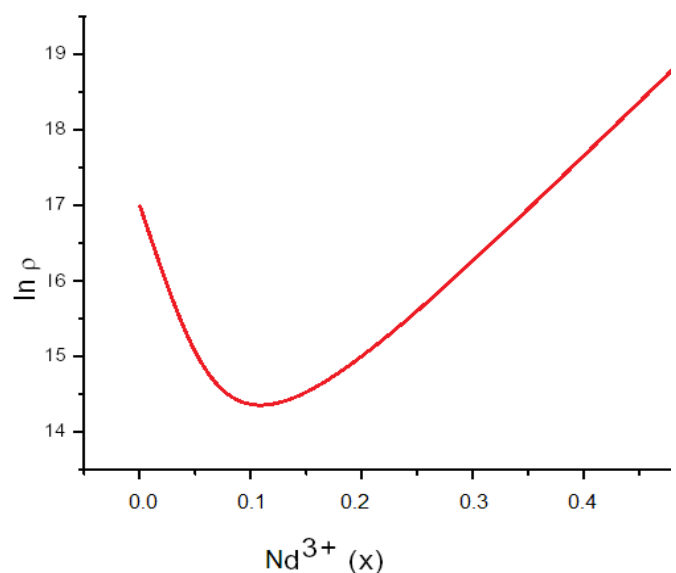


Fig. 5. Variation of resistivity with concentration

Magnetic study

To study the effects of Nd³⁺ doping on magnetization, coercivity, remanent magnetization (Mr) and magneto-crystalline anisotropy (HK) of NiFe, M-H hysteresis loops were recorded using VSM under the applied magnetic field of

10 kOe at room temperature. Fig. shows the hysteresis curves for all the samples under investigation. It is clear that all the samples show a fine s shape loops with a decrease in magnetization (Ms) and coercivity (Hc) with increase in Gd³⁺ concentration. This is due to the substitution of Nd³⁺ in place of Fe³⁺ at B-sites. Nd³⁺ ions have a higher magnetic moment (7 μ B) in place of Fe³⁺ that has 5 μ B. The order in magnetic moments of rare earth ions is below room temperature; due to this at room temperature Nd³⁺ ion behaves as non magnetic that causes a decrease in saturation magnetization and coercivity. This substitution causes a lattice distortion that alters the magnetic characteristics of materials. Magnetic properties of ferrite materials largely based upon the grain size, cation substitution, and A-B exchange interactions. Increase in the grain size and decrease of A-B super exchange interaction causes canting spins at the surface of nano particles that decreases the magnetic characteristics of the present samples. Spin arrangement for the Ni-Nd ferrites were analyzed by measuring the value of Bohr's magneton by the relation

$$n_B = \frac{\sigma_s}{5585} M_w$$

Where

$$n_B = (6+x)\cos\alpha_{Y-K} - 5(1-x)$$

Where x represents the composition of doping ion. Anisotropy field (H_k) can be calculated by following equations

$$H_k = \frac{2K_1}{\mu_0 M_s}$$

Where μ_0 is the permeability of the free space, M_s is saturation magnetization from the fit.

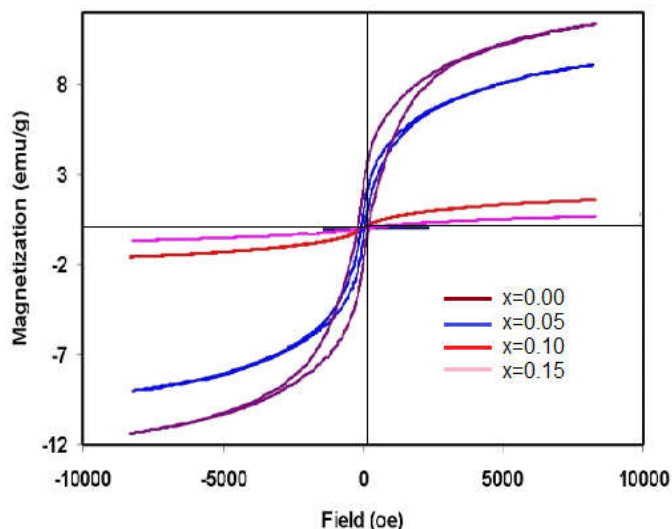


Fig.6. Hysteresis loop of NiNd_xFe_{2-x}O₄ at different x (x=0.0, 0.05, 0.1, 0.15)

Pure nickel ferrite is has high anisotropy constant and field due to occupation at B-sites. It decreases due to Nd³⁺ occupation at B-sites. Cubic anisotropy constant also decreased doping neodymium showing a decrease in coercivity for present studied samples. The anisotropy parameters (anisotropy

constant and field) are not decreasing monotonically but more abruptly due to differing concentration of doping ion. This behaviour is showing a strong lattice distortion due to Nd³⁺ substitution. It is clear from the data that cubic anisotropy decreases with increase in neodymium concentration.

Conclusion

Nano spinal NiFe_{2-x}Nd_xO₄ with x in step increment 0.05 has been synthesized by sol-gel Combustion method. All the studied samples are pure cubic spinal phase ferrites without any impurity metal oxides. Lattice constant and crystallite size increases with increase in Nd³⁺ concentration, due to increase ionic radii and atomic weight of neodymium as compared to Fe³⁺. Dielectric constant and loss tangent decreases to 4.92 and 0.016 respectively with increase in the dopant concentration showing that the material with x=0.15 is a low loss dielectric. Magnetic studies by Vibrating Sample Magnetometer (VSM) shows that magnetization (M_s) decreases with increase in Nd³⁺ concentration. Coercivity (H_c) first decreases for x=0.05, after which it increases up to x=0.15.

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