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Effect of sintering temperature on structural and magnetic properties of Ni-Zn spinel ferrite nanoparticles

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Abstract

Effects of sintering temperature on the magnetic properties of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles were studied. Ferrite samples were synthesized by sol gel auto combustion method and sintered at various temperatures (500, 700 and 900 ^oC) in air. Their structural and magnetic properties were studied by X-ray diffraction and pulse field hysteresis loop technique. The X-ray diffraction patterns reveal that all the samples consist of nanocrystalline single cubic phase structure. Sintering temperatures (T_s) significantly affect on crystallite size of samples. The crystallite size increases from 12 nm to 31 nm with increase in sintering temperature. The M-H loop of the samples was measured at room temperature. The saturation magnetization increases where as coercivity decreases of the samples with increase in sintering temperature.
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Keywords: *XRD; Sintering temperature, Magnetic material.*

1. Introduction

The spinel ferrites with formula MFe_2O_4 are technologically important magnetic materials from the point of view of their potential applications and novel properties. Recently, there has been an increase interest in these magnetic nanoparticles because of their unique physiochemical, electrical, magnetic, dielectric and optical properties [1-4]. Many researchers have studied magnetic nanoparticle for various applications. The most conventional magnetic nanoparticles are iron oxide, γ-Fe_2O_3 and spinel ferrites. In the nano region spinel ferrite nanoparticles exhibit interesting, unusual and superior properties as compare to their bulk counterpart. Crystallite size and specific surface area of spinel ferrites are responsible for their superior properties. Nanoparticles of spinel ferrites have great potential for catalytic degradation of organic and inorganic pollutants, as a catalyst and as a sensor [5, 6]. Recently, they have also attracted considerably for biomedical applications such as contrast agents for magnetic resonance imaging (MRI), hyperthermia applications and in drug delivery system, microwave absorbance, magnetic fuel, Catalyst, multilayer chip inductor (MLCI), electromagnetic interference (EMI), suppression, gas sensing, transformer cores, antenna rods, inductors, recording heads etc. [7-11]. The high electrical resistivity, low eddy current and dielectric losses, moderate saturated magnetization and high Curie temperature are the important properties of Nickel ferrite. These properties are sensitive to various parameters like method of preparation, preparative parameters and conditions, nature of dopant and cation distribution. Due to their comparatively low losses at high frequencies, they are extensively used in specific applications such as switch mode power supply (SMPS). They are also used in high frequency circuits, high quality filters, read and write etc. [12-14].

Nickel ferrites are one of the versatile and technologically important soft ferrite materials due to their typical ferromagnetic properties, low conductivity and thus lower eddy current losses, high electrochemical stability, catalytic behaviour, abundance in nature etc. Soft magnetic materials with their remarkable electrical, magnetic and optical properties have wide range of applications in various areas [1, 3-4]. Among the soft magnetic materials mixed spinel ferrites like Ni-Zn is of great importance because of their scientific and industrial application. Ni-Zn spinel ferrite has been extensively used in many electronic devices because of their moderate electrical, magnetic properties and good chemical stability.

The properties of the ferrite nanoparticles can be further tuned by controlling their shape, size and surface structure. Various techniques have been developed for the synthesis of ferrite nanoparticles. These method includes, sol-gel method, thermal decomposition, chemical co-precipitation, hydrothermal, mechanical milling etc. [15-19]. Among these methods sol-gel auto combustion method have shown promising results in controlling the particle morphology and their size distribution. The sol-gel auto combustion methods yields particles of nanometer dimension, gives better homogeneity and high quality powder. Various stabilizing agent/fuel such as citric acid, glycine, urea, polyethylene oxide, polyglycol etc are reported to be used for the controlled growth, prevention of agglomeration and stabilization of nanoparticle.

However, the properties of spinel ferrite nanoparticles are also dependent on sintering temperature, sintering time and sintering atmosphere. The sintering temperature affects the microstructure there by change in the properties can be seen.

In the literature, mixed Ni-Zn spinel ferrites were developed by different methods, by number of researchers [16-18, 20]. The structural and magnetic properties of nanocrystalline Ni-Zn spinel ferrites in the context of cationic distribution have been reported by J. Jadhav et al. [21]. The influence of calcination temperature on the morphology and magnetic properties of mixed Ni-Zn ferrite is reported by A. C. F. M. Costa et al. [22]. The synthesis of mono dispersed pH-sensitive $Ni_{0.65}Zn_{0.35}Fe_2O_4$ magnetic nanoparticles for medical applications is reported in the literature by Ling-Hui Nie et al. [23].

In summary, though mixed Ni-Zn spinel ferrite have been studied by many researchers in nanocrystalline form but effect of sintering temperature on various properties of mixed Ni-Zn ferrites is reported less in the literature. The increase in sintering temperature may affect the particle size there by influences the structural, morphological, electrical, dielectric and magnetic properties of mixed Ni-Zn spinel ferrite. In this work we report the synthesis of mixed Ni-Zn spinel ferrite by sol-gel auto combustion method using L-Ascorbic acid as a fuel. The resultant Ni-Zn powder of composition $Ni_{0.65}Zn_{0.35}Fe_2O_4$ is sintered at three different temperatures 500°C, 700°C and 900°C for 6 hr and its influence on structural and magnetic properties were studied and results obtained were presented in this work.

2. Experimental

Sol-gel auto combustion method is based on oxidation reduction process. The energy to form the ferrite nanoparticles of nanometer dimensions is provided by oxidation reduction process of chemical precursors and fuel used in the sol-gel auto combustion method. Metal nitrates of constituent ions i.e. nickel nitrate, zinc nitrate and ferric nitrate of AR grade supplied by Merck were used as a raw materials. L-ascorbic acid was used as a fuel. The metal nitrate to fuel ratio was taken to be 1:3. The as prepared powder of Ni-Zn Spinel ferrite is sintered at different temperatures viz. 500°C, 700°C and 900°C and effect of sintering temperature on various properties of Ni-Zn ferrites was studied.

Characterizations

The prepared spinel ferrite system was subjected to various characterization technique like X-ray diffraction (XRD). Moreover, the magnetic measurements were carried out by using pulse field hysteresis loop tracer technique at room temperature. X- ray diffraction (XRD) measurements are obtained at room temperature (Bruker Advanced D8), operated at 40 kV and 20 mA, utilizing a Cu-Kα radiation source (λ = 1.54056 Å) in the 2θ range 20-80^0 with step of 0.01^0 and time/step 2 s. Pulse field hysteresis loop tracer supplied by Magneta pvt. Ltd. Mumbai was used to measure the magnetic properties of the samples at room temperature. The field applied during measurements was ± 5000 Oe.

3. Results and Discussion

X-Ray diffraction (XRD) is an important tool to check the phase purity and crystal structure of the prepared samples. In the present study, the X-Ray diffraction technique was used to characterize $Ni_{0.65}Zn_{0.35}Fe_2O_4$ spinel ferrite sintered at 500°C, 700°C and 900°C for its phase purity, crystal structure and structural parameters. The X-Ray diffraction pattern recorded at room temperature for all the samples is shown in Fig. 1. A close observation of XRD pattern suggest that the planes with Miller indices (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) belonging to cubic spinel structure are observed. These planes have sufficient intensity and are broader. The analysis of the XRD pattern revealed that $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticle with cubic spinel structure was formed at 500°C, 700°C and 900°C. Further, it is observed from Fig. 1 that the broadening of the observed planes decreases with increase in temperature, the intensity increases with increase in sintering temperature. With increasing sintering temperature, the crystallization quality of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles was improved. Based on $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles phased standard diffraction pattern data of JCPDS [24] with sintering temperature up to 900 °C, the $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles has formed non impurity phases of typical single phase cubic spinel structure. The decrease in broadening of the various planes may be due to the increase in crystallite size [25, 26].

The bulk density is found to be less as compared to X-Ray density. It is found that the bulk density increases with increase in annealing temperature. The increase in bulk density may be due to the fact that with increase in temperature the sample becomes denser [27, 28].

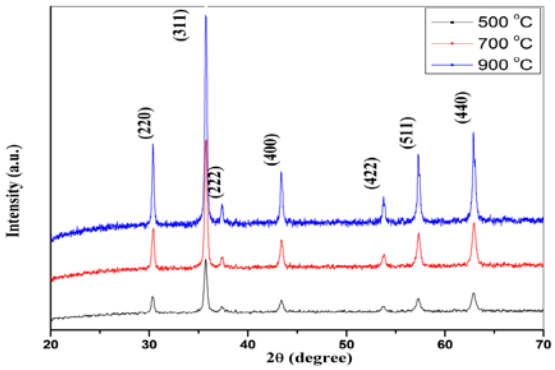

Fig.1. X-ray diffraction pattern of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500 °C, 700 °C and 900 °C)

Table 1: Lattice parameter (a), X-ray density (d_x), Bulk density (d_B) and Crystallite size (t) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500 °C, 700 °C and 900 °C)

Sintering temp. maintain at	**a (Å)**	**d_x (gm/cm^3)**	**d_B (gm/cm^3)**	**t (nm)**
500 °C	8.335	5.4312	4.5449	12
700 °C	8.324	5.4527	4.6317	18
900 °C	8.333	5.4351	4.7893	31

The magnetic properties of prepared Ni-Zn ferrite nanoparticles were measured by pulse field hysteresis loop technique at room temperature. The variations of magnetization with static magnetic field (up to 5 kOe) of samples are shown in Fig. 2 for 500 °C, 700 °C and 900 °C sintering temperature. The hysteresis loop is deviated from rectangularity, and hence illustrates magnetic behavior of soft ferrite.

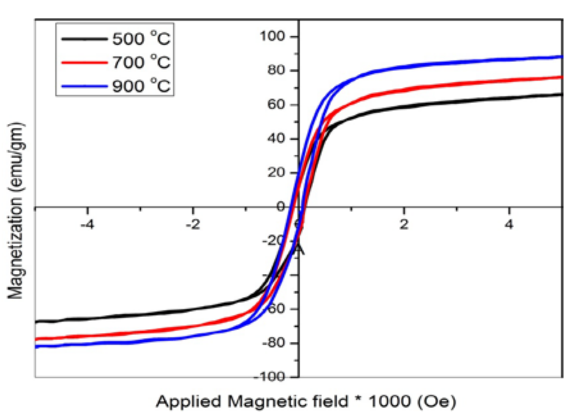

Fig. 2. M-H plot recorded at room temperature for $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500 °C, 700 °C and 900 °C)

It can be seen that magnetization increases with the field and attains near saturation near 5 kOe for all the samples. The values of saturation magnetization (M_s), coercivity (H_c), and remanence (M_r) are given in Table 2. The values of *M*s obtained for the present samples goes on increasing as sintering temperature increases. However, the values of coercivity (H_c) decrease as sintering temperature increases. The remanence ratio increases with sintering temperature. The increases in saturation magnetization can be attributed mainly due to increase in grain [29].

Table 2: Saturation magnetization (Ms), Remanence magnetization (Mr), coercivity (H_C), Remanence ratio (Mr/Ms) and Magneton number (n_B) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500°C, 700°C and 900°C)

Sintering temp. maintain at	**M_S (emu/g)**	**Mr (emu/g)**	**H_C (Oe)**	**n_B**
500 °C	66.50	15.74	114.15	0.67
700 °C	76.68	17.43	99.64	0.74
900 °C	88.57	9.54	79.36	0.40

The reduction of coercivity (H_c) with increasing sintering temperature can be attributed to an increase in grain sizes. Larger grains tend to consist of greater number of domain walls, and hence, contribution of wall movement to magnetization or demagnetization is greater than that of domain rotation. The magnetization/demagnetization caused by domain wall movement requires less energy than that required by domain rotation [30]. Maximum retentivity of 17.43 emu/gm was found for sample obtained at a sintering temperature of 700 °C.

4. Conclusions

The nanocrystalline $Ni_{0.65}Zn_{0.35}Fe_2O_4$ ferrite system for different sintering temperature viz. 500 °C, 700 °C and 900 °C was successfully synthesized via sol-gel auto combustion technique. The X-ray diffraction results for all samples showed the formation of single phase cubic spinel structure. The crystallite size confirms the nanocrystalline nature of the samples. The crystallite size of the samples calculated using the Debye-Scherrer's formula. The crystallite size increases from 12 nm to 31 nm because of the increase in annealing temperature. All the structural parameters changes as sintering temperature increases. The M-H curves recorded at room temperature exhibits a typical hysteresis loop indicating that the sample exhibits magnetic nature for all the samples. The values of M_s obtained for the present samples goes on increasing as sintering temperature increases. However, the values of coercivity (*H*c) decrease as sintering temperature increases.

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