ORIGINAL RESEARCH PAPER

Physics

Volume - 7 | Issue - 2 | February - 2017 | ISSN - 2249-555X | IF : 3.919 | IC Value : 79.96

Structural and magnetic properties of sol-gel synthesized zinc ferrite nanoparticles

KEYWORDS

Sol-gel, Zinc ferrite, Structural properties, Magnetic properties.

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ABSTRACT $ZnFe_2O_4$ nanoferrites were synthesized using sol-gel technique. As-prepared and the annealed samples were examined for their structural and magnetic properties using X-ray diffraction, IR spectroscopy, scanning electron microscopy and vibrating sample magnetometer. The XRD analysis showed that the as-prepared sample was non-homogenous were as the annealed sample showed single phase and ferrite phase. The grain size in both the samples was observed to be 15 nm and 30 nm. The nanosize natures in the prepared samples were identified using SEM analysis. IR analysis was used to identify the ferrite phase and associated chemicals during the synthesis. Magnetization measurements revealed that pure sample showed paramagnetic behavior and impure sample showed ferromagnetic behavior.

Introduction

Ferrites are ferrimagnetic materials containing iron oxide as the main constituent with different metal oxides. Ferrites have received considerable attention in experimental, theoretical and computational solid state physics due to their rich and often unusual experimental behaviour. Ferrites and substituted ferrites find extensive applications in high frequency devices, RF transformers, toys, TV yokes, loud speakers and in many of the materials which are used in daily life. Ferrites have greater applications in the current science and technology like in the field of biotechnology for drug delivery, self-assembly and memory devices [10].

The structural and magnetic properties of ferrites depend mainly on the grain size, synthesis techniques and cation distribution. Zinc ferrite is a normal spinel with the tetrahedral (A) sites almost exclusively occupied by Zn^{2+} ions, while the octahedral [B] sites by Fe^{3+} ions and results in normal spinel. Synthesis of nanocrystalline ferrite material like $ZnFe_2O_4$ is growing interest because of wide possible magnetic and catalytic properties. $ZnFe_2O_4$ can be used as starting materials for obtaining substituted ferrites [9]. ZnFe2O4 is commercially very important magnetic materials [2, 9] having applications such as gas sensors [11], absorbent materials [12] catalyst [13], etc. The present work has been taken up to study the effect of annealing temperature on the structural and magnetic properties of prepared $ZnFe_2O_4$ samples. So, the research on $ZnFe_2O_4$ is of utmost interest in understanding the structural and magnetic behaviour in nano meter size level.

2. Experimental

$ZnFe_2O_4$ nanoferrites were prepared by using sol-gel method [7]. The a.r. grade citric acid ($C_6H_8O_7H_2O$), zinc nitrate ($Zn(NO_3)_26H_2O$), ferric nitrate ($Fe(NO_3)_39H_2O$) from SD fine chemicals were used as starting materials. The synthesis technique is described in detail elsewhere [7]. The obtained raw powder was annealed at 500°C temperature for 5 hours.

XRD analysis was measured using Philips PW 3020 Bragg-Brentano diffractometer using Cu Kα radiation (wave length λ = 1.54 Å). The morphology of powder was observed using scanning electron microscopy (SEM) from Carl Zeiss. The structural changes were observed using ABB Bomem MB 102 infrared spectrometer. The samples were mixed with KBr and made in the form of pellets and recorded at 4 cm^{-1} resolution giving the spectra in the 2000 - 200 cm^{-1} range. Room temperature magnetization was measured using ADE magnetics DMS 4 Vibrating Sample Magnetometer (VSM) Ltd.

3. Results and discussions

Figure 1 shows the XRD patterns of $ZnFe_2O_4$ as-prepared and annealed samples. Both the samples show the formation of spinel structure. Apart from the main peak of $ZnFe_2O_4$ spinel structure, the as-prepared sample also shows few minor and low intensity peaks corresponding to unidentified impurity phases. When the as-prepared sample was annealed at 500°C the impurity phases disappeared and the $ZnFe_2O_4$ showed homogenous spinel structure. The lattice constant for as-prepared sample was observed to be 8.432 Å and for annealed sample it was 8.419 Å. The XRD patterns of as-prepared samples looks little broad compared to the annealing sample. The calculate grain for as-prepared sample was observed to be 15 nm and for annealed sample it was 22 nm. The difference in the lattice constant and grain size between the as-prepared and annealing samples may be due to the higher annealing temperature. The as-prepared sample was prepared at very low temperature therefore the grain size shows very less value compared to annealing sample. Therefore the annealing temperature showed significant effect on the structural properties. The XRD analysis is further supported with the IR analysis.

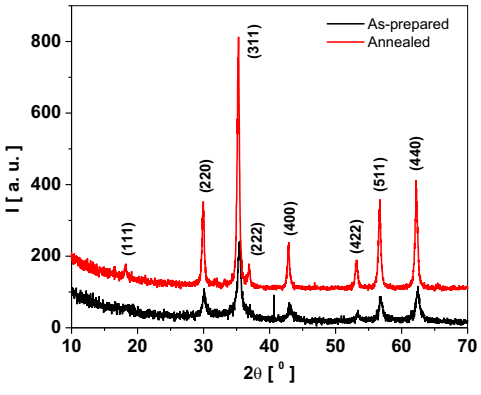

Fig.1. X-ray diffraction patterns of $ZnFe_2O_4$ as-prepared and annealed samples

Figure 2 shows the IR spectra of $ZnFe_2O_4$ as-prepared and annealed

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samples. The as-prepared sample shows the bands corresponding to Zn, Fe ions at 400 cm-1 and 600 cm-1 [8]. Apart from the main ferrite bands it also contains the absorption bands at 1350 cm-**1** correspond to NO^{3}- ions and at 1600 cm^{-1} corresponding to carboxyl (COO-) groups. When the $ZnFe_2O_4$ sample is annealed at 500°C, except the bands corresponding to ferrite phases all other phases corresponding to NO3- and COO- disappeared. Therefore it is observed that proper annealing temperature will lead to the ferrite phase formation. Therefore the XRD and IR data agrees with each other.

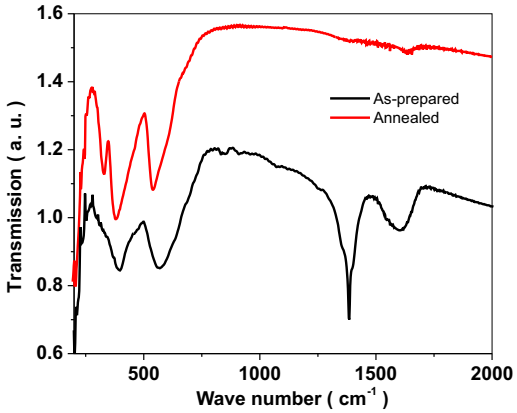

Fig.2. IR spectra of $ZnFe_2O_4$as-prepared and annealed samples.

Further the evidence of nano size nature in the prepared samples and the morphology of $ZnFe_2O_4$ samples has been verified using SEM analysis. Figure 3 shows the SEM images of as-prepared and annealed $ZnFe_2O_4$ samples. The as-prepared sample Figure 3(a) shows the rough surface with no clear appearance of nano nature in the sample. With the annealing temperature the surface of $ZnFe_2O_4$ samples became smooth and the nano size grains seems to appear. When the sample is annealed properly the unwanted phases disappears and helps in the crystallization of ferrites. There are many evidences that annealing temperature helps in the grain growth and in obtaining the single phase ferrite structures. Therefore in the present study it is clear that the annealing temperature significantly contributed in achieving the phase pure $ZnFe_2O_4$ samples.

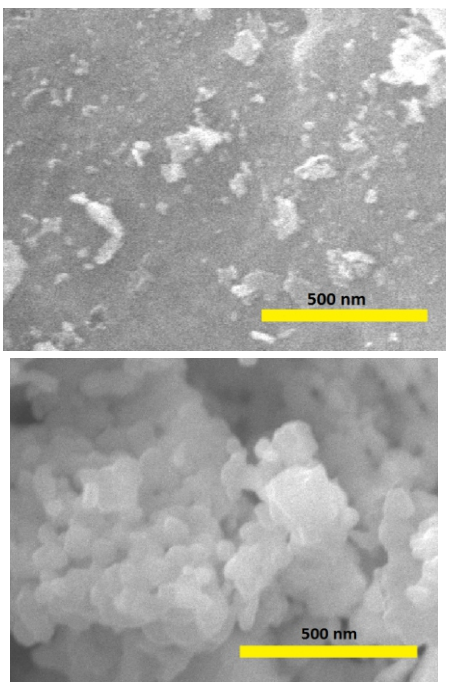

Fig.3. SEM micrographs of $ZnFe_2O_4$ (a) as-prepared and (b) annealed samples.

Figure 4 shows the magnetization curves of $ZnFe_2O_4$as-prepared and annealed samples measured at room temperature with the maximum applied field of 10 kOe. The as-prepared sample showed a ferromagnetic like behavior having very small magnetization values. Whereas, the annealed sample showed a straight line passing through origin signifying the typical paramagnetic like behavior. Higher annealing temperature on the $ZnFe_2O_4$ material will result in the paramagnetic behavior. Such kind of behavior was previously observed in $ZnFe_2O_4$ that it is antiferromagnetic at room temperature so there should be no additive magnetic contribution at room temperature and the antiferromagnetic spin order of $ZnFe_2O_4$ is not homogeneous [9]. Therefore higher annealing temperature showed significant effect on the magnetic properties of $ZnFe_2O_4$ nanomaterials.

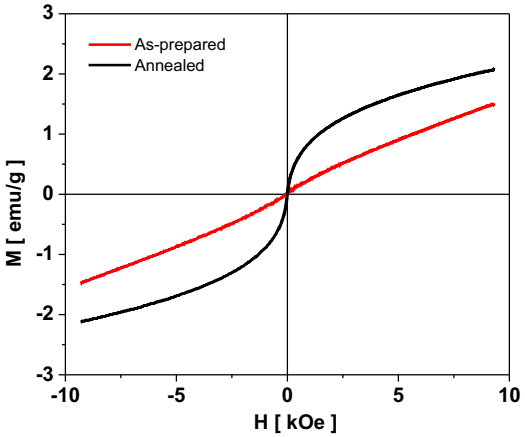

Fig.4. SEM micrographs of $ZnFe_2O_4$ (a) as-prepared and (b) annealed samples.

Conclusions:

$ZnFe_2O_4$ nanoferrites were successfully synthesized using sol-gel technique. Two samples with different annealing temperatures were examined for the structural and magnetic properties. XRD analysis confirmed the ferrite phase formation and it is supported with IR analysis. Nanosize behavior in the prepared samples is verified using SEM analysis. As-prepared behavior ferromagnetic and the annealed samples showed paramagnetic behavior.

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