Contents lists available at ScienceDirect



Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm



Formation of latent tracks and their effects on the magnetic properties of nanosized zinc ferrite



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ARTICLE INFO

Article history: Received 6 June 2013 Received in revised form 13 September 2013 Available online 11 October 2013

Keywords: Nanosized zinc ferrite Latent track High resolution of transmission electron microscope X-ray diffraction Vibrating sample magnetometer

ABSTRACT

In present work we have studied the effect of latent tracks on the structural and magnetic properties of nanosized zinc ferrite. These tracks were created by irradiating the system with 200 MeV Ag¹⁵⁺ beam at the fluence of 4×10^{12} ions/cm². Theoretical estimation of latent tracks shows that the track radius is \sim 3 nm. These tracks were observed in the system with the help of transmission electron microscope. Beside these tracks, lattice distortion also appeared in the irradiated specimen. The formation of hysteresis after irradiation is due to the presence of latent tracks in the system. The change in blocking temperature after irradiation may be attributed to the change in magnetic ordering/cation inversion. Related mechanism has been discussed on the basis of thermal spike model.

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1. Introduction

Various reports are available showing the formation of latent tracks in materials after irradiation with swift heavy ions [1,2]. Latent tracks are the trail of defects inside the materials created in the path of heavy ions during a typical irradiation experiment. Formation of these tracks depends on the amount of energy lost inside the material. Process of energy loss is generally governed by two processes and the corresponding energy loss per unit length inside material is known as (i) electronic stopping (S_e) and (ii) nuclear stopping (S_n) . Role of S_e has been studied in ferrites and it is observed that S_e should be almost 10^3 times greater than S_n for producing defects by electronic excitations inside the materials [3–7]. Depending upon value of S_e, different types of defects are produced in the materials. The defects are known as (i) point/cluster defects and (ii) latent tracseks/columnar defects. Latent tracks are produced only when *S*_e value is greater than a threshold. Although the presence of these tracks is indirectly evidenced by various experimental techniques such as X-ray diffraction (XRD), Raman Spectroscopy etc. [8] but can be visualized only by microscopic methods like high resolution

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transmission electron microscopy (HRTEM), scanning electron microscopy (SEM) and atomic force microscopy (AFM) [9–11].

Recently, there has been great interest in irradiation studies of ferrites for understanding of underlying physics [3–7]. Ferrites are the magnetic insulators having general formula $[M_{\delta}Fe_1 - \epsilon]_A$ $[M_{1-\delta}Fe_{1+\delta}]_AO_4$, where, M is divalent transition metal ion and Fe is in trivalent state and are generally preferred for various technological applications like transformer cores, high frequency application etc [12]. Apart from their various applications, ferrites in nanoregime, exhibit superior properties compared to bulk counterpart that are affected by the crystallite size and the methods of synthesis. Among the various ferrites, zinc ferrite is an important example which exhibit normal spinel structure ($\delta = 1$) and is antiferromagnetic having Néel temperature of 10 K. Nanoparticles of zinc ferrite exhibits superparamagnetism at room temperature transforming it's structure from normal to mixed spinel. Various methods not only produce different order of magnitude of magnetization but also different type of magnetic ordering in the synthesized ferrite of similar particle size [12-18]. Different values of blocking temperature $(T_{\rm B})$ and saturation magnetization are reported depending upon the crystallite size and method of synthesis [18,19]. It has been seen that crystallite size of these materials also plays dominant role in modifying the properties after irradiation [20–23]. Studer et al. (1993) investigated irradiation induced effects in bulk zinc ferrite irradiated with heavy ions of inert gases. In this work the induced

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^{0304-8853/\$ -} see front matter © 2013 Elsevier B.V. All rights reserved. http://dx.doi.org/10.1016/j.jmmm.2013.09.050

magnetization and enhancement in Néel temperature after irradiation has been attributed to the cation redistribution [24]. Apart from this study, decrease in magnetization was observed in nanosized zinc ferrite system after irradiation with 100 MeV oxygen beams which has been attributed to decrease in cation inversion and presence of defects in the system [7,21,25]. Magnetic resonance in nanosized zinc ferrite generally appears at higher value compared to pristine after irradiation. The electron paramagnetic resonance (EPR) line-shape and derived parameters from the EPR spectra show size dependent changes in irradiated specimen [20,24]. Another important feature observed in these studies is that changes in magnetic properties are affected by the crystallite size of pristine samples. These several studies are confined to investigate irradiation induced changes when point defects are produced in the system. With these several observations, a study which includes investigation carried out if the nature of defects is changed will be an ambiguous effort to extend this work. In the present investigation, formation of latent tracks is observed with the help of high resolution transmission electron microscopy (HRTEM) and their effects on structural and magnetic properties of nanoszied ZnFe₂O₄ system has been studied. To the best of our knowledge no reports are available on the formation of latent tracks in nanosized zinc ferrite, although few reports exist that shows the presence of latent tracks of diameter \sim 2–3 nm in bulk zinc ferrite and other similar systems [2].

2. Experimental

2.1. Sample synthesis and irradiation of ion beam

Zinc ferrite nanoparticles were synthesized by using nitrate route [16]. In this method zinc nitrate and ferric nitrate were taken

Table 1

Estimated parameters from most intense peak (311) of XRD pattern and HRTEM micrographs.

Irradiation		Sintering temperature (°C)		
		300	800	1000
Pristine	$D_{ m XRD} (nm) \ D_{ m TEM}(nm) \ a(m \AA) \ ho(g/cc)$	$\begin{array}{c} 13 \pm 1 \\ 13 \pm 7 \\ 8.39 \pm 0.01 \\ 5.4 \pm 0.1 \end{array}$	$\begin{array}{c} 32\pm 2\\ 90\pm 45\\ 8.42\pm 0.01\\ 5.3\pm 0.1\end{array}$	$\begin{array}{c} 61 \pm 4 \\ 350 \pm 100 \\ 8.43 \pm 0.01 \\ 5.3 \pm 0.1 \end{array}$
Irradiated	$D_{ m XRD} (nm)$ $D_{ m TEM} (nm)$ a(Å) ho(g/cc)	$\begin{array}{c} 14 \pm 1 \\ 15 \pm 8 \\ 8.41 \pm 0.01 \\ 5.4 \pm 0.1 \end{array}$	$\begin{array}{c} 37 \pm 2 \\ 102 \pm 50 \\ 8.44 \pm 0.01 \\ 5.2 \pm 0.1 \end{array}$	$52 \pm 3 \\ 279 \pm 120 \\ 8.42 \pm 0.01 \\ 5.4 \pm 0.1$

 $^{*}D_{XRD}$ and D_{TEM} represents size obtained from XRD pattern and TEM micrographs.

in stoichiometric ratio and dissolved in double distilled water separately. Both solutions were further dissolved in citric acid solution by keeping the cations to citric acid molar ratio 1:3. This solution was put on a magnetic stirrer at 85 °C for 3 h. This viscous solution was heated in a furnace at 100 °C for overnight to form the precursor. This precursor was sintered at 300, 800 and 1000 °C for 1 h to obtain zinc ferrite samples of different crystallite size. These samples were irradiated with 200 MeV silver beam by Pelletron Accelerator at Inter University Accelerator Centre (IUAC), New Delhi at the fluence of 4×10^{12} ions/cm². Stopping and Range of Ions in Matter (SRIM) code calculation shows that values of S_{e_1} S_n and projected range (R_p) are 27 keV/nm, 0.007 keV/nm and 11 μ m respectively. In present case S_e/S_n ratio is $\sim 10^3$ which indicate that damage due to nuclear cascades is negligible. Value of S_e is larger than the threshold value (~13 keV/nm), hence latent tracks are expected to produce in the system [8].

2.2. Characterization techniques

The crystalline phase and structure of pristine and irradiated samples were estimated by using Rigaku Japan X-ray diffractometer. The scan was made from 20° to 75° with an interval of 0.02°. JEM2100F electron microscope operated at 200 kV was used for observation of particle size, morphology and irradiation induced defects/latent tracks inside materials. To estimate average particle size bar plot between number of particles and particle size is fitted by Gausian distribution function. Hysteresis (σ -H) curves of pristine and irradiated samples at room temperature were recorded on Princton-105 vibrating sample magnetometer (VSM). Further, (σ -H) curves at room temperature (RT) and 10 K were recorded on a Quantum Design 100 PPMS vibrating sample magnetometer. Thermal magnetization (σ -T) measurements in zero-field (ZFC) and field cooled (FC) mode at two different fields of 0.1 and 1 kOe were also carried out.

3. Results and discussion

All pristine samples exhibit cubic spinel phase. Irradiated counterpart of sample sintered at 300 °C exhibits presence of α -Fe₂O₃ and ZnO phases along with spinel phase. However no phase change is observed for sample sintered at 800 and 1000 °C after irradiation. The detailed discussion on presence of impurity phase in samples with low sintering has been carried out elsewhere [23]. The values of crystallite size and lattice parameters for pristine and irradiated samples have been collated in Table 1.



Fig. 1. HRTEM image of sample sintered at 300 °C (a) distribution of particles and (b) crystallite size (encircled).

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