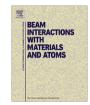
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# Effect of thermal spike energy created in CuFe<sub>2</sub>O<sub>4</sub> by 150 MeV Ni<sup>11+</sup> swift heavy ion irradiation

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#### ABSTRACT

Effects of 150 MeV Ni<sup>11+</sup> swift heavy ion (SHI) irradiation on copper ferrite nanoparticles have been studied at the fluences of  $1\times10^{11}$ ,  $1\times10^{12}$ ,  $1\times10^{13}$ ,  $1\times10^{14}$  and  $5\times10^{14}$  ions/cm<sup>2</sup>. The XRD pattern shows the irradiation fluence dependant preferential orientation. Scanning electron microscope analysis displays fine blocks of material for pristine while partial agglomeration on irradiation. Notably, a large number of holes are present at the fluence of  $5\times10^{14}$  ions/cm<sup>2</sup>. The magnetization measurements performed in these samples exposes that the coercivity and remanence magnetization value increases due to the magnetocrystalline anisotropy up to the fluence of  $1\times10^{13}$  ions/cm<sup>2</sup>. At  $1\times10^{14}$  ions/cm<sup>2</sup> fluence, the induced thermal energy overcomes the magnetocrystalline anisotropy constant and causes a decrease in coercivity and remanence values. The saturation magnetization decreases up to the fluence of  $1\times10^{13}$  ions/cm<sup>2</sup> and then it increases for further irradiation. The change of crystalline orientation observed from XRD, the creation of holes from SEM and the change in magnetic properties are discussed on the basis of electro-phonon coupling and it invokes the thermal spike theory.

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#### 1. Introduction

Swift heavy ion (SHI) irradiation plays a vital role to change the physical and chemical properties in magnetic insulators [1–3]. When an energetic ion beam passes through a material, the modifications are caused due to the Coulombic interactions. The energy losses of the incident ion arise along its path in two ways: (i) electronic excitation and ionization i.e. electronic slowing down  $S_{\rm e}$  (dE/ dx<sub>e</sub>, or electronic energy loss – (dE/dx)<sub>e</sub>; and (ii) elastic collisions with the nucleus of the target atoms i.e. nuclear slowing down  $S_{\rm n}$ , or nuclear energy loss –  $(dE/dx)_{\rm n}$  [4]. Both electronic and nuclear energy loss depends on the target material, the mass of the projectile ion, its energy and fluence [5]. It is well established that electronic stopping power and nuclear energy play a vital role in the defect creation mechanism. SHI penetrates deep into the materials and produces a long narrow disordered zone and sometimes creates perceptible path way (holes) along its trajectory [6,7]. The nature of damaged structures depends upon the electronic energy loss  $S_e$  and the threshold value of electronic energy loss  $S_{eth}$ . When these values satisfy the condition  $S_e < S_{eth}$ , defects will be generated in the materials and modify the structural and magnetic properties [8]. In the electronic slowing-down regime  $S_{\rm e}\gg S_{\rm n}$ , most of the energy of the incident ions is transferred to the host electrons, results in a high electronic ionization (ionic spike) and/or a temperature release of electronic subsystem (thermal spike) [1].

Copper ferrite is unique among the common ferrites and is widely used as magnetic material at high frequencies due to its low cost and high performance in the soft magnetic field industries. It is used to fabricate ferrofluids, magnetic cores, magnetic refrigeration, optomagnetic devices and bubble memory devices [9,10]. Copper ferrite in its bulk form is a mixed spinel and the unit cell consists of eight formula units (8 × CuFe<sub>2</sub>O<sub>4</sub>). The 32 oxygen ions form a face centred cubic (fcc) lattice in which two kinds of interstitial sites are present, namely (i) 64 tetrahedral sites, surrounded by four oxygen (A sites) and (ii) 32 octahedral sites, surrounded by six oxygen ions (B sites) [11]. Various methods have been reported for preparation of ferrite nanoparticles [12–16]. In this paper co-precipitation technique has been used to synthesize the material because it is simple, rapid and one can control the particle size [17].

In the present study, we have investigated the change in crystallite nature, morphology and magnetic properties due to 150 MeV Ni<sup>11+</sup> ion irradiation at various fluences ( $10^{11}$ ,  $10^{12}$ ,  $10^{13}$ ,  $10^{14}$ ,  $5 \times 10^{14}$  ions/cm<sup>2</sup>) in spinel copper ferrite using X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometer (VSM) and reported.

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#### 2. Experimental

#### 2.1. Preparation of material

All chemicals and solvents used were of analar grade obtained from E. Merck. Ferrite nanoparticles of  $\text{CuFe}_2\text{O}_4$  were prepared by chemical co-precipitation route. In order to obtain the desired compositions, stoichiometric amounts of 0.5 M  $\text{Cu}(\text{NO}_3)_2\cdot 3\text{H}_2\text{O}$  and 1 M  $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$  were dissolved in 250 ml of double distilled water with constant stirring. The neutralization was carried out with 4 M sodium hydroxide solution. The reaction temperature was kept at 85 °C for one hour. The precipitate was thoroughly washed with distilled water till the pH value reaches 7. The product was dried in an oven at 100 °C for 12 h to remove the water contents. The out coming sample was grinded in a cleaned agate mortar and pestle, sintered at 250 °C for two and half hours and then cooled to room temperature with the same rate as that of heating. The final product was used for irradiation purpose.

#### 2.2. Irradiation experiment

For irradiation, the targets in the form of thin layer of ferrite material having thickness of about 20 mg/cm<sup>2</sup> were prepared by spreading fine copper ferrite powders in an aluminium foil, and uniform thickness was achieved by fixing the powder using liquid GE varnish. The irradiation experiment was performed using 150 MeV Ni<sup>11+</sup> ions as projectiles under high vacuum with the aluminium foil encrusted material. The beam current used was 5 pnA. From the stopping range of ions in matter (SRIM) calculations, the range of the projectile ions of Ni<sup>11+</sup> for 150 MeV Ni<sup>11+</sup> is 8.65 μm. In the present study, the ions are expected to pass through the material and there will be no implantation of these ions [18]. Using 150 MeV Ni<sup>11+</sup> swift heavy ion, CuFe<sub>2</sub>O<sub>4</sub> was irradiated with various fluences of  $1 \times 10^{11}$ ,  $1 \times 10^{12}$ ,  $1 \times 10^{13}$ ,  $1 \times 10^{14}$  and  $5 \times 10^{14}$ 10<sup>14</sup> ions/cm<sup>2</sup>. The ion beam was magnetically scanned over a  $1 \times 1$  cm<sup>2</sup> area covering the complete sample surface for uniform irradiation. The samples were mounted on a copper target ladder using conducting carbon tapes. To prevent sample heating during irradiation, the ion flux was kept low. After irradiation the material was collected from the aluminum foil and that was used for further analysis.

#### 2.3. Characterization techniques

The structural characterization of pristine and irradiated copper ferrite was carried out by analyzing the X-ray diffraction patterns, obtained using Cu K $\alpha$  radiation ( $\lambda$  = 1.5406 Å) in an X'Pert PRO multipurpose diffraction system in the  $2\theta$  range  $10^{\circ}$ – $70^{\circ}$ . JEOL JSM-5610 scanning electron microscope with an Energy dispersive X-ray analysis attachment was used to record the micrograph of pristine material to study the composition analysis. The magnetic studies were carried out using Vibrating Sample Magnetometer of Digital Measurement Systems – Model EV 9 for both unirradiated and irradiated samples at the applied field from +20,000 to –20,000 Oe.

#### 3. Results

#### 3.1. XRD analysis

The XRD pattern of pristine and  $Ni^{11+}$  ion irradiated copper ferrite material for various fluences are shown in Fig. 1. The pristine copper ferrite XRD has  $(3\,1\,1)$  peak and it is the prominent peak for the spinel structure. Along with that  $(1\,1\,1)$ ,  $(2\,2\,0)$ ,  $(2\,2\,2)$ ,  $(4\,0\,0)$  and  $(4\,4\,0)$  peaks are also noticed. The observed XRD

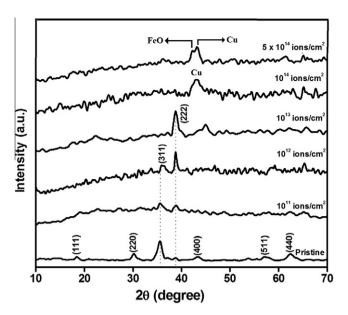


Fig. 1. XRD pattern of CuFe<sub>2</sub>O<sub>4</sub> at various fluencies.

**Table 1**XRD parameters for pristine CuFe<sub>2</sub>O<sub>4</sub>.

Sl. No.	hkl	2θ (°)	d-spacing (Å)	
			Measured	Standard
1	111	18.44	4.81	4.83
2	220	30.19	2.96	2.96
3	311	35.58	2.52	2.52
4	222	38.80	2.32	2.42
5	400	43.39	2.09	2.09
6	511	57.16	1.61	1.61
7	440	62.44	1.49	1.48

parameters are given in Table 1 and it is in well agreement with the standard JCPDS card No. 77-001. It proves that the pristine copper ferrite has spinel face centred cubic structure. The cell constant and cell volume of the pristine sample are calculated using UNIT CELL software [19] and found to be 8.352(9) Å and 582.5 Å<sup>3</sup> respectively. The crystallite sizes (D) for all the samples are calculated using Debye–Scherrer's formula [20],  $D = \frac{0.9\lambda}{\beta \cos \theta}$ , where  $\beta$  is the full width at half maximum of the peak in radians,  $\lambda$  is the wave length in nanometers,  $\theta$  the Bragg angle of the X-ray peak. The average crystallite size of pristine sample is found to be 10 nm. In the XRD pattern of the irradiated material the intensity of the prominent X-ray peak (3 1 1) is decreasing continuously on irradiation at the fluence of 10<sup>11</sup> and 10<sup>12</sup> ions/cm<sup>2</sup>. Moreover the X-ray peaks (1 1 1), (2 2 0), (4 0 0), (5 1 1) and (4 4 0) vanish on irradiation. The intensity of the X-ray peak (222) increases with the increase in fluence up to 10<sup>13</sup> ions/cm<sup>2</sup> and then suddenly vanishes at 10<sup>14</sup> ions/cm<sup>2</sup>. It indicates that irradiation influence the alignment of crystallites along [2 2 2] direction. Moreover the full-width at half maxima (FWHM) of this (222) peak is found to increase significantly with increasing irradiation ion fluence and their values are given in Table 2. The increase in intensity along with the peak broadening indicate the stress induced agglomeration due to swift heavy ion irradiation. On further irradiation, at the fluence of 1 and  $5 \times 10^{14}$  ions/cm<sup>2</sup>, all the X-ray peaks of CuFe<sub>2</sub>O<sub>4</sub> vanishes. However, a broad weak X-ray peak is observed at  $2\theta$  = 43.22° at the fluence of  $1\times 10^{14}\ \text{ions/cm}^2$  and it is in agreement with copper (1 1 1) X-ray peak. Mean while at  $5 \times 10^{14} \, \text{ions/cm}^2$ , two X-ray peaks overlapped having  $2\theta$  value at  $43.22^{\circ}$  and  $42.13^{\circ}$  is present and it is not due to copper ferrite material. On further analysis, it

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