



EFFECT OF SINTERING ON STRUCTURAL PROPERTIES OF MAGNESIUM FERRITE ($MgFe_2O_4$)

P. S. Bongale¹, R. M. Shedam², A. M. Bagwan³ and A. B. Gadkari⁴

Department of Physics and Electronics, GKG College, Kolhapur – 416012, (MS) India

bagwan.azeem.ab@gmail.com

Abstract:

Magnesium ferrite $MgFe_2O_4$ synthesized by oxalate co-precipitation method using high purity sulphates. The synthesized powder is characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), Fourier transform infrared radiation (FTIR) technique. The sample were sintered at different temp $400^\circ C$, $500^\circ C$, $600^\circ C$ for 5hr. The X-ray diffraction analysis confirms the formation of single phase cubic spinel structure of the sample. The Lattice constant, X-ray density, physical density, porosity, crystallite size, site radii (r_A , r_B) Bond length (A-O, B-O) were calculated. The grain size increase with temperature. The FTIR spectra show two strong absorption bands around the range of $350cm^{-1}$ and $800cm^{-1}$ to tetrahedral and octahedral respectively.

Keywords: Synthesis; crystallite size; XRD; Grain Size.

Introduction:

Ferrites are ferromagnetic oxides consists of iron oxide. Ferrite materials are extensively used in electronic microwave devices, computer memory chips, magnetic recording media etc. [1]. Depending on the crystal structure ferrites classify in to three classes such as spinel ferrite, garnet and hexagonal ferrite. Among the ferrites the class of spinel ferrites is a group of technologically important material [2]. Magnesium ferrite ($MgFe_2O_4$) is one of the important magnetic oxides with spinel structure. At low crystallite size, $MgFe_2O_4$ possesses unique super magnetic property at room temperature and it has promising application in transformer, ferro-fluids and magnetic cores of coils [3–5]. In addition, $MgFe_2O_4$ can be used in verity of applications including semiconductors [6], catalysts [7], gas sensors [8] and humidity sensors [9]. It is well known that the magnetic properties of the ferrite depend on microstructure, size and morphology of particles which depends on the preparation techniques.

In the present study, we report preparation of $MgFe_2O_4$ by oxalate co-precipitation method and their characterization by XRD, SEM and FTIR techniques.

Experimental:

The $MgFe_2O_4$ ferrites were prepared by the oxalate co-precipitation method. The high purity starting materials $MgSO_4 \cdot 7H_2O$ (purity 99.99%, Sd fine), and $FeSO_4 \cdot 7H_2O$ (purity 99.5%, Thomas Baker) were used for preparation of samples. These chemicals were weighed in desired stoichiometric proportion and dissolved in distilled water. The pH of the solution was maintained by drop wise addition of concentrated H_2SO_4 [10]. The precipitating reagent was prepared in distilled water by adding required proportion of AR grade ammonium oxalate. The

precipitating reagent was added by drop into metal sulphate solution with constant stirring until the process of precipitation was complete. The precipitate along with solution was digested on sand bath for 1 h in order to settle down the precipitate at the bottom of the beaker. The precipitate was filtered by Whatman filter paper no. 41 using suction flask operating with the vacuum pump. The precipitate was thoroughly washed with distilled water in order to remove sulphate ions. The absence of sulphate ion in the filtrate was confirmed by barium chloride test. The powders were finally sintered at $400^\circ C$, $500^\circ C$, $600^\circ C$ for 5 h followed by slow cooling in the furnace. Then the samples were characterized by XRD, SEM and FTIR techniques.

The X-ray powder diffraction (XRD) patterns were obtained at room temperature by using a philips PW-3710 X-ray powder diffractometer operated at 40kV and 30mA using $CuK\alpha$ radiation ($k = 1.5424 \text{ \AA}$). The diffraction patterns were recorded at step size of 0.02 in angular range $20^\circ - 80^\circ$ for (2h). The crystallite size was calculated by Scherrer formula. The scanning electron microscopy was carried to analyze microstructure of fractured surface the power using the SEM, JEOL-JSM 6360 model, Japan. Infrared absorption spectra of powdered samples were recorded in the range of $350-800 \text{ cm}^{-1}$ using Perkin-Elmer FTIR spectrum one spectrometer by KBr pellet technique.

Results and Discussion:

The X-ray diffraction pattern of $MgFe_2O_4$ at $500^\circ C$ is presented in Fig. 1. The X-ray analysis reveals the formation of single phase cubic spinel structure of all the samples. The XRD pattern shows that peak width decreases with intensity with increasing sintering temperature. The diffraction peaks are corresponding to (220), (311), (400), (422), (511)

and (440) planes. The calculated and observed values of inter planer distances (d) are in good agreement with each other. The lattice constant 'a' (Å) for all the samples was calculated by using the prominent (311) XRD peak using the equation,

$$a = \sqrt{h^2 + k^2 + l^2} \tag{1}$$

The calculated value of lattice constant is presented in the table. It is observed that the lattice constant decreases with increasing temperature. Similar behavior is also reported by S. Maensiri *et al.* [11]. The average crystallite size for all samples using (311) plane was determined by Debye Scherrer formula [12].

$$D = \frac{0.94\lambda}{\beta \cos \theta} \tag{2}$$

Where, λ is the wavelength of X-ray, β is the full width at half maxima, and θ is the Bragg's Diffraction angle. The average crystallite size 'D' is presented in the Table. From this table it is observed that, the crystallite size of the sample lies in the nano particle range of 37.18 to 38.51 nm and increases with temperature [7,13].

$$\rho_x = \frac{8M}{Na^3} \tag{3}$$

Where, M is the molecular weight of the sample. N is the Avogadro's number and 'a' is the lattice constant. The bond lengths (A-O, B-O) and ionic radii (r_A , r_B), on tetrahedral (A) and octahedral (B) sites of cubic spinel structure are calculated by using the following equations suggested by Standely [14],

$$A - O = (u - 1/4)a\sqrt{3} \tag{4}$$

$$B - O = (5/8 - u)a \tag{5}$$

$$r_A = (u - 1/4)a\sqrt{3} - r(O^{2-}) \tag{6}$$

$$r_B = (5/8 - u)a - r(O^{2-}) \tag{7}$$

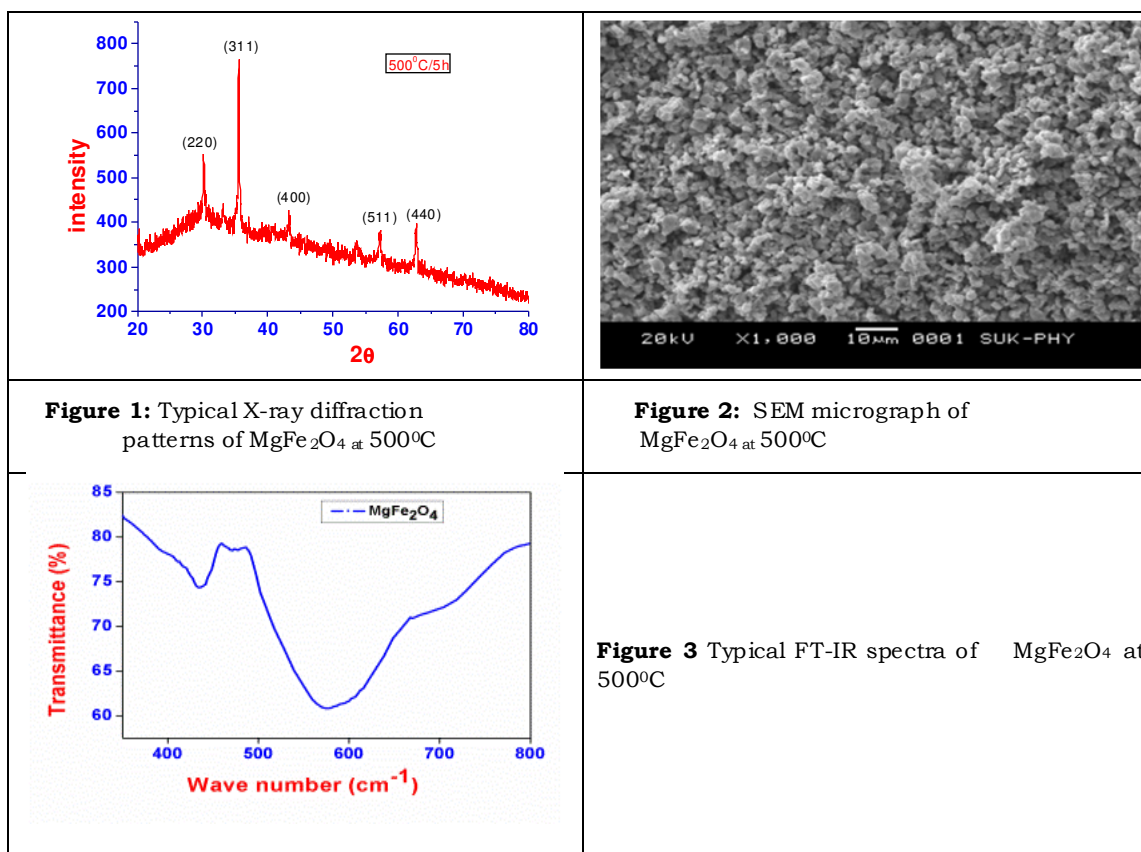
where 'a' is the lattice constant, $r(O^{2-})$ is the radius of oxygen ion (1.35 Å) and 'u' is the oxygen ion parameter (u =0.382 for MgFe₂O₄). The bond lengths (A-O, B-O) are ionic radii (r_A , r_B) on tetrahedral and octahedral sites for each composition are summarized in Table 1. The ionic radii on A-site and B-site are found to be in good agreement with those reported by Standley [14]. It can be seen that the bond lengths (A-O, B-O) and ionic radii (r_A , r_B) It is well known that there is co-relation between ionic radius and lattice constant.

SEM micrograph of fractured surface of pallets sintered at 500°C for MgFe₂O₄. The average grain size of the sample is calculated by linear intercept method and presented in table. The grain size lies in the range 0.15 to 0.18 μm. It is observed that average grain size of the sample increases with increase in temperature. It is noticed that their change in morphology of particle with increase in temperature [11].

FTIR analysis of Magnesium ferrite was taken in the range 350 to 800 cm⁻¹. The FTIR spectra of sample sintered at 500°C is shown in Fig.3. The typical low frequency band at around 469.61cm⁻¹ corresponds to Fe-O vibration in tetrahedral and octahedral size and the band at around found to be better than developed through other method [15]. The higher frequency absorption band (ν_1) is caused by scratching vibration of tetrahedral metal oxygen bond and lower frequency bond (ν_2) is caused by metal - oxygen vibrations in octahedral position the band position of ν_1, ν_2 are presented in the table.

Table: 1. Parameters of MgFe₂O₄ sintered at 400°C, 500°C and 600°C.

Temp.	Crystallite size (nm)	Lattice constant (Å)	Grain Size (μm)	Absorption bands		X-ray Density (ρ_x)	Ionic radii (Å)		Bond length (Å)	
				ν_1	ν_2		r_A	r_B	A-O	B-O
400	37.18	8.36	0.15	469.61	424.61	4.53	0.562	0.683	1.912	2.033
500	37.21	8.34	0.17	565.12	435.81	5.64	0.556	0.676	1.906	2.026
600	38.51	8.31	0.18	571.54	456.37	6.30	0.558	0.663	1.901	2.016



Conclusion:

The XRD confirms single phase cubic spinel structure. The crystal size lies in the range is 37.18nm to 38.51nm and increases with increase in sintering temperature of the samples. Lattice constant decreases with increase in temperature. The grain size increases with increase in temperature. The FTIR spectra show two strong absorption bands around the range of $350cm^{-1}$ and $800cm^{-1}$.

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