Electrical and magnetic properties of nano-sized magnesium ferrite

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Abstract. Nano-sized magnesium ferrite was synthesized using sol-gel techniques. Structural characterization was done using X-ray diffractometer and Fourier Transform Infrared Spectrometer. Vibration Sample Magnetometer was used to record the magnetic measurements. XRD analysis reveals the prepared sample is single phasic without any impurity. Particle size calculation shows the average crystallite size of the sample is 19nm. FTIR analysis confirmed spinel structure of the prepared samples. Magnetic measurement study shows that the sample is ferromagnetic with high degree of isotropy. Hysteresis loop was traced at temperatures 100K and 300K. DC electrical resistivity measurements show semiconducting nature of the sample.

1. Introduction
Spinel magnesium ferrite is having a wide range of applications as catalyst, humidity sensors and magnetic memory devices. Nowadays nanostructured spinel ferrites has invoked more attention due to its novel material properties when compared with its bulk counter parts [1-2]. The interesting physicochemical properties of ferro-spinels arise from their ability to distribute the cations among the available tetrahedral (A) and octahedral [B] sites[3-4]. Magnesium ferrite is a pertinent magnetic material showing inverse spinel structure for wide applications owing to its high resistivity, high Curie temperature and environmental stability. The degree of inversion of spinel structure depends upon the heat treatment due to high diffusibility of Mg$^{2+}$ ions$^7$. At present, several chemical methods including the solid state reaction, co-precipitation, sol–gel, citrate method have been developed to synthesize small particle sized ferrites [5-7]. Due to good stoichiometric control and production of ultrafine particles in nanorange at relatively low temperature, sol-gel technique is an attractive method of preparation [8]. In addition to this, sol–gel-derived nanoparticles generally possess good chemical homogeneity, high purity and lower calcination temperature [9-10].

2. Experimental methods
2.1 Synthesis
Sol-gel combustion method was used for preparation of magnesium ferrite nanoparticles. AR grade magnesium nitrate and ferric nitrate were used as chemical precursors in ethylene glycol as solvent. Nitrates in the required stoichiometric ratio were dissolved in minimum amount of ethylene glycol at room temperature and the sol was heated at 60°C to obtain a wet gel. This gel was dried at 120°C, which self ignites to form a fluffy product. This is then ground to form fine powder and was fired at
400°C for 3 hours. For doing the d.c. conductivity studies the samples were pelletized in cylindrical disc-shape using a hydraulic press by applying a uniform pressure of 5 tons and were sintered at 500°C in the furnace for 24 hours.

2.2 Characterization
Structural characterization was done using XRD analysis. X-ray powder diffractometer (Rigaku make RINT 2000) with Cu-Kα radiation (λ=1.54059Å) at 40kV and 30mA performed a scanning from 20° to 80° at a step size of 0.02° per second for each sample. The crystal structure, lattice parameter, crystallite size, X-ray density, bulk density and porosity were determined from the XRD pattern. The Fourier Transform Infrared (FTIR) absorption spectra of the samples were recorded using FTIR spectrometer (Thermo Nicolet, Avatar 370) in the wave number range 4000 to 400 cm⁻¹ with Potassium bromide (KBr) as solvent. Magnetic characterization of the sample was carried out by vibration sample magnetometer at temperatures 40K and 303K with maximum applied field of 20kOe using Lakeshore VSM 7410. Keithley 6221 current source and 2182A voltmeter was used to determine the d.c. conductivity of the samples at temperature from 300K to 400K.

3. Results and discussion
3.1 Structural characterization
3.1.1 X-ray diffraction analysis The XRD pattern of the prepared samples S1 is shown in figure 1. The XRD pattern confirms the formation of single phase fcc spinel structure with no extra lines corresponding to any other crystallographic phase (compared with JCPD card no: 88-1935). The lattice constant ‘a’ calculated using Bragg’s equation for prominent (311) peak is 8.40Å which is comparable with reported value of 8.37Å[11]. The average crystallite size calculated from the XRD peak broadening of the peaks using Scherrer formula [14] corrected for micro strain estimated from the Hall-Williamson plot (figure 2) [12] is 19.8nm. Calculated value of x-ray density is 4.47 gm cm⁻³.

![Figure 1. XRD pattern of MgFe₂O₄](image1)
![Figure 2. Hall-Williamson plot of MgFe₂O₄ from XRD](image2)

3.1.2 FTIR analysis IR spectra of MgFe₂O₄ ferrite system is shown in figure 3. FTIR spectrum analysis helps us to confirm the formation of spinel structure in ferrite ferrites. Two major absorption bands are found in the range 600-550 cm⁻¹ and 450-385 cm⁻¹ which confirm the spinel structure formation of the prepared samples [13]. The bands corresponding to 3400cm⁻¹ and 1400 cm⁻¹ represent stretching and bending vibrations of H-O-H which indicates the presence of free or absorbed water in the samples. This is confirmed in the d.c. conductivity studies also.
3.2 Electrical characterization

3.2.1 DC conductivity studies  The temperature dependence of DC conductivity in magnesium ferrite is studied in the temperature range 300K to 473K and is shown in figure 4. The variation of conductivity with temperature shows semiconducting nature of the ferrite. But initially till the temperature reaches 323K from room temperature there is a sudden decrease in $\sigma_{dc}$ value which is due the absorbed water in the sample. The dc conductivity of the sample varies from $5 \times 10^{-7}$ to $5 \times 10^{-4}$ S/m in the temperature range 323K-473K. The conductivity in ferrites can be explained based on the popular electron hopping model [18]. According to this model, the conductivity is mainly due to the hopping of electrons between $\text{Fe}^{2+}$ and $\text{Fe}^{3+}$ ions present at the octahedral B sites.

3.2.2 Magnetic properties Magnetic characterizations of the samples were carried out by vibration sample magnetometer at room temperature with maximum applied field of 20kOe. Typical magnetic hysteresis loop of sample S1 at temperatures 303K and 40K is shown in figure 5. The saturation magnetization ($M_s$), coercivity ($H_c$) and remanance ($M_r$) of the samples are shown in table 1.
Table 1. Saturation magnetization ($M_s$), coercivity ($H_c$) and remanence ($M_r$) of MgFe$_2$O$_4$ at temperatures 40K and 303K

| Sample at Temperature | Saturation magnetization ($M_s$) in emu/gm | Coercivity ($H_c$) in Oe | Remanence ($M_r$) in emu/gm | $M_r/M_s$ |
|-----------------------|------------------------------------------|--------------------------|----------------------------|-----------|
| 303K                  | 21.4                                     | 349.9                    | 8.2                        | 0.38      |
| 40K                   | 25.8                                     | 461                      | 12.7                       | 0.49      |

The remanent ratio $R=M_r/M_s$ shows the ease with which the direction of magnetization reorients to the nearest easy axis direction after the field is removed. The low value of $R$ indicates the isotropic nature of the material [17]. According to Stoner Wohlfarth model [19], a theoretical value of $M_r/M_s$ is 0.5 for non interacting uniaxial single domain particles with the easy axis being randomly oriented. $M_r/M_s$ value observed is found to be 0.49 for sample at low temperature which shows its isotropic nature.

4. Conclusions

Magnesium ferrite particles were prepared successfully using the sol gel method. The XRD pattern confirms the formation of single phase fcc spinel structure with no extra lines corresponding to any other crystallographic phase. FTIR spectrum confirms the spinel formation and presence of water in the sample. The temperature variation of dc conductivity indicated the semiconducting behavior of the ferrite. Decrease in conductivity till temperature 323K is due to water absorbed in the sample. The magnetic study reveals that the sample is ferromagnetic at room temperature and low temperature (40K). At low temperature the sample is highly isotropic in nature.

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References

[1] Santin M, Montana S, and Amporn W, 2009 Nanoscale Res. Lett. 4:221-228.
[2] S. Wang, Y. Zhou, Guan, B. Ding, 2008 Nanoscale Res. Lett. 3, 289
[3] G. Blass, 1965 Philips Res. Rept 20 pp 528.
[4] J.B. Goodenough, 1963 Mag. Chem. Bond, John Wiley, New York, London 120.
[5] A. Verma, T.C. Goel, R.G. Mendiratta, R.G. Gupta, 1999 J. Magn. Magn. Mater. 192 pp 271–276.
[6] P.P. Hankare, P. D. Kamble, M. R. Kadam, K. S. Rane, P. N. Vasambekar, 2007 Mater. Lett. 61:2769–2771.
[7] Cui-Ping L, Ming-Wei L, Zhong C, Juan-Ru H, Yi-Ling T, Tong Lin, Wen-Bo Mi, 2007 J. Mater. Sci. 42, 6133–38.
[8] Manish Srivastava, S. Chaubey, Animesh K Ojha, 2009, Materi. Chem. Phys. 118:1, 174–180
[9] Abdel-Mohsen FF, Emira HS 2005, Pigment Resin Technol 34 pp 312
[10] Cui H, Zayat M, Levy D 2005 J Sol–Gel Sci Technol 35:175
[11] Viswanathan B, Murthy VRK, Ferrite material science & technology, 1990 Narosa Publishing, p6.
[12] Klug H P & Alexander L e 1974 X-ray diffraction procedures for polycrystalline & amorphous materials (Wiley: New York)
[13] Waldron R D, Infrared Spectroscopy of ferrites, Phy rev 1955,99,1727-35.
[14] Binu PJ, Smitha Thankachan, Sheena X, E M Mohammed, 2011 Phys. Scr. 84 045702 (6pp)
[15] Franco, Jr. and M. S. Silva, 2011 Journal of Applied Physics 109, 07B505
[16] S.S.Khot, N.S.Shinde, B P. Ladaonkar, B.B.Kale and S C. Watawe, Advances in Applied Science Research, 2 (4),460-471
[17] Shirshath S E, Toksha B G and Jadhav K M 2009 Mater. Chem. Phys. 117, 163-8
[18] Veena G E, Al-Omari I A, Malini K A, Joy P A, Sakti Kumar D, Yasuhiko Y, Anantharaman M R 2008 J. Magn. Magn. Mater. 321 1092-9
[19] E. C. Stoner , E. P. Wohlfarth, Philos. 1948 Trans. R. Soc. A 240, 599-642