

INFLUENCE OF ANNEALING TEMPERATURE ON THE PROPERTIES OF MAGNESIUM FERRITE SYNTHESIZED BY SOLUTION COMBUSTION METHOD

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Abstract

Magnesium ferrite was prepared by solution combustion method using glycine as the fuel. The influence of annealing temperature on the structural properties was done using PXRD and FT-IR. The crystallite size sample show sigmoidal type behavior and the value of lattice parameter close to bulk value. Magnesium ferrite annealed at 1173K has a value saturation magnetization 31 emu/g indicates the phase purity of the sample prepared by solution combustion method

Keywords: *Solution combustion, Spinel ferrites, X-ray diffraction.*

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Introduction

Nanosized materials play an important role in the physical, chemical and biological field due to its unexpected properties compared to its bulk counterpart. Among these materials, oxides with spinel structure with chemical formula AB_2O_4 have been intensively studied due to its technological importance. Variation in distribution of cations across the different symmetric sites presents in the spinel influence the properties. In normal spinel the tetrahedral sites are (A-site) occupied by a divalent cation and the octahedral sites (B-site) are occupied by a trivalent cation. The entire divalent cation occupies at B-site then the inverse spinel forms[1]. Magnesium ferrite has near inverse spinel in which 90% of magnesium ions occupy at B-site. This percentage of octahedral occupancy magnesium indirectly introduces magnetism in this ferrite.

The method of preparation has direct influence on the cation distribution and hence influences the properties. There are number of methods such as co precipitation, sol-gel, sono chemical, micro emulsion, solid state reaction, solution combustion etc[2], were used by different researchers around the globe for the preparation of magnesium ferrites. In which solution combustion is the one of the most suitable method which results a high purity and highly crystalline nano powders[3]. In the present study we report influence of annealing temperature on the properties of $MgFe_2O_4$ prepared by the solution combustion method using glycine as the fuel.

Experimental

Analytical grade ferric nitrate nonahydrate and magnesium nitrate hexahydrate were used as oxidants and glycine as a fuel to accomplish the combustion reaction. In this preparation the stoichiometric amount of metal nitrates were dissolved in distilled water. The amount of fuel was kept at particular value to maintain the fuel to oxidizer ratio at 0.8. The mixture was turned into the slurry due to hygroscopic nature of metal nitrates. The beaker was then kept on hot plate preheated at 473 K. During combustion, the spark was occurred at one position which spread over the entire volume resulting brown fluffy product that was powdered using mortar and pestle[4]. The as prepared samples were divided into four and annealed at four different temperatures (573, 773, 973 and 1173 K) for 2hrs.

Powder X-ray diffraction (PXRD) pattern of all the samples were recorded with the help of Rigaku Miniflex-600 X-ray diffractometer using $Cu-K\alpha$ radiation. Rietveld refinement was carried out using MAUD software to get structural parameters[5]. FTIR spectra of all the samples were carried out using Agilent Cary 630 FTIR spectrometer using KBr optics, in the range 4000-400 cm^{-1} . DC magnetization of 1173 K annealed sample was performed using Lakeshore 7304 vibrating sample magnetometer.

Results and discussion

Rietveld refined PXRD pattern of the samples were shown in figure 1. All samples have peaks corresponds to spinel phase only. The lattice parameter and crystallite size of the samples were obtained from the refinement and was given in table 1. The lattice parameter of the samples are very close to bulk value (8.389 Å)[6]. On the other hand crystallite size show gradual increase with increase in annealing temperature and become constant (Figure 2). The increase in temperature increases the diffusion of atoms at the boundary of the particles that cause increase in crystallite size.

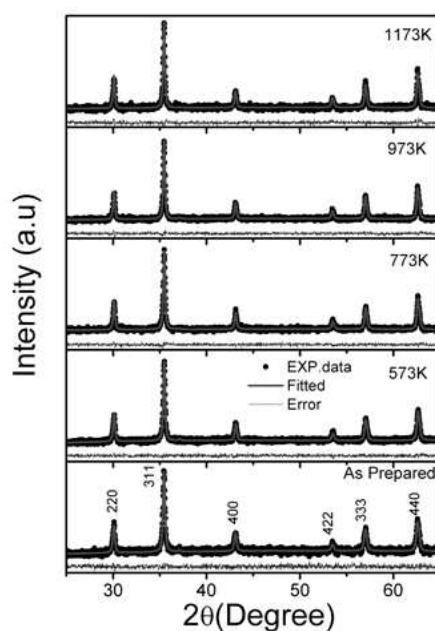


Figure.1 PXRD pattern of the magnesium ferrite samples.

Table.1 Variation in lattice parameter and crystallite size with annealing temperature.

Samples	Lattice parameter (Å)	Crystallite size (nm)
Asprepared	8.388	69
573 K	8.383	81
773 K	8.383	84
973 K	8.386	92
1173 K	8.386	92

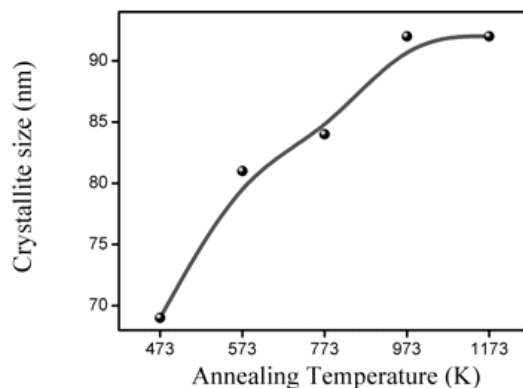


Figure 2. Variation in crystallite size vs. annealing temperature

The FTIR spectra of the samples were shown in figure 3. The spinel phase has mainly two stretching mode corresponds to A and B sites. In the present samples also stretching mode sue tetrahedral site band at 570 cm^{-1} . The mode due octahedral were not completely visible but the spectra show absorption near 400 cm^{-1} .

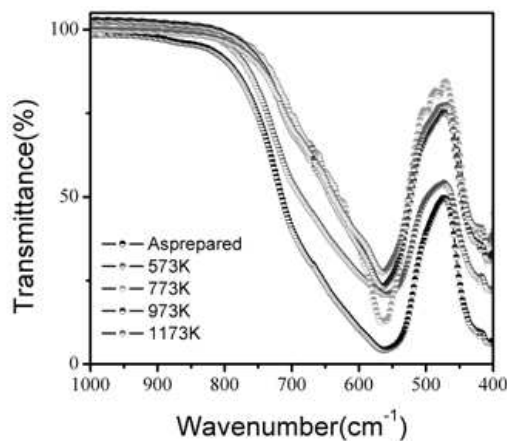


Figure.3 FT-IR spectra of as prepared and annealed MgFe₂O₄

Room temperature hysteresis curve of the annealed (1173 K) sample were shown in figure 4. The magnetic properties of the sample were obtained from the room temperature curve. The value saturation magnetization is 30 emu/g and coercivity 90 Oe. The bulk magnesium ferrite has saturation magnetization 31 emu/g [7]. The sample prepared by this method has similar magnetic structure as that of bulk magnesium ferrite.

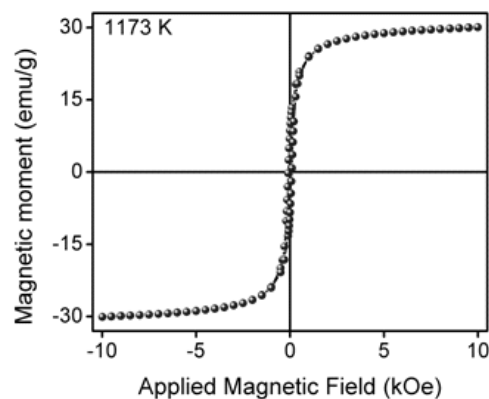


Figure.3 Room temperature hysteresis curve of samples annealed at 1173 K.

Conclusion

Magnesium ferrite samples were prepared by solution combustion method using glycine as fuel. The spinel phase was obtained in all the samples. Lattice parameter show a little variation with annealing temperature, but crystallite size gradually increases. The value of saturation magnetization of as prepared and annealed samples were close to the bulk value i.e. 31 emu/g. Thus, using solution combustion method nano sized samples can be obtained with magnetic properties comparable to that of bulk counterpart.

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References

1. J. Smit, H.P.J. Wijn, Ferrites: Physical properties of ferromagnetic oxides in relation to their technical application, Philips' technical library, Eindhoven, 1959.
2. J. Chandradass, A.H. Jadhav, K. Hyeon, H. Kim, Influence of processing methodology on the structural and magnetic behavior of $MgFe_2O_4$ nanopowders, *J. Alloys Compd.* 517 (2012) 164-169.
3. A. Varma, A.S. Mukasyan, A.S. Rogachev, K. V Manukyan, Solution Combustion Synthesis of Nanoscale Materials, *Chem. Rev.* 116 (2016) 14493-14586.
4. V.D. Sudheesh, J. Nehra, A. Vinesh, V. Sebastian, N. Lakshmi, D.P. Dutta, V.R. Reddy, K. Venugopalan, A. Gupta, Investigation of structural and magnetic properties of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nano powders prepared by self combustion method, *Mater. Res. Bull.* 48 (2013) 698-704.

5. L. Lutterotti, S. Matthies, H.-R. Wenk, A.S. Schultz, J.W.J. Richardson, Combined texture and structure analysis of deformed limestone from time-of-flight neutron diffraction spectra, *J. Appl. Phys.* 81 (1997) 594-560.
- 6 C.M.B. Henderson, J.M. Charnock, D.A. Plant, Cation occupancies in Mg , Co , Ni , Zn , Al ferrite spinels?: a multi-element EXAFS study, *J. Phys. Condens. Matter.* 19 (2007) 76214.
- 7 B.D. Cullity, C.D. Graham, *Introduction to Magnetic Materials (2nd Edition)*, Second, John Wiley & Sons, Inc., Hoboken, New Jersey, 2009.