

Synthesis and structural properties of Mg-Mn ferrites prepared by chemical co-precipitation method.

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Abstract: In the present study, $\text{Mg}_x\text{Mn}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.00, 0.01, 0.03, 0.05$) ferrites were prepared by chemical co precipitation method using ammonia as a precipitating agent. XRD study of prepared samples reveals that all the samples have single phase cubic spinel structure. Broad peaks in XRD studies suggest that the prepared samples are in nano regime. Electron microscopic analysis confirm the nano-crystalline nature of prepared samples.

1. Introduction

An ideal ferrite with all its characteristics properties improved and is capable of covering a complete spectrum of applications is impossible to prepare. It is found that on substitution of impurity ions a few properties improved but some of other are adversely effected. Therefore, a compromise has to be made in improving one property at the expense of other by substituting suitable ions in various ferrites. The properties of ferrites are highly sensitive to the type and amount of impurity. A number of workers have studied the effect of divalent, trivalent, tetravalent impurities on their magnetic properties [1-4]. The Mg-Mn ferrites prepared by the chemical co-precipitation techniques have been studied by several workers [5-8]. These ferrites highly suitable for memory and switching circuits in digital computers and phase shifters as they have characteristic rectangular hysteresis loop, The micro-structural properties of the Mg-Mn ferrites can be upgraded by preparing these ferrites by various preparation methods. The aim of present investigations is to synthesis Mg-Mn ferrite using chemical co-precipitation method and studying its structural properties using XRD, SEM and TEM.

2. Experimental:

All the compositions of nanocrystalline $Mg_xMn_{1-x}Fe_2O_4$ ($x = 0.00, 0.01, 0.03, 0.05$) system were prepared from Merck Germany GR grade chemicals viz. $Mg(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, $Mn(NO_3)_2 \cdot 6H_2O$ and aqueous NH_3 (Merck India, 30%). The samples were prepared using stoichiometric quantities of $Mg(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ then dissolving them separately in 100mL deionized water and adding simultaneously into a flask containing 200mL deionized water. Ammonia solution was added drop wise till the pH value 10 was attained. The solution was continuously stirred by a magnetic stirrer for 1 h and aged at room temperature overnight. The precipitates were filtered and washed with deionized water and then dried at $120^\circ C$ for 16 h in a hot air oven. The dried samples were calcined at $950^\circ C$ in air in a tube furnace programmed at a fixed heating rate of $5^\circ C/min$ for 8 h. The Mn doped derivatives of $Mg_xMn_{1-x}Fe_2O_4$ system were also prepared by adding the appropriate stoichiometric quantities of dopant salt $Mn(NO_3)_2 \cdot 6H_2O$ and following the same procedure as stated above.

3. Results & discussions

X-ray diffraction patterns were obtained for all the $Mg_xMn_{1-x}Fe_2O_4$ ($x = 0.00, 0.01, 0.03, 0.05$) samples prepared by chemical co-precipitation method. The structural analysis and detection of phase present in these ferrites samples was done using Rigaku –Denki X-ray Diffractometer (Cu-K α , $\lambda = 1.154 \text{ \AA}$). The variation of x-ray intensities were studied between the ranges of 2θ from 10 to 80° . All the samples showed single phase spinel structure with several broad peaks. The most intense peak was observed at $2\theta = 36.75^\circ$. The lattice parameter of the $Mg_xMn_{1-x}Fe_2O_4$ ferrites was found to reduce progressively from 8.482 \AA to 8.317 \AA with the increase in the concentration of the Mg^{2+} ions from $x = 0.00$ to $x = 0.05$. The decrease in the lattice parameter as tabulated in Table 1 is attributed to the replacement of the Mn^{2+} ions of ionic radius 0.91 \AA , by the Mg^{2+} ions of smaller ionic radius 0.78 \AA . The average grain size was found using Scherrer's formula, $D = K\lambda/\beta \cos \theta$, where β is the full width at half maximum (FWHM) of diffraction peak, λ is the wavelength of X-ray, θ is the angle of diffraction, and K is the Scherrer's constant of the order of unity for usual crystal. However, in case of nanoparticles, sizes calculated from Scherrer's formula can vary from the real particle size because nanoparticles often produce associated structures [9]. The average grain thus found varies from 9 to 6 nm in the present studies.

Microstructural analysis of $Mg_xMn_{1-x}Fe_2O_4$ ($x = 0.00, 0.01, 0.03, 0.05$) ferrite samples prepared by chemical co-precipitation exhibited the formation of spherical shape grains, hence revealing the

crystalline nature of the material. The average grain size has shown a significant decrease with increase in concentration as observed from SEM images shown in figures 2 and 3. This was in accordance with the XRD results where crystallite size was found to be decreasing. This can be attributed to the dissolution of grain boundaries due to the confinement of the heat energy released during sample preparation and thus forming small grains. A similar decrease in grain growth was evidenced from TEM analysis in the prepared samples. Here increasing dopant concentration was found to have significant decreasing effect on the grain size.

4. Conclusion

Chemical co-precipitation method is a very suitable technique for the formation of ferrites at relevant temperatures. Ferrites made by this technique have remarkably shown fine spinel crystalline structure in all the prepared samples. The same microstructural properties were observed in scanning electron microscopy (SEM) and Transmission electron microscopy (TEM) with decreasing grain size when concentration increases which was also revealed from X-ray diffraction studies. Hence we can conclude that this technique is very useful for the fabrication of Mn-Mg based ferrites for application purpose where magnetic properties and electrical properties can be further explored.

5. References

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Table 1. Variation of lattice constant and crystallite size with Mn concentration in $\text{Mn}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$

Concentration (X)	Lattice Constant (Å)	Crystallite Size (nm)
0.00	8.384	8.640
0.01	8.391	8.590
0.03	8.406	7.982
0.05	8.429	6.513

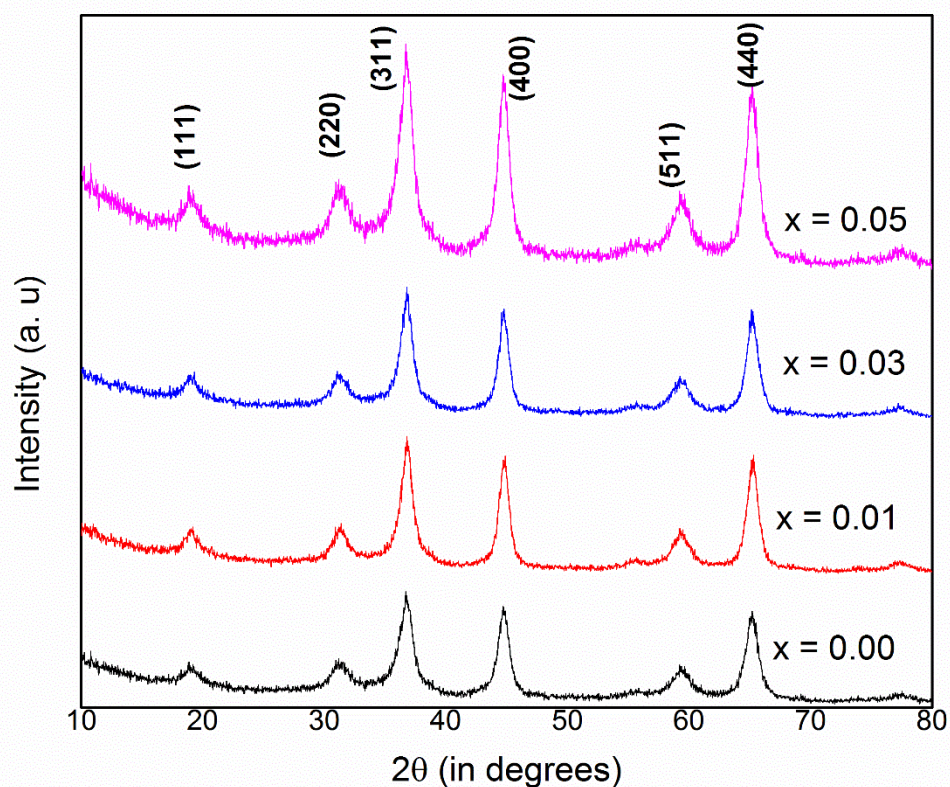


Figure 1: X-ray diffraction studies of $\text{Mn}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.00, 0.01, 0.03, 0.05$)

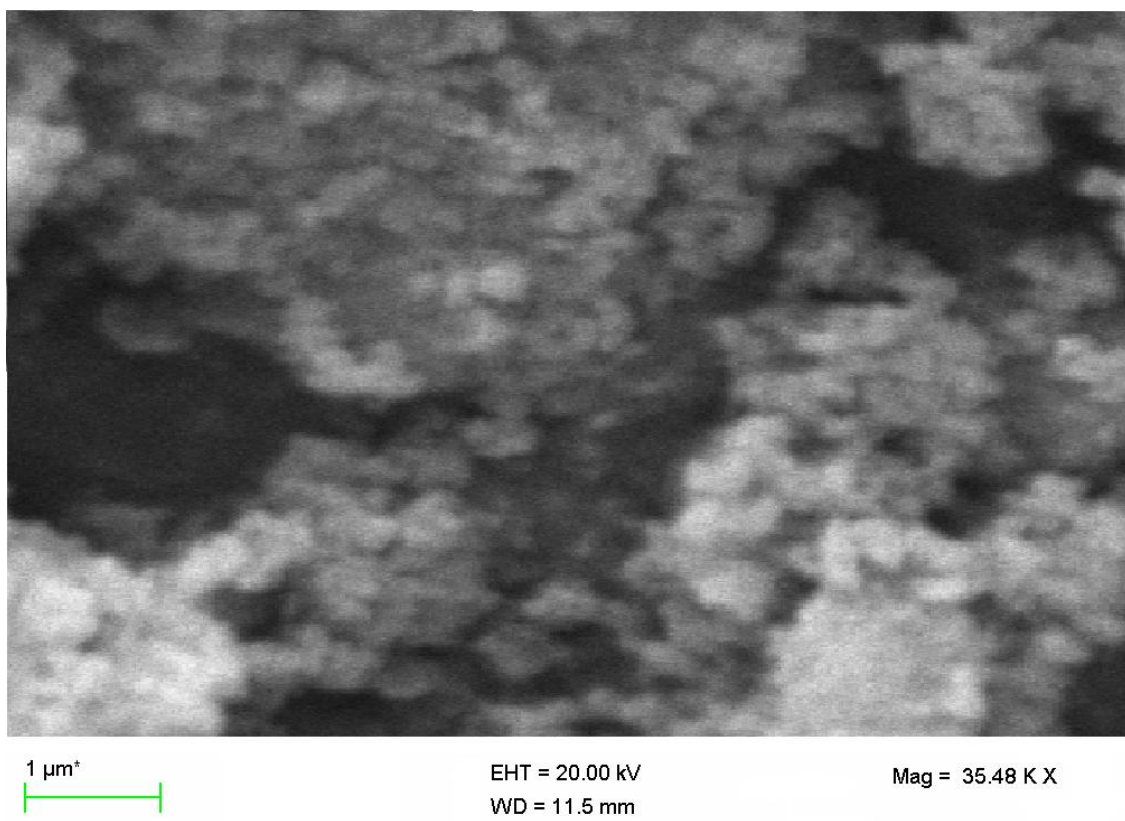


Figure 2: Scanning Electron Microscopy of $\text{Mn}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.05$)

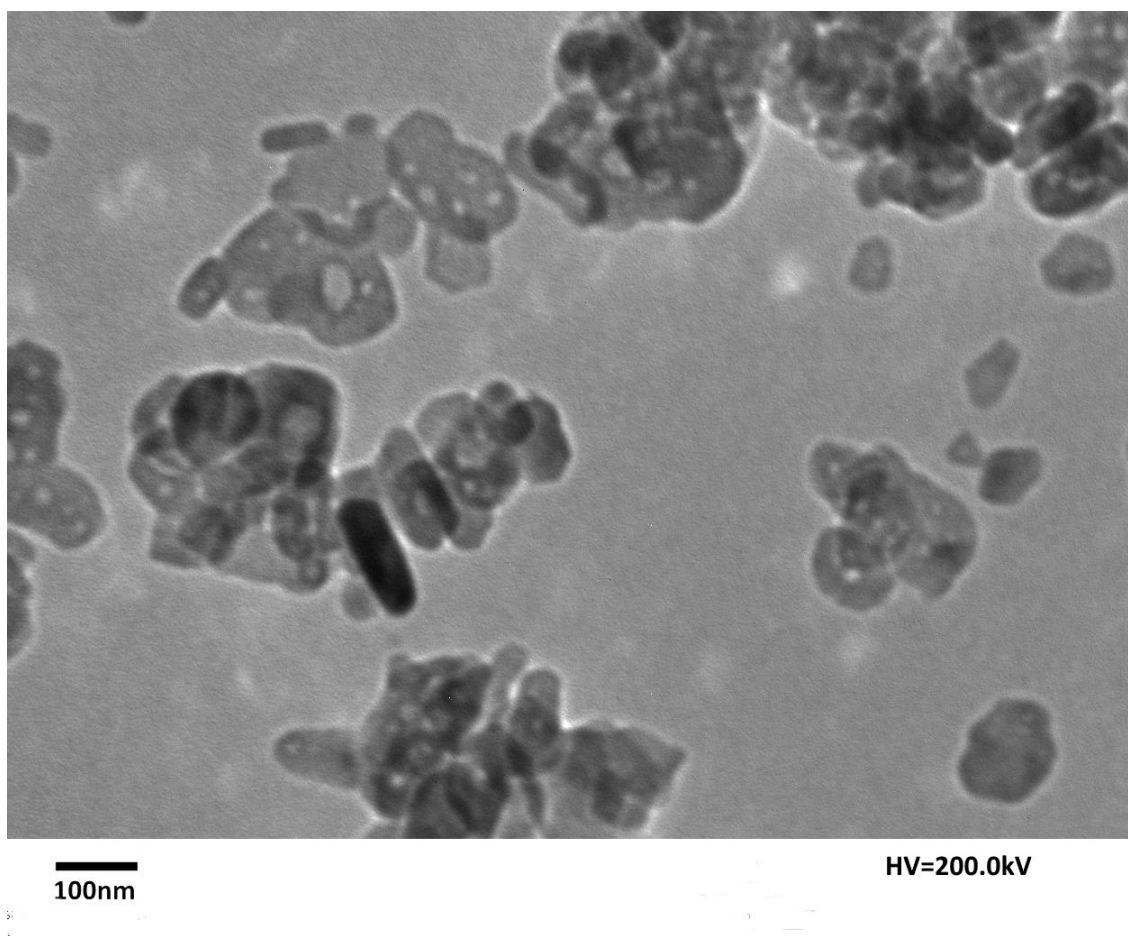


Figure 3: Transmission Electron Microscopy of $\text{Mn}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.05$)