Mössbauer Studies of Ferrite Nanoparticles Prepared by Coprecipitation Method

Sanjib Dey

Department of Chemistry, Y.S. Palpara Mahavidyalaya, Palpara 721458, Purba Medinipur, West Bengal, India Corresponding Author: Sanjib Dey

Abstract: Nanocrystalline $C_{0,2}Z_{n_0,8}Fe_2O_4$ sample was prepared by Co-precipitation method and sintered at 573K. X-ray and Transmission electron microscopy show that the samples are nanocrystalline spinel oxides with an average crystallite size of about 4 nm. Magnetization measurements at 0.1 T show that the $C_{0,2}Z_{n_0,8}Fe_2O_4$ is superparamagnetic having a blocking temperature 73K. The Mössbauer spectra of $C_{0,2}Z_{n_0,8}Fe_2O_4$ sample measured at 55K, 125K and 303K. The spectrum at 55K and show hyperfine split sextet patterns. When the temperature is increased to 125K, a paramagnetic doublet appears at the centre of the sextet. At 303K also only the paramagnetic doublet is present. These results indicate that $C_{0,2}Z_{n_0,8}Fe_2O_4$ is superparamagnetic below 73K. This has also been indicated in the magnetization measurements. **Key words:** Nanocrystalline, Superparamagnetism, Blocking temperature, Mössbauer spectra

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I. Introduction

Spinel ferrite nanoparticles have been extensively investigated in recent years not only due to their unusual properties different from their corresponding bulk materials, but also from their technological applications (1-3). The interesting and useful magnetic properties of the spinel ferrites are governed by the choice of the cations along with Fe^{2+} , Fe^{3+} ions, and their distribution between tetrahedral (A) and octahedral (B) sites of the spinel lattice. In spinel oxides, generally, the antiferromagnetic superexchange A-O-B interaction is much stronger than the ferromagnetic A-A and B-B interactions. Thus, the spins of magnetic ions in the same sublattice are parallel and the spins of cations on dissimilar sublattices, A and B, are antiparallel. As the octahedral sites are twice those of the tetrahedral sites, a net, non-compensated, magnetic moment is expected if all the cations are magnetic in nature (i.e. ferrimagnetism). If disorder is introduced in the structure through dilution by nonmagnetic ions, there will be a competition between ferromagnetic and antiferromagnetic interactions, leading to a spin-glass or cluster-glass behavior (4). In nano ferrites below a critical size, magnetic particles become single domain and exhibit superparamagnetism [5]. Superparamagnetism is an important magnetic property of nano spinel ferrite materials and they show a size dependent blocking temperature above which the particles display paramagnetic characteristics [6]. In this new state of magnetism the collective behaviour of the magnetic nanoparticles is the same as that of paramagnetic atoms but there is a well defined magnetic order within each nanoparticle [7]. The superparamagnetic properties of the spinel ferrite nanoparticles have also been studied by temperature dependent Mössbauer spectroscopy [8,9]. In contrast to the bulk ferrites, in which below T_N , a hyperfine pattern is observed in the Mössbauer spectra, the nanocrystalline ferrite materials show paramagnetic doublet patterns below T_{N} , when the particle size is smaller than a critical value [8]. Some studies on ferrites have shown that at lower temperatures a small sextet pattern along with a doublet is observed due to short-range order within the ultrafine particles and absence of long range order between the particles, and on lowering the temperature below the blocking temperature, the sextet appears due to long-range order, between the particles [9].

II. Experimental

The $\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$ sample was prepared by co-precipitation method and sintered at 573K. X-ray diffraction analyses of the sample was carried out with a Phillips X-ray diffraction unit (model PM 1710) using CoK_{α} ($\lambda_{=1.79\ \text{\AA}}$) radiation with a Ni filter. The crystallite size of the nanocrystalline samples were measured from X-ray line broadening analyses applying Scherrer formula [10]. Transmission ⁵⁷Fe Mossbauer spectrometry analysis was performed at 55K , 125K and 290K.



Fig. 1 . XRD patterns of Co_{0.2}Zn_{0.8}Fe₂O₄ samples sintered at a) 573K

The XRD patterns in Fig. 1 show that samples is single phase spinel oxides. The average crystallite size of these samples were found to be in the range of 3nm to 5 nm. The particle size of the sample calculated from the TEM photograph is 7.8nm.



Fig.2 Zero field cooled (ZFC) and field cooled (FC) magnetization plots of $\rm Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample

The temperature dependence of dc magnetization in Fig. 2 is shown that the ZFC magnetization M_{ZFC} gradually increases with increasing temperature and then a broad maxima appears, followed by decrease of M_{ZFC} for 573K sintered samples. On heating the samples, the FC magnetization curve does not follow the ZFC curve below the temperature of maxima in the ZFC plots. The experimental results from ZFC-FC magnetization measurement indicate that $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered nanoparticles have superparamagnetic behavior. The temperature at which the ZFC and FC curves meet appears to the blocking temperature of these samples. The blocking temperature, T_B is 73K.



Fig.3. Mössbauer spectra of Co_{0.2}Zn_{0.8}Fe₂O₄ 573K sintered sample at 303K



Fig. 4. Mössbauer spectra of Co_{0.2}Zn_{0.8}Fe₂O₄ 573K sintered sample recorded at a) 55K and b) 125K

Fig. 3 shows the mössbauer spectra of $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample, sample recorded at 303K. Fig 3 shows, presence of only paramagnetic doublet. Earlier mössbauer studies on bulk Co-Zn ferrite samples have shown the presence of a sextet at 303K (11). Absence of sextet and the presence of only a doublet in these nanocrystalline sample is possible only if the samples is superparamagnetic and the mössbauer patterns are recorded above the blocking temperature (12). Magnetization measurements have indicated that the $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample is superparamagnetic and their blocking temperatures T_B is 73K. Thus, mössbauer spectra of the $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample is 73K sintered sample was recorded below and above their T_B . The T_B of $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample is 73K and the mössbauer spectra at 55K shows the appearance of a sextet and at 125K a paramagnetic doublet (Fig.4 a,b). These results confirm that $Co_{0.2}Zn_{0.8}Fe_2O_4$ 573K sintered sample is superparamagnetic.

IV. Conclusion

From these results it may be concluded that $Co_{0.2}Zn_{0.8}Fe_2O_4$ sample sintered at 573K are superparamagnetic and the blocking temperature is 78K. Mossbauer spectra of these samples shows paramagnetic doublets above blocking temperature and sextet pattern below the blocking temperature which indicate sample is superparamagnetic in nature.

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