Magnetic properties of cobalt ferrite nanoparticles synthesized by sol-gel method

To cite this article: T George et al 2015 IOP Conf. Ser.: Mater. Sci. Eng. 73 012050

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Abstract. Cobalt ferrite nanoparticles of average size 18 nm are synthesized by sol-gel method and investigated the magnetic properties. The saturation magnetization value calculated from vibration sample magnetometer (VSM) studies for CoFe$_2$O$_4$ is lower than the reported value for the bulk. The magnetization curves demonstrate a trend towards the superparamagnetic behavior of the as-prepared CoFe$_2$O$_4$ nanoparticles. The microwave magnetic parameters show a decreasing trend with the increase of frequency.

1. Introduction

Nanosized ferrite magnetic materials are the subject of intense research work because of their numerous applications in various fields such as, magnetic disc drives, spintronics, ferrofluids, catalysts, medical diagnostics, drug delivery systems, pigments in paints and ceramics [1-3]. The high resistivity and low hysteresis loss makes ferrites, suitable to use in microwave applications and radio electronics [4-5]. Various methods have been developed for the synthesis of nanoferrites, like sonochemical reactions, sol-gel, microwave plasma, host template, coprecipitation, microemulsion and chemical vapour deposition [6-8]. We have synthesized cobalt ferrite nanoparticles of average size 18 nm by sol-gel method as described elsewhere [9-10] and investigated their magnetic and microwave properties.

2. Experimental

The precursors are Co(NO$_3$)$_2$ 6H$_2$O (0.4 M, 99% SD Fine Chem), Fe(NO$_3$)$_3$ 9H$_2$O (0.8 M, 99% SD Fine Chem) and aqueous solution of poly acrylic acid (Acros organics), used without further purification, and synthesized by sol-gel method [10]. The phase structure characterization of the synthesized powder was carried out by X-Ray diffraction (XRD) using PANalytical X’Pert PRO diffractometer with CuK$_\alpha$ radiation. Studies on the surface morphology and homogeneity of the powders were carried out using a field emission scanning electron microscope (NOVA NANOSEM 600 FEI).
The magnetic properties of the synthesized nanoparticles were measured using a VSM (Lakeshore) at room temperature and the hysteresis loop was drawn with the magnetic field, cycled between –10000 G to 10000 G. The experimental set up for the determination of microwave magnetic properties consists of a transmission type S-band rectangular cavity resonator and a network analyzer [11-12]. The rectangular cavity is connected to the Agilent 8714 ET network analyzer (frequency range 300 KHz - 3GHz). To find the complex permeability, the sample powders were tightly filled in thin capillary tubes of low loss material and sealed. The tube was then inserted through the non-radiating slot on the broad wall of the wave-guide cavity, and held with a thin sample holder made of low loss material. Resonant frequencies and the corresponding Q values of the sample loaded cavity and of the air filled cavity were measured in the TE mode. Then the magnetic parameters such as the real and imaginary part of complex permeability (table 1) were evaluated according to the theory explained elsewhere [11-12].

3. Results and discussion

Figure 1(a) shows the XRD pattern of the synthesized cobalt ferrite nanoparticles. All diffraction peaks are indexed to a pure cubic phase (JCPDS Card No 22-1086) of CoFe$_2$O$_4$ with space group Fd3m. The estimated lattice parameter, $a = 8.388$ Å well agrees with the JCPDS file. The average crystallite size estimated using the Scherrer equation for CoFe$_2$O$_4$ is 18 nm. Figure 1(b) shows the FE-SEM images of CoFe$_2$O$_4$ nanoparticles. It can be seen that the particles are almost spherically shaped and uniformly arranged. The particle size estimated from the FE-SEM analysis agrees well with the XRD results. The extremely fine crystallite size and regular shape of the materials are evident from the FE–SEM studies.

![Figure 1. (a) XRD pattern (b) FE-SEM images and (c) hysteresis curves of the CoFe$_2$O$_4$ nanoparticles.](image)

A hysteresis curve of CoFe$_2$O$_4$ nanoparticles is shown graphically in figure 1(c). The saturation magnetization (Ms) value calculated for CoFe$_2$O$_4$ nanoparticles is 30 emu/g, which is lower than the reported value for the bulk samples (80 emu/g) [13], which is a consequence of superparamagnetic nature of the magnetic nanoparticles. The decrease in the density of magnetization of the nanoparticles with respect to the bulk can be attributed to surface defects and morphology. The surface defects are the results of finite-size scaling of nanocrystallites, which in turn leads to a non-collinearity of magnetic moments on their surface. These effects are more intense in ferromagnetic system, where the super-exchange interaction occurs through the oxygen ion, O$^{2-}$ [14]. The coercivity recorded is approximately 1100 G for CoFe$_2$O$_4$ nanoparticles. The superparamagnetic behavior should be attributed to the extremely fine crystallite size and regular shape of the material [15], which makes it easier for them to be thermally activated to overcome the magnetic anisotropy.

Microwave magnetic parameters of CoFe$_2$O$_4$ nanoparticles (sample density = 0.46x10$^3$ kgm$^{-3}$) at various resonant frequencies are shown in table 1. The real part of complex permeability $\mu'_r$, imaginary part of complex permeability $\mu''_r$ and the corresponding loss tangent of CoFe$_2$O$_4$ nanoparticles decrease continuously as the frequency increases from 2248 MHz to 2972 MHz. The eddy current loss in a material can be expressed in terms of Tan$\delta$. It is known that ferrites possess feeble eddy currents, which agrees very well with the experimental results. The decrease in loss
tangent is due to the fact that, when the frequency of the field increases the dipoles cannot follow the rapid variation of the field. The values of permeability are not really constant for a given material but usually have a strong dependence on frequency [16]. When the frequency increases the magnetic dipoles of the crystallites cannot follow the fast variation of the applied field. The superparamagnetic nature of the CoFe$_2$O$_4$ nanoparticles might have affected the experimental values of permeability.

Table 1. Variation of magnetic parameters with frequency of CoFe$_2$O$_4$ nanoparticles.

<table>
<thead>
<tr>
<th>Frequency (MHz)</th>
<th>Real part of Complex permeability $\mu'_r$</th>
<th>Imaginary part of complex permeability $\mu''_r$</th>
<th>$\tan \delta = \mu''_r/\mu'_r$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2248</td>
<td>8.327</td>
<td>0.4162</td>
<td>0.0500</td>
</tr>
<tr>
<td>2440</td>
<td>7.941</td>
<td>0.3861</td>
<td>0.0486</td>
</tr>
<tr>
<td>2685</td>
<td>7.612</td>
<td>0.2923</td>
<td>0.0384</td>
</tr>
<tr>
<td>2972</td>
<td>7.308</td>
<td>0.2672</td>
<td>0.0365</td>
</tr>
</tbody>
</table>

4. Conclusions

Cobalt ferrite nanoparticles with average particle size 18 nm are synthesized by sol-gel method. The average crystallite size and the particle size are calculated both by XRD and FE-SEM are found to be in good agreement with each other. The saturation magnetization value calculated for CoFe$_2$O$_4$ is lower than the reported values for the bulk. The magnetization curves demonstrate that CoFe$_2$O$_4$ nanoparticles at room temperature tend to be superparamagnetic. The microwave magnetic parameters show a decreasing trend with the increase of frequency. The superparamagnetic nature of the CoFe$_2$O$_4$ nanoparticles might have affected the experimental values of magnetization and permeability.

Acknowledgements

Authors thank University Grants Commission (UGC), New Delhi and Kerala State Council for Science, Technology and Environment (KSCSTE), Thiruvananthapuram for financial support.

References