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An Approach to Study Applications of Doped Calcium Hexaferrites

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ABSTRACT : *Hexaferrites show favorable properties to be used for the applications of EMI shielding , like high values of resonance frequencies , relatively high permeability at microwave frequencies, high electrical resistivity etc.[1] Keeping in view above stated properties, Zirconium-Cobalt doped calcium hexaferrites are synthesized using microwave induced sol-gel combustion route for different doping concentrations. Structural and Morphological properties are studied by X-ray diffraction analysis and Transmission Electron Microscopy (TEM). Structural analysis confirms the formation of calacium Hexaferrites. Electrical and dielectric properties of the synthesized samples are studied by Impendence Analyzer as a function of temperature and frequency. Magnetic properties are analyzed using Vibrating Sample Magnetometer(VSM).Measured values of the properties show favorable order from the point of view of designing and developing new low cost customized EMI shielding materials.*

Keywords - *Hexaferrites, Eielectric Properties, Magnetic Properties, Electromagnetic Interference*

1. INTRODUCTION

With the advancement of the communication technology, issues related to Electromagnetic Interference (EMI) has got the special importance. There is need to synthesis materials for shielding from electromagnetic radiation which are efficient, inexpensive, lightweight and the same time low cost[1]. The shield from EM radiation can be either reflection or absorption phenomena based. The shielding layer should be as thin and light weight as possible, operating in a broad frequency range, and independent of the incident angle and polarization. The usual solution for antireflection coating is a sequence of layers with progressively changing electromagnetic parameters, matching the impedance between air and substrate. Hexagonal Ferrites received a great deal of attention as microwave absorbing materials [1]. Hexaferrites have an internal anisotropy field up to 35 kG, magnetization up to 5 kG, and high Neel temperature of around 500^{o}C. Due to the presence of Fe^{3+} ions hexaferrites are good insulators, having very low dielectric losses. The temperature dependence of the magnetization is rather weak, guaranteeing good stability with respect to temperature.
Extensive work has been done on the Barium –Hexaferrite regarding their applications in various fields. As Calcium hexaferrites are less attended [2], an attempt is made to synthesis and carry out experimentation to find the utility of Zirconium-Cobalt substituted calcium hexaferrites as microwave absorbing or EMI shielding material.

2. EXPERIMENTAL

The synthesis of $CaFe_{11}(ZrCo)O_{19}$ and $CaFe_{10}(ZrCo)_2O_{19}$ is done using microwave induced sol-gel combustion route.[3,4]. The reactive oxidants such as $Ca(NO_3)_2$, $Fe(NO_3)_2.9H_2O$, $Co(NO_3)_2.6H_2O$, $ZrO(NO_3)_2.H_2O$ were dissolved into an unionized distilled water at the temperature of 50^{o}C for 15-20min, urea is used as fuel which gives requisite energy to initiate exothermic reaction. The gel produced is then kept for an hour in the room temperature and then it's combustion is carried out by microwave oven for 15-20 min. The gel get burnt and finally gets converted in homogeneous powder. The sample is then sintered by giving moderate heat treatments for further few minutes with further grinding. Pellets of diameter 15mm are formed using hydraulic press. Polyvinyl acetate (PVA) is used as binder for pellets and the sintering is done at 400^{o}C for 2 hours by sequentially increasing and decreasing temperature at the moderate rate of 4^{o}C/min.

3. RESULT AND DISCUSSION

Table 1 gives the values of the Structural, Electric, Dielectric and Magnetic properties of the samples.

3.1 STRUCTURAL PROPERTIES

The diffraction patterns of samples are taken with Philips X'pert Diffractometer, Philips X'pert Diffractometer and Cu Kα radiation with wavelength λ=1.542 **(Å).** Lattice parameters *a* and *c* are found to be 5.8256 **(Å)** and 22.1244 **(Å)** respectively for $CaFe_{10}(ZrCo)_2O_{19}$ and 5.8234 **(Å)** and 22.1286 **(Å)** for $CaFe_{10}(ZrCo)_2O_{19}$ respectively. Nanosize confirmation is done by TEM analysis using Philips CM200, Operating at voltage range 20-200kv. Fig.1 (a-b) shows the TEM images of the samples. The particle size is found to on an average 100nm. The grains are found to be have sharp edges.[5,6]

3.2 ELECTRIC AND DIELECTRIC PROPERTIES

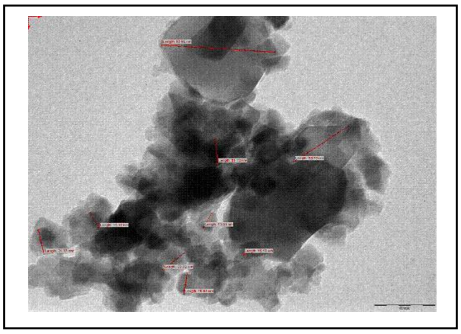
The variation of Conductivity with temperature is studied using precision impendence Analyser 6500B, Waynekerr Electronics. For controlled temperature variation, the sample is kept in a digitally controlled furnace. Four probe method is used to measure dielectric properties of the samples in palletized form. Variation of conductivity with respect to temperature is shown in Fig.2 (a-b). Increase in conductivity with increase in temperature shows the semiconducting nature of the materials. The conductivity in ferrites explained by Verwey's hopping mechanism [6,7]. The dielectric loss (tanδ) gives energy loss within the material during conduction of electrons. Values of loss tangent at room temperature are shown in Table 1. Loss tangent (tanδ) is found to decrease with increasing degree of substitution. Lagging of the polarization with applied ac electric field is the main cause of the dielectric loss [2].

3.3 MAGNETIC PROPERTIES

Fig. 3 shows hysteresis curves for the sample depicting magnetic behavior of the sample in the presence of external magnetic field.

Values of saturation magnetization (Ms), remnant magnetization (Mr) and coercivity (Hc) found to decreases with the substitution of cations as mentioned in Table 1. Decrease in the values of Ms, Mr with increase in values of substitution is due to the substitution of diamagnetic ions not only into the spin-down sub lattice site $4f_1$ of Fe^{3+} ions but also into other octahedral spin-up sub lattice sites 12k, 2a, 2b of Fe^{3+} ions. Also the area under hysteresis curves decreases with increase in substitution level which indicates that the ferrite become soft with increasing degree of substitution. [7].

3.4 FIGURES AND TABLES



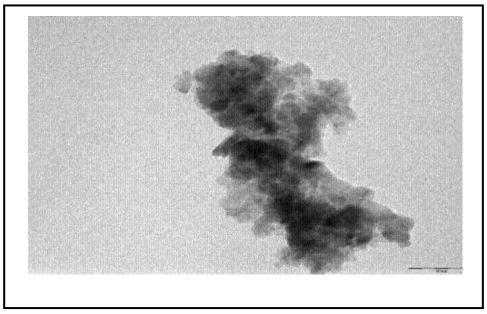
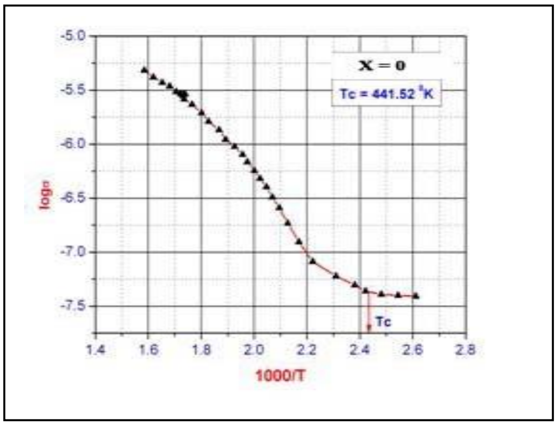


Figure 1 (a-b) : Transmission electron micrographs (TEM images) of the samples



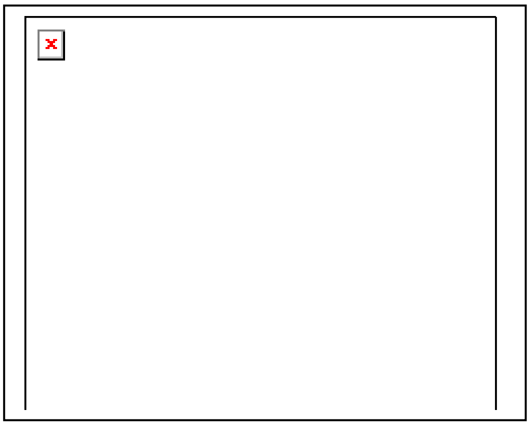


Figure 2 (a-b) : Plot of conductivity Vs Temperature for $CaFe_{11}(ZrCo)O_{19}$ and $CaFe_{10}(ZrCo)_2O_{19}$

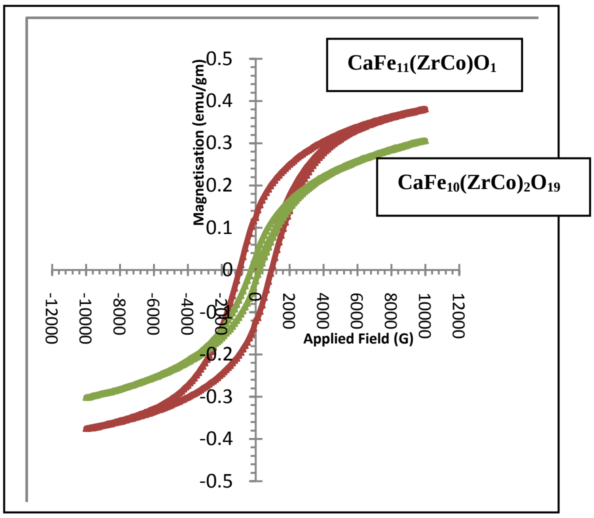


Figure 3 : Hysteresis curves for the samples $CaFe_{11}(ZrCo)O_{19}$ and $CaFe_{10}(ZrCo)_2O_{19}$

TABLE 1 : Lattice parameters (a and c) , Resistivity (ρ), Values of saturation magnetization (M_s) , remnant magnetization (M_r) and coercivity (H_c)

Samples	a (□)	c (□)	□ at 373K (Ω-m)	tan □ at 1 kHz	Ms (emu/gm)	Mr (emu/gm)	Hc (G)
$CaFe_{11}(ZrCo)O_{19}$	5.8256	22.124	$1.2292*10^7$	2.6238	0.3805	0.1245	875
$CaFe_{10}(ZrCo)_2O_{19}$	5.8234	22.128	$1.7718*10^6$	0.1314	0.3055	0.0305	250

4. CONCLUSION

The substitution of Zirconium and -Cobalt in calcium hexaferrites by replacing Fe^{3+} is found to be successful by the Microwave Induced sol-gel combustion route. The X-ray diffraction studies confirm the formation of hexaferrites and the a & c values of the sample supports this confirmation. From TEM data analysis, particle size is found to be in nano range. Dielectric losses are observed to decrease with increase in degree of substitution at constant frequency. Conductivity plots show the semiconducting nature of the synthesized samples. With high values of electrical resistivity and low values for dielectric loss tangent, the synthesized calcium hexaferrites dopped with Zirconium and Cobalt can prove to be a promising material in the microwave absorbing, EMI shielding applications [1]. Also the nano size of the particles helps to reduce the noise produced which occur due to displacement of domain boundaries [8]. The variation in the values of magnetic parameters shows the possibility of tailoring of the magnetic properties according to the need for different applications [5,6]. The reduction of particle size of hexaferrites to nanorange helps to improve magnetic and electric properties mentioned earlier.

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