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Development and investigation of physicochemical properties of $CoFe_2O_4$/SiC/epoxy nanocomposites

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Abstract : This work emphases on the study ofconsequences of adding different wt% of SiC on physical and chemical properties of $CoFe_2O_4$/SiC/epoxy nanocomposites. To obtain $CoFe_2O_4$/SiC nanocomposites along with epoxy polymer which acts as matrix, combination of Chemical co-precipitation method & Solid-State approach were employed. The Morphological, Structural, Dielectric Properties& elemental analysis of manufacturedbuilding blocks were estimated using Scanning electron microscopy (SEM), X-ray diffractometer (XRD), Impedance Spectroscopy & Energy dispersive x-ray analysis (EDX) respectively. The inverse spinal crystal structure was revealed through XRD and the particle size were calculated using Scherrer's formula and estimated ~3nm±2nm for $CoFe_2O_4$ (CFO) nanoparticles and ~5nm±2nm, 6nm±2nm, 11nm±2nm and 18nm±2nm for CFO/SiC nanocomposites with different wt% of SiC respectively. The agglomeration of nanoparticles with epoxy polymer matrix were evident from SEM images and EDX images illustrates the existence of Co, Fe and O. The dielectric properties were deliberated at room temperature ( ) for proposed nanocompositeswith varying frequency from 20 Hz to 1MHz. The highest dielectric constant and loss were discovered at initial frequency, which perceived to be falling as frequency raising.A rise in relative permittivity and fall in loss tangent were evident on growing the wt % of SiC in CFO/SiC/Epoxy nanocomposites.

Keywords: Ferrites; Nanocomposites; Polymer; Co-precipitation; Solid-State method.

Introduction

In this paper, the nano sized fillers are comprised of magnetic nanoparticles using $CoFe_2O_4$ (CFO). Here we are focusing on CFO/SiC polymer nanocomposites because CFO nanoparticles are very useful for magnetic recording such as audio and video tape, microwave devices and electromagnetic shielding fields caused by theirtop-notch saturation magnetization, great coercivity[1]. The properties possessed by CFO nanoparticles depend upon their chemical composition and cation distribution at tetrahedral A and octahedral B sites as CFO possess inverse spinel structure[1–3].There are various techniques used to synthesize CFO nanoparticles such as hydrothermal, micro-emulsion, chemical co-precipitation and sol-gel methods[4]. Here we have used chemical co-precipitation method to synthesize ultrafine and homogeneous nanoparticles of CFO. From this method, we can easily control some parameters such as reaction temperature, molar concentration and pH value precisely.We haveprepared CFOnanoparticles by co-precipitation method[1,2]. The concentration and temperature parameters were controlled precisely and superparamagnetic CFO nanoparticles were found without using capping agent. The preparedCFONanoparticles acts as a promising materials for gas sensing applications towards different toxic gases[5]. Silicon carbide (SiC) is a high-quality semiconducting material due to which its technology has advanced rapidly over the year, resulting in advancement in sensing devices, wafer growth technology, and electronic devices[6]. CFO and SiC are interesting candidates for a inclusive applications, including sensors, memories, actuators, electromagnetic interface (EMI) shielding etc.[6,7]. Epoxy is an environmentally friendly inexpensive polymer which has a good capability to incorporate nanoparticles. Therefore, we have included Epoxy as binder in CFO/SiC polymer nanocomposite. The combination of such materials leads to change in electrical polarization which enhance the dielectric properties[8].The suggested $CoFe_2O_4$/SiC/epoxy

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nanocomposites are novel to the best of author's knowledge and not been reported by other scientists or researchers so far.

Experimental details

The chemical co-precipitation approachwaslabored for production of cobalt ferrite nanoparticles. The solution of $CoCl_2.6H_2O$ along with $FeCl_3.6H_2O$ precursors were added into the alkaline solution of NaOH during stirring. The precipitation was formed followed by drying at 60 , grinding and sintering at 200 . To synthesize the composite samples, firstly mix different weight percent (80/20, 60/40, 40/60 and 20/80) wt% of CFO/SiCby adding ethanol which were grinded till the evaporation of ethanol and form CFO/SiC nanocomposites in powder form.Then CFO/SiC nanocomposites of different wt% were mixed using Bisphenol-A epoxyas a matrix and the hardener was mixed in epoxy resin just before mixing of CFO/SiC mixture. For the preparation of composite epoxy and hardener has been taken in the ratio 10:1and mixed with accurately weighted CFO/SiC/epoxynanocomposites. Circular shaped mould of diameter 10 mm was used for making composite pellets. Silicon grease was coated in the inner surface of mould for smooth release of composite pellets. The combined materialsweretransferred in the grease coated circular mould and formation of air traps was dodged by applying load on the upper side of the cast. It took 24 hr to cure the sample. After 24 hours the samples were removed from the mould and cut into appropriate size for testing.The structural characterization and phase identification of CFO/SiC powder wasdetected by X-ray diffraction (XRD). The appearance of elemental composition of CFO nanoparticles were estimated usingenergy dispersive X-ray spectroscopy (EDX). Scanning electron microscope (SEM) were used to study the surface morphology of CFO/SiC nanocomposite. The dielectric properties including dielectric constant (εr) and loss tangent ( ) is measured by using E4980A LCR Meter (Agilent technologies).

Results and discussion

Structural Properties

Thephase identification& structural properties of CFO/SiC powder wereestimated using X-ray diffraction (XRD) technique. The phase formation and crystalline size of different samples were studied at room temperature.The XRD pattern of developed CFO nanoparticles and CFO/SiC with various wt% can be illustrated from Fig.1. As per the standard JCPDS data &XRD pattern, all samples contain single phase of cubic spinel structure. The reflections planes (311), (400), (440) and (511) confirms the development of cubic spinel structure of single phase of CFO nanoparticles matched with standard file (JCPDS-22-1086) and planes (101), (103), (104), (109), (110) and (202) of SiC were coordinated with JCPDS-29-1129.The crystalline size was determined using Scherrer's formula

$$t = \frac{0.89\lambda}{\beta\cos\theta} \qquad (1)$$

where't' illustrates the crystalline size (nm), 'λ' represents the wavelength of X-ray, 'θ'shows the Bragg's diffraction angle and'β'denotesthe difference between the Full Width at Half Maximum (FWHM).For the biggest particle size, the sharp and fierce reflections were noticed, which indicates the large CFO/SiC nanocrystalline particles exist. Thelattice constant and grain size of CFOnanoparticles& CFO/SiCweretabulated in Table 1.

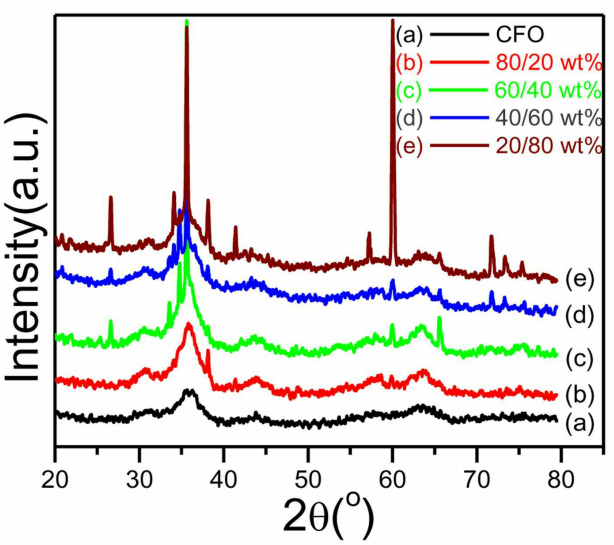

Figure1. Comparison of XRD dataof (a) CFO nanoparticles and (b-e) CFO/SiC with different (80/20, 60/40, 40/60 and 20/80) wt %.

Table 1. The values of structural parameters of CFOnanoparticles and CFO/SiC with various compositions

Sample	Crystalline size (t) nm	Lattice constant (a) Å
CFO	3.5	8.34
CFO/SiC wt% 80/20	4.7	8.31
CFO/SiC wt% 60/40	6.3	8.30
CFO/SiC wt% 40/60	11	8.30
CFO/SiC wt% 20/80	18.7	6.7

Surface Morphology

The morphology and microstructurethe CFO/SiC epoxy nanocomposites were studied using scanning electron microscopy (SEM) technique. The granules seem to consolidate into bigger and smoother grain as the concentration of Co drops. Fig.2(a–e)illustrates the SEM micrographs of $CoFe_2O_4$ in the form of powder andCFO/SiC in the shape of pellets with various compositions. The analysis of SEM image confirmshomogeneous distribution of nanoparticlesand their agglomeration. The micrograph of CFO nanoparticles in Figure 2 (a) exhibits the evenly distributed, pore less and continuous contrast morphology of particles.

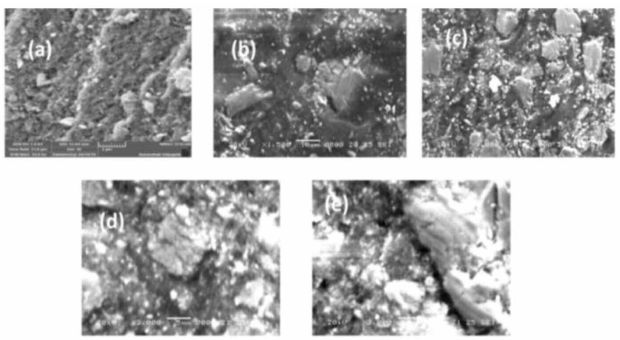

Figure2.(a) SEM micrograph of CFO nanoparticles.(b–e)SEM micrographs of CFO/SiC nanocomposites with 80/20,60/40, 40/60 and 20/80 wt %.

Elemental Analysis

The compositions of CFOnanoparticles were determined using the EDX analysis. The individual peaks of Co, Fe and O of CFOnanoparticles wereappeared in EDX images which was illustrated in Figure 3. The qualitative discussion for better understanding of chemical quality of CFOnanoparticles can be done using EDX measurements. The peaks are identified as Co & Feat their corresponding energy levels, demonstrating the presence of Fe, Co in the specimen. The composition of CFOnanoparticles is given in Table 2.

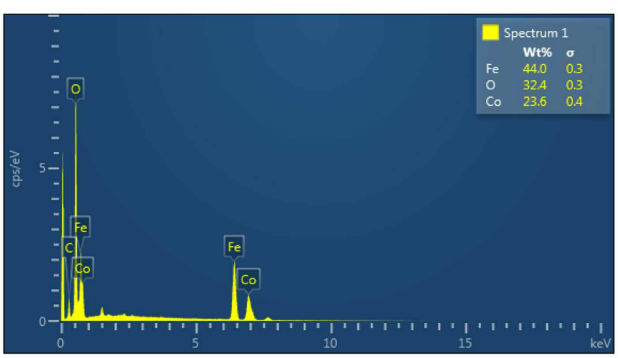

Figure3.EDX spectrum of CFOnanoparticles.

Table 2. The elemental composition of synthesized CFOnanoparticles.

S. No.	Element	wt %
1	Co	23.6
2	Fe	44.0
3	O	32.4

Dielectric Properties

Dielectric measurements including frequency dependence of capacitance (C), dielectric constant dielectric loss ( ) was recorded simultaneously with varying frequency from 20 Hz to 1MHz at room (28) temperature for CFO/SiC epoxy nanocomposites.The relativepermittivity(εr) of CFO/SiC pellets is determined using the formula

$$\varepsilon_r = \frac{Cd}{\varepsilon_\circ A} \qquad (2)$$

where, the capacitance of the pellets was represented by 'C', thickness was indicated by 'd',the cross-sectional area was denoted by 'A' and the absolute permittivity was represented by ' (8.854×10^{-12} F/m). The dielectric constant as function of frequency were appeared in Fig. 4. The decrement was observed in relative permittivity with raise in frequency range which was evident from Fig. 4.

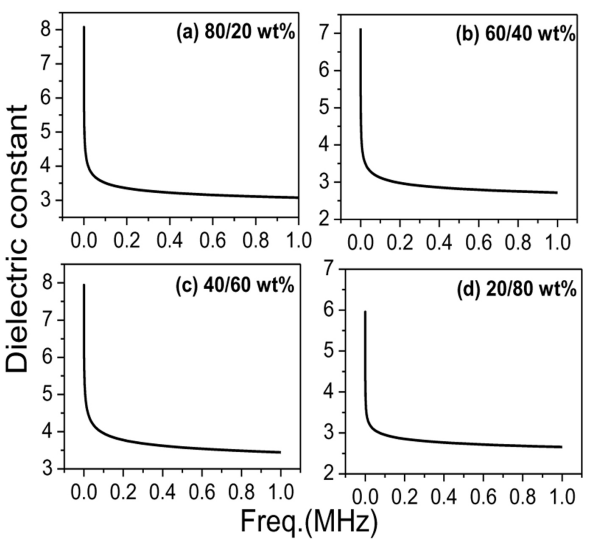

Figure4. (a–d) Dielectric constant (εr) with varying frequency of CFO/SiC epoxy nanocomposite in range 20 Hz to 1MHz.

The change in loss tangent (with frequency was illustrated in Figure 5. Structural homogeneity, Fe^{+2} content & stoichiometry are the factors which depends on the fabrication methods and compositions affects the value of dielectric loss. The values of dielectric loss and permittivity at lowest and highest frequency are listed in Table 3.

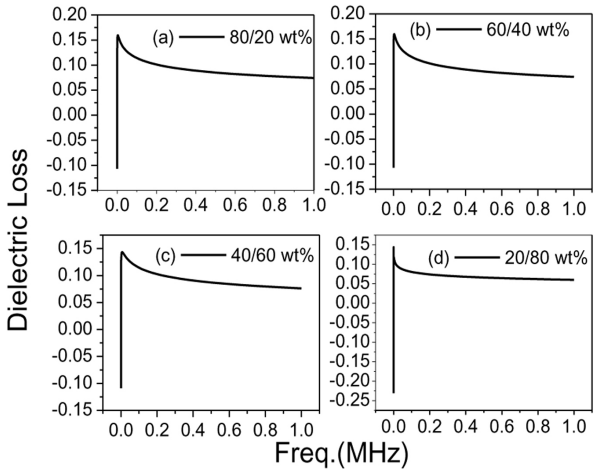

Figure5. (a–d)Change in loss tangent with varying frequency range 20 Hz to 1MHz.

Table3. The dielectric behavior of CFO/SiC/epoxy nanocomposite at frequency 20 Hz to 1MHz.

	f= 20Hz		f=1MHz	
Sample	ε_r	$tan\delta$	ε_r	$tan\delta$
CFO/SiC wt %	**148.12**		**59.50**	
80/20	**-84**	**4.1**		
CFO/SiC wt %	**126.90**		**48.39**	
60/40	**-13.6**	**3.5**		
CFO/SiC wt %	**147.08**		**68.84**	
40/60	**-16.0**	**5.2**		
CFO/SiC wt %	**177.24**		**80.37**	
20/80	**-41.6**	**5.1**		

Conclusion

In a nutshell, we can conclude thatthe CFO nanoparticles were successfully createdusing chemical co–precipitation approach. The inverse spinel crystal structure of cobalt ferrite nanoparticles was confirmed by X–ray diffraction (XRD). Particle size was calculated using Scherrer's formula which was estimated ~3nm±2nm andCFO/SiC nanoparticles with different wt% having crystalline size 5nm±2nm, 6nm±2nm, 11nm±2nm and 18 nm±2nm respectively. The lattice parameters of preparedCFO nanoparticles and CFO/SiC nanocompositeswith varying wt% are 8.34Å, 8.31Å, 8.30Å, 8.29Å and 6.7Å respectively. SEM micrographs revealed that on raising the concentration of cobalt nanoparticles, they coalesce to create larger grains with smooth surface. The existence of Co, Fe, and O in proper proportions of ratio 1:2 was deep–rooted in their corresponding energy locations by EDX analysis.With the frequency region from 20 Hz – 1MHz, the relative permittivity & loss tangent were calculated as a function of frequency at room temperature. All the CFO/SiC nanocomposites at various compositions shows a fall in permittivity and loss tangent with greater frequency. To reduce dielectric losses in electrical circuits, materials with good dielectric constant are required which can work on higher frequencies applications.

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