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Synthesis and Characterization of $MnFe_2O_4$ / Graphene / Epoxy Nanocomposites

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Ternary $MnFe_2O_4$/graphene/epoxy nanocomposites have been synthesized using chemical co-precipitation and solid state methods having different concentration of graphene. The obtained ternary nanocomposites and pure $MnFe_2O_4$ have been characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and impedance spectroscopy to obtain the structural, morphological and dielectric properties. The crystallite size of $MnFe_2O_4$ has been calculated using Scherer formula, which was found to be 1.3 nm. The agglomeration of particles with surface roughness was obtained by SEM analysis. The device has been fabricated using silver coating in terms of capacitor to characterize dielectric properties. It has also been observed from dielectric analysis as concentration of graphene increases from 1% to 10%, the value of gradient of dielectric decreases and also loss tangent decreases on increasing concentration of graphene.

Keyword: Co-precipitation, Ternary composites, Dielectric constant, Loss tangent

Introduction

Recently, graphene has become a great facilitating material for metal oxide nanoparticles due to its enormous specific area and attractive conductivity. The two dimensional structure of graphene provides large surface area to disperse widely the metal oxide nanoparticles and gives a proper network to transport electrons[1]. For designing the graphene/metal oxides based composite, the choice of a metal oxide with synergetic effect is also a great deal. Therefore, a ferrite is being in consideration because of its normal and inverse spinel structures. The spinel structure can be defined by tetrahedral (A) and octahedral (B) lattice sites in a spinel cube[2,3]. The cation distribution in both lattice sites decides the magnetic properties of ferrites. Further, a suitable polymer must be introduced to give the strength of new architect composite. Additionally, the selection of polymer is also a great challenge. In this report, authors have selected epoxy with hardener to design ternary $MnFe_2O_4$/Graphene/epoxy nanocomposites having different wt % of graphene to study the dielectric properties.

Experimental Detail

The solution of $MnCl_2.4H_2O$ (Sigma-Aldrich, 99.9 % pure) and $FeCl_3.6H_2O$ (S.D. Fine Chem, 99.9 % pure) was prepared separately using distilled water and mixed with the ratio[4] of Mn to Fe as 1:2. Another solution of NaOH was prepared as an alkaline precipitant agent. The metal chloride solution was mixed drop by drop in the alkaline solution. Concentration and reaction temperature were kept constant at room temperature. The reaction time of 2 h was found to be sufficient for the dehydration and atomic rearrangement involved in the conversion of the hydroxide into the ferrite compound. Further, to synthesize $MnFe_2O_4$/Graphene/ Epoxy nano- composites, 5 mL Epoxy was added into the 0.5 mL hardener with 10:1 by weight. Then, continuous stirring was maintained for proper mixing of epoxy and hardener. After that, the 0.3 gm MnFe2O4 with respect to 1% (0.003 gm), 2% (0.006 gm), 5% (0.015 gm) and 10% (0.030 gm) graphene by weight was added to the epoxy matrix material. And final sample was measured at 0.79 g.

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Results and Discussion

Structural properties

XRD patterns revealed that the sample is identified in Fig. 1 (a) as $MnFe_2O_4$ (JCPDS No.00-074-2403) having a single phase cubic structure. The average crystallite size of the $MnFe_2O_4$ nanoparticles was found to be 1.3 nm using Scherrer formula[5]. It was observed from Fig. 1 (b, c, d & e) that the peaks at 2θ values of 17.8º are of graphene. It was also observed that as wt % of graphene increases, another peak generated at 36.2º in 5 % and 10 % concentration.

Surface morphology

The surface morphology of all samples in pellet form was illustrated in Fig. 2. Agglomeration of particles with roughness of surface was observed from Fig. 2.

Dielectric properties

The dielectric properties of $MnFe_2O_4$/Graphene/ epoxy ternary composites were measured using (Agilent LCR meter E4980A) in the frequency range (2.0×10^5-1.0×10^6) Hz at room temperature.

It was observed from Fig. 3 (a) that dielectric constant is found to be higher at lower frequency whereas it is falling down with higher frequency having less concentration of graphene. As concentration of graphene increases from 1 % to 10 %, the value of gradient of dielectric decreases. Figure 4 illustrates that loss tangent is decreasing as

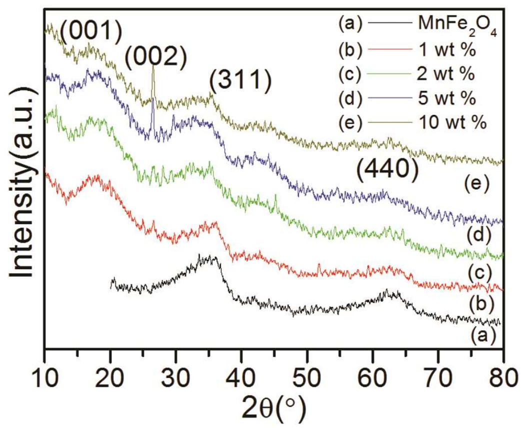

Fig. 1 – XRD patterns of $MnFe_2O_4$/graphene/ epoxy nano-composites.

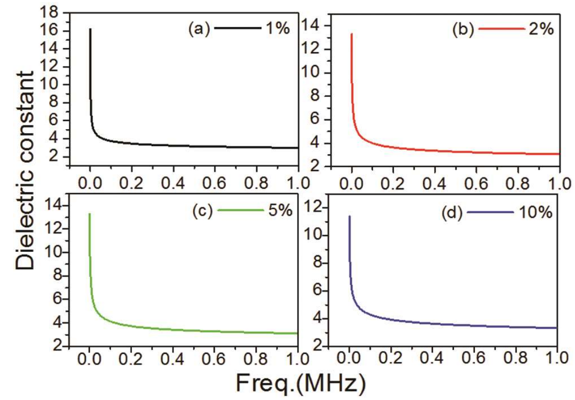

Fig. 3 – Variation of dielectric constant with frequency for (a) 1%, (b) 2%, (c) 5% and (d) 10% ternary $MnFe_2O_4$/graphene/epoxy composite.

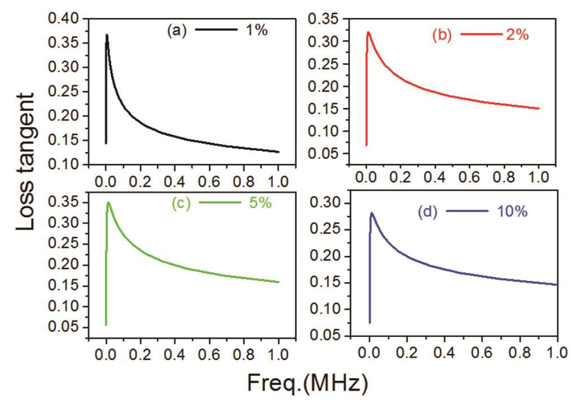

Fig. 4 – Loss tangent as a function of frequency for (a) 1%, (b) 2%, (c) 5% and (d) 10%, ternary $MnFe_2O_4$/graphene/epoxy composites.

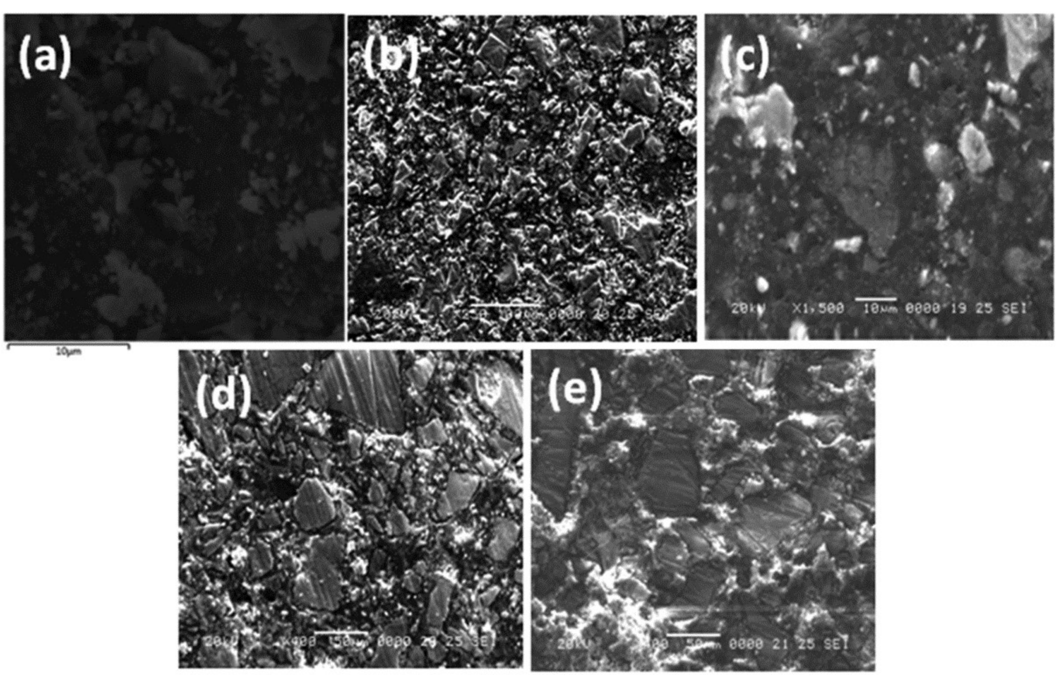

Fig. 2 – SEM images of (a) $MnFe_2O_4$, (b) 1 %, (c) 2 %, (d) 5 % and (c) 10 % ternary $MnFe_2O_4$/graphene/epoxy composites.

concentration of graphene increasing from 1 % to 10 % at all studied frequencies.

Conclusions

The ternary $MnFe_2O_4$/ Graphene/ Epoxy composites have been synthesized using co-precipitation and solid state method, which have been confirmed by XRD analysis. The crystallite size of pure $MnFe_2O_4$ nanoparticles has been found to be 1.3 nm. From SEM analysis, the agglomeration of particles has been obtained on the surface of the devices with roughness. It was observed from impedance spectroscopy that as concentration of graphene increases from 1 % to 10 %, the value of gradient of dielectric decreases and loss tangent decreases on increasing concentration of graphene.

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