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Preparation and Electrical Properties of Epoxy Resin Reinforced with Functionalized Carbon Nanotubes

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***Abstract**: In this study, dielectric properties of nanocomposites of epoxy as a based and multi walled carbon nanotubes before and after functionalization u-CNTs ,f-CNTs respectively as a reinforced materials have been investigated. The solution processing with help of acetone as a solvent have been used to fabricate u-CNTs/epoxy composites, while functionalized MWCNTs with nitric and sulfuric acids used to fabricate f-CNT/epoxy composite with volume fraction(0.0, 0.1 and 0.2) V_p % of CNTs. The effects of u-MWNT and f-MWNT with volume fraction loading on dielectric properties (dielectric constant, dielectric loss and impedance) as a function of the frequency (10^2 - $5x10^6$ Hz) at room temperature of an epoxy based nanocomposite system were investigated. The studies showed that dielectric constant, dielectric loss and impedance increased with CNT loading and decreased with frequency increasing. And f-CNT/epoxy composite shows a significant improvement in the dielectric properties when compared with pure epoxy and u-CNTs composites.*
Keywords:** polymer composites, carbon nanotubes, epoxy,* functionalization*, dielectric properties.**

I. Introduction

Epoxy resin is one of the most important thermosetting polymers used in different industrial applications owing to their excellent properties. Its use as an insulator extends to the encapsulation of integrated circuits and for the fabrication of printed circuit boards[1]. Epoxy resin also has a good balance mechanical properties allowing it to also find application in environments such as dry type transformers, coil insulators within rotating machines[2] and as spacers within Gas Insulating Switchgear (GIS)[3].Because of widespread usage of epoxies, researchers have conducted numerous investigations on different types of filler for enhancing their properties [4]. In recent decades, carbon nanotubes (CNTs) [5] has been intensively studied as promising candidates for epoxy resins at low contents. The addition of CNTs into polymers at very low volume could lead to considerable increase in their mechanical properties [6], electrical properties [7], thermal conductivity [8] and flame retardancy [9].

Numerous studies have shown already, that an effective performance of the carbon nanotubes in composites for a variety of applications strongly depends on the ability to disperse the CNTs homogenously throughout the matrix [10,11]. Good interfacial bonding and interactions between nanotubes and polymers are also necessary conditions for improving properties of the composites. Due to the nanoscale size of the CNTs the active CNT/matrix interface is significantly higher than that of other conventional fillers. Functionalization enhances the dispersion and interfacial bonding between CNTs and polymer matrix. It is suggested that, functionalization of CNT can play an important role for the further development of CNT composites. The aim of this work was to investigate the effect of surface functionalization of CNTs on dielectric properties of epoxy-nanotube composites with different loading.

II. Experimental

a- Materials

The polymer matrix consisted of part A epoxy resin (Quickmast 105) and part B hardener (poly amine) Epoxy polymer matrix was prepared by mixing 1:3 parts by volume of epoxy resin (Quickmast 105) with hardener (amine). Two different types of MWCNTs, pristine MWCNT and functionalized MWCNT, were used in this work. The MWCNTs employed in this study were supplied by Materials and Electrochemical Research (MER) corporation and its purity was higher than 95%. The MWCNTs had diameter of 140 ∓ 30 The MWCNTs had diameter of 140 ∓ 30 nm, and the length ranging of 7 ∓ 2 μm. Epoxy resin and hardener having the density of 1.04 g/cm^3.

b- Nanocomposite preparation

In order to study the effect of CNTs in the polymer matrix, pure epoxy, MWNT/epoxy (u-CNT /epoxy) and functionalized MWNT/epoxy (f-CNT/epoxy) composites were fabricated separately with volume fraction of the carbon nanotubes was 0.1 and 0.2 V_p %). The u-CNT /epoxy composites were prepared by desperation a desired a mounted of u-CNTs kinetically by ultrasonication. To achieve better state of desperation first the nanotube were treated with acetone in an ultrasonic bath at room temperature for (30 min) for the demarcation of the tube bundles. in order to evaporate the acetone , the mixtures are treated in a vacuum oven at (70 ℃) for (1 h) , then the treated tubes added the epoxy resin and sonicated for (1h) at room temperature . Were MWCNTs functionalized by acid treatment of MWCNTS, typically, 7.7g of MWCNTs,64 ml of 60% HNO_3,and 193 ml of 98% $H_2SO_4$was added into 1000ml flask equipped with a condenser with vigorous stirring. The flask was then immersed in a sonication bath (40 kHz)for 10 min . Then both mixture cured under by hardener addition (resin / hardener ratio 3:1) and sonicated for 3 mints in water bath for homogenization. The resultant mixture was cast in to mould, the mould is smeared by wax paper before the mixture was poured in to the mould.

The dielectric properties of samples (pure epoxy u-CNT/Epoxy and f-CNT/epoxy composites) are measured by (Agilent impedance Analyzes 4294A) . The samples were made in the form of circular discs and smooth surfaces. Samples with a diameter of (40 mm) and thicknesses (3 mm) were placed between two parallel plated electrodes. The measurement was carried out at frequencies from 100 Hz to 5 MHz at room temperature in order to examine the dielectric properties for samples pure, u-CNT, and f-CNT loading epoxy composites respectively.

III. Results and Discussion

Figs. (1) and (2) show dielectric constant (real permittivity) (ε') and dielectric loss (imaginary permittivity) (ε") as a function of the frequency (10^2 to $5x10^6$ Hz) at room temperature for cured epoxy and its cured PNCs reinforced with different u-MWNT(u-CNT) and f-MWNT(f-CNT) loadings, respectively.

The variations of dielectric constant with respect to frequency for the epoxy composites with CNTs and functionalized CNTs-fillers and at different filler concentrations are shown in Fig.(1). It can be seen from Figure that the dielectric constant of epoxy composites increase with decreasing frequency and increasing of CNTs loading. Dielectric constant is a frequency dependent parameter in polymer systems. At lower frequencies of applied voltage, all the free dipolar functional groups in the epoxy chain can orient themselves resulting in a higher dielectric constant value at these frequencies. As the electric field frequency increases, the bigger dipolar groups find it difficult to orient at the same pace as the alternating field, so the contributions of these dipolar groups to the dielectric constant goes on reducing resulting in a continuously decreasing dielectric constant of the epoxy system at higher frequencies[12], this behavior is in agreement with[13] , while the cured pure epoxy is observed to be a constant around 3.3 and nearly independent of the frequency within the entire measured frequency scale, indicating a stable dielectric performance of cured epoxy upon frequency variation[14] this result is in good agreement with other worker[15] . The ε' value for the cured u-CNT PNCs increases as the u-CNT loading increases is considered a consequence of interfacial polarization. Similar results have also been reported by [15], while the ε' value for the cured f-CNT PNCs increases as loading of f-CNTs increase, may be due to that as the loading increase in the epoxy ,the effect of filler permittivity. The ε' of f-CNT composites are higher than those of u-MWNT composites, this is due to that polymer/carbon nanotube composites is strongly influenced by several factors such as dispersion, aspect ratio, purity and alignment. Dispersion is probably the more fundamental issue. The strong van der Waals interactions between the nanotubes of u-CNTs bundles them together. This bundling of the nanotubes reduces their aspect ratio and decreases the dielectric constant. The PNCs with a high dielectric constant can be used in high charge-storage capacitors [16].But the ε" value for the cured u-CNT PNCs increases as the u-CNT loading increases, while the ε" value for the cured f-CNT PNCs decreases as the f-CNT loading increases as shown in Fig. (2) due to a phase lag between molecular orientation and electric field.

Figure (3) shows the variation of impedance of epoxy and its composites with different loading of u-CNT and f-CNT as a function of frequency.

As it is shown, the impedance decreases as frequency is increased, indication that the impedance of the samples is dominated by the capacitance of the epoxy matrix according to equation of impedance Z [17]:

$$Z = \frac{R}{\sqrt{1+(2\pi F)^2 R^2 C^2}} \qquad \text{-------1}$$

Where R is resistance, C is capacitance and F is frequency. Also the results indicate that the impedance of composite increase as CNTs loading increase and impedance of f-CNT composites is higher than that u-CNT composites due to several factors such as orientation, dispersion and interfacial of CNTs in the matrix.

IV. Conclusions

The dielectric properties of epoxy, u-CNT/epoxy and f-CNT/epoxy composites were evaluated. The f-CNT/epoxy composite shows a significant improvement in the dielectric properties when compared to the other two composites. Functionalization enhances the dispersion and interfacial bonding between CNTs and polymer matrix. It is suggested that, functionalization of CNT can play an important role for the further development of CNT composites.

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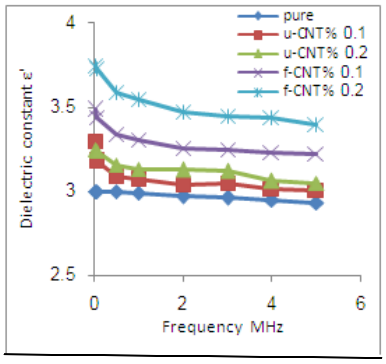
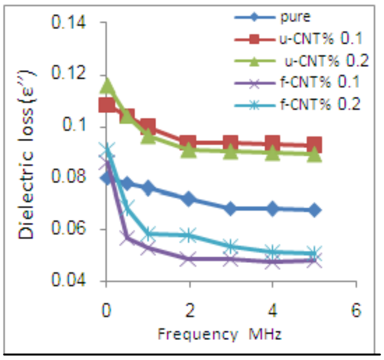
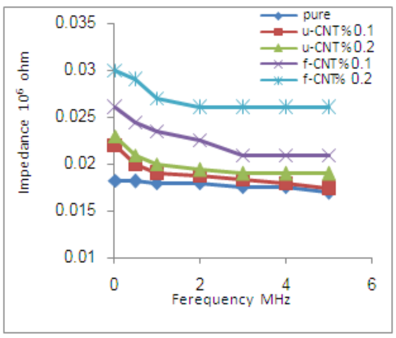


Fig(1) Variation of dielectric constant as a function of frequency for different loading of CNTs



Fig(2) Variation of dielectric loss as a function of frequency for different loading of CNTs



Fig(3) Variation of impedance as a function of frequency for different loading of CNTs