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Original Research Paper

Enhanced microwave processing of epoxy nanocomposites using carbon black powders

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ABSTRACT

The conversion of electromagnetic energy into heat depends largely on the dielectric properties of the material being treated. Therefore, the knowledge of dielectric properties of the test specimen is required in order to understand the extent of curing using microwaves. In this study, a detailed investigation is carried out by considering a number of carbon black (CB) samples having particulate sizes in the range of 15–65 nm. The dielectric properties of the synthesized CB/epoxy nanocomposites, before and after microwave curing, are measured using the advanced cavity perturbation method (CPM). It is observed that the CB/epoxy nanocomposite having smallest particulate size i.e., 15 nm attains the maximum value of dielectric constant (ε_r') and loss tangent ($\tan\delta$) of 10.79 and 0.05, respectively. These results indicate that the epoxy reinforced with the CB having least particulate size would interact more effectively with microwaves, which are confirmed by the experimental data showing that the nanocomposite with smallest CB particle size of 15 nm requires the minimum curing time. The dielectric properties especially the loss factors of fully cured samples are found to decrease after curing indicating that the dielectric properties of post cured samples can provide an idea about the extent of curing. At last, thermal, mechanical and morphological analyses are also performed on all the microwave cured epoxy samples.

1. Introduction

Advanced polymer nanocomposite materials with good mechanical strength, light weight, and excellent high-temperature characteristics have gained considerable interest in the recent years and have widely been used for various commercial applications such as aerospace, automotive, sport, energy, infrastructure, marine industry, etc. [1–4]. The huge demand for light weight, high strength materials with improved electrical and thermal properties is a major driving force for improved composite technologies [5–7]. The epoxy-based resin is now a day's quite often being used as a matrix for synthesizing the advanced composites due to the light weight, superior adhesion quality, environment resistant, excellent thermal and electrical properties [8–11]. This type of resin is basically a thermosetting epoxide polymer containing two or more oxirane rings or epoxy groups in their molecular structure [12]. The performance of an epoxy-based nanocomposite significantly depends on its curing circumstances especially in terms of mechanical and thermal properties [12,13]. Therefore, curing of epoxy plays a crucial role in the manufacturing of composite parts. However, apart from this improved performance manufacturing costs as well as time for production are also important parameters in the selection of manufacturing processing chain [14]. Curing of thermosetting epoxy resins is usually carried out by conventional thermal curing method, which quite often takes long time requiring a huge amount of energy to obtain good quality cured product. Hence, this has prompted interest to develop a fast curing method to manufacture products based on epoxy resins [15,16].

It is mainly due to the aforementioned reasons that the microwave heating has, in recent years, emerged as an alternate heating method, which has proven to be rapid and cost-effective curing procedure to produce polymer composite parts [16–18]. Microwave (MW) curing offers several advantages over the conventional curing procedure such as the reduced curing time, more efficient curing, and more uniform cure as compared to the conventional curing [18,19]. It is to be noted that although direct heating by

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microwaves can offer advantages over the conventional heat transfer, the different mechanism of energy transfer in the microwave heating has also led to new processing challenges [20]. The major challenge stems from the fact that the microwave heating is a selective process, which basically means that only those materials having a significant amount of dielectric losses can be efficiently heated using microwaves. Now, the problem is that majority of polymers including epoxy resins exhibit very low dielectric losses in the MW (GHz) region, which may lead to uncompleted and uneven curing of composites [21]. To address this issue the use of suitable filler as the microwave absorber in the insulating matrix has been proposed, which basically enhances the response of polymeric materials to microwave energy [21,22]. With the addition of microwave absorbing filler, heat supplies directly inside the sample, which led to faster curing and absolutely cured product.

Recently, a huge and significant research has been established to alter the electromagnetic properties for various applications viz. radar absorbing materials, electromagnetic interference (EMI) shielding, phantom materials, etc. Mehdizadeh and Jahangiri have reported the use of carbon black (CB) as dielectric loss absorbent with epoxy to improve the electromagnetic properties in the broad X band (8.2–12.4 GHz) for different engineering applications especially for polarimetric radar, space radar and stealth purposes [23]. Moon et al. have reported the use of CB/epoxy/dielectric powder composites for developing a phantom material in order to see the biological effects of electromagnetic radiation on human body [24]. In radiology, phantoms are utilized to estimate radiation dose delivered to patients and evaluate the quality of imaging systems. Hence, the material phantom should closely mimic the human tissue; in particular the dielectric properties of the material must be similar to that of tissue. Therefore, it is very important to understand the dielectric properties of the materials in constructing the phantom model. Luhyna et al. have shown that the addition of carbon nanotubes (CNTs) to the epoxy could provide more energy efficient microwave curing of epoxy nanocomposites, but they have not provided any measurement data regarding electromagnetic properties of resulting epoxy nanocomposites due to addition of CNTs [25]. It is important to note here that hardly any work has been reported on the influence of filler material on the dielectric properties and MW heating behavior of composites. The physics of interaction of material with electromagnetic field is of primary importance in composite material processing, which can be experimentally quantified in terms of dielectric properties and loss tangent of the resultant composites [20]. Most of the research in this area focuses on the kinetics of microwave curing and comparisons are mainly between energy and time for conventional and microwave curing techniques as well as by comparing the thermal and mechanical properties of composites cured using both conventional and microwave curing methods [8,26–28]. Nanya et al. have used microwave radiation for curing of carbon fiber/bismaleimide composites to shorten the production cycle time [29]. Yingguang et al. have developed a temperature distribution model of carbon fiber reinforced composites during microwave cure in order to closely monitor the temperature rise [17]. This study systematically reports the effect of addition of CB particles on the dielectric properties of epoxy nanocomposites. The measured dielectric properties of these composites are then related with the processing time using a typical microwave system, and the improved thermal and mechanical properties of these nanocomposites are also closely studied in order to understand the complete microwave curing procedure. In this study the CB powder of varying particle sizes (grades) in epoxy to establish the optimum parameters for the microwave curing is used. To the best of author's knowledge, such kind of systematic approach making use of experimental dielectric properties data to explain the MW curing process of CB based epoxy nanocomposites has not be presented earlier in literature.

Among the available fillers, the carbon black (CB) is commonly being used because of its ability to provide higher effective dielectric loss to an insulating matrix at relatively low filler content [12,30]. In addition to the benefits offered during the microwave processing of polymers, the incorporation of nanoscale fillers in the composites may also enhance the performance of the composite produced after curing [31,32]. It should be, however, noted that in order to take maximum advantage of the carbon black fillers to assist the microwave processing of composites, a detailed and systematic study regarding the effect of various parameters such as particle size of the CB on the electrical and mechanical properties of the resultant composite should be carried out. It has been shown that the properties of resultant epoxy nanocomposites are usually altered by the characteristics of the filler including its shape, size, volume fraction, etc. in the resin, as well as the modification of filler surfaces [26,32]. Therefore, the aim of the present work is to study the effect of particle size of CB on dielectric properties and microwave curing of epoxy nanocomposites. It is to be noted that the addition of CB to the epoxy increases its capacity to absorb microwaves, and reduces the cure cycle time as the CB can acts as nano reinforcement in the composite system. It is mainly due to this reason that a detailed study of the effect of particle size of CB on dielectric, mechanical and thermal properties of the resultant nanocomposite is carried out in this work. The dielectric properties of all the composite samples in this study were measured at 2.45 ± 0.05 GHz in S band [33].

2. Microwave dielectric theory

The dielectric properties of materials usually describe the behavior of test specimens when they are subjected to microwave field for the purpose of processing of materials. Therefore, the characterization of dielectric properties is vital for understanding the response of a material to microwaves [20]. The fundamental electrical property, which describes the ability of material to interact with microwaves and accordingly absorb the electromagnetic power, can be described in terms of the complex relative permittivity of materials represented by $\varepsilon_r^* = \varepsilon_r' - j\varepsilon_r''$. The real part of the complex relative permittivity, ε_r', represents the ability of the material to become polarized under the applied electric field. The imaginary part of the complex relative permittivity, ε_r'', represents the loss factor, which measures the ability of the material to convert electromagnetic energy into the heat. The ratio, $\varepsilon_r''/\varepsilon_r'$ is called the loss tangent or dissipation factor, an important dielectric parameter, which is used as an index of the material to generate heat. The ε_r' and ε_r'' are frequency dependent and the extent to which the material will interact with the microwaves is controlled by their magnitudes. It is mainly due to the aforementioned reasons that the measurement of dielectric properties of materials is quite important in order to determine the suitability of the test samples for the microwave assisted curing and processing [34].

3. Materials and methods

3.1. Materials

Epoxy resin was purchased from M/S Resinova Chemie, India. It is a two part system. Part A is PG100, (diglycidylether of bisphenol-A) and Part-B is PHY161 (aromatic amine-based curing agent). The density of PG100 is 1.1 g/cc. The mixing ratio was Part A (10): Part B (1) by weight. Here, the electrically conductive CB of four different grades i.e., N774, N660, N330 and N220 have been used