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MUHAMMAD RAZLAN ZAKARIA[1,2], NUR AISHAHATUL SYAFIQA MOHAMMAD KHAIRUDDIN[3], MOHD FIRDAUS OMAR[1,2*], HAZIZAN MD AKIL[3], MUHAMMAD BISYRUL HAFI OTHMAN[4], MOHD MUSTAFA AL BAKRI ABDULLAH[1,2], SHAYFULL ZAMREE ABD RAHIM[2], SAM SUNG TING[1,2], AZIDA AZMI[1]

MECHANICAL AND DIELECTRIC PROPERTIES OF HYBRID CARBON NANOTUBES-WOVEN GLASS FIBRE REINFORCED EPOXY LAMINATED COMPOSITES VIA THE ELECTROSPRAY DEPOSITION METHOD

Herein, the effects of multi-walled carbon nanotubes (CNTs) on the mechanical and dielectric performance of hybrid carbon nanotube-woven glass fiber (GF) reinforced epoxy laminated composited are investigated. CNTs are deposited on woven GF surface using an electrospray deposition method which is rarely reported in the past. The woven GF deposited with CNT and without deposited with CNT are used to produce epoxy laminated composites using a vacuum assisted resin transfer moulding. The tensile, flexural, dielectric constant and dielectric loss properties of the epoxy laminated composites were then characterized. The results confirm that the mechanical and dielectric properties of the woven glass fiber reinforced epoxy laminated composited increases with the addition of CNTs. Field emission scanning electron microscope is used to examine the post damage analysis for all tested specimens. Based on this finding, it can be prominently identified some new and significant information of interest to researchers and industrialists working on GF based products.

Keywords: Glass fibre; Carbon Nanotubes; Hybrid Material; Epoxy Laminated Composites

1. Introduction

Until recently, the progress of technology development is still relatively depending on the advancement in the field of materials [1]. A significant number of research projects focus mainly on the creation of new materials that can fulfil various consumer needs [2]. Fibre-reinforced polymer composites (FRP) are two-phase composites consisting of fibres and polymer matrixes [3]. They have been widely used in aviation, aerospace, navigation, transportation, reinforcement, and damage repair of construction structures due to their excellent characteristics, such as high specific strength and stiffness, good electrochemical performance, superior corrosion resistance, and special design versatility [4]. However, FRP shows significant disadvantages, such as weak transverse strength and poor interface between fibre and matrix [5]. Researchers have studied a range of approaches to compensate for such weaknesses in FRPs, including the use of micrometre materials and nanomaterials as a third-phase reinforcement material [6].

Nanomaterials, such as carbon nanomaterials are preferred compared to micrometre materials because of their excellent mechanical, chemical and physical properties [7]. According to their dimensions, carbon nanomaterials can be classified into 1D, 2D and 3D carbon nanomaterials [8]. Graphene is a 2D structure with strong interlayer interactions and lacks functional groups, making it easy to agglomerate in solvents [9,10]. Carbon black is a 3D spherical structure with a hydrophobic surface, which makes it difficult to disperse in solvents or polymers [11]. Compared to the materials described previously, carbon nanotubes (CNTs) are 1D structure with a special hollow morphology and extremely large aspect ratio, making them suitable third-phase reinforcement materials [12]. Iijima and his co-workers discovered CNTs with characteristics of lightweight, high strength, high modulus and good toughness in 1991 [13]. In accordance with their number of layers of graphene sheets, CNTs divided into single-walled carbon nanotubes (SWCNTs), double-walled carbon nanotubes (DWCNTs), and multi-walled carbon nanotubes (MWCNTs) [14]. The SWCNTs and DWCNTs have greater

1 UNIVERSITI MALAYSIA PERLIS (UNIMAP), FACULTY OF CHEMICAL ENGINEERING TECHNOLOGY, PERLIS, MALAYSIA
2 UNIVERSITI MALAYSIA PERLIS (UNIMAP), GEOPOLYMER & GREEN TECHNOLOGY, CENTRE OF EXCELLENT (CEGEOGTECH, PERLIS, MALAYSIA
3 UNIVERSITI SAINS MALAYSIA, SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING, ENGINEERING CAMPUS, 14300 NIBONG TEBAL, PULAU PINANG, MALAYSIA.
4 UNIVERSITI SAINS MALAYSIA, SCHOOL OF CHEMICAL SCIENCES, 11800 MINDEN, PENANG, MALAYSIA

* Corresponding author: firdausomar@unimap.edu.my

inertness and less active surface functional groups, rendering mixing with other substances difficult [15]. However, more active functional groups like hydroxyl and carboxyl are found on the surface of MWCNTs, which makes it easier to combine them with other materials [16]. Therefore, MWCNTs were chosen to modify glass fibre-reinforced polymer composites (GFRP) in the present study in consideration of mechanical performance and cost-effectiveness.

There are two common methods for the production of hierarchical composites based on MWCNT. One is to disperse MWCNTs into the matrix and then inject the modified resin into fibre cloth to form composite laminates [17]. Second, MWCNTs are deposited directly on the fibre surface, and then the modified fibre cloths are combined with resin to form composite laminates [18]. The introduction of MWCNT particles in the matrix will lead to the high viscosity of the resin and low solvent resistance, which will greatly reduce the performance of the resin. However, if the MWCNTs are deposited directly on the surface of the fibre, the MWCNTs on the fibre surface will be distributed randomly in various directions, increasing the effective diameter of the single fibre and extending the contact area between the fibre and the matrix and enhancing the efficiency of load transfer between the fibre and matrix [19,20].

Therefore, in this study, from the perspective of glass fibre (GF) modification, which has rarely been studied before, MWCNTs are deposited on the GF surface using the electrospray deposition method, and the responses of deposition of MWCNT to the mechanical and dielectric properties of GFRP are further discussed.

2. Experimental Set-up

Multi-walled CNT (external diameter of 20-30 nm, internal diameter of 5-10 nm, length of 10-30 µm) was purchased from Sky Spring Nanomaterials Inc., Houston, TX, USA (95% pure and used as-received). N-Methyl-2-Pyrrolidone (NMP) was purchased from Sigma-Aldrich, St. Louis, MO, USA, and used as received.The epoxy resin and curing agent were used in polymer nanocomposites are DER 331 and Epoxy Hardener Clear. The DER 331 and Epoxy Hardener Clear were purchased from Eurochemo Pharma Sdn. Bhd.

For electrospray preparation, 0.005 g CNTs were dispersed in 5 ml of the DMF by using sonicator at a frequency of 50 kHz for 30 minutes. Then, the epoxy resins DER 331 were mixed with the Epoxy Hardener Clear at 10:6 by weight ratio. After that, 0.003 g of the resulting mixture was added to a CNT dispersion and ultrasonicated for another 30 minutes in order to improve attachment strength of CNT onto GF surface. The general view of the electrospray setup is shown in Figure 1. The equipment consists of a stainless-steel needle (with 0.3 mm inner diameter and 0.55 mm outer diameter) and a steel platform as a ground electrode. The precision high voltage power supply (Model type ES20P-20W) which capable of delivering an applied voltage of 20 kV with a resolution of 0.1 kV is connected to the stainless-steel needle (with 0.3 mm inner diameter and 0.55 mm outer diameter). The needle is fitted to a syringe of 20 ml and the syringe sits firmly on a precision syringe pump (Model type NE-1600, New Era Pump Systems, Inc., USA), which can deliver low flow rates up to 0.001 µL/hr. Woven GF fixed onto the roller and the steel platform connected with an earth wire. The electrospray deposition process was then carried out as explained. By rotating the roller at 120 rpm, the woven GF was sprayed by turn to achieve an even deposition and coating. The distance between the needle and roller was fixed to be 10 cm, and the flow rate was 0.02 mL/hr with applied voltage 15 kV. On completion of the spray process, the woven GF was left to dry for 24 hours, and the spray process was repeated on the opposite side. A field emission scanning electron microscope (FESEM) (ZEISS Supra 55 SEM VP) was used to analyse the morphology of the woven hybrid GF-CNT. The woven hybrid GF-CNT was coated with a thin layer of gold to improve their conductance for observations using FESEM.

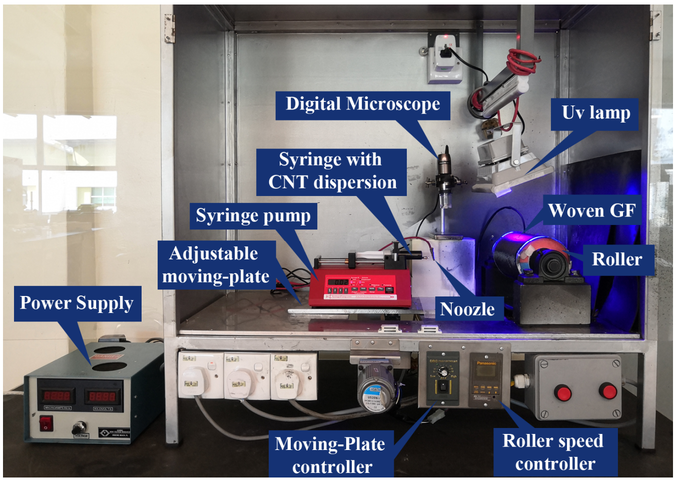

Fig. 1. General view of electrospray deposition process

Woven hybrid GF-CNT epoxy laminated composites were prepared by using the vacuum assisted resin transfer moulding method (VARTM). The layers of woven hybrid GF-CNT were stacked together into four plies and were put into the mould. The vacuum was used to get rid of the air trapped inside the mould and to assist the resin flow throughout the VARTM. After the impregnation process is completed, the woven hybrid GF-CNT epoxy laminated composites were left to cure for 1 day of post-cured at room temperature. The cured woven hybrid GF-CNT epoxy laminated composites were removed from the mould and cleaned. The same process was used to prepare woven GF epoxy laminated composites. The description of the samples was presented in TABLE 1.

The tensile test was conducted using a universal testing machine (Model: 5982, Instron, USA) by following the ASTM 3039 standard. Samples were cut into pieces with a measurement of dimension 250 mm × 25 mm × 2.5 mm. Five specimens of each composite were measured tested to ensure the test results were reliable. The flexural test was carried out by using a univer-

TABLE 1

Description of the samples

Sample	Description
Epoxy	Epoxy without reinforcement
GF/Epoxy	Epoxy reinforced with 4 layers of woven GF
Hybrid GF-CNT/Epoxy	Epoxy reinforced with 4 layers of woven hybrid GF-CNT

sal testing machine (Model: 5982, Instron, USA) by following ASTM D 790. Samples were cut into pieces with measurement of dimension 80 mm ×13 mm × 32 mm. Five specimens of each composite were measured tested to ensure the test results were reliable. The fracture surface of the tensile specimen was coated with a thin layer of gold and observed using FESEM. Dielectric properties of the samples were measured using Agilent 4284A LCR meter at room temperature over a frequency range from 20 Hz to 1 MHz.

3. Results and Discussion

3.1. Morphology analysis

The surface morphologies of the woven GF and woven hybrid GF-CNT were characterized by using FESEM as depicted in Figure 2. Figure 2a) shows the morphological structure of the GF with diameter of about 13 μm. In addition, the smooth surface of GF along their axis also can be clearly seen. Figure 2b) shows the SEM images of the woven hybrid GF-CNT produced at voltage 15 kV. It was experimentally proven that the CNTs were homogeneously distributed and covered the entire surface of the GF.

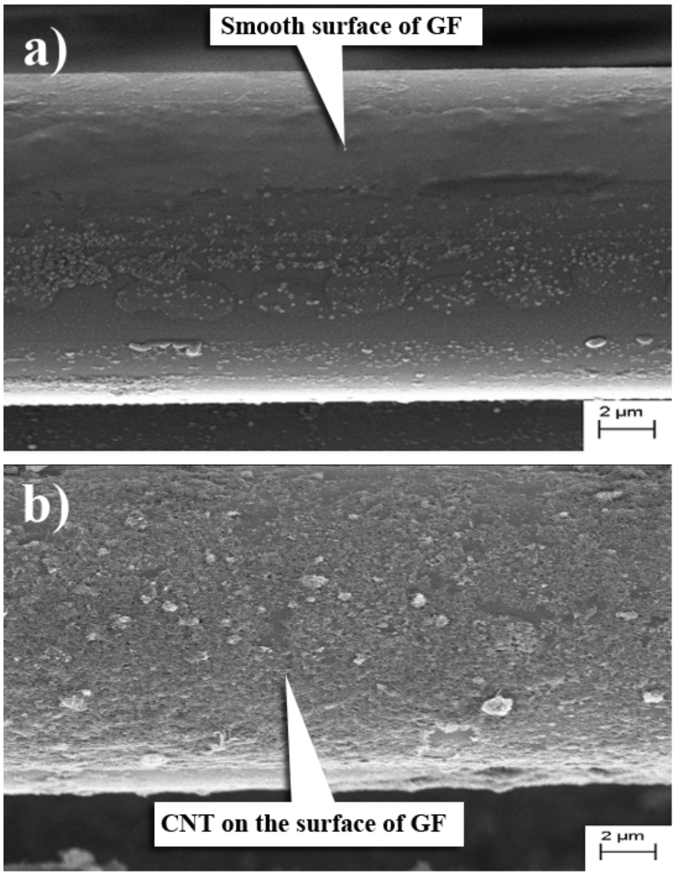

Fig. 2. SEM images of (a) woven GF and (b) woven hybrid GF-CNT

3.2. Tensile Properties

Tensile strength is a measurement of the material ability to resist breaking under tensile stress. It is one of the most important properties of materials that are being used in a composite's applications [21]. The tensile test was used to evaluate the effect of CNTs on the mechanical properties of the epoxy laminated composites. Figure 3 illustrates the comparative study of the typical tensile stress-strain curve of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy. From the graph, it demonstrates the increasing value of tensile stress experience suddenly dropped to a minimum value which represents the brittle fracture of the epoxy laminated composites. This phenomenon occurs most probably due to the matrix-reinforcement interaction which has changes the matrix deformation from ductile to brittle material through the interfacial reinforcing mechanisms [22]. The experimental results reveal that the Hybrid GF-CNT/Epoxy samples give better tensile properties than Epoxy and GF/Epoxy. For the tensile strain, the Epoxy without reinforcement failed at a very low strain of about 2.3%. For GF/Epoxy tensile strain increased by 26% and the tensile strain of the Hybrid GF-CNT/Epoxy increased by 17%. The tensile strain of Hybrid GF-CNT/Epoxy was slightly lower than GF/Epoxy due to the addition of the CNT, which increased the rigidity of the epoxy laminated composites.

Figure 4 shows the tensile strength and tensile modulus of the Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy. From the result potrayed in Figure 4, it is found that the tensile strength and tensile modulus of the Hybrid GF-CNT/Epoxy is higher than that of both Epoxy and GF/Epoxy. The Hybrid GF-CNT/Epoxy demonstrated the highest tensile strength of 1.74 GPa and the highest tensile modulus of 57.3 GPa. For the tensile strength, the addition of CNT on the GF surface improved interfacial bonding leads to an improvement in the interfacial strength between CNT and fibre and results in an effective transfer of stress between CNT and fibre [23]. The presence of CNTs, which alter the behavior of the interface, is responsible for the increased tensile strength. CNT deposition on the GF surface increases the surface roughness of GF and induces mechanical interlocking, which improves stress transfer between the GF and the epoxy matrix [24]. For the tensile modulus, the presence of CNTs on the GF surface provides a strengthening mechanism by serving as a bridge to the defect, increasing the flexural modulus of the Hybrid GF-CNT/Epoxy. Furthermore, the presence of CNTs on the surface of fiber has resulted in the formation of transition layers in the interface area, which affect the strength and toughness of the epoxy matrix [25]. The stress transfer efficiency also improved significantly between the fiber and the matrix that lead to the higher flexural modulus.

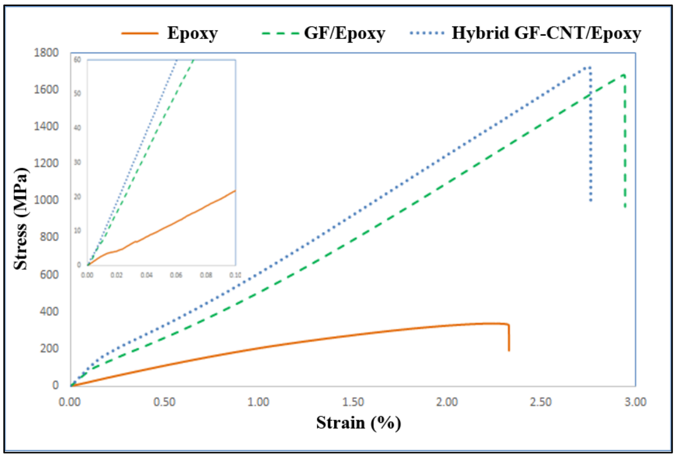

Fig. 3. Tensile stress-strain curves of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy

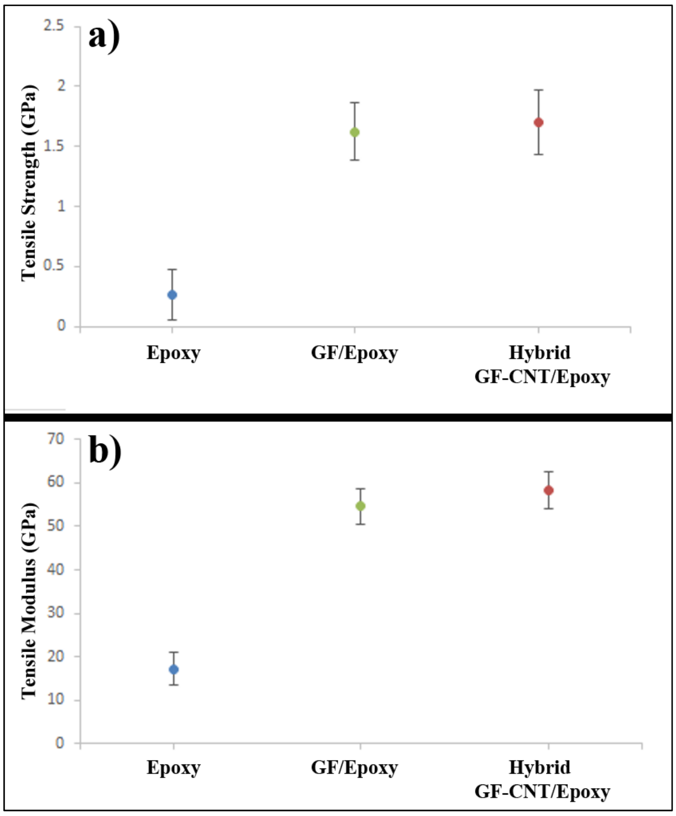

Fig. 4. Tensile properties of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy in terms of (a) tensile strength, and (b) tensile modulus

3.3. Flexural properties

Flexural properties of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy are shown in Figure 5. Flexural properties of Hybrid GF-CNT/Epoxy were significantly improved by depositing the CNT on the surface of GF. From the graph in Figure 5, the value of flexural strength for Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy were 25 MPa, 124 MPa, and 174 MPa, respectively. Flexural strength of the Hybrid GF-CNT/Epoxy increased about 40% as compared to the GF/Epoxy. Improving the flexural strength of the Hybrid GF-CNT/Epoxy may have been related to the significant presence of the CNT on the GF surface, which also shared the stress transfer with the GF. For the flexural modulus, the deposition of CNT has improved the flexural modulus of the Hybrid GF-CNT/Epoxy about 52% as compared to the GF/Epoxy. Such an enhancement could be attributed to the deposition of the CNT that filled the voids on the woven GF [26]. Generally, there are gaps between the GF tows that cross each other on the woven GF, which can contribute to air bubbles becoming trapped during the production of the composite. As a result, the CNT deposition on the woven GF seemed to be accountable for this change and also resulted in improved interlocking between the GF and the epoxy matrix.

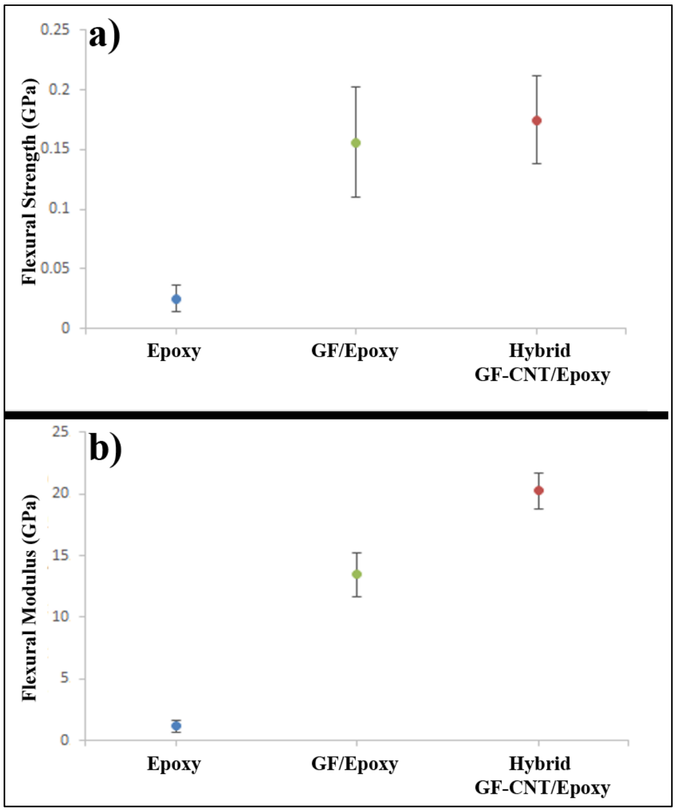

Fig. 5. Flexural properties of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy in terms of (a) flexural strength, and (b) flexural modulus

The fracture morphology of epoxy laminated composites demonstrated the difference in the failure structure and effects of the CNT deposition on the woven GF surfaces. Figure 6 shows the fracture surfaces Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy. For Epoxy, a smooth fracture surface can be seen clearly without any GF. For GF/Epoxy and Hybrid GF-CNT/Epoxy, it can be seen the GF bundles were break up into broom like structure in the epoxy matrix after tensile test. The result also revealed the features of many epoxy fragments and some gaps present between GF and epoxy matrix. For comparison, Hybrid GF-CNT/Epoxy shows good fibre-matrix bonding compared to the GF/Epoxy. Generally, the deposition of CNT on the GF surface was able to help improve the interfacial bonding between the matrix and fibre interface and also improved the transfer of load between the fibre and the matrix.

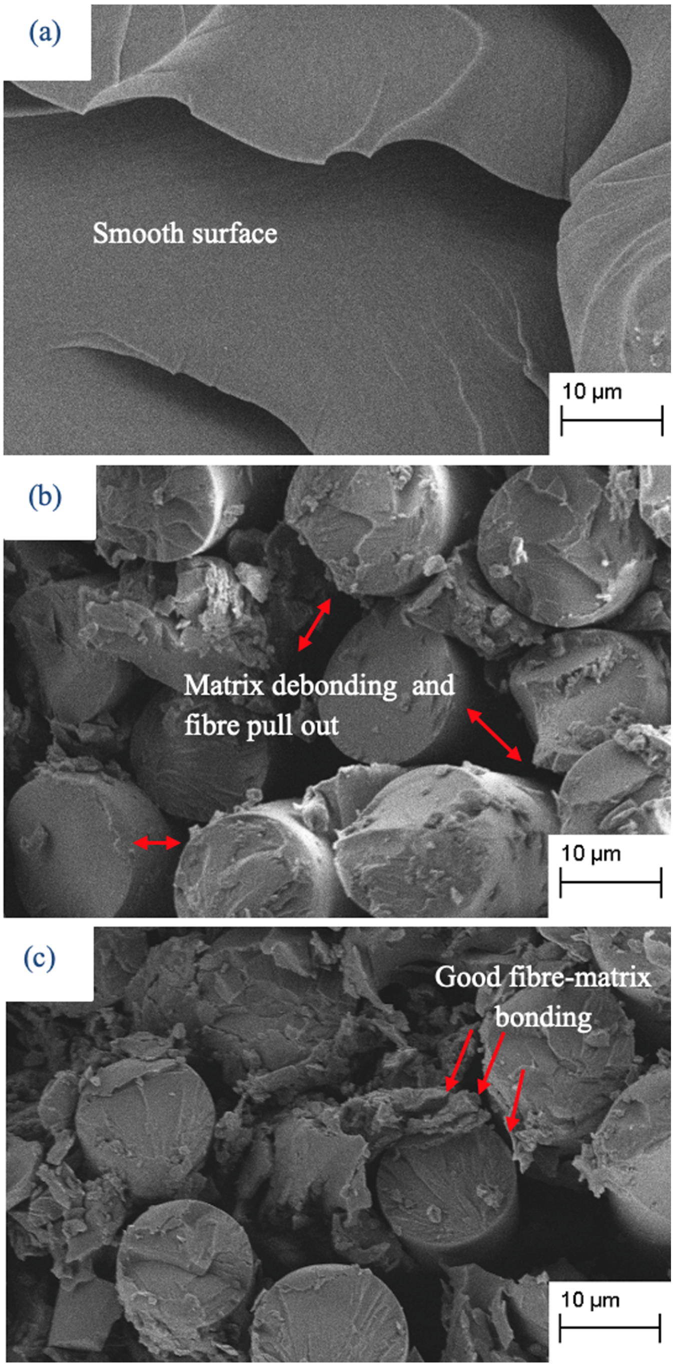

Fig. 6. SEM images of the tensile fractured surfaces of (a) Epoxy, (b) GF/Epoxy and (c) Hybrid GF-CNT/Epoxy

3.4. Dielectric properties

Dielectric constant measures the materials capability to store charge whereas the tan delta (loss tangent) is used to express the material ability in energy dissipation [27]. Figure 7 shows the dielectric constant (real permittivity) (ε') as a function of the frequency at room temperature for Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy. The dielectric constant is one of important parameter that offers insights into the filler and its applicability in electronic applications. The CNT is well-known for its high dielectric constant filler, which is introduced into the epoxy matrix to produce high dielectric constant composite materials [28].

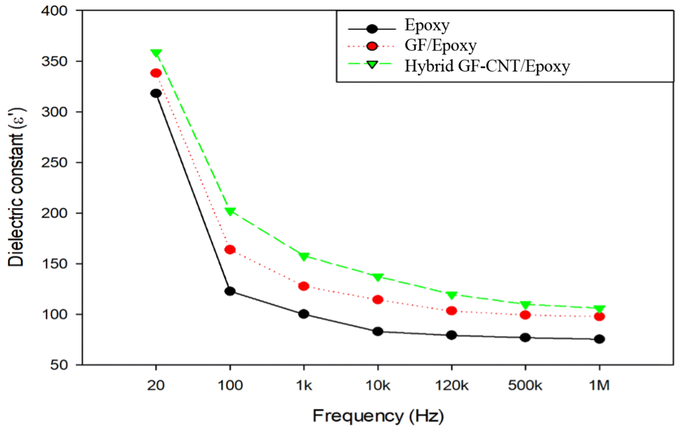

Fig. 7. Frequency dependent dielectric constant of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy

The result demonstrates that the incorporation of CNT into Hybrid GF-CNT/Epoxy increase the dielectric constant values of the epoxy laminated composites. The dielectric constant value of Hybrid GF-CNT/Epoxy is higher than GF/Epoxy and Epoxy. The dielectric constant for Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy decreased as frequency increased. It was exciting to reveal that the dielectric constant of all samples varied with frequency. The changes of the dielectric constant was associated to the free charge carriers that are moving at constant motion in these frequency range. The slight reduction of dielectric constant under higher frequency is due to the effect of the polarisation reduction when the frequency is higher [29]. At lower frequencies, the interfacial polarisation would be high and changes in the electric field are slower. In this situation, the free dipolar functional groups that exist in the epoxy chain may aligned themselves freely. At higher frequency however, the very fast action of electric field changes resulting to lesser time for surface charges to polarise. Therefore, the surface charges polarisation decreases at higher frequency and subsequently resulted in reduction of dielectric constant.

As shown in the graph, the incorporation of CNT on woven glass fibre improves the dielectric constant of epoxy laminated composites significantly. At a frequency of 20 Hz, the GF/Epoxy shows a 6.23% increase over Epoxy. Meanwhile, at a frequency of 20 Hz, the Hybrid GF-CNT/Epoxy shows a significant increase of about 12.90% when compared to the Epoxy. The leading of an increment of dielectric constant achieved by Hybrid GF-CNT/Epoxy dielectric is due to the good CNT dispersion, which gives the epoxy nanocomposite some polarity and generates significant amounts of micro-capacitors [12]. The vast number of microcapacitors therefore results in higher dielectric constant for the epoxy nanocomposites. The variation dielectric loss (ε") value as a function of frequency for Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy are shown in Figure 8. Dielectric loss is the measurement of the energy lost into the system according to the amount of frequency applied and the charge carrier movement. According to Figure 8, the dielectric loss of all these samples decreases as the frequency range increases (20 Hz-1 MHz) and then nearly remains constant at higher frequencies. When the frequency was increased further, the effect of dipole polarization reduced the orientation polarization because the chain motion of the polymer could not maintain phases with the higher oscillating electric field, causing the loss factor value to decrease accordingly. The low value of dielectric loss is beneficial in electronic application due to the low energy loss.

At a frequency of 20 Hz, the dielectric loss of the Hybrid GF-CNT/Epoxy is 0.58, compared to 1.10 and 1.64 for the GF/Epoxy and Epoxy, respectively. The low value of dielectric loss is beneficial in electronic application due to the low energy loss. This indicates that Hybrid GF-CNT/Epoxy has favorable dielectric loss compared to other GF/Epoxy and Epoxy samples. The presence of a barrier at the charge transport in a polymeric system accounts for the low dielectric loss. This phenomenon can occur as a result of interchain charge transport and interface transport. Furthermore, polymer chain entanglements and the presence of several interfaces hinder charge movement in the system and reduce dielectric loss [30].

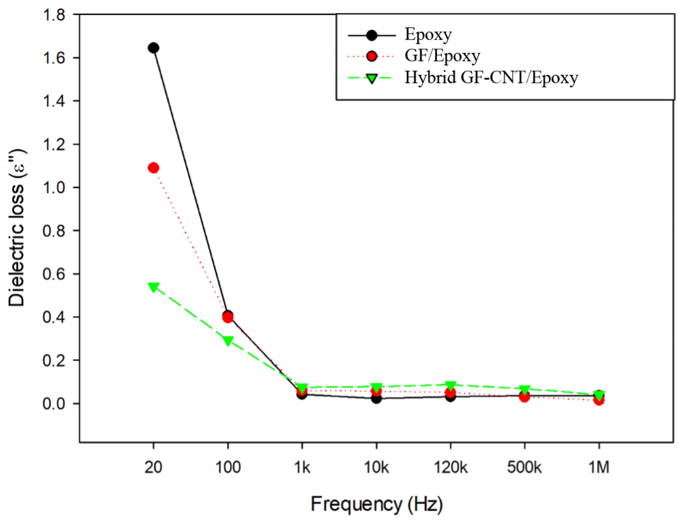

Fig. 8. Frequency dependent dielectric losses of Epoxy, GF/Epoxy and Hybrid GF-CNT/Epoxy

4. Conclusion

The main objective of this study is to investigate the enhancement of the mechanical and dielectric properties of woven GF epoxy laminated composites by incorporating CNT. The approach used in this study for the introduction of CNT is less studied in the literature. The method is to deposit CNT on the woven GF surface using the electrospray deposition method. The results demonstrate the introduction of the CNT were greatly enhanced the mechanical and dielectric properties of Hybrid GF-CNT/Epoxy. FESEM observations show that CNT deposited on the GF surface effectively increases the interface region, resulting in strong interfacial bonding. FESEM observations show that CNT deposited on the GF surface effectively expand the interface area, resulting in strong interfacial bonding. Thus, the mechanical and dielectric are improved, which approximately matches the experimental results. The Hybrid GF-CNT/Epoxy dielectric properties have been improved, creating an opportunity for printed circuit board applications. Since each component on the PCB requires a different dielectric constant, varying the amount of CNT deposition can be used in the future to modify the dielectric constant of the PCB.

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