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Flexural properties for two-ply Glass Fiber Reinforced composites with different loading of CNT/Epoxy film Produced by different Methods

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ABSTRACT

Background: Composite materials reinforced by glass fibers are widely used in various applications ranging from aerospace to sporting goods due to their remarkable properties including high strength to weight ratio and high modulus to weight ratio. However, the addition of nanoparticles as reinforcement shows promising results for properties enhancements. Unique properties of nanoparticles such as carbon nanotubes (CNTs) make them potential candidates for many applications from mechanical enhancements to electrical and thermal conductivities. **Objective:** In this work, the effect of CNTs loadings and different methods used to fabricate CNT/epoxy film on flexural properties of two-ply glass fiber reinforced composites were investigated. Various CNTs loadings ranged from 0 vol% to 2.0 vol% were used to fabricate the films by using casting and hot press methods. The films were then stacked in between 2-ply of glass fiber reinforced epoxy composite by vacuum bagging process. **Results:** The results shows that the 0.5 vol% cured CNTs/epoxy film fabricated by using hot press method demonstrated better flexural properties compared to other CNTs loadings and fabrication methods. Hot-press sample shows improvements in flexural strength and modulus which were 190.5% and 368.6% respectively followed by casting (187.2% and 360.0%) and prepreg (169.8% and 330.8%) compared to the neat glass fiber composite. However, the glass fiber reinforced composite with 2.0 vol% CNTs film shows lower improvement (72.0% and 152.7%). **Conclusion:** Casting and hot press methods were used to fabricate film and the flexural properties of two-ply glass fiber reinforced composites were investigated. The results exhibited that the flexural properties were influenced by properties of the film, methods to produce film and the adhesion between film and glass fiber mats. For future works, it is aimed to optimize film and laminates processing parameters to enhance structural properties of the fiber reinforced composites.

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INTRODUCTION

Glass fiber-reinforced plastics (GFRPs) composite are widely used in various structural applications such as aerospace, automotive, sporting goods and marine due to their remarkable properties including high strength to weight ratio and high modulus to weight ratio. Composites materials are commonly used due to their adaptability to various conditions and ease of combination with other materials in order to perform specific purposes and achieve desirable properties. However, performance of final composite depends on various parameters such as matrix materials, reinforcing fibers, fiber alignments and fiber directions.

Recent developments in fiber reinforced polymer composites shifted direction towards application of nanoparticles for properties enhancements. The addition of nanoparticles as reinforcement shows promising results for properties enhancements and thus will maximize their applications. Unique properties of nanoparticles such as carbon nanotubes (CNTs) in terms of high mechanical strength and stiffness, exceptional thermal conductivity, low density and high aspect ratio make them very good candidates for many applications from mechanical enhancements to electrical and thermal conductivities (Baughman *et al*., 2002; Zhidong *et al*.,

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2011; Kesong *et al*., 2014). Excellent mechanical properties of CNTs make them potential candidates as advanced filler materials for polymer composite reinforcement. In another area, combination of their conductivity and high aspect ratio were predicted to create conductive polymer with exceedingly low percolation thresholds (Kilbride *et al*., 2002). Biercuk *et al*. (Biercuk *et al*., 2002) reported that their massive thermal conductivity can be exploited to produce thermally conductive composites. However, it is foreseen that the most promising area of polymer using CNTs as reinforcing fillers will be more significant on mechanical enhancement (Jonathan *et al*., 2006). Christopher *et al*. (Christopher *et al*., 2008) have incorporated epoxy with CNTs into glass fiber polymer composites by using wet lay-up method and cured the samples by using heated platen press. It was reported that the CNTs exhibit a relatively uniform distribution in the polymer with little agglomeration and clumping. Ramlee *et al*. (Ramlee *et al*., 2013) investigated the effect of CNT content and hybrid flame-retardant filler on the properties glass fiber/epoxy laminated composites and found out that CNT-filled epoxy/glass fiber composite exhibit higher tensile properties and burning rate than hybrid flame-retardant system. Wan Dalina *et al*. (Wan Dalina *et al*., 2014) compared the performance of the glass fiber and carbon fiber/epoxy laminated composites. The results shows that addition of 0.5 vol% of CNT in the 4-ply system of glass fiber increases the flexural properties compared to carbon fiber laminated composite.

This study aims to investigate the effect of CNTs loadings and different methods used to fabricate CNT/epoxy film on flexural properties of two-ply glass fiber reinforced composites. Various methods have been reported in previous works to fabricate films composite such as layer by layer (LBL), casting, spin coat, electrophoretic deposition (EPD) and buckypapers (Mamedov *et al*., 2002; Martone *et al*., 2010; Ghaleb *et al*., 2014; Zhang *et al*., 2009; Wang *et al*., 2004). CNTs loadings ranged from 0 vol% to 2.0 vol% were dispersed in epoxy system and the films were fabricated by using casting and hot press methods. The films were then stacked in between 2-ply of glass fiber reinforced epoxy composite by vacuum bagging process.

MATERIALS AND METHODS

Materials:

The materials used in this study were an epoxy resin D.E.R.^{TM} 332 (diglycidyl ether of bisphenol-A) cured with polyetheramine D230 hardener, with weight ratio of 100:32, both supplied by Penchem Technologies Sdn. Bhd. The density of materials were 1.16 g/cm^3 and 0.946 g/cm^3 respectively. The multi-walled carbon nanotubes (MWCNTs) used in this study were purchased from USAINS Holding, Universiti Sains Malaysia. The MWCNTs were produced via catalytic chemical vapour deposition (CCVD) process with an average diameter and length ranged 10 ± 1 nm (mean ± standard deviation) and 1-5 µm, respectively. Sodium dodecyl sulfate (SDS) was used as the dispersant supplied by Sigma-Aldrich Corporation. The reinforcement was woven type-E glass fibers with a linear density of 200 g/m^2 supplied by Saint-Gobain Ltd.

Composite fabrication:

The general process of composites fabrication were divided into three stages; solution preparation, film composites fabrication and glass fiber reinforced composites fabrication. Various CNTs loadings (0 vol% to 2.0 vol%) were dispersed in acetone and dispersant, then placed in the ultrasonic bath for 30 min. The mixture was then placed in the oven at 60°C for approximately 1h to evaporate the solvent. Then, the mixtures were suspended in epoxy resin and sonicated using Hielscher UP200S Ultrasonicator at 24 kHz and 50% amplitude for 10 min at room temperature. A hardener was added at a ratio of 100:32 by weight (epoxy: hardener) and the mixture was sonicated for 10 min in an ice bath. Thereafter, the mixture was vacuumed at room temperature for 30 min to remove entrapped air. The controlled samples (neat epoxy) were prepared following the same procedure for comparison purposes.

Film composites with dimensions of 155 mm × 245 mm were fabricated based on three different conditions; (1) *Casting (fully cured)*. The mixtures were cast into silicon mold and cured at room temperature for 24h followed by post-cured at 80°C for 2h, (2) *Casting (prepreg)*. The mixtures were cast into silicon mold and partially cured (prepreg) at 50°C for 2h and (3) *Hot-press (fully cured)*. The mixtures were moulded into a flat steel plate and pressed using hot press GOTECH GT-7014-A at 300 psi and 80°C for 2h. The thicknesses of thin film composites produced were within ranges of 0.3-0.5mm. Glass fiber/epoxy composite laminates were fabricated by stacking up thin film composite (casting-fully cured/ casting-prepreg/hot press-fully cured) in between two-ply of glass fibers. The composite laminates were consolidated for 30 min using the vacuum bagging technique. Then, the composite laminates were cured at room temperature for 24h, followed by post cure at 80°C for 2h.

Characterisation:

The flexural properties of 2-ply glass fiber reinforced epoxy composite were performed using three-point bending tests according to the ASTM D790 standard test method at a crosshead speed of 2 mm/min (INSTRON series IX/s Automated Material Tester Version 8.25.00). Five specimens with a dimension of 50.8 mm x 12.7

mm (for sample with thickness, t < 1.6mm) and the span length is 25.4 mm were prepared and tested. The fracture surfaces of the thin film were examined using scanning electron microscopy (FESEM Zeiss SUPRA 35VP) at an acceleration voltage of 10kV. The samples surface were vacuum coated with a thin layer of gold palladium to provide conductive surface for testing.

RESULTS AND DISCUSSION

Flexural tests were performed to evaluate the flexural strength and modulus of the two-ply glass fiber reinforced composites. Figure 1 shows the comparison of the representative flexural stress-strain curves for two-ply glass fiber reinforced composites with different loading of CNTs/epoxy film produced by different methods. The stress-strain curves of the tested samples represents the effect of CNTs loadings on the mechanical behavior of the composites. As the CNTs loadings increased, the areas under the stress-strain curve increases. It shows that the toughness of the composites increased with the addition of CNTs. However, at 2.0 vol% CNT the areas under the curve decreases which due to the agglomeration of CNTs in epoxy film deteriorates the overall flexural properties of the composites. The addition of CNTs more than 0.5wt% was very challenging due to the massive surface area of CNTs and leads to increase in viscosity (Njuguna *et al.*, 2007). Amongst the tested samples, 0.5 vol% CNTs exhibits better performance compared to neat epoxy and 2.0 vol% CNTs. Hot press methods shows higher flexural properties followed by casting and prepregs.

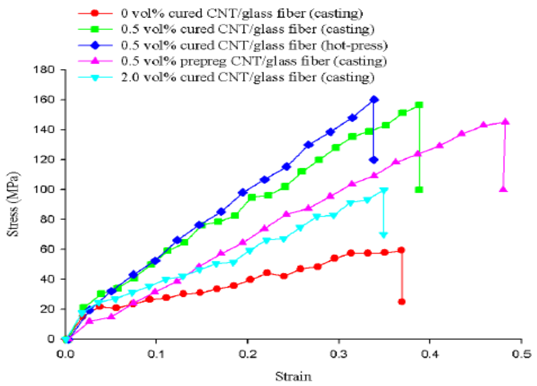

Fig. 1: Comparison of the flexural stress-strain curves for two-ply glass fiber reinforced composites with different loading of CNTs/epoxy film produced by different methods.

Table 1 and Figure 2 shows the flexural properties of two-ply glass fiber reinforced composites with different loading of CNTs/epoxy film produced by casting and hot-press methods. The results shows that the flexural properties increased consistently as the CNTs loadings increased. Similar trend was reported by the previous works (Wan Dalina *et al*., 2014; Luen *et al*., 2012; PC Ma *et al*., 2007; Mora *et al*., 2009; Breton *et al*., 2004). The composites containing 0.5 vol% CNTs loadings exhibited maximum values in flexural strength and modulus compared to neat epoxy and 2.0 vol% CNTs loadings. Hot-press sample shows slightly higher in flexural strength and modulus ranged 156.6 ± 27.19 MPa and 79.2 ± 34.65 GPa respectively followed by composite samples prepared by casting (154.8 ± 52.03 MPa and 77.9 ± 19.40 GPa) and prepreg (145.4 ± 28.77 MPa and 72.8 ± 13.86 GPa) methods. In this work, the hot press methods shows improvements in flexural strength and modulus which are 190.5 % and 368.6% compared to the neat glass fiber composite. However, the glass fiber reinforced composite with 2.0 vol% CNTs film shows lower improvement (72.0% and 152.7%). This scenario was due to the agglomeration of CNTs in epoxy film deteriorates the overall flexural properties of the composites. For prepreg, the variation of thickness causes non-uniform stress was not been transferred uniformly in the composites and leads to lower flexural properties compared with hot press and casting samples. The thickness of prepreg was uneven due to the shrinkage of epoxy matrix during curing stages (Koran *et al.*, 1983). Tack is one of the prepreg properties that have to be monitored. The epoxy content of prepreg influences the tack and as well as the final strength of the laminate. Tack refers to the ability of a prepreg to adhere to itself or to other material surface. In many prepreg systems, the resin content of the prepreg is higher than desired in the finishing part. This improves tack and drape but the excess resin must be removed in the manufacturing process. The removal of this excess resin assists the removal of trapped air in the laminate. It is essential to remove trapped air, since voids within laminate have a negative effect on interlaminar strength (Peterson *et al.*, 1992). The defect caused by trapped air was observed in Figure 3.

Figure 4 represents the morphologies of composites containing 0 vol%, 0.5 vol% and 2 vol% CNTs loadings. Figure 4a shows the cleavage plane between two cleavage steps that were flat and smooth at different magnifications. This indicated a typical fractography feature of brittle fracture behavior for neat epoxy. The fracture surface of the CNTs composite (Figure 4b-c) shows different fractographic features; rougher with the CNTs added into epoxy matrix, which indicates ductile deformation and smooth fracture surface suggests that it had a brittle failure mode. Additionally, the white points indicates the CNTs dispersed in the epoxy resin. The white points increases as the loadings of CNTs increased. In this work, the 0.5 vol% CNTs film shows better dispersion due to low agglomeration in the mixture. Hence, the glass fiber reinforced composite with 0.5 vol% CNTs film shows higher improvement in flexural properties as compared to the neat glass fiber composite. However, the glass fiber reinforced composite with 2.0 vol% of CNTs film gives lower improvement (72.0%) due to high agglomeration occurs in the film. From the SEM images, microvoid and void were also observed which will initiates the propagation of cracks. This scenario occurs due to the insufficient degassed application to remove entrapped air from the mixture during solution processing.

Table 1: Flexural properties of two-ply glass fiber reinforced composites with different loading of CNTs/epoxy film produced by different methods.

CNTs Loading (vol%)	Flexural Strength (MPa)	Flexural Modulus (GPa)
0 vol% cured CNT/glass fiber (casting)	53.9 ± 10.79	16.9 ± 2.07
0.5 vol% cured CNT/glass fiber (casting)	154.8 ± 52.03 (+187.2%)	77.9 ± 19.40 (+360.0%)
0.5 vol% cured CNT/glass fiber (hot-press)	156.6 ± 27.19 (+190.5 %)	79.2 ± 34.65 (+368.6%)
0.5 vol% prepreg CNT/glass fiber (casting)	145.4 ± 28.77 (+169.8 %)	72.8 ± 13.86 (+330.8%)
2.0 vol% cured CNT/glass fiber (casting)	92.7 ± 12.02 (+72.0%)	42.7 ± 7.38 (+152.7%)

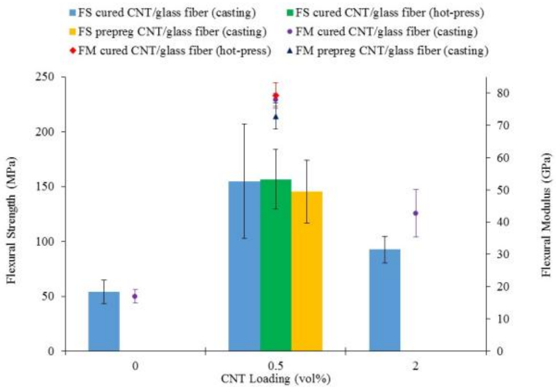

Fig. 2: The bar charts of flexural strength (FS) and modulus (FM) for two-ply glass fiber reinforced composites with different loading of CNTs/epoxy film produced by different methods.

Fig. 3: Void caused by trapped air in sample prepared by preimpregnated CNTs/epoxy thin film and glass fiber mats at 45x magnification.

Conclusion:

Flexural properties for two-ply glass fiber reinforced composites with different loading of CNT/Epoxy film produced by different methods were investigated. The increase of CNTs loading increases the flexural properties where 0.5 vol% exhibited better performance compared to other CNTs loadings. The flexural properties of composites are depending on the dispersion and presence of defects in the CNTs/epoxy film. Higher CNTs loading increases the tendency of agglomeration of CNTs in the epoxy system which causes poor dispersion of CNTs and creates stress concentration. Methods used to fabricate the CNTs/epoxy film will also influence the performance of the composites. Hot-press sample shows improvements in flexural strength and modulus which were 190.5% and 368.6% respectively followed by casting (187.2% and 360.0%) and prepregs (169.8 % and 330.8%) compared to the neat glass fiber composite. However, the glass fiber reinforced composite with 2.0 vol% CNTs film shows lower improvement (72.0% and 152.7%). The results shows that the 0.5 vol% cured CNTs/glass fiber composites fabricated by using hot press method demonstrated better flexural properties. The flat surface of hot-pressed film has smaller contact areas with glass fiber and this scenario creates smaller air gaps and minimize voids after curing and hence improve the adhesion between the surface contacts.

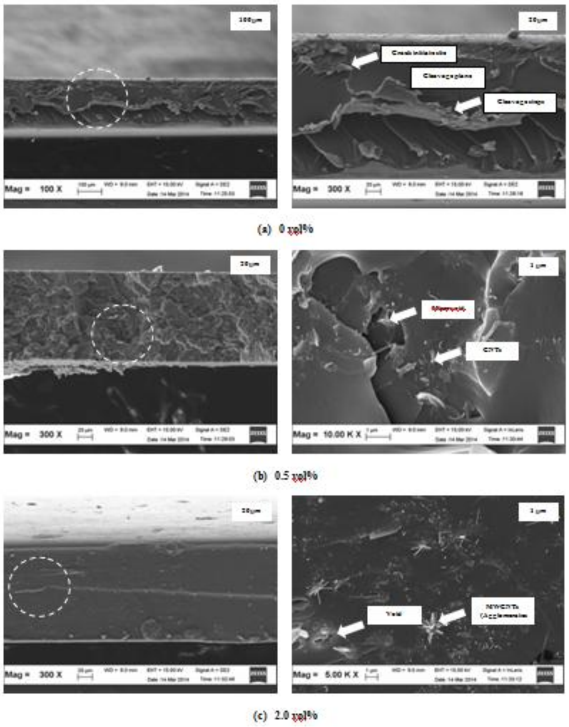

Fig. 4: SEM images of the fracture surfaces of two-ply glass fiber reinforced composites with different CNTs loadings at different magnifications: (a) 0 vol%, (b) 0.5 vol% and (c) 2.0 vol%.

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