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Carbon-nanotube-grafted glass-fiber-reinforced composites: Synthesis and mechanical properties

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ABSTRACT

Glass fibers (GFs) are commonly used as reinforcements for advanced polymer composites. To improve the interfacial shear properties and mechanical properties of GF-reinforced composites (GFRPs), carbon nanotubes (CNTs) are directly grafted onto GFs using chemical vapor deposition (CVD). However, this process requires high temperatures, which causes thermal degradation of GFs, deteriorating their mechanical properties. In this study, a low-temperature CNT-grafting process was investigated using a bimetallic catalyst introduced onto a GF fiber surface via precursor solutions. The mechanical properties of the CNT-grafted GFs fabricated at different CVD temperatures were evaluated; they consistently showed low tensile strengths at temperatures above 400 °C. Subsequently, various CNT-grafted GFRPs were manufactured, and their mechanical properties were characterized. Interestingly, the flexural strengths of the composites increased with maintained tensile strength, despite a deterioration of the CNT-grafted GF reinforcements due to the CVD process. This could be attributed to the improved interfacial shear strength (IFSS) of the CNT-grafted GFs at the fiber level, and the enhanced compressive strength and interlaminar shear strength (ILSS) of CNT-grafted GFRPs at the composite level. Considering the properties of GF through CVD processes, particularly in relation to temperature, and factors such as IFSS, ILSS, tensile, compressive and flexural properties of composite materials, grafting CNTs on GF via a CVD system demonstrated its highest optimality at 450 °C.

1. Introduction

Owing to their low weight, good conductivity, and excellent mechanical and physical properties, carbon nanotubes (CNTs) are considered ideal reinforcements for several types of composites [1–4]. Hybridizing CNTs onto the surfaces of advanced carbon and glass fibers (GFs) enhances the fiber-matrix interfacial adhesion, improving the mechanical properties of the fiber composites [5–8]. In contrast, CNTs tend to aggregate on dispersal in polymer resins [9]. To overcome this limitation, CNTs are grown directly on the fiber surface. Electrophoretic deposition and fiber sizing methods involving CNTs are widely used. However, CNTs are not uniformly grown directly along the radial direction of the fibers. This non-uniform growth could potentially lead to a reduced bridging effect between CNTs and the fibers in CNT-fiber reinforced composites. In contrast, the direct growth of CNTs on the fiber surface ensures the radial growth of CNTs onto the fiber, leading to beneficial effect such as improved interfacial shear strength in composite level. CNT-grafted fibers are primarily synthesized by chemical vapor deposition (CVD) [10].

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Carbon fibers (CFs) have been extensively used as substrates for CNT-grafted fibers due to their ability to exhibit synergistic effects when used as composite reinforcements; moreover, their carbon structures can withstand the harsh conditions of CVD. CF-reinforced composites exhibit high interfacial shear strength (IFSS) [11], tensile strength [12], and tensile modulus [13]. To ensure the synthesis of CNT-grafted CF composites with high tensile strength, the CFs undergoing the CVD process should undergo negligible/no deterioration. The CVD temperature has been lowered (to 500 °C) using bimetallic catalysts, to generate the optimal reaction conditions [14]. The increased tensile strength of CNT-grafted CF composites can be attributed to the enhanced IFSS of such systems, along with a delayed initiation of splitting cracks [15]. The excellent electrochemical performances of CNT-grafted CF electrodes, e.g., CNT-CF hybrid supercapacitor electrodes, along with their rate and cycling stability, have been widely reported [16].

Despite being heavier than CFs, GFs have been used as reinforcements in polymer composites, owing to their acceptable strength and stiffness at relatively lower costs [17]. CNTs have been used to modify GFs to fabricate composites with excellent mechanical properties. The resin-dispersal of CNTs generates GF-reinforced composites (GFRPs) with high IFSS values [18]. CNT-deposited GFs synthesized by electrophoretic deposition, with strong CNT-glass fiber chemical bonding, also exhibit high IFSS values [19]. Nevertheless, there are very few reports on the direct grafting of CNTs onto the surface of GFs, possibly due to the thermal degradation of GFs at the temperatures required for the grafting process [20]. As a result, there is a limited amount of research on the mechanical properties of CNT-grafted GFRPs.

In this study, we investigate the mechanical properties of CNT-grafted GF composites synthesized at various temperatures using CVD, along with the effects of the reaction conditions on the mechanical properties of single GF fibers. We fabricated CNT-grafted GFRPs and the morphology of the CNT-grafted GFs was also investigated. The mechanical behavior of the composites was characterized using uniaxial tensile, compressive, and three-point bending tests, as well as fractography. While there have been numerous studies on CNT-grafted-CF and its composites, the investigation of CNT-grafted GF, especially within the context of its composites, is rare. In this regard, this study presents several noteworthy contributions. First, CNT-grafted GF was fabricated, and its composites were manufactured and characterized—an area that has remained largely unexplored. Second, the limited research on CNT-grafted GF composites can be attributed to the degradation of their properties. However, this paper systematically delved into the causes behind the changes in GF's properties, revealing their underlying factors as a second notable contribution. Lastly, despite the property deterioration in CNT-grafted GF due to the thermal effects during the CNT grafting process, we observed an increase in IFSS and ILSS due to the presence of CNTs. This led to the preservation of tensile properties and an elevation in flexural properties at the composite level, highlighting the multifunctional potential of GF composite materials—a third significant contribution.

2. Experimental section

2.1. CNT grafting on glass fibers and fabrics

GFs were isolated from yarns in woven fabric preforms (HD 318-03, plain weave; Entra Korea, 193 g/m2, thickness = 0.2 mm) and soaked in a bimetallic catalyst precursor solution, which was prepared by mixing ethanolic solutions of iron (III) chloride hexahydrate (0.5 M) and nickel (II) nitrate hexahydrate (1 M) for 1 h, followed by drying at 70 °C for 2 h. A CVD-furnace system with hydrogen (25 % v/v) and acetylene (12.5 % v/v) gas-flow in an argon atmosphere was used for the direct growth of CNTs onto the GF surface. The total gas flow rate was 800 sccm, and the flow of each gas was controlled by a mass-flow controller. CVD temperatures of 400 °C, 450 °C, 500 °C, 600 °C, and 700 °C were used to determine the effect of CVD temperature on CNT formation and the mechanical properties of the GFs. Starting from 400 °C, the glass fibers experienced a decrease in the tensile strength. We aimed to understand the changes in the mechanical properties of the glass fibers and also their composites by introducing CNTs in increments of 100 °C. However, CNTs were not grown at 400 °C, leading to investigate the lowest temperature at which CNT growth takes places, which was found to be 450 °C.

CNT-grafted GF fabric preforms were prepared by the same procedure as that employed for GFs described above. The GF fabrics were immersed in a solution of bimetallic catalyst precursor, which was prepared by mixing ethanolic solutions of iron (III) chloride hexahydrate (0.5 M) and nickel (II) nitrate hexahydrate (1 M) for 1 h. Subsequently, they were dried at 70 °C for 2 h. CVD temperatures of 400 °C, 450 °C, 500 °C, 600 °C, and 700 °C were used to facilitate the growth of CNT on the GF fabric. The same CVD-furnace system (gases and their flow) as used for the aforementioned GFs was also utilized in this process.

2.2. Fabrication of CNT-grafted GF composites

CNT-grafted GF composites were manufactured using vacuum-assisted resin transfer molding (VARTM). Four layers of the asreceived woven and CNT-grafted GF fabrics were stacked in a mold, followed by the infiltration of a mixture containing an epoxy resin (Epofix) and hardener (Struers) in a 25:3 ratio into the mold. Different numbers of woven fabric layers (4, 4, 8, and 24) were stacked for tensile, flexural, compressive, and short-beam bending tests. Finally, the preforms were pressed and cured at 100 °C for 2 h.

Single-fiber glass composites for IFSS measurements were prepared using the droplet method. The epoxy compound with the hardener was wetted onto the tip of a small pin, followed by the transfer of epoxy droplets onto the GF surface. These epoxy droplets were cured at room temperature for one day before experimentation.

2.3. Morphological and mechanical characterization

Field-emission scanning electron microscopy (FE-SEM) (SUPRA 55VP) was used to investigate the morphology of the CNT-grafted

GFs. First, the GF samples were coated with a 5-nm-thick platinum layer using a sputter coater (BAL-TEC/SCD 005). Subsequently, the microstructures of the CNT-grafted GFs were observed by high-resolution transmission electron microscopy (HR-TEM) (3000F; JEOL).

To measure the tensile strengths of the as-received GFs and CNT-grafted GFs, a single-fiber tensile test was carried out using a universal tensile machine (UTM, R&B RB307 Chamber) with a 100-g load cell following the ASTM D3822. Over 30 single fibers with a gauge length of 20 mm were tested for each sample; each fiber was pulled at a strain rate of 1 mm/min.

A microdroplet debonding test was used to estimate the IFSS of the single-fiber composites. For this test, a single fiber was pulled out from the epoxy droplet. The mechanism of the microdroplet debonding test is shown in Fig. 1; F_d indicates the force required to pull the filament out, d is the diameter of the fiber, and L is the length of the epoxy droplet.

The tensile and compressive properties of the CNT-grafted GF composites were investigated using a universal tensile testing machine (UTM, Instron 5582). Specimens with dimensions of 60 mm \times 10 mm \times 1 mm were used for the tensile test; a tensile speed of 1 mm/min was used during testing. For the compressive tests, specimens with dimensions of 13 mm \times 13 mm \times 2 mm were prepared and compressed at a nominal rate of 1.3 mm/min.

Three-point bending tests, according to the ASTM D7264 and D2344, were used to investigate the flexural strength and interlaminar shear strength (ILSS), respectively. Samples with dimensions (length \times width \times thickness) of 70 mm \times 10 mm \times 1 mm and 21 mm \times 7 mm \times 3.5 mm, respectively, were used for the flexural strength and ILSS tests. From the load-displacement curve, the flexural strength and ILSS were calculated using the following Equation (1).

$$\sigma_{flexural} = \frac{3PL}{2bh^2}, \sigma_{ILSS} = \frac{3P}{4bh} \tag{1}$$

where σ is the flexural strength (or ILSS), P is the maximum load, L is the length of the span, b is the width of the specimen, and h is the thickness of the specimen.

3. Results and discussion

3.1. Morphology of CNTs grown on GFs

Fig. 2 shows the morphology of CNTs grown on GF surfaces at different CVD temperatures. At CVD temperatures above 450 °C, CNTs were grown and grafted radially and uniformly on the GF surfaces. At 400 °C, some carbon soot was formed on the GFs, indicating no development of CNTs (Fig. 2(a)). Thus, under the catalyst system used in this study, although a CVD temperature of 400 °C was sufficient to catalyze the decomposition of acetylene gas into carbon, it was not sufficient to induce carbon-atom diffusion into the catalyst. Up to a CVD temperature of 700 °C, a uniform growth of CNTs was observed on the GF surface (Fig. 2 (c), (e), (g), and (i)). High-resolution images (Fig. 2(b), (d), (f), (h), and (j)) indicate that long CNTs grew into very fine fibers. Previous publications indicate fiber decomposition at extremely high temperatures; thus, CVD temperatures higher than 700 °C were not used for experimentation [13].

TEM was used to investigate the microstructure (particularly the wall-structure) of the CNTs grafted onto GFs. Fig. 3(a), (c), (e), and (g) indicate CNT growth during tip growth; this can be attributed to weak interactions between the catalyst particles and CF surface. Strong interactions between substrate and catalyst particles (such as those observed between CFs and catalysts) lead to CNT growth by the bottom-growth mode, which causes CNT growth on top of the catalyst, whereas weak interactions result in tip growth [21]. Fig. 3



Fig. 1. Schematic of the microdroplet debonding test of single-fiber composites [15].



Fig. 2. SEM images of CNT-grafted GFs fabricated at CVD temperatures of (a) 400 $^{\circ}$ C, (c) 450 $^{\circ}$ C, (e) 500 $^{\circ}$ C, (g) 600 $^{\circ}$ C, and (i) 700 $^{\circ}$ C at low resolution, and their corresponding high-resolution images ((b), (d), (f), (h), and (j).

(b), (d), (f), and (h) indicate the formation of a multi-walled structure. This confirms that multi-walled CNTs were formed by the CVD system-catalyst combination used here. Notably, CNTs grafted onto the GF surface at 600 °C and 700 °C showed similar structures; in both cases, multi-walled CNTs were formed in the tip growth mode.

3.2. Mechanical properties of CNT-grafted single fibers

Single-fiber tensile tests were used to measure the tensile strengths of the CNT-grafted GFs. As shown in Fig. 4, the tensile strength of the fibers decreased on increasing the CVD temperature; the deterioration of the tensile modulus was not as severe as that of the tensile strength. This deterioration in tensile strength was observed at all the CVD temperatures selected for CNT grafting, possibly due to the elevated temperatures used for CVD [20]. Further experimentation validated this hypothesis. Single GFs were heat-treated at various temperatures (300 °C, 350 °C, and 400 °C) under the experimental conditions of this study, and at a specific CVD temperature without any catalyst and gases. The tensile strengths of these heat-treated GFs were subsequently compared, as shown in Fig. 5. Their tensile modulus values changed negligibly up to 400 °C; however, their tensile strengths significantly deteriorated, with no growth or grafting of CNTs (see Section 3.1). According to heat-treatment experiments, the CVD temperature should be lower than 400 °C for no degradation of the GF tensile strength; however, this is not feasible under the experimental conditions (catalyst and CVD system) of this study.

The IFSS values of the CNT-grafted GFs was measured and compared with that of the as-received GF; as shown in Fig. 6(a), the IFSS of the former (\sim 75 MPa) is almost three times that of the latter (23.7 MPa). A CVD temperature of 400 °C was not considered during IFSS characterization, due to the absence of CNT growth at this temperature. The increase in the IFSS value of the GF on CNT grafting can be explained by the radial growth and grafting of CNTs onto the GF surface, which increases the mechanical interlocking between the epoxy matrix and GF surface. A similar interlaminar interface effect is observed on analyzing the ILSS values of composite specimens (rather than fiber specimens) estimated through a short-beam shear test. As shown in Fig. 6(b), the CNTs grown and grafted



Fig. 3. TEM images of CNTs grafted onto GFs at CVD temperatures of (a) 450 °C, (c) 500 °C, (e) 600 °C, and (g) 700 °C at low resolution, and their corresponding high-resolution images ((b), (d), (f), and (h).

onto the GFs contributed only to a slight increase in the ILSS. This could be due to incomplete mechanical locking caused by CNT entanglement between the laminae, owing to the length of the CNTs formed in this study. According to previous studies on CF systems, long CNTs and their entanglements cause a mechanical locking that positively affects the ILSS value of such systems [22].

3.3. Mechanical properties of CNT-grafted GF composites

The tensile stress-strain curves of the CNT-grafted GF composites are shown in Fig. 7, and the tensile strengths and moduli of the composites are listed in Table 1. The tensile strength and modulus values of the CNT-grafted GF composites decreased on increasing the CNT-grafting temperature. Up to 500 °C, a slight deterioration was observed in the tensile strength of the CNT-grafted GF composites; at higher temperatures, this reduction was significantly more severe. The tensile strength of all the CNT-grafted GFs and their composites deteriorated beyond 400 °C as mentioned in Section 3.2. An increased IFSS induces delayed splitting behavior, thus increasing the tensile strength of fiber composites [23]. Here, the increased IFSS values delayed the tensile strength reduction of the CNT-grafted GF composites up to 500 °C; beyond this temperature, the tensile strength of the CNT-grafted GFs and composites underwent severe deterioration. Cross-sectional images of the fractured specimens are shown in Fig. 8. A fractography of the as-received GFRPs showed fibers pulled out of the epoxy matrix (Fig. 8(a)); the lengths of the pulled-out and fractured fibers were all different. In contrast, a fractography of the CNT-grafted GFs at 700 °C (Fig. 8(b), (c), (d) and (e)) indicated fewer pulled-out fibers, with bulkier fractured surfaces than Fig. 8(a). The CNT-grafted GFs at 700 °C (Fig. 8(e)) were pulled out as the bulky form, where the fibers were stuck with the epoxy matrix. These differences can be attributed to CNT grafting, which binds the epoxy matrix by enhancing the interfacial interactions of the fibers.

The compressive properties of the CNT-grafted GF composites are shown in Fig. 9. The highest compressive strength (162.7 MPa) was observed for the CNT-grafted GF composites fabricated at 450 °C; the compressive strengths of the composites synthesized at



Fig. 4. Tensile strengths of the as-received and CNT-grafted GFs fabricated at different CVD temperatures. The number after 'CVD' represents the heat-treatment temperature.



Fig. 5. Tensile strengths and moduli of the as-received and heat-treated GFs prepared at different CVD temperatures without any catalyst and gases. The number after 'GF' represents the heat-treatment temperature.

higher temperatures were considerably lower, as summarized in Table 1. Notably, all the composites showed higher compressive strengths than that of the as-received GF composite. The radial growth and grafting of CNTs onto GF surfaces possibly hinder the buckling. Notably, the ILSS values of all the CNT-grafted GF composites were greater than that of the as-received GF composite, and the compressive modulus values of the GFs reduced slightly after CNT grafting. It was assumed that the ILSS did not influence the small-load case; therefore, little variation was observed in the compressive modulus. Cross-sectional images of the fractured specimes are shown in Fig. 10. A fractography analysis of the as-received GFRP shows fibers pulled out of the epoxy matrix; the pulled-out fibers and traces of fibers remaining in the epoxy matrix are shown in Fig. 10(a). CNT grafting increased the fiber-resin binding; therefore, Fig. 10 (b), (c), (d) and (e) show fewer pulled-out fibers than Fig. 10(a). For CNT-grafted GFs prepared at higher grafting temperatures, the adhesion of the fibers to the epoxy resin increased due to the longer and more extensive growth of CNTs, resulting in a higher IFSS and making the fibers appear bulkier. The CNT-grafted GFs at 700 °C were stuck with the epoxy matrix, forming a bulky mass, possibly because the CNTs on the fibers acted as an adhesive between the fibers and epoxy resin.

The flexural properties of the CNT-grafted GF composites are shown in Fig. 11. The flexural strength and modulus values increased significantly after CNT grafting. The CNT-grafted GF composite undergoing CVD at 450 °C showed a 74 % increase in flexural strength. Subsequently, the flexural strength of the CNT-grafted GF composites decreased on increasing the CVD temperature, which is



Fig. 6. (a) IFSS of the as-received and CNT-grafted glass fibers at different CVD temperatures. (b) ILSS of the as-received GFRP and CNT-grafted GFRPs fabricated at different CVD temperatures. The number after 'CVD' represents the CVD temperature.



Fig. 7. Stress and strain curves of the as-received GFRP and CNT-grafted GFRPs fabricated at different CVD temperatures obtained by tensile tests. The number following 'CVD' represents the heat-treatment temperature.

Table 1

Mechanical properties of GFRP and CNT-grafted GFRPs prepared at different CVD temperatures. The values in parenthesis represent standard deviations.

	GFRP	CVD 400	CVD 450	CVD 500	CVD 600	CVD 700
Tensile strength (MPa)	239.8 (27.6)	212.2 (19.8)	230.4 (20.9)	228.4 (21.4)	188.8 (29.1)	156.0 (22.5)
Tensile modulus (GPa)	23.3 (2.0)	17.8 (2.1)	21.5 (1.8)	20.1 (1.6)	19.8 (3.1)	18.5 (1.9)
Flexural strength (MPa)	131.5 (23.1)	199.3 (17.5)	229.3 (18.7)	194.0 (18.1)	162.4 (21.6)	192.8 (19.4)
Flexural modulus (GPa)	5.4 (1.8)	7.8 (2.4)	10.3 (2.1)	7.9 (2.2)	7.9 (3.4)	10.2 (2.0)
Compressive strength (MPa)	128.1 (18.9)	147.7 (19.1)	162.7 (16.2)	145.5 (17.1)	135.2 (18.6)	148.8 (16.0)
Compressive modulus (GPa)	18.0 (1.7)	15.9 (2.0)	17.2 (1.9)	17.5 (1.8)	17.5 (2.1)	16.2 (1.6)
IFSS (MPa)	23.7 (1.69)	-	70.1 (2.33)	72.6 (2.21)	71.8 (7.08)	74.9 (5.61)
ILSS (MPa)	33.1 (1.29)	-	40.3 (2.46)	37.4 (1.60)	37.3 (3.58)	41.0 (1.93)



Fig. 8. Fractography of tensile composite specimens composed of the (a) as-received GFs and CNT-grafted GFs at (b) 450 °C, (c) 500 °C, (d) 600 °C and (e) 700 °C.



Fig. 9. Stress and strain curves of the as-received GFRP and CNT-grafted GFRPs fabricated at different CVD temperatures obtained by compressive tests. The number following 'CVD' represents the heat-treatment temperature.



Fig. 10. Fractography of compressive composite specimens composed of the (a) as-received GFRP, and CNT-grafted GFs at (b) 450 °C, (c) 500 °C, (d) 600 °C and (e) 700 °C.

consistent with the trend shown by the compressive strength of the composites. Flexural strength indicates the bending nature of materials, and is determined by its tensile and compressive properties. The asymmetric behavior of the tensile and compressive properties observed in this study indicates a neutral-surface relocation from the middle surface (which is observed for isotropic and symmetric cases). In cases where the tensile properties are higher than the compressive properties, the neutral surface moves to the tension side. Here, the compressive properties were predominant while analyzing the flexural strength; in particular, the tensile properties varied slightly. As listed in Table 1, the tensile strengths of the CNT-grafted GF composites decreased with increasing CVD temperature; it decreased slightly up to 500 °C. In contrast, the compressive properties of all the CNT-grafted GF composites were higher after grafting, regardless of the CVD temperature (including the CVD temperature at which the tensile properties severely deteriorated). Thus, the improved compressive properties, IFSS, and ILSS of all the CNT-grafted GF composites possibly influenced their flexural properties, suppressing the reduced contribution of tensile strength to these properties. The cross-sectional images of the fractured specimens are shown in Fig. 12. The as-received GFRP exhibited fibers pulled out of the epoxy matrix (Fig. 12(a)); fewer fibers were pulled out after CNT grafting, possibly because the CNTs grafted onto the fiber surface acted as an adhesive between the fiber and resin (Fig. 12(b), (c), (d), and (e)).



Fig. 11. Stress and strain curves of the as-received GFRP and CNT-grafted GFRPs fabricated at different CVD temperatures obtained by flexural tests. The number following 'CVD' represents the heat-treatment temperature.

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In summary, the tensile strength of the GFs decreased monotonically with increasing CVD temperature. However, in CNT-grafted GF composites, the CNTs grafted onto the fiber surface caused the deterioration to deviate from a uniform trend with temperature. For instance, at 450 °C, most composite properties reached their peak. Yet, at higher temperatures, the decline in GF properties was considerably more pronounced, to the extent that the presence of CNTs couldn't offset the degradation in GF properties.

4. Conclusions

In this study, CNT-grafted GFs were fabricated via CVD, using bimetallic catalysts (Ni and Fe) under the mixture of argon, hydrogen and acetylene gas conditions at different temperatures, and their composites were manufactured using VARTM with an epoxy resin. Owing to the degradation of GFs at temperatures above 400 °C, the tensile strength of the CNT-grafted GFs was lower than that of the GF at all CVD temperatures. However, CNT-grafted on GF which varied with the CVD temperature, increased its IFSS. The CNT-grafted GFRPs showed good compressive and flexural properties at all CVD temperatures, possibly due to the increased ILSS caused by CNT bridging. The decrease in properties of GF due to the CNT grafting temperature, however, resulted in an increase in IFSS and ILSS due to the grafted CNT, maintaining tensile properties at the composite level and even enhancing flexural properties. Additionally, the introduction of CNTs will enable the development of multifunctional composite materials. The optimum CNT growing temperature that brings about these characteristics is currently 450 °C, and further research is being conducted to lower this temperature using plasma.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time due to legal or ethical reasons.

CRediT authorship contribution statement

Ga-Young Kim: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **Geunsung Lee:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Data curation. **Woong-Ryeol Yu:** Writing – review & editing, Writing – original draft, Validation, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.



Fig. 12. Fractography of flexural composite specimens composed of the (a) as-received GFRP and CNT-grafted GFs at (b) 450 °C, (c) 500 °C, (d) 600 °C and (e) 700 °C.

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