Research Article

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The multiscale enhancement of mechanical properties of 3D MWK composites via poly(oxypropylene) diamines and GO nanoparticles

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Abstract: Interfacial bonding between the fibers and matrix plays a large role in mechanical properties of composites. In this paper, poly(oxypropylene) diamines ($D_{400}$) and graphene oxide (GO) nanoparticles were grafted on the desized 3D multi axial warp knitted (MWK) glass fiber (GF) fabrics. The surface morphology and functional groups of modified glass fibers were characterized by scanning electron microscopy (SEM) and fourier transform infrared spectra (FT-IR). Out-of-plane compression properties and the failure mechanisms of composites at different temperature were tested and analyzed. The results revealed that GO nanoparticles were successfully grafted on fibers under the synergistic effect of $D_{400}$. In addition, $D_{400}$-GO-grafted composite possessed the highest mechanical properties than desized composite and GO-grafted composite. Their strength and modulus were improved by 10.16%, 10.06%, 8.92%, 8.75%, 7.76% and 40.38%, 32.74%, 29.85%, 26.98%, 25.16% compared to those of desized composites at 30°C, 60°C, 90°C, 120°C, 150°C, respectively. The damage to $D_{400}$-GO-grafted composite was yarns fracture accompanied with fibers breakage, matrix cracking, interface debonding. At higher temperature, interlayer slipping with matrix plasticization was the main failure mode.

Keywords: surface modification; GO; out-of-plane compression; damage mechanics; high temperature

1 Introduction

Graphene oxide (GO) as a kind of graphene derivative has attracted significant attention due to its unique two-dimensional nano-laminated structure, large specific surface area, and abundant carboxyl, hydroxyl and carbonyl functional groups distributed on the surface and edges, which enables various covalent chemical modification simple and convenient [1]. Das et al. [2] synthesized multi-layer GO via electrochemical exfoliation method based on organic liquid-assisted solvent. Chen et al. [3] summarized that GO could be further used to fabricate graphene quantum dots which is a new kind of fluorescent carbon materials with unique structure of a single atomic layer. GO has been widely used in medicine field owning to its large plain surface area which provides advantages for drug and protein delivery [4]. Jiao et al. [5] loaded different amounts of drugs on carboxy-methyl cellulose-grafted graphene oxide, and high drug-loading efficiency and encapsulation efficiency were achieved, which could reach 39.33% and 29.50%. Innovative nano-drug (Ori@GE11-GO) was prepared to recognize the specific tumour cells, and improve anticancer efficiency [6]. Graphene-based substrates was applied as adsorbent and photocatalyst to remove oil and organic/inorganic contaminants [7]. Yu et al. [8] constructed thermally evolved and boron bridged GO framework on hollow fibers to separate organic matters, indicating the potential application in wastewater treatment. N-doped FeOOH/RGO hydrogels was researched to enhance removal rate of organic pollutants [9]. Visible-light-induced self-cleaning functional fabrics was fabricated based on graphene oxide/carbon nitride materials [10]. GO was also widely applied in electronic fields of electromagnetic interference (EMI) shielding [11, 12], electrochemi-
The mechanical properties of composites fabricated with modified MWK fabrics have not been discussed. By combining the advantages of GO with ample functional groups and MWK fabrics with good mechanical properties, GO modified fabrics were investigated, and their mechanical properties were discussed for the first time. In this paper, HATU was chosen as condensation agents. To improve interfacial strength between GO and fibers, D_{400} with highly active amino groups at both ends was grafted on fibers of MWK fabrics in advance. Then, GO nanoparticles were grafted on D_{400} grafted fibers in which flexible D_{400} also acted as a bridge to improve the interface bonding between fiber and matrix, and further improve mechanical properties of MWK composites. The out-of-plane compression curves, mechanical properties and failure mechanisms of desized composite, D_{400}-GO-grafted composite and GO-grafted composite were correspondingly compared. The effect of D_{400} and GO nanoparticles on interface bonding of fiber/matrix were analyzed.

2 Experimental

2.1 Materials

The schematic drawings of 0°/90° MWK used in this study were shown in Figure 1. Epoxy resin TDE-90 (4,5-epoxycyclohexane-1,2-dicarboxylic acid diglycidyl ester) was used as matrix. m-phenylenediamine was used as curing agent (Shanghai Macklin Biochemical Co., Ltd., Shanghai, China). Poly(oxypropylene) Diamines (D_{400}, molecular mass Mn= 400) was purchased from Addin industrial corporation. N-[(dimethylamino)-1H-1,2,3-triazolo[4,5,6]-pyridin-1-ylmethylene]-N-methylmethana dinium hexafluoro-phosphate N-oxide (HATU) (Shanghai Macklin Biochemical Co., Ltd., Shanghai, China) was used as coupling agent. The graphene oxide (GO, Thangshan Jianhua Technology Development Co., Ltd., Tangshan, China) was dispersed in deionized water to prepare GO solution (5 mg/mL). The SEM morphology of GO was presented in Figure 2.
The multiscale enhancement of mechanical properties of 3D MWK composites

2.2 Surface modifications of the 3D MWK fabrics

The fabrics were firstly immersed into acetone for 48h to remove the sizing agents, and marked as GF fabrics. Then, they were repeatedly washed with deionized water and dried to obtain the desized GF fabrics. To graft more GO nanoparticles, D₄₀₀ was used as bridge reagent. In this case, desized fabrics were immersed into solution with 70 mL D₄₀₀, 140 mg HATU, and 840 mL deionized water, and they were put into oil bath and reacted for 4 h at 90°C. The obtained D₄₀₀-grafted fabrics were flushed with excess deionized water to remove the unreacted organics. At last, to graft GO nanoparticles on fiber surface, D₄₀₀-grafted fabrics were immersed into 5 mg/mL GO solution with 140 mg HATU, and reacted for 4 h at 90°C. The relevant samples were flushed with deionized water and dried in oven, and they were denoted as D₄₀₀-GO grafted fabrics. Desized fabrics were also separately immersed into GO solution with 140 mg HATU, and also reacted for 4 h at 90°C, and the modified fabrics were named GO-grafted fabrics. The grafting schematic diagram was shown in Figure 3.
Figure 3: The scheme of grafting process for three kinds of composites: desized fabrics, D400-GO-grafted fabrics, GO-grafted fabrics

Figure 4: The photographs of the prepared 3D MWK composite: (a) desized composite; (b) D400-GO-grafted composite; (c) GO-grafted composite

2.3 Fabrication of GF/epoxy composites

3D MWK composites were prepared by the vacuum-assisted resin transfer molding method, in which the matrix were injected into the preforms and then consolidated in oven (90°C for 2 h, 120°C for 2 h). Six layers of fabrics were laid up alternately in 0° and 90° direction. The desized fabrics, D400-GO-grafted fabrics and GO-grafted fabrics were used in each composite type. The composites were then cut into specimens with size of 10mm×10mm×4.0mm. The fiber volume fractions were 53.45%, 53.32% and 52.34% for desized composites, D400-GO-grafted composites and GO-grafted composites, respectively. Figure 4 showed the photographs of prepared composites. The composites fabricated with GO modified fabrics were black due to GO nanoparticles were grafted on fabrics compared with desized composite in Figure 4a.
2.4 Characterization

The chemical structure of the grafted fabrics was examined with a KBr pellet in the range of 500-4000 cm\(^{-1}\) on the Fourier transform infrared spectra (FT-IR, Nicolet 6700, USA).

The morphologies of the fabrics and composites were observed using a scanning electron microscope (SEM, Quanta 250 FEG, Czech Republic).

The out-of-plane compression tests were conducted following the HB 7571-1997 standard [37] with the high temperature mechanical machine (Figure 5). Compression tests were conducted at five different temperatures (30\(^\circ\)C, 60\(^\circ\)C, 90\(^\circ\)C, 120\(^\circ\)C, 150\(^\circ\)C), respectively. The crosshead speed was set to 0.5 mm/min. During the tests, the load and deformation of all samples were automatically recorded by computer. At least three samples were tested at each temperature.

3 Results and discussion

3.1 Morphology of GF with D\(_{400}\) and GO nanoparticles

The SEM morphologies of modified GF fabrics were shown in Figure 6. It could be observed that the surface of desized GF was smooth after acetone cleaning (Figure 6a). In Figure 6b, pleat structure of GO on fibers was obvious indicating that layered graphene was successfully grafted on D\(_{400}\)-grafted fabrics. This kind of structure could promote stress transfer between carbon fiber and matrix. In Figure 6c, GO nanoparticles coated on fabrics in abundance, and reaction groups of fibers were mostly covered which limited their contact area with matrix in the next curing stage.

3.2 Chemical group analysis of fabrics

The surface functional groups of desized fibers, D\(_{400}\)-GO-grafted fibers and GO-grafted fibers were characterized by FT-IR and the images were presented in Figure 7. It could be observed that peaks of O-H stretching vibration at 3434 cm\(^{-1}\), C-H stretching vibration 2925 cm\(^{-1}\) existed in all three kinds of fibers. Comparing with desized GF, peaks at 1417 cm\(^{-1}\) of C=O stretching, 1044 cm\(^{-1}\) of C-O stretching were obvious for D\(_{400}\)-GO-grafted fabrics and GO-grafted fabrics, which demonstrated that GO was successfully grafted on desized fabrics. In terms of GO-grafted fabrics, peak at 1630 cm\(^{-1}\) of C=C stretching vibration appeared, which revealed that most of GO were just coated on the surface of fibers and not adequately reacted with surface organic groups of fibers. While for D\(_{400}\)-GO-grafted fabrics, peak of C-O-C stretching at 1260 cm\(^{-1}\) was visible, which indicated that GO nanoparticles reacted well with fibers since D\(_{400}\) acted as a bridge reagent. Other functional groups were mostly removed after cleaning with acetone. New groups were grafted on the GF fabrics again during GO nanoparticles surface modification process.

3.3 Compression stress-strain curves

Out-of-plane compression stress-strain curves of desized composite, D\(_{400}\)-GO-grafted composite, GO-grafted composite at 30\(^\circ\)C, 60\(^\circ\)C, 90\(^\circ\)C, 120\(^\circ\)C and 150\(^\circ\)C were displayed in Figure 8. As the figure shown, curves increased slowly with similar slopes at initial stage, then increased linearly up to the peak quickly with different slopes, and finally dropped immediately for all three kinds of composites. The reason is that the matrix supported the loading at the initial stage, and the modulus was similar at different temperatures; As the load increased, the matrix cracked, the interface bonding force of fiber/matrix bore the main load; At final stage, fibers broke resulting in samples complete failure. In addition, the performances of MWK composites were temperature sensitive, and the maximum stress decreased with increasing temperature. It was due to different thermal-expansion coefficient (CTE) of fibers, matrix and GO. The CTE of glass fiber, matrix and GO were 5.0 \times 10^{-6}/^\circ\text{C} [38], 31.7 \times 10^{-6}/^\circ\text{C} [39] and \(-67\) \times 10^{-6}/^\circ\text{C} [40], respectively. In this case, stress could be effectively transmitted between fibers and matrix at room temperature. With increasing temperature, due to the large difference of CTE among the matrix, glass fibers and GO, the crystallization rate of the matrix around glass fibers and the surface of glass fibers were different. The crystallization zone with gradient distribution led to the ex-
Figure 6: SEM images (left, low magnification; right, high magnification) of the glass fiber fabrics: (a) desized GF; (b) D$_{4000}$-GO-grafted GF; (c) GO-grafted GF
istence of residual thermal stress, thus the final performance of composites decreased. Then, the fiber/matrix interface bonding got weaker, and stress could not be transmitted effectively among fibers and matrix any more. From 30°C to 90°C, the slopes of curves were higher, and the failure strain was shorter. This is because interface bonding between fiber and matrix was well before 90°C which was also gelation temperature of matrix. While at 120°C and 150°C, the failure strain got longer when the curves reached to the maximum stress since the matrix came to be plastic and softening, and the mechanical properties decreased at the same time.

Slopes of D400-GO-grafted composite curves was the highest and maximum stress was the largest comparing to that of desized composite and GO-grafted composite at each temperature. The reason is that GO nanoparticles were grafted on fibers with chemical bond instead of van der Waals force, and interface bonding force of fiber/matrix was improved. -NH₂ of D400 at one end reacted with -COOH of fiber, another -NH₂ at the other hand acted with -COOH of GO nanoparticles [41]. At the same time, amino could also participate in curing reaction of matrix. Intermolecular bond between fiber and matrix was strengthened. In addition, the structure of D400 is flexible, which could promote the uniform transfer of stress among interface between fibers and matrix. In Figure 8c, the maximum stress of GO-grafted composite was the lowest while its slopes were higher than desized composite at different temperature. Most GO nanoparticles just coated on the surface of fibers through the strength of weak van der Waals force. Therefore, the interfacial bonding between fibers and matrix decreased due to surface of a part of fibers were occupied with GO nanoparticles which limited their contact area with matrix. However, the addition of GO nanoparticles benefited for modulus improvement.
Table 1: Out-of-plane compression strength and modulus of composites

| Temperature (°C) | Desized composite | D<sub>400</sub>-GO-grafted composite | GO-grafted composite |
|-----------------|-------------------|-------------------------------------|---------------------|
|                 | Strength (MPa)    | Modulus (GPa)                       | Strength (MPa)      | Modulus (GPa) |
| 30              | 579.87            | 3.17                                | 638.80              | 4.45          |
| 60              | 559.59            | 2.81                                | 615.90              | 3.73          |
| 90              | 516.09            | 2.68                                | 562.11              | 3.48          |
| 120             | 438.95            | 2.15                                | 477.34              | 2.73          |
| 150             | 340.60            | 1.59                                | 367.03              | 1.99          |

3.4 Compression properties

Out-of-plane compression properties of desized composite, D<sub>400</sub>-GO-grafted composite and GO-grafted composite were shown in Figure 9 and Table 1. D<sub>400</sub>-GO-grafted composite exhibited the highest strength comparing to desized composite and GO-grafted composite (Figure 9a). Comparing to desized composite, the strength was improved by 10.16%, 10.06%, 8.92%, 8.75%, 7.76% from 30 °C to 150 °C, respectively. This could be attributed to effective graft of D<sub>400</sub> and GO nanoparticles on fiber surface. Graft of D<sub>400</sub> on fiber surface improved interfacial bonding between fiber and matrix in which high mechanical properties of GO nanoparticles were fully realized [42]. However, comparing to desized composite, the strength of GO-grafted composite decreased by 7.02%, 9.21%, 10.14%, 12.37%, 0.75% from 30 °C and 150 °C, respectively, which manifested that single GO nanoparticles surface modification could not improve mechanical properties of desized composite. It was because D<sub>400</sub> played a role of bridge which connected the fibers and GO nanoparticles, and further increased interfacial bonding force of GO nanoparticles, fibers, and matrix. The single GO nanoparticles could not effectively graft on fibers and were easy to peel off.

In Figure 9b, the modulus of D<sub>400</sub>-GO-grafted composite presented the highest value when temperature changed from 30 °C to 150 °C comparing to desized composite, they were improved by 40.38%, 32.74%, 29.85%, 26.98%, 25.16%, respectively. This is because high mechanical properties of GO nanoparticles which is with excellent stiffness performance improved the mechanical properties of composites [43]. Meanwhile, the modulus of GO-grafted composite was improved by 16.40%, 9.96%, 6.34%, 3.26%, 1.26% comparing to desized composite from 30 °C to 150 °C. This because many GO nanoparticles with high mechanical properties were introduced into composite. However, the effects were not good as that of D<sub>400</sub>-GO-grafted composite owning to weak interfacial bonding force without D<sub>400</sub> acting as bridge agent.
3.5 Failure mechanism

Figure 10 showed out-of-plane compression fracture morphologies of composites tested at different temperatures (30°C, 90°C and 150°C). During compression, warp knitting yarns were easy to broken which finally led to yarns prone to be extruded under stress for MWK composites. At 30°C, it could be observed that the failure mode was yarns fracture for all three kinds of composites. Surface fabrics were seriously crushed, and cracks appeared on the surface layer. At 90°C, extrusion of yarns could be seen due to the fracture of warp knitting yarns, and interlayer slipping feature also began to take place. This is because of that matrix began to soften and layer-to-layer bond strength.
came to lose their effect. At 150°C, macro-appearance of three kinds of composites maintained integrity, and the main failure mode was interlayer slipping since matrix got soft [44]. The matrix could not transmit stress to fibers effectively since its decreased mechanical properties at high temperature, resulting in fibers could not fully supporting load. The plasticity of matrix led to the final failure of composites. The damage to D_{400}-GO-grafted composite was obvious at each temperature, and then were the desized composite and the GO-grafted composite. This was because synergistic effects of D_{400} which improved the interfacial bonding of fiber/matrix and GO nanoparticles which im-

Figure 11: SEM photographs of out-of-plane compression fracture for three kinds of composites: (a) desized composite; (b) D_{400}-GO-grafted composite; (c) GO-grafted composite
proved mechanical properties, and finally led to stress was transmitted effectively. Thus, surface cracks, yarns extrusion phenomenon was the most serious. The damage to GO-grafted composite was small owing to weak interface bonding between fiber and matrix, and stress transmission was not efficient. At the same time, interlayer slipping phenomenon was serious owing to the weakest interface bonding of fiber/matrix.

SEM morphologies in Figure 11 further demonstrated compression failure mode of desized composite, $D_{400}$-GO-grafted composite and GO-grafted composite (30°C, 90°C and 150°C). For desized composite in Figure 10a, at 30°C, matrix cracking and fibers breakage were obvious resulted from warp knitting yarns breakage during compression. However, fibers and matrix maintained the good adhesion since CTE of matrix and fiber matched well at 30°C. At 90°C, fibers fracture, layers delaminating was obvious caused by softening matrix and weak interface bonding strength. At 150°C, matrix plasticization got more serious which led to weaker fiber/matrix interface bond. For $D_{400}$-GO-grafted composite in Figure 10b, 0° fabric and 90° fabric crushing damage were the main failure mode at 30°C. This is because that graft of $D_{400}$ and GO nanoparticles improved interfacial bonding force of fiber/matrix. The fibers bore the main load, the fracture feature of fibers was severe, and multiple breaks occurred in a single fiber comparing with desized composite. At 90°C, matrix deformation and fiber shear fracture appeared. At 150°C, the softening matrix was extruded. For GO-grafted composite, fibers buckling phenomenon was seen at 30°C. At 90°C, fibers pull-out, interface debonding, fibers fracture led to the failure of composite. At 150°C, some GO nanoparticles remained on the surface of fibers since weak bonding force with matrix without grafting $D_{400}$. In addition, it could be seen that interface debonding occurred on interface of fiber/matrix for desized composite while on interface GO/matrix for $D_{400}$-GO-grafted composite and GO-grafted composite.

4 Conclusions

GO was successfully grafted on 3D MWK fabrics based on $D_{400}$, and $D_{400}$-GO-grafted composite was fabricated. Out-of-plane compression stress-strain curves increased linearly at initial stage with decreased slopes with increasing temperature for desized composite, $D_{400}$-GO-grafted composite and $D_{400}$-grafted composite. The mechanical properties decreased with increasing temperature due to softening matrix and declined interfacial bonding. $D_{400}$ and GO nanoparticles synergistically improved the binding force of 3D MWK fabrics with matrix due to reaction between matrix, $D_{400}$ and GO nanoparticles took place during consolidation stage. The mechanical properties of $D_{400}$-GO-grafted composite were improved especially at room temperature.

The main failure mode for MWK composite was yarns broken with fibers breakage, matrix cracking, interface debonding at low temperature. At higher temperature, the main damage was interlayer slipping owing to matrix got soft. The damage to $D_{400}$-GO-grafted composite was most serious since its high interfacial bonding force between $D_{400}$-GO-grafted fibers and matrix, and fibers could effectively bear load. The destruction to GO-grafted composite was less serious comparing with $D_{400}$-GO-grafted composite since the bonding way was van der Waals force, and stress could not be effectively transmitted. Interface debonding between fibers and matrix mainly led to failure of composite. Interface debonding occurred on the interface between fiber and matrix for desized composite while on GO and matrix for $D_{400}$-GO-grafted composite and GO-grafted composite.

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