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Effect of fiber surface modification on water absorption and hydrothermal aging behaviors of GF/pCBT composites

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ABSTRACT

Water absorption and aging behaviors of fiber reinforced polymerized poly (cyclic butylene terephthalate) (GF/pCBT) composites are investigated. We coated nano-silica on glass fiber surface by physical vapor deposition (PVD) method. Subsequently, we immersed pCBT composites reinforced with nano-treated/untreated fibers in 25 °C and 60 °C distilled water until their saturated moisture. We also exposed some specimens in various hydrothermal aging environments. We tested the mechanical performance of these test specimens and found that the mechanical performance of both pCBT cast and GF/pCBT composites reduces obviously after water absorption and hydrothermal aging. However, nano-silica modified fiber reinforced composites have higher remaining strength than GF/pCBT. Scanning electron microscope (SEM) is used to study the microscopic phase and nanoparticle modified mechanism, and better interface characteristic between fibers and matrix is observed.

1. Introduction

Compared with their thermoset counterparts, thermoplastic resins have significant advantages including high toughness and recycling options. However, their high melt viscosity (50–2000 Pa s) could hamper fiber impregnation in the fabrication of thermoplastic composites. Recently, cyclic butylene terephthalate resin (CBT) has drawn tremendous interests mainly due to its low melt viscosity (17 mPa s) which leads to the super manufacture ability and recycling potential when used as thermoplastic matrix [1,2]. Fiber reinforced CBT composites are good candidates for many applications such as automobile industries, marine engineering, and aerospace field etc. However, fiber reinforced CBT composites must experience serious service condition to achieve their wider application scope, because it has been verified that the performance of polymer composites would deteriorate under prolonged immersion in water or exposure in hydrothermal environment [3–5].

Generally, two aging mechanisms can explain the degradation of polymeric composites in hydrothermal conditions: one is the physical aging induced reversible changes, such as plasticization and swelling. The other is the chemical aging induced irreversible changes, such as hydrolysis [6–8]. These two degradation mechanisms are affected by water (moisture) content and temperature, and the aging process is usually accelerated by high ambient temperature and moisture content experimentally [9–13]. The accelerated hydrothermal environment could reduce the mechanical performance of fiber, matrix and especially the interface between fiber and matrix. This is because the mismatch expansion in interface area could generate interface stress between fiber and matrix in moisture and temperature environment [14–16]. In aging process, water absorption is a main factor. Scientist and technologist have adopted various methods to change water absorption behaviors of composite materials by modification of matrix, fiber or both components [17–19]. Fiber surface coating technology is an important modification method. Many fiber surface coating treatments have been used to modify the performance of composites, and it has been proved that these methods could enhance the composite interface strength between fiber and matrix [20–24]. In terms of CBT resin, there are many literatures about its matrix modification in order to enhance its mechanical and thermal

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performance [25–27]. The objective of this paper is to investigate the effect of fiber surface treatment on GF/pCBT composites by measuring its water absorption and hydrothermal aging behavior. In the experiment, immersion, hydrothermal aging and mechanical test are performed. The modified fiber surface and fiber/matrix interaction are investigated and some conclusions are drawn.

2. Experimental details

2.1. Materials

The polymer is CBT-100, supplied by Cyclic Corporation. CBT-100 resin has a big-ring paucity of polyester structure with the molecular weight $M_w = (220)_n$ (n = 2–7) g/mol. It could be transformed into linear high molecular weight engineering thermoplastic poly (butylene terephthalate) (pCBT) plastic at 190 °C via entropically-driven ring-opening polymerization mechanism [28,29]. The catalyst selected in the ring-opening polymerization reaction of CBT resin is butyltin chloride dihydroxide (PC-4101). The unidirectional glass fiber cloth is used with surface density of 800 g/m^2. Hydrophobic nano-silica (DNS-3) is used with the diameter from 5 nm to 15 nm. All the materials are dried for 10 h in a vacuum oven as pretreatment prior to processing.

2.2. Manufacturing process

Physical vapor deposition (PVD) method is adopted to deposit catalyst and/or nano-silica on the glass fiber surface. The specific preparation processing is described as follow: firstly, catalyst powder (0.6 wt.% of the resin) is added in 200 ml isopropanol aqua, and we stir the mixture by magnetic stirrer at 70 °C and rotation rate of 1000 r/min for 10 min until completely dissolved (Kind I). Subsequently, we take half specimen of Kind I and fill 2 wt.% (percentage of resin) nano-silica into the mixture. To ensure the homogeneous dispersion of nano-silica, the mixture is stirred by magnetic stirrer at rotation rate of 1000 r/min for 2 h, and then the mixture is dispersed by an ultrasonic agitator for 1 h (Kind II). Then, we immerse 12-ply unidirectional glass fiber [0_{12}] into these two kinds of mixed solution in a metal square plate holder, respectively. The system is kept in an incubator for 3 h at room temperature to ensure the fiber fully wet. Finally, the glass fiber is dried at 140 °C to evaporate isopropanol aqua and coat catalyst and/or nano-silica on fiber surface. Glass fiber coated by catalyst and catalyst/nano-silica are observed by SEM in Fig. 1. From the figure, we can see that the catalyst is effectively cohered on fiber surface (Fig. 1a), and surface roughness of fiber enhances significantly after nano-silica treatment by PVD approach (Fig. 1b).

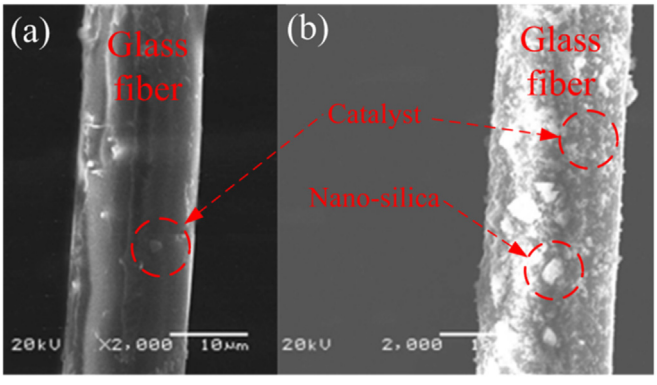

Fig. 1. SEM microphotographs of glass fiber surface treated by catalyst (a), and catalyst and nano-silica (b).

Three groups of test specimens are manufactured: pCBT casts and its nano-treated/untreated glass fiber reinforced composites. In the fabrication process of pCBT casts, 0.6 wt.% catalyst is added in melted CBT resin. The mixture is poured into a metal mold and cures for 1 h in a vacuum oven at 220 °C. Fiber reinforced pCBT composite laminates is manufactured via vacuum-assisted molding pressing (VAMP) process, as shown in Fig. 2. During the process, the curing temperature is a stage-like shape: keep 1 h at 230 °C, then decrease temperature to 190 °C and keep for another hour. Photographs of the obtained pCBT casts and its nano-treated/untreated glass fiber reinforced composites are shown in Fig. 3. The dimension of prepared laminates is 280 × 180 × 2.4 mm^3 with 68 vol.% of fiber content tested by ablation method. We observed the polished cross-section of GF/pCBT composites using SEM, as shown in Fig. 4. From the figure, we can find that no fiber dislocation or voids between fibers are observed at micro level, which indicates the feasibility of VAMP process used in this research.

2.3. Immersion/hydrothermal environment experiment

In order to study the water absorption behavior, the immersion test is performed in 25 °C and 60 °C distilled water, respectively. 60 °C is adopted to accelerate the immersion process, and 25 °C is used as reference. Dimension of pCBT is 72 × 12 × 2.8 mm^3, and size of composite laminates is 60 × 60 × 2.8 mm^3. In the experiment, firstly specimens are dried in oven at 60 °C for 12 h. Then, these specimens are immersed into distilled water for 3 months until saturation during which the weight of these immersed specimens is recorded periodically. To investigate the influence of hydrothermal aging on the macroscopic mechanical behavior of these specimens, another group of specimens is exposed in chambers to suffer the hydrothermal condition. These cases including humidity and temperature are listed in Table 1. Specimens are exposed in the chamber for up to 4 weeks. One group (5 specimens) is taken out from the chamber and tested every week to study the performance degradation. Compression and three-point-bending tests are performed to measure the mechanical property of these specimens taken out from the chamber. The dimension of these specimens is defined according to ASTM standard (D7264/D7264M-07, D695-02). pCBT cast in compression and bending test has the dimension of 25 × 10 × 10 mm^3 and 60 × 10 × 6 mm^3, while composites in bending test have dimension of 60 × 12.5 × 2.8 mm^3. All tests are accomplished using Instron-4505 servo-electric testing machine, and the crosshead speed is 2 mm/min. Furthermore, to evaluate the quality of interfacial adhesion between the fiber and matrix, scanning electron microscope (Hitachi S-4300, Tokyo, Japan) is adopted to observe the fracture surfaces with special attention on the interface region.

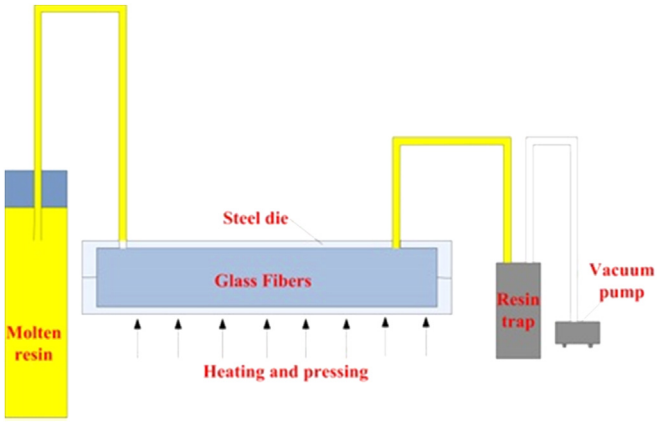

Fig. 2. Schematic diagram of VAMP.