Composites Part B 153 (2018) 306–314

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Effects of hydrothermal aging on moisture absorption and property prediction of short carbon fiber reinforced polyamide 6 composites

Lin Sang, Chuo Wang, Yingying Wang, Wenbin Hou*

School of Automotive Engineering, State Key Laboratory of Structural Analysis for Industrial Equipment, Dalian University of Technology, Dalian 116024, China

ARTICLE INFO

Keywords:
Carbon fiber
Thermoplastic resin
Environmental degradation
Mechanical properties

ABSTRACT

The present work focuses on the effects of hydrothermal aging on moisture absorption and mechanical durability of short carbon fiber reinforced polyamide 6 (CF/PA6) composites by immersion in water at 20 °C, 40 °C and 60 °C. Tensile strength, Young's modulus and impact strength of PA6 and CF/PA6 specimens are monitored, and a predictive model based on Arrhenius methodology has been utilized to estimate the retention of tensile strength. By gravimetric experiments, moisture uptake and diffusion coefficients of PA6 and CF/PA6 composites are analyzed by Fickian model, with good exponentially decaying fit with tensile strength.

1. Introduction

Currently, short fiber reinforced polyamide composites have been widely used in automotive industry due to their high strength-to-weight ratio and ease processing of complex geometries at high production rate [1,2]. Nevertheless, polyamide resin is highly sensitive to moisture condition, and moisture attack from harsh environment could induce the physical and chemical changes of exposed short fiber reinforced polyamide composites [3–5]. During the operating life, these changes would result in both moisture uptake and deterioration of the mechanical performance, thus, assessing the evolution of mechanical properties and prediction of environmental durability of such materials under hydrothermal aging condition is a very important issue.

Polyamide is a semi-crystalline thermoplastic with polar amide groups. When exposed to the hydrothermal conditioning, the absorbed water molecule replaces the existing inter-chain amide-amide bonding with amide-water bonding. This would induce the plasticization of the polyamides, which highly impacts the mechanical properties such as a reduction of strength and modulus while increasing the ductility [6–8]. There have been several studies regarding the hygrothermal behavior of glass fiber reinforced polyamides (GF/PA) composites on the water diffusion and mechanical properties [9–12]. Thomason et al. [9] investigated the mechanical performance and weight in glass-fiber reinforced polyamide 66 (GFPA66) at temperature of 70 °C. Haddar et al. [10] studied the sorption mechanism of PA6/GF50 at high temperatures (90 °C) in two types of fluids (water and salt solution) placed in different fluids and temperatures. Bergeret and Vlasveld et al. [11] showed that the diffusivity of water within PA was easier and faster at high temperature of 70 °C, roughly 17 times smaller at 20 °C, denoting temperature acted like an activator of water diffusion. Despite the recent progress towards the hygrothermal aging of GF/PA composites, prediction of long-term mechanical properties of carbon fiber reinforced polyamide (CF/PA6) composites have not been investigated, and the correlation between absorbed water with mechanical performance have not been yet proposed.

Recently, the use of carbon fiber reinforced composites become popular since they provide a good balance between lightweight and superior mechanical properties [13–15]. The focus of this work is to study the mechanical response of short fiber reinforced PA6 composites subjected to hydrothermal aging by immersion into water at 20, 40 and 60 °C. The evolution of mechanical properties of PA6 resin and CF/PA6 composites are evaluated, and SEM observations are further performed to identify the damage mechanisms of CF/PA6 composites. On the basis of monitoring tensile strength of specimens, analytical model is adopted and predicted the retention of tensile strength of PA6 and CF/PA6 composites, respectively. Based on moisture absorption curves, correlations between moisture uptake and tensile strength retention are further developed for PA6 and CF/PA6 composites.

2. Experimental study

2.1. Materials

The polyamide 6 resins, a standard viscosity grade product, were produced by Yueyang Petrochemic Co., Ltd., China. The characteristics of PA6 are as follows: the density is 1.13 g/cm^3, the tensile strength is

* Corresponding author.
E-mail address: houwb@dlut.edu.cn (W. Hou).

https://doi.org/10.1016/j.compositesb.2018.08.138
Received 29 April 2018; Received in revised form 23 August 2018; Accepted 30 August 2018
Available online 31 August 2018
1359-8368/

86 MPa, the tensile modulus is 3.4 GPa, and the melting temperature is 220 °C. The carbon fiber (CF) tow (commercial grade: HTS40-6 k) is provided by Toho Tenax Co., Ltd. The characteristics of the CF are as follows: the diameter 7 μm, the density is 1.77 g/cm^3, the tensile strength is 4.4 GPa, and the tensile modulus is 230 GPa. 3-Aminopropyltriethoxy silane coupling agent (KH550, $NH_2CH_2CH_2CH_2Si(OC_2H_5)_3$, 97% purity), acetone (99.5% purity), alcohol (95% purity) were purchased from Tianjin FuYu chemical co., LTD.

2.2. Samples preparation

In our previous work [16], the silane-treated CF reinforced PA6 composites increased the interface adhesion between matrix and fiber and improved mechanical properties in contrast with bare CF reinforced PA6 composites. Therefore, carbon fibers were preliminary treated by silane agent in this work, and the process of silane treatment was according to our previous work.

Polyamide 6 composites with 20 wt% of carbon fibers (CF/PA6) were prepared by melt compounding using a co-rotating twin screw extruder (*SHJ*20-*X*40, $L/D = 40$, $D = 40$ mm, Nanjing Giant Machinery Co., Ltd. China). PA6 was fed from the hopper, and the continuous carbon fibers were automatically fed through the fiber feeding port by rotation of the screw. The CF was sheared to be short and dispersed in PA6 matrix during extruding, with ramping temperature profile from feeding zone to die as 235, 240, 245, 240, and 235 °C, respectively. Then the extruded material was graduated into pellets to a length of 4–5 mm. These pellets were oven dried for 24 h at 80 °C to remove the residual water originating from the cooling step in the compounding process prior to injection molding. Neat PA6 without reinforcement was set as control.

Standard dumbbell-shaped tensile specimens (GB/T 1040:2–2006) and impact specimens (GB/T 1843–2008) were prepared on an injection molding machine (XTK 1200, Xiatian General Machinery, Ningbo, China). Geometry of PA6 and CF/PA6 specimens for mechanical tests were illustrated in Fig. 1. Molding conditions were as follows: the temperature was from feeding zone to injection port as 245, 243 and 241 °C, and the pressure during heating was set from feeding zone to injection port as 35, 45 and 65 bar, respectively. The specimens before hydrothermal aging were stored under ambient temperature (20 °C). For aged specimens, testing specimens were re-dried in oven at 50 °C for at least 100 h to ensure that they reached a constant weight condition. The specimens for the water uptake test were cut from the plates which were fabricated by hot press method. The pellets were together placed onto the hot press machine and pressed with 10 MPa pressure at 250 °C for 10 min.

2.3. Experimental and characterization

2.3.1. Moisture sorption measurements: water absorption

Specimens with a dimension of 50 × 50 × 2 mm were cut for the water uptake test in accordance with ASTM D 5229. The test specimens of PA6 and CF/PA6 were immersed into distilled water in a sterilized water bath kettle. The exposure temperature of the specimens was controlled at three levels, 20 °C (room temperature), 40 °C and 60 °C (summer weather). The weight of specimens were measured at predetermined time intervals through gravimetric method. At the time intervals, specimens were removed from the distilled water, their surface water was wiped off using filter water and immediately weighed the surface-dried specimens with an electronic analytical balance with a relative precision of 10^{-3}. The mass or the moisture content of a sample at a given duration, M_t, was determined as follow by Eq. (1):

$$M_t(\%) = \frac{W_t - W_o}{W_o} \cdot 100 \quad (1)$$

where W_o and W_t denote the dry weight (initial dry sample) and the wet weight sample at a given time t, respectively. The moisture absorption tests at room temperature (20 °C) was stopped after 1896 h, whereas that at 40 °C after 888 h (37 days) and 60 °C after 648 h (27 days).

2.3.2. Mechanical test

The tensile test of all specimens was carried out using a universal tester (Sans UTM5105X, Shenzhen) according to ISO 527–2 procedure at a cross-head speed of 5 mm/min at room temperature. The impact strength was measured by XQZ-II impact tester (JJ-Test, Chengde, Hebei, China) equipped with a pendulum of 2.75 J according to ASTM D 256 standard. The notch was 2.5 mm and the angular radium was 0.25 mm. All the tests were performed at a constant temperature of 25 °C, and each data point was the average from five specimens. Before the mechanical testing, the specimens were post-conditioned for a period of 24 h in a circulating-air oven at a temperature of 50 °C based on ASTM D 638 to make sure all specimens have the same moisture content at the time of the mechanical testing. Then the post-conditioned specimens were removed from the oven and were cooled to the room temperature for a period of at least 5 h prior to the mechanical testing.

2.3.3. Dynamic mechanical analysis (DMA)

DMA was conducted on a Q800 analyzer (TA Instruments). DMA samples were cut from the notched izod impact samples. The dimension of the test specimens was 40 × 10 × 4 mm^3 (length × width × thickness). The frequencies used were 1 Hz at a heating rate of 3 °C/min. The temperature was programmed from 25 °C to 200 °C and the amplitude was set 10 μm in the bending mold.

2.3.4. Microstructure observation

The fracture surface of tensile tested specimens were observed using a SEM (QUANTA 450, FEI). The fractured surface of the specimens were cut and attached to carbon adhesive discs, placed on aluminum mounts and sputter-coated with gold eliminating the charge effect.

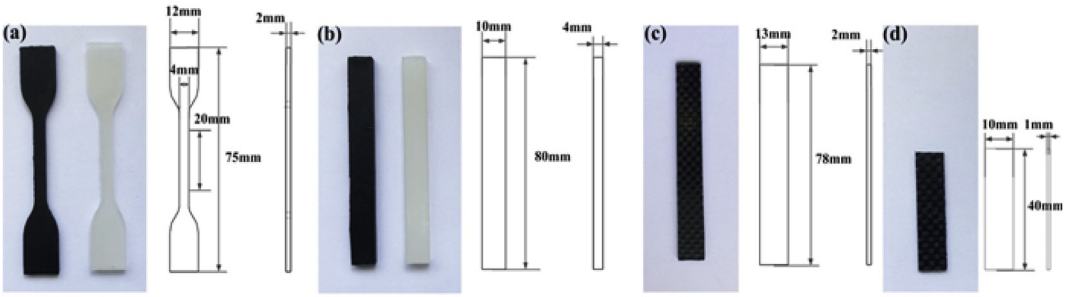

Fig. 1. Specimen geometry used for mechanical tests.