Composites Part B 99 (2016) 398–406

Contents lists available at ScienceDirect

Composites Part B

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

Microwave radiation effects on carbon fibres interfacial performance

Ben Wang, Yugang Duan*, Jingjing Zhang, Xinming Zhao

State Key Lab for Manufacturing Systems Engineering, Xi'an Jiaotong University, Xi'an, Shaanxi Province, 710049, China

ARTICLE INFO

Article history:
Received 2 June 2015
Received in revised form
6 September 2015
Accepted 3 June 2016
Available online 8 June 2016

Keywords:
Carbon fibre
Interface/interphase
Surface analysis
Surface treatments

ABSTRACT

In this paper, microwave radiation, a fast and cost-effective industrial surface pretreatment method without chemicals, was employed to pretreat T300 carbon fibres and improve the carbon fibres/matrix interfacial properties. The microwave pretreatment mechanism on carbon fibres was studied in an experiment divided microwave treated carbon fibres into a microwave radiation section and a pure current section. The polarization current in the carbon fibre induced by microwave radiation helped microwave effects interact on the morphology, compositions, and structure of carbon fibres. Carbon fibres pretreated with short time microwave radiation had an increased thermodynamic work of adhesion and the interfacial shear strength, indicating microwave radiation pretreatment of carbon fibre promises to be an effective method of fiber treatment.

1. Introduction

Carbon fibres reinforced polymer composites are widely used in aerospace, energy, marine and automobile industries due to their outstanding properties such as high specific strength and stiffness, and lower density [1,2]. Composite properties are not only determined by the fibre and the resin, but also influenced by the interface between the two constituents. An effective adhesion of the carbon fibres/matrix interface has been considered as high-performance composite preconditional factor to obtain better stress transfer and crack resistance [3,4]. The carbon fibres/matrix interface formation in the composite preparation process could be considered in two stages. In the first impregnating stage, the resin spreads and impregnates on the fibre surface more easily with favorable wettability and high surface energy; In the second interface formation stage, the interface would be formed by the interaction between the resin and fibre surface, which is strongly influenced by the amount of functional groups and active surface area [5,6]. Commercially available carbon fibres are normally coated by a surface sizing layer to connect with the matrix and protect fibres from the damage. However, the tailored sizing is usually limited to match a singular kind of polymer besides the complicated sizing procedure [7,8]. To overcome the disadvantage of sizing, some carbon fibres surface pretreatments/modifications have been applied to provide an alternative interphase to improve the carbon fibres/matrix interface cohesive force in the industrial processes while remaining cost-effective. They adopted gas [9], liquid [10], plasma [11], ultrasonic [12] and electrochemical approaches [3] as the fibre modification. But all these pretreatment methods are time-consuming, and some chemicals are needed, which increases the cost of the production. For example, aqueous ammonia was employed to pretreat carbon fibres to increase the surface roughness, but the pretreatment time of 48 h was needed for the optimized results [13]. Also, plasma with O_2 required plasma generator and high O_2 flux to increase the surface oxygen content of carbon fibres in a short time [14].

Microwave, a high-frequency electromagnet wave ranging from 100 MHz to 300 GHz, has been used in some applications according to its microwave effects including thermal and non-thermal effects. For thermal effects, dielectric materials can be heated fast due to dielectric loss, which could activate molecule to relax or break chemical bonds, and form new chemical bonds. Non-thermal microwave effects have been postulated to result from a direct stabilizing interaction of the electric field with specific (polar) molecules in the reaction medium that is not related to a macroscopic temperature effect [15]. For example, the focusing of electric fields at particle interfaces caused by microwaves can form plasma and generate polarity groups [16,17]. Other than microwave effects, conductive carbon fibres could be regarded as an electric dipole according to the skin effect (Electromagnetic radiation at high frequencies penetrates the near surface region of an electrical conductor), because the diameter of monofilament is only 7 μm far smaller than microwave wavelength (122 mm). Consequently, it

* Corresponding author.
E-mail address: ygduan@mail.xjtu.edu.cn (Y. Duan).

http://dx.doi.org/10.1016/j.compositesb.2016.06.032
1359-8368/

could activate polarization current on carbon fibres surface during the microwave radiation [18]. Since these characters of carbon fibres, polarization current effect and microwave effects induced by microwave pretreatment are supposed to perform on carbon fibres simultaneously. However, the mechanism of these effects for the carbon fibres pretreatment on the fibre surface performance is not so clear. Therefore, the microwave radiation was employed in present work to pretreat T300 carbon fibres within a short time, and microwave effects were analyzed by dividing microwave treated carbon fibres into a microwave radiation section and a pure current section. The carbon fibres surface after being microwave treated was investigated through morphology, compositions and surface structure. The interface performance was characterized through the carbon fibres wettability against solvents and resin and the interfacial shear strength (IFSS).

2. Material and methods

2.1. Materials

Polyacrylonitrile-based T300 carbon fibres with sizing agent were produced by Toho Tenax in Japan, which was coded as CFs. A mild desizing procedure extracted the CFs by acetone for 48 h at 70 °C was adopted to avoid deteriorating the morphology and properties of original fibres [13], and the obtain fibre was coded as CF. The mixture of diglycidyl ether of bisphenol A epoxy (DER331, Dow, USA) and amine-based curing agent (Isophorone, 2855-13-2, Evonik Degussa, German) with a 3:1 wt ratio was used as the resin matrix.

2.2. Surface pretreatment by microwave radiation

A tow of carbon fibres including 3000 filaments was irradiated using a microwave generator (BMR-01, Kairui, China) with a emitting frequency of 2.45 GHz at 300 W. Due to the anisotropic dielectric behavior of carbon fibres, the fibre direction was perpendicular to the electric field direction for the maximum microwave absorbance [19]. The height between the generator and carbon fibres was 180 mm. To prevent carbon fibres from being ablated, the radiation time was arranged in cycles: 90 s for radiation followed by 60 s without radiation. In addition, the specimens treated by different radiation time were coded as CF-T (T stands for radiation time).

2.3. Methods

2.3.1. Polarization current test

In order to evaluate the polarization current effect on carbon fibres during the microwave radiation, the polarization current was transmitted from the irradiated monofilament, and recorded as shown in Fig. 1. Both ends of the monofilament (200 mm) were pasted on a glass plate to maintain a straight line, and then connected with copper wires (1.5 mm^2) by conductive adhesive (4×10^{-4} Ω cm, Jiuqi, YC-01, China) to form a conductive path. A 1MΩ resistor was used to magnify the voltage signal obtained by oscilloscope (maximum Sampling rate 4×10^9 Sa/s, RIGOL, DS4012, China).

2.3.2. Atomic force microscopy

The carbon fibres morphologies were observed by Atomic Force Microscopy (AFM, NanoScope MultiMode III, USA) with a scanning speed of 1 μm/s and a scanning scope of 3 μm × 3 μm. At least 3 samples was provided for measuring the roughness of carbon fibres.

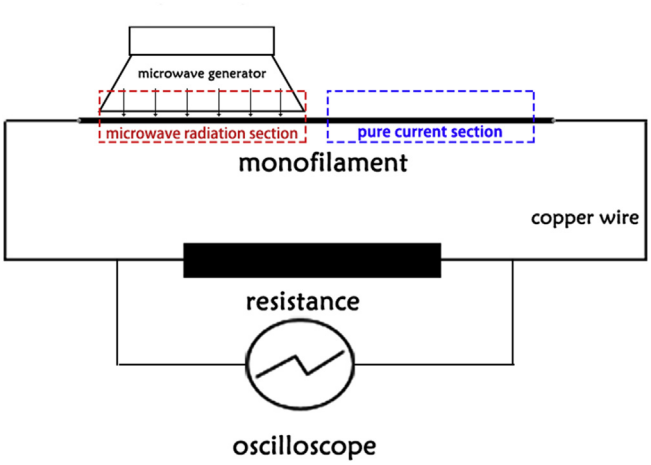

Fig. 1. Polarization current test illustration during the microwave radiation.

2.3.3. X-ray photoelectron spectroscopy

The carbon fibres surface chemical composition was determined by X-ray photoelectron spectroscopy (XPS, Kratos AXIS ultra DLD, UK) with a monochromate Al Kα source (hν = 1486.6 eV) at a power of 150 W (15 kV × 10 mA). To compensate for the surface charging effect, all binding energies were referenced to Cls neutral carbon peak at 284.6 eV.

2.3.4. Raman spectroscopy

The carbon fibres microscopic structure was analyzed by Raman Spectroscopy (Lab-RAM Aramis, JOBIN YVON HORIBA, France) using a He–Ne 20 mW laser with an excitation source of 514.5 nm.

2.3.5. Single fibre tensile testing

The single fibre tensile testing was according to ASTM D3379-75. The specimen ends were attached to a cardboard tab using a quick-drying adhesive (Instant Krazy Glue, KG82048SN, USA) while under slight tension to ensure a consistent gauge length. The tab ends were gripped in the jaws of the testing machine, an INSTRON 1195 Tester with a 100 g load cell. The tab was then cut so that only the fibre was loaded during the test. Specimens with approximately 20 duplicate samples per set were tested under quasi-static tensile loading at a deformation rate of 0.1 mm/min.

2.3.6. Wettability

The carbon fibres surface wettability was evaluated by contact angle and surface energy using a Dynamic Contact Angle Meter (DCAT 21, Germany). Deionized water (γ = 72.8 mN/m, γ^d = 21.8 mN/m, γ^p = 51.0 mN/m) and ethylene glycol (γ = 48.3 mN/m, γ^d = 29.3 mN/m, γ^p = 19.0 mN/m) were chosen as the testing liquid. The dispersive and polar components of carbon fibres are described in Eq (1):

$$\gamma_1(1+\cos\theta) = 2\sqrt{\gamma_1^p \gamma_f^p}$$
$$\gamma_f = \gamma_f^d + \gamma_f^p \quad (1)$$

where θ is the contact angle between carbon fibres and the testing liquid; γ is the surface tension; γ^d is the dispersive component; γ^p is the polar component; subscript l stands for the testing liquid, and subscript f stands for fibres.

The wettability of carbon fibres/epoxy was directly measured by the contact angle, and the work of adhesion (W_A) was calculated by the revised Young-Dupre [20] Eq (2).