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Effects of hydrothermal aging on carbon fibre/epoxy composites with different interfacial bonding strength

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HIGHLIGHTS

- Surface treatment enhanced the hydrothermal durability of fibre/epoxy interface.
- Moisture absorption and desorption tests were carried out for CFRP plates.
- Dynamic mechanical thermal analysis provides insight for degradation of interface.
- Damping at interface (tan δ_{in}) was linked with the interlaminar shear strength (ILSS).

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ABSTRACT

This paper presents an investigation on the effects of hydrothermal aging on carbon fibre reinforced polymer (CFRP) composites with different interfacial bonding strength. The combination of electrochemical oxidation and sizing treatments effectively enhanced the long-term interlaminar shear strength (ILSS) retention from 0.24–0.38 to 0.74–0.86 for CFRP in hydrothermal environment, which basically met the specified environmental reduction factor C_E (0.85) given in ACI 440.2R-08. The improved durability of fibre/epoxy interface was also evidenced by the moisture absorption and desorption tests and scanning electron microscopy (SEM). In addition, the dynamic mechanical thermal analysis (DMTA) was directly used to characterize the degradation of interface adhesion for CFRP, and the damping at fibre/epoxy interface (tan δ_{in}) was evaluated and finally linked with the ILSS results.

1. Introduction

Recently, carbon fibre reinforced polymer (CFRP) composites are increasingly being used to strengthen and repair existing concrete structures in civil engineering due to their high tensile strength and modulus, light weight, chemical corrosion resistance and easy construction [1–4]. Considering the application of CFRP in complex and variable service environments including humidity, temperature variations, water, and other aqueous solutions [2,5,6], and the most important environmental factors are moisture and temperature [7], the long-term properties of CFRP in hydrothermal condition are of primary importance. However, the durability of CFRP mainly depends on the durability of carbon fibre/resin interface since the interface is generally the weakest area in composites, which is more easily attacked and degraded by the absorbed water than carbon fibre and resin matrix with the diffusion of moisture [8–10]. Therefore, to improve the hydrothermal durability of carbon fibre/resin is the key to promote the application of CFRP composites in civil engineering.

Surface treatment of fibre is a widely used method to enhance the interfacial adhesion for CFRP composites [11]. Through surface treatments, the increased surface roughness and reactive functional groups on the fibre surface can effectively improve the mechanical interlocking and chemical interaction between carbon fibre and resin, respectively [12,13]. The common surface treatments include: electrochemical oxidation, plasmas etching, gas phase treatment, sizing treatment, carbon nanotubes grafting and graphene oxide modification and so on [11,12,14–17]. Among these methods, the combined technology of electrochemical oxidation and sizing treatment is the most preferable industrial option because it allows continuous process and is easy to control during the manufacturing of carbon fibre [8,13,18].

The interfacial shear strength (IFSS) and interlaminar shear strength (ILSS) were generally used to evaluate the interfacial

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adhesion behaviour between carbon fibre and resin. The IFSS was confirmed through testing the bonding strength between single fibre and resin, and the common test methods include microbond pull-out test [8,19], single-fibre fragmentation test [20] and so on. The ILSS was confirmed through testing the bonding strength between fibres layer and resin layer inside FRP which contain large amount of fibres, and the common test methods include short-beam shear test [21], in-plane shear test [22,23] and transverse flexural test [24,25] and so on. Therefore, the research specimens for IFSS and ILSS are totally different in scale. Previous researches have shown that the combination of electrochemical oxidation and sizing treatment can clearly increase the IFSS or the ILSS of carbon fibre/epoxy composites by 14–75% and 15–41%, respectively [8,13,26–28]. Only limited work involved the effect of surface treatments on the hydrothermal aging behaviour of carbon fibre/resin interface [8,15,20]. Wang et al. reported that the combination of electrochemical oxidation and sizing treatment increased the IFSS retention from 59% to 70% for the microbond pull-out specimens after exposure to 95% relative humidity (RH) at 40 °C for 24 days [8]. Pérez-Pacheco et al. also suggested that the silane coupling agent sizing treatment increased the finial IFSS retention (from 65% to 71%) for the single-fibre fragmentation test specimens in 95% RH at room temperature, while slightly affected the finial moisture absorption content [20]. However, above studies only focused on the micromechanical behaviour of carbon fibres, while the effect of surface treatments on the long-term behaviour of macroscopic CFRP composites in hydrothermal condition is not yet comprehensively understood.

Dynamic mechanical thermal analysis (DMTA), which sometimes referred to as dynamic mechanical analysis (DMA), is a powerful technique [29–32] used to characterize the viscoelastic response [31], cure degree [2,29] and internal molecular mobility [29] of matrix and composites, and the measured parameters includes [5]: glass transition temperature (T_g), dynamic modulus (i.e. storage modulus E' and loss modulus E'') and the loss factor (tan δ). Meanwhile, DMTA was also adopted to directly investigate adhesion between fibre and resin [26,33,34]. The damping at the interface area was successfully separated from the whole composite, and was proven to be much close to the interface bonding behaviour [33,34]. To the authors' best knowledge, there is still a lack of systematic study on the hydrothermal durability of carbon fibre/epoxy interface using DMTA.

Our previous study [8] has proven that fibre surface treatments can effectively enhance the durability of interface between single fibre and epoxy resin in a relative modest environment (i.e. 95% RH at 40 °C). To realize the application of CFRP in civil engineering, the present study further focuses on the long-term durability of macroscopic CFRP plates with/without fibre surface treatment [8] in the harsher hydrothermal condition using accelerated test method. In this study, the interlaminar shear strength (ILSS), moisture absorption and desorption, scanning electron microscopy (SEM) and dynamic mechanical thermal analysis (DMTA) were adopted to characterize the degradation mechanism of carbon fibre/epoxy interface. The long-term behaviour of two kinds of CFRP plates in distilled water at room temperature was also predicted with the Arrhenius relation theory. Finally, DMTA results in the hydrothermal condition are linked to ILSS results.

2. Experimental program

2.1. Materials

In this study, two kinds of PAN-based carbon fibres (800 tex) with average diameter of 7 μm were provided by Sinopec Shanghai Petrochemical Co. Ltd (Shanghai, China). One was chosen after the carbonisation, without any surface treatments during the manufacturing process, and named as untreated carbon fibre. The other one was chosen after the electrochemical oxidation and further sizing treatments, and was named as oxidized + sized carbon fibre, which possesses the similar mechanical properties to the commercial Toray T300 carbon fibre. The detailed oxidation and sizing process and parameters were reported in previous research [8]. For simplicity, the oxidized + sized carbon fibre was redefined as the treated carbon fibre in contrast to the untreated carbon fibre in this study. Accordingly, the following two kinds of CFRP plates produced with the corresponding fibres (i.e. untreated carbon fibre; oxidized + sized carbon fibre) were defined as untreated CFRP and treated CFRP, respectively. The diglycidyl ether of bisphenol-A (DGEBA) utilized in this study is a commercial epoxy with a brand name of E51 purchased from Xing-Chen Chemicals Co. Ltd. (Wuxi, China). The hardener is methyl tetrahydrophthalic anhydride (MeTHPA) purchased from Qing-Yang Chemistry Co. Ltd (Jiaxing, China). The accelerator is a tertiary amine tris (dimethylaminomethyl) phenol (DMP-30) purchased from Shan-Feng Chemical Industry Co. Ltd (Changzhou, China). The mixture ratio of epoxy, hardener and accelerator is 100:80:2 by weight. The detailed chemical formulas of epoxy, hardener, accelerator and cured epoxy resin [35] are shown in Fig. 1.

Accordingly, two kinds of unidirectional CFRP (i.e. untreated CFRP and treated CFRP) plates were prepared with the corresponding carbon fibres respectively by a filament winding machine (4FW500 × 2000, Harbin FRP Institute, Harbin, China) [36] using the same production parameters. The dimension of each CFRP plate was 100 mm × 100 mm × 2 mm, with approximately 50% volume of carbon fibres. During winding, a fibre bundle was saturated with the epoxy resin when going through the resin tank, and then wound on the metal plate mold coated with the release agent. No pretension was added on the fibre bundle because the friction force formed between the fibre bundle and the impregnation rollers was high enough. The winding speed was set as 0.15 m/s, and the winding interval was 2.7 mm for both carbon fibres. The winding angle of plates was 89.2° and the nominal thickness of both CFRPs was controlled as 2 mm. A total of six carbon fibre layers were wound for both CFRP plates, and the nominal thickness of a carbon fibre layer was ~0.165 mm. The nominal thickness of the fibre layer was calculated. With the winding interval (2.7 mm), the weight per unit area could be obtained based on the linear density (800 g/km), and then the nominal thickness was calculated from the volume density (~1.8 g/cm^3). In theory, both prepared CFRP plates contain ~13,000 filaments per mm^2 along the cross section of specimen. The wound CFRP plates were cured in oven at 120 °C for 3 h followed by 150 °C for another 3 h with the mold. In addition, the epoxy resin plate with 100 mm × 100 mm × 2 mm size was also prepared under the same curing condition (3 h at 120 °C followed by 3 h at 150 °C) with the aluminium mold. More detailed parameters on both CFRP plates are listed in the Table 1.

Cured CFRP and epoxy specimens were cut with a water-cooled diamond saw to the dimensions required by the following tests.

2.2. Interlaminar shear strength (ILSS) tests

The mechanical tests were carried out with a universal machine (WDW100D model, Jinan Shijin Co. Ltd., Jinan, China). In this study, three kinds of typical test methods were adopted to characterize the interlaminar shear strength (ILSS) of CFRP plates, i.e. short-beam shear method, transverse (90°) flexural method and in-plane shear method. Each test was repeated at least five times. The loading speed was 1.0 mm/min.