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Characterisation of the hygro-thermo-mechanical behaviour of organic matrix composites instrumented with optical fibres: A study of interfacial bonding

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ABSTRACT

Moisture diffusion can decrease the mechanical stiffness and strength of organic matrix composites. Recently, Fibre Bragg Grating (FBG) sensors have been used in order to study the hygroscopic ageing of polyester/glass composites at room temperature. In the present study, the hygroscopic strain measurement of polyester/glass composite has been achieved at room temperature. Furthermore, measurements have been done at higher temperatures in order to better understand the combination of both hygroscopic ageing and varying temperatures on the mechanical properties of these composite samples. The Bragg wavelength (λ_b) was found to shift linearly over a temperature range from room temperature to 35 °C. Beyond 35 °C, the Bragg wavelength does not linearly vary as a function of the temperature. A strong variation of the Bragg wavelength above a specific temperature threshold was found. This could be explained according to two mechanisms. Firstly, hygroscopic ageing could result in a decrease of the glass transition temperature (T_g) of the polymer matrix. Therefore viscoelastic behavior may appear beyond this temperature. Hygroscopic ageing could also degrade the interfacial shear strength between the fibre and the resin. Differential Scanning Calorimetry (DSC) analyses showed that hygroscopic aging does not affect the Tg of the polyester resin. Furthermore, it has been found that the hygroscopic aging degrades the interfacial adhesion of the optical fibre/polyester according to the considerable decreases of the interfacial shear strength observed in practice.

1. Introduction

The organic matrix composites (OMC's) have been used widely in structural applications over the past three to four decades [1]. The service life of such structural components is strongly associated to their durability, which itself depends on the environmental conditions they are subjected to. Recently, the effects of humidity on the mechanical properties of polymer matrix composites have been reported by several authors [2–5]. Glass fibers and polyester resin, the two constituents of the composite samples used in this study, present heterogeneous hygroscopic properties. For example, the coefficient of moisture expansion (CME) and the maximum moisture absorption capacity (Ms) are entirely different for these two materials. As a result of this, moisture absorption gives rise to the hygroscopic swelling of the matrix, which, coupled with the strong heterogeneity between the properties of both the matrix and the hydrophobic E-glass reinforcements, induces heterogeneous local stresses within the composite [6]. Considerable efforts have been made by researchers to develop analytical models that can predict the mechanical states occurring during both the transient stage and the permanent regime of the moisture diffusion process of organic matrix composites submitted to hygro-mechanical loads [7,8]. Several studies highlight that the resulting mechanical states bring along structural damage [9–11]. Therefore, the determination of internal mechanical states of polymer matrix composite submitted to the hygro-mechanical loads is critical for reliably predicting the long term behavior and durability of these composite structures. Different techniques have been developed in order to measure the hygro mechanical strain [12,13]. Recently, optical fibre sensors have been used to achieve the measurement of strain and temperature [14]. Optical fibre sensors provide many advantages compared to other strain measurement techniques, such as electrical and piezoelectric sensors, due to their ability to investigate the internal mechanical states within the bulk of the specimen [15–17]. The FBG is an optical filtering device that reflects light of a specific wavelength and is present within the core

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of an optical fiber waveguide. The reflected wavelength depends on the spacing of a periodic variation or modulation of the refractive index that is present. The sensing function of an FBG originates from the sensitivity of both the refractive index of the optical fiber and the grating period within the fiber to externally applied load and temperature. In the authors' previous study, the hygroscopic properties of glass/polyester composites have been identified by using FBG sensors [18]. Authors used FBG sensors in order to characterize the evolution of the hygroscopic strain field in unidirectional fibre reinforced composites, immersed in deionized water, during the moisture diffusion process. Materials properties such as the Coefficients of Moisture Expansion (CME) as well as the transverse diffusivity D_2 were identified from the treatment of the collected data [19]. Additionally, the effect of temperature change on the Bragg wavelength (λ_b), and consequently on the hygroscopic strain, was presented. In this study, the shift in the fiber Bragg wavelength (λ_b) due to the influence of temperature has been examined for the glass/polyester composite specimen. It was observed that the response of Bragg wavelength shifting with temperature is linear over a range from 23 °C to 35 °C. On the contrary, the Bragg wavelength does not exhibit same behavior beyond 35 °C. This could be explained by several phenomena. Among them, the two main factors are, firstly, the influence of the hygroscopic ageing of composite samples which could lead to a fall in T_g, and secondly, a viscoelastic behavior appearing at higher temperatures. Moreover, the absorbed water can induce the debonding of the fibre-resin interface.

For that purpose, the characterisation of the glass transition temperatures of elaborated polyester resin and acrylate coating before and after hygroscopic aging has been achieved. In addition, the determination of the interfacial debonding stress of the silica optical fibre/polyester interface before and after hygroscopic aging has been carried out using the single-fibre pull-out test.

2. Materials and testing methods

2.1. Materials and specimens

An ortho-phthalic polyester resin (POLYLITE 420–731) has been used in this study. The gelling point of this resin is relatively short (about 20 min). Methyl Ethyl Ketone Peroxide (MEKP) has been used as a catalyst for initiating the polymerization of this polyester resin. The employed optical fibres are standard SMF-28e single mode fibres with a 250 μm diameter. This fibre is composed of a silica core and an acrylate coating. The acrylate coating is often used in standard optical fibres in order to protect the silica fibre core. Despite having a hydrophilic behavior, it ensures the protection of fibre against micro-bending and external physical stresses. The optical fibres used in this study were supplied by IX-fiber (SMF28-N/A-0.4 m). The polyester resin/optical fibres samples have been manufactured by a fairly simple method. In this method, a metallic mold, which consists of two steel plates, has been employed. The optical fibres have been placed between two plastic chocks - themselves covered by rubber gaskets (Fig. 1A). The gaskets have the role of protecting the optical fibre against external stress, and consequently prevent fibre breakage. The metal mold was placed into an environmental chamber and heated up close to 50 °C in order to reduce the temperature gradient between the mold and polyester resin. The prepared resins were then transferred into the mold and kept in an environmental chamber for 4 h at ambient temperature (23 °C). After polymerization, the optical fibre/resin specimens were withdrawn from environmental chamber and cut in order to carry out the pull-out test. After cutting and polishing, the specimen surfaces were cleaned with ethanol in order to remove any residual oil and dirt resulting from milling operation. The final dimensions of the specimens prepared for the pull-out test stated below were $(25 \times 10 \times 2)$ mm^3. A series of specimens, some containing optical fibres, and others without the fibres, were used for the ageing tests.

The tested unidirectional composite samples were made of E glass fibres embedded in an ortho-phthalic polyester (POLYLITE 420–731). The specimens were manufactured through the vacuum assisted resin infusion (VARI) method. Several composites, as well as neat resin square plates (1.3 mm thick) were fabricated. The fibre volume fraction (v_f) of the elaborated composites varied from 17% to 22%. The initial weights and dimensions of the specimens were recorded. The FBGs employed in this study were printed on standard SMF-28e single mode optical fibres with a 250 μm diameter. Bragg gratings (10 mm long) were uniform, and centered on a 1555 ± 0.2 nm wavelength. The use of FBGs in composite samples is limited by the ability to insert these sensors in these materials (so as to retrieve the optical signal) whilst minimizing their effect on the composites mechanical behavior. In order to address this issue, the optical fibre containing Bragg grating was inserted between two plates, bonded by polyester resin. The FBGs were either aligned with the reinforcing fibres or set perpendicularly to them. In the area of the grating, the acrylate coating was removed from the FBG in order to get the most direct strain provided by the surrounding material. The final size of the obtained instrumented samples is $90 \times 90 \times 3$ mm^3. In addition, this kind of architecture enables us to limit the stresses experienced by optical fibres during the fabrication process. Two series of specimens were manufactured. The first subset of samples were equipped with FBGs sensors. This group of samples was intended to provide the time-dependent evolution of the internal strain states throughout the moisture diffusion process. The second subset of specimens, which did not contain any Fibre Bragg Grating, were devoted to moisture uptake characterization by means of periodic mass measurements. These samples have the same final size as the instrumented specimens (90*90*3 mm^3). The experimental conditions that these samples were subjected to (with regards to the hygroscopic ageing process) is detailed in the next section.

2.2. Hygroscopic ageing process

2.2.1. Moisture absorption experiments

The hygroscopic ageing tests were carried out on both the composite and neat resin samples in order to identify their diffusive behavior. The initial weights of the samples were recorded. Thereafter, the specimens were immediately placed into deionized water maintained at controlled ambient temperature (20 °C). The change in mass was measured using an analytical electronic scale, marketed by SARTORIUS®, with an accuracy of 0.1 mg. The weight gain versus the square root of time ($\sqrt{t}$) curves for the composite and neat resin samples were then determined in order to follow their moisture absorption kinetics. The results (detailed in Section 3.1) show that the studied samples exhibit a Fickian diffusive behavior.

2.2.2. Identification of moisture diffusion parameters

The identification method used in this study for determining the diffusion parameters was explained in a previous work [19]. This numerical method was intended for identifying both the macroscopic transverse moisture diffusion coefficient (D_2) and the maximum moisture absorption capacity (M_s) of the samples. The numerical method was based on the comparison between the 3D Fick's solution and the experimental measurement of the weight gain occurring during the diffusion process [20]. The method consists of finding out the unknown values of the problem by minimizing the standard deviation $\boldsymbol{q}$ (Eq. (1)) using a Gauss-Newton algorithm:

$$\boldsymbol{q} = \sum_i [\boldsymbol{M}(t_i) - \boldsymbol{M}_i]^2 \tag{1}$$

where $\boldsymbol{M}(t_i)$ is the moisture content calculated at time t_i from the analytical solution to Fick's model, whereas $\boldsymbol{M}_i$ is the corresponding experimental point.