INTERNAL STRAIN MEASUREMENT OF GLASS-POLYESTER COMPOSITES UNDER HYGRO-THERMAL AGEING TEST USING FIBER BRAGG GRATINGS

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Abstract
This study is devoted to the identification of the moisture expansion coefficients of composite materials by means of a novel method of measurement. This method is based on the insertion of Fiber Bragg Grating (FBG) sensors in the composite structure. The sensor enables to measure the hygroscopic strain induced by the diffusion of moisture in the composite. The evolution of the strain, due to the moisture absorption, was represented according to the water uptake, in order to characterize the moisture expansion coefficient.

1 Introduction

Organic matrix composites are extensively used in several engineering applications due to their good stiffness to weight ratio. However, when such materials are exposed to humid environments, they absorb moisture. Moisture diffusion greatly affects the hydro-mechanical properties [1] as well as the multi-scale internal mechanical states experienced by the constituents of polymer matrix composites. Consequently, the durability and reliability of structural parts made with such materials can be impaired in humid environments. In glass/polyester composites, which were used in this study, the matrix absorbs a significant amount of water whereas fibers absorb almost no moisture. This results into a significant mismatch in the moisture induced volumetric expansion experienced by the matrix and the fibers. Such inhomogeneous strain field may lead to high localized stresses and eventually, to the failure of the material [2]. Numerous authors have used Fiber Bragg Grating (FBG) sensors [3, 4] in order to achieve the measurement of the local strain, temperature and other quantities of engineering interest, experienced within the bulk of the studied samples. A FBG clearly has many advantages over other strain sensors, since it enables to investigate the internal mechanical states in the depth of the specimen.

The main objective of the present work consists in demonstrating the suitability of FBG sensors for investigating the hygroscopic ageing of organic matrix composites. This was achieved by an experimental study of both the time-dependent moisture uptake and strain field in composite containing FBG sensors.

The Fiber Bragg Grating (FBG) sensors were used to characterize the evolution of the hygroscopic strain field in unidirectional fiber-reinforced composites specimens immersed in liquid water, during both the transient and the permanent stage of the moisture diffusion

process. Besides, the time-dependent average moisture uptake over the volume of the samples will be followed through the classical gravimetric method until the permanent regime is reached. Eventually, the longitudinal and transversal moisture expansion coefficient (CME) of these composite samples will be determined by using the data collected through the optical sensors, positioned either in a parallel or perpendicularly to the reinforcing fibers.

2 Materials and testing methods

2.1 Materials and specimens

The tested unidirectional composite samples were made of E-glass fibers embedded in an ortho-phtalic polyester resin (POLYLITE 420-731 from REICHOLD) polymerizing at room temperature. The specimens were manufactured through the vacuum assisted resin infusion (VARI) method. Several composite, as well as neat resin plates with cross section 90x1.3 mm^2 were realized. After cutting and polishing, the samples surfaces were cleaned with ethanol in order to remove any oil residual and dirt resulting from machining. The initial weights and dimensions of the specimens were recorded. The FBGs employed in this study were inscribed in the standard SMF-28e single mode optical fiber with a 250 μm diameter. The Bragg gratings (10 mm long) were uniform, and centered on a 1535 nm wavelength. The optical fiber containing Bragg grating was inserted between two plates, bonded by polyester resin. The FBG were either aligned with the reinforcing or set perpendicularly to them (as shown on figure 1 below). This type of specimen architecture enabled us to obtain instrumented samples, the final size of which is 90x90x3 mm^3.

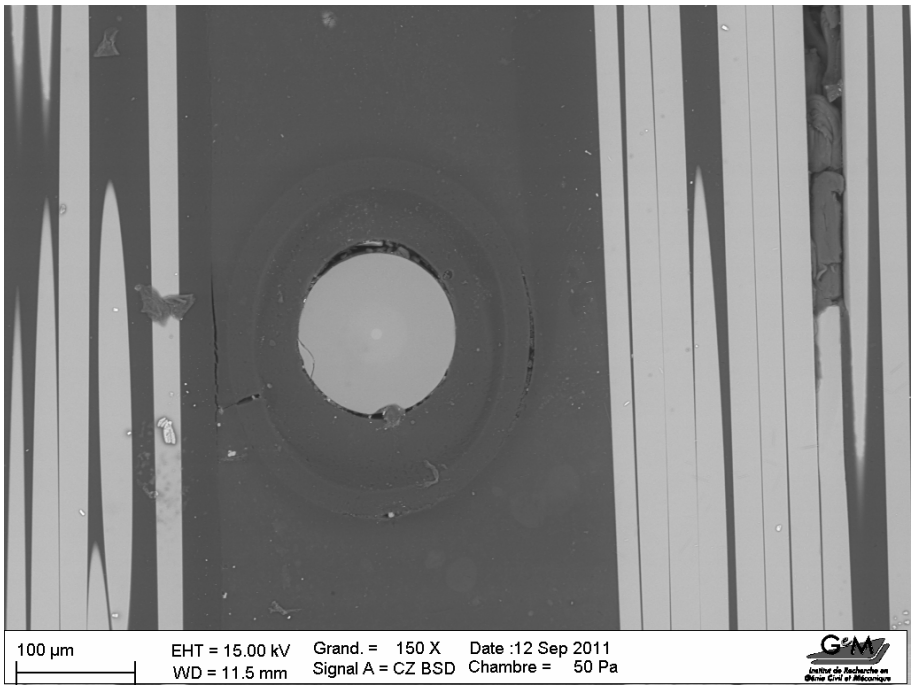

Figure1. *SEM micrograph showing an optical fiber embedded perpendicularly to the reinforcing fibers.*

The main parameters controlling the moisture diffusion kinetics, namely the diffusion coefficients and the maximum moisture absorption capacity do strongly depend on the volume fractions of the constituents (i.e. those of the fibers and the polymeric matrix). Therefore, the reinforcing fibers content of the composite affects the magnitude of the multi-scale mechanical states to be monitored during the present work.

As a consequence the precise determination of the volume fraction occupied by the glass fibers in each investigated sample was mandatory in the context of the present work. Several techniques have traditionally been employed in order to determine the fiber volume fraction

(v_f) of organic composites. In the present work, the identification of the glass-fiber volume fraction in the manufactured samples will be handled according to the dimension and weight analysis method.
Optical microscopy and Scanning Electron Microscopy were used for investigating the microstructure of the manufactured samples (see figure 1). According to both characterization methods, the fabricated specimens are void-free. As a result, the total volume of the composite (V_t) is equal to the sum of the volume occupied by the glass fibers (V_f), on the one hand and by the resin (V_r), on the other hand:

$$V_t = V_f + V_r \tag{1}$$

A similar relation is also valid for the corresponding masses:

$$M_t = M_f + M_r = \rho_f V_f + \rho_r V_r = \rho_f V_t \tag{2}$$

Where the symbol ρ denotes the mass density.
The reinforcing fibers volume fraction corresponds to the following ratio:

$$v_f = \frac{V_f}{V_t} = 1 - v_r = 1 - \frac{V_r}{V_t} \tag{3}$$

Considering (2) and (3) enables to write:

$$v_f = \frac{\rho_t - \rho_r}{\rho_f - \rho_r} \tag{4}$$

Dimensions and mass of the specimens were measured in order to determine their total volume (V_t) and their total mass (M_t), from which the total mass density calculation is straightforward. The same method was applied to the neat resin samples, so that the numerical value of ρ_r could be deduced. Glass fibers mass density is a well known parameter that is widely available in the literature [5]. The results obtained through this calculation method, for the glass fibers volume fraction, are displayed in Table 1.

Specimen group N°	1	2	3
v_f (%)	17	21	22

Table 1. Fiber volume content of the manufactured samples.

2.2 Experimental set up and hygroscopic ageing process
Two kinds of specimens were manufactured for the purpose of the present study. The first subset of samples was instrumented by FBGs sensors. This group of samples was intended to provide the time-dependent evolution of the internal strains states throughout the moisture diffusion process. The second subset of specimen, which did not contain any optical fiber, was intended to be used to follow the moisture uptake, instead, owing to periodic mass measurements. The samples were eventually immersed in de-ionized water.
Each subset of samples contains specimens taken from the specimen groups listed in table 1, so that the moisture diffusion kinetics as well as the mechanical strains would both be available for a given value of the reinforcing fiber content of the manufactured specimen.

2.3 Technique of measurement

Mechanical strains measurement through FBG has been presented in various papers, such as [6]. A FBG consists in a series of grating planes formed along the fiber axis. If the Bragg condition is fulfilled, a light signal propagating into the device can interfere constructively to the waves reflected by each of the grating planes. As a result, a back reflected signal with a center wavelength commonly known as the Bragg wavelength λ_b is formed. In the case when the FBG is submitted to a homogeneous axial strain ε_z and uniform temperature change ΔT, the Bragg wavelength experiences a deviation $\Delta\lambda_b$ from the reference value λ_{b0} corresponding to the unloaded state ($\varepsilon_z = 0$; $\Delta T = 0$):

$$\frac{\Delta\lambda_b}{\lambda_b} = \frac{\lambda_b - \lambda_{b0}}{\lambda_b} = a\varepsilon_z + b\Delta T \qquad (5)$$

Coefficients a and b depend on the nature of the optical fiber and the FBG inscription parameters. According to relation (5), at isothermal conditions, the Bragg wavelength shift $\Delta\lambda_b$ is proportional to the axial strain (ε_z).

In the present study, the strong hygroscopic strain experienced by organic matrix composites exposed to moisture diffusion is expected to induce an axial elongation of the optic fiber containing the Bragg grating [7], resulting in a significant Bragg wavelength shift according to equation 5).

3. Results and discussion

3.1 Hygroscopic aging tests

The weight gain versus the square root of time curves obtained for the neat resin as well as those corresponding to the composite samples are shown on figure 2a and 2b, respectively).

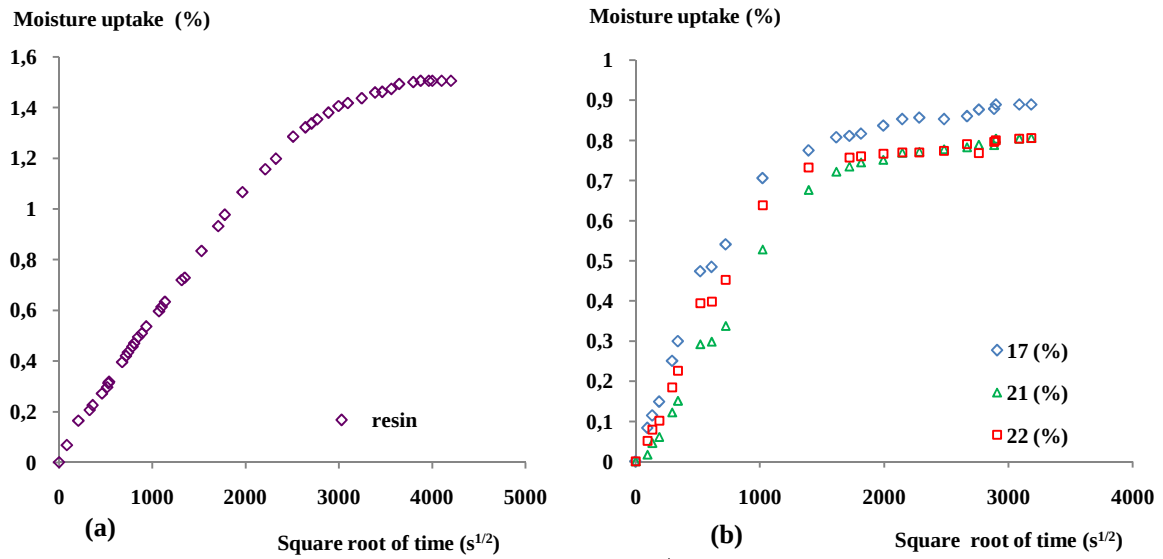

Figure 2. The weight gain versus the square root of time ($\sqrt{t}$) curves for all composite and resins specimens.

The neat resin (figure 2a) exhibits a time-dependent moisture uptake typical from Fickian kinetics. According to figure 2b, the three composite samples exhibit an almost linear evolution of their moisture uptake for several months until a pseudo-plateau indicating that the saturation of the diffusion process is almost reached. Thus, it is realistic to consider a Fickian diffusion behavior for the investigated composite samples.

The identification of the diffusion parameters was based on the comparison between the classical 3D Fick's analytical solution published by Crank [8] and the experimental

measurement of the weight gain occurring during the diffusion process. The method consists in seeking the unknowns of the problem by minimizing the standard deviation between the calculated and measured quantities using a Gauss-Newton algorithm.
The moisture diffusion parameters of composite and neat resin specimen obtained owing to this method of identification are presented in the Table 2:

	$D_{apparent}$ (mm^2/s)	D_{11} (mm^2/s)	D_{22} (mm^2/s)	Ms (%)
Composite (v_f = 17 %)	$2.2*10^{-7}$	$2.31*10^{-7}$	$2.07*10^{-7}$	0.85
Composite (v_f = 21 %)	$2.1*10^{-7}$	$2.31*10^{-7}$	$1.74*10^{-7}$	0.78
Composite (v_f = 22 %)	$1.83*10^{-7}$	$2.31*10^{-7}$	$1.97*10^{-7}$	0.79
Bulk resin	$2.31*10^{-7}$	$2.31*10^{-7}$	$2.31*10^{-7}$	1.5

Table 2. Moisture diffusion parameters of composite and neat resin.

3.2 Internal strain measurement

The measured Bragg's wavelength shift enables to determine the strain experienced in the center of the instrumented samples, from relation 5, provided that the thermal contribution, namely, the product b.ΔT is negligible by comparison to the quantity of interest: $a.\varepsilon_z$. Figure 3(a) shows the evolution as a function of square root of time of the hygroscopic strain obtained for the three composite specimen instrumented by FBG positioned perpendicularly to the reinforcing fibers. The temperature change of the liquid water surrounding the samples is displayed on the same figure.
One can notice that, the influence of the fiber volume fraction on the hygroscopic strain is almost undistinguishable. However, the time-dependent evolutions of the hygroscopic strains do present a significant scattering. This scattering almost follows the temperature change of the ambient fluid, which ranges from 0°C to 6°C throughout the course of the aging test.
Such a temperature variation is actually not negligible as regard to the Bragg peak shift.
Figure 3(b) shows the evolution as a function of moisture uptake of the hygroscopic strain obtained for the three composite specimen instrumented by FBG positioned perpendicularly to the reinforcing fibers. Such a curve has to be considered in order to deduce the coefficient of moisture expansion of the studied specimens.
Nevertheless, since the temperature variation contributes to the Bragg peak shift, it is necessary to correct this effect before analyzing more precisely figure 3(b).

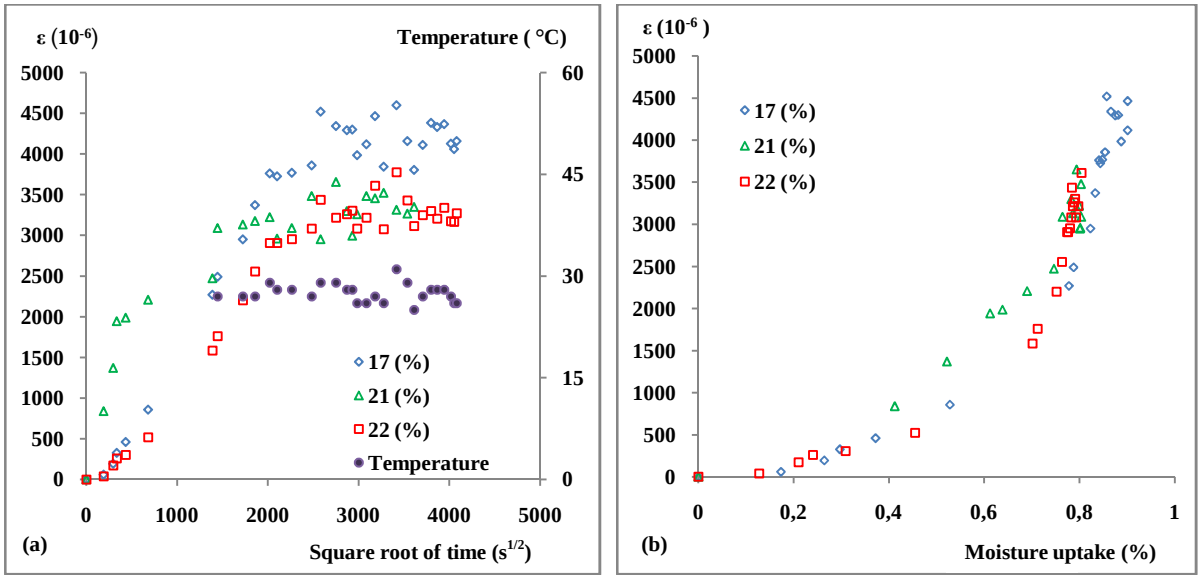

Figure 3. Evolutions of the strain as a function of square root of time (a) or moisture uptake (b) for the instrumented composite specimens.

3.3 Separation of thermal and hygroscopic effects

From the scattering of the strains deduced from the Bragg peak shifts, one can expect that the temperature change during the experimental investigation had a significant influence on the measurements. In such a situation, it is necessary, to separate the contribution due to the thermal strain from the collected peak shifts in order to determine a more realistic estimation of the hygroscopic strains.

The shift of Bragg wavelength as a function of an imposed, controlled temperature is presented in figure 4.

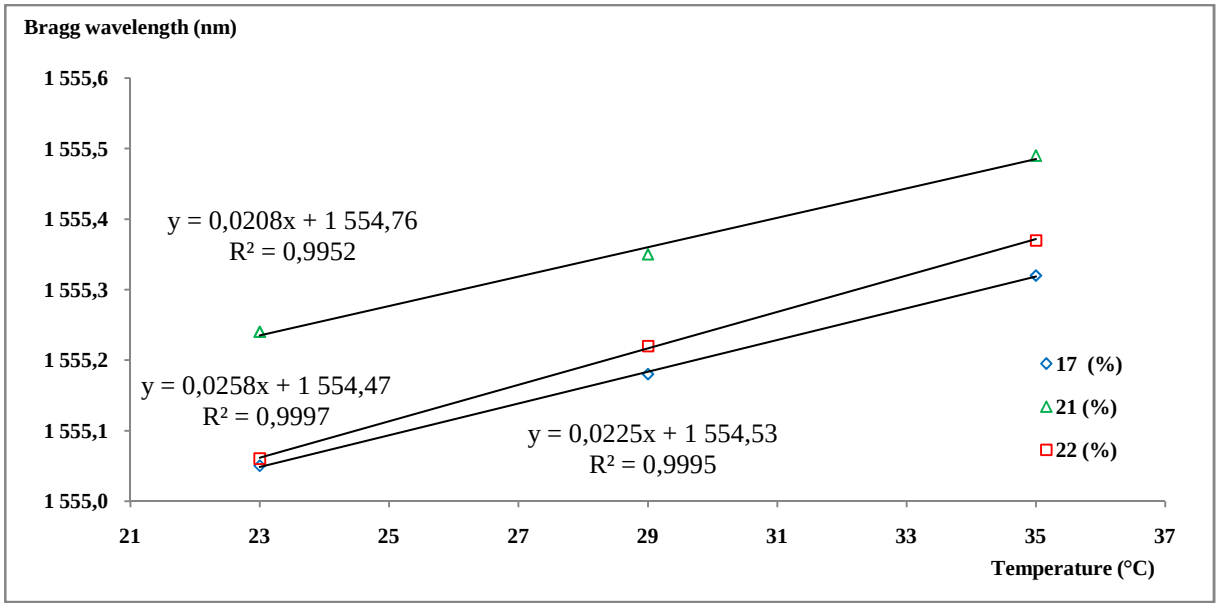

Figure 4. Bragg wavelength shifting as a function of temperature.

From the slope of the curves presented on figure 4, one can separate the effects induced by the temperature change from that of the hygroscopic strain on the measured Bragg wavelength shifting λ_b.

Figure 5(a) represents the strains after correction as a function of the square root of time for the composites with different fiber contents. The corrected strains of these composite specimens were plotted as a function of moisture content (figure 5(b)).

We can observe a high linearity for the three curves of strain, especially, on the saturation pattern compared to the initial measurements (before correction). On the other hand, the deformation of the composite specimens with 17 % of reinforcements remains disturbed after correction of the effect of temperature. Knowing that this disturbance appears in a saturated state, consecutive evolution of the deformation is probably associated with a defect of the microstructure, such as a cracking. This anomaly will be investigated by a micrographic study on this sample after the aging test.

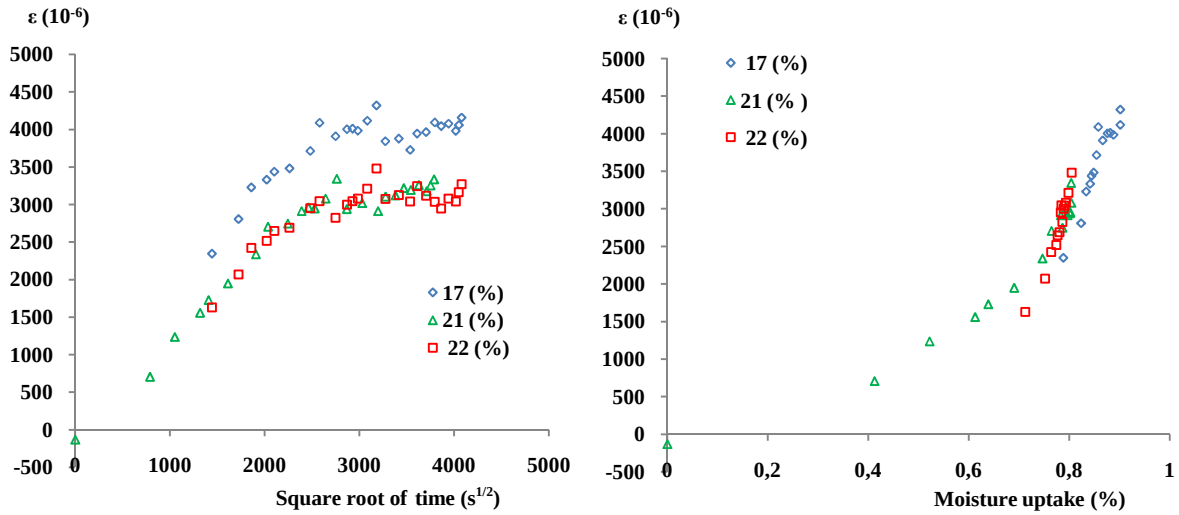

Figure 5. Time dependent evolutions of (a) the hygroscopic strain (a) and (b) the moisture content.

From the slope of the hygroscopic strain as a function of the water uptake, one can also determine the coefficients of moisture expansion of the studied samples. The precise values of these coefficients are gathered in Table 1.

v_f (%)	ΔM (%)	$\Delta\varepsilon_{11}$ (10^{-6})	$\Delta\ \varepsilon_{22}$ (10^{-6})	β_{11}	β_{22}
17	0.85	180	4000	0.022	0.472
21	0.79	330	3140	0.042	0.397
22	0.78	200	3100	0.026	0.397

Table 3. Coefficients of Moisture Expansion.

4. Conclusions

In this work, the hygroscopic strains experienced by composite samples immersed in de-ionized water were followed from the transient to the permanent stage of the diffusion process. The measurements were carried out owing to Fiber Bragg Grating (FBG) sensors.
The combination of the obtained results to the knowledge of the moisture uptake enables the identification of the macroscopic coefficients of moisture expansion of the studied samples, also.

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