



# Studies of hygrothermal degradation of a single fiber composite: An iterative approach with embedded optical sensors and numerical analysis



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## ABSTRACT

The hydrothermal ageing of glass/epoxy interface is investigated using an experimental-numerical approach on cylindrical epoxy specimens with centrally located optical fibers. A 24 mm long Bragg grating sensor is inscribed on the optical fiber and used to monitor strains along the fiber, due to processing and subsequent ageing in water at 50 °C. The distributed strains are used to: (a) evaluate the residual strain field developed during processing, employing a parametric finite element identification scheme, (b) monitor the evolution of the moisture induced strains during ageing using linear and non-linear responses for the epoxy recorded experimentally, (c) track debond growth at the interface, generated during ageing, by adopting a concentration dependent cohesive finite element model. Good agreement is found between experimental data and simulations until 47 days of immersion (or 63% of saturation). Afterwards, the model is not quantitatively accurate but indicates well the trend of the experimental data.

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## 1. Introduction

It is well known that material's ageing takes place at the molecular level by inducing important changes in their chemical structures [1–5] affecting, among others, the interfaces in composites materials [6–8]. Damage in the interface reduces significantly the load transfer capability of the matrix and reinforcement and, by lowering the interfacial shear strength [9], triggers extensive fiber/matrix debonding impairing the overall mechanical and fracture properties of composites [10,11].

An important question to be addressed is the role of residual strain on ageing and failure of composites. For example, in high temperature composites, where the residual strain field can be considerable, it is often found that microcracks are generated which act as paths for contaminants to penetrate even before mechanical loading [10]. A wide range of destructive and non-destructive methodologies is available that attempts to link the local distribution of residual stresses to damage evolution and composite's failure [11]. Such information is of primary importance in the efforts to characterize the ageing phenomenon since a redistribution of stresses is generated by the hygric expansion of the material [12].

Although a large amount of research has been conducted to determine the effects of hygrothermal ageing on mechanical properties and damage [13–15], a comprehensive analysis is very difficult to carry out because of the inherent complexity of the phenomenon and difficulties in extracting quantitative experimental data. In this work, the matrix response and interface debonding are investigated by exposing a glass-epoxy, single fiber composite (SFC), to hygroscopic strains at 50 °C. The reinforcement of the SFC is a centrally located optical fiber having a long fiber Bragg grating (FBG) sensor inscribed on it which allows to record the axial strains along its axis after postcuring, drying and during water absorption. In such a configuration, the fiber is the reinforcement and a sensor over a certain length along its axis. A point-wise deformation in the direction of the fiber is obtained by interrogating the sensor with an optical low coherence reflectometry (OLCR) based technique [16]. Thus strain data due to processing, radial cuts, and hydrothermal ageing are obtained and exploited to determine: (a) a residual stress field due to curing/postcuring (Section 6.1), (b) the mechanical response of the epoxy (Section 6.2) and (c) the evolution of debonding during ageing (Section 6.3). Pertinent numerical modeling is carried out and used to analyze the data at each step of the thermal/hygric loading and damage events (i.e. fiber fracture and interface damage).

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## 2. Materials and specimens

The epoxy used in the present studies is a mixture of two resins DER330®, DER732® and the hardener DEH26® provided by the DOW chemical company. This mixture, in fixed weight proportion of 70:30:10, respectively, is casted in a vertical mould in which the fiber is kept aligned axially by a holder. The resin is subjected sequentially to a curing plateau at 30 °C for 24 h, a post-curing phase at 70 °C for 9 h and a drying dwell at 50 °C for 7 days to ensure the specimen dryness before immersion to a water bath at 50 °C. More details on the fabrication process can be found in [12]. The resulting specimen is a cylinder of 12 mm in diameter and length of 40 mm (Fig. 1a), with a centrally located optical fiber without any coating and surface treatment. Two groups of SFC specimens are produced:

- The first one of two specimens is used for the determination of the three dimensional residual strain field generated during processing only and is labeled as “CC” followed by the specimen number.
- The second one of four specimens is used to monitor the evolution of the hygroscopic strains and interfacial damage during ageing. This group is labeled as “SFC” followed by the specimen number. The water absorption of epoxy in time is studied by ageing several control specimens [12].

The optical fibers used herein are standard single mode telecommunication fibers of 125/9 μm cladding to core diameter and grating length of 24 mm (Fig. 1a) obtained from Avensys Inc. Such fibers exhibit Young's modulus  $E_f = 72$  GPa, Poisson ratio  $\nu_f = 0.19$ , elongation at break of more than 5%, and average ultimate tensile strength  $\sigma_f^u = 6$  GPa [17] at room temperature. Note that these characteristics decrease with increase in temperature and humidity, especially if the fiber is simultaneously subjected to mechanical loads [18]. Moreover, the inscription of FBGs lowers the fibers strength because of the introduction of a higher subcritical crack density region during irradiation over the FBG length [19]. The properties of the epoxy are obtained by testing, dog-bone-like specimens made of the same resin, aged in the same conditions and tested in tension, to retrieve the evolution of the material properties as a function of the water content. The measured resin baseline elastic properties, at room temperature, in dry condition, are: Young modulus  $E_m \cong 2.4$  GPa and the Poisson's ratio  $\nu_m = 0.38$ .

## 3. Experimental methods

### 3.1. Strains measurements

An FBG is a periodic modulation of the refractive index of the core of an optical fiber that allows a permanent capability to reflect a narrow light spectrum. This spectrum is characterized by a peak centered on a particular wavelength,  $\lambda_{BO}$ , called Bragg wavelength, which is normally taken as reference signal. In isothermal conditions any change in this wavelength is proportionally related to the applied strain on the fiber.

In this work long length FBGs are interrogated using the OLCR based method. A description of the setup, methodology and its working principle can be found in [16]. The technique has an accuracy of about  $\pm 50 \mu\epsilon$  and a spatial resolution of less than 20 μm over more than 25 mm grating length. In fact, using the OLCR setup a position dependent wavelength,  $\lambda_B(z)$ , is determined and the mechanically induced axial strain on the fiber,  $\epsilon_z(z)$  as a function of the position along the sensor, is obtained from the following relation:

$$\frac{\lambda_B(z) - \lambda_{BO}}{\lambda_{BO}} = (1 - p_e)\epsilon_z(z) \quad (1)$$

where  $p_e = 0.215$  is the grating gage factor. A compensation to  $\epsilon_z(z)$  is required if thermal strains are present. In this work the fiber axial deformation, as a function of position along the grating when the SFC is subjected to residual strain and swelling, is reported.

### 3.2. Residual strain analysis

To obtain an approximate process induced residual strain field, a modified crack compliance methodology, in combination with strain data on the fiber obtained from three radial cuts, is implemented. Thus, two specimens (i.e. CC1 and CC2) are cut radially, using a lathe and a diamonded disk, at different  $z$  positions shown schematically in Fig. 1a (dashed lines). The machining process is performed without water irrigation in order to avoid resin swelling and a low cutting speed is used to limit the development of heat during the process. The depth,  $a_T$ , at each position, is increased by 1 mm at a time and after each cut the fiber's axial deformation is retrieved and related to the distribution of residual strain in the epoxy (Section 5.2).

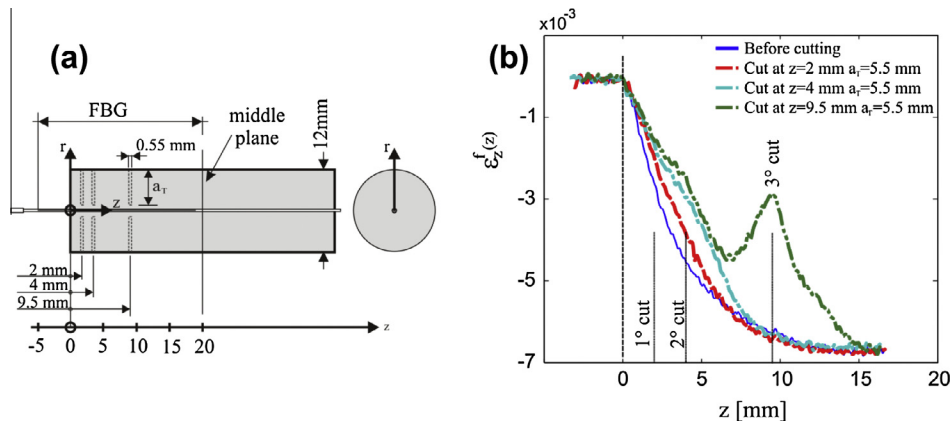


Fig. 1. (a) Cylindrical specimen, dashed lines indicate the circular cuts machined for the analysis of the residual strains field, (b) fiber axial deformation evolution on specimen CC1 (see text for details).

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