



Combined experimental/numerical investigation of directional moisture diffusion in glass/epoxy composites



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ABSTRACT

A combined experimental and numerical investigation is conducted on the anisotropic water diffusion behaviour of unidirectional glass/epoxy composites. Experimental diffusivity values are obtained by immersing thin material slices for each of its planes of orthotropy extracted from a thick composite panel and interphase measurements are performed using thermal analysis. In order to elucidate the observed anisotropy, the diffusion process is modelled at the microscale using a representative volume element (RVE) of the material with random fibre distribution. Water concentration gradients are applied to the micromodel and a homogenisation procedure is used to retrieve the macroscopic diffusivity coefficients. The influence of the interphase around the fibres on the diffusion process is modelled by making the matrix diffusivity a function of the distance to the nearest fibre using a level set field. The models are used to fit the experimental data and test a number of hypotheses that may explain the observed anisotropy. The effect of fibres acting as barriers for water movement is found to partially explain the observed transverse diffusivity. However, a fit is only obtained by allowing faster diffusivity at the interphase. In the longitudinal direction, a fit can only be found by allowing for orthotropic interphase diffusivity.

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1. Introduction

The influence of hygrothermal ageing, a combination of high temperatures and moisture ingress, on the mechanical behaviour of composite materials is an important design driver for structures such as wind turbine rotor blades, which are required to withstand extreme service environments. Experimental characterisation of the hygrothermal ageing phenomenon remains a challenging research area, despite the already large body of literature on the subject. Many of the current knowledge gaps stem from the fact that hygrothermal ageing is a complex combination of physical and chemical phenomena that operate at different time and spatial scales [1]. Furthermore, the incurred degradation strongly depends on the material components involved [2] and on the exposure environment.

Over the past few years, a number of experimental studies on hygrothermal ageing has been performed with focus on elucidating the underlying microscopic degradation mechanisms, such as differential swelling, plasticization and interface debonding [3–6], responsible for the impact of ageing on macroscopically measured material properties. Additionally, with the advent of novel numerical analysis techniques such as concurrent multiscale finite element analysis (FE²) [7] and the development of advanced material models [8], such degradation mechanisms can be modelled and their nonlinear interactions can be captured across spatial scales.

However, since the ageing process is driven by water ingress, it is also important to understand the process of water diffusion across scales. For fibre reinforced polymers, the material microstructure brings complexity to the diffusion phenomenon since water molecules have to go around the fibres for diffusion in transverse direction [9], giving rise to orthotropic diffusion behaviour. Furthermore, chemical interaction between the epoxy and fibre sizing creates an interphase region around the fibres

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where water diffusivity can be different from the one in bulk resin regions [3].

In this work, a combined experimental and numerical approach was used to elucidate the water diffusion process in composite laminates, expanding upon early results published by the authors as a conference paper [10]. For the present contribution, new experimental interphase measurements are included, the numerical formulations are presented in greater detail, new calibration examples are presented and additional results and discussion are included. For the experimental part, a thick unidirectional glass/epoxy composite panel was manufactured and thin slices were cut along the three orthotropy planes of the material. The slices were immersed in demineralised water at 50°C and weighed at regular intervals. By fitting a unidimensional analytical solution of Fick's second law of diffusion to the experimentally obtained water uptake curves, the orthotropic diffusivity constants were retrieved. Using a combination of scanning thermal microscopy and local thermal analysis, the existence of an interphase was confirmed for the present composite system and its approximate thickness was measured.

The diffusivity coefficients were also numerically obtained through homogenisation of the microscopic diffusion behaviour. For this purpose, a three-dimensional Representative Volume Element (RVE) was subjected to water concentration gradients in each direction. By integrating the flux components in the microscopic volume, homogenised diffusivity values were obtained. In order to account for the impact of the fibre/matrix interphase on the diffusion process, the resin diffusivity was made a function of the distance to the nearest fibre. A parametric study on the RVE size and mesh density was performed and the model was used to verify the validity of a number of hypotheses put forward in literature that may explain the observed anisotropy.

2. Experiments

2.1. Materials

The material system considered in the present study consists of the epoxy resin EPIKOTE 135/1366, manufactured by Momentive [11], with embedded unidirectional glass fibre fabrics (Saertex PPG 2002 2400tex [12]). For the resin preparation, the monomer and hardener were mixed in a 100:30 ratio and the resultant mixture was degassed in vacuum in order to minimise void content. Unidirectional glass fibre plies were stacked and infused through vacuum bagging in a heated flat mould.

In order to experimentally investigate the anisotropic diffusion behaviour through weight measurements, specimens must be devised in such a way that the water movement in a certain direction is promoted while diffusion in the other directions is minimised. This can be achieved by selectively sealing specimen edges [13] or by cutting thin material slices in each direction [9]. Here, the latter procedure was chosen and a thick laminate of 50 unidirectional (UD) plies (total thickness of 30 mm) was manufactured in order to obtain specimens with high width/thickness and length/thickness ratios for three different orientations.

Each UD fabric ply used in the manufacturing was mainly composed of fibres oriented at 0° (95% in weight) with stability rovings oriented at 90° accounting for 5% of their weight. In order to obtain a purely UD laminate, the stability rovings were manually removed prior to infusion. The panel was cured for 3 h at 30°C and 5 h at 50°C, with a subsequent post-curing period of 10 h at 70°C. After curing, 30 × 30 mm² slices were cut from the panel along the three orthotropy planes of the material, as shown in Fig. 1.

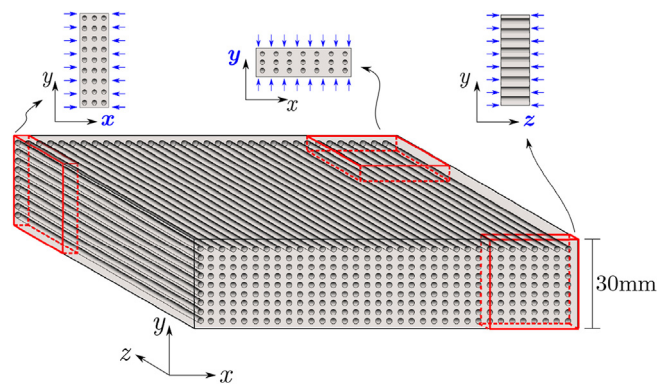


Fig. 1. Material slices and preferred diffusion directions.

Three slices were cut in each direction using a water-cooled diamond saw and were grinded with a Labopol 30 polishing machine using progressively higher sanding grits ranging from 320 to 1200, with a resulting average thickness of 0.94 ± 0.06 mm and an average width/thickness ratio of 31.9 ± 2.3 . Finally, the surfaces were polished using a suspension of diamond particles with average diameter of 1 μ m. The final surfaces were inspected for defects using a Motic BA210 optical microscope. It must be stated that the occurrence of microscopic failure events such as interface debonding during specimen preparation cannot be completely ruled out. Using three additional specimens, average values of fibre volume fraction of $53.10 \pm 0.27\%$ and void content of $0.85 \pm 0.21\%$ were obtained through loss on ignition, according to the ISO 1172 standard.

2.2. Conditioning

In order to obtain accurate measurements of the maximum water uptake, the slices were first dried for approximately 200 h in a desiccator at 50°C, after which a stable weight was achieved. The choice of the reference state in diffusion experiments on polymer matrix composites can be a complex one, since additional ageing processes such as physical ageing and oxidation can occur during the initial drying phase. Due to changes in the polymer structure caused by these additional ageing mechanisms, the diffusivity and the maximum uptake may suffer both reversible and irreversible changes [14,15]. In this work, oxidative reactions were avoided by applying vacuum to the desiccator during the drying process, while the short duration of the drying phase, enabled by the use of thin slices, helped to minimise the influence of physical ageing.

The slices were subsequently immersed in demineralised water kept at a temperature of $50.0 \pm 1.5^\circ\text{C}$. This immersion temperature was chosen in order to accelerate water uptake while keeping a safety margin of 20 °C from the glass transition temperature (T_g) measured in saturated resin specimens (70 °C). The T_g of the unaged resin is 87 °C [16]. Dependency of the diffusivity on the immersion temperature is well documented in literature [17] and will not be treated in the present work.

Water uptake was individually tracked for each slice through weight measurements, according to the ASTM D5229/D5229M-14 standard, using a Kern ALJ 160-4NM analytical balance with 0.1 mg resolution. Hourly weighings were performed in the first 8 h of immersion, two weighings were performed on the second day and the frequency was decreased to one weighing per day for the remainder of the experiment. Only one slice was taken out of immersion at a time, with the complete weighing procedure for each individual slice taking approximately 1 min.

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