



Study of the moisture/stress effects on glass fibre/epoxy composite and the impact of the interphase area



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ABSTRACT

This paper shows the influence of interphase areas in a UD glass fibre/epoxy composite on water diffusion process and mechanical properties thanks to experimental, analytical and numerical analysis. The interphase is first characterized in terms of molecular mobility by micro-thermal analysis and in terms of mechanical properties by force measurements with atomic force microscopy. The measurements show an increase in elastic modulus and in glass transition temperature around each fibre. Optical microscopy reveals an important heterogeneity of fibre distribution inside the matrix with many contacts between fibres. A realistic microstructure including the interphase size (defined by μTA) and fibre distribution has then been considered to model the composite accurately. Diffusion parameters resulting from water absorption measurements on both the resin and the composite were also used in the finite element analysis. The comparison between experimental data, analytical and finite element models shows that the water diffusion coefficient is about five times higher in the interphase area than in the matrix whereas the gradient in modulus around the fibres barely modify the composite mechanical response.

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1. Introduction

The properties of composite materials at initial state and their evolutions under wet service conditions are highly influenced by the interface or interphase created between the matrix and the reinforcement [1–3]. The means of investigation of this area require specific techniques operating at sub-micron scale [4,5]. Among these techniques, AFM and nanoindentation have been frequently used in the last decade to characterize the interphases in organic matrix composites [6–12]. In most cases, due to the lateral resolution of the probe and to high indentation depth, boundary effects often lead to an increase of the apparent modulus close to fibre surface, suggesting that the interphase is much harder than the matrix. Nevertheless, in some cases and with the use of specific techniques, the interphase appears softer than the matrix, due to an incomplete curing [13–15] or to plasticizing effects resulting from fibre silane treatment [16–19]. The interphase can then be characterized by a molecular mobility higher than the bulk matrix one, which influences water diffusion and solubility inside the composite material [20].

Many authors [21–25] have studied the water uptake of epoxy resins and obtained different diffusion coefficient values. Indeed, the resin diffusion kinetics depends directly on many parameters such as, the chemical composition of the resin, the curing

temperature and/or the curing agent nature. In the case of composite, the water uptake depends on fibre volumic fraction and on fibres arrangement [23,26,27]. So, many values have been proposed in literature. To describe the diffusion kinetics several analytical models can be used. The most simple is based on Fick's law [21,25,28,29] and allow a good description of water uptake for short times of diffusion. At longer times, a divergence between Fick's model results and experimental values is often observed. It can be explained by the assumptions made in Fick's model that water molecules are not chemically bonded to the matrix network.

In the literature, some finite element models are proposed to simulate the water diffusion based on Fick law [30,31]. The impact of fibre spatial distribution on water diffusion kinetics has been studied by many authors [26,27,32]. For high fractions of fibre in the composite it is really important to take into account the real distribution especially the areas where fibres are in contact [25].

When immersed in water, the fibre/epoxy composite undergo differential swelling due water absorption. This volumetric expansion generates stresses in the composite which decrease the durability of the material. Many authors have observed and measured the swelling vs. the water uptake for epoxy composite. The value of the moisture expansion coefficient is between $10^{-3}/\% \text{ H}_2\text{O}$ and $10^{-2}/\% \text{ H}_2\text{O}$ [33–40]. A finite element approach enables the calculation of stress levels resulting from water uptake (i.e., swelling). As example, Vaddadi et al. [40] obtained for carbon/epoxy composite a maximal stress around 30 MPa at fibre/matrix interface or in areas where the matrix is confined between fibres.

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The aim of this study is to use properties determined at (sub) micron scale to model the macroscopic behaviour of a glass fibre/epoxy composite. The influence of glass fibres and more especially of the interphase areas on water diffusion kinetics is precisely described. The numerical models developed are based on the real fibre distribution which includes barrier effects and interphases around each monofilament. The mechanical and thermal properties of the interphase area have been obtained from microthermal analysis and atomic force microscopy measurements. The Fick's model has been chosen to determine the kinetics of water diffusion in the matrix. Different numerical models have been developed in this work to simulate water uptake, coupling effects between water uptake and stress generated and the three-point bending test.

2. Experimental part

2.1. Material preparation

Two types of samples materials have been prepared: a composite plate based on epoxy matrix with glass fibres and an epoxy resin plate (without fibres).

The thermosetting matrix is a DGEBA-based epoxy resin (Araldite® LY556 from Ciba-Geigy) with aliphatic polyamine (XB3486 from Huntsman) selected as hardener at 33 parts by weight of resin. This resin has a long pot life compatible with the filament winding process at ambient temperature used to obtain laminas. E-glass fibres (300 Tex, 14 μm) have been treated with a commercial sizing. The composite plates were realized by filament winding at 120 °C for 2 h followed by a post curing stage at 140 °C to reach an infinite glass transition temperature of 104 °C (± 2 °C) (measured by DSC at 10 °C/min). The porosity resulting from filament winding process remained lower than 1 vol.% for a fibre content of 50 vol.% The fibres distribution has been observed by optical microscopy (Fig. 1).

Epoxy resin plates (i.e., without fibres) were also moulded following the curing cycles recommended by the manufacturer.

2.2. Atomic force microscopy measurements

A small probe (radius around 10 nm and height of 10 μm [41]) is fixed at the extremity of a cantilever beam. AFM measurements are dependent on the cantilever stiffness (between 10^{-2} and 10^2 N/m). During a force measurement, the sensitivity is increased when the bending of the cantilever is limited. The mechanical behaviour of the cantilever-probe can be roughly compared to the mass-spring system where k_{spring} and $k_{\text{cantilever}}$ are respectively the

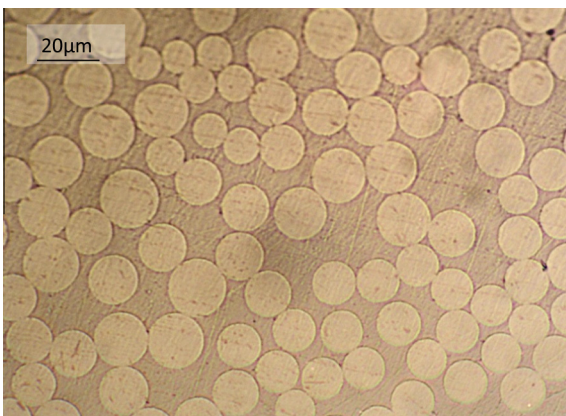


Fig. 1. UD composite with 50 vol.% glass fibres as observed.

stiffness of the spring and the cantilever. The deflection of the cantilever is measured by an optical method. A laser beam is focused on the extremity of the cantilever and is reflected on a dial photodiode. The displacement of the reflected signal allows to measure the cantilever deflection (Fig. 2).

Atomic force microscopy measurements in force mode are performed on a multimode Nanoscope V from Bruker AXS with a scanner type 8610 JVL. Tapping probes (type RTESP from Bruker) were chosen for force measurements because of their adequate spring constant range, between 40 N/m and 80 N/m, adapted to epoxy stiffness.

The system sensitivity and cantilever spring constant k_c are successively determined from force measurements on a rigid sample and from the thermal tune method [42], implemented in Bruker Nanoscope (V7.3) software.

From the slope of the force–displacement curve (Fig. 3) of our sample in linear elastic range, the apparent stiffness measured k_{eff} is linked to the stiffness of the sample k_s by [43]:

$$k_s = \frac{k_c \cdot k_{\text{eff}}}{k_c - k_{\text{eff}}} \quad (1)$$

In the case of a perfectly elastic tip with a spherical end and a homogeneous sample, with no adhesive effects, Hertz model can give an estimation of Young's modulus from force measurements [43,44]:

$$k_s = \frac{3}{2} a E_{\text{tot}} \quad \text{with} \quad a = \left(\frac{R \cdot F}{E_{\text{tot}}} \right)^{1/3} \quad (2)$$

where a is the tip-sample contact radius under a force F and E_{tot} is the reduced Young's modulus given by:

$$\frac{1}{E_{\text{tot}}} = \frac{3}{4} \left(\frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_t^2}{E_t} \right) \quad (3)$$

here ν_t , E_t , ν_s and E_s are the Poisson's ratio and the Young's moduli of tip and sample, respectively.

When testing rigid polymers, the tip is much stiffer than the sample, so that the former equation can be approximated by:

$$\frac{1}{E_{\text{tot}}} \cong \frac{3}{4} \left(\frac{1 - \nu_s^2}{E_s} \right) \quad (4)$$

and finally:

$$E_s \cong \frac{3}{4} (1 - \nu_s^2) \left(\frac{2k_s}{3} \right)^{3/2} \frac{1}{\sqrt{R \cdot F}} \quad (5)$$

The curvature radius of the tip R is estimated using Eq. (5) from force measurements on polymer samples with known modulus:

$$k_s = \frac{3}{4} a E_{\text{tot}} \quad k_s = \frac{3}{2} a E_{\text{tot}} \quad \text{with} \quad (6)$$

From local AFM measurements an effective Young modulus has been calculated at increasing distance from the fibre surface. Five measurements were performed on each point. To define the interphase area, the ratio between the local Young modulus and a reference Young modulus is represented: ratio = $E_{\text{calculated}}/E_{\text{reference}}$. $E_{\text{reference}}$ value is calculated from an arithmetic average of the matrix Young modulus values beyond 1 μm . The experimental results show a decrease of the effective Young modulus below 1 μm (the distance 0 is the localization of fibre/matrix interface, Fig. 4). A maximal drop around 60% from $E_{\text{reference}}$ is observed at 50 nm of the interface. The mechanical interphase area can be divided in two parts. Between 500 nm and 1 μm , the properties drop down to a plateau values (corresponding to a decrease of around 30% compared to $E_{\text{reference}}$). Between 50 nm and 500 nm from fibre surface, the values drop drastically (and linearly) down to a ratio of around 30%.

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