Polymer Testing 48 (2015) 160–168

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Material properties

# Effect of relative humidity on mechanical properties of a woven thermoplastic composite for automotive application

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ARTICLE INFO

*Article history:*
Received 9 September 2015
Accepted 15 October 2015
Available online 19 October 2015

*Keywords:*
Woven composites
Water ageing
Acoustic emission
Tensile behaviour

ABSTRACT

The purpose of this work is to characterise the influence of moisture content in a woven glass fibre reinforced polyamide 6,6 composite. Two different stacking sequences are studied: $[(0/90)_3]$ and $[(\pm45)_3]$ as well as the neat PA6,6 matrix. Samples have been conditioned through three ways: either water immersed, left at ambient temperature and humidity or dried in 35 °C oven. A one dimensional Fick's law has been used to model the water uptake in immersed samples. The glass transition temperature is highly affected by the presence of water and has been measured using modulated DSC technique. Finally, the effects of water on these composite materials have been investigated through tensile tests instrumented with acoustic emission monitoring (AE). Mechanical properties are highly affected by the presence of water in the composite. This result is even more visible on $[(\pm45)_3]$ sample because of the preferential loading of the matrix.

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## 1. Introduction

Glass-fibre-reinforced thermoplastic (GFRTP) are of interest for the automotive industry in the current context of $CO_2$ emission reduction. The development of this type of material is mainly due to their ease of design, light weight, low cost and good mechanical properties [1–3]. If reinforced with woven fabrics, these composite materials exhibit high stability of mechanical properties such as strength and stiffness in the weft and warp directions. This is not the only advantage over unidirectional composites, since woven fabrics make GFRTP more damage tolerant and impact resistant [4–7].

GFRTP have found many applications in different fields such as automotive, marine, aircraft and civil engineering [8,9]. This is not only instigated by their high strength-to-weight ratio, but also due to their capacity to be recycled [10]. Thus, many examples of woven GFRTP studies can be found in the literature with polypropylene [11], PET [12,13], polyurethane [14] and nylon [3,15–18] resins.

However these thermoplastic resins are sensitive to moisture absorption. The water absorption can lead to different types of degradation. The first is an increase in chain mobility (plasticizing effect), visible at the macro-scale through the decay of elastic modulus, the increase of elongation at break and the decrease of glass transition temperature [19]. Swelling has also been reported as well as matrix hydrolysis [20]. Arif et al. [3] have more precisely studied the water effect on short glass fibre reinforced PA6,6. They studied three moisture conditions: RH0, RH50 and RH100 which, respectively, correspond to the glassy, glass transition and rubbery states of the PA6,6. For this short fibre composite, it has been shown that damage mechanisms and chronology are highly influenced by moisture content.

The present work is focused on a woven GFRTP made with PA6,6 resin (referred as GFRPA6,6). To our knowledge, there is no publication about this material so far: only short-glass-fibre-reinforced PA6,6 have previously been reported [3,21,22].

This study characterises the behaviour of neat PA6,6 resin and GFRPA6,6 for three different ageing conditions: water immersed, left at ambient temperature and humidity or dried in 35 °C oven. Two composite layups have been studied, designated as $[(0/90)_3]$ and $[(\pm45)_3]$. The water intake was modelled by Fick's law and the mechanical behaviour of each type of samples was studied by performing tensile tests. Post-mortem failure modes were analysed through scanning electron microscopy (SEM) observations of the

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http://dx.doi.org/10.1016/j.polymertesting.2015.10.010
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fracture surface. In order to reach an extensive understanding of damage mechanisms, some tensile tests were also instrumented with acoustic emission monitoring.

## 2. Materials and methods

### 2.1. Tested material

The composite material studied is made of three plies of a 2/2 twill woven glass fabric impregnated with polyamide 6,6 resin (Fig. 1). The glass fibre fabric has a weight of 600 g/m$^2$ and a warp to weft ratio of 50/50. The fibre mass fraction ($m_f$) is equal to 0.63 and the void content is below 1%. The resulting composite plates are characterised by a density of 1.78 g/cm$^3$. The material was provided as plates of 1.53 mm thick, and rectangular coupons with dimensions of 200 mm × 20 mm × 1.53 mm were cut using a milling machine. The influence of fabric orientation on mechanical properties was studied using two different stacking sequences. The first, designated as $[(0/90)_3]$, had the warp direction of each ply oriented at 0° from the tensile axis (x axis). The second, designated as $[(\pm45)_3]$, had the warp direction of each ply oriented alternately at +45° and −45° from the tensile axis.

40 mm of each end of the specimen was gripped in the jaws of the test machine. The specimen edges were polished to remove mechanical damage caused by the cutting and 80 grit sand papers were used in the jaws to improve gripping.

Neat PA6,6 specimens were also been studied. They were made of the same grade as the resin of the GFRPA6,6 composite. The polymer material was provided as plates of 1.53 mm thick and dumbbell coupons were cut using a milling machine, following ISO 527-2. The overall length of the specimen was 100 ± 0.5 mm with a gauge length between the fillets of 40 ± 0.5 mm. The gauge width was equal to 7 ± 0.5 mm.

### 2.2. Ageing conditions

Two types of ageing have been studied for both neat resin and composite. Samples were either dried in an oven at 35 °C without humidity monitoring or immersed in 20 °C water. These two conditions are, respectively, referred to as “dry” state and “wet” state. The reference state is defined as ambient temperature and humidity (respectively, about 20 °C and 48 ± 5% RH). Samples were then tested at different ageing states.

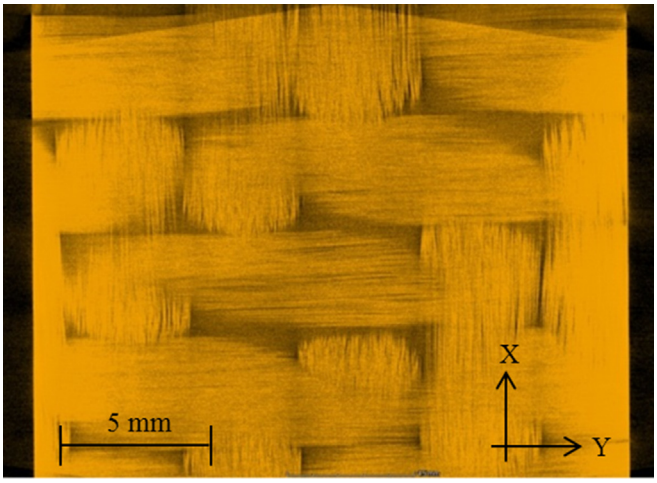


**Fig. 1.** Micro-tomography scan of $[(0/90)_3]$ specimen (resolution 18 μm).

### 2.3. Experimental procedure

#### 2.3.1. Measurement of water absorption

Water uptake was measured by weighing samples periodically at ambient temperature with a precision balance (sensitivity of 0.01 mg). Relative weight gain ($M_t$) is defined as given by Equation (1).

$$M_t = \frac{M(t) - M(t=0)}{M(t=0)} \times 100 \tag{1}$$

$M_t$ is then plotted versus the square root of time to allow data analysis.

#### 2.3.2. Data analysis

The water uptake kinetic of immersed samples has been studied using the one dimensional Fickian model. Thus, the weight gain can be expressed as follows [23]:

$$\frac{M_t}{M_\infty} = 1 - \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(\frac{-D(2n+1)^2\pi^2 t}{h^2}\right) \tag{2}$$

where $M_t$ is the relative weight gain during ageing, $M_\infty$ is the relative weight gain at equilibrium, D is the diffusion coefficient, t is the time and h is the thickness of the sample. According to [23], Equation (2) can be approximated by the expression (3):

$$\frac{M_t}{M_\infty} = 1 - \exp\left(-7.3\left(\frac{Dt}{h^2}\right)^{0.75}\right) \tag{3}$$

where the diffusion coefficient can be evaluated using the initial slope (k) of the $M_t$ versus $\sqrt{t}$ experimental curve:

$$D = \pi\left(\frac{hk}{4M_\infty}\right)^2 \tag{4}$$

#### 2.3.3. DSC measurements

Polyamide 6,6 glass transition temperature has been determined using a differential scanning calorimeter (DSC) Q20 from *TA Instruments* on pure resin samples. The glass transition temperature is hard to detect, hence modulated DSC (M-DSC) was used in this study. Two aluminium pans were used, the first was empty and used as the reference while the second was filled with the matrix sample (between 5 mg and 10 mg of material was used). The method used was as follows:

1. Equilibrate at −50 °C;
2. Modulate ±1.272 °C every 60 s;
3. Isothermal for 5.0 min;
4. Ramp 2 °C/min to 280 °C.

Modulation amplitude was chosen according to the modulation period and the heating rate in order to stay in “heat only” conditions. In these conditions, the modulation does not imply any cooling phenomenon during the test.

#### 2.3.4. Mechanical testing

Quasi static tensile tests were performed using an INSTRON 4505 electromechanical machine with a cross-head speed of 1 mm/min, strain being measured by a 25 mm gauge length extensometer. In order to obtain detailed analysis of the damage process, some tests were instrumented with acoustic emission (AE) monitoring. The acoustic emission monitoring was performed by using an AE

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