EXPERIMENT AND MODELLING OF INJECTED CARBON-FIBRE-REINFORCED-PEEK BEHAVIOUR UNDER TENSILE LOADING

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Abstract
Variations of injection-moulding parameters have a great impact on mechanical and damaging properties of short-fibre reinforced thermoplastics. The aim of the study is to predict the behaviour and damaging of an injected specimen under monotonic quasi-static tensile loading depending on injection parameters thanks to a design of experiments carried out on two different commercial 40%-wt carbon-fibre-filled PEEK®. Static tensile tests enable to determine the damageable elastic behaviour of the composite. A Scanning Electron Microscopy analysis reveals two main damage modes: matrix cracking and debonding. A mean-field homogenization-based model (Mori-Tanaka) featuring elastoplastic matrix and elastic fibres was developed, including damageable matrix/fibre interface.

1 Introduction

Composite materials are of main interest in transport fields such as in aeronautic manufacturing [1] due to their good strength-to-density ratio. They are composed of a matrix and reinforcing parts (mostly fibres). PEEK (poly-ether-ether-ketone) has very good mechanical and chemical properties [2]. Moreover, it has a glass transition temperature of 143°C and a melting temperature of 393°C that enables it to be used at high temperature [3]. Carbon fibres exhibit good compromise between density and strength. Comparing to unreinforced matrix, short-fibre-thermoplastics (SFT) show an improvement in stiffness and strength and a reduction in the ductility related to fibre volume fraction [2]. These materials are commonly used by automotive industry because of the capacity of the process (reliability, speed, cost).

2 Experiments

2.1 Materials and processing

2.1.1 Materials

Two commercial grades of carbon-fibre-reinforced-PEEK were used: 90HMF40 provided by Victrex® and LarPeek 10K/40HM provided by Lati®. Both materials feature 40-wt% of short carbon fibres (high modulus carbon fibres with mean length of 200μm) and a similar PEEK grade. Tests specimen corresponding to ASTM D3641 - 10a [4], have been moulded with a DK 65/160 injection –moulding press.

2.1.1 Design of experiments

In order to understand the influence of processing parameters on mechanical and damaging properties, a L9 Taguchi table was used [5]. It enabled to vary four parameters on three levels. Each parameters were chosen according to physical limits and previous works [6,7,8]. Finally, the mould temperature (T_{mould}) was chosen to be above the PEEK glass transition temperature and under the PEEK degradation temperature 400°C [9]; the holding pressure (P_{hold}) was chosen in order to avoid shrink (low holding pressure) and flash (high holding pressure), the holding time (t_{hold}) (time applying holding pressure) and cooling time (t_{cool}) (time from the end of holding pressure to part ejection) were also chosen in order to avoid cold slug. All parameters levels are gathered on Table 1. For T_{mould} the levels are those achieved during manufacturing. Temperature of the hopper was 50°C, barrel profile temperature was 385°C-390°C-395°C, and the nozzle temperature was 405°C for all experiments.

Injection parameters	**Low level**	**Medium level**	**High level**
T_{mould} (°C)	178	188	203
P_{hold} (MPa)	60	70	80
t_{hold} (s)	6	9	12
t_{cool} (s)	35	55	80

Table 1. Levels of injection-moulding parameters for the design of experiments.

Thanks to a multiple linear regression, it is able to have access to parameters effects on different results obtained from tests, as Young modulus, tensile strength, fracture strain, yield stress and strain.

2.2 Quasi-static monotonic tensile tests

Tensile tests were carried out on a MTS 810 (Figure 1) with hydraulic grips respecting NF ISO 527-2 norm [10], with a crosshead speed of 5mm/min (≈ $2e^{-3}s^{-1}$).

Figure 1. Photograph of MTS 810

2.3 Damaging characterization

Observations of fracture surfaces were made on a XL30 ESEM Tungsten (Figure 2).

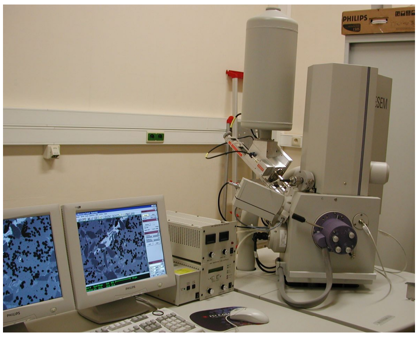

Figure 2. Photograph of XL30 ESEM Tungsten

3 Modelling

3.1 Mean-field homogenization: Mori-Tanaka's model

The homogenization scheme chosen to predict the mechanical behaviour of the carbon-fibre-reinforced-PEEK is the Mori-Tanaka's model firstly developed by Mori and Tanaka [11] and improved by Benveniste [12]. It is based, as major all mean-field homogenization scheme, on Eshelby's inclusion problem [13], that relates the accommodation of an inclusion in a matrix. The theory considers an inclusion (fibre) embedded in the matrix. Thus, it is an explicit model which equation for the stiffness tensor (L_{MT}) is given by the equation (1). The resolution of the equation can be made by summing effects of all phases:

$$L_{MT} = L_m \left[I + \langle A_r \rangle \left(I + \langle (E_r - I) A_r \rangle^{-1} \right) \right]^{-1} \tag{1}$$

where $A_r = -\left[(L_r - L_m) E_r + L_m \right]^{-1} (L_r - L_m)$, $\langle X_r \rangle = \sum_{r=1}^{N} v_r X_r$ is the volume average operator over the N phases, v_r is the volume fraction of the phase r, L_r is the stiffness tensor of the phase r, E_r is the Eshelby tensor of the phase r depending on its aspect ratio (length/diameter) and matrix stiffness tensor.

3.2 Nonlinear behaviour: elastoplastic phase

In order to take account of the nonlinear behaviour of the matrix (elastoplasticity) in the homogenization scheme, it is necessary to linearize the stiffness tensor. It can be calculated by different method described by Rekik [14], the one chosen for the proposed model is the modified secant method detailed by Suquet [15]. The principle is that the secant stiffness tensor depends on the second-order average of the local equivalent strain of the matrix $L_m^{sct} = L_m^{sct}\left(\bar{\bar{\varepsilon}}_{eq}^{m} \right)$, it is expressed in the equation (2). This extension of the secant method coincides with the variational approach of Ponte-Castañeda [15,16] (upper bound for the effective energy).

$$\bar{\bar{\varepsilon}}_{eq}^{m} = \sqrt{\frac{1}{3 v_m} \varepsilon_{ij} \frac{\partial L_{ijkl}^{SC}}{\partial \mu_m} \varepsilon_{kl}} \tag{2}$$

where μ_m is the shear modulus of the matrix.

The plastic matrix follows a Ramberg-Osgood law of plasticity whose parameters are the yield stress σ_0, yield strain ε_0 and the plastic exponent α. Equation (3) is the relation between matrix Von Mises equivalent stress and strain:

$$\sigma_{eq}^m = \frac{\sigma_0}{\varepsilon_0}\left(\frac{\varepsilon_{eq}^m}{\varepsilon_0}\right)^\alpha \tag{3}$$

where $\varepsilon_{eq}^m = \sqrt{\frac{3}{2} e_{ij}^m e_{ij}^m}$, $e_{ij}^m = \varepsilon_{ij}^m - \delta_{ij}\frac{\varepsilon_{kk}^m}{3}$, $\sigma_{eq}^m = \sqrt{\frac{3}{2} s_{ij}^m s_{ij}^m}$, $s_{ij}^m = \sigma_{ij}^m - \delta_{ij}\frac{\sigma_{kk}^m}{3}$, δ_{ij} being the Kronecker delta.

3.3 Damage criteria

Considering elastic fibres and elastoplastic matrix, it was obvious to model fibre/matrix interface damage. This micromechanical damage depends on fibre orientation according to load orientation. Fitoussi determined a quadratic tridimensional failure criterion for discontinuous-reinforced composite [17] (equation (4)). It requires the identification of two parameters: σ_c and τ_c, the normal and shear failure stresses of the interface. Thus, the criterion has to be calculated on the diameter of the interface to take into account the anisotropy of the damage. A damage variable D can be calculated as the percentage of achieved criteria over angle decomposition of the interface.

$$\left(\frac{\sigma}{\sigma_c}\right)^2 + \left(\frac{\tau}{\tau_c}\right)^2 = 1 \tag{4}$$

4 Results and Discussions

4.1 Quasi-static global tensile behaviour

The two grades do not exhibit the same behaviour against quasi-static tensile load, as it is shown in Figure 3. Analysis of tensile tests gave Young modulus (E), fracture strength (σ_r) and strain (ε_r), mean values are gathered in Table 2.

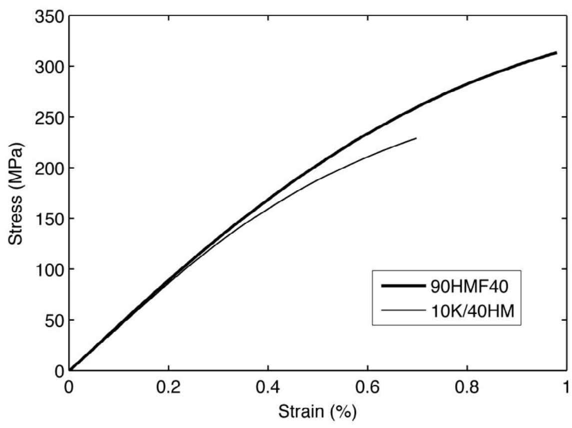

Figure 3. Results of quasi-static tensile tests on 90HMF40 and 10K/40HM.

Material properties	90HMF40	10K/40HM
E (MPa)	43450	43320
σ_r (MPa)	317	235
ε_r (%)	1.0	0.7

Table 2. Tensile tests analysis for 90HMF40 and 10K/40HM.

2.2 Design of experiment analysis

Thanks to a multilinear regression, effects of injection parameters were determined (related to their levels). Table 3 shows the effects of each injection parameters on tensile properties of both materials.

Injection parameters	90HMF40	10K/40HM
T_{mould}	E = / $\sigma_r \nearrow$ / $\varepsilon_r \nearrow$	E = / $\sigma_r \nearrow$ / $\varepsilon_r \nearrow$
P_{hold}	No effects	No effects
t_{hold}	No effects	E = / $\sigma_r \nearrow$ / ε_r =
t_{cool}	No effects	E = / $\sigma_r \nearrow$ / $\varepsilon_r \nearrow$

Table 3. Injection parameters effects on tensile properties for 90HMF40 and 10K/40HM.

According to injection parameters chosen levels, holding pressure does not have a significant effect on tensile properties for both materials whereas, mould temperature is the most significant parameter, affecting failure stress and strain only. Comparing to 90HMF40, both temperature, holding and cooling, have similar lower effects on 10K/40HM.

2.3 Nonlinear behaviour: damage and plasticity

In order to understand the damaging behaviour of both materials, SEM microscopy were done, (Figure 4). Two different mechanisms are observable: debonding (a) and matrix plasticity (b). Those two pictures were taken on the same specimen fracture surface but at different width. It has been seen that in the middle of tests specimen, debonding is the main damage, whereas near surfaces, matrix plasticity is more present. Effectively, PEEK transcrystallinity (growth of crystallinity perpendicularly to fibre interface), has a significant effect on damage behaviour, as observed by Friedrich and Kim [18,19]. This crystallinity is highly influenced by cooling rates (thermal history), which itself affects mechanical properties of matrix/fibre interface. It can explain why mould temperature has a very significant effect on fracture strength and strain. The higher the mould temperature is, the lower cooling rate is, and so, there is a stiffer matrix/fibre interface.

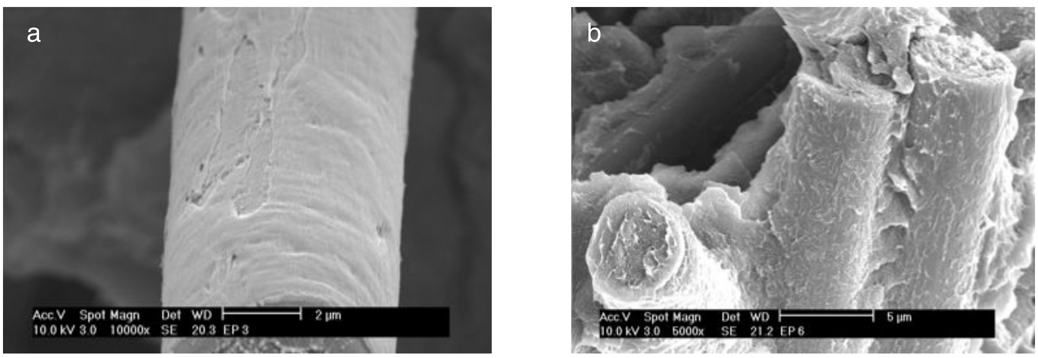

Figure 4. SEM microscopy of fracture surfaces of 90HMF40 (debonding (a), matrix plasticity (b)).

That is why the model features a damage criteria and an elastoplastic behaviour for the matrix.

2.4 Model

Firstly, Young modulus is well predicted by Mori-Tanaka's model using parameters gathered in Table 4. Where l_f and r_f are respectively fibre length and radius.

Material	**Matrix**		**Fibre**			
Parameters	E (GPa)	ν	E (GPa)	ν	l_f (m)	r_f (m)
Parameters Value	3.8	0.325	450	0.27	$200.e^{-6}$	$5.e^{-6}$

Table 4. Model parameters input for Mori-Tanaka's model.

Secondly, the non-linear behaviour induced by both plasticity and damage needs a good understanding of the material. Tensile tests with releases at different stress levels can give essential information of damage and plasticity law of evolution (Figure 5-a).

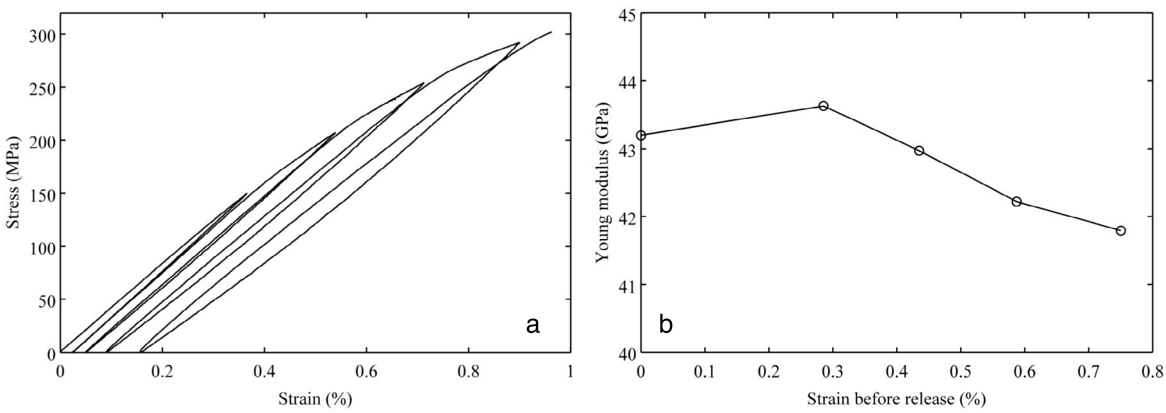

Figure 5. Tensile release tests for 90HMF40 (a), evolution of Young modulus of loading after releases at different strain value (b).

Plasticity seems to appear before damaging phenomena, as loading slope is altered in the last cycle (Figure 5-b). Thus, it is convenient to take into account this effect on damage criteria and plasticity parameters. The proposed model features a macro plasticity law of evolution, for simplicity purpose. The plasticity function is calculated after applying damage criteria on elements and actualizes elastic and plastic strains. An exponent law of hardening, based on experimental data, has been chosen in order to model plasticity.

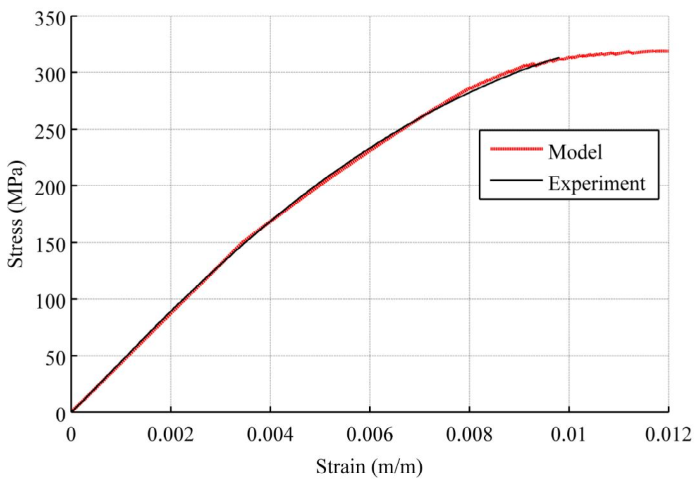

Figure 6. Experimental and modelling of quasi-static tensile test for 90HMF40

Finally, the proposed model has a good prediction (Figure 6), but the fracture has not been taken into account yet.

5 Conclusions

Thanks to a design of experiments, it has been possible to determine process parameter effects on microstructure, thus, on mechanical properties. Tensile tests and interrupted tensile tests enabled to understand the scenario of damaging in the material. The nonlinear behaviour is due to debonding (interface failure) and matrix plasticity. A micromechanical model based on Mori-Tanaka's theory was developed featuring micro-damage and global plasticity and gives a good prediction for quasi-static tensile tests. An extension to interrupted tensile tests is necessary to check the model accuracy.

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