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EXPERIMENTS AND ANALYSES FOR DETERMINING FIBRE/MATRIX INTERFACE PARAMETERS – UNDERSTANDING DEBONDING PROBLEMS

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ABSTRACT

A new experimental technique is developed to monitor the initiation and propagation of a debond crack during a fibre pull-out experiment. The advanced experimental setup consists of a high resolution video camera and a laser extensometer mounted at a tensile test machine. The test setup enables the measurement of the fibre/matrix displacement and debond length. A micromechanical model is used for analysing the experimental data. It allows the evaluation of the following parameters: the interface debond energy G_{IIc} and the frictional sliding shear stress τ_s at the fibre/matrix interface, and the misfit strain $\Delta\varepsilon^T$, accounting for initial residual stresses. Specimens of a single steel fibre embedded centrally in a polyester matrix are tested using the experimental setup and the model. A practical experimental procedure for establishing the interface parameters is suggested, and an example demonstrates the procedure and yields a set of interface parameters.

1. INTRODUCTION

Interfacial properties of a composite are of utmost importance for an understanding of composites overall properties. The stress transfer between fibre and matrix occurs through the fibre/matrix interface. To characterize and understand the interface phenomena, several techniques are available like single fibre fragmentation test, fibre peel test, single fibre pull out test and micro indentation test. Out of these, single fibre pull out test is one of the best suited for the study of composite interfacial behavior. The two most important mechanisms that take place are interfacial debonding and frictional sliding along the debonded interface. The parameters that control these mechanisms are the interfacial sliding shear stress, the debond energy and residual stresses for any fibre/matrix composite system.

Marshall (1992) used the advanced shear-lag models derived by Hutchinson and Jensen (1990) for evaluating the interface properties for ceramic matrix composites experimentally. Kharrat et al. (2006) used an analytical model based on shear lag analysis (for elastic load transfer) for a stainless steel/epoxy system. The shear lag model is effective as long as the fibre has not debonded, after complete debonding the fibre is extracted against interfacial friction. The SiC-Glass system was used by Kerans and Parthasarathy (1991) to extract the interface parameters such as frictional resistance, residual axial strain considering the Poisson's effect. They found that after debond initiation the curve of load versus fibre/matrix displacement show nonlinear behaviour, and unstable failure occurs when the debond initiation load is high or when residual stress is low. Levasseur et al. (1997) obtained the interfacial parameters of a SiC/pyrex composite from pull out tests.

This paper targets the latest advancements made in the fibre pull out process to measure debond lengths and fibre/matrix displacements in a steel/polyester system. The procedure defined in this study aims to determine the interface parameters and to evaluate the mechanical effects of surface coatings applied on fibre surface. This evaluation procedure has been derived from the basic model of Hutchinson and Jensen (1990), applying the fibre debond-length measurements and correlating the analytical results with the experimental data. The calculation of the frictional shear stress is done using the load versus debond length measurements. The data obtained can be used further to calculate the residual stresses and the debond energy G_{IIc} (fracture toughness).

2. MATERIALS AND MANUFACTURING

The steel fibres used in the current research study are high carbon steel fibres with a fibre diameter of 0.21mm. The surface coating on the steel fibre is zinc layer (to protect from corrosion). The fibre surfaces are otherwise untreated (no sizings). The fibre was cleaned with acetone before infusing the polyester resin. The fibre was embedded in a thermoset polymer, a standard polyester resin (Polylite 400-499). Test specimens designed for pullout experiments have a matrix block with dimensions 100 mm x 5 mm x 5mm. A silicone mould was designed having a cavity 5 mm x 5 mm with a pin hole to place the steel fibre exactly at the centre line of the cavity. The test setup for processing the specimens is shown in Fig. 1.

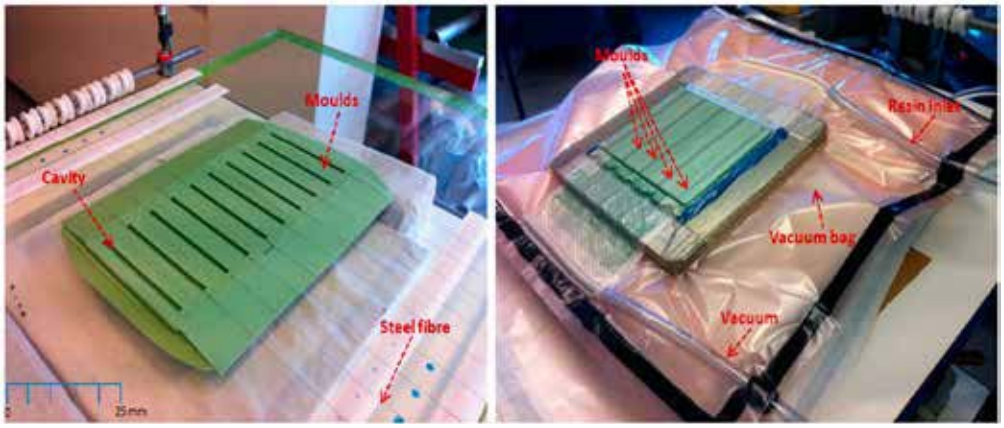

Fig. 1. Processing setups for pull-out specimens: placement of fibre in the mould and closed silicone moulds with vacuum bagging.

Specimens were made using vacuum infusion in order to obtain high quality specimens for pullout test avoiding processing issues like bubbles, voids or shrinkage problems. The silicone mould has cavities for producing 10 samples. In order to obtain perfectly placed steel fibre at centre of the specimen, a weight of 200 grams is used to preload the steel fibre after the fibre has been placed at the centre of the mould cavity. This ensures steel fibre placement and straightness of the fibre after processing. After demoulding, the specimens are inspected for bubbles and damages in the fibres. Finally the specimens are post cured in an oven at 60℃ for 24 hours. This is to ensure total cure of the matrix material.

3. EXPERIMENTAL SETUP

3.1 State of the art – pull-out test: The test method chosen to characterize the fibre/matrix interface is single fibre pullout using fixed bottom gripping as shown in Fig. 2. The testing method was chosen to fit to the boundary conditions of the advanced micromechanical models of Hutchinson and Jensen (1990).

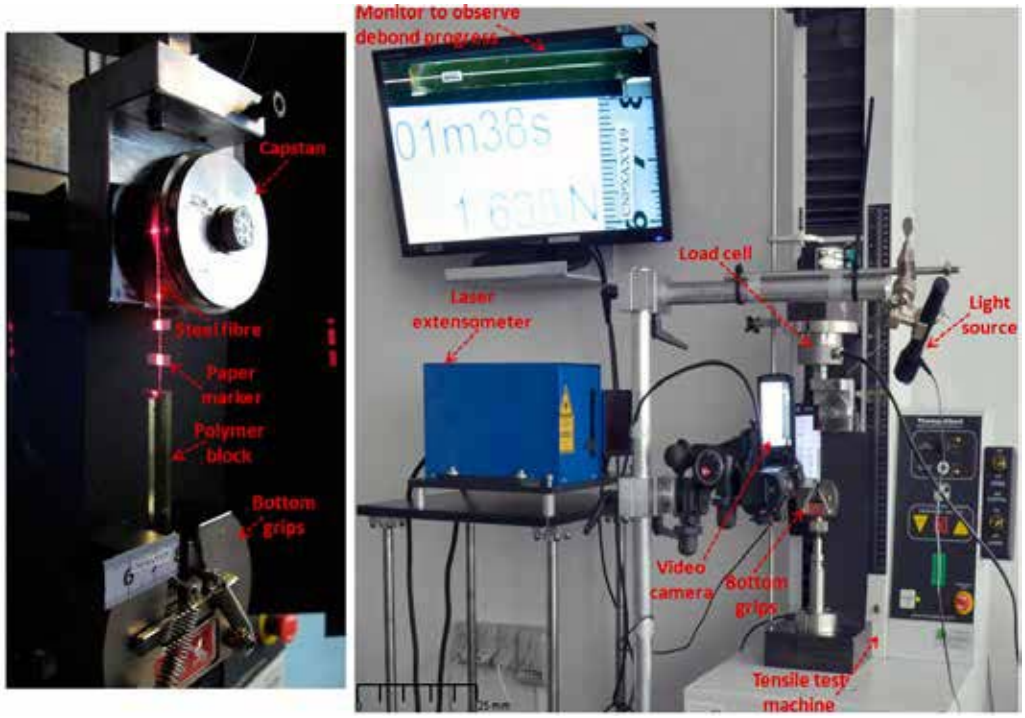

Fig. 2. Pull-out test – Advanced experimental setup.

3.2 Advanced experimental setup: The polyester block is gripped from the bottom and the fibre is pulled using a circular capstan having a radius 25 mm at the top. A laser extensometer (P-50, Fiedler Optoelektronik GmbH) is used for measuring fibre/matrix displacements. A video camera (Panasonic, HDC–TM 900) is used to record the interface crack initiation and progression while loading the specimen as shown in Figure 2. The resolution of the image is 1920 x 1080 pixels, and images were recorded with 50 frames per second. The target is to measure the debond lengths from the video clips. Tests are performed at a displacement rate of 1

mm/min with a data sampling rate of 10 Hz. The experiments are terminated, when the debond length approaches the clamped end (i.e. within 10mm from the bottom grip).

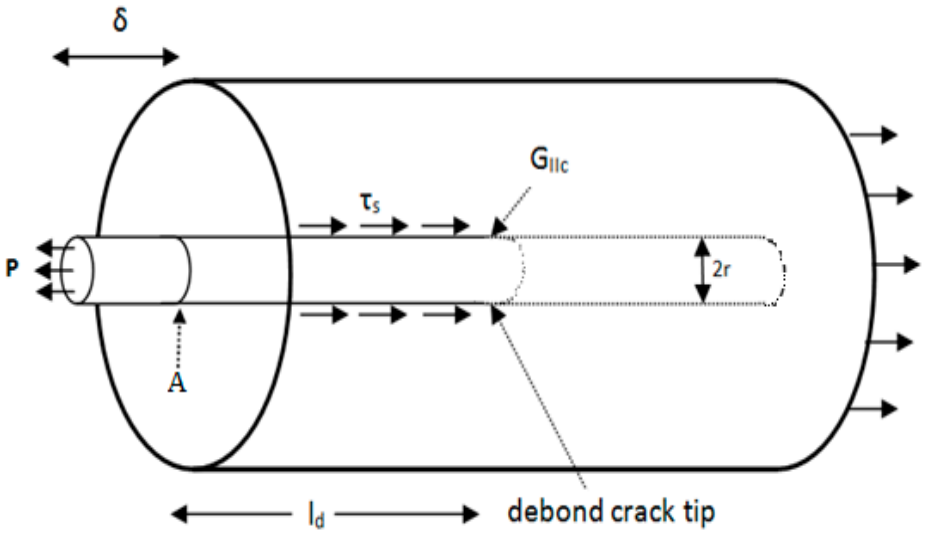

Fig. 3. The model for a fibre in a block of matrix, with debond and pull-out conditions. The parameters recorded during the pullout experiment are the applied force, P, the fibre/matrix displacement δ, and the debond length, l_d.

4. MODEL FOR DEBOND AND PULL-OUT

The experiments are designed to correlate closely with the model for the single fibre debond and pull-out mechanism, the model and analysis is based on the theoretical work of Hutchinson and Jensen (1990).

The material system parameters which characterize the basic materials and geometry are: fibre stiffness E_f, matrix stiffness E_m, fibre radius r, specimen cross sectional area A_c, fibre volume fraction V_f, matrix volume fraction V_m, and composite stiffness E_c. The composite stiffness is calculated from the law of mixtures for unidirectional composites.

The parameters which characterize the debond and pull-out mechanism are the misfit strain $\Delta\varepsilon^T$, accounting for initial residual stresses, the mode II interface debond energy G_{IIc}, and the frictional sliding shear stress τ_s acting along the fibre/matrix interface after debonding.

4.1 Model geometry: The model constitutes a single cylindrical fibre in a block of matrix, and is axisymmetric with respect to the fibre axis, see Fig. 3. The fibre sticks out at one end of the specimen, such that load can be applied to the fibre. At the other end of the specimen the matrix (composite) is held by a conventional gripping system, as shown in Fig. 2.

4.2 Experimental aspects: The experiment is carried out by applying the load axially along the fibre at a constant loading rate. The steps during the experiment are the following: (i) elastic loading of the composite (fibre and matrix), (ii) at load P_o initiation of a mode II debond crack occurs at position A, and controlled by debond energy G_{IIc}, (iii) displacement δ increases with

load P, and debond length l_d increases with load P. The analysis of experimental data are the following: (i) the debond lengths l_d as a function of load P are measured from the video recordings, (ii) the relative displacements between fibre and matrix δ as a function of load P are calculated from the laser extensometer recordings.

4.3 Analysis of data: The procedure for deriving the (three) parameters τ_s, $\Delta\varepsilon^T$, and G_{IIc} is based on equations of the micromechanical model.

1) The relation between load P and debond length l_d is:

$$P = \left(2\pi\, r \tau_s \frac{E_c}{V_m E_m} \right) l_d \;+\; 2\pi\, r \sqrt{\left(r\, G_{IIc} \frac{E_f E_c}{V_m E_m} \right)} \;-\; \pi\, r^2 E_f \Delta\varepsilon^T \tag{1}$$

According to the micromechanical model P is linearly related to l_d. The slope of a P versus l_d curve (dP/dl_d) is only dependent on τ_s and thus allows evaluation of τ_s.

2) The equation for $\Delta\varepsilon^T$ is related to both the displacement δ and the debond length l_d and τ_s :

$$\Delta\varepsilon^T = \frac{\delta}{l_d} \;+\; \frac{\tau_s}{r} \frac{E_c}{E_f V_m E_m}\; l_d \;-\; \frac{1}{E_f} \frac{P}{\pi r^2} \tag{2}$$

Knowing τ_s from the first analysis step, the above equation is used to calculate $\Delta\varepsilon^T$ for combined sets of δ, l_d and P (all known experimental data), and to plot $\Delta\varepsilon^T$ versus P. This should give a constant value for $\Delta\varepsilon^T$.

3) The final step is then to use $\Delta\varepsilon^T$ together with P_o, in the equation for G_{IIc}:

$$G_{IIc} \;=\; \left(\frac{P_0}{\pi\, r^2 E_f} + \Delta\varepsilon^T \right)^2 \frac{r\, E_f V_m E_m}{4 E_c} \tag{3}$$

4) Finally, the experimentally determined values for τ_s, $\Delta\varepsilon^T$, and G_{IIc} are used in Eq. (4) to calculate the curve of load P versus displacement δ, for comparison with the experimental curve of P versus δ. The relation between the load P and the displacement δ is:

$$P = \pi\, r^2 E_f \sqrt{\left(\delta\; \frac{4\, \tau_s E_c}{r\, E_f V_m E_m} \;+\; \frac{4\, G_{IIc}}{r} \frac{E_c}{E_f V_m E_m} \right)} \;-\; \pi\, r^2 E_f \Delta\varepsilon^T \tag{4}$$

Eq. (4) shows that P is expected to be related to the fibre/matrix displacement δ, with a nearly square-root dependence (i.e. $P \sim \sqrt{\delta}$).

5. AN EXPERIMENTAL EXAMPLE

To illustrate the evaluation procedure, one experiment is used in the following paragraphs. The basic material system parameters are listed in Table 1. Fig. 4 shows a plot of load P versus debond length l_d, and the slope of the line yields a value for τ_s using Eq. (1). Fig. 5 shows a plot of $\Delta\varepsilon^T$ versus load P calculated by Eq. (2) (using the determined value of τ_s and simultaneous values of δ, P and l_d); this yields a (nearly) constant value for $\Delta\varepsilon^T$. The initial debond load P_o is obtained by extrapolation of the P-l_d relationship to $l_d = 0$ as shown in Fig. 4. Having determined

τ_s, $\Delta\varepsilon^T$, and P_0, the debond energy G_{IIc} is then calculated from Eq. (3). Fig. 6 is the plot of load P versus displacement δ, based on Eq. (4) using the determined the interface parameters. This curve is compared with the direct experimental curve of P versus δ.

The procedure seems to be operational, and gives reasonable results, although at present the actual values for τ_s, $\Delta\varepsilon^T$, and G_{IIc} cannot be evaluated against independent measurements or other data, nor against more experiments which will give a statistical qualification of the values.

Table 1. Material and interface parameters – (steel fibre/polyester system, CNPXAXV08).

Material parameters			
Fibre stiffness	E_f	GPa	210
Matrix stiffness	E_m	GPa	3
Fibre radius	r	mm	0.155
Specimen area	A_c	mm^2	25
Fibre volume fraction	V_f		0.003
Matrix volume fraction	V_m		0.997
Composite stiffness	E_c	GPa	3.625
Interface parameters			
Shear stress	τ_s	MPa	1.24
Misfit strain	$\Delta\varepsilon^T$		0.0029
Initial load	P_0	N	13
Debond energy	G_{IIc}	J/m^2	93

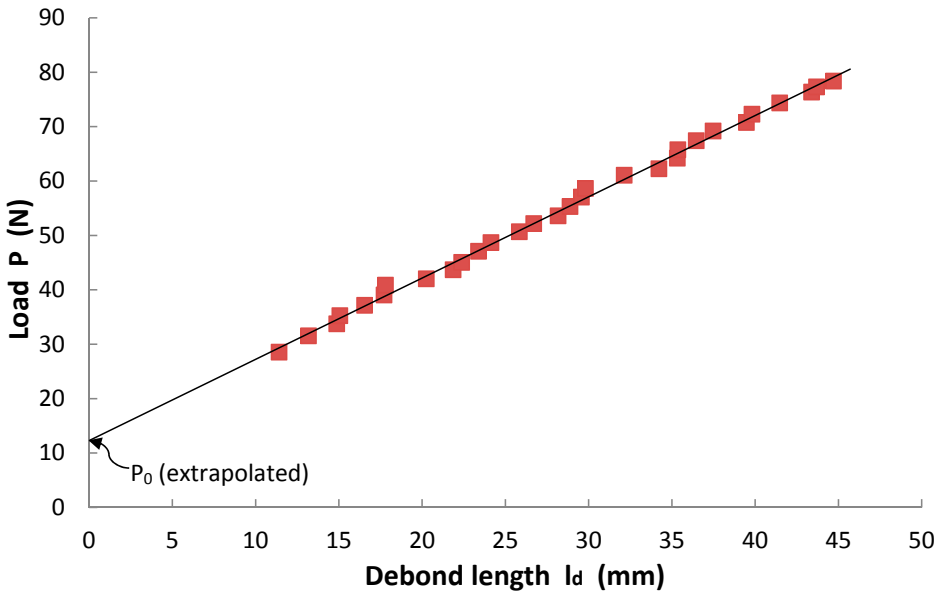

Fig. 4. Load as a function of debond length; the slope is used to evaluate interfacial frictional sliding shear stress τ_s, and the intersection with the y-axis P_o is used in the calculation of G_{IIc}.

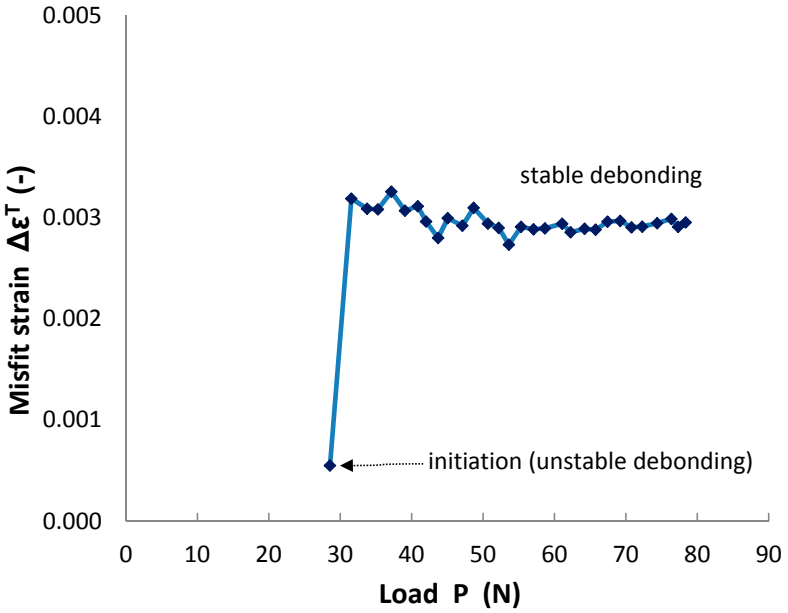

Fig. 5. Misfit strain as a function of load, the (nearly) constant value establishes $\Delta\varepsilon^T$ for the material system.

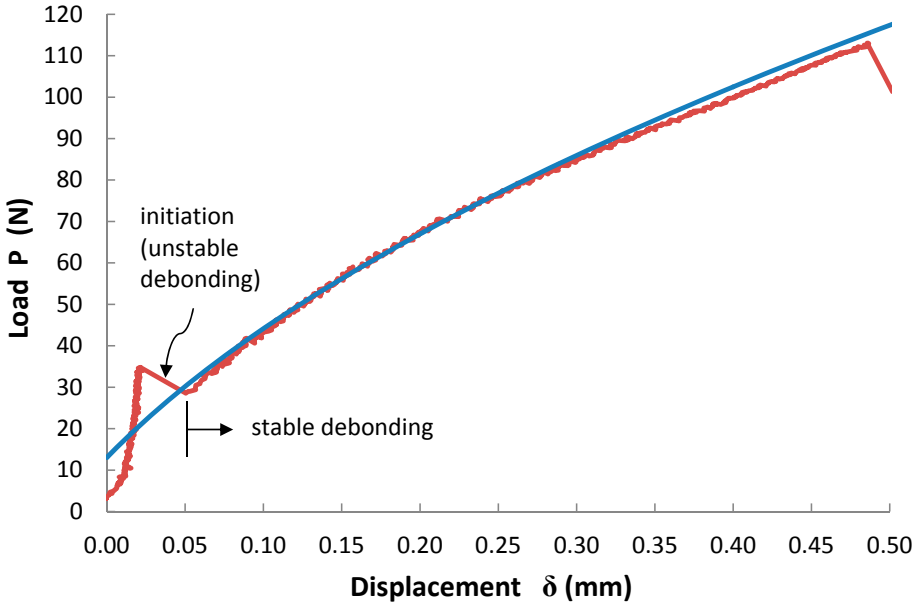

Fig. 6. Load as a function of displacement, the blue curve is calculated on the basis of experimental values for τ_s, $\Delta\varepsilon^T$, and G_{IIc}, and the red curve is based on the experimental data from the laser extensometer.

6. SUMMARY AND CONCLUSIONS

A new experimental technique is developed to monitor the initiation and propagation of a debond crack during a fibre pull-out experiment. The advanced experimental setup consists of a high resolution video camera and a laser extensometer. The test setup enables the measurement of the fibre/matrix displacements and the debond length. A micromechanical model is used for analyzing the experimental data. It allows the evaluation of the following interface parameters: the misfit strain $\Delta\varepsilon^T$, the interface debond energy G_{IIc} for mode II cracking, and the frictional sliding shear stress τ_s acting along the fibre/matrix interface during the debonding. Specimens of a single steel fibre embedded centrally in a polyester matrix are used to demonstrate the experimental setup and the use of the micromechanical model. A practical experimental procedure for establishing the interface parameters is suggested, and an example demonstrates the procedure and yields a set of parameters. At present, the validity of these results cannot be established.

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