Non-destructive characterization of fibre–matrix adhesion in composites by vibration damping

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Adhesion at the fibre–matrix interface in fibre-reinforced composites plays an important role in controlling the mechanical properties and overall performance of composites. Among the many available tests applicable to the composite interfaces, the vibration damping technique has the advantages of being non-destructive as well as highly sensitive. An optical system was set up to measure the damping tangent delta of a cantilever beam, and the damping data in glass fibre-reinforced epoxy-resin composites were correlated with transverse tensile strength which are also a qualitative measurement of adhesion at the fibre–matrix interface. Four different composite systems containing three different glass fibre surface treatments were tested and compared. Our experimental results showed an inverse relationship between damping contributed by the interface and composite transverse tensile strength.

1. Introduction

It is well known that the fibre–matrix interfacial adhesion has a major effect in achieving superior mechanical properties of a composite. The tensile strength of the composite is dependent on the ability of the composite to transfer the tensile load from the broken fibres to the surviving ones through shear in the matrix and at the interface. Thus, a method that is capable of determining the interfacial adhesion strength is needed to evaluate the mechanical performance of composite materials.

Numerous experimental techniques have been developed for measuring interfacial adhesion strength in fibre-reinforced composites. These methods include the single-fibre pull-out test [1, 2], microbond test [3–5], the single-fibre fragmentation test [6–9], the microindentation test [10, 11], and some non-destructive evaluation techniques, such as vibration damping [12]. Vibration damping is a promising non-destructive technique because it is simple and quite sensitive to the interfacial region. The method has a potential to be used by the materials industry for *in situ* monitoring of the mechanical performance of composites.

According to the theory of energy dissipation [13], the quality of the interfacial adhesion in composites can be evaluated by measuring that part of energy dissipation contributed by the interfaces; the interface part can be obtained by separating the fibre and matrix from the total composites. Zorowski and Murayama [12] were the first to develop a method for the quality of the interfacial adhesion in the reinforced rubber through energy dissipation measurements based upon the following relationship

$$\tan \delta_{in} = \tan \delta_{comp} - \tan \delta_s \tag{1a}$$

$$\tan \delta_s = \frac{\tan \delta_f E_f V_f + \tan \delta_m E_m V_m}{E_m V_m + E_f V_f} \tag{1b}$$

where $\tan \delta_{in}$ is the internal energy dissipation due to poor adhesion from the interface, which can be used for evaluating the interfacial adhesion; $\tan \delta_s$ is the effective loss tangent for a composite with perfect interfacial adhesion, $\tan \delta_{comp}$ is the measured internal energy dissipation of the composite system. E is the Young's modulus and V represents volume fraction. Subscripts f and m refer to the fibre and matrix, respectively. By measuring the total system energy dissipation in terms of $\tan \delta$, and knowing $\tan \delta$ and the dynamic moduli of the components, as well as the volume fraction of fibres, the dissipation due to the poor interfacial adhesion can be determined.

In this paper, we describe an optical system for measuring vibration damping of a cantilevers specimen. The system was used to measure the damping of glass fibre-reinforced polymer composites, and the damping data were correlated with the transverse tensile strength of composites.

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TABLE I A list of the samples used in this study

Specimen type	Fibre volume fraction	Description of surface treatment
A	0.610	Untreated fibres
B	0.677	366 size without silane
C	0.695	366 size with silane
D	0.716	158B size with silane

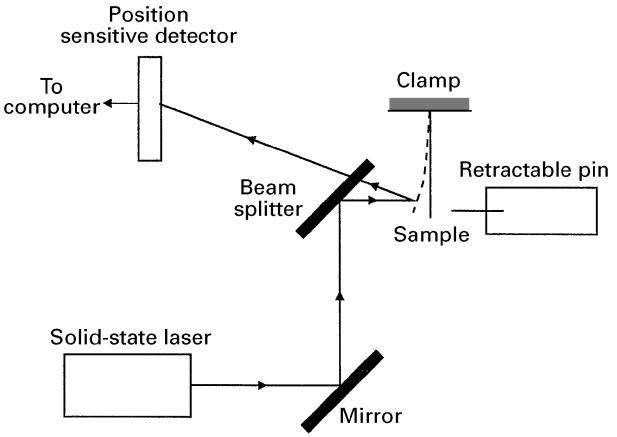

Figure 1 Schematic diagram of the optical system.

2. Experimental procedure

2.1. Composite sample preparation

Unidirectional composite laminate specimens were fabricated at the Owens-Corning Science and Technology Center. D. E. R. 331 epoxy resin (Dow Chemicals Company) and Lindride 66 curing agent (Lindau Chemicals Inc.) were selected as the matrix material commonly used in a filament winding process. The reinforcements were E-glass fibres with a diameter of 16 μm. Four fibre systems that contained different surface treatments were investigated in this study, as listed in Table I. To make a composite laminate, D.E.R. 331 epoxy (100 parts by weight) was mixed with Lindride 66, curing agent (85 parts by weight). Composite laminates were fabricated through a filament winding machine and samples were cured for 2 h at 120 °C and 2 h at 180 °C under a hot-press machine with a 1.43 MPa constant pressure. The composite laminates were then cut into 30 mm × 4 mm × 0.5 mm specimens. The actual length of the cantilever beam was 25 mm.

2.2. Optical system

A schematic illustration of the optical equipment designed to measure the deflection and vibration dynamics of a cantilever beam is shown in Fig. 1. The construction consists of a 1 mW solid-state laser (670 nm), a mirror, a beam splitter and a position sensitive photodetector. A sample is mounted by clamping it vertically between two plates such that the protruding part forms a cantilever beam. An electronically triggered pin is used to generate an initial deflection on the sample; vibration of the sample is initiated by retracting the pin. Vibration curves are obtained by bouncing a laser beam off the sample to the photodetector.

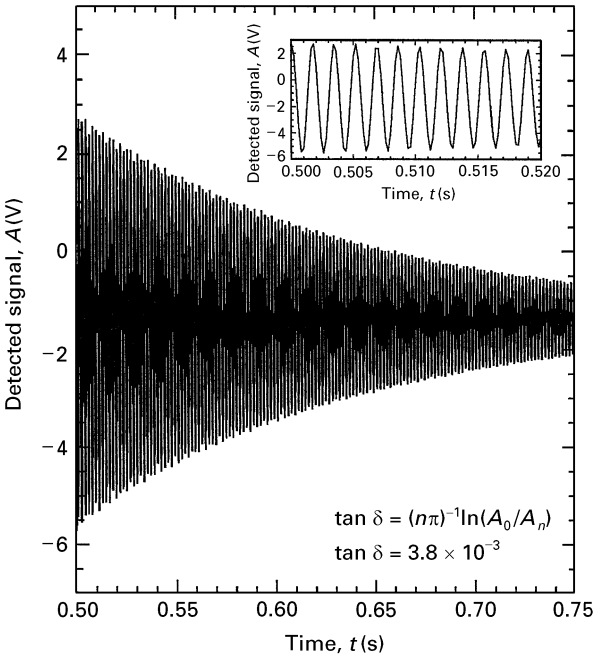

Figure 2 An example of the vibration damping curve from the optical system.

The damping factor, tan δ, is calculated from the decaying-oscillatory damping curve using the following equation [14]

$$\tan \delta = \frac{\ln(A_0/A_n)}{n\pi} \quad (2)$$

where n is the number of cycles of the vibration, A_0 is the amplitude of the first vibration, and A_n is amplitude of the n^{th} vibration. The term $\ln(A_0/A_n)/n$, also known as the logarithmic decrement Δ, can be obtained by fitting the experimental data to the following formula [14]

$$A(t) = B_0 \exp(-\zeta\omega_r t)\cos(\omega_d t - \phi) + B_1 \quad (3)$$

where ω_r is the resonant frequency of vibration, $\zeta = \Delta/[(2\pi)^2 + \Delta^2]^{1/2} \cong \Delta/2\pi$ when damping is small, $\omega_d = (1 - \zeta^2)^{1/2}\omega_r$, B_0, B_1 and ϕ are constants.

3. Results and discussion

Fig. 2 is a typical example of the results that were obtained from the optical system described above, in which a composite specimen without any fibre surface treatment (Type B specimen) was tested. The value of tan δ was found to be 3.81×10^{-3}. This value is typical for this material system. Other experimental results are summarized in Table II. The values presented here are the averages obtained from at least five specimens.

From the Bernoulli-Euler beam equation [14–16], it can be shown that the Young's modulus, E, of the material is related to its frequency of vibration. The equation used for calculating E for a beam specimen is described as follows [14]

$$E = \frac{12\rho\omega_r^2 L^4}{1.875^4 t^2} \quad (4)$$

TABLE II Measured resonant frequencies > ω_r, damping factors, tan δ_{in}, tan δ_{comp}, and transverse tensile strength, σ_{tr}

Specimen type	ω_r (s^{-1})	tan δ_{comp} ($\times 10^{-3}$)	tan δ_{in} ($\times 10^{-3}$)	σ_{tr} (MPa)
A	3733 (2.0%)	3.74 (1.6%)	2.24	28 (14.7%)
B	3980 (2.7%)	2.75 (3.6%)	1.37	45 (8.5%)
C	4061 (1.2%)[a]	2.46 (4.9%)	1.11	56 (8.8%)
D	4038 (1.2%)	2.73 (0.7%)	1.42	46 (9.3%)

[a]Numbers in parentheses represent the coefficient of variation.

where ω_r is the resonant frequency of the first mode of vibration, L and t are the length and the thickness of the beam, and ρ is the density. The density of the epoxy resin is 1.115 g cm^{-3} [17].

It has been reported that the damping factor also varies with frequency [18, 19]. By changing the beam length, a resonant frequency of about 3900 s^{-1} was obtained, which is comparable to the frequencies of other composites that were tested. The measurements from our optical system show that $E_m = 2.4$ GPa and $\tan \delta_m = 25 \times 10^{-3}$. It is also known that $E_f = 72.3$ GPa and $\tan \delta_f = 1 \times 10^{-3}$ [20]. These data were used to calculate the values of tan δ_{in} which are also given in Table II.

Equation 1 shows that with a higher value of tan δ_{in}, poorer adhesion is exhibited in the system. Results show that untreated specimens appear to have the weakest fibre–matrix interfacial adhesion among the four composite systems. However, the 366 size with silane specimens show the best interfacial adhesion. It is also interesting to note that the 366 size without silane and 158B size with silane specimens seem to have an equal magnitude of interfacial adhesion.

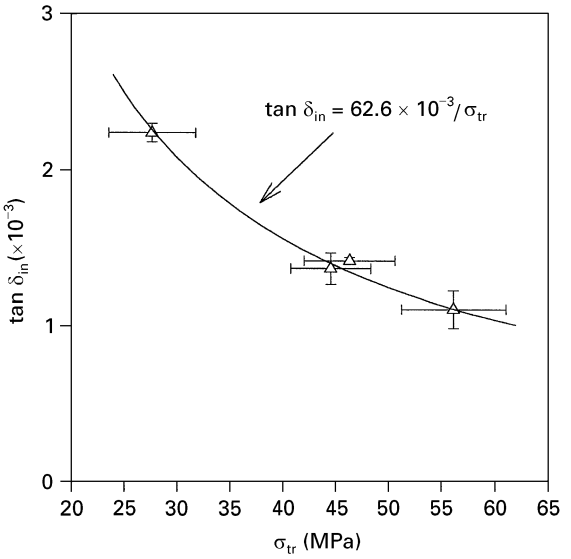

Figure 3 The relationship between tan δ_{in} and σ_{tr}. Error bars represent ± 1 standard deviation.

It is known that the poor interfacial adhesion exhibits a low transverse tensile strength, σ_{tr}. The

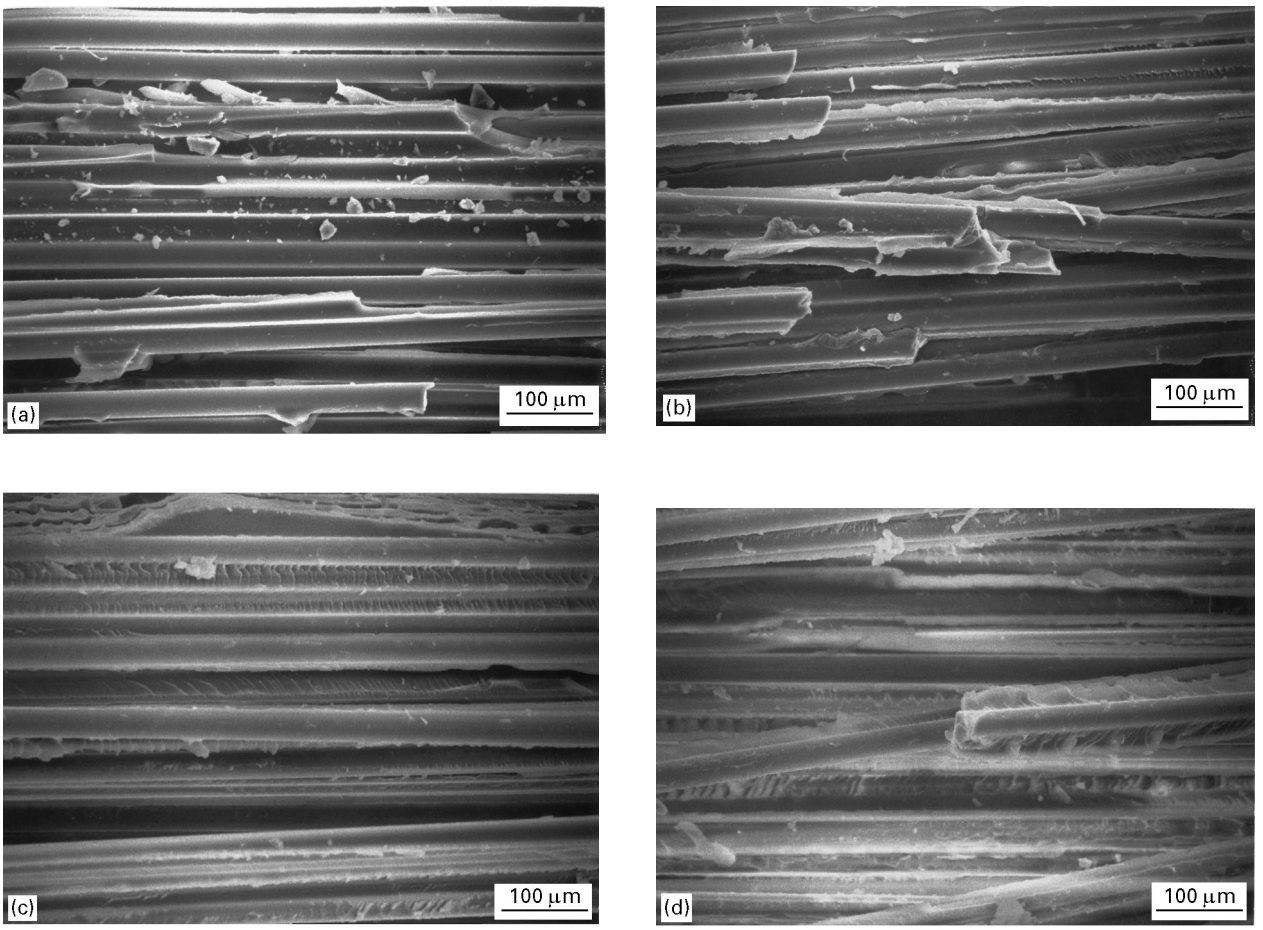

Figure 4 Scanning electron micrographs of the transverse tension fracture surfaces in the glass/epoxy composites: (a) Type A, (b) Type B, (c) Type C, and (d) Type D specimens.

measured σ_{tr} from the same composite systems are also listed in Table II. It can easily be seen that the observations from tan δ_{in} are consistent with the results from σ_{tr}, and both show the same magnitude of the interfacial bonding. In other words, tan δ_{in} and σ_{tr} are highly inversely correlated, as shown in Fig. 3.

In order to support the arguments, a comparison of the microstructures of different types of bonding mechanisms is presented in Fig. 4a–d. It is obvious that 366 size with silane and 158B size with silane specimens show better fibre–matrix wet-out than other types of specimens. Fibre–matrix debonding is clearly observed in the untreated fibre specimens.

This suggests a common process among the defect-related mechanisms involving (a) transfer of kinetic energy of structural motion to potential energy of a defect and (b) dissipation of the potential energy in the form of heat to its surroundings. Therefore, a sample containing more defects should have a higher damping factor; and also the more loosely configured a defect is, the higher is its contribution to damping. At a strongly bonded interface, there are fewer loosely bonded defects or centres which can easily absorb kinetic energy; therefore, it should have smaller damping. At a weak interface, there are probably more loosely bonded defects or centres to absorb kinetic energy; thus, it should have higher damping.

4. Conclusion

An optical system was constructed to measure the damping factor of a cantilever beam and used to characterize adhesion at fibre–matrix interfaces in glass fibre-reinforced polymer–resin composites. The tested samples had three different fibre–surface treatments and a control sample. The results show that the composite system having 366 size with silane exhibits the best fibre–matrix interfacial adhesion. The system having 366 size without silane exhibits an equal magnitude of interfacial adhesion to the system having 158B size and silane. Samples with untreated fibres have the weakest interfacial adhesion. Transverse tension test results and scanning electron micrographs clearly show that fibres with silane surface treatment have the best fibre–matrix interfacial adhesion. The experimental results showed a strong inverse relationship between the damping characteristics of the fibre–matrix interface and transverse tensile strength of composites.

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