DEFORMATION MICROMECHANICS OF GLASS-FIBRE-REINFORCED COMPOSITES

Marek Hejda*, Robert J. Young*, Stephen J. Eichhorn*
marek.hejda@postgrad.manchester.ac.uk
* Northwest Composites Centre, School of Materials, University of Manchester

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Abstract

The current work presents for the first time the application of luminescence spectroscopy in following the micromechanics of deformation in glass-fibre reinforced composites; this has been achieved using luminescence-active glass fibres prepared from glass doped with Sm^{3+}. Glass prepared in this way exhibited several relatively sharp and intense luminescence peaks observed in the range 550 nm to 700 nm. The luminescence band located at 648 nm was used for the calibration of the local strain state of the fibre due to its shift towards lower wavenumbers with increasing strain. The fragmentation of Sm-doped glass fibre has been followed in detail and the behaviour analysed using a classical shear-lag analysis.

1 Introduction

It is a well-established fact that the physical properties of the interface between a fibre and a matrix material in a composite are very important as this governs the stress-transfer process, which in turn is responsible for the effectiveness of the reinforcement [1]. Raman spectroscopy is a commonly used technique in analysing the deformation micromechanics and the strength of the interface between composite matrices and reinforcing fibres [2]. Although a wide range of fibre-matrix systems have been studied using this technique, glass-fibre-reinforced polymer-matrix composites have not been characterised since it has not been possible to obtain well-defined Raman spectra of glass. Attempts have been made to overcome this problem by coating the fibres with a strain sensitive material such as polydiacetylene-urethane copolymer [3] or using carbon nanotubes as stress sensors in the vicinity of the glass fibres [4]. However, a recent study has reported that the use of lanthanides (such as Sm^{3+}, Er^{3+}) enable the local stress state in glass to be observed using photo and electroluminescence [5-6].

2 Experimental

2.1 Materials

Glass of composition (wt %) 64 % SiO_2, 6.5 % Al_2O_3, 1.6 % CaO, 10.7 % Na_2O, 0.4% MgO, 16.3 % B_2O_3, and 0.5 % SmF_3 was produced using a standard melting process. Glass rods approximately 20 cm long and 0.5 cm diameter were drawn from the melt which were subsequently used to form the individual fibres.

The resin matrix used in these experiments was an epoxy Araldite® LY/HY5052 (Ciba-Geigy 5052). The butan-1,4-diol diglycidyl ether resin (LY5052) was mixed with the isophorone diamine hardener (HY5052) in a 50/19 weight ratio.

2.2 Composite preparation

Dumbbell shaped single-fibre composite was prepared of dimensions illustrated in Fig. 1. Firstly, some of the resin mixture was poured into a mould and allowed to cure partially for 4 hours at room temperature then a glass fibre (approximately 1 cm long) was carefully placed onto the surface of the resin in the centre of the sample. The second part of the resin was then poured into the mould and the samples left to cure for at least seven days at room temperature.
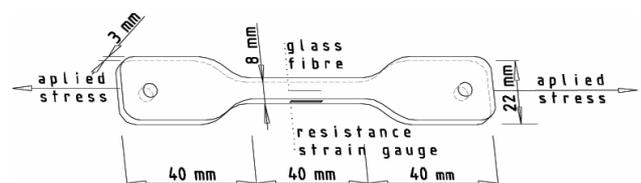

Fig. 1. Schematic representations of the dumbbell specimen used in fragmentation test.

2.3 Luminescence spectroscopy

A Renishaw System-1000 spectrometer was employed to record spectra, using a 514 nm Ar laser (Spectra-Physics Lasers) and a ×50 lens. This lens focused the laser to a spot size of about 2 μm on the fibre surface. A highly sensitive Peltier-cooled charge-coupled (CCD) camera was used to collect the luminescence spectra using an exposure time of 10 s. All spectra were fitted using a Gaussian/Lorentzian function to determine the peak position at each strain level.

In order to calibrate the 648 nm luminescence band shift of the glass fibres, a single glass fibre was encapsulated onto the epoxy resin sample of dimensions 60×10×3 mm. The specimen was left to cure at room temperature for at least one week. A strain gauge (type EA-06-240LZ-120, produced by Vishay®), of gauge factor 2.08, was attached parallel to the fibre in order to monitor the surface strain applied to the system. The specimen was then placed in a small four-point bending rig situated on the microscope stage of an Olympus optical microscope, which was part of the Renishaw 1000 system. The strain was increased in increments of 0.1% in tension, and spectra taken.

The dumbbell specimen was mounted on a Miniature Materials straining rig (MINIMAT™, Polymer Laboratories Ltd, UK), which was, in turn, placed on the microscope stage. The strain in the matrix was simultaneously measured using a strain gauge attached to the matrix surface adjacent to the fibre. Spectra were recorded along the length of the fibre within the composite at various matrix strains, up to failure of the dumbbell specimen which occurred at about 1.1% matrix strain.

3 Mechanical testing

The tensile properties of the glass fibres were measured using an Instron universal testing machine (model 1122) at a strain rate of 0.0167 %s^{-1} (cross-head speed of 5 mm/min, gauge length 50 mm). For statistical reasons approximately 40 fibres were tested.

The fibres were found to have a mean Young's modulus of 99±3 GPa, a failure strength of 0.88±0.06 GPa and a strain to failure of 0.80±0.05 %. The Young's modulus of these fibres is quite high compared to literature values for E-glass (*cf.* 70 GPa in ref. 1). This may be due to the high level of SiO_2.

4 Single fibre deformation

As depicted in Fig. 2, several relatively sharp and intense peaks of luminescence spectrum of glass fibres were observed in the range 550 nm to 700 nm.
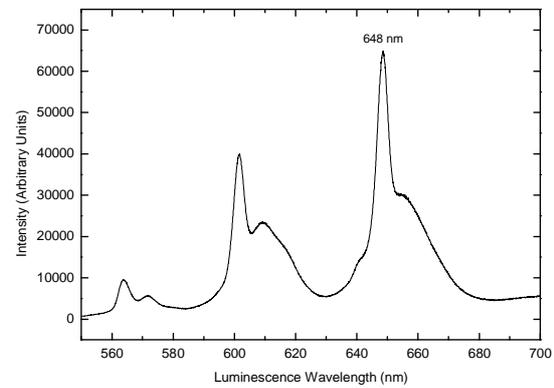
Fig. 2. The luminescence spectra of Sm-doped glass with the 648 nm peak highlighted.

The luminescence band located at 648 nm was used for subsequent stress assessment due to its high intensity and distinctive linear shift toward lower wavelengths during tensile experiments as shown in Fig 3. The shift in the peak position, $\Delta\nu$, with respect to the applied strain, was found to be linear with slope $\Delta\nu/\Delta\varepsilon$ = -0.08 nm%$^{-1}$ (R^2 = 0.99) as illustrated in Fig 4.
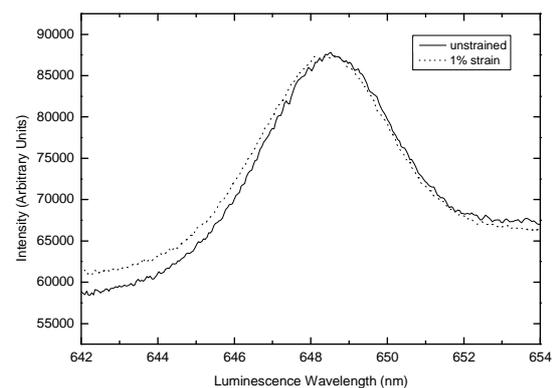
Fig. 3. Shift in the 648 nm central maximum position with a strain of 1% (dotted line).
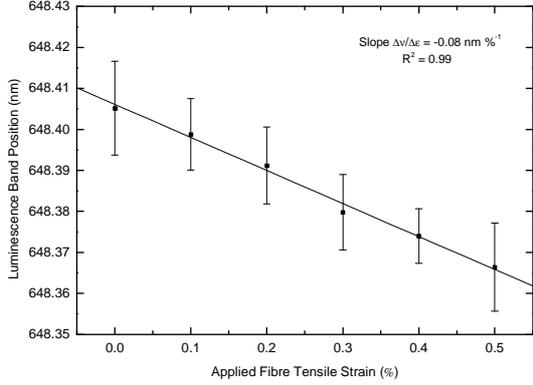
Fig. 4. The luminescence band shift of the 648 peak as a function of applied strain on a single Sm-doped glass fibre.

5 Single fibre composite test

Several methods have been developed to determine the properties of the fibre/matrix interface. One of the most commonly used is the single-fibre fragmentation test introduced by Kelly and Tyson [7]. In this test, a single fibre is embedded in a large amount of resin matrix and the specimen is loaded in tension. As the load is increased, the fibre breaks into fragments. It has been demonstrated that luminescence spectroscopy can be used to study the micromechanics of load transfer during fragmentation testing [8].

In this study luminescence spectroscopy was employed to measure the axial stress along a studied fibre by monitoring changes in stress sensitive luminescence band positions at each stage of matrix load. A shear-lag approach was used to analyse the results.

5.1 Shear-lag model

The shear-lag model was originally proposed by Cox [9]. The model assumes that the fibre and the matrix are in an elastic state, there is a perfect adhesion along the interface, and there is no stress transferred across the ends of the fibres [1]. The variation of the axial fibre stress (σ_f) and strain (ε_f) with position (x) along an embedded fibre is given as follows [10]:

$$\sigma_f = \varepsilon_m E_f \left[1 - \frac{\cosh \beta(l/2 - x)}{\cosh \beta l/2} \right] \quad (1)$$

and

$$\varepsilon_f = \varepsilon_m \left[1 - \frac{\cosh \beta(l/2 - x)}{\cosh \beta(l/2)} \right] \quad (2)$$

where ε_m is the matrix strain, E_f is the fibre modulus, r_f is the fibre radius, and l the fibre length. The parameter β is dimensionless and can be expressed as follows:

$$\beta = \left[\frac{2G_m}{E_f r_f^2 \ln(R/r_f)} \right]^{1/2} \quad (3)$$

where G_m is the shear modulus of the matrix and R is the radius of a solid cylinder of matrix around the fibre that is influenced by the applied stress.

The interfacial shear stress along the fibre can be calculated from the equilibrium of forces acting over a small area on the surface of the fibre [10], which gives:

$$\tau = \frac{r_f}{2} \frac{d\sigma_f}{dx} \quad (4)$$

Hence, an expression for the variation of the interfacial shear stress along the fibre can be derived as:

$$\tau = E_f \varepsilon_m \left[\frac{G_m}{2E_f \ln(R/r_f)} \right]^{1/2} \frac{\sinh \beta(l/2 - x)}{\cosh \beta l/2} \quad (5)$$

5.2 Fragmentation analysis

Fig. 5 shows the strain distribution along the Sm-doped glass fibre at a matrix strain of both 0.5% and 1.0%. The local fibre strains are low at the fibre ends which indicate the transfer of shear stress from the epoxy resin matrix to the fibre [10]. The fibre strain then increased to a value approximately equal to the matrix strain.

At 1% matrix strain it can be seen that the fibre broke into two fragments as the failure strain of the fibre was exceeded. The fibre strain at the fibre break point drops to about zero.

All of the solid lines through the experimental data represent the theoretical calculations based on Eq. 2. The value of R/r was calculated as 70 based on the specimen dimensions (~3 mm thickness) and the fibre diameter (~42 μm), which gives a close fit to the experimental data.

Closer inspection of Fig. 5 shows that the data points, more noticeable at a matrix strain of 0.5%, do not exhibit a plateau region at the central area of

the fibre; instead, the fibre strain increases gradually from one fibre end to the other. This gradual increase was postulated to be due to a variation in the fibre diameter along its length, as was observed in some cases. The fibres were manually hand drawn therefore rendering it difficult to maintain a constant fibre diameter. As the fibre diameter is related to the load transfer, the strain is therefore also affected.

The corresponding interfacial shear stress distributions derived from Eq. 5 are shown in Fig. 6. It can be seen that the interfacial shear stress is highest at the fibre ends as predicted by the shear-lag analysis. The maximum interfacial shear stress, τ_{max}, for Sm-doped fibre was found to be 36 MPa.
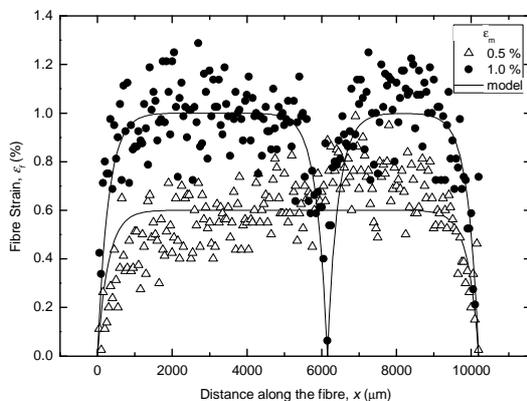

Fig. 5. The strain profile along a short Sm-doped glass fibre embedded in a model composite at two strain levels.
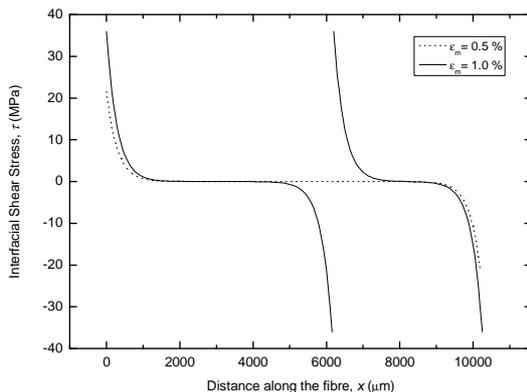

Fig. 6. The variation of interfacial shear stress along the Sm-doped glass fibre at two strain levels.

6 Conclusions

The interfacial deformation micromechanics of glass-fibre-reinforced epoxy composites have been determined directly using fibres prepared from glass doped with Sm^{3+} and luminescence spectroscopy.

Well defined luminescence spectra from the glass with a linear shift with strain of the 648 nm band were obtained. Using the above technique it was possible to construct calibration curves for their use in evaluating the strain distributions along fibre within model composite.

The micromechanics involved in the fragmentation behaviour of Sm-doped glass fibre was studied in detail using classical shear lag analysis. From this analysis the maximum interfacial shear stress was determined to be 36 MPa.

The current work demonstrates for the first time the mapping of a glass fibre-resin composite interface, thus highlighting the potential for using doped fibres as strain gauges within related bulk materials.

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