CHARACTERIZATION OF MODIFIED INTERPHASE PROPERTIES IN E-GLASS/EPOXY MODEL COMPOSITES USING DILA

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Introduction

Assume a model composite consists of fiber, matrix, and interphase (Fig. 1a). The interphase functions as a bridge between the fiber and the matrix. If the interphase bonds strongly to the fiber and the local shear stresses exceed the yield stress of the matrix, plastic deformation increases and hence the energy dissipation should increase and the failure will be cohesive. If the interactions between the interphase and the fiber are weak, the local shear stresses are not sufficient to exceed the yield stress of the matrix and hence there will be no plastic deformation. In this case, the energy dissipation will be small and the failure will be adhesive at the fiber surface. Thus, the selection of the interphase material plays a crucial role in the mechanical properties and fracture behavior of the composites.

The objectives of this work are :

- Control the interphase properties
- Design of interphase material with properties that can meet the requirements for high strain rate applications.

Some predictions on the behavior of force-displacement curve (Fig. 1b) were suggested for different types of interphase:

- For water-sized fiber composites, the fiber matrix-interaction is weak (poor adhesion), the failure will be brittle and energy absorption is small.
- For sized fiber composites, if the compatibility, roughness, and plastic deformation induced in the matrix and in the interphase increase, the energy absorption and the shear strength increase. The upper band (black curve) where shear strength and high energy dissipation (frictional energy) are very high, is an ideal case for high rate applications.

Interphase Effect Predictions

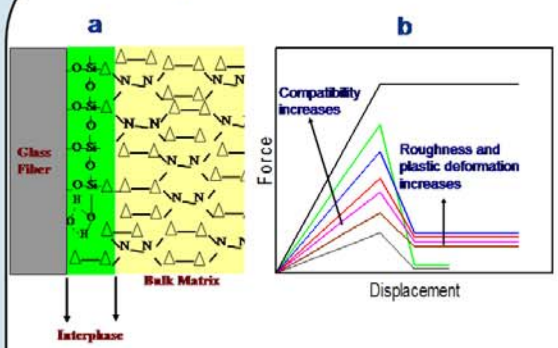

Figure 1: a) Schematic structure for glass fiber/epoxy model composite.
b) Predictions of force-displacement behavior for different types of interphase properties.

Sizing Systems

Three sizing systems were prepared: Tri-methoxy, Di-Trimethoxy(1/1), Di-Tetraethoxy(1/1) and Di-etraethoxy(1/1) copolymer (Fig. 2). The selection of these systems was based on the following reasons:

- A copolymer of Di- and Tri- or Di- and Tetra-ethoxy will increase the function groups through which the interfacial interactions should increase and hence the interfacial shear strength increases.
- As the ratio of -Δ/OH per copolymer unit decreases the interphase becomes more ductile and the energy absorption increases.

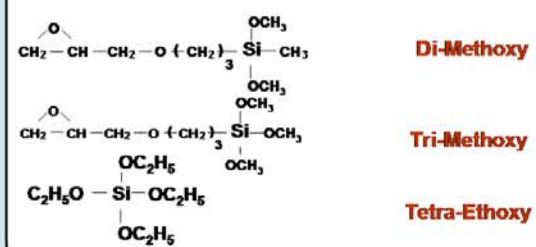

Figure 2: Chemical structures for (3-Glycidoxypropyl)Methyldimethoxysilane (Di-Methoxy), (3-Glycidoxypropyl) Trimethoxysilane (Tri-Methoxy) (GPS) and Tetraethoxysilane (Tetra-Ethoxy).

Predictions On Interphase Properties

The predictions on ratio -Δ /OH, compatibility with matrix (C-M), compatibility with fiber (C-F) and failure modes are given in Table 1.

Table 1: Predictions on interphase properties

Sizing system (Mix. Ratio)	-Δ /OH	C-F	C-M	Predicted failure modes
Trimethoxy (100%)	1/3	2	4	Adhesive
Di-Trimethoxy (1/1)	2/5	3	5	Adhesive or cohesive
Di-Tetraethoxy (1/1)	1/6	4	2	cohesive
Di-Tetraethoxy (3/1)	3/10	5	3	Cohesive
Water sized	-	1	1	Brittle, adhesive

- Highest compatibility can be obtained by Di-Tri and the lowest obtained by Water sized.
- Highest compatibility with fiber can be obtained by Di-Tetra3/1 and the lowest by water sized.

Specimen Preparation

- The materials used are: Epon-Epoxy 828; and E-glass fiber.
- Glass fiber/epoxy model composites were prepared (Fig. 2).
- Curing cycle: at RT for 24 hours, 90° C for 2 hours and post curing at 160° C for 2 hours
- Slicing and polishing to thickness of ~100 mm

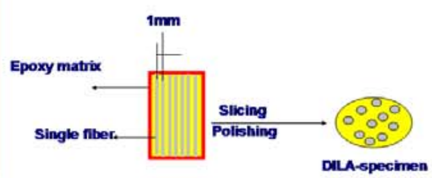

Figure 3: Schematic diagram for specimen preparation of glass fiber/epoxy model composite.

DILA Measurements

The interphase properties were measured using Dynamic Interphase Loading Apparatus (DILA)(Fig. 4). The measurements were performed at displacement rate of 110 mm/s and the support radius was 65mm.

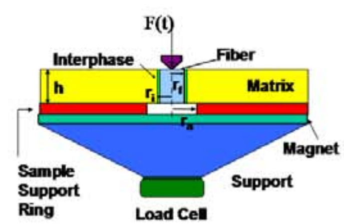

Figure 4: Schematic diagram for micro-indentation test

Data Reduction

Shear strength (τ)

τ was calculated as

$$\tau = Fu/A;\ A = \pi Rh \quad (1)$$

where Fu is the failure load, R is the fiber diameter, and h is the specimen thickness.

Shear modulus (G_i)

G_i was calculated as

$$G_i = ((-K_e)\ a\ \coth(ah)\ \ln(r_i\,(1+B_1)/r_f))/2\pi \quad (2)$$

where

$$a^2 = 2G_i/E_f r_f^2 \ln(r_i(1+B_1)/r_f),\ B_1 = G_i \ln(r_m/r_i)/G_m \ln(r_i/r_f) \quad (3)$$

K_e is the slope of in the elastic region of load-displacement curve; r_i is the radius of the interphase, r_m is the radius of the matrix and G_m is the shear modulus of the matrix.

Elastic Energy E_e

The elastic E_e was calculated from load-displacement data

$$E_e = F^2/2K \quad (4)$$

$$K = K_e - (K_e - K_n)(\delta - \delta_e)/\delta \quad (5)$$

K_n is the slope in the nonlinear region, δ is the displacement, and δ_e is the displacement at the end of elastic region (Fig.5).

Data Reduction

Absorbed Energies

The load-displacement curve obtained by DILA (Fig. 5) can be divided into three regions. In Region 1, progressive debonding and yield process can occur. In Region 2, a transition between debonding and pure frictional sliding processes takes place. The frictional sliding process evolves in Region 3.

From the load-displacement curve, the absorbed energy density due to debonding, (ΔE_D), the transitional absorbed energy density (ΔE_{trans}), and the normalized absorbed energy due to frictional sliding (ΔE_{fs}) can be calculated as

$$\Delta E_D = (\Delta E(\delta_D) - \Delta E(\delta_e))/A \quad (6)$$

$$\Delta E_{trans} = (\Delta E(\delta_D) - \Delta E(\delta^*))A \quad (7)$$

$$\Delta E_{fs} = h\,(\Delta E(\delta_{fs}) - \Delta E(\delta_D))/(A(\delta_{fs} - \delta_D)) \quad (8)$$

where the absorbed energy ΔE at any δ is given by

$$\Delta E(\delta) = E_{total}(\delta) - E_e(\delta) \quad (9)$$

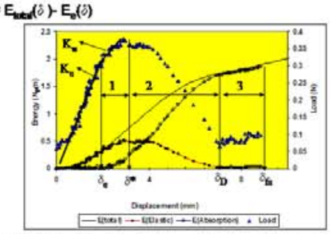

Figure 5: Calculating the absorbed energies from the load-displacement measured by DILA.

Stress-Displacement Curves

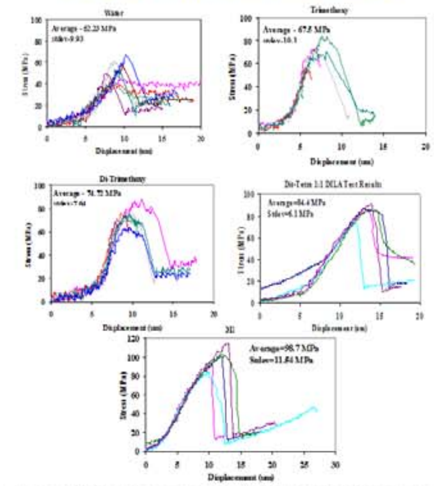

Figure 6: Stress-displacement results for different sizing systems

Average Shear Strength

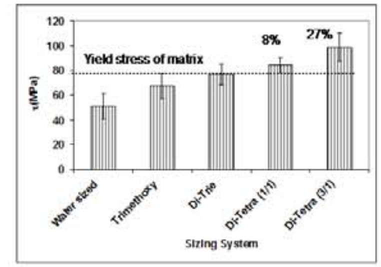

Figure 7: Average shear strength for different sizing systems.

Since Di-Tetra (1/1) and Di-Tetra (3/1) have a higher compatibility with the fiber (C-F) (Table 1) than other sizing systems, they show higher shear strength values. Moreover,the strong interface bonds in theses two sizings cause the shear strength to exceed the strength exceeds the yield stress of the matrix with 8% for Di-Tetra (1/1) and 27% for Di-Tetra (3/1).

Shear Modulus

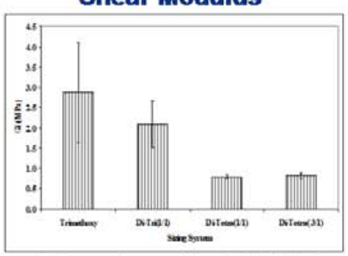

Figure 8: Average shear modulus G_i of the interphase for different sizing systems.

Di-Tetra (1/1) and Di-Tetra (3/1) have lower shear modulus values than other sizing systems. This decrease in shear modulus may be due to the decreases in the ratio of -Δ/OH in copolymer unit and in compatibility with the matrix (C-M) (Table 1) for these tow sizing systems.

Absorbed Energies

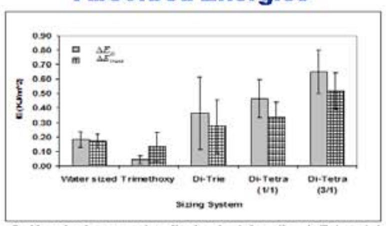

Figure 9: Absorbed energy density due to debonding (ΔE_D) and transitional absorbed energy density (ΔE_{trans}) for different sizing systems.

The absorbed energies ΔE_D and ΔE_{trans} show an increasing trend for Di-Tetra (1/1) and Di-Tetra (3/1). The increase in ΔE_D values is due to the plastic deformation, decrease in -Δ/OH in copolymer unit. The strong interfacial interactions (high shear strength)can support the local stresses that can exceed the yield stress of the matrix (Fig. 7) causing a plastic deformation in the interphase. The high energy release due to the strong interfacial bonds causes the absorbed energy ΔE_{trans} to increase for Di-Tetra (1/1) and Di-Tetra (3/1).

Frictional Sliding Absorbed Energy

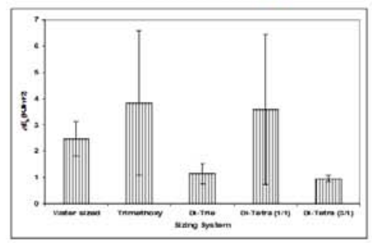

Figure10: Normalized absorbed energy due to frictional sliding (ΔE_{fs}) for different sizing systems.

The normalized absorbed energy density due to the frictional sliding (ΔE_{fs}) has a tendency to increase for the sizing systems Trimethoxy and Di-Tetra (1/1). This may be related to degree of roughness of the texture on the fiber surface.

Conclusion

- The interfacial shear strength and energy absorption in the glass fiber composites can be increased by using silane-based copolymers.
- The sizing system of Di-Tetra (1-1) and (3-1) show higher energy absorption and shear strength than other systems.
- The decrease in the ratio of -Δ/OH in copolymer unit can cause the shear modulus to decrease and the energy absorption due to debonding to increase.
- The absorbed energy due to debonding increases through the plastic deformation that can be induced in the interphase when local shear stresses exceed the yield stress of the matrix.
- A high degree of adhesion between the fiber and matrix can lead to high transitional absorbed energy.

Acknowledgements

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