MECHANICAL PROPERTIES OF BORON AND KEVLAR-49 REINFORCED THERMOSETTING COMPOSITES

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1. Abstract

Glass reinforced thermosetting composite materials are now widely usUUed in building and construction applications. However, the adoption of Boron and Kevlar based composite materials in this industry has been very slow, and the main reasons are lack of previous experiences to assess the durability of such composite products and understanding of their properties by designers. In order to fill this gap, the understanding of Boron and Kevlar fibre reinforced thermosetting composites becomes necessary for the design of structures. This study is aimed towards characterizing the mechanical properties of four selected reinforced composites using these fibres to benefit designers and manufacturers in a direct and an easily understandable manner. The properties of these composites were evaluated using micromechanics models based on classical lamination theory, self-consistent approach and Rosen and Xu-Refsnider models. The model estimations are compared with the experimentally obtained mechanical properties. An attempt is made to rationalize the results with respect to the basic properties of the constituents of these composites so that newer composites can be confidently estimated.

2. Introduction

The use of thermosetting composites has gained steady favour but the properties obtained vary significantly from those predicted by mathematical models for stiffness and strength of this type of materials [1].Characterization of the tensile, flexural and compressive properties as well as the anisotropic nature of composites is more complicated compared to conventional materials [2].

Although many researchers have made efforts to analyze the mechanical behaviour of composite structures, there are limited studies on properties of Boron and Kevlar-49 thermosetting composite laminates which exhibit excellent mechanical properties [3]. Boron and Kevlar-49 fibre reinforced composites have high modulus, high strength, and toughness. They have recently become available for use as advanced load-bearing structures due to excellent strength-to-cost benefits.

Boron fibres are not only strong in tension but also lead to composites that are strong in compression, probably due to the large diameter which inhibits buckling. Theoretically, Boron fibres exhibit a linear axial stress-strain relationship from room temperature to 650 ^{o}C. Kevlar-49 reinforcement has been seldom used in high-performance structural applications but its good mechanical properties combined with low density need further exploration. The performance of these composites depends largely on the quality of the matrix–reinforcement interface, which determines the way load is transferred from the polymer to the fibre.

Polyimide is a high-performance polymer owing to its high thermal stability up to 300 ^{o}C, low dielectric constant and high chemical resistance. Accordingly, it found applications in the composite and microelectronics industries [4]. Also, polyimide based composites possess high mechanical strength, acceptable wear resistance, good thermal stability, high-stability, good anti-radiation, and good solvent resistance [5]. LM unsaturated polyester resin is commercially most important, accounting for around 80% of the relevant market. Reactions with it generally occur quickly, but in a controllable manner to give a cross-linked thermoset composite structure and the matrix itself is inexpensive.

The adhesion between fibre and matrix plays a critical role in the performance of polymer composites. Strong interfacial adhesion strength must be necessary to improve the mechanical properties of composites. A number of studies have been conducted to enhance the adhesion between Kevlar-49 and polymer through surface treatments of fibres, and the most successful one is chemical treatment of fibres [6].

3. Models for estimation of mechanical properties

Micromechanical models have been widely used to predict composite properties from their constituents' properties, especially in the case of continuous fibre composites applying traditional laminate theories.

Classical Lamination Theory (CLT)

The rule of mixture (ROM) equation for the apparent Young's modulus in the fibre direction is:

$$E_1 = E_f V_f + E_m V_m \tag{1}$$

where E_f, E_m, V_f and V_m are the moduli and volume fractions of the fibre and matrix materials respectively. This model is used with the basic assumption of equal strain in the two constituents, and its modified version is:

$$E_1 = E_f V_f + \kappa E_m V_m \tag{2}$$

where κ is geometrical and interfacial bonding factor. A value of κ = 0.919 is used for long fibre composites under axial tension or κ = 0.27 to 0.375 for short fibres to account for the random orientation and variation of aspect ratio of the fibres [7].

The elastic modulus of the composite in the transverse direction (E_2) is determined by an inverse rule of mixtures (IROM) equation:

$$E_2 = \frac{E_f E_m}{E_f V_m + E_m V_f} \tag{3}$$

E_2 can also be determined using Halpin-Tsai semi-empirical relationship [8] and Reuss's assumption [9], and this equation is called modified IROM.

Self-Consistent Model for Tensile Properties

This approach is generally credited to Hill [10] and Budiansky [11], whose original work focused on spherical particles and continuous and aligned fibres.

In self-consistent or embedding models, a single particle in an infinite matrix is considered for determining the average properties of the composite. The general form of such models is as follows.

$$\overline{G} = G_r + \sum_{i=1}^{N} \frac{\upsilon_i (G_i - G_r)\overline{G}}{\overline{G} + 2\overline{S}_{1212}(G_i - \overline{G})} \tag{4}$$

$$\overline{K} = K_r + \sum_{i=1}^{N} \frac{\upsilon_i (K_i - K_r)\overline{K}}{\overline{K} + \frac{1}{3}\overline{S}_{ijkl}(K_i - \overline{K})} \tag{5}$$

where

$$\overline{S}_{1212} = \frac{3\overline{K} + 6\overline{G}}{15\overline{K} + 20\overline{G}} \quad \text{and} \quad \overline{S}_{ijkl} = \frac{9\overline{K}}{3\overline{K} + 4\overline{G}} \tag{6}$$

Rosen and Xu-Refsnider models

Rosen [12] model is based on the elastic buckling analysis of fibres embedded in matrix using traditional energy method [13]. The model, which is two-dimensional, treats the fibres as layers (plates) supported by an elastic matrix. This model assumes that the layers are perfectly straight and evenly spaced, and that both the fibres and matrix may be described as linearly elastic. When the unidirectional composite of infinite extent undergoes failure, Rosen [13] envisages two possible modes of failure, which are called the extension mode (refer to Eqn. 7) and shear mode (refer to Eqn. 8). In the extension mode, the matrix material is predominantly in extension and adjacent layers deform π radians out of phase with each other. In the shear mode, adjacent layers deform in phase, and the matrix material is predominantly in shear.

Extension mode: $$\sigma_{CIC} = 2V_f \sqrt{\frac{V_f E_m E_{f1}}{3(1 - V_f)}} \tag{7}$$

Shear mode: $$\sigma_{CIC} = \frac{G_m}{1 - V_f} \tag{8}$$

Xu and Reifsnider [13] model is also based on the use of micro-buckling model of a representative volume element using a beam on elastic foundation. The effects of matrix slippage and fibre-matrix bond condition are included by two factors, namely, ξ and η. The model expression in terms of the constituent properties and micro-geometrical parameters is given in Eqn. (9).

$$\sigma_{CIC} = G_m \left[V_f + \frac{E_m}{E_{f1}}(1 - V_f) \right] \times \left\{ 2(1 + v_m) \sqrt{\frac{\pi \sqrt{\pi} \eta r_f}{3\left(\frac{E_m}{E_{f1}}\right)\left(V_f \frac{E_m}{E_{f1}} + 1 - V_f\right)\left(1 + V_f v_f + v_m (1 - V_f)\right)}} + 1 - \xi - \frac{\sin \pi\xi}{2\pi} \right\} \tag{9}$$

4. Experimental

Materials

In this study, continuous fibres of BORON 5521 (fibre **F1**), prepared by chemical vapour deposition using high-modulus carbon fibre as the substrates (from Specialty Materials Inc. [14]) and unidirectional Kevlar-49 fibre monofilament (fibre **F2**) from Dupont™ de Numours were selected as reinforcements. Thermosetting materials used as matrix were (i) Polyimide resin PMR-15 (matrix **S1**) (Kapton HN® DuPont Nemour) which required a temperature of 315 °C for curing, and (ii) Unsaturated polyester resin (matrix **S2**) (Scott Bader® Crystic™ 196E) which is a low modulus thermosetting matrix that can be cured at 35 °C after preparing the composite. The basic physical and mechanical properties of the fibres and the matrix materials used in this study are listed in Table 1.

Table 1: Basic properties of fibres and matrix

Fibre Type	d_f (μm)	ρ_f (kg/m^3)	E_{f1} (GPa)	E_{f2} (GPa)	σ_{fT} (MPa)	σ_{fC} (MPa)
Boron	140	2629.6	399.9	399.9	4136.9	4826.3
Kelvar-49	12	1467.0	151.7	4.1	2757.9	517.1

Matrix Type	ρ_m (kg/m^3)	E_m (GPa)	σ_{mT} (MPa)	σ_{mC} (MPa)	τ_m, (MPa)
Polyimide	1218	3.5	103.4	206.8	89.6
LM Polyester	1163	2.2	55.2	103.4	55.2

Notations for the prepared composites are as follows:
F1S1 : fibre **F1** (Boron) with **S1** (Polyimide)
F1S2 : fibre **F1** (Boron) with **S2** (LM Polyester)
F2S1 : fibre **F2** (Kevlar-49) with **S1** (Polyimide)
F2S2 : fibre **F2** (Kevlar-49) with **S2** (LM Polyester)

Preparation of Composites

The architecture, manufacturing, and quality control in preparing the test specimens had followed the established recommendations [15]. The four types of composite panels were fabricated for the evaluation of mechanical properties. The volume fraction of Boron and Kevlar-49 fibres used in all the composites was maintained at 61% of the composite volume. The chemical treatment of Kevlar-49 fibres was done using 10 wt.% phosphoric acid (H_2PO_4) solution using a laboratory scale apparatus. The Kevlar-49 fibres were then washed several times with distilled water and dried in a vacuum oven at 80 °C for 24h. Melt and mold temperature of 260 °C and 80 °C, respectively, have been used.

Mechanical Testing

Tension tests: Five dog-bone shaped specimens were tested following BS 2792 Part 3 Method 321:1994 for each type of composite laminate at the ambient conditions using an extension rate of 1 mm/min on a computer controlled 30 kN MTS Alliance RT/30 testing machine equipped with a digital controller and computer data acquisition for tensile tests. Instantaneous load P and displacements were recorded at a rate of one set per second. The elastic modulus (E) was calculated using data regression, and the tensile strength was calculated from the maximum load and the actual specimen cross-sectional area.

Compression tests: The tests were carried out using specimen of 5mm nominal thickness with gage length (between grips) of 10mm as described in BS 2792 Part 3 Method 345A:1993. Five specimens were tested for each sample. From the test record, the compressive modulus and the compressive strength corresponding to the maximum load at failure could be determined.

Flexure tests: Five specimens of each composite type were tested in three point bending configuration using the same testing machine. The radius of the loading roller tip was 5mm. The span length L was kept as 40mm to maintain a span length to thickness ratio 8. Instantaneous load P and crosshead displacement δ measured by a linear variable differential transformer (LVDT) were recorded by a computerized data acquisition system at one second intervals. The flexural modulus (E_{CIB}) and strength (σ_{CIB}) were calculated following BS 2782 Part 3 Method 335A:1993.

5. Results and Discussions

Density of the Prepared Composites

The densities of the prepared composites were found to be about 90% of the theoretical values:
F1S1 : 1,875 kg/m^3 F1S2 : 1,850 kg/m^3
F2S1 : 1,235 kg/m^3 F2S2 : 1,215 kg/m^3

The difference can be attributed to the void content and imperfections in orientation and packing of fibres. These results are in a range of densities obtained in such type of composites studied [16].

Longitudinal Tensile Properties

Fig. 1 illustrates typical load-displacement (P-δ) responses of Boron and Kevlar-49 fibre reinforced samples. F1S1 and F1S2 samples showed higher peak loads or strengths than that of F2S1 and F2S2 composites. All composites exhibited some non-linear responses in their tensile properties. The high strength of Boron fibre led to higher levels of strength compared to those of Kevlar-49 composites. The theoretical and experimental tensile properties of the four Boron and Kevlar-49 reinforced thermosetting composites are presented in Tables 2 and 3. The ROM equation assumed that fibres and matrix are perfectly bonded together and will be stretched/strained by the same amount under an applied load. Actually, the tensile strength of fibres is not uniform.

Rosen's model of cumulative damage, which is based on the Weibull distribution [17] of the strength-length relationship, provides better estimation of longitudinal tensile strength when the fibres and the matrix exhibit brittle behavior. The ROM model assumes that the composite consists of N fibres of original length L and the weaker fibres fracture due to the applied tensile stress.

Theoretical prediction for the four reinforced composites estimated by the model equations are approximately an order of magnitude higher than those experimentally measured values. In addition, fibre and matrices are considered to behave elastically. Besides, fibre breakage and matrix failure are possibly responsible for the large reduction in strength properties. In addition, the effect of lateral constraint imposed by the strain compatibility leads to higher transverse moduli.

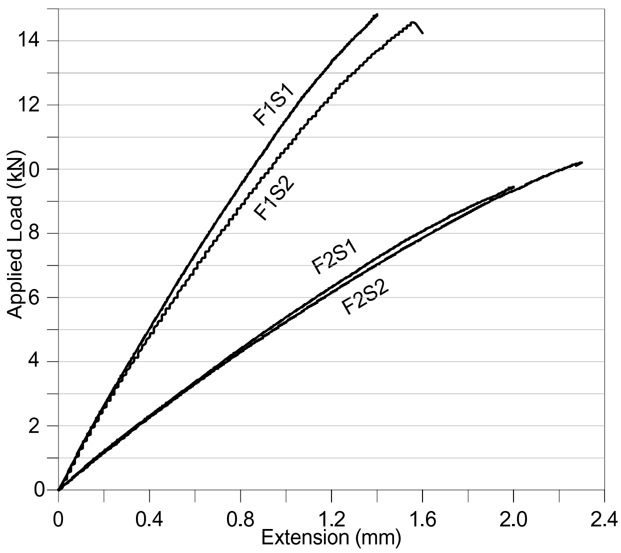

Fig. 1: Load-displacement curves obtained from uniaxial tensile tests of the composites.

Piggott [18] suggested that high volume fractions combined with reduction in the average fibre and matrix stresses must be considered to obtain predictions which are closer to the decreased composite strength. Simple ROM strength models (Eqn. 1 and 2) predict a linear increase in composite strength with volume fraction, indicating their use restrictive and incorrect.

Longitudinal Compressive Properties

The failure of composites in compression is usually triggered by fibre microbuckling when individual fibres buckle inside the matrix. The buckling process is controlled by fibre misalignment. The compressive load versus displacement responses of the tested composites loaded along the in-plane direction are shown in Fig 2. The slopes of these curves indicate that the compressive moduli of composites manufactured by Polyimide matrix are generally higher than the LM polyester composites. The load-displacement behavior of specimens shows nearly linear elasticity up to the yield point. A sudden drop of the stress after the maximum yield stress and the rapid occurrence of failure indicate the loss of composite integrity for both composite types. Prior to the yield point, F1S1 exhibited nearly double the compression strength of F1S2. Kelvar-49 based composites reached the yield strength with small compressive deformation compared to Boron-based ones. It can be also found that predications of Rosen model in both shear and extension modes are found to disagree with the experimental data. The oscillating nature of the load-displacement curves immediately after yielding and continuous drop of load up to failure is the common feature for all the tested composites (ref to Fig. 2). When a composite material is subjected to a compressive load, several mechanisms contribute to failure, and the major ones are: matrix yield followed by fibre micro-buckling, local fibre micro-bucking within an elastic matrix, shear failure, and pure fibre compressive failure.

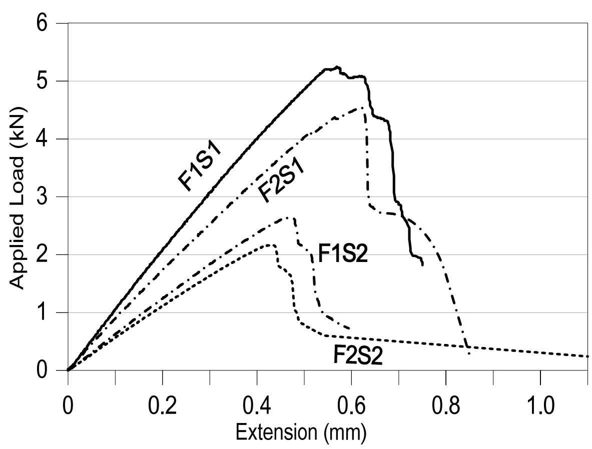

Fig. 2: load-displacement curves obtained from the compressive tests of the composites.

Table 2: Theoretically predicted values of mechanical properties of the composites

Loading Type	Model	(Unit) *Notation*	Composite type			
			F1S1	F1S2	F2S1	F2S2
Tension	ROM	(MPa) σ_{C1T}	2563.8	2545.0	1722.7	1824.3
		(GPa) E_{C1T}	245.3	244.8	93.9	100.2
	Modified ROM	(MPa) σ_{C1T}	2359.4	2340.6	1586.4	1703.8
		(GPa) E_{C1T}	225.7	225.2	87.1	93.4
	Eshelby's Model	(MPa) σ_{C1T}	2711.9	2686.0	1851.6	1567.6
		(GPa) E_{C1T}	262.5	258.4	101.7	86.1
	Self-Consistent Model	(MPa) σ_{C1T}	1746.5	1729.8	1192.5	1174.9
		(GPa) E_{C1T}	169.0	166.4	65.5	64.5
Compression	Rosen – Extensional	(MPa) σ_{C1C}	32960.0	27290.0	20290.0	16810.0
	Rosen – Shear	(MPa) σ_{C1C}	3280.0	5670.0	3280.0	5670.0
	Xu-Reifsnider Model	(MPa) σ_{C1C}	118.2	57.3	95.0	45.3
Transverse	IROM	(MPa) σ_{C1B}	370.1	238.3	162.8	110.3
		(GPa) E_{C1B}	8.7	5.6	3.84	3.1
	Modified IROM	(MPa) σ_{C1B}	584.2	378.6	163.4	114.8
		(GPa) E_{C1B}	13.8	8.9	3.9	3.2

Table 3: Experimentally measured mechanical properties of the composites

Composite	Tensile		Compressive		Flexural	
	σ_{C1T} MPa	E_{C1T} MPa	σ_{C1C} MPa	E_{C1C} MPa	σ_{C2B} MPa	E_{C2B} MPa
F1S1	237	22.9	106	10.8	316	7.5
F1S2	235	22.6	51	4.8	205	4.8
F2S1	162	8.9	85	6.1	135	3.2
F2S2	159	8.8	41	2.9	95	2.2

F1:Boron; F2:Kevlar; S1:Polyimide; S2:LM Polyester

During pultrusion process, not all fibres are perfectly aligned in uniaxial direction. Such fibre misalignment plays an important role in affecting the compressive strengths. In addition, matrix may hinder the specimen's ability to bend by making load contribution to the fibres. The misaligned fibres begin to buckle when the matrix is yielding. The matrix surrounding the fibres harden after yielding occurs. This failure process repeats itself and this mechanism is responsible for the oscillating nature of the load-displacement curve.

Transverse Flexural Properties

The prediction of flexural properties is based the assumption that all fibres are aligned parallel to one another and the composite is loaded in the perpendicular direction of the fibres. The IROM equation assumes that the fibres and matrix are equally stressed. Such predictions are in practice complicated by uncertainties about in situ strengths, interfacial properties, residual stresses, etc. Consequently, these models tend to overestimate the properties, particularly at high volume fraction concentration where correlation effects become important [19]. Fig. 3 shows the experimentally obtained stress-strain curves from the three-point bending tests for the four thermosetting composites under normal orientation. They showed that the tested composites failed gradually and strains at maximum stress remained nearly the same. F1S1 exhibited highest strength and flexural modulus, followed by F1S2, F2S1 and F2S2. Kevlar-49 based composites generally failed gradually at comparatively larger deflections.

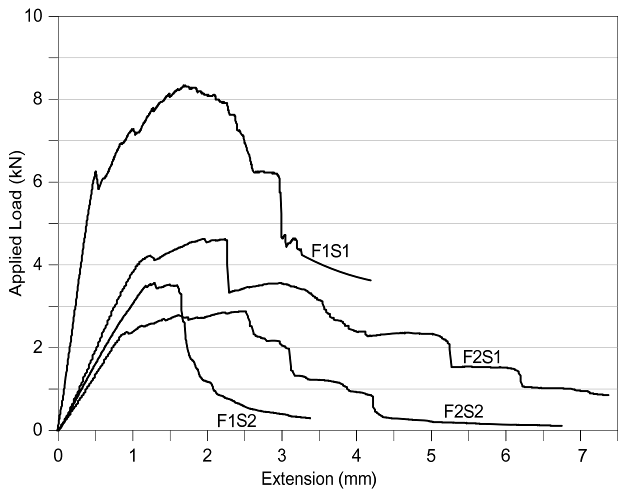

Fig. 3: Typical load-displacement curves obtained from flexural tests of the prepared composites.

5. Comparison of model predictions

Table 4 shows a comparison of the experimentally obtained values of tensile, compressive and flexural strengths with respect to the properties of their respective constituents. The theoretically calculated tensile strengths are an order of magnitude higher compared to those obtained experimentally. The ratio between experimental tensile strength values to the fibre strength is nearly constant for all the four thermosetting composites (approximately 0.10). This clearly indicates that the tensile strength is controlled by fibre alone and the matrix contribution is negligible. The compressive strengths obtained experimentally are only about 10% lower than those estimated using Xu-Reifsnider model [13] for all the composites. This small difference can be attributed to the expected deficiencies including fibre bundling, interfacial debonding, waviness, misalignments, material nonlinearity and void formation, etc. reducing the strength of composites. Experimental data indicates that the flexural strengths obtained are about 14-17% lower than the corresponding theoretical values using IROM model for all four composites.

Table 4: Comparison of model predictions with the measured properties of composites.

Composite	Tensile Measured / ROM	Compressive Measured / XR model	Flexural Measured / IROM
F1S1	0.092	0.89	0.85
F1S2	0.092	0.89	0.86
F2S1	0.094	0.90	0.83
F2S2	0.094	0.90	0.86

ROM: Rule of mixtures; XR: Xu-Reifsnider; IROM: Inverse rule of mixtures

6. Conclusions

Experimental studies were carried out to evaluate the mechanical properties of selected Boron and Kevlar-49 thermosetting composites with 61% fibre fraction. Tensile strengths of the four thermosetting composites were predicted using ROM, modified ROM, Halpin-Tsai and self-consistent models. The predicted tensile properties are approximately an order of magnitude higher than the experimentally obtained values. The predictions of compressive strenghs by Xu and Reifsnider model are in very good agreement with the experimental values for all composites whereas Rosen model predictions are extremely high. The compression strengths of thermosetting composites are significantly lower than their tensile strengths. Kevlar-49 composites exhibited lower strength in flexure and compression compared with Boron composites. The strength of composites using Polyimide matrix are higher than that using LM polyester. The flexural properties obtained using IROM model are about 15% higher than the experimentally obtained values. The compressive strengths are the lowest compared to tensile and flexural values. The modulus in tension is the highest for all the composites.

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