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A method for characterizes the interface between carbon fiber and epoxy resin: three-parameters exponential pattern

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Abstract

A new method for characterizing the interfacial performance between fiber and matrix was introduced in this paper, which was called three-parameters exponential pattern method (TPEPM). TPEPM belongs to neither macro- nor micro-measurement method. It was able to characteristic both macro- and micro-interfacial performance of composites efficiently at the same time. It could be attributed to meso mearsurement method. After verified by traditional method (short beam shear), it proved that the method of three-parameters exponential pattern has high reliability. The periodic time of experiment was shorter and could obtain more effective datum than before. The process of producing specimens as well as the procedure of testing was described detailedly. The way of testing the shear strength was according to the following formula:$\tau = P/bl$. In this formula, τ is the shear strength (MPa), P the shear force (N), bl express the area of adhesion. The dispersion pattern graph of shear strength (τ) and adhesion area (s) was made. It can be found that they have the similar variation regularity with exponential curve, which was proved by a great deal of experiment. The three-parameters exponential curve was fitted by the method of least squares. The variable regularity of the parameters τ_0, τ_1 and λ were compared with interlaminar shear strength (short beam shear). It was proved that the method was reliable.

Keywords: Carbon fiber; Interface; Exponential pattern; The method of least squares; Fitted

1. Introduction

A strong and durable interface is required between the fiber and matrix for the resulting composite to have good mechanical properties. Hence it is necessary to have better measurement techniques that are capable to quantifying the level of interfacial adhesion between the fibers and matrix. Characterization techniques of interface mechanical properties between carbon fiber and matrix could be divided into macro- and micro-measurement. Macro-measurements included short beam shear (also called three-point bend test) [1–4], Iosipescu shear [5,6] and cross direction tensile test [7], etc. And at least three different micro-mechanical test methods have been developed in order to determine the strength of the interface in high-performance polymer-based composites [8]. The test methods are fragmentation [9–19], pull-out [20–24] and microbond tests [2,19,25–27], etc. The schematic of these methods were shown in Fig. 1. All of them were have their merits and drawbacks. Prepare for the specimens, which were tested by macro-measurements would need a long time for solidifying periodicity and large quantity of materials were consumed for producing samples. Only could them be acquired a little effective data. The situation of micro-measurements, however, they often give different results even with the same fiber/matrix systems prepared under identical condition. One reason for this is that the state of stress in different tests may be different but a more fundamental problem is that the determination of fiber stress or strain using these test methods is usually indirect. It normally relies on assumptions about material properties and the use of analytical equations, which may or may not give an accurate picture of the state of stress in the system.

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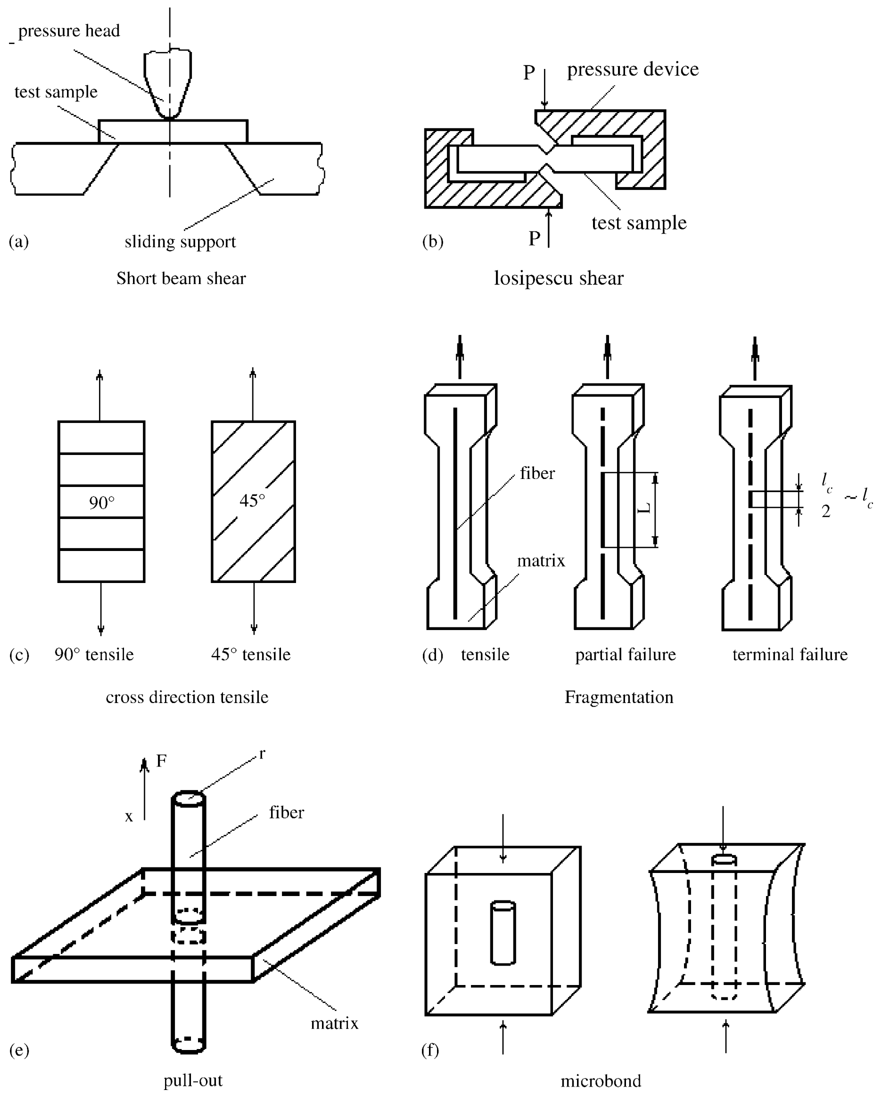

Fig. 1. Schematic method for testing interfacial strength of CF composites.

In this paper it will be introduced a new method for characterizing the interfacial performance between fiber and matrix, which it was called three-parameters exponential pattern method (TPEPM). This method belongs to neither micro- nor macro-measurements. It indicated that TPEPM probably belongs to a meso measurement. How to produce specimens as well as the procedure of testing were described detailedly in this paper as well as the steps of fitting exponential curve by least squares techniques. The reliability of TPEPM was verified by comparing with other characteristic method.

2. Fundamental

The specimen of unidirectional lamination composite was made by carbon fiber and epoxy resin, which was shown in Fig. 2. The area of adhesion between the two laminations was only the part that represented by the shadow in Fig. 2.

The conventional way of using the test is to increase the stress along the carbon fiber axis on the two layers of the specimen, at this moment would be occurred shearing action, the shear strength could be calculated by the following formula (1):

$$\tau = \frac{P}{bl} \tag{1}$$

In this formula, τ is the shear strength (MPa), P the shear force (N), b the adhesive width (mm) and l the adhesive length (mm). The area of adhesion (s) could be calculated by $s = bl$. Shear strength (τ) and adhesive area (s) made the graph of dispersion pattern that was shown in Fig. 3. It can be found that they have the similar variation regularity, which was proved by a great deal of experiment. For the distribution of data resembled exponential curve through observation measurement, the equation of regression was decided using exponential curve. Consider of when $s \to \infty$, as the adhesive area increasing, the shear strength would approach to a high-

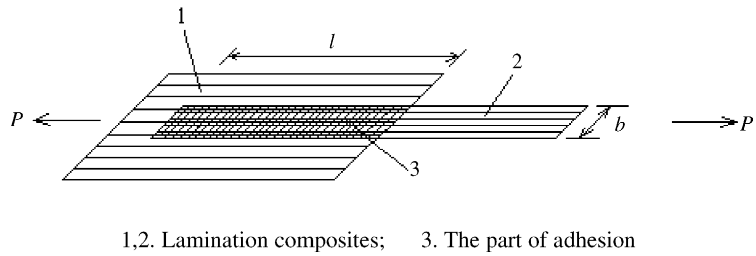

Fig. 2. The specimen of meso composite for experiment principle: (1 and 2) lamination composites; (3) the part of adhesion.

positive constant, this denoted the interlaminar shear strength (ILSS); when $s \to 0$, the value of shear strength approximated interface shear strength (IFSS) between fiber and matrix. And then a three-parameters-which were undetermined exponential equation could be created, just like formula (2)

$$\tau = A + B\exp(-\lambda s) \tag{2}$$

In the formula, A, B and λ are undetermined coefficients.

The values of A, B and λ acquired by simple regression analysis according to formula (2). After the curve fitting, obtained the correlation coefficient R^2 by the fitting curve, check the significance and reliability. As $s=0$, $\tau=\tau_0$, then $\tau_0=A+B$, its physical conception could be approximated evaluation the IFSS between fiber and matrix; as $s=\infty$, $\tau=\tau_1$, then $\tau_0=A$, its physical conception was the shear strength of macroscopy composite, interlaminar shear strength, i.e. different surface performance of fibers and resins decided different values of τ_0 and τ_1. If the treated carbon fiber could improve the interfacial performance of composite, the values of τ_0 and τ_1 will be increased. The curve expressed exponential relationship because of the interfacial tension between the interfacial films. The larger adhesive area decided the greater interfacial tension. So λ is the correlated variable corresponding the strength of interfacial tension. The greater value of λ result in the greater influence of shear action by interfacial tension.

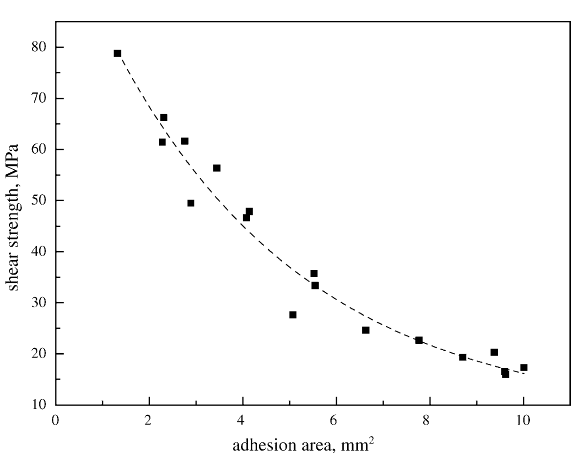

Fig. 3. The data point dispersion pattern about shear strength and adhesive area.

3. Materials and methods

3.1. Prepare for specimen

The fiber used for this study was conventional PAN-based fiber (Jilin, China). The matrix used was E618 epoxy resin (Wuxi, China). The interlaminar shear strength of composite specimen was tested by the universal-testing machine (Changchun, China).

The meso composite specimen should be ready made two-layers-adhesion shape. According to the following method could obtain a group of specimens taken on different adhesive areas.

First, The continuous bundle of carbon fibers were impregnated into epoxy resin, then were enwind the first layer around a cylinder metal mandrel by lapping machine, the schematic plan was shown in Fig. 4. The bundle of fibers in this layer should be close contact arrange and ensure laid full of the cylinder metal mandrel seamlessly and uniformly.

Teflon separate layer should be laid on the first fiber layer before enwinding the second, one side of the separate layer was paralleled with the bus bar of cylinder metal mandrel, the other side was leaned few angle (about 7°) to the bus bar. A few areas of mandrel sidewall were remained without separate layer for adhesion, the schematic plan was shown in Fig. 5, the shadow section remarked the Teflon separate film and the white section remarked the adhesive zone between the double layers. Thus, different length of adhesive ribbon was formed due to the leaning deposited Teflon separate film.

When the second fiber layer enwinding, the separation between adjacent fiber bundles was about 5 mm. After enwinding fibers the cylinder metal mandrel was put into oven waiting for heat-curing solidification.

In succession, cut the solidified composite along with the bus bar and take it down from the mandrel. After smoothing the lamellar composite, put the second fiber layer upwards. And then cut-off the double sides of every bundle of carbon fiber of the second layer along the fiber arranged direction, it was shown in Fig. 6. A group of meso composite specimens were acquired which have different adhesive areas. According to Fig. 7, the paper intermediate plates were adhered to the two heads of every sample; one head is the first fiber layer (close arrange) the other head is the second fiber layer (single bundle).

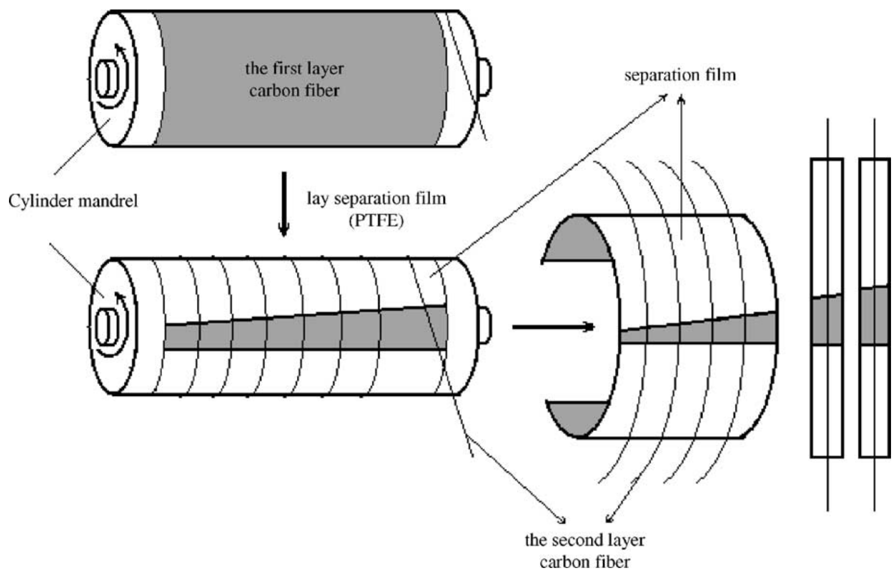

Fig. 4. The process chart of producing meso composite specimen.

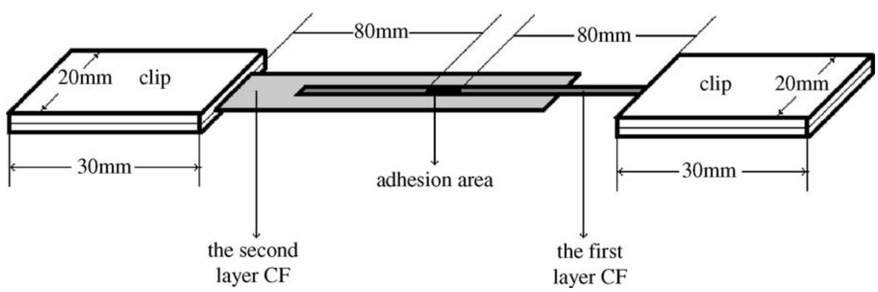

Fig. 5. Specimen of meso composite for tensile test.

3.2. Curve fitting

Used the method of least squares fitting the curve through Matlab software. For example, there were 22 datum in Fig. 3, the procedure as following:

(a) analysis this model. As $\lambda \to \infty$, $\tau = A$; as $\lambda = 0$, $\tau = A + B$. Due to $B > 0$ and $\exp(-\lambda s)$ is high-positive from beginning to end, the numerical values of this function are monotonic. So A less than all of the output datum values, i.e., less than the minimum of τ among these test datum. It could be found that the minimum value of τ was 15.9, i.e., $A < 15.9$.

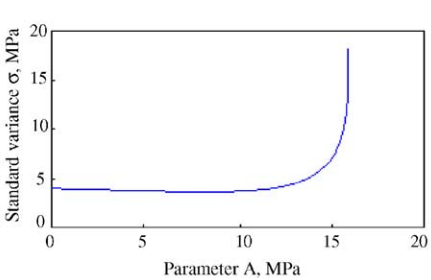

Fig. 6. The curve of A and standard deviation σ for regression.

(b) Evenly divided A to 1000 portion from 0 to 15.9, calculated B, λ and the standard deviation (σ) of τ which corresponded with each A through the method of least squares, it was shown in Fig. 6. Look for the point of

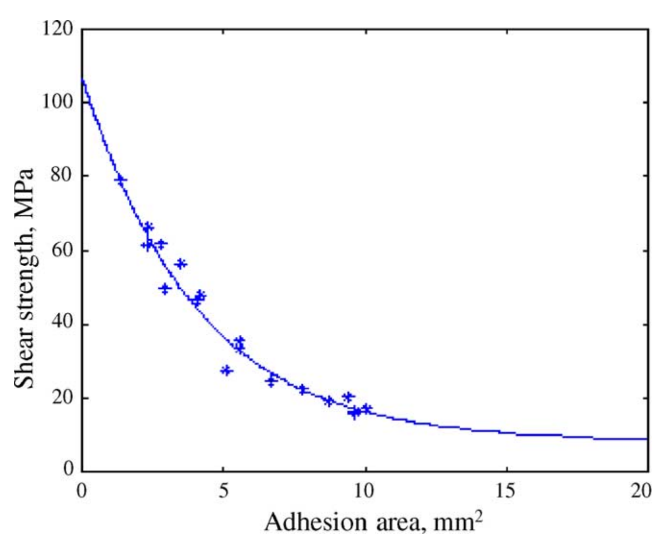

Fig. 7. The regressed curve for minimum standard deviation.

Table 1
The comparison of ILSS and three-parameter model for composite specimens with different interfacial properties

Processing Time (min)	ILSS (MPa)	τ_0 (MPa)	τ_1 (MPa)	λ
0	60.58	106.4101	8.2362	0.2491
4	65.41	116.8615	10.0417	0.2431
8	69.70	127.5812	11.3647	0.2376
12	75.48	135.4052	12.1478	0.2374
15	78.32	139.1349	13.2863	0.2327

minimum standard deviation from curve, determined the value of A. The conclusions from the group of datum were $\sigma = 3.6237$, $A = 8.2362$.

(c) $B = 98.1739$, $\lambda = 0.2491$, they were identifiable through the method of least squares. The equation could be written as following:

$$\tau = 8.2362 + 98.1739 \exp(-0.2491 s) \quad (3)$$

The fitting curve was shown in Fig. 7 It could be obtained the correlation coefficient (r) from the fitting curve, i.e., $r = 0.9014$. In this experiment the quantity of specimens was 22 and examine level was 0.001, looked-up the correlation coefficient critical value $r_{0.001} = 0.6524$ from coherent references. For the correlation coefficient value acquired from fitting curve much greater than critical value, so the regression equation was significant and the exponential curve of fit was acceptable.

3.3. Examine reliability

For examining the reliability of the three-parameters exponential pattern method, a contrast test was necessary. The virgin carbon fibers were treated by plasma method with different processing time, 4, 8, 12 and 15 min. According to the method that introduced in this paper and traditional method (short beam shear), respectively, produced carbon fiber/epoxy resin composite specimens. Compared with the variable regularity of testing results, interlaminar shear strength (short beam shear) and the parameters τ_0, τ_1 and λ (three-parameters exponential pattern), respectively. It could be seen in Table 1 that as increased plasma treating time the ILSS of short beam shear enhanced, so as to τ_0 and τ_1. It could be proved that three-parameters exponential pattern method characteristic interfacial performance of composite was reliable.

4. Conclusion

After verified by traditional method (short beam shear), it proved that the method of three-parameters exponential pattern has high reliability and was able to characteristic both macro- and micro-interfacial performance of composites efficiently. The periodic time of experiment was shorter and obtained more effective datum than before. It has much practical value and would be well developed in the future.

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