MEASUREMENT AND INFLUENCE FACTOR'S ANALYSING OF INTERFACIAL STRENGTH OF COMPOSITES①

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ABSTRACT A Nicalon SiC fiber reinforced 7740(Corning Code) boro-silicate glass composite was made by slurry-infiltration-hot-pressing process. The interfacial strength(τ_s) was measured using pyramidal diamond indentation method and micro de-bonding method respectively. The relationship between interfacial strength and various factors, such as, the heat-exposure time(t) of the fiber at 600 ℃ in air, the fiber diameter(D_f), and the spacing from the tested fiber to its nearest neighbouring fiber(S_m). The agreements and the difference of the two methods were discussed. It was discovered that, in the experiment range, τ_s increased as t and S_m rising and decreased as D_f increasing. In addition, the relationship between interfacial strength and fracture toughness(K_{Ic}) was also studied, it was also discovered that a relatively stronger interfacial strength led to a lower fracture toughness of composite.

Key words composites interfacial strength SiC fiber

1 INTRODUCTION

Interfacial strength includes interfacial shear strength and interfacial tensile strength, but under general condition the former is indicated as the interfacial strength unless specially pointed out. Interfacial strength is one of the most important factors that determine the composite behaviors, especially the mechanical properties; It also reflects the microscopical mechanisms of composites such as the physical and chemical compatibility between fiber and matrix, interface reactions and interface microstructure in a most direct way.

Most as-reported methods of determining interfacial strength are indirect ones and the results are hard to reflect the actual interfacial strength of real composites. Nevertheless, both the pyramidal diamond indentation method developed by Marshall[1] in 1984 and the micro de-bonding method by Mandell[2] in 1986 can be used for measuring in-situ interfacial strength of composites, the former measures the sliding frictional stresses while the latter measures the de-bonding stresses directly and both apparatus were set up in our lab.

As we know, for the real composite, the interface compositions and structures are very complex, which are remarkably affected by fiber distribution, fiber alignments, fiber spacings, fiber diameters and interface defects, etc. And up to date, quantitative investigations of interfacial strength are still rare.

2 SPECIMEM PREPARATION AND EXPERIMENTAL METHODS

2.1 Specimen preparation

The tested composites were all manufactured by hot-pressing the mixtures of fiber and powdered matrix material. Nicalon SiC fiber was obtained from the Nippon Carbon Company. As the fibers were oxidized obviously above 600 ℃ in air, this temperature was chosen as the heat-exposing temperature and the soak time at this temperature were 5, 10, 15, 20, 25, 30 and 120 min, respectively. Matrix material 7740 (Corning Code) boro-silicate glass was first ball

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milled, cleaned and then screened through a 200-mesh sieve. The compositions and properties of 7740 glass and Nicalon SiC fiber are shown in Table 1 and 2 respectively.

Table 1 The compositions of 7740 glass and Nicalon SiC fiber

Constituents	7740 glass	SiC fiber
Free carbon		11.7
SiC		65.3
SiO_2	80.2	23.0
B_2O_3	12.5	
Na_2O	4.0	
Al_2O_3	2.2	
CaO	< 0.6	

Table 2 The physical and chemical properties of 7740 glass and Nicalon SiC fiber

Properties	7740 glass	SiC fiber
Density/ $g \cdot cm^{-3}$	2.23	2.55
Tensile strength/ MPa	100	2 500~ 3 000
Elastic modulus/ GPa	60	180~ 200
Thermal expansion coef./ $10^{-6}K^{-1}$	3.2	3.1
Fracture toughness / $MPa \cdot m^{1/2}$	0.83	–
Rupture strain/ %	–	1.5

The tape of as-received or head-exposed fibers was made by so called slurry-infiltration technology. After drying, the tape was cut into appropriate length then laid up in a graphite mould and hot pressed at 1 200 ℃ for 30 min under the pressure of 6.7 MPa in N_2 atmosphere. The hot-pressed blanks were cut into suitable sizes by a diamond saw and the slices of composite were then ground and polished for testing.

2.2 Measurement of interfacial strength

The principles of diamond indentation method are shown as Fig. 1. The calculating formula of interfacial strength τ_s is calculated as

$$\tau_s = F^2/(4\pi^2 \mu R^3 E_f) \quad (1)$$

where $\mu = (b - a)\cot\Phi$; $F = 2a^2H$; (here, H —the hardness of the fiber), E_f —the elastic modulus of the fiber(taken as 200 GPa), 2Φ — the angle between opposite edges of the Vickers indenter.

During test, the largest load applied to fiber end was 0.98 N, ten to fifteen seconds was taken for the load raising from zero to the largest and then it was held another ten seconds. The average value of ten times of measurements was regarded as the indentation load.

The schematic diagram of the measurement device self-manufactured for micro de-bonding method is illustrated as Fig. 2, its loading precision was 9.8×10^{-4} N and loading rate was controlled at 1.5 μm/ min. The load was taken as de-bonding load when fiber was just de-bonded from matrix, and the interfacial strength was calculated according to [3].

Fig. 2 Schematic diagram of micro de-bonding apparatus

2.3 Determination of fracture toughness

The test was carried out using a 32 mm-span (S) in three-point bend. The notched specimen was 4 mm × 8 mm × 40 mm (B × W × L) in size and the notch depth (a) was approximately 4 mm. The loading velocity was about 1 mm/min. And the calculation formula[4] is

$$K_{Ic} = \frac{P_q S}{4BW^{3/2}}[7.31 + 0.21 \times \sqrt{(S/W) - 2.9}]\sec\left(\frac{\pi a}{2W}\right) \times \sqrt{\mathrm{tg}(\pi a/2W)} \quad (2)$$

where P_q —load; B, W, L —width, thickness and length of the specimen. For each test point, four samples were tested and the mean value was taken as the result.

3 EXPERIMENTAL RESULTS AND DISCUSSIONS

3.1 Relationship between the interfacial strength and thermal exposure time of the fiber

Upon exposure of Nicalon SiC fiber at 600 ℃, the following reactions may happen[5]:

$SiC + 2O_2 = SiO_2 + CO_2 \uparrow$

$2SiC + 3O_2 = 2SiO_2 + 2CO \uparrow$

$SiO_2 + C = SiO(g) + CO \uparrow$

With the binder on the fibers being removed and a new protective layer of SiO_2 formed by reactions above, the chemical compatibility and wettability between the fiber and matrix were improved, therefore, the interfacial strength increased with the extending of thermal exposure time as shown in Fig. 3.

3.2 Relationship between the interfacial strength and the diameter of the fiber

The experimental result is shown as Fig. 4, in which S_m is the spacing between the tested fiber and its nearest neighbouring fiber, D_f is the fiber diameter, S_m/D_f may be regarded as relative fiber spacing. Fig. 4 reveals that, no matter how S_m/D_f is changed over a large range 0.1~0.70, the interfacial strength decreased rapidly as the fiber diameter increased. Taking the logarithm of D_f and τ_s, there is an approximate linear relationship between $\lg\tau_s$ and $\lg D_f$ (see Fig. 5), which could be expressed as:

$$\tau_s = AD_f^K \quad (3)$$

where K was −1.86~−2.30 which was calculated from experimental data

Equation (1) can be transformed to:

$$\tau_s = 8a^4HD_f^{-3}/[\pi^2(a-b)\cot\Phi E_f)] \quad (4)$$

For a given composite system, $8a^4H/[\pi^2(a-b)\cot\Phi E_f]$ can be regarded as constant and thereby equations (3) and (4) are in agreement in form.

However, as the interfacial strength is

Fig. 4 Relationship between interfacial strength τ_s and fiber diameter D_f

Fig. 5 Plot of $\lg \tau_s$ against $\lg D_f$
(Symbols are the same as those in Fig. 4)

influenced by many factors, the K value may varies with the change of systems, processes and measurement methods.

3.3 Relationship of the interfacial strength to the fiber spacing

Choosing those fibers, which are 18 μm in diameter while have various spacings with their individually nearest fiber, measuring their τ_s, then we can found that the interfacial strength has an approximate linear relationship to the fiber spacing, as demonstrated in Fig. 6. This relationship is in agreement with the conclusion obtained in Ref. [2].

The reason why the data scattered so much may be resulted from the difference between the real experiment condition and the model, at least there are the following factors varying from theoretic model: (1) S_m, reflects only one location on the circumference around a fiber; (2) the stress field surrounding a fiber is asymmetrical other than axis-symmetric; and (3) the neighbouring fibers around the tested one are distributed discontinuously. Ref. [6] had discussed the strain nonuniformity of the matrix between the neighbouring fibers. Supposing four fibers located at each vertex of a square respectively (see Fig. 7), the strain magnification along line AB could be deduced as:

$$\beta = 2 + 1/[1 + (2E_m/E_f)/(S/r)] \quad (5)$$

where S/r is equivalent to S_m/D_f in the present paper. So, similarly, with the increase of S_m/D_f, the strain magnification along line AB increases, that is in agreement with the conclusion of the present experiment.

Fig. 6 Effect of fiber spacing on interfacial strength
(by de-bonding method)

Fig. 7 Fiber square model[5]

3.4 Relationship between interfacial strength and fracture toughness

The relationship of interfacial strength and fracture toughness of SiC(f) – 7740 glass composite measured by indentation and micro de-bonding methods are shown in Fig. 8.

From Fig. 8, it can be seen that the fracture toughness decreases as the interfacial strength increases. Because, if a weaker interfacial bond

Fig. 8 Relationship between fracture toughness K_{I_C} and interfacial strength τ_s

(a) —measured by indentation method; (b) —measured by micro de-bonding method

exists, in the courseof cracking, when the crack tip meets the interface, the fiber is easier to de-bond and pull out. These mechanisms absorb fracture energy greatly, thereby, lead to a higher toughness. If the interfacial bond strength is higher, the fracture stress concentrated at the crack tip is not easier to make the fiber de-bonding and be pulled out, leading the crack to run through the fiber and the fiber to break down. Comparing the energy absorbed by fiber de-bonding and pulling out with the energy absorbed by breaking, it is apparent that, the former is far bigger. So, a weaker bond leads to a higher toughness, while a stronger bond resulting in a lower toughness, of course in a certain range.

The reason why the interfacial strength value in Fig. 8 differs almost by one order may be that τ_s in (a) was obtained from the frictional stress upon the fiber having slide a distance, while τ_s in (b) was calculated from the transient de-bonding force of the fiber. As frictional stress is smaller, the result is naturally reasonable and is in agreement with the results reported.

4 CONCLUSIONS

(1) Using the pyramidal diamond indentation method and the micro de-bonding method equipped with self-manufactured apparatus, the interfacial strength of the Nicalon SiC(f)-7740 boro-silicate glass composite has been successfully measured.

(2) Under our experimental conditions, the interfacial strength increases with extending thermal exposure time of the as-received fibers at 600 ℃ in air.

(3) For Nicalon SiC(f)-7740 glass composite, the interfacial strength decreases with increasing fiber diameter, while increases with increasing fiber spacing.

(4) For a given system, with the increase of the interfacial strength, the fracture toughness reduces and the material becomes brittle.

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