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The measurement and characterization of the interfacial toughness of Si_3N_4/BN composites by a three-point bending test

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Abstract

In this paper, a simpler method has been proposed for measuring and characterizing the interfacial toughness of Si_3N_4/BN composites. A sandwiching material, with one preset crack source connecting directly to a BN interphase in the middle of a single half-Si_3N_4 matrix, were designed and fabricated by sandwiching a thinner BN layer with two Si_3N_4 green bodies obtained by die compaction at room temperature. The BN layer had been made by tape casting. The sandwiched sample bars measured with $3\times4\times50$ mm^3 were cut and machined. The interfacial toughness of Si_3N_4/BN composites was measured by three-point bending test for pure BN interphase, and interphases modified by different amounts of Si_3N_4 or Al_2O_3. The interfacial toughness values were calculated based on three-point bending fracture mechanics model. The interfacial toughness values we obtained were 38.20, 104.03 and 116.14 J/m^2 for pure BN, BN + 15vol.%Si_3N_4 and BN + 25vol.%Si_3N_4 interphases; 46.86, 53.90, 73.64 J/m2 for BN + 16vol.%Al_2O_3, BN + 36vol.%Al_2O_3 and BN + 63vol.%Al_2O_3 interphases, respectively. When the amounts of modified Si_3N_4 or Al_2O_3 increased, the interphase was strengthened and crack deflection and propagation within interphase could not occur, the interfacial toughness was not obtainable for the corresponding samples, but it could be obtained by extrapolation based on the values obtained in each kind of interphase systems. The results show that the method is simple and effective for measuring and characterizing interfacial toughness, compared with some other related works in literatures.

Keywords: Composites; Interfaces; Toughness; Measurement; Si_3N_4–BN; Testing

1. Introduction

Si_3N_4 ceramic is a very promising, high-temperature structural material with its excellent mechanical properties, but due to its brittleness, its wide application has long been restricted. With the emergence of the two structures Si_3N_4/BN composites, i.e. laminated and fibrous monolithic ceramics,[1–4] the toughness of the Si_3N_4 has been greatly improved. Haiyan Liu et al.[5] made the multilayer Si_3N_4/BN ceramics, which has 430 MPa of average bending strength and about 6500 J/m^2 of average work of fracture. Halloran et al.[6,7] manufactured the Si_3N_4/BN composites with the two structures respectively, the materials had bending strength of about 400–600 MPa and work of fracture values of >4000 J/m^2. Huang et al.[8,9] also fabricated the materials with the two structures, the materials had bending strength of about 600–800 MPa and work of fracture >4000 J/m^2. Among all the studies mentioned earlier, most of them lacked of interface design and control. More often it was followed by manufacturing material, measuring and characterizing material properties, and finally evaluating interface by the properties of the materials. This was a very passive process to fabricate material. In order to design, tailor the material, and optimize its bending strength and work of fracture, we must know how to characterize interphases with different compositions and give a quantitative evaluation on its bonding strength. Usually, the fracture toughness of this kind of composite was characterized indirectly by measuring the work of fracture. Sometimes, it was also characterized by Single Edge Notched Beam (SENB) method. However, values obtained from both of the two methods cannot reflect the toughness of interface

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directly. Meanwhile, the reliability of the SENB method is influenced by notch retard effect and the highly anisotropy in composite due to the existence of multi weak interfaces. So the interfacial toughness, i.e. the interfacial strain–energy release rate or interfacial resistance, was proposed for characterizing the interfacial bonding strength. Unfortunately, data on interfacial toughness of the composite have been seldom reported in literatures. In addition, so far, there are few reliable methods for measuring interfacial toughness. Although Kovar et al [6] gave a method for measuring the interfacial toughness, the specimens they used were different with what the model based on, with which interfacial toughness was calculated,[10] and the method was not so reliable. Based on the model of Charalambides et al.,[10] Phillips et al.[11] measured the interfacial toughness of a laminated SiC/C composite using an SiC/C/SiC sandwich sample with a single interphase. However, the sample dimensions adopted by those researchers were too large ($3.5 \times 18 \times 140$ mm^3) for the sample to be easily manufactured and machined, making the method inconvenient for characterizing interfacial toughness and also limiting its flexibility. Besides the big size, the sample was notched and precracked under three-point bending, with a short loading span, before the test, but that process is difficult to control and also makes it very difficult to obtain a crack starting from the tip of the notch and just exactly reaches the interphase or deflects toward two sides a little there.

In this paper, we improved the method used by Phillips et al.,[11] and gave a method that is suitable for measuring and characterizing interfacial toughness of the Si_3N_4/BN composite, which is simple and easy to be extended. By using this method, the interfacial toughness of the BN interphase systems strengthend by Si_3N_4 and Al_2O_3 respectively was measured and characterized.

2. Experimental

2.1. Experiment principle

Based on the model of the fracture of laminated composites under three-point bending given by Phillips et al.,[12] a single-interlayer type of sandwiching material, with one preset crack source connecting directly to a BN interphase in the middle of a single half-Si_3N_4 matrix, was designed and fabricated for measuring and characterizing interfacial toughness. The upper and lower Si_3N_4 matrixes height were kept as equal as possible to fix the effect of h_1/h_2 on the phase angle ψ, i.e. to make the ratio of shearing to opening stress-intensity factors tended to be constant.[11,13,14] The three-point loading system of the sample is shown in Fig. 1. According to the model,[12] the through-thickness cracks occur in the centre of the beam and the interfacial cracks propagate symmetrically from the centre, also a debonded layer between the through-thickness crack and the tip of an interfacial crack cannot undertake load. The compliance corresponding to the loading stage is calculated according to the remained section as shown in Fig. 1. In addition, it was assumed that the value of the critical interfacial toughness G_{ic} is a constant, uniform throughout the specimen.[11,12]

From Fig. 1, the formula for calculating the interfacial toughness can be deduced. Because the interlayer is just in the middle of the upper and lower Si_3N_4 matrixes, it is exact the position of the neutral axis. In region ①, the beam can carry stress, there exists corresponding deflection curve equation:

$$\frac{d^2y_1}{dx^2} = -\frac{Px}{2\Sigma_c} \tag{1}$$

where y_1 is the displacement from the neutral axis and Σ_c the beam stiffness of region ①.

In region ②, only the half side Si_3N_4 matrix layer is assumed to carry the applied stress, so it is related with the following equation:

$$\frac{d^2y_2}{dx^2} = -\frac{Px}{2\Sigma_s} \tag{2}$$

where y_2 the displacement from the neutral axis of the lower Si_3N_4 matrix, Σ_s the beam stiffness of the matrix layer in region ②.

There exist the following boundary conditions:

$$y_{1|x=0} = 0$$

$$\frac{dy_1}{dx}|_{x=(L-a)} = \frac{dy_2}{dx}|_{x=(L-a)}$$

$$y_{1|x=(L-a)} = y_{2|x=(L-a)}$$

$$\frac{dy_2}{dx}|_{x=L} = 0$$

According to the partial differential equations of (1) and (2), and the earlier-mentioned boundary conditions, we can obtain the relationship between the displacement of the central loading point and the propagating crack length.

$$y = \frac{P}{6\Sigma_s}L^3 + \frac{P}{6}\left(\frac{1}{\Sigma_c} - \frac{1}{\Sigma_s}\right)(L-a)^3 \tag{3}$$

since $y = CP$, hence

$$C = \frac{1}{6\Sigma_s}L^3 + \frac{1}{6}\left(\frac{1}{\Sigma_c} - \frac{1}{\Sigma_s}\right)(L-a)^3 \tag{4}$$

so the crack length can be expressed by compliance.

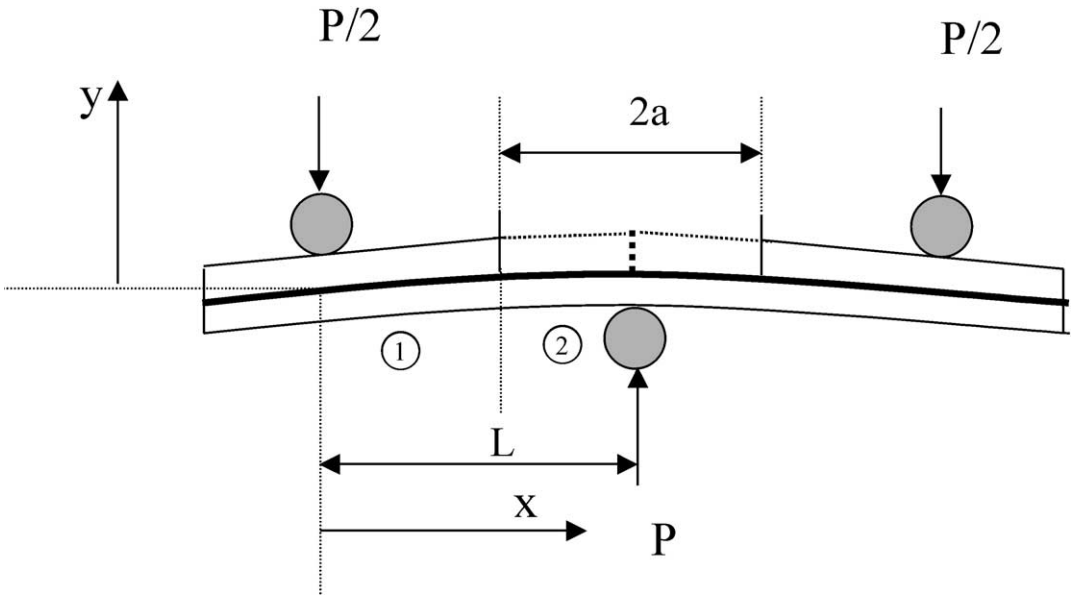

Fig. 1. The schematic of loading and remained effective section for the three—point loading system.

$$a = L + \left[\frac{\Sigma_c}{\Sigma_c - \Sigma_s}\left(6\Sigma_s C - L^3\right)\right]^{1/3} \tag{5}$$

According to interfacial fracture mechanics, meanwhile considering here the crack is assumed to be propagating symmetrically on either side of the central notch, interfacial toughness can be expressed as:

$$G_i = \frac{P^2}{4b}\frac{dC}{da} \tag{6}$$

From Eq. (4), an expression for the first derivative of C with respect to a can be written as:

$$\frac{dC}{da} = -\frac{1}{2}\left(\frac{1}{\Sigma_c} - \frac{1}{\Sigma_s}\right)(L-a)^2 \tag{7}$$

combining Eqs. (6) and (7), we can have

$$G_i = -\frac{P^2}{8b}\left(\frac{1}{\Sigma_c} - \frac{1}{\Sigma_s}\right)(L-a)^2. \tag{8}$$

In our experiments, the interlayer of sandwich specimen is very thin, so the beam stiffness can be approximately expressed as:

$$\Sigma_c = \frac{1}{12}bEh^3 = I_c E \tag{9}$$

where h is the thickness of the specimen, E the Young's modulus of the Si_3N_4 matrix, I_c the moment of inertia of the specimen.

Substituting Eq. (9) into Eq. (8), meanwhile, $\Sigma_s = I_s E$, the final interfacial toughness expression can be deduced.

$$G_i = -\frac{P^2}{8Eb}\left(\frac{1}{I_c} - \frac{1}{I_s}\right)(L-a)^2 \tag{10}$$

where I_s is the moment of inertia for the Si_3N_4 matrix layer.

When the loading state is the plain strain case, the Young's Modulus in the Eq. (10) will be substituted by $E' = E/(1-\nu^2)$, in which ν is the Poisson's ratio of the Si_3N_4 matrix.

2.2. Specimen design and preparation

A Si_3N_4/BN/Si_3N_4 sandwiched sample with a single BN interlayer and the thickness of the upper and lower sides of the Si_3N_4 matrices as equal as possible was designed for the present study. On one side of the matrix, a crack source directly connecting to the BN interphase was preset during material preparation. First, α-Si_3N_4 powders (Founder High Technology Ceramic Co., Beijing, China) combined with 8 wt.% Y_2O_3 (>9.99% purity, Hokko Chemical Industry Co., Ltd., Tokyo, Japan), 2.5 wt.% Al_2O_3 (>99.9% purity, Beijing Chemical Plant, Beijing) and 1.5 wt.% MgO (>99.9% purity, Beijing Hong Xing Chemical Plant,-Beijing) were milled in an ethanol medium. Then, 20 wt.%SiC whiskers dispersed by ultrasonic in ethanol media (TWS-400, Hokko Chemical Industry) were added to the mixture, and the milling step was repeated. The twice-milled mixture was filtered and dried, then sieved through a 60-mesh screen.

A green body with a Si_3N_4 matrix was obtained by die compaction. The BN interlayer was prepared by tape casting. Mixed powders with different interfacial compositions were prepared by incorporating BN with different amounts of α-Si_3N_4 or Al_2O_3 powders and milling the mixtures in ethanol for 24 h, then filtering, drying, and sieving the milled mixtures through a 60-mesh screen. The sieved powders were mixed with some

water, glycerin, and paraffin, milled, and incorporated into a 20 wt.% polyvinyl alcohol solution; this mixture was milled again and then degassed, under vacuum, at -1.013×10^5 Pa pressure. The homogeneous slurry was used for tape casting, and green sheets 40–60 μm thick was obtained.

Single-interface samples were prepared by sandwiching two green products of the Si_3N_4 matrix around a thinner BN interfacial sheet. The samples then were stacked, placed in a graphite die, and sintered, by hot pressing, at 1820 °C for 1.5 h in an atmosphere of N_2, under a pressure of 22 MPa. Initially, the heating rate was slow, to allow the binder in the interlayer tape to pyrolyze and burn out below 500 °C. Strict control of the heating rate was not necessary, because the interfacial layer was so thin. After sintering, the thickness of the interfacial layer was ~15 to 30 μm.

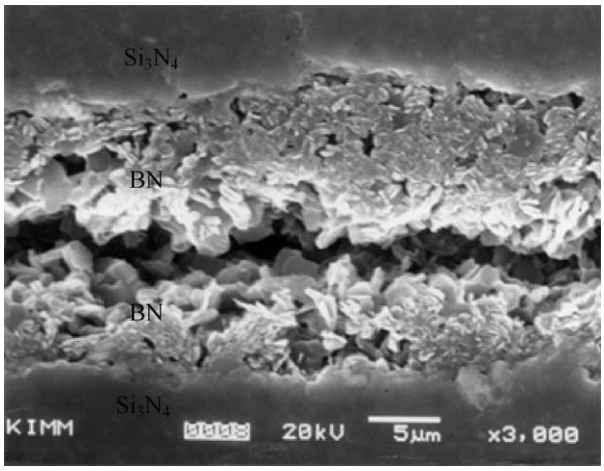

Fig. 2. SEM micrograph of the BN interphase crack.

2.3. Experimental method

Test samples measuring $3 \times 4 \times 50$ mm^3 were machined. Because the phase angle of loading, ψ, defined as the angle having a tangent equal to the ratio of the shearing to the opening stress-intensity factors,[13] was influenced by sample dimensions, that value varied with the thickness ratio, h_1/h_2 .Test errors resulting from fluctuation of the sample dimensions were reduced and the effect of phase angle ψ on interfacial toughness was fixed in the present study by making the thickness of the upper and lower beams as identical as possible. The sample was notched along the source of the crack to a certain depth, where it was near the interphase. The loading system as illustrated in Fig. 1 was adopted to

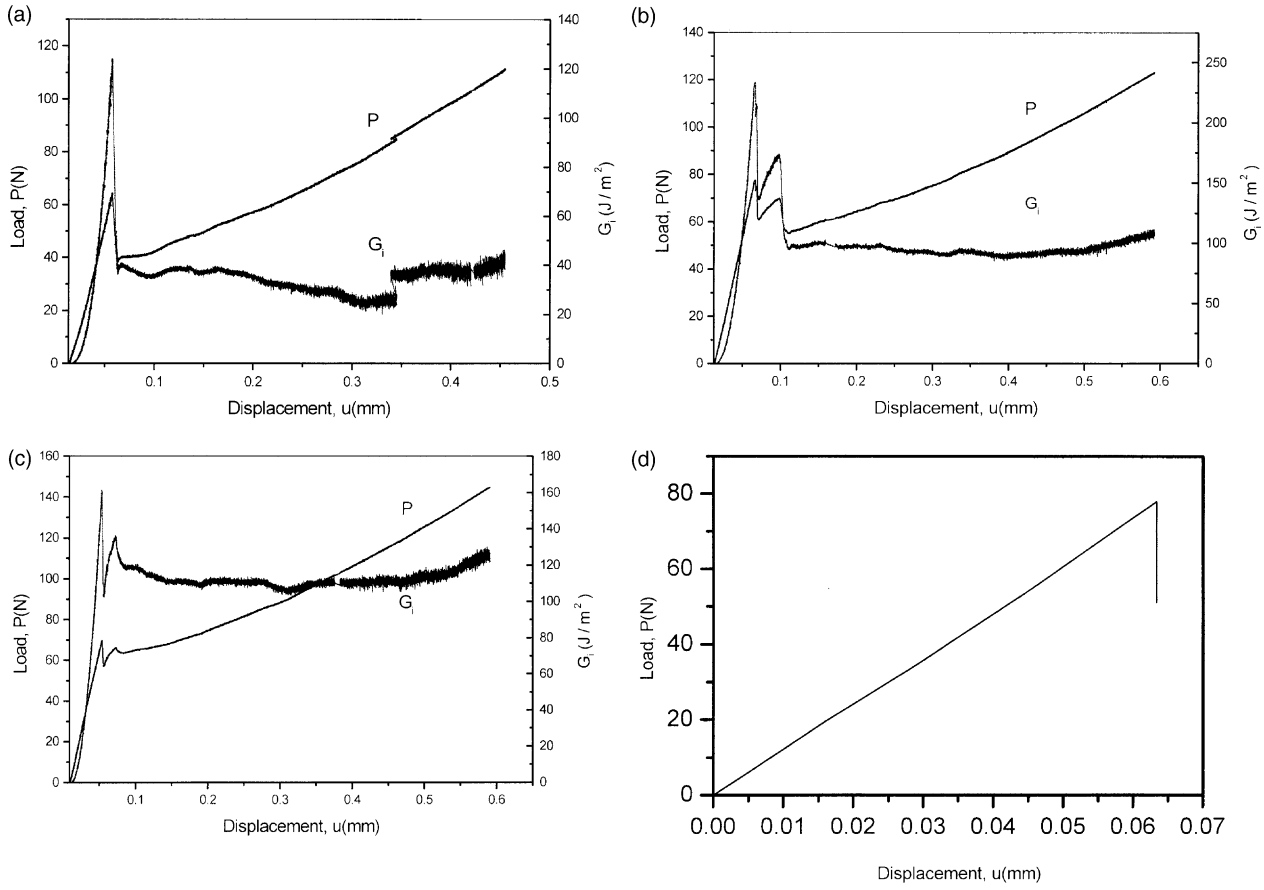

Fig. 3. The three-point bending test and interfacial toughness calculation results for the BN + Si_3N_4 interphase system. (a) BN; (b) BN + 15vol.%Si_3N_4; (c) BN + 25vol.%Si_3N_4;(d) BN + 50vol.%Si_3N_4.

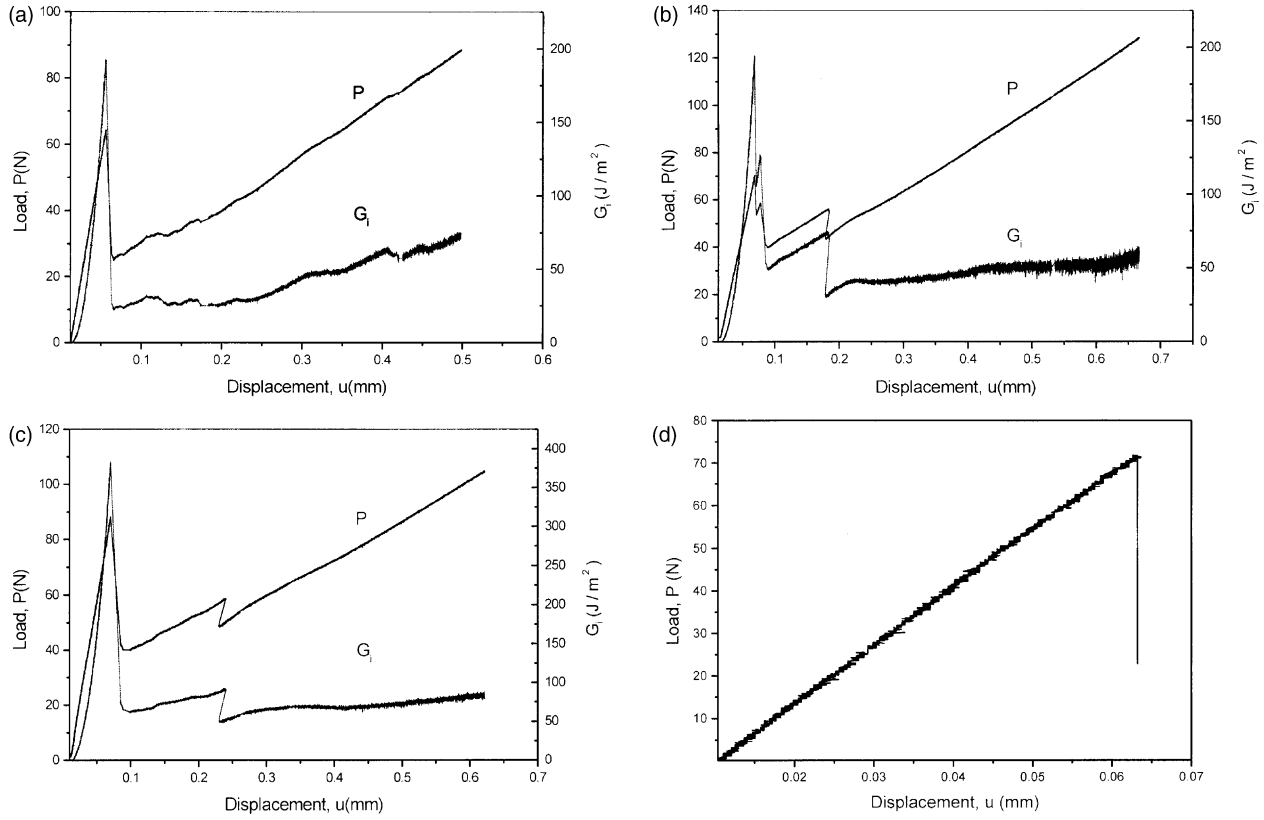

Fig. 4. The three-point bending test and interfacial toughness calculation results for the BN + Al_2O_3 interphase system. (a) BN + 16vol.%Al_2O_3; (b) BN + 36vol.%Al_2O_3; (c) BN + 63vol.%Al_2O_3; (d) Al_2O_3.

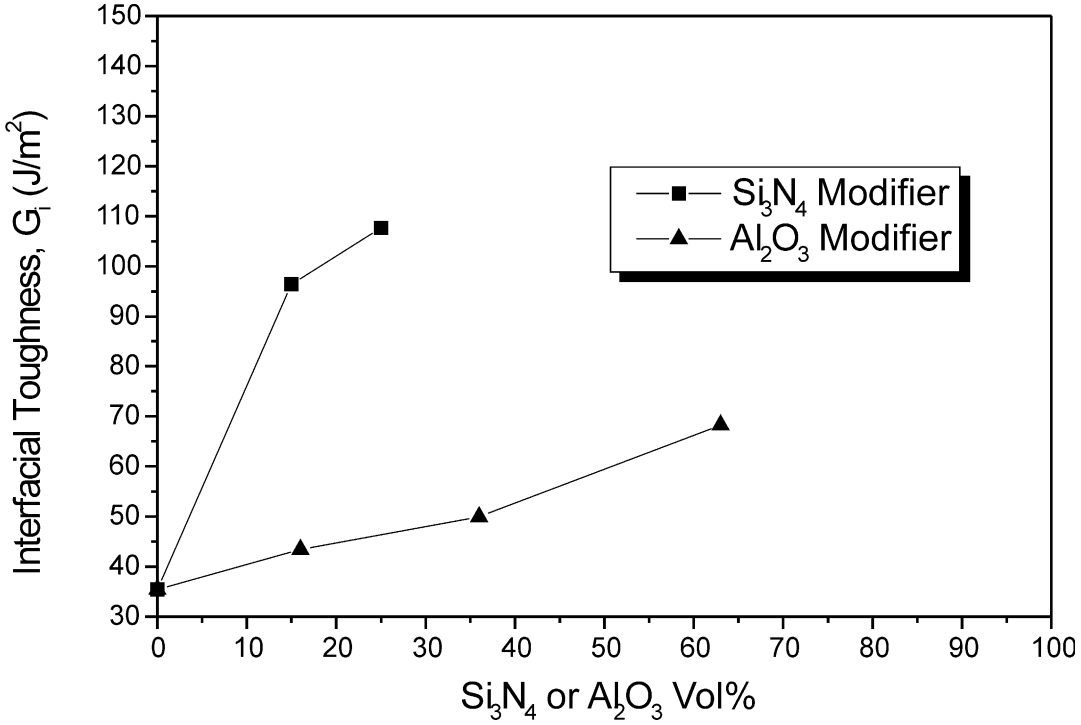

Fig. 5. The dependence of interfacial toughness on volume fraction of Si_3N_4 or Al_2O_3 modifier.

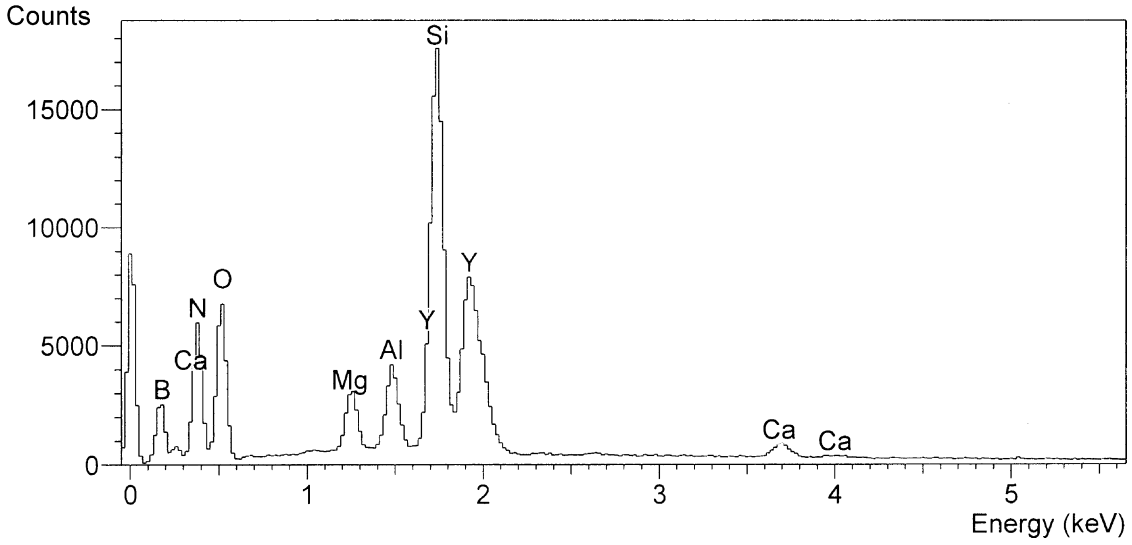

Fig. 6. The energy dissipation spectrum on any of one point in BN interphase.

carry out the tests, with 40 mm span. At least two or three samples were tested for each type of interphase composition for the loading experiments. A universal materials testing machine (model 2000, Shimadzu Corp., Kyoto, Japan) was used. To reduce the friction between roller and sample surface, the aluminum foil was used for the purpose. According to the sample size, the loading state was regard as plain strain case, the Poisson's ratio (ν) was taken as 0.27.[6]

Samples that has the same composition with the matrix Si_3N_4 was also prepared by the same process for measuring Young's modulus by three-point bending test. The sample is 4 mm wide and 20 ratio of span to thickness, the tests were conducted with 40 mm loading span. The average value was obtained from the results of 20 samples.

3. Results and discussion

The experiment results indicate the crack deflection and propagation occurred inside the interphase, rather than at the interface between matrix Si_3N_4 and BN interphase (Fig. 2), this also confirmed the results obtained by Kovar et al. [6] The load-displacement curves with different interphases of BN + Si_3N_4 system (Fig. 3) show that crack deflection and interfacial crack propagation occurred in the interphases of BN, BN + 15vol.%Si_3N_4 and BN + 25vol.%Si_3N_4. As the Si_3N_4 modifier increased to 50vol.%, the crack in thickness went across the interphase directly, leading to catastrophic fracture. This was because the interfacial toughness now was too large to allow crack deflection and propagation in the interphase. According to the Eqs. (5) and (10), the interfacial toughness versus displacement curves of the samples with the BN, BN + 15vol.%Si_3N_4 and BN + 25vol.%Si_3N_4 interphases were obtained (Fig. 3). When crack propagation in interphase began, the interfacial toughness tended to be a constant. Its values for the three interphases were obtained by taking an average value within the corresponding stable region, they were 35.42, 96.45, and 107.67 J/m^2, respectively. The interfacial toughness of the specimen without crack deflection and propagation in interphase could not be obtained by the same way.

The load–displacement curves of the samples with interphases modified by different amount of Al_2O_3 (Fig. 4) indicates that crack deflection and propagation occurred in the interphases of the BN + 16vol.%Al_2O_3, BN + 36vol.%Al_2O_3 and BN + 63vol.%Al_2O_3. In addition, the extent of the load descending was larger than that of the BN interphase modified by Si_3N_4. Therefore

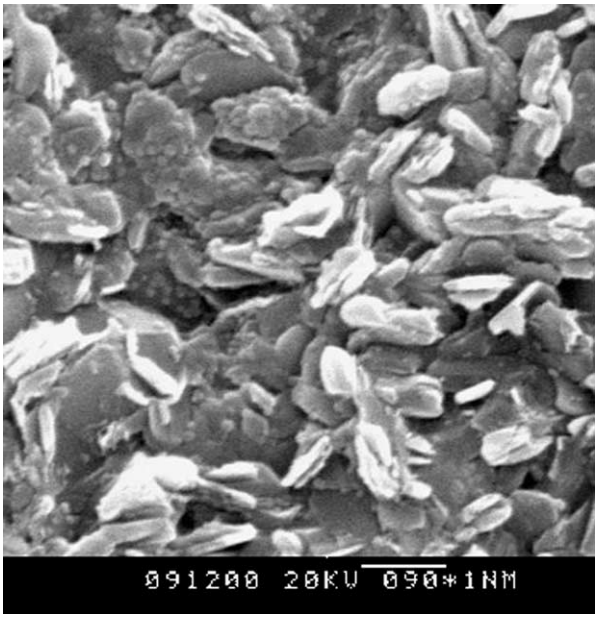

Fig. 7. The SEM micrographs of BN interphase.

the interfacial toughness versus displacement curves of these three interphases were obtained according to the Eqs. (5) and (10) (Fig. 4). Similar with the $BN + Si_3N_4$ system, there also existed an approximate constant interfacial toughness region after the abrupt load descending for each specimen in the $BN + Al_2O_3$ system. The interfacial toughness of this three interphases were obtained by taking an average value within each of these regions, they were 43.44, 49.97 and 68.27 J/m^2, respectively. The interfacial toughness value increased with the increase of volume fraction of Al_2O_3, as the interphase consisted of pure Al_2O_3, its strength was too high to allow inside crack propagation, the crack in-thickness crossed the interphase directly resulting in brittle fracture.

The interfacial toughness for the $BN + Si_3N_4$ and $BN + Al_2O_3$ interphase systems are listed in Table 1, the

Table 1
The interfacial toughness measured by three-point bending test for two interfacial modifier systems with different interphase compositions

Interface system	$BN + Si_3N_4$				$BN + Al_2O_3$			
	Volume fraction of Si_3N_4(vol.%)				Volume fraction of Al_2O_3(vol.%)			
Interphase composition	0	15	25	50	16	36	63	100
Interfacial toughness (J/m2)	35.42	96.45	107.67	–	43.44	49.97	68.27	–

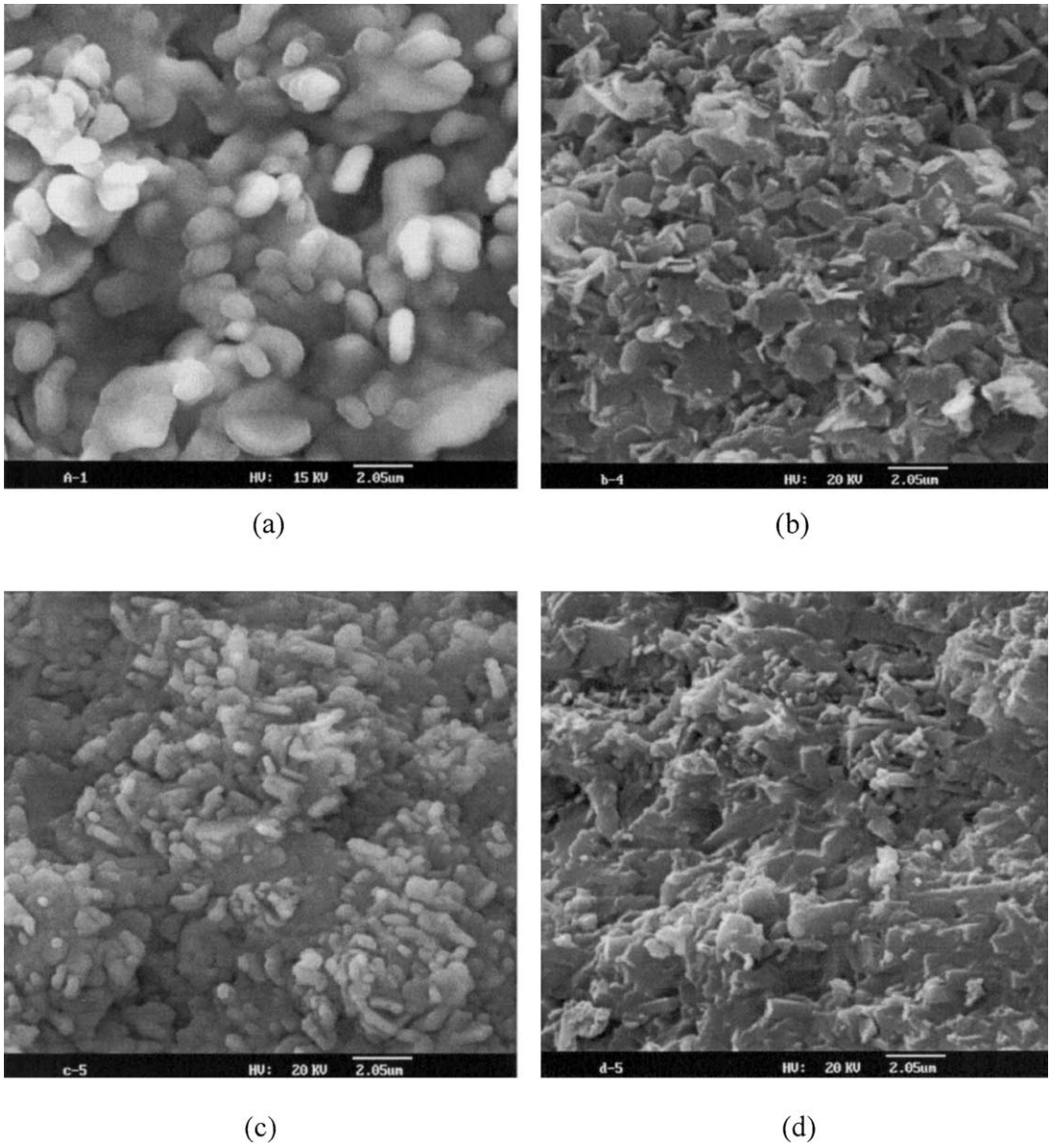

Fig. 8. The SEM interface fracture morphology of BN interphases modified by Si_3N_4. (a) Pure BN; (b) BN +15vol.% Si_3N_4; (c) BN + 25Vol% Si_3N_4; (d) BN + 50Vol% Si_3N_4.

results show that the values of the former system was bigger than that of the later in the whole composition range (Fig. 5). The Si_3N_4 strengthened interphase is much stronger than the Al_2O_3 strengthened interphase. This is agreement with the four-point bending test results.[15]

Studies on the microstructure of BN interphase have shown that grain boundary glass phase in Si_3N_4 matrix usually diffuses into the interhase,[16] the same case was observed in our samples. The energy dissipation spectrum (EDS) on any of one point in pure BN interphase indicates (Fig. 6) that there existed Si, Y, Al, Mg, Ca and O elements and some glass phase in matrix Si_3N_4 moved into the interphase. This was also confirmed by the SEM surface morphology of the BN interphase (Fig. 7), some of the plate like hexagonal BN grains were wrapped by glass phase and some of pores were filled. On the other hand, the micrograph also demonstrates that the BN interphase had a porous structure. This is the reason that doping with Si_3N_4 or Al_2O_3 could strengthen the BN interphase. The earlier-obtained results show the interfacial toughness was considerably influenced by the quantity of the doped Si_3N_4 or Al_2O_3. The regularities of the dependence of the interfacial toughness on volume percentage of Si_3N_4 or Al_2O_3 added into the BN interphase can be explained by the SEM micrographs of the fracture surface due to interface delamination (Figs. 8 and 9). As the volume percentage of Si_3N_4 increases, the BN interphase was densified gradually and quickly, this led to the increase of the interfacial toughness. When the percentage reaches to 50%, the interphase already seemed to be

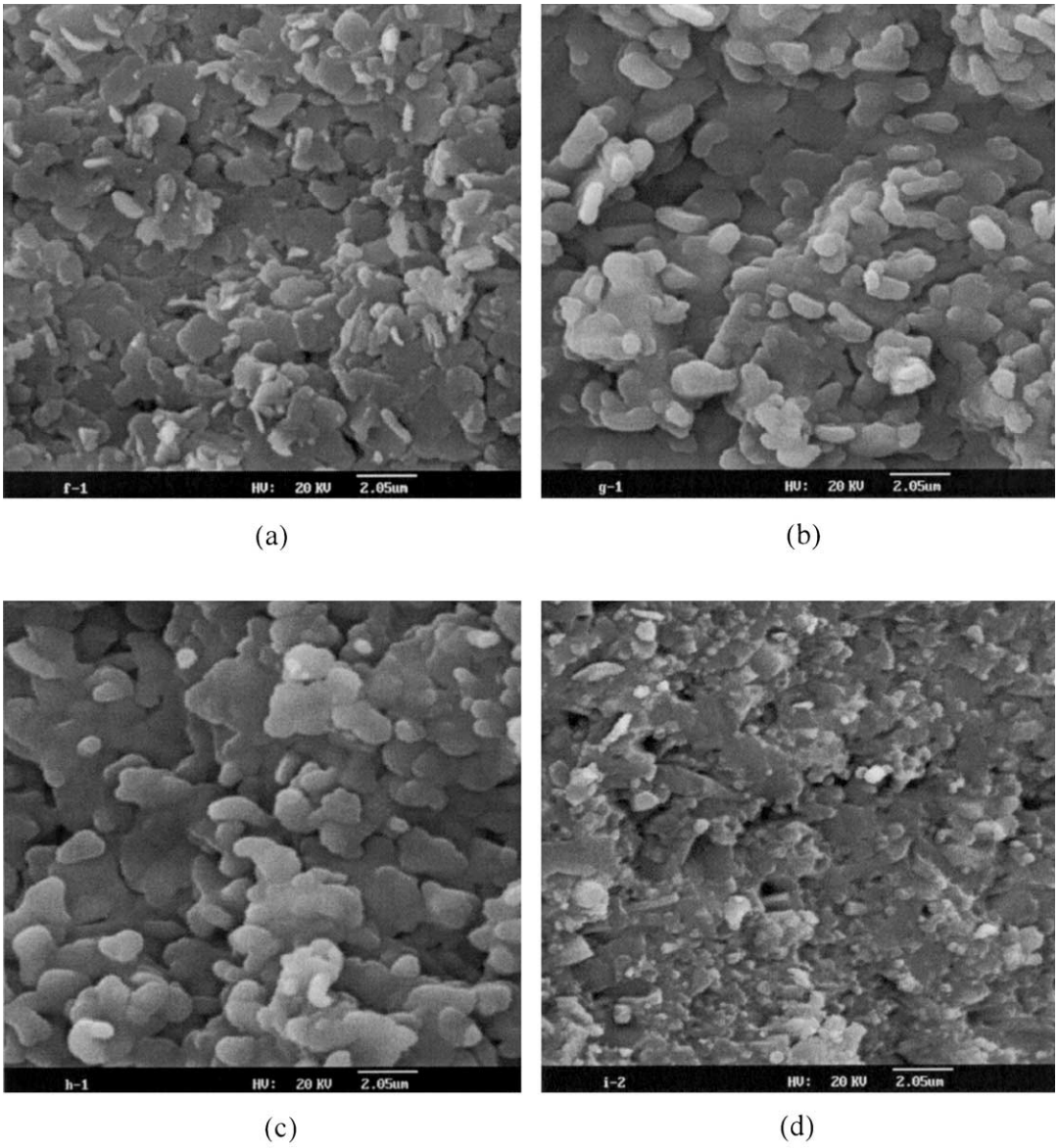

Fig. 9. The SEM interface fracture morphology of BN interphases modified by Al_2O_3. (a) BN + 16vol.%Al_2O_3; (b) BN + 36vol.%; (c) BN + 63Vol% Al_2O_3; (d) Pure Al_2O_3.

denser, and no eminent big pores existed (Fig. 8d), correspondingly, the interfacail toughness was too big to allow crack deflection and propagaton in the interphase. However, in the case of the BN interphase modified by Al_2O_3, the densification rate was not so fast as that of Si_3N_4 modified interphase. With the increase of volume percentage of Al_2O_3, the BN interphase was densified gradually and slowly, and the interfacial toughness also increased. Even if the percentage approaches 63%, the interphase still seemed to be loosen and porous (Fig. 9c), this is the reason why crack deflection and propagation still could happen in this interphase. As the interphase was pure Al_2O_3, the interfacial toughness was big enough to hinder the occurrence of crack deflection and propagation. From the microstructure analysis, it is also clear that the strengthening effect of Si_3N_4 is much bigger than that of Al_2O_3, at the same volume percentage of modifier, the interfacial toughness value of Si_3N_4 doped BN interphase is much higher that of Al_2O_3 doped one.

The earlier-mentioned results indicate that the method we used to measure and characterize interfacial toughness is viable. Compared with the method Phillips et al.[11] used for measuring the interfacial toughness of a laminated SiC/C composite, the present method allowed us to measure the interfacial toughness using smaller-sized samples, which were much easier to obtain. Thus, the method of presetting a crack directly connecting to the interphase is viable, making the experimental testing simpler and more flexible.

For the method presented here, the most important step is to determine the crack propagation length by compliance method (5). According to the equation, the crack length is also determined by the moment of inertia of half Si_3N_4 matrix (I_s). Because the matrix thickness (h_2) was obtained by measuring its thickness in different positions under common optical microscopy and then taking an average value, this introduced some errors due to irregularities of the matrix surface contacted to the interphase. Because the inside surface of Si_3N_4 matrix layer was not absolutely flat and its thickness had some fluctuations after the specimen was fabricated. The crack propagation length was considerably affected by the fluctuation of I_s and finally the interfacial toughness was influenced indirectly. Interfacial toughness was also influenced by the I_s [see Eq. (10)], so there existed a double influencing factors in three-point bending method, this was the cause of giving rise to deviation of the result. Another important factor that affected the measurement results was the Young's modulus of the matrix. For several samples, the measured E value could not stand for that of the real Si_3N_4 matrix in the sample for measuring interfacial toughness, leading to a result that was not reasonable. Sometimes the crack propagation length was negative, or the interfacial toughness value had a big fluctuation instead of tending to be a constant. Obviously, these were contradictory with the initial assumption for the three-point bending fracture model, and they were not correct results. To solve this problem, we concentrated all the deviations from the effecting factors upon the Young's Modulus in Eqs. (5) and (10). In this way, The E value was successfully adjusted to offset the earlier-mentioned influence. The adjustment was not finished until the interfacial toughness value almost tended to be a constant after the occurrence of crack deflection and propagation within interphase. To obtain a preliminary E value that is near to the prospective one, sometimes the critical condition, i.e. $a=0$ or $a=L$, was employed for the purpose according to Eq. (5). As $a=0$, it was corresponding to the initiation point of interfacial crack propagation, there was a sudden load descent in the load–displacement curve. For $a=L$, it was only applicable to the case in which the interfacial crack reached the ends of the loading span. Based on the obtained preliminary E value, the prospective E value was determined easily to make the interfacial toughness nearly tend to be a constant. Comparing with the four-point bending test to measure the interfacial toughness, the three-point bending test measurement results would be less accurate.

4. Conclusions

1. By considerably reducing sandwiching sample size, and presetting a crack source connecting directly to a BN interphase in the middle of a single half-Si_3N_4 matrix, a new method was presented for measuring interfacial toughness of the Si_3N_4/BN composite.
2. The interfacial toughness of the Si_3N_4/BN composite with different interphases strengthened by Si_3N_4 or Al_2O_3 were measured, the method turned out to be viable, and is simpler and more flexible compared with other method in literatures.
3. The interfacial toughness for Si_3N_4/BN composites with BN, BN + 15vol.% Si_3N_4, and BN + 25vol.% Si_3N_4 interphases was 35.42, 96.45, and 107.67 J/m^2, respectively. The composite with BN + 16vol.% Al_2O_3, BN + 36vol.%Al_2O_3, and BN + 63Vol% Al_2O_3 interphases was 43.44J/m^2, 49.97J/m^2 and 68.27J/m^2 respectively. For the both interphase systems, as the amount of the modifier Si_3N_4 or Al_2O_3 increased, the interphase was over-strengthened, the crack deflection and propagation no longer happened, and resulted in brittle fracture.
4. The method was affected by the fluctuation of the moment of inertia of half Si_3N_4 matrix (I_s) due to irregularities of the matrix surface contacted to the interphase. Also it was influenced by the

Young's modulus of the Si_3N_4 matrix in the sandwiching sample due to its indirectly determination. However, this was solved by concentrating all the deviations from the effecting factors upon the Young's Modulus in Eqs. (5) and (10). In this way, The E value was successfully adjusted to offset these influences.

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