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The characterization and measurement of interfacial toughness for Si_3N_4/BN composites by the four-point bend test

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Abstract

The interfacial toughness of Si_3N_4/BN composites with different interphase compositions has been characterized. A single-interlayer type of sandwich material Si_3N_4/BN/Si_3N_4, with one preset crack path connecting directly to the BN interphase in the center of one side of the Si_3N_4 matrix, was designed and fabricated. Sandwiched sample bars measuring 3 mm×4 mm×50 mm were cut and machined. The load–displacement curves for the samples were obtained using the four-point bend test, and the interfacial toughness was calculated, based on the model for a specimen with bimaterial interface. The interfacial toughness was measured for composites with interphases strengthened by Si_3N_4 or Al_2O_3 modifiers. The method was proved to be viable. The effectiveness of the method was further discussed and the reasons for its effectiveness explained.

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

1. Introduction

Silicon nitride (Si_3N_4) is a very promising, high-temperature structural material, with excellent mechanical strength; but brittleness limits its applications. Based on the structures of natural biomaterials, such as mollusk shells, bamboos, trees, and bones, the idea of biomimetic design has been introduced to materials processing within the past 10 years. The principle of structural design introduced by that idea is to decrease, as much as possible, the dependence of the mechanical properties of a material on its original natural crack population, by a mechanism of energy dissipation, thus producing a material with flaw tolerance.

Si_3N_4/BN composites with multilayer laminates and in situ synthesized fibrous monolithic structures have been fabricated according to the biomimetic principle.[1–4] Such composites possess higher apparent fracture toughness, and they also maintain a high level of strength. The design of the interphase is the most important factor in fabricating materials with such properties. Usually, the interfacial toughness, i.e., the interfacial strain–energy release rate or interfacial fracture resistance, is used to characterize the interfacial combining strength. Unfortunately, data on the interfacial toughness of Si_3N_4/BN composites with different interphase compositions have seldom been reported in the literature, especially for Si_3N_4 composites reinforced with SiC whiskers. Kovar et al.[2] measured the interfacial toughness of Si_3N_4/BN composites with different interphase compositions modified by Si_3N_4; in their experiment, a laminated sample with multiple interphases was notched at the middle position and then loaded, under four-point bending. However, the model with which the interfacial toughness was calculated was that of a bimaterial interface.[5] Thus, the interphase crack-propagation behavior was not accurately reflected. Based on the model of Charalambides et al.,[5] Phillips et al.[6] studied the fracture behavior of laminated SiC/C composites and measured the interfacial toughness of the composites using an SiC/C/SiC sandwich sample with a single interlayer. However, the sample dimensions adopted by those researchers (3.5 mm×18.3 mm×140 mm) and in another work by Howard et al.[7] (3 mm×20 mm×160 mm) were too large for the sample to be easily manufactured and machined, making it difficult to characterize the interfacial toughness and also

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limiting its flexibility. In addition, the sample was notched and precracked under three-point bending, with a short loading span, a process which is difficult to control with ceramic materials and which also makes it very difficult to obtain a crack that reaches the interphase; the sample tends to fracture directly instead of obtaining a precrack that reaches the interphase. This is the most important thing that limits the application of the method.

Based on the above-mentioned previous work, we improved their methods by adopting a sandwich specimen of small size, and by introducing a weak crack path connecting to the interphase for solving the precrack problem, and used the improved method for characterizing and measuring the interfacial toughness of Si_3N_4/BN composites with SiC whisker toughened Si_3N_4 matrix. Interfacial toughness is measured for various interphase compositions strengthened by Si_3N_4 or Al_2O_3.

2. Experimental

2.1. Experimental principle

Usually, two types of crack propagation occur: (1) stable propagation, also known as *crack creeping*, and (2) nonstable propagation, also called *crack bursting*.[6,7] What type of propagation will happen depends on the variation of interfacial toughness with position (possibly caused by microstructural inhomogeneities) and on factors dependent on the beam stiffness.[7] During stable crack propagation, interfacial toughness does not vary considerably along the interface. Under the displacement control of four-point loading, stable crack propagation occurs between two inner loading points after a critical load has been reached.[7] In such a case, the load is almost constant, but displacement increases continuously. For symmetrical propagation along both sides of an interphase, interfacial toughness can be expressed by the following equation,[7]

$$G_i = \frac{P^2 S^2}{8b}\left(\frac{1}{\sum_s} - \frac{1}{\sum_c}\right) \tag{1}$$

where G_i is the interfacial toughness (J/m^2), P the load corresponding to crack propagation (N), b the sample width (mm), S the spacing between the inner and outer loading lines (mm), $\sum_s$ the beam stiffness of the single-layer Si_3N_4 matrix (N·mm^2), and $\sum_c$ the beam stiffness of the single-interlayer sandwiched sample (N·mm^2). The interfacial toughness given by Eq. (1) is a transient one, because the load P is not absolutely constant due to the interphase ununiformity. The beam stiffness can be defined by the following integral:

$$\sum = \int E(y) y^2 dA \tag{2}$$

here, y is the displacement from the neutral axis, E (y) Young's modulus at displacement y, and A the cross-sectional area of the beam. For a sandwiched sample with a single, thin interlayer, the corresponding beam stiffness can be expressed approximately as

$$\sum = \frac{1}{12} bh^3 E = I_c E \tag{3}$$

where h is the total thickness of the sample (mm), b the sample width (mm), E Young's modulus for the Si_3N_4 matrix (kgf/mm^2), and I_c the moment of inertia of the sample (mm^4). It implies that the Young's modulus of the sample is approximately equal to that of the Si_3N_4 matrix layer.

Substituting Eq. (3) into Eq. (1) gives

$$G_i = \frac{P^2 S^2}{8bE}\left(\frac{1}{I_s} - \frac{1}{I_c}\right) \tag{4}$$

where I_s is the moment of inertia of the single-matrix beam contacting the two inner loading points (mm^4).

When crack bursting occurs, the critical interfacial toughness, G_{ic}, or critical load, P_c, corresponding to crack propagation, varies considerably with crack position, and compliance increases quickly under the constant roller displacement, so that the applied load, P, decreases with the crack advance. According to the crack-propagation criterion:

$$G_i \geqslant G_{ic} \tag{5}$$

when P_c decreases more rapidly than P, unstable crack propagation occurs, and the interfacial toughness is characterized by bursting initiation and termination loads:[7]

$$\bar{G}_{ic} = \frac{P_1 P_2 S^2}{8bE}\left(\frac{1}{I_s} - \frac{1}{I_c}\right) \tag{6}$$

Here, $\bar{G}_{ic}$ is the average value of critical interfacial toughness and P_1 and P_2 the bursting initiation and termination loads, respectively (N). P_e is the effective load (N), i.e., the geometric mean of the initiation and termination loads [$(P_1 P_2)^{1/2}$].

According to the sample dimensions, if it is in plane strain condition, E in Eqs. (4) and (6) will be substituted by the plane strain modulus $E' = E(1-\nu^2)$.

2.2. Sample design and material preparation

A Si_3N_4/BN/Si_3N_4 sandwich 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 sandwich, 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 were dispersed by ultrasonic in ethanol media (TWS-400, Hokko Chemical Industry) and 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 (Commercial Product, Chemical Reagent, average size 1 μm, Hexagonal), with different amounts of α-Si_3N_4 (the same powder with the matrix, average size 0.69 μm) or Al_2O_3 (the same with the additive Al_2O_3, average size 0.5 μm) powders and by 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 were obtained.

Single-interphase samples were prepared by sandwiching two round green plates (diameter 70 mm) 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, under a pressure of 22 MPa and an atmosphere of N_2. 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.

2.3. Experimental method

Test samples measuring 3 mm×4 mm×50 mm were machined (the ratio of height to width is 0.75). For this kind of sample, plane strain condition was adopted. Poisson's ratio (ν) of the Si_3N_4 has been reported as 0.27,[2] here we used this value approximately. The measured interfacial toughness is an average value corresponding to the average load in the stable propagation region for crack creeping and to the geometric mean of the initiation and termination loads for crack bursting. 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,[8] was influenced by sample dimensions, that value varied with the thickness ratio, h_1/h_2. When h_1/h_2 was increased gradually, ψ decreased, tending to level off as $h_1/h_2\rightarrow1$. Thus, the ratio of shearing to opening stress–intensity factors tended to be constant (Fig. 1).[8,9]

Test errors resulting from fluctuation of the sample dimensions were reduced in the present study, to make the thicknesses of the upper and lower beams as identical as possible. In accordance with the four-point loading model proposed by Charalambides et al.[9] and Cao and Evans,[10] the loading system illustrated in Fig. 2, with a 20 mm inner span and a 40 mm outer span, was adopted to conduct the tests. According to Charalambides et al.,[11] relatively small h/s values are preferred to minimize uncertainties associated with unknown frictional effects, values in the interval $0.25\leqslant(h/s)\leqslant0.5$ seem practical. In this case, the current study is satisfied with the requirement due to $h/s=0.3$. In addition, one kind of aluminum thin foil was employed as a media between the specimen and the loading rollers to reduce the friction coefficient in the loading system. The sample was notched along the preset source of crack to a certain depth by using an inner round cutting machine, where it was near the interphase. Three samples were tested for each type of interphase composition. A universal material

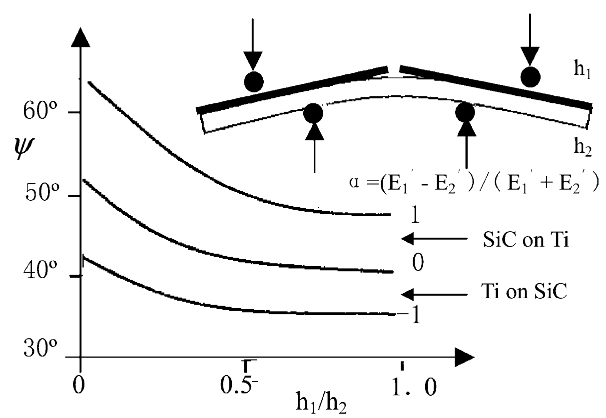

Fig. 1. The dependence of phase angle ψ on specimen size and Dundurs parameter in four-point bending test.

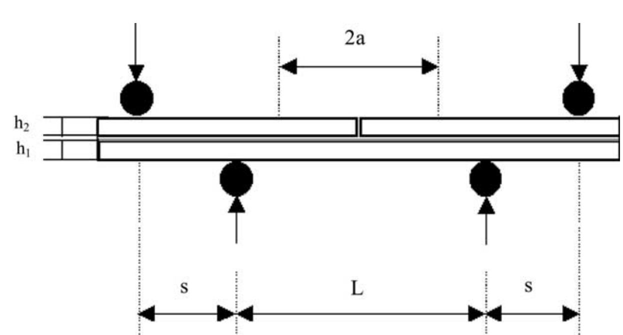

Fig. 2. The loading schematic of four-point bending test (The through-thickness crack initiates in the center of the specimen and the interfacial cracks propagate symmetrically from the center, the total crack length is 2a.)

testing machine (model 2000, Shimadzu Corp., Kyoto, Japan) was used for the loading experiments.

Samples that had the same composition as the matrix Si_3N_4 were also prepared for measuring Young's modulus by three-point bending test using the same method of fabricating the sandwich specimens. 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 load–displacement curves of the samples with pure BN and BN + Si_3N_4 interphases are shown in Fig. 3. With pure BN and 15 vol.%, 25 vol.% Si_3N_4 modifier added interphases, stable crack propagation occurred in the interphase. As the amount of Si_3N_4 modifier was increased, the load corresponding to crack propagation increased [Fig. 3(a)–(c)], and the interfacial toughness increased (Table 1). When the amount of Si_3N_4 added to the BN approached to 50 vol.%, no crack propagation occurred in the BN + 50% Si_3N_4 interphase; as in the case of monolithic ceramics, the sample fractured [Fig. 3(d)], and crack-propagation resistance at the interphase increased, making crack deflection impossible. The measurement of Young's modulus of the Si_3N_4 matrix layer gives an average value of 29 532 kgf/mm^2. Eq. (4) was used to obtain the interfacial toughness values related to the different interphase compositions (Table 1).

In the load–displacement curves of the Fig. 3(b) and (c), there exist overloading phenomena. This is because the notch was somehow not deep enough, and preloading process was also not enough for the crack initiated from the tip of the notch to reach the interphase. It still had to propagate a short distance along the weak crack source preset beforehand to arrive at the interphase, where it was then deflected and propagated within the interphase. It was the propagation in that short distance that led to the occurrence of overloading. Therefore, the preset weak crack source made the initiated crack from the tip of the notch easily propagate into the interface instead of a catastrophic fracture happening, which occurs more often under the condition without the preset crack source. Although we have made our efforts to make sure that the notch must be deep enough to be near the interphase, our cut machine could not be precisely controlled with its cutting length. For the sake of safety, several specimens were cut to a depth where it

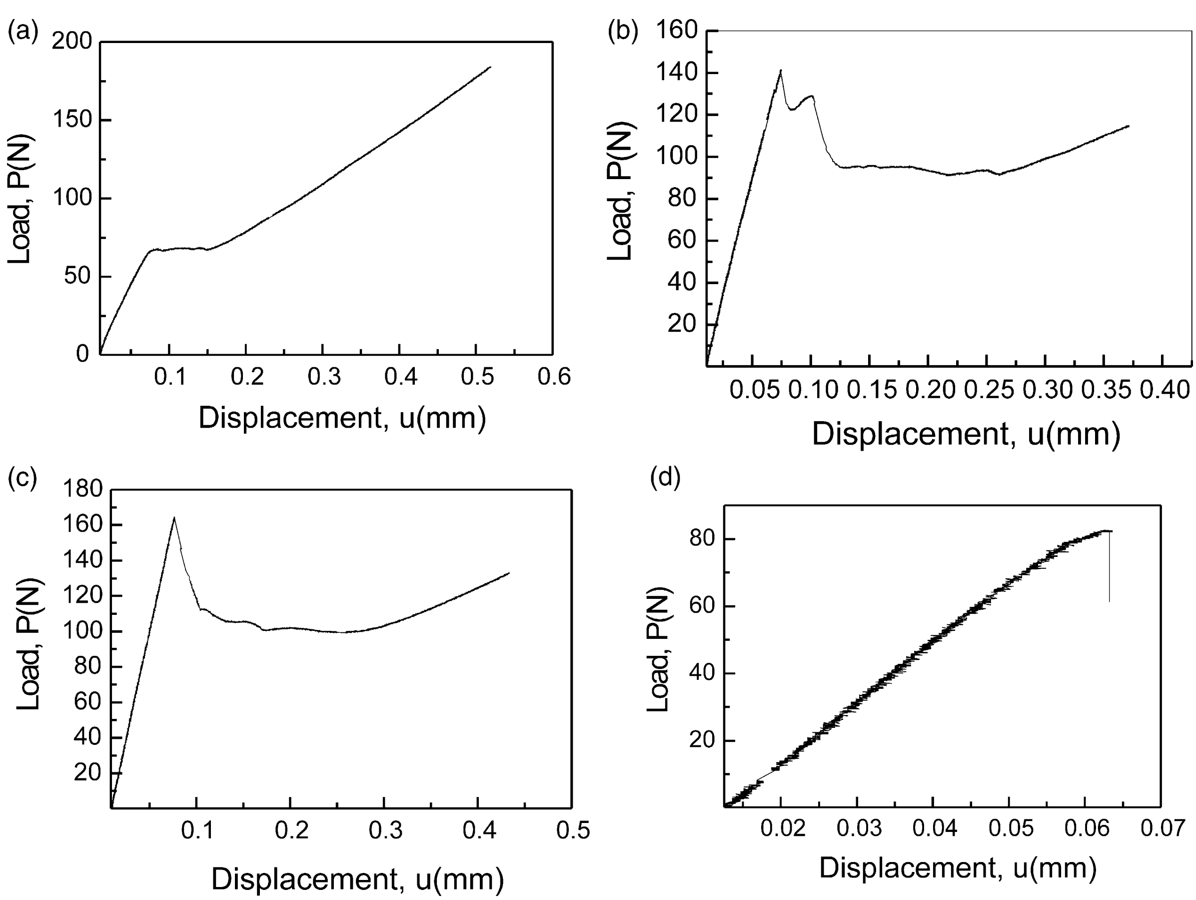

Fig. 3. The load–displacement curves of Si_3N_4/BN/Si_3N_4 sandwiching specimens with different composition of Si_3N_4 modifier. (a) BN. (b) BN + 15 vol.%Si_3N_4. (c) BN + 25 vol.%Si_3N_4. (d) BN + 50 vol.%Si_3N_4.

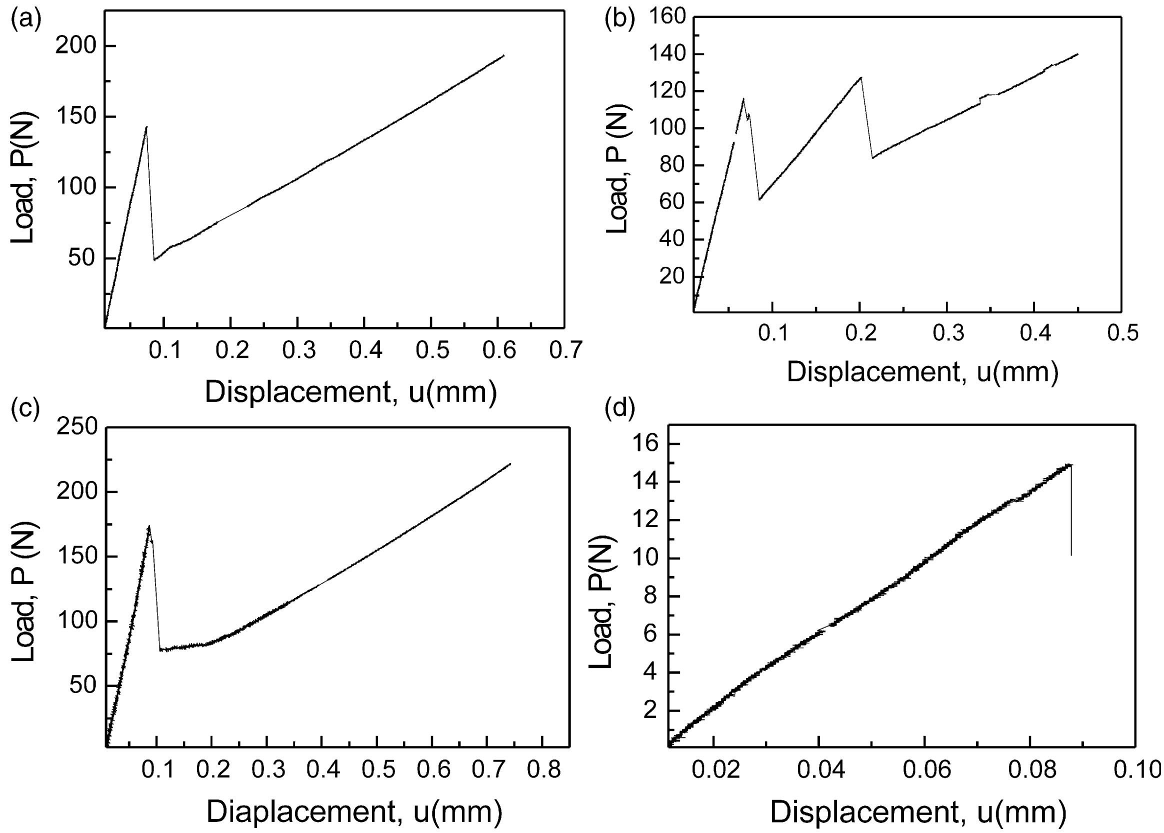

Fig. 4. The load–displacement curves of Si_3N_4/BN/Si_3N_4 sandwiching specimens with different composition of Al_2O_3 modifier. (a) BN + 16 vol.%Al_2O_3. (b) BN + 36 vol.%Al_2O_3. (c) BN + 63 vol.%Al_2O_3. (d) 100% Al_2O_3.

Table 1
Interfacial critical crack propagation loads and interfacial toughness of Si_3N_4/BN composites with different interphase composition modified by Si_3N_4 under four-point bending test

Parameter	Interphases modified by different volume fraction of Si_3N_4			
	0	15%	25%	50%
P (N)	74.79	107.41	114.42	–
G_i (J/m^2)	37.16	90.58	117.76	–

was still a short distance away from the interphase. Nevertheless, we made preloading before every formal test to initiate the crack from the tip of the notch and to propagate it to the interphase. On the other hand, in the case of the crack creeping, the load corresponding to the stable crack propagation should be the same with that of the sample without overloading, i.e., they should have the same plateau corresponding to stable crack propagation whether there exists the overloading phenomenon or not. Only the slope in the elastic loading region of the sample without overloading is lower than that of the same sample with overloading. Even if overloading happened, as load increased to the maximum point, it yielded and finally fell down to a load corresponding to crack deflection and propagation in the interphase [Fig. 3(b) and (c)]. According to Eq. (4), the overloading will not influence the measured interfacial toughness. So for the samples with crack creeping, there was no need to ensure in the process of preloading that the crack reached the interphase and deflected there.

The load–displacement curves of the Si_3N_4/BN/Si_3N_4 samples modified with different volume percentage of Al_2O_3 show that unstable crack propagation tended to occur when Al_2O_3 was the interphase modifier (Fig. 4). Similarly to addition of Si_3N_4, as the amount of added Al_2O_3 increased, the interphases were strengthened; the bursting initiation and termination loads increased and the interfacial toughness also increased gradually. In Fig. 4(a), there occurred one time of crack bursting with the BN + 16 vol.%Al_2O_3 interphase. With the increase of the modifier Al_2O_3, there occurred two times of crack bursting with the BN + 36 vol.% Al_2O_3 interphase [Fig. 4 (b)]. Even as the amount of Al_2O_3 reaches 63 vol.%, crack deflection and propagation still occurred with the BN + 63 vol.% Al_2O_3 interphase, giving two times of crack propagation, but different from the

Table 2
Interfacial critical loads and interfacial toughness of Si_3N_4/BN composites with different composition modified by Al_2O_3 under four-point bending test

Parameter	Interphases modified by different volume fraction of Al_2O_3			
	16%	36%	63%	100%
Effective load				
P_e (N)	80.14	86.08	94.13	
$\bar{G}_{ic}$ (J/m^2)	53.95	73.95	83.62	–

behavior of the former two interphases, in this case, first occurred crack bursting, second a short period of crack creeping. This implies the interphase properties were undergoing some transformation as it was strengthened by increasing amount of Al_2O_3 modifier gradually, and tended to have some similar interfacial behavior with the BN interpase and interphases modified by Si_3N_4. When the interface consisted entirely of Al_2O_3, the interfacial toughness was too high to allow interfacial crack advance, and the thickened crack crossed the interphase directly, resulting in brittle fracture.

The critical interfacial toughness of the Si_3N_4/BN composite with an Al_2O_3-modified interphase was obtained using Eq. (6) with crack bursting and (4) with crack creeping. An average value was taken for each kind of interphase that produced two times of crack bursting or first crack bursting and second crack creeping (Table 2). The interfacial toughness of the composite with the pure Al_2O_3 interphase was not obtainable, because no crack propagation occurred along the interphase during loading [Fig. 4(d)], but the value could be deduced by extrapolation, after several values of the Al_2O_3-modified BN interphase had been obtained.

In this case, due to occurrence of the crack bursting, interfacial toughness is influenced by the initiation load. We took measures to make sure that the crack reached the interhpase and deflected there during the course of preloading. Actually, after the weak crack source had been preset, the precrack process was much easier to do compared with that of Phillips et al.;[9] the crack was inclined to propagate along the crack source to the interphase and deflected there instead of giving brittle fracturing. The process was repeated for several cycles until the expected results were obtained.

The interfacial toughness values for the BN + Si_3N_4 and BN + Al_2O_3 interphase systems are listed in Table 3, the 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 in agreement with the three-point bending test results.[11]

The above-mentioned results indicate that the method we used to characterize the interfacial toughness is viable. The load–displacement curves corresponding to crack propagation in the interphase could be recorded during the bend test, because the interphase was still not much strengthened. Whether stable or unstable crack propagation occurred, the initiating load related to crack advance and the terminating load of crack bursting could be obtained from the curves. As the amount of interphase modifier increased, interfacial bonding was enhanced. Both the load corresponding to stable crack propagation and the effective load of unstable crack bursting tended to increase rapidly, and the values obtained for interfacial toughness followed the same trend.

In addition, the effect on the experimental results of residual stress resulting from the mismatch of two materials with different thermal expansion coefficients cannot be omitted with a multiple-interlayer laminated sample. On the other hand, this residual-stress effect can be eliminated for a sandwiched sample, because a BN interlayer sandwiched between two thick Si_3N_4 matrices is much thinner. In Charalambides et al.'s work, they thought that the residual stress existing in the thin bond layer does not contribute to the mixed mode fracture resistance.[12] BN is highly anisotropic on thermal expansion. The thermal expansion coefficient along *c* direction is much bigger than that of along *a* direction. During hot pressing, the BN plate grains are inclined to have an orientation parallel to Si_3N_4 matrix layer, i.e., the *a* axis is parallel to the matrix layer, and *c* axis is perpendicular to it. Because the thermal expansion coefficient of BN along *c* direction is also bigger than that of the Si_3N_4 matrix, there exist residual tensile

Table 3
The interfacial toughness measured by four-point bending test for two interfacial modifier systems with different interphase compositions

Interface system	BN + Si_3N_4				BN + Al_2O_3			
Interphase Composition	Volume fraction of Si_3N_4(vol.%)				Volume fraction of Al_2O_3(vol.%)			
	0	15	25	50	16	36	63	100
Interfacial toughness (J/m^2)	37.16	90.58	117.76	–	53.95	73.95	83.62	–

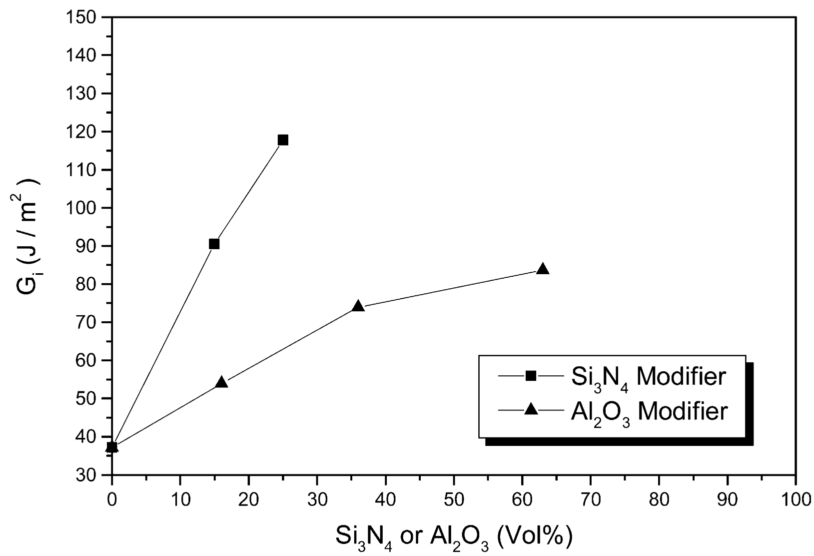

Fig. 5. The dependence of interfacial toughness on volume fraction of Si_3N_4 or Al_2O_3 modifier.

stresses in BN-containing interphase along c direction. This is beneficial for cleavage along the basal plane of BN. It is for this characteristic that BN is usually used as weak interface in composites. So even if there exists residual stress in BN-containing thin interphase, it was not considered in this case. Then, keeping the upper and lower matrices the same height as possible, i.e., $h_1/h_2 \rightarrow 1$, we can reduce the value of ψ and fix its effect on interfacial toughness, decreasing measurement error.

In our samples, the top and bottom layers are all Si_3N_4 matrix reinforced by SiC whiskers with randomly orientation, which can be regarded as linear elastic homogeneous and isotropic layers. Usually, the common feature of sandwich specimens devised for experimental determination of interfacial toughness is that each of them is homogeneous except for a very thin layer of second material which is sandwiched between the two halves comprising the bulk of the specimen,[13] According to Eqs. (4) and (6), the interfacial toughness depends on the elastic modulus of the Si_3N_4 matrix layer. If the matrix layer is inhomogenous, it means the Young's Modulus is dependent on the sample orientation. In this case, the interfacial toughness was strongly influenced by the determination of E value, and an interfacial toughness value that can reflect the real properties of the interface will be not available.

Eqs. (4) and (6) indicate that I_s has a significant effect on G_{ic}, because errors occurring during fabrication of the material and machining of the rectangular bars lead to thickness fluctuations of the unnotched half-Si_3N_4 matrix. This also influences the accuracy of the measurements. When one of the Si_3N_4 layers is much thinner than 1.5 mm, the value obtained for the interfacial toughness corresponds to that of another phase angle, which differs from the one of $h_1/h_2 \rightarrow 1$. If the specified dimensions during each stage of sample manufacturing and machining can be guaranteed, such influence is decreased.

Compared with the method used by Phillips,[9] 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 path directly connecting to the interphase is viable, making the experimental testing simpler and more versatile. The measurement results under such a method are credible, as long as the interphase is flat and the thickness of the upper and lower matrices almost the same.

4. Conclusions

1. Using a sandwich sample with small dimensions, and with a weak crack path preset, the method used for measuring interfacial toughness of the ceramic matrix composites was improved. The difficult point of precracking a crack in this kind of measurement was successfully solved.
2. The interfacial toughness of the interphases in Si_3N_4/BN composites strengthened by Si_3N_4 and Al_2O_3 was successfully characterized and measured by the improved method, making possible the design of interfaces and model numerical calculations for Si_3N_4/BN composites.
3. The results showed that there exists difference of interface fracture behavior between Si_3N_4 and Al_2O_3 strengthened BN interphases, the former is characterized with crack creeping and the later is mainly crack bursting.
4. The measurement results showed that the inter-

facial toughness of Si_3N_4/BN composites increases as the volume fraction of Si_3N_4 or Al_2O_3 interphase modifier increases; the improved method is proved to be effective.

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