Applicability of Fracture Toughness Concept to Fracture Behavior of Carbon/Carbon Composites*

Hiroshi HATTAA**, Yasuo KOGO***, Hideyuki ASANO**** and Hiroyuki KAWADA****

Carbon/Carbon(C/C) composites have attractive mechanical properties such as superior specific strength and high elastic modulus at high temperature exceeding 2000°C in an inert atmosphere. However, mainly due to lack of knowledge of design criteria, C/C composites have not been used in primary heat resistant structures. For example, almost no unified explanation has been given about the fracture behavior of C/C composites. The objective of this paper is to examine the adequacy of the linear elastic fracture mechanics (LEFM) as the fracture criterion of notched C/C composites. Thus the LEFM was tried to be applied to fracture behavior in tensile tests of double-edge-notched and compact tension specimens and in four-point bending tests of single-edge-notched specimens. It was found that the results of three kinds of fracture tests can be consistently and rationally explained in terms of the LEFM concept with the aid of R-curve behavior if the pre-crack length is long enough to be able to neglect the notch tip radius. From fractographic observation it was found that R curve behavior of C/C composites was mainly attributed to the fiber-bridging effect near the notch tip.

Key Words: Carbon/Carbon Composites, Fracture Criterion, Fracture Toughness, Stress Intensity Factor, Net Section Stress, Point-Stress Criterion, R Curve Behavior

1. Introduction

Carbon fiber reinforced carbon matrix composites, C/Cs, are unique materials possessing high heat resistance over 2000°C in an inert atmosphere as well as low density, high specific strength and stiffness, and superior thermal shock resistance. Because of these properties C/Cs have been expected to be applied to high temperature structures, such as space planes and hypersonic airplanes. However C/Cs have been rarely used in structures if high durability is required, mainly due to lack of data bases for structural design. Thus it has pivotal importance to establish fracture criteria of C/Cs in various situations.

As fracture criteria for structures including stress concentration sources such as cracks or holes, net stress criterion(1) and fracture toughness criterion(2) have been proposed. These criteria, however contradict each other because the former suggests complete notch insensitivity while the latter is on the premise of notch sensitivity. Fracture behavior of C/Cs has been generally discussed in comparison with that of ceramic matrix composites, CMCs, which exhibit similar fracture patterns. For example, Evans et al.(3)-(4) classified the fracture behavior of CMCs in three basic modes discussing the relation between fracture modes and constituent material properties of the CMCs. In these papers C/Cs exhibit the third fracture mode and the fracture toughness criterion was reported to be effective. In the same papers, they pointed out that C/Cs possess extremely low shear strength and thus prone to form a process zone near the notch tips by shear mode micro-fracture. This behavior leads to relaxation of stress concentrations around notch tips and thus C/Cs were said to show

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notch insensitivity. In other word, the fracture mechanics approach is not effective for them. Similar contradicting conclusions were reported from other authors(1).

In view of the above discussion fracture criteria of C/Cs are hardly established. In this paper the fracture mechanics criterion was attempted to be applied to various fracture behaviors of the cross-ply and quasi-isotropically laminated C/C composites. In order to observe fracture patterns in various situations, three geometrically different fracture tests were carried out.

2. Experimental Procedure

2.1 Materials

The C/C composites used in this work were fabricated by Acer Tech Co. by a processing route of the preformed yarn method(9). The C/Cs were of a lamination type with stacking sequences of 0°/90° (cross ply; CP) and 0°/45°/90°/−45° (quasi-isotropy; QI). The mechanical properties of these C/Cs are listed in Table 1.

The C/C composites were supplied by 300 mm square plates, from which specimens were cut into shapes shown in Fig. 1. The thickness of the specimens was 10 mm for the compact tension, CT, and single-edge-notched-bending, SENB, specimens and 3 mm for the double-edge-notched, DEN, specimens. The notches for all specimens were cut in by a diamond wire saw and finished into a notch tip radius equal to 0.15 mm. An Instron type testing machine, Autograph AG5000A by Shimazu Co., was used for the tests under a cross head speed of 0.1 mm/min. Acoustic emission, AE, signals were monitored during the tests to identify the onset of crack propagation near the notch tips.

2.2 Fracture toughness measurement

2.2.1 CT test To evaluate the dependence of fracture toughness parameters on the pre-introduced notch length, \( a \), test pieces with \( a/W \) equal to 0.4, 0.5, and 0.6 were prepared, where \( W \) is the width of the CT specimen. In the CT test, the loading and unloading cycle was repeated to obtain a crack resistance, \( R_\alpha \), curve. The energy release rate \( G_\alpha \) was determined by use of the compliance method:

\[
G_\alpha = \frac{P^2C}{2B} a
\]

where \( P \), \( B \), \( C \) and \( a \) are applied load, specimen width, compliance, and crack length, respectively. The compliance was calculated from the relation of \( P \) and the crack opening displacement, COD, which was obtained by a clip gauge attached at the notch mouth. The crack length was measured on an enlarged image obtained by a CCD camera. To identify clearly the crack propagation the specimens were thinly white-painted on their polished surfaces by emery paper No. 1200. The observed relation between the compliance and the crack length was compared with calculated one. For later convenience the energy release rate \( G_\alpha \) obtained was converted into the stress intensity factor \( K_\alpha \) by use of Eq. (2)(10).

\[
G_\alpha = K_\alpha \left( \frac{S_{11} + S_{66}}{2} \right)^{1/2} \left( \frac{S_{12}}{S_{11}} \right)^{1/2} \left[ 2S_{12} + S_{66} \right]^{1/2}
\]

where \( S_{ij} \) are components of the compliance tensor.

2.2.2 Tensile test of DEN specimen Both the fracture toughness and the net-area-averaged stress have been proposed as fracture criteria of C/C composites. In order to discuss the adequacy of these

<table>
<thead>
<tr>
<th>Property</th>
<th>0°/90°</th>
<th>QI</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Strength MPa</td>
<td>150</td>
<td>125</td>
</tr>
<tr>
<td>Young's Modulus GPa</td>
<td>75.8</td>
<td>54.0</td>
</tr>
<tr>
<td>Shear Modulus GPa</td>
<td>6.87</td>
<td>23.8</td>
</tr>
<tr>
<td>Poisson's Ratio</td>
<td>0.272</td>
<td>0.345</td>
</tr>
</tbody>
</table>

Table 1 Mechanical properties of C/C composites

Fig. 1 Geometry and dimensions of specimens
criteria, tensile fracture tests of the double-edge-notched, DEN, specimens with changing notch length but fixed relation $2a/W = 0.5$ were carried out, where $a$ and $W$ denote the notch length and the specimen width. The critical stress intensity factors of the C/Cs were determined by use of Eq. (3)\(^{(b)}\)

$$K_{\text{max}} = \sigma \sqrt{\pi a} \left(1 + 0.122 \cos^4 \frac{\pi \xi}{2}\right) \sqrt{\frac{2}{\pi \xi}} \tan \frac{\pi \xi}{2}$$

where $\sigma$ and $\xi$ represent the fracture tensile stress in the far field and $2a/W$, respectively.

2.2.3 Bending test of SENB specimen

Four-point-bending tests were carried out by use of single-edge-notched specimens varying the ratio $a/W$ from 0.4, 0.5, to 0.6. The loading and unloading cycles were applied to obtain the $R$-curve by use of Eqs. (1) and (2).

3. Experimental Results

3.1 Compact tension test

A typical load vs. crack-opening-displacement ($P$-$\Delta$) curve during a compact tension test is shown in Fig. 2. The $R$ curves obtained by use of Eqs. (1), (2) and data like in Fig. 2 are shown in Fig. 3. The onset of crack extension was estimated at the beginning of the nonlinearity in the $P$-$\Delta$ curve as shown by arrow in Fig. 2. The estimated initial value of the crack extension resistance $K_{\Delta}$ was about 5 to 6 MPa $\sqrt{\text{m}}$. As shown in Fig. 3, $K_{\Delta}$ increased rapidly up to its maximum of about 22 MPa $\sqrt{\text{m}}$. The initial and maximum values were observed to be independent of the pre-crack length, $a$, and the stacking sequences, CP or QI.

3.2 Double-edge-notched tests

Tensile fracture stresses of double-edge-notched specimens for CP and QI stacking sequences are

![Fig. 2](image-url) Relation between load and crack opening displacement in a CT test of the $0^\circ/90^\circ$ C/C composite

![Fig. 3](image-url) $R$-curve behavior in the CT and the SENB tests

![Fig. 4](image-url) Gross fracture stress in the DEN tests as a function of pre-crack length

shown in Fig. 4(a) and (b), respectively. In these figures the ordinate, gross stress, represents the apparent stress defined as fracture load divided by the gross-sectional area of the SEN specimen while the horizontal solid line stands for the net stress criterion. This criterion assumes that ultimate fracture occurs when the averaged stress over the minimum cross-section reaches the fracture stress of smooth specimens, \(\sigma_0\). It is noted here that \(\sigma_0\) was determined from an average value of over 30 specimens and was confirmed to be not dependent upon the width\(^b\). It is obvious from these figures that fracture occurs at a higher or lower value than that of the averaged stress criterion for the case of short or long notch length, respectively.

3.3 Bending test of single-edge-notched specimen (SEN)

A typical \(P-COD\) curve obtained by a single-edge-notched test for the CP laminate is shown in Fig. 5. Using this result and Eqs. (1) and (2), the \(R\)-curve was determined as shown in Fig. 3. In this test, the onset of crack propagation was also estimated from the beginning of nonlinearity in the \(P-COD\) curve. Thus the initial value of the \(R\)-curve was determined to be 5 to 6 MPa/\(\sqrt{m}\). This value agrees well with that obtained by the CT test. In the SENB tests, fracture behavior was affected by stress concentration at the loading point. Because of this effect, the crack propagation resistance initially increased more rapidly than that in the CT tests and then decreased. Hence the \(R\)-curve by the SENB tests was regarded as meaningful only near the onset point of the crack propagation.

4. Discussion

4.1 Onset of crack propagation

For materials giving rise to \(R\)-curve behavior, it has pivotal importance to identify the onset load of crack propagation. Since there is possibility to start crack propagation before reaching a maximum load, attempt was made to identify the onset load by use of the AE method.

The load and AE event rate during the CT and SENB tests are plotted in Fig. 6(a) and (b), respectively, as a function of test time. The onset of crack propagation can be identified from the event rate beginning to increase, indicated by the meshed region in these figures. Thus the crack propagation resistance was calculated at this point resulting in 5 to 6 MPa/\(\sqrt{m}\). This is identical with the value obtained from the starting point of nonlinearity in the \(P-COD\) curve.

The wave form ratio is defined by the risetime of
(σ UN : Strength for the smooth specimens)

Fig. 7 Explanatory drawing for the point stress criterion

AE signals divided by the duration. The onset of the crack propagation can also be identified from the waveform ratio as a point of stabilization, i.e., the point from which the amplitude of the waveform ratio decreases. Stochastically saying, stabilization of this ratio implies that AE counts due to damage of material increase to surpass those due to noise. This is due to the fact that in case of noise the waveform ratio should have a high amplitude while damage should have a stabilized one.

4.2 Point stress criterion

For stress distribution near the notch tip, the region with higher stress than static strength σ\textsubscript{UN} tends to extend its area with increasing external load. The point stress criterion\textsuperscript{(10)} presumes as shown in Fig. 7 that the ultimate fracture of a notched specimen occurs when the extension of the region exceeding σ\textsubscript{UN} reaches the characteristic dimension d\textsubscript{0}, which depends only on the kind of material. This criterion was reported to be effective for the prediction of ultimate fracture in polymer matrix composites with stress concentration sources\textsuperscript{(11)}. The point stress criterion was applied for the prediction of fracture loads of DEN specimens of C/Cs and results are shown in Fig. 8(a) and (b). In this calculation the stress distribution near the notch tips was approximated by Eq.(4), in which the crack tip shape is assumed to be a prolate ellipsoid with an equal semiaxis radius.

\[
\frac{\sigma_{EL(x,y)}}{\sigma_{UN}} = \frac{1}{2 + \left( \frac{b}{\rho} \right) \left( 1 - \frac{x^2}{b^2} \right)} \left( 1 + \left( \frac{b}{\rho} \right) \left( 1 - \frac{x^2}{b^2} \right)^{3/2} \right)
\]

where \( b, \rho, \) and \( a \) represent half of the ligament length, notch tip radius, and stress concentration factor, respectively. It is obvious from this figure that the point stress criterion explain all the data if \( d_0 \) is assumed to be 0.35 mm. At fracture, the stress \( \sigma_0 \) in \( d_0 \) exceeded \( \sigma_{UN} \) so that the material in this region must have suffered from some kind of damage. Therefore \( d_0 \) should be a measure of the damaged zone extension.

The Irwin's rectification\textsuperscript{(11)} is known to be effective for metal with small plastic deformation regions near the notch tips. Irwin showed that a stress intensity factor is given by Eq.(5) when the extension of plastic deformation zone is \( r^*_p \).

\[
K_i = \sigma \sqrt{\pi a} \left( \frac{a^*}{W} \right) = \sigma \sqrt{\pi (a + r^*_p)} \left( \frac{a + r^*_p}{W} \right).
\]

Equation (5) is originally formulated for the rectification of a plastic deformation zone. However the damage zones in the present experiments are considered to induce similar stress relaxation. In addition, \( d_0 \) in the present paper is much smaller than the ligament or pre-crack length. Thus estimation of the effect of the damage zone can be approximately made by use of Eq.(5).

The extension of a plastic deformation zone is evaluated by
Fig. 9 Application of the Irwin’s rectification for small scale damage zones to the results of the DEN tests

\[ r^* = 2r_0 = \frac{1}{\pi} \left( \frac{K_i}{\sigma_y} \right)^2, \quad (6) \]

where \( \sigma_y \) represents the yield stress. Then substitution of the critical stress intensity factor and strength of the C/C into \( K_i \) and \( \sigma_y \) in Eq. (6) resulted in about 0.5 mm of the damage zone, which agrees reasonably well with the damage zone extent from the point stress criterion, 0.35 mm. Substituting this value of \( r^* \) into Eq. (5), the effect of the damage zone can be evaluated and the calculation results are shown in Fig. 9. In this figure, solid, dashed, and dotted lines represent the fracture stresses obtained by the simple linear fracture mechanics, point stress criterion and Irwin’s rectification, respectively. It is obvious from this figure that even in the short notch length region this estimation gives a good estimation.

It follows from the above results that the damage zone formed around the notch tip is sufficiently small so that the situation of the present experiments is similar to that of the small scale yielding in the field of fracture mechanics. This is why the concept of fracture mechanics can be effectively applied to the C/C composites despite of its microstructure including a lot of deficits. Hence in this work the fracture mechanics approach is shown to be effective but this can be applied only for specimens with a sufficiently small notch tip radius compared to the notch length.

4.3 \( R \)-curve behavior

As shown in Fig. 3 the \( R \)-curve behaviors obtained by the CT and SENB tests were much different though their initial values coincided. This is due to different fracture patterns caused by the different geometry of specimens. Figure 10 shows a typical SEM photograph of a cross-sectional view near the notch root of a compact tension specimen. In this figure, two large holes formed by pulling out the fiber bundles can be observed in the vicinity of the notch root. The area of the holes is nearly equal to that of the fiber bundle, 6 k. Consequently an initial rapid increase of the \( R \)-curve can be explained in terms of the bridging mechanism by the fiber bundles. This bridging mechanism operates in a distance of ca. 0.5 mm from the notch tip. This is a value similar to the characteristic dimension in the point stress criterion. It implies that damage occurring in the notch tip damage zone is debonding of whole fiber bundles and pure matrix carbon.

While the initial values of the CT and SENB tests are about 5 to 6 MPa\(\sqrt{m} \), the critical stress intensity factor in the DEN test is 7.5 MPa\(\sqrt{m} \). The difference can be settled down if we consider that the value of the DEN might be evaluated after formation of the damage zone. The crack propagation resistance after 0.35 mm progression of damage is estimated to be 7.5 MPa from the \( R \)-curve of CT specimen in Fig. 3.

4.4 Mechanism of strength increase

When the notch length is short in the DEN tests, it was observed in Fig. 4 that the fracture stress averaged over the minimum cross section, net fracture stress, surpassed the strength measured by use of smooth specimens. Though surprising for ordinary materials, this behavior can be reasonably explained in case of C/C composites as follows.

It is well known that strength of C/C composite is generally much weaker than that predicted by the simple rule of mixture (123). This low strength is ordinarily explained in terms of low fracture strain of the matrix carbon, i.e., ultimate fracture of C/C composites occurs at the low fracture strain of the matrix (123). The fracture strain of the C/C composite
in this paper is about 0.2% but that of the reinforcing fiber is 0.6%. If constraint by the matrix to the fibers prevents to exert elongational ability of the fibers, the relief of the matrix constraint by fracture of the matrix and/or interfaces possibly results in higher fracture strain of C/C composites. In this study, above mentioned release of the matrix restriction might partly occur in the damage zones induced near the notch tips. This induces strength increase and, when the damage zones cover the whole of the ligament, the high strength phenomenon might be observed as shown in Fig. 4.

5. Conclusion

Fracture toughness criterion of C/C composites was studied by using specimens with various geometries. Main conclusions obtained are as follows:

1. When notch length is sufficiently long compared with the notch tip radius, C/C composites exhibit notch sensitive behavior and their fracture can be predicted by the criterion of the linear fracture mechanics.

2. The extension of the damage zones near the notch tips in this study was about 0.35 mm. This small damage zones enabled the application of the linear fracture mechanics.

3. The onset of fracture could be identified by use of the AE method, event rate and wave form ratio.

4. Fracture toughness of C/C composites examined was 5 - 6 MPa\sqrt{m}.

References


(9) NF Electronic Instrument Music, Application soft for AE Instruments.

