

# EFFECTS OF TEMPERATURE AND FIBER ORIENTATION ON THE MODE I INTERLAMINAR FRACTURE TOUGHNESS OF CARBON/EPOXY COMPOSITES

Hyoungh Soo Kim <sup>1</sup>, Wen-Xue Wang <sup>2</sup> and Yoshihiro Takao <sup>2</sup>

<sup>1</sup> *Department of Aeronautics and Astronautics, Graduate School of Engineering, Kyushu University, 6-10-1, Hakozaki, Higashi-ku, Fukuoka 812-8581, Japan*

<sup>2</sup> *Research Institute for Applied Mechanics, Kyushu University, 6-1, Kasuga-koen, Kasuga, Fukuoka 816-8580, Japan*

**SUMMARY:** Delamination is a predominant failure mechanism in composite structures. In the present study, double cantilever beam tests were performed to investigate the effects of temperature and fiber orientation on the Mode I interlaminar fracture toughness,  $G_I$ , of carbon fiber-reinforced epoxy composites, T800H/#3631. The values of  $G_I$  for three kinds of laminates,  $[0_{12}/0_{12}]$ ,  $[22.5/-22.5/0_8/-22.5/22.5//22.5/22.5/0_8/22.5/-22.5]$  and  $[45/-45/0_8/-45/45//45/45/0_8/45/-45]$ , with a pre-cracked interface, that is // in each laminate, were obtained at various temperatures, *i.e.*, -100°C, 20°C and 150°C. Fracture surface observation was also carried out by a scanning electron microscope and optical microscope. It is shown that  $G_I$  is obviously affected by the temperature and fiber orientation. In the case of the specimen with delamination at 0//0, the fracture morphology changes as the delamination increases, but the value of fracture toughness for initiation  $G_{IC}$  is not so different from the one for propagation  $G_{IR}$ , and the fracture toughness of the specimen with 0//0 is little affected by the temperature. However,  $G_{IR}$  of the specimen with 22.5//22.5 shows a remarkable temperature dependence at a large crack length region. In case of the specimen with 45//45,  $G_{IR}$  is considerably affected by both failure mechanisms of crack jumping and fiber bridging, and the effect of temperature on  $G_{IR}$  is less than the case of 22.5//22.5.

**KEYWORDS:** temperature, fiber orientation, composite materials, DCB testing, Mode I interlaminar fracture toughness.

## INTRODUCTION

Fiber reinforced plastic (FRP) laminates have been widely used in aerospace and other application fields because of their high specific modulus and high specific strength, and they are expected to be applied to a wide range of temperature environments.

Recently, the authors have investigated experimentally the effects of the temperature on the

microscopic damage for quasi-isotropic carbon/epoxy composites at low (-100°C), room (25°C) and high (150°C) temperatures under tensile loading[1], where it was reported that there was temperature dependence in damage behaviors and that nonlinearity observed in the stress-strain response was caused by a large-scale interlaminar delamination as the separation between adjacent plies throughout the length of the specimen. Delamination, which is the most predominant and life-limiting failure mechanism in composite structures, has been recognized as one of the frequently observed damage modes in composite laminates. This interlaminar delamination significantly decreases structural integrity, such as stiffness and strength. Thus, it is very important to understand and predict the composite's resistance to interlaminar fracture.

A double cantilever beam (DCB) has been commonly used to measure the Mode I interlaminar fracture toughness,  $G_I$ , of composite laminates. Evaluation of  $G_I$  for the unidirectional laminates has been performed by many researchers [2-7] using a DCB specimen with a pre-cracked interface at its center at room and various temperatures. However, in practical application FRP laminates are frequently used in a form of multidirectional laminates in order to meet various required material properties, which leads to the interlaminar delamination between layers with different fiber orientations. Therefore, in order to understand the interlaminar fracture behavior in structural composites, it is essential to accurately evaluate the composite's resistance to interlaminar fracture of multidirectional laminates with a delamination between  $\theta$  and  $-\theta$  degree layers, that is,  $\theta//-\theta$ . Several researchers [8-13] have reported  $G_I$  in multidirectional laminates at room temperature. However, there are few studies about the influence of the temperature and fiber orientation on  $G_I$ .

In the present study, three kinds of DCB specimen,  $[0_{12}/0_{12}]$ ,  $[22.5/-22.5/0_8/-22.5/22.5//22.5/22.5/0_8/22.5/-22.5]$  and  $[45/-45/0_8/-45/45//45/45/0_8/45/-45]$ , with a pre-cracked interface are loaded at three temperatures, *i.e.*, -100°C, 20°C and 150°C to investigate the effects of the temperature and fiber orientation on  $G_I$  of Carbon/Epoxy composites. Fracture surface observation is carried out by a scanning electron microscope (SEM) and optical microscope.

## EXPERIMENTAL DETAILS

### Material and Specimens

Three types of lay-ups were prepared as listed in Table 1. A 25 mm thick Kapton film sprayed with a release agent was inserted at the center to yield 0//0, 22.5//22.5 and 45//45 interfaces with a delamination due to a pre-crack. It should be noted that the stacking sequence of each half of the specimen is symmetric and warping does not occur due to delamination if it is located on the pre-cracked interface. The laminates used in this study were made of Toray P2212-15 prepregs

Table 1: Stacking sequence of DCB specimens

Specimen	Stacking sequence
Type [0//0]	$[0_{12}/0_{12}]$
Type [22.5//22.5]	$[22.5/-22.5/0_8/-22.5/22.5//22.5/22.5/0_8/22.5/-22.5]$
Type [45//45]	$[45/-45/0_8/-45/45//45/45/0_8/45/-45]$

// : pre-crack

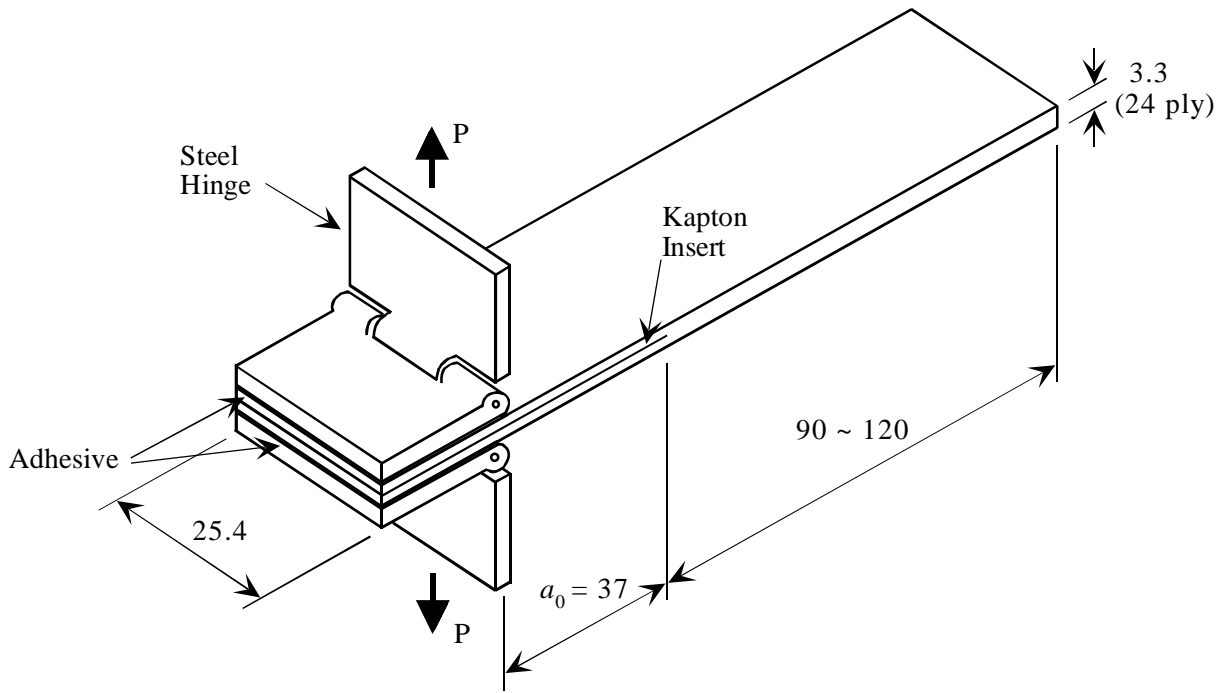


Fig. 1: Geometry of DCB specimen (Dimensions in mm)

which consists of carbon fiber (T800H) and epoxy resin (#3631), composed of 24 plies and cured in an autoclave for 2 hours at 180°C. The fiber volume fraction  $V_f$  and final thickness of the laminates were approximately 62% and 3.3 mm, respectively. Specimens having a size of 145~175 mm long and 25.4 mm wide were cut out of the laminates. The DCB test specimen geometry used to obtain the Mode I interlaminar fracture toughness,  $G_I$ , is shown in Fig. 1. Steel hinges were adhered to transfer the external load into the specimen. The crack length of  $a_0$  was measured from the center of the hinge pivot pin. The side edge of the specimen was painted with typewriter correction fluid to facilitate the observation and measurement of crack growth process during the DCB testing.

### Test Procedure

The DCB tests were carried out under the displacement control condition using a servo-hydraulic testing machine (MTS 320.34) equipped with a thermostatic chamber which keeps the temperature between -180°C and 320°C. Both a heater and nitrogen were used to control the temperature. Before loading, the DCB specimen was set in the thermostatic chamber for one hour at the testing temperature. Then, it was loaded in tension perpendicular to their length (as shown in Fig. 1) at a constant crosshead speed of 1mm/min. After the crack extension,  $\Delta a$ , of about 10 mm, the specimen was unloaded with a crosshead speed of 1~3mm/min. This procedure was repeated until the crack length became to approximately 70 and 100 mm for multidirectional and unidirectional laminates, respectively. Crack growth process during the DCB testing was monitored in-situ by a stereoscopic microscope and recorded by a VTR through a CCD camera. Crack length could be measured with precision up to 0.001~0.005 mm. Failed specimens were observed in detail by both a SEM and optical microscope.

### Data Reduction

The conventional compliance method [14] was used to evaluate the Mode I interlaminar fracture toughness,  $G_I$ , of the composite laminates as follows:

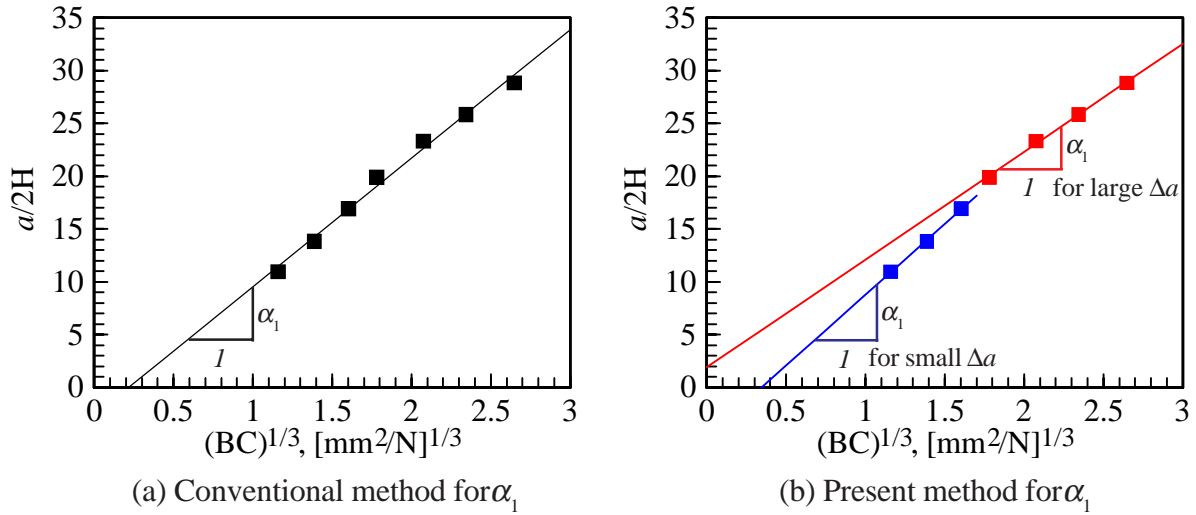


Fig. 2: Relationship between  $a/2H$  and  $(BC)^{1/3}$  for multidirectional laminates

$$\frac{a}{2H} = \alpha_1 (BC)^{1/3} + \alpha_0 \quad (1)$$

$$G_I = \frac{3}{2(2H)} \left(\frac{P}{B}\right)^2 \frac{(BC)^{2/3}}{\alpha_1} \quad (2)$$

where  $P$ ,  $a$ ,  $C$ ,  $B$ ,  $2H$  are applied load, crack length, compliance, specimen width and specimen thickness, respectively and  $\alpha_1$  is calculated from Eqn 1 based on the experimental data of  $a$  and  $C$  (as shown in Fig. 2). Fig. 2 shows a typical experimental relationship between crack length and compliance obtained from a type [45//45] specimen. It is seen that this experimental relationship could not be represented well by a single line. This rather complicated relationship for multidirectional laminates has been reported [9, 12] at a room temperature and concluded to be due to crack jumping and fiber bridging. In the previous studies,  $\alpha_1$  is determined by fitting a line to experimental data as shown in Fig. 2 (a). This single  $\alpha_1$  yields  $G_{IC}$  at initiation and  $G_{IR}$  at propagation larger and less than real ones. Here, two  $\alpha_1$  were determined as shown Fig. 2 (b) by considering the change of the relationship.

## RESULTS AND DISCUSSION

### Type [0/0] Specimen

Typical load-displacement curves and the Mode I interlaminar fracture toughness at initiation  $G_{IC}$  and at propagation  $G_{IR}$ , obtained for the unidirectional specimen at various temperatures, *i.e.*, -100°C, 20°C and 150°C are shown in Figs 3 and 4, respectively.

A few studies [7] reported that  $G_I$  at cryogenic temperatures is higher than  $G_I$  at room temperatures. In general,  $G_I$  is a product of the fracture toughness of the matrix or fiber/matrix interface and the fracture surface area, being related to failure mechanisms, such as fiber bridging and fiber breakage. Fig. 4 shows that  $G_{IC}$  is high in order of -100°C, 20°C and 150°C and the same phenomena may be observed. The scattering of data is a little large for  $\Delta a$  less than 20 mm, which leads to the idea that the change of the fracture morphology, such as fiber bridging and fiber breakage appeared. It was recognized that the change of  $G_{IR}$  at -100°C is small for various  $\Delta a$ , and that there is not so much difference between  $G_{IC}$  and  $G_{IR}$ . Generally  $G_{IR}$  increases by the

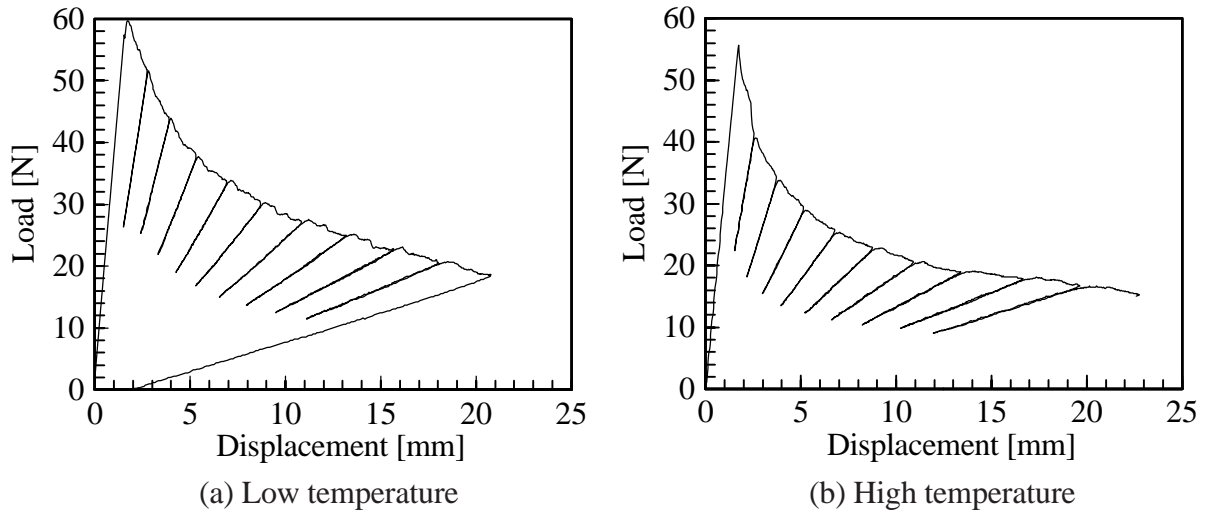


Fig. 3: Typical load-displacement curves for type [0//0] specimen

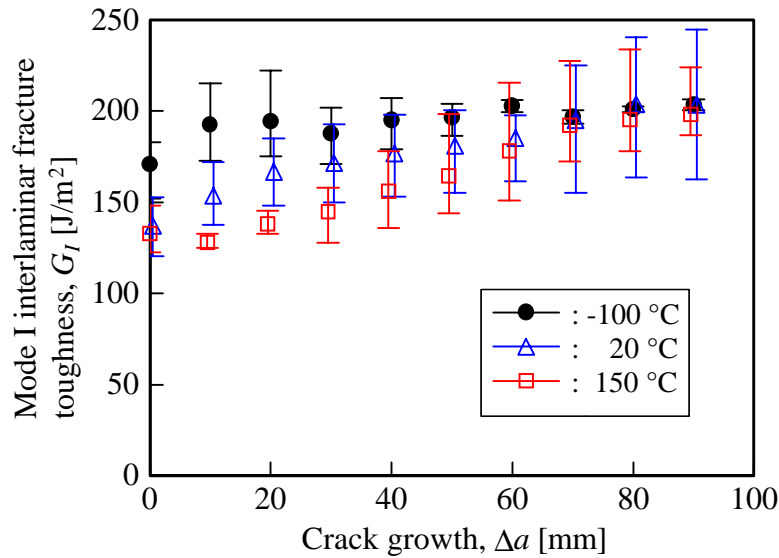


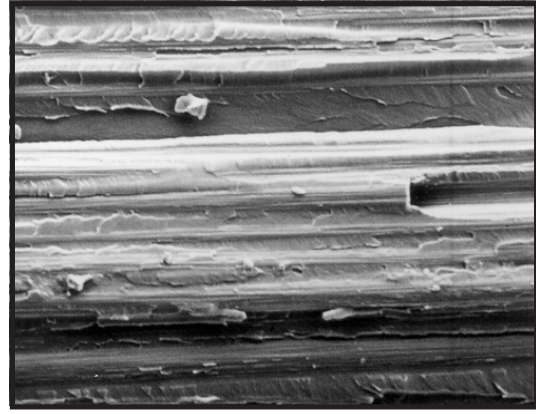
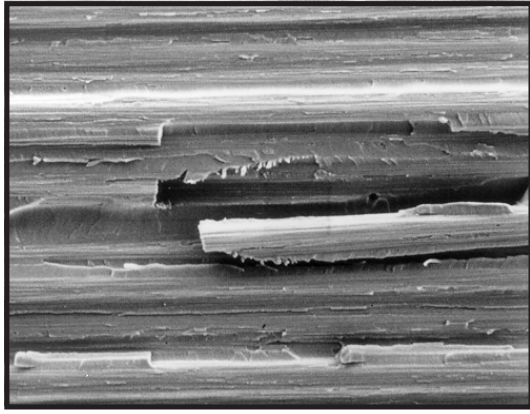
Fig. 4: Mode I interlaminar fracture toughness for type [0//0] specimen at three temperatures

development of fiber bridging. Thus, it is predicted that the fiber bridging hardly occurred for large  $\Delta a$  at low temperature.

On the other hand,  $G_{IC}$  at 150°C was small. The degradation of the matrix and fiber/matrix interface at high temperature environment is conceivable. Since the glass transition temperature of the matrix #3631 is approximately 180°C, the degradation at 150°C is an appropriate conjecture. The  $G_{IR}$  increases with  $\Delta a$  increasing and its rate is large among three temperatures, which makes us think that the fiber bridging is remarkably exhibited.

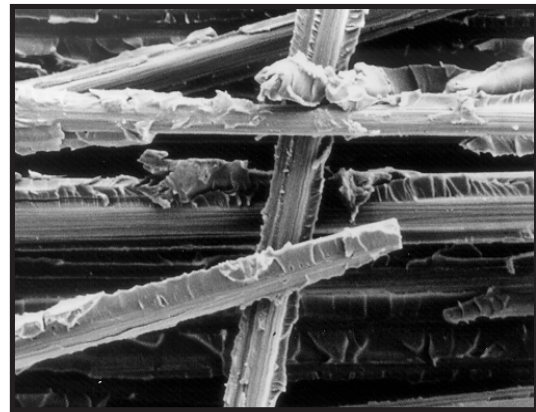
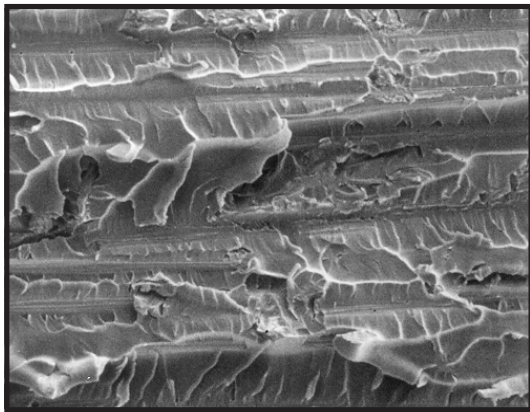
The  $G_{IC}$  at 20°C showed the intermediate tendency.

The fracture surface of the type [0//0] specimen was examined using a SEM in order to figure out characteristic morphologies. The SEM photographs of the typical fracture surfaces at low and high temperatures are presented in Fig. 5. At a low temperature, as above mentioned (see Fig. 4), it is recognized that the fiber bridging and fiber breakage occurred at the initiation and the fiber bridging was not exhibited at the propagation. At a high temperature, it is found that the failure at the initiation occurred mainly in the matrix as shown in Fig. 5 (b). The small  $G_{IC}$  at high



(a) -100°C (left:initial, right:propagation)

10μm

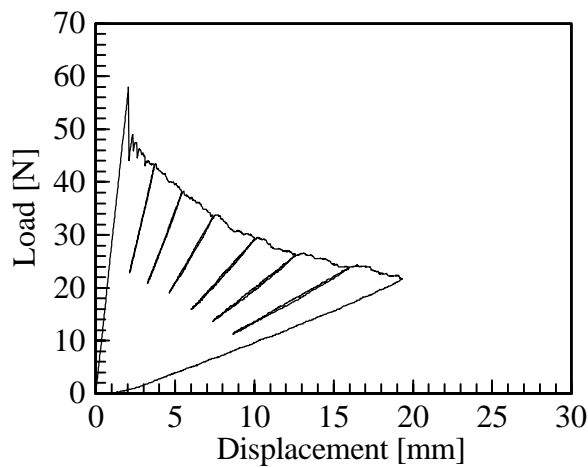


(b) 150°C (left:initial, right:propagation)

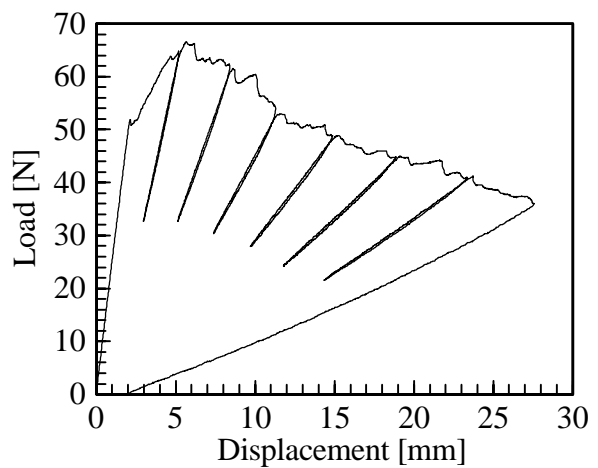
Fig. 5: SEM photographs of fracture surfaces for type [0//0] specimen; crack growth L to R temperature means that the fracture toughness of the matrix is relatively low. However, in propagation stage the fiber bridging occurred and conspicuously increased  $G_{IR}$ .

### Type [22.5// -22.5] Specimen

Fig. 6 shows typical load-displacement curves obtained at -100°C, 20°C and 150°C for the



(a) Low temperature



(b) High temperature

Fig. 6: Typical load-displacement curves for type [22.5// -22.5] specimen

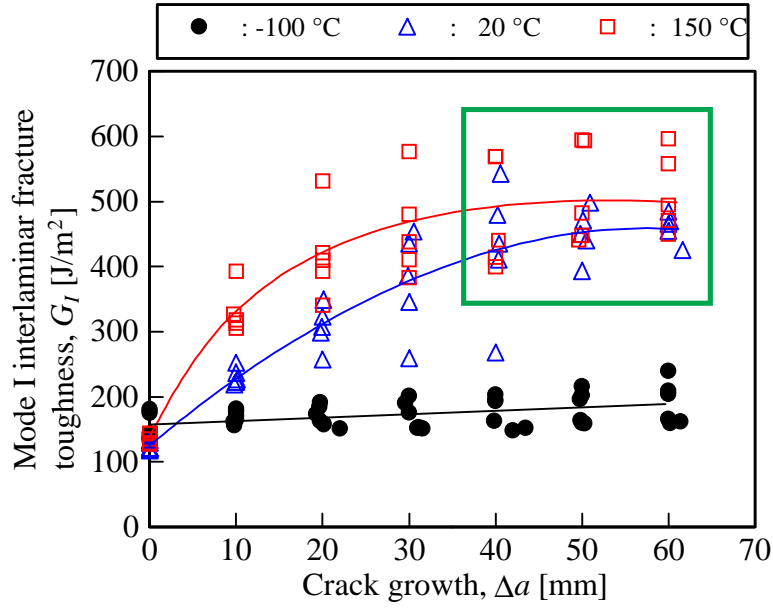


Fig. 7: Mode I interlaminar fracture toughness  $G_I$  for type [22.5// -22.5] specimen at three temperatures

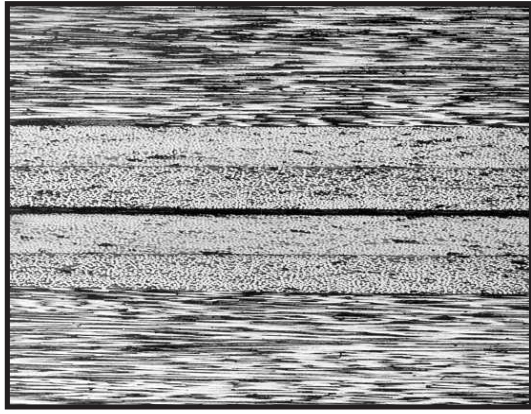
multidirectional specimen with a [22.5// -22.5] interface. These two curves are fairly different and it is expected that both  $G_{IC}$  and  $G_{IR}$  are affected by temperatures.

The  $G_I$  at three temperatures is shown in Fig. 7. The  $G_{IC}$  at -100°C is large. Near the initial crack tip (*i.e.* pre-crack tip or  $\Delta a = 0$  mm), a resin rich area exists and the strength of matrix increases at low temperature. Thus,  $G_{IC}$  becomes large. As shown in Fig. 6 (a), the maximum load at low temperature corresponds to the crack propagation. This is in accordance with the above mentioned result. The change of  $G_{IR}$  is small and  $G_{IR}$  is nearly the same to  $G_{IC}$ . It is said that  $G_{IR}$  of multidirectional laminates is strongly affected by the fiber bridging and crack jumping. The crack jumping is a failure mechanism frequently observed in multidirectional composites, where the delamination crack propagates through an adjacent ply or into the other interface. Thus, the fiber bridging and crack jumping are seemed to be suppressed. This may be explained by the thermal shrinkage of the matrix, which is expected to resist the fiber pull-out mechanism.

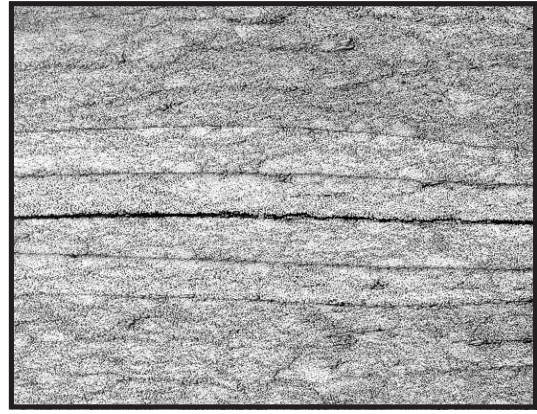
At high temperature,  $G_{IC}$  is lower than the low temperature case. In the load-displacement curve, nonlinearity appears before the maximum load. Since the point of nonlinearity is related to  $G_{IC}$ ,  $G_{IC}$  becomes small. However,  $G_{IR}$  becomes remarkably large for  $\Delta a$  larger than 10 mm. Both the degradation of a matrix and fiber/matrix interface and the expansion of the matrix are expected to accelerate the fiber pull-out process at high temperature, and the change of the fracture morphology, such as fiber bridging and the increasing of fracture surface, appears. After all, it is conceivable that  $G_{IR}$  is considerably large. However, for  $\Delta a$  larger than 40 mm, the crack jumping appears, which increases  $G_{IR}$ . A semitransparent region (quadrangular region) in Fig. 7 indicates that crack jumping occurred.

It is found that  $G_{IR}$  at a room temperature remarkably increases with  $\Delta a$  increasing as at a high temperature, though it is smaller than the high temperature case.

Fig. 8 shows photographs of the free edge and cross section in perpendicular to the direction of the crack propagation. At a low temperature, the delamination crack propagated clearly along the [22.5// -22.5] interface. It implies that the change of  $G_{IR}$  does not occur. On the other hand, the

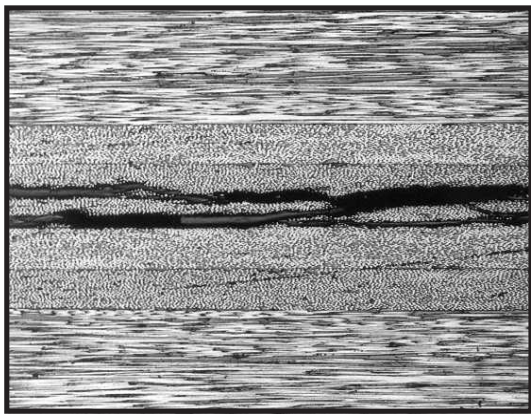


Free edge

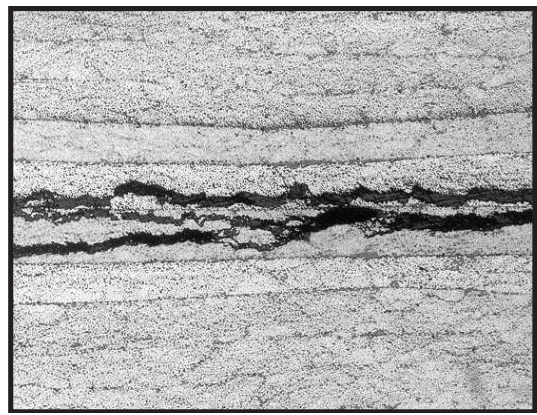


Cross section

(a) -100 °C



Free edge

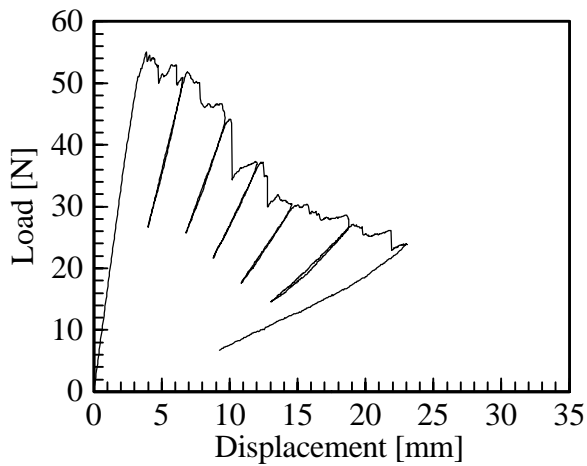


Cross section

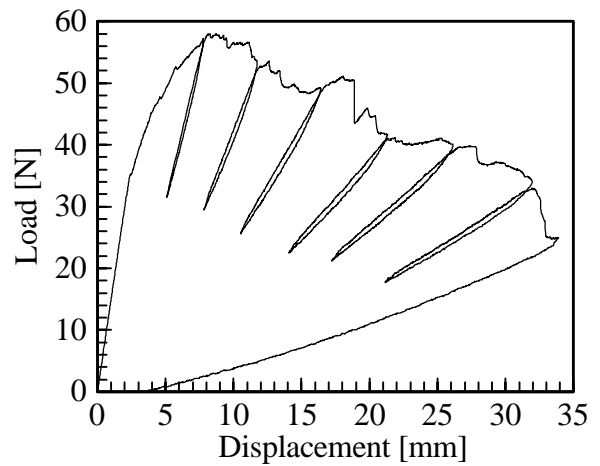
(b) 150 °C

Fig. 8: Typical side view and cross section of type [22.5// -22.5] specimen

crack jumping and the increasing of the fracture surface are observed at high temperature. These explain the considerably increase of  $G_{IR}$ .



(a) Low temperature



(b) High temperature

Fig. 9: Typical load-displacement curves for type [45// -45] specimen



## Type [45//−45] Specimen

Typical load-displacement curves and  $G_{IC}$  and  $G_{IR}$  for the type [45//−45] specimen at −100°C, 20°C and 150°C are depicted in Figs 9 and 10, respectively. It is found that the  $G_{IC}$  is high in order of −100°C, 20°C and 150°C as in Fig. 7. The same discussions for  $G_{IC}$  of the type [45//−45] specimen are true to the present case. The load of the nonlinearity in Fig. 9 is lower than the type

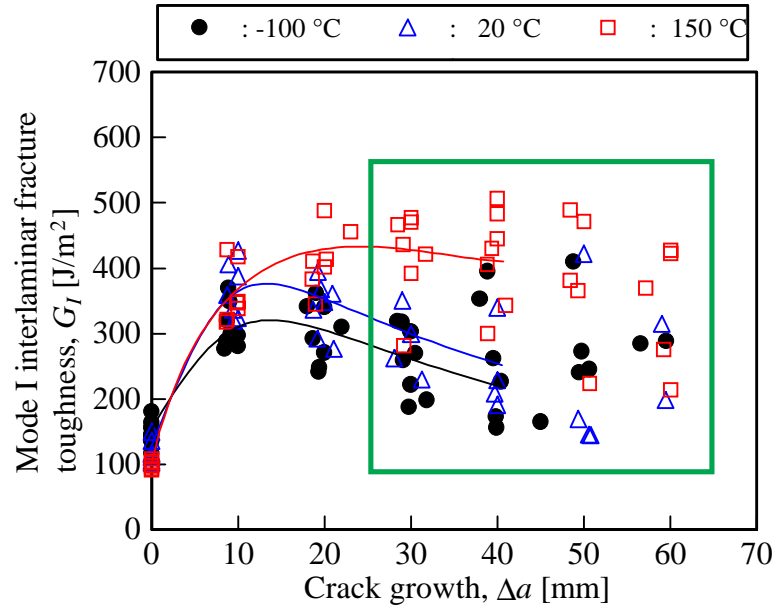


Fig. 10: Mode I interlaminar fracture toughness  $G_I$  for type [45//−45] specimen at three temperatures

[22.5//−22.5] case and  $G_{IC}$  of this type is the smallest among three types of specimen. And the behavior of the interlaminar fracture propagation shows the same tendency in all temperatures. A semitransparent region (quadrangular region) in Fig. 10 shows that the crack jumping occurs. It occurs earlier than the type [22.5//−22.5] specimen. For  $\Delta a$  larger than 20 mm all  $G_{IR}$ 's decrease. Since the crack, which propagated both in 45 ply (or in −45 ply) and along a [−45/45] interface, propagates only along the [−45/45] interface, the amount of fracture surface and  $G_{IR}$  for  $\Delta a$  larger than 30 mm gradually decrease.

## CONCLUSIONS

In the present study, three kinds of DCB specimen,  $[0_{12}/0_{12}]$ ,  $[22.5/-22.5/0_8/-22.5/22.5//22.5/22.5/0_8/22.5/-22.5]$  and  $[45/-45/0_8/-45/45//45/45/0_8/45/-45]$ , with a pre-cracked interface are loaded at three temperatures, *i.e.*, −100°C, 20°C and 150°C to investigate the effects of the temperature and fiber orientation on  $G_I$  of Carbon/Epoxy composites. The following conclusions are obtained.

It is shown that  $G_I$  is obviously affected by the temperature and fiber orientation. In the case of the specimen with delamination at 0//0, the fracture morphology changes as the delamination increases, but the value of fracture toughness for initiation  $G_{IC}$  is not so different from the one for propagation  $G_{IR}$ , and the fracture toughness of the specimen with 0//0 is little affected by the temperature. However,  $G_{IR}$  of the specimen with 22.5//−22.5 shows a remarkable temperature dependence at a large crack length region. In case of the specimen with 45//−45,  $G_{IR}$  is considerably affected by both failure mechanisms of crack jumping and fiber bridging, and the effect of temperature on  $G_{IR}$

is less than the case of 22.5//22.5.

## REFERENCES

1. Kim, H. S., Ogi, K., Matsubara, T., Wang, W. X. and Takao, Y., "Microscopic Damages of Quasi-Isotropic Carbon/Epoxy Laminates at various Temperatures", (in Japanese) *Journal of the Society of Materials Science, Japan*, 47(6), 1998, pp. 599-605.
2. Wang, W. X., Takao, Y., Yuan, F. G., Potter, B. D. and Pater, R. H., "The Interlaminar Mode I Fracture of IM7/LaRC-RP46 Composites at High Temperatures", *Journal of Composite Materials*, Vol. 32, No. 16, 1998, pp. 1508-1526.
3. Hashemi, S., Kinloch, A. J. and Williams, J. G., "The Effects of Geometry, Rate and Temperature on the Mode I, Mode II and Mixed-Mode I/II Interlaminar Fracture of Carbon-Fibre/Poly (ether-ether ketone) Composites", *Journal of Composite Materials*, Vol. 24, 1990, pp. 918-956.
4. Nakai, Y., Sakata, N., Kadowaki, T. and Hiwa, C., "Delamination Crack Growth of Unidirectional CFRP in Thermo-Mechanical Fatigue", *International Conference on Materials and Mechanics*, 1997, pp. 653-658.
5. Garg, A. and Ishai, O., "Hygrothermal Influence on Delamination Behavior of Graphite/Epoxy Laminates", *Engineering Fracture Mechanics*, Vol. 22, No. 3, 1985, pp. 413-427.
6. Kawada, H., Sugiura, W., Miyano, K., and Hayashi, I., "Evaluation of Mode I Interlaminar fracture Toughness in Unidirectional GFRP at Low Temperature", *Proceedings of ICCM-10*, Whistler, B.C., Canada, August 14-18, 1995, Vol. I, pp. 93-100.
7. Lau, H., Jiang, K. and Rowlands, R.E., "Fracture Behavior of Extren at Room Temperature and 77K", *Journal of Composite Materials*, Vol. 24, 1990, pp. 326-344.
8. Robinson, P. and Song, D. Q., "A Modified DCB Specimen for Mode I Testing of Multidirectional Laminates", *Journal of Composite Materials*, Vol. 26, No. 11, 1992, pp. 1554-1577.
9. Choi, N. S., Kinloch, A. J. and Williams, J. G., "Delamination Fracture of Multidirectional Carbon-Fiber/Epoxy Composites under Mode I, Mode II and Mixed-Mode I/II Loading", *Journal of Composite Materials*, Vol. 33, No. 1, 1999, pp. 73-100.
10. Nicholls, D. J. and Gallagher, J. P., "Determination of  $G_{IC}$  in Angle Ply Composites Using a Cantilever Beam Test Method", *Journal of Reinforced Plastics and Composites*, Vol. 2, 1983, pp. 2-17.
11. Chai, H., "The Characterization of Mode I Delamination Failure in Non-Woven, Multidirectional Laminates", *Composites*, Vol. 15, No. 4, 1984, pp. 277-290.
12. Nailadi, C. L. and Adams, D. F., "Determination of Mode-I Fracture Toughness of Angle-Ply Composites Using the Double Cantilever Beam Test Method", *Proceedings of the 13<sup>th</sup> Annual Technical Conference on Composite Materials*, September 21-23, 1998 Baltimore, Maryland.
13. Chou, I., Kimpara, I., Kageyama, K. and Ohsawa, I., "Effects of Fiber Orientation on the Mode I Interlaminar Fracture Behavior of CF/EPOXY Laminates", (in Japanese) *Journal of the Society of Materials Science, Japan*, Vol.41, No.467, pp.1292-1298.
14. Japanese Industrial Standard Group, "Testing Method for Interlaminar Fracture Toughness of Carbon Fiber Reinforced Plastics," (in Japanese) *JIS K 7086-1993*, pp. 651-665.