INTERLAMINAR REINFORCEMENT TO COMPOSITE LAMINATES BY DISTRIBUTING WHISKERS ALONG THE INTERFACE

Wen-Xue Wang[1], Yoshihiro Takao[1], Terutake Matsubara[1] and Hyoung Soo Kim[2]

[1] *Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasuga-Koen, Kasuga, Fukuoka 816-8580, Japan*
[2] *Graduate School, Kyushu University, 6-10-1, Hakozaki, Higashi-ku, Fukuoka 812-8581, Japan*

SUMMARY: In the present study, an attempt is made to improve the interlaminar fracture toughness of composite laminates by distributing whiskers along the interface of composite laminates. A commercially available T800H/3631 CFRP prepreg and β-SiC whisker are employed. A simple spray method is developed to distribute whiskers on the prepreg. An unidirectional laminate of 24 plies with a whisker reinforced interlayer is made for conventional DCB (double cantilever beam) testing. The DCB testing is conducted to investigate the effects of the whisker on the Mode I fracture toughness. Microscopic analysis of the cross-section and fracture surface of specimens is performed by the SEM (scanning electron microscope) and EPMA (electron probe micro-analyzer) to investigate the distribution of whiskers and effects of whiskers on the fracture toughness.

KEYWORDS: β-SiC Whisker, Composite laminates, Whisker reinforced interface, Microscopic analysis, DCB testing, Mode I interlaminar fracture toughness,

INTRODUCTION

Fiber reinforced composite materials are well known for their high stiffness/weight and strength/weight ratios. These advanced characteristics enable composite laminates to be used positively in aerospace and aeronautical structures, as well as in other wide applications. However, the low interlaminar strength of composite laminates is one of major shortcomings, which delayed the widespread use of composite laminates in primary aircraft structures. Interlaminar fracture or delamination becomes a fatal damage frequently observed in composite laminates in service. For this reason, many efforts have been made to improve the interlaminar strength.

The study on stitching method [1,2] seems to be one of the most reported researches. Stitching through the thickness of a laminate is valid in the improvement of interlaminar fracture toughness. However, it may reduces the strength of other directions. Another well known research is the development of 3-D braided composites (for example, [3]). Doubtless, the 3-D braided composite has relatively high strength in all directions because of no obvious layers existing. The major shortcomings of 3-D composites are high cost and the complexity of manufacturing process. Furthermore, a particulate interlayer toughening technology[4] has been developed in recent years to improve the interlaminar toughness and is employed practically in the aeronautical structures. High impact resistance is achieved by the use of this technology. Whiskers and short fibers were also employed to improve the interlaminar toughness by adding whiskers (or short fibers) with ferromagnetic coating in matrix during the manufacturing process of prepreg [5], where a magnetic moment method was developed to control the orientation of whiskers. Much improvement was obtained for the Mode I interlaminar fracture toughness and little effects were found for the Mode II one. But, high cost and complexity in the manufacturing process is still a problem for practical application. It is believed that many other researches, which are not described here because of limitation of pages, are also conducted. However, up to now, all of these methods seems to have not been widely used in practical applications mainly due to their high cost and negative effects on the other strength factors.

In the present study, an attempt is made to improve the interlaminar fracture toughness of composite laminates by distributing whiskers along the interface of composite laminates, where a simple spray method is developed to distribute whiskers on the prepreg. An unidirectional laminate of 24 plies with whiskers distributed along the mid-plane is made. The conventional DCB testing is conducted to investigate the effects of the whisker on the Mode I fracture toughness. Microscopic analysis of the cross-section and fracture surface of specimens is performed by the use of SEM and EPMA to investigate the distribution of whiskers and effects of whiskers on the fracture toughness.

EXPERIMENT

Materials and Specimens

A commercial TORAYCA T800H/3631 prepreg and TOKAIWHISK TWS-2 β-SiC whisker are used as the basic material and reinforcement, respectively. The diameter and length of whiskers are around 0.3-0.6 μm and 5-15 μm, respectively. A spray method is developed to distribute whiskers randomly on the prepreg surface by using a high pressure sprayer. In general, whiskers may be distributed selectively to the prepreg surfaces during the layout process. The curing processes of the laminate are the same to the conventional ones without adding whiskers. In order to investigate the effects of whiskers on the mode I interlaminar fracture toughness, an unidirectional laminate $[0]_{24}$ of 300 by 170 (mm) is made to conduct a DCB testing. Whiskers are deposited along the mid-plane of the laminate. A Kapton film of 25 μm thickness is inserted between the plies at the mid-plane to make a initial crack of 50 mm length. The thickness of the laminate is nearly 3.3 mm. Before DCB test, microscopic analysis of the side-view and cross-section of the laminate is performed by using the SEM and EPMA to obtain the distribution of whiskers in the laminate after curing process.

Figure 1 shows a SEM image of a side-view of the laminate. It is seen that a whisker reinforced interlayer of nearly 20 μm is formed along the mid-plane. White spots and short lines within the interlayer are understood to represent the whiskers. Good coherence between whiskers and matrix can be observed. Moreover, it is understood that the different shapes of white spots and lines describe the random distribution of whiskers in the interlayer. In order to confirm that these white spots and lines represent whiskers, EPMA analysis is performed and an image is shown in Fig. 2. The silicon is analyzed by EPMA and the strip consisting of bright spots shows the region with much amount of silicon. It is seen that bright spots concentrate in the interlayer, which is coincident with the observation by SEM.

Furthermore, the distribution of whiskers around the initial crack tip (i.e. the Kapton film

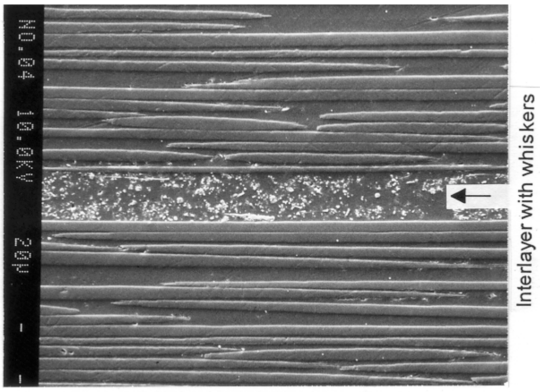


Fig.1 Side-view of the specimen by SEM

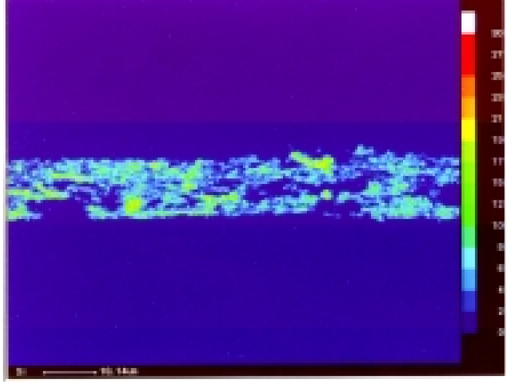


Fig. 2 Mapping analysis of Si by EPMA

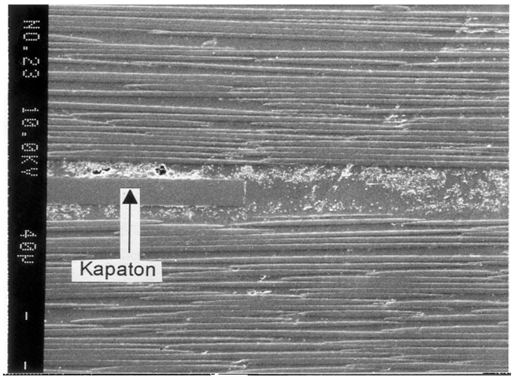


Fig. 3 Distribution of whiskers around the Kapton film tip

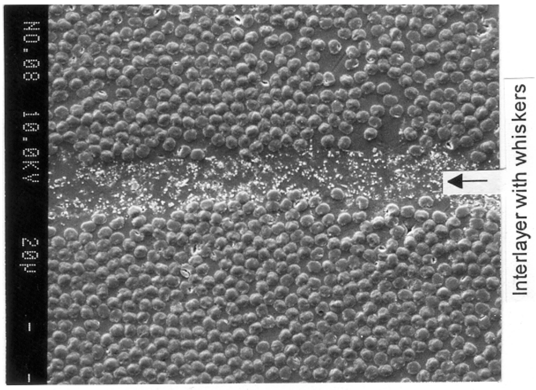


Fig. 4 Cross section image of distribution of whiskers in the interlayer.

film tip), which is artificially made by inserting a Kapton film along the mid-plane during the layout process, is shown in Fig. 3. Here whiskers are found to be concentrated relatively in the upper and lower regions around the Kapton film tip because whiskers are originally sprayed on the surfaces of prepregs. The whiskers near the corners of Kapton film tip are expected to enhance the initial fracture toughness since the crack initiates usually from one of these corners. The thickness of the interlayer near the Kapton film tip is about 40 μm, it decreases with the distance from the tip increasing and becomes nearly 20 μm after the distance is equal to about 10 mm. A relatively small region of matrix rich is observed ahead the Kapton film tip at the center of interlayer. Figure 4 shows the distribution of whiskers in the interlayer from a cross sectional view quite away from the Kapton tip. Relatively large circles represent cross sections of fibers and small white spots in the interlayer represent whiskers. It is also seen that whiskers are randomly distributed in the interlayer.

DCB Testing

Specimens for DCB testing made from the laminate are straight-sided, approximately 25 mm wide and 150mm long. The edges of specimen are painted in white and scaled to enable easy observation of the crack growth length. Two hinges are bonded to the specimen to transfer the loading. The crack length are measured from the center of the hinge pivot pin to the crack tip.

DCB fracture tests are conducted by using a material testing machine of MTS system. Eight specimens are tested. The specimen is loaded under a cross-head rate of 1 mm/min. After the crack is extended by about 10 mm the specimen is unloaded by applying the same cross-head rate. The cycle of loading and unloading is repeated until the crack is extended by about 100 mm. An optical microscope and video recorder are employed to record the crack growth process. Mode I fracture toughness G_{IC} is reduced from the conventional compliance method [6] as follows:

$$G_{IC} = \frac{3}{2(2H)}\left(\frac{P}{B}\right)^2 \frac{(BC)^{2/3}}{\alpha_1}\ , \quad \frac{a}{2H} = \alpha_1 (BC)^{1/3} + \alpha_0 \qquad (1)$$

where P is applied load, a is crack length, C is compliance, B is specimen width, $2H$ is specimen thickness and α_1 is calculated from Eq. (1) based on the experimental data of crack length a and compliance C.

EXPERIMENTAL RESULTS AND DISCUSSION

Experimental results are shown from Figs.5 to 9. Typical load to displacement curves from two different specimens are depicted in Fig.5. Two specimens are cut from the same laminate. It is seen that the peak values of two curves exhibit different variation tendency with displacement increasing. The load for specimen-a decreases rapidly during the early crack growing, in contrast, the load for specimen-b decreases slowly. This difference is also reflected in the values of fracture toughness of two specimens. The specimen-a exhibits a low

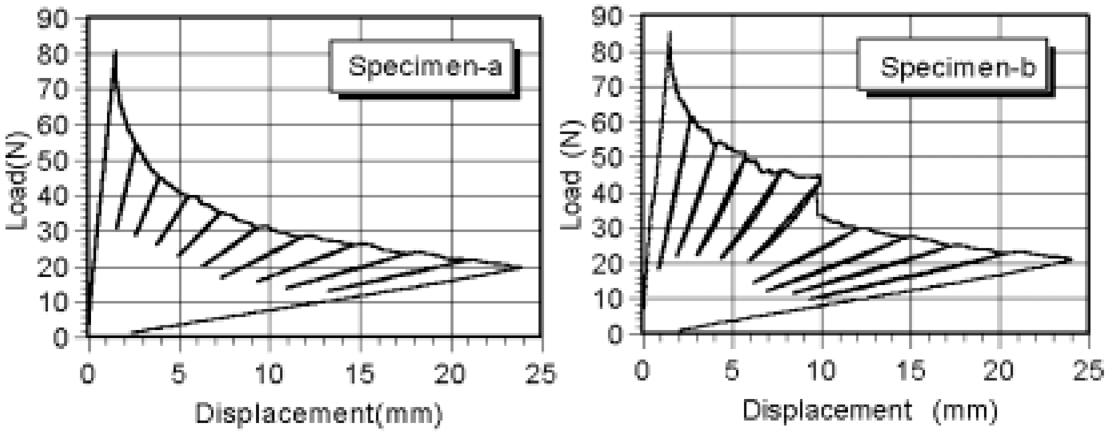
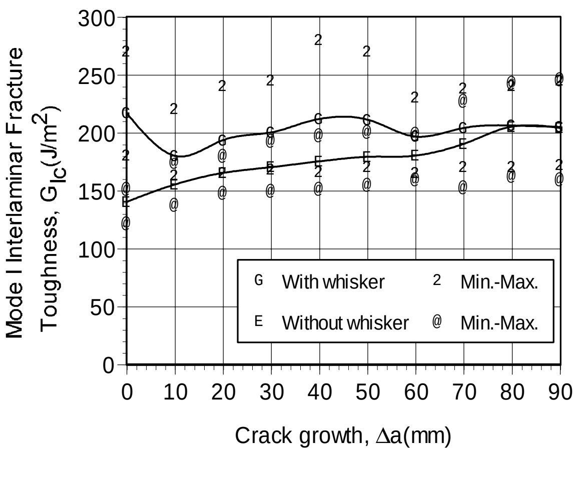


Fig. 5 Typical load to displacement curves for two different specimens.

fracture toughness, while the specimen-b gives a relatively high one. Figure 6 present the results of the mode I fracture toughness G_{IC} together with those without whiskers addition. The solid curve for whisker reinforced specimens represent an average of eight specimens and the solid one for specimens without whiskers is an average of 10 specimens. Cross marks indicate the maximum and minimum values of G_{IC} for whisker reinforced specimens and sharp marks indicate those of specimens without whiskers. From these results, Two obvious characteristics can be observed. First, the G_{IC} with whiskers exhibits a higher initial value than that of specimens without whiskers and it arrives at the same value to that without whiskers for a large crack extension. Next, The G_{IC} with whiskers is always larger than that



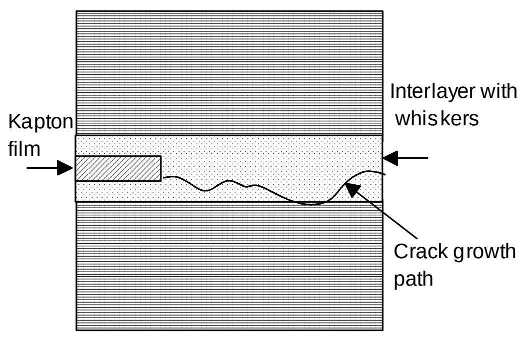


Fig. 6 Mode I interlaminar fracture toughness Fig. 7 Illustration for crack growth

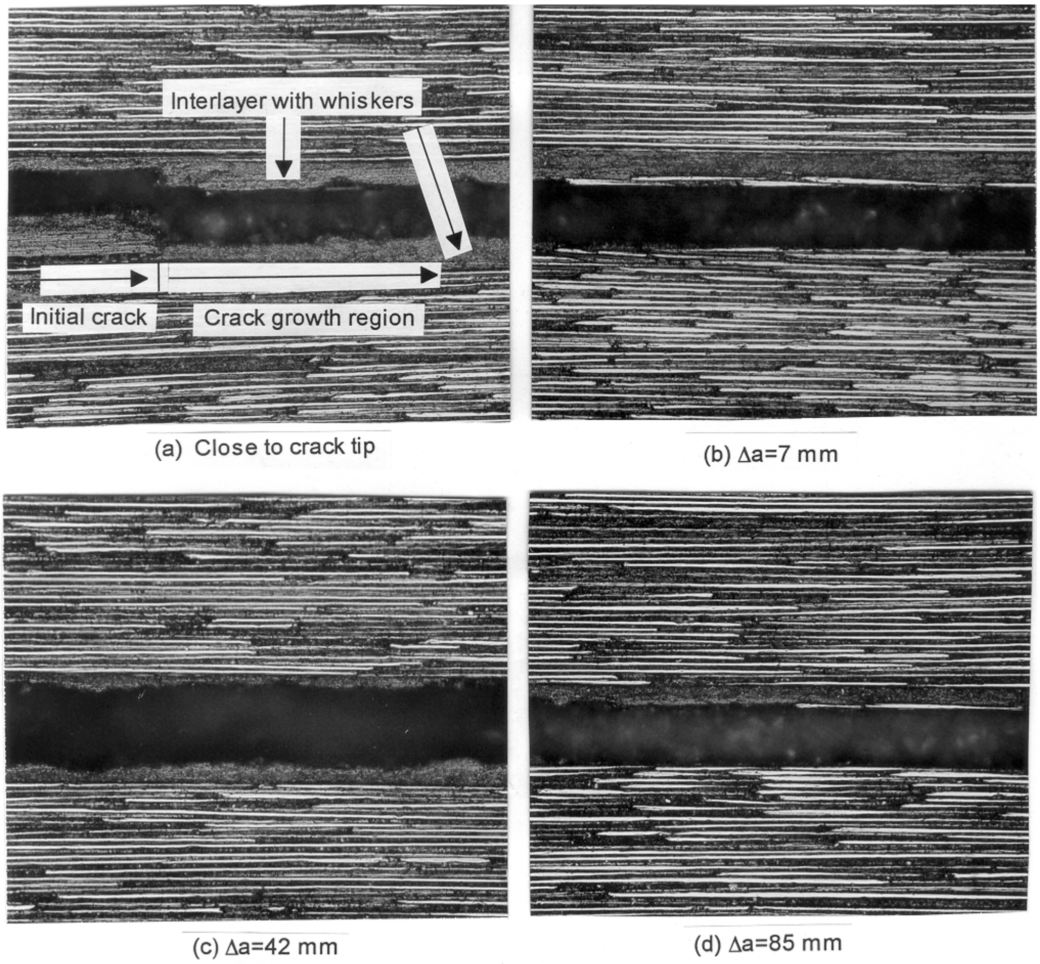
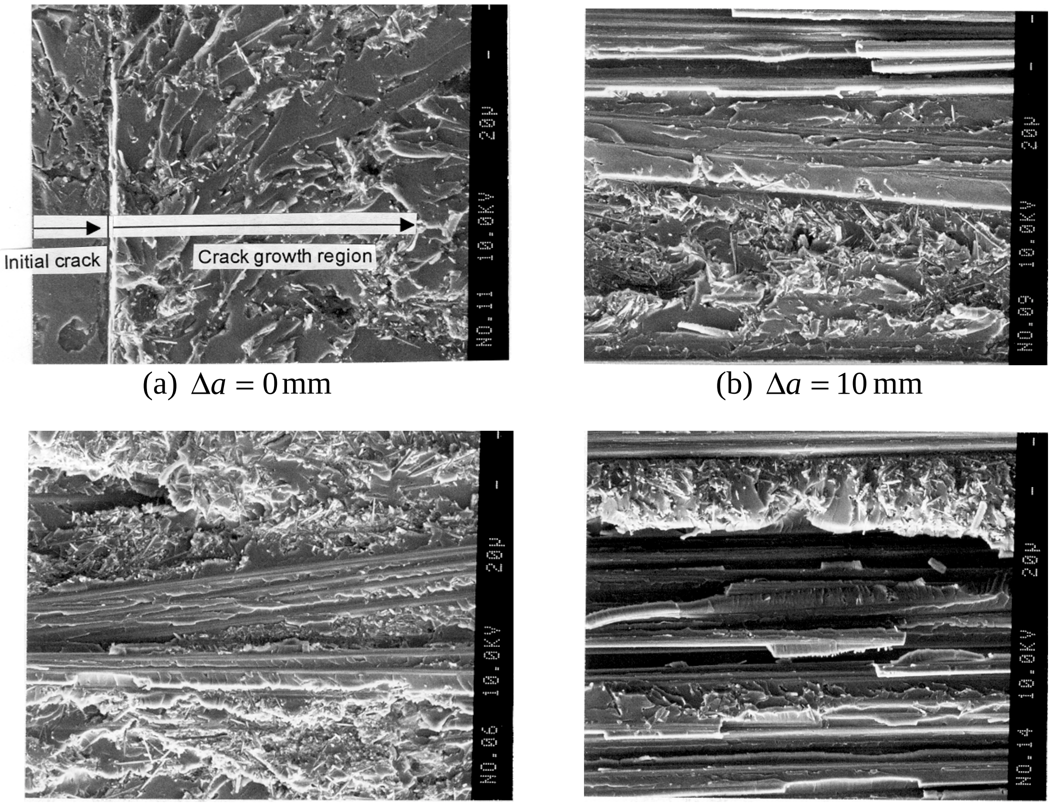


Fig. 8 Side-view of crack growth path by means of an optical microscope.

without whiskers except for a large crack extension (80 to 90 mm). The initial G_{IC} exhibits an improvement of nearly 50 % and the propagation G_{IC} increases by nearly 20 % compared with those without whiskers. The scatter range of G_{IC} between the maximum and minimum values also shows the same tendency. It is well known that the G_{IC} without whiskers shows a R-curve variation with crack extension mainly due to the fiber bridging, and that the initial fracture toughness is almost equal to the that of matrix. However, in the present cases of whisker reinforced specimens, high initial G_{IC} appears and R-curve is not clear. It is considered that this difference is caused by whiskers. In order to understand the effects of whiskers on the fracture toughness, cracked specimens are investigated by means optical microscope and SEM.

Figure 7 depicts a typical crack growth path based on the observation of cracked specimens. The crack usually initiates from one corner of the Kapton tip and grows firstly in the whisker reinforced interlayer. Then, the crack grows curvedly in the interlayer or along interface between the interlayer and fiber reinforced matrix layer. These characteristics of crack growth can be observed from a side-view of a typical cracked specimen by means of an optical microscope, as shown in Fig. 8. Where, Δa denotes crack extension length. It is seen that the crack grows in the interlayer when Δa =0 (i.e. near the Kapton film tip), along the interface or in the fiber reinforced layer when crack extension is around 7 mm, again in the interlayer when crack extension is around 42 mm, and then along the interface when crack extension is around 80 mm. These characteristics of crack extension are correlated well with the results of G_{IC} given in Fig. 6.

For instance, in the case of crack initiation, the whisker bridging near the Kapton film tip , as show in Figs. 3, plays an important role in resisting the crack extension and a high initial G_{IC} appears, in the case of 42 mm, the whisker bridging together with fiber bridging leads to a high propagation G_{IC}, while in the cases of 7 mm and 85 mm, the effect of whisker bridging is relatively weak compared with that of fiber bridging since the crack grows along the interface and low values of G_{IC} are observed.



(a) $\Delta a = 0\,\text{mm}$ (b) $\Delta a = 10\,\text{mm}$

(c) $\Delta a = 40\,\text{mm}$ (d) $\Delta a = 85\,\text{mm}$

Fig. 9: SEM images of fracture surfaces at different regions of crack extension.

The following SEM images of fracture surfaces of a typical cracked specimen are also helpful to understand the variation of G_{IC} as shown in Fig. 9.

Figure 9 shows SEM images of fracture surface at different regions of crack extension. Where, the initial crack region in Fig. 9(a) denotes the inserting region of Kapton film. It is seen that many whiskers appear on the surface and the pullout and breaking of whiskers are considered to be the major reason which causes a high initial G_{IC}. In the cases of Figs. 9 (b) to (d), two partial surfaces of whisker reinforced interlayer and fiber reinforced layer are observed. The surface of fiber reinforced layer is similar to the conventional one without whiskers and pullout traces of fibers and fiber breaking are observed. In contrast, the surface of whisker reinforced interlayer is rather complicated, numerous whiskers are observed, the surface appears quite rough and many pullout traces of whiskers are observed. Especially, in the case of (c), the partial surface of whisker reinforced interlayer is relatively large, then it is considered that much energy is needed to forme such surface during crack extension. These images are coincident with the results of G_{IC} given in Fig.6 and they reveal that the whisker bridging plays an important role in enhancing the interlaminar fracture toughness compared with that without whiskers.

CONCLUSIONS

In the present study, an attempt is made to improve the interlaminar fracture toughness of composite laminates by distributing whiskers along the interface of composite laminates. An unidirectional laminate of 24 plies with β-SiC whiskers distributed along the mid-plane is manufactured for DCB testing. A spray method is developed to distribute whiskers on the prepreg. The manufacture process is simple and applicable in practical structures. A microscopic analysis of the edge and cross section of the laminate is performed. The SEM and EPMA images of the side-view and cross section reveal that a whisker reinforced interlayer is formed along the mid-plane. Whiskers are found to be distributed randomly throughout the interlayer except for the small region ahead the Kapton tip and good coherence between whiskers and matrix is observed. Hence, the present spray method is considered to be valid for distributing whiskers on prepregs.

The DCB testing is conducted to investigate the effects of the whisker on the Mode I fracture toughness. The initial G_{IC} exhibits an improvement of nearly 50 % and the propagation one increases by nearly 20 % compared with those without whiskers. The scatter range of G_{IC} between the maximum and minimum values also shows the same tendency. Microscopic analysis of the crack growth path and fracture surface of cracked specimens by the optical microscope and SEM. Exhibits that the whisker bridging plays an important role in enhancing fracture toughness G_{IC} of the interlaminar. Hence, it is concluded that the present method with whiskers distributed on prepregs is simple and valid to improve the interlaminar fracture toughness of laminated composites.

Further researches are necessary to investigate the effects of whiskers on the interlaminar strength of multidirectional laminates and on the mode II fracture toughness.

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