NOVEL TEST METHOD FOR CHARACTERIZING ACCURATE INTRA-LAMINAR FRACTURE TOUGHNESS IN CFRP

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Abstract
A novel initial crack insert method, "intra-laminar film method", in which a release film is inserted inside a single lamina prepreg, was proposed for intra-laminar fracture toughness test. Mode I intra-laminar fracture toughness tests (DCB test) were carried out for unidirectional CFRP laminates, in which the initial crack was introduced using intra-laminar film method. For comparison, two conventional methods were used to introduce initial cracks. "Inter-laminar film method" is to insert release film between prepregs in lay-up process. "Machined slit method" is to machine slit in parallel to layer of CFRP laminates. As a result, it was demonstrated that the proposed method "intra-laminar film method" is the only experimental way to correctly evaluate the intra-laminar fracture toughness of CFRP laminates from the initial value to the propagation value.

1 Introduction

Delamination is one of the design limited factors for CFRP laminates, and many researchers have discussed how to characterize delamination resistance for several decades[1,2]. Test method for inter-laminar fracture toughness has already been standardized by ISO, ESIS, JIS, and ASTM[1]. Here, the initial crack was introduced between prepreg layers using release films. On the other hand, the testing methods for intra-laminar fracture toughness have difficulties introducing initial crack at proper location in an intra-layer region. Here, the delamination should propagate inside a prepreg layer without deviating to the inter-layer region. When machined slit was inserted in intra-layer region, initial values of the fracture toughness were higher owing to rather large slit root radius. When pre-crack was introduced from the slit by using knifes, large amount of bridging fibres brought higher initial values of the fracture toughness[1]. The other methods were also proposed, in which the mechanical slit was introduced in transverse direction, ie. perpendicular to the lamination plane[3,4]. However, in this method, the crack face is not coincided with the plane of the laminate, the fracture toughness measured by this method was probably different from the in plane fracture toughness of the laminate.

In this study, a novel initial crack insert method, "intra-laminar film method", was proposed for measuring intra-laminar fracture toughness in the out-of-plane direction, and Mode I intra-laminar fracture toughness tests (DCB test) were carried out for unidirectional CFRP laminates, where almost natural intra-laminar initial crack was introduced by inserting film inside a single lamina prepreg. For comparison, two conventional methods were used to introduce initial cracks. "Inter-laminar film method" is to insert release film between prepregs in lay-up process. "Mechanical slit method" is to machine slit in parallel to layer of CFRP laminates.

2 Materials and testing methods

2.1 Materials

For the reinforced fibre, PAN-based intermediate modulus, high tensile strength fibre (diameter 6 μm, tensile modulus 290 GPa) was used. For the matrix resin, Di-Glycidyl Ether of Bisphenol A epoxy (EEW 189 g/mol) was mixed with 4,4'-diamino diphenyl sulfone (DDS) and Polyethersulfone(PES) in ratios of 100:33:15 by weight. Using these materials, the prepregs were fabricated by using drum winding method. A nominal cured ply thickness of the prepreg was 0.2mm. These prepregs were stacked unidirectionally, and the CFRP laminate was cured in an autoclave at 180 degree. The nominal fibre volume fraction of the CFRP laminate was 60%.

2.2 Proposal of concepts of intra-laminar film method

In order to discuss the potential of the proposed method, initial cracks for fracture toughness test were inserted by these 3 methods, (a) Inter-laminar film insert method, (b) Intra-laminar film insert method, and (c) Mechanical method. Figure 1 shows the schematic drawing of cross-sections of each specimen. The stacking sequence for Inter-laminar film insert method was (0_{20}), and PTFE film (thickness 13μm) was inserted in the centre of the laminate in lay-up process. The stacking sequence for Intra-laminar film insert method was (0_{19}), and the PTFE film was also inserted in the centre of the laminate during the lay-up process. The detail of inserting method of the PTFE film is described in next section. The stacking sequence for Mechanical method was (0_{20}). Mechanical notch (width 0.4mm) was inserted in the centre of the specimen out of plane direction using a diamond coated rotating blade. The widths and heights of each specimen are (a) 12.7mm and 4.0mm, (b) 12.7mm and 3.8mm, and (c) 4.0mm and 4.0mm. Here, the width of the specimen of mechanical method was smaller than others. The reason is that in mechanical method, it was difficult to introduce an initial crack parallel to the lamination plane. In all cases, a pre-crack was not installed.

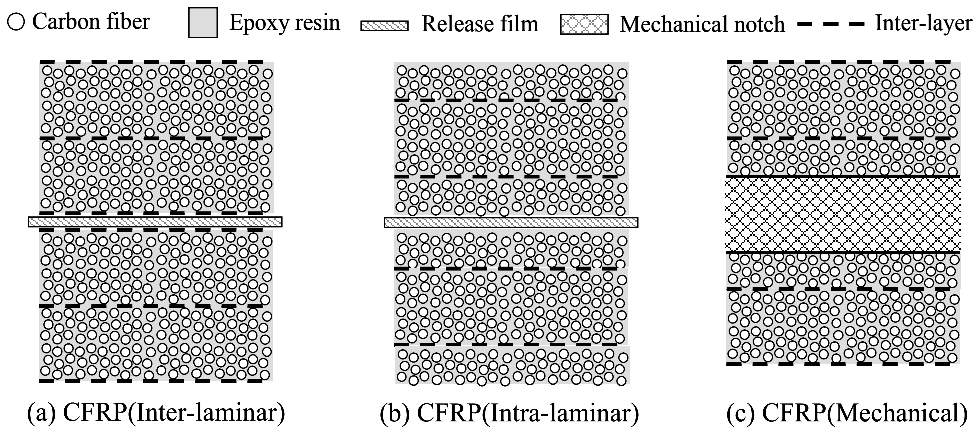

Figure 1 Schematic drawings of cross-section of CFRP.

After this section, for the sake of simplicity, a CFRP plate, in which the initial crack is inserted by inter-laminar film inserted method, was called as "CFRP(Inter-laminar)", a CFRP plate, in which the initial crack was inserted by intra-laminar film inserted method, is called as

"CFRP(Intra-laminar)", a CFRP plate, in which the initial crack was inserted by mechanical method, is called as "CFRP(Mechanical)"

2.3 The detail of Intra-laminar film insert method

In the proposed intra-laminar film insert method, a release film is directly inserted in the intra-layer region for the initial crack of fracture toughness test. In conventional prepreg manufacturing process, a fibre sheet is fabricated by aligning fibre tows in one-direction. The resin is warmed up to reduce the viscosity of the resin, and the resin is impregnated completely in the fibre sheet. Then, it is quite difficult to open the fibre sheet in the out of plane direction and insert a release film. On the other hand, the resin temperature is kept lower than one of the conventional process in this study, in order to keep the high viscosity during the prepreg fabrication process. Here, the resin is impregnated only in the surface regions of the fibre sheet. This semi-impregnated prepreg is opened in the out-of-plane direction, and a release film is inserted in the intra-layer region. The concrete procedure is as follows.

Figure 2 shows the schematic drawing of cross-section of the prepreg for each process in Intra-laminar film insert method. In the first step, carbon fibre tows were aligned in one-direction as a sheet, and this carbon fibre sheet was placed between resin films (Figure 2(a)). In the second step, the temperature of this sheet was kept at 40 °C for 3 minutes in order that resin was impregnated into the surface region of the carbon fibre sheet (Figure 2(b)). Then, the temperature of the sheet was reduced to room temperature again. While the surface regions of the sheet were impregnated with the epoxy resin, the intra-layer region was not impregnated yet. In the third step, this semi-impregnated prepreg was opened in the out of plane direction, and a PTFE film (thickness 13mm) was directly inserted in the intra-layer region (Figure 2(c)). Finally, the temperature of the semi-impregnated prepreg was kept at 70 °C for 10 minutes, the resin was enough impregnated in the sheet (Figure 2(d)). This prepreg was located at the centre of lay-up, and unidirectional laminates were cured in an autoclave. In this way, CFRP laminates, in which release film was inserted in intra-layer region, were fabricated.

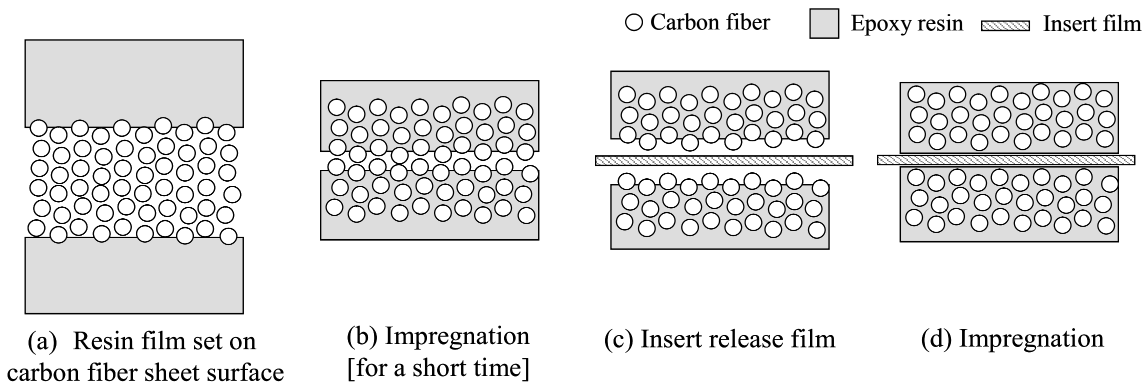

Figure 2 The schematic drawing of the method to insert a release film into intra-layer region.

2.4 Mode I Fracture toughness test method

Mode I fracture toughness tests were carried out using double cantilever beam (DCB) specimens according to JIS K 7086[5]. Aluminum blocks (10mm x 10mm) with diameter 4 mm hole at the centre were glued at the loading points. The length between the loading line and the edge of the initial crack was 25mm. The edge of specimen was painted with a white marker in order to enhance the crack length measurement. The cross head speed was slightly changed during DCB test, from 0.5mm (Δa<20mm, Δa : the increment of crack length) to

1.0mm (Δa≥20mm). The energy release rate was calculated by the modified compliance calibration method (MCC)[5]. The initial value of fracture toughness was defined as the fracture toughness at nonlinear point of load displacement curve. For each film insert method, DCB test was carried out for 3 specimens.

3 Result and discussion

3.1 Comparison between intra-laminar and inter-laminar film insert method

Figure 3 compares the average values of the initial values of fracture toughness of CFRP(Inter-layer), and CFRP(Intra-layer), and the error bars indicates the maximum values and the minimum values. Though the initial value of CFRP(Intra-laminar) was slightly higher than the CFRP(Inter-layer), the difference was only 20 J/m^2. There was no significant difference between CFRP(Inter-layer) and CFRP(Intra-layer).

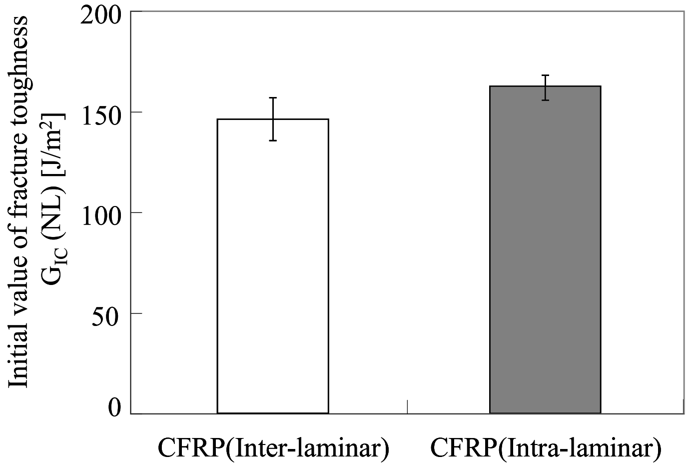

Figure 3 Initial value of fracture toughness (Comparison with Inter-laminar film insert method).

Figure 4 shows SEM images of the fracture surfaces at the initial stage of crack propagation. In these images, flat faces on the left hand side correspond to the location of the insert PTFE film. The arrow indicates there crack growth direction. In both the samples, most of the fracture surfaces are covered with carbon fiber surfaces, trace of fiber debonding, and fracture surfaces of epoxy resin.. There was no significant difference in the area of fibre exposure and depth of fibre debonding, between CFRP(Inter-laminar) and CFRP(Intra-laminar). The alignment of fibres on the fracture surface of CFRP(Intra-laminar) was lower than that of CFRP(Inter-laminar). In the intra-laminar film insert method, the prepreg was wedge-opened and the release film was inserted at the location without resin impregnation in the intra-layer region. Thus, some fibres on the contact face of the release film were easy to move, and therefore, the alignment of fibres became slightly low. This lower alignment probably improve the fracture toughness of CFRP(Intra-laminar) compared to CFRP(Inter-laminar).

Figure 5 shows the relationship between the increment of crack length Δa from the tip of the release film and the fracture toughness G_{IC} (R-curve). Each plot indicates averaged value for every Δa=2mm, and error bars indicate the maximum and the minimum values. For both laminates, the fracture toughness increased owing to the fibre bridging effect with the increase of the crack length[6]. The gradient of the R-curve of CFRP(Intra-laminar) was higher than that of the CFRP(Inter-laminar), and the saturated propagation values of the fracture toughness of CFRP(Intra-laminar) were also higher than those of CFRP(Inter-laminar).

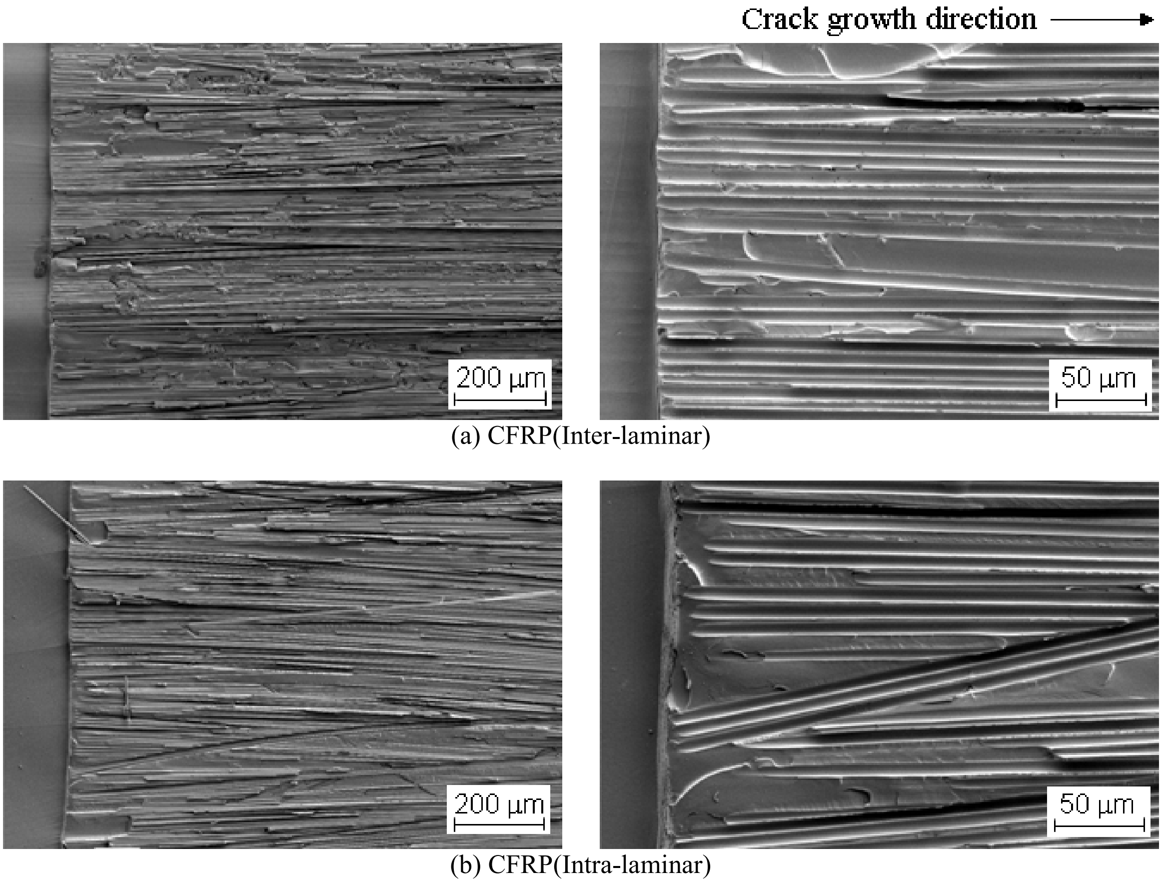

Figure 4 SEM images of fracture surface of CFRP(Inter-laminar) and CFRP(Intra-laminar).

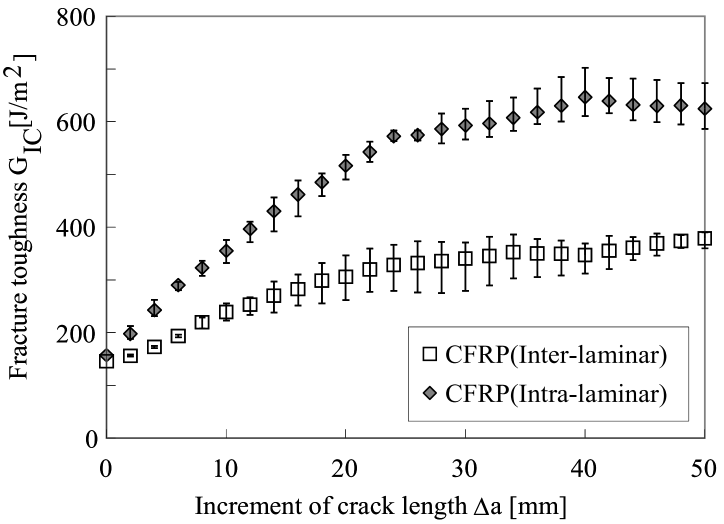

Figure 5 R-curve (Comparison with Inter-laminar film insert method).

In order to evaluate fibre bridging effect more quantitatively, the number of bridged fibres was counted directly by observing the cross section of the specimen after DCB test. In particular, we separate the number of this bridged fibre bundle and the number of fibres within this fibre bundle. Here, a bridged fibre bundle indicates fibres connected by matrix resin each other.

In order to count the number of bridged fibres more accurately, the space between upper and lower fracture surfaces was filled with cyanoacrylate adhesive, the bridged fibre was fixed

with instant glue filled in fracture face after DCB test, and the cross-section was polished with diamond paste. Figure 6 shows one of the typical optical micrographs of the cross section.
Figure 7 shows the relationship between the distance from the edge of the release film to the observed cross section and the total number of bridged fibres per unit width in the cross section. In all cross sections, number of bridged fibres of CFRP(Intra-laminar) was more than one of the CFRP(Inter-laminar).
Figure 8 shows the relationship between number of fibres in each bridged fibre bundle and the probability of bridged fibre bundles. Here, the probability of bridged fibre bundles indicates the number of bridged fibre bundles standardized by the total number of bridged fibre bundles. The observed cross-section was Δa = 30mm. In both cases, 10 to 15 % of all bridged fibre bundles contained only one fibre. The number of bridged fibre bundles decreased with increasing of number of fibres within the fibre bundle. The number of bridged fibre bundles of CFRP(intra-laminar) was higher than the number of CFRP(Inter-laminar) without respect to the number of fibres within fibre bundles. In particular, when the number of fibers within fibre bundles, are more than ten, a certain numbers of bundles were observed only for CFRP(Intra-laminar). It was theoretically demonstrated that the propagation values of the fracture toughness increase with increasing number of bridged fibre bundles and thickness of the fiber bundle[6]. Thus, these differences in number of bridged fibre bundles and thickness of the fiber bundle(number of fibres within the fibre bundle) probably caused the difference between the propagation values of fracture toughness.

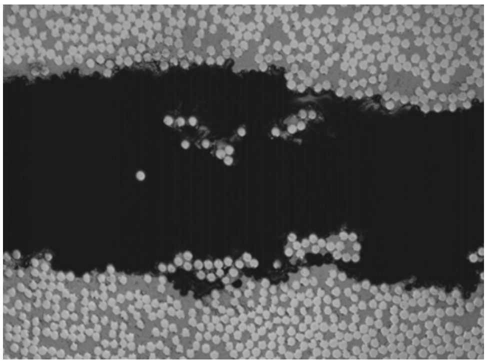

Figure 6 Optical micrographs of cross section of CFRP(Inter-laminar), Δa=30mm.

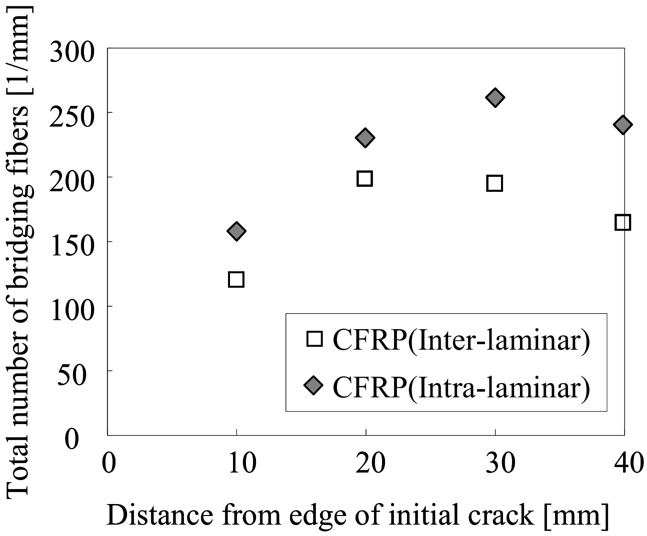

Figure 7 Total number of bridged fibres per unit width length

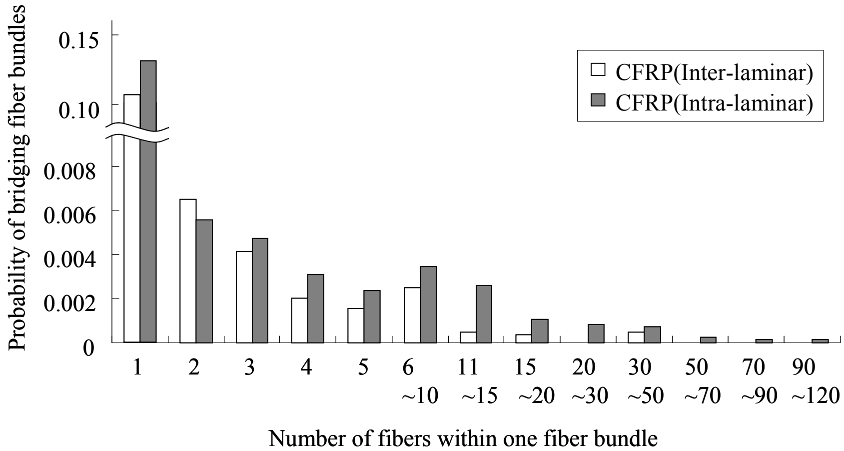

Figure 8 Relationship between the number of fibres within one fibre bundle and probability of bridged fibre bundles

3.2 Comparison between Intra-laminar film insert method and Mechanical method

Figure 9 shows the average of the initial value of fracture toughness of CFRP(Mechanical). For comparison, the those of CFRP(Inter-laminar) and those of CFRP(Intra-laminar) are also indicated in Figure 9. The initial value of fracture toughness of CFRP(Mechanical) was 260J/m^2. This value was twice as much as that of CFRP(Inter-laminar) and CFRP(intra-laminar). The thickness of the initial defect in CFRP(mechanical) was 0.4mm, and this thickness was 30 times as high as that of CFRP(Intra-laminar) and CFRP(Inter-laminar). The larger radius of crack tip of CFRP(mechanical) was responsible for the higher initial values of the fracture toughness of CFRP(mechanical).

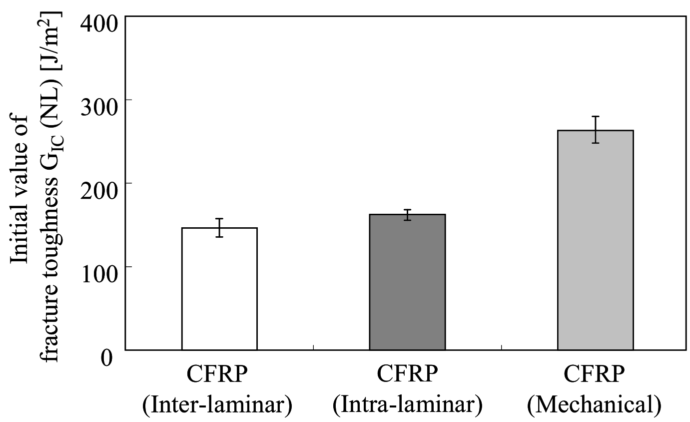

Figure 9 Initial value of fracture toughness (Comparison with Mechanical slit method).

Figure 10 shows the R-curve of CFRP(Mechanical). The R-curve of CFRP(Inter-laminar) and the R-curve of CFRP(Intra-laminar) were reproduced in Figure 9 for comparison. The error bars indicate the maximum and minimum value of the fracture toughness. In the case of CFRP(Intra-layer) and CFRP(Inter-layer), the initial value of fracture toughness was lowest in each R-curve. On the other hand, in the case of CFRP(Mechanical), the propagation value of the fracture toughness(Δa=4mm) was the lowest. From the point of view of composite structural design, minimum value of fracture toughness is important in order to prevent crack propagation completely. Thus, it should be important to point out that the initial value of fracture toughness is higher than the potential value of the material system when the mechanical slit method was employed. For these reasons, the proposed method ”intra-laminar film method” is the ideal experimental way to evaluate the inter-laminar fracture toughness of CFRP laminates from the initial value to the propagation value.

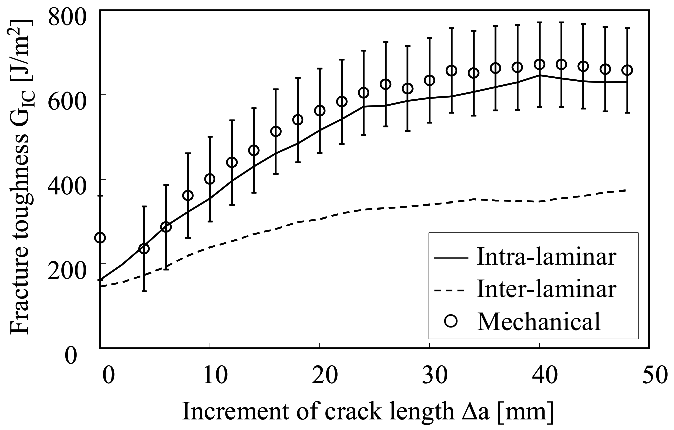

Figure 10 The R-curve (Comparison with mechanical slit method)

4. Conclusion

The novel initial crack insert method, "intra-laminar film method", was proposed for intra-laminar fracture toughness test, and Mode I intra-laminar fracture toughness tests were carried out for unidirectional CFRP laminates, in which the initial crack was introduced using intra-laminar film method. For comparison, two conventional methods were used to introduce initial cracks. "Inter-laminar film method" is to insert release film between prepregs in lay-up process. "Machined slit method" is to machine slit in parallel to layer of CFRP laminates. The results were summarized as follows:

1. There is no significant difference for the initial value of fracture toughness between inter-laminar film insert method and intra-laminar film insert method. On the other hand, the initial value of fracture toughness of mechanical slit method was twice as much as that of the other two methods. It should be noted, that in the case of mechanical slit method, initial value of fracture toughness gives non-conservative value.
2. As for the propagation value of the fracture toughness, the fracture toughness of the intra-laminar film insert method was higher than that of the inter-laminar film insert method. The number of bridged fibres of intra-laminar film insert method was higher than that of the inter-laminar film inserted method.Increased number of bridged fibers was responsible for the higher fracture toughness of CFRP(Intra) with Intra-laminar film insert method.
3. It was demonstrated that the proposed method "intra-laminar film method" is the only experimental way to evaluate the inter-laminar fracture toughness of CFRP laminates from the initial value to the propagation value.

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