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Fractographical analysis on the mode II delamination in woven carbon fiber reinforced epoxy composites

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Mode II delamination phenomena of woven fabric carbon/epoxy composites were investigated by scanning electron microscopy. End notch flexural (ENF) test was used to examine the mode II delamination. Woven fabric composites showed two peculiar crack propagation patterns due to the complexity of woven geometry. In warp yarn region, crack propagated with forming a shear band and breaking the fiber/matrix interface. In fill yarn region, however, no shear band was observed. Considering these crack patterns, matrix shear property and fiber/matrix interfacial strength played an important role in enhancing the delamination properties of woven fabric carbon/epoxy composites. Due to the woven geometry, matrix rich positions, which are interstitial and undulated region, were formed in woven carbon/epoxy composite. In these regions, matrix fracture and complex crack path were mainly observed. © *1999 Kluwer Academic Publishers*

1. Introduction
Carbon/epoxy composites have been widely used as advanced composite materials due to their excellent properties, such as high specific tensile modulus and strength, good abrasion resistance, fatigue properties and good chemical resistance [1, 2]. These excellent properties made it possible for carbon/epoxy composites to be used in aerospace and civilian and military aircraft [3].

Since carbon/epoxy composites for aircraft applications are apt to suffer the shear stress in use, improving the delamination resistance of carbon/epoxy composite to shear stress is indispensable for enlarging its applications. Therefore, controlling the crack propagation between laminates by shear stress has become one of the most important research issues in carbon/epoxy composites [4–9].

There have been many papers concerning on the mode II delamination fracture behavior of carbon/epoxy composites. These are mainly focused on the unidirectional carbon fiber reinforced composites [4–7]. As for woven fabric composites, however, little investigation has been performed [8, 9]. This is partially because woven fabric composites have been used less frequently than unidirectional composites and partially because the delamination phenomenon of woven fabric composites is more complicated than that of unidirectional composites due to the geometrical characteristics of woven fabric.

Nowadays, woven fabric composites are used more frequently than before due to their easiness in composites manufacturing and uniform strength in two directions. With this reason, although it is difficult to interpret the mechanical behavior of woven fabric composites, the necessity for understanding it grows larger and larger. Therefore, much attention is paid to the woven fabric composites.

In this paper, end notch flexural (ENF) test was carried out to examine the crack propagation phenomena in woven fabric carbon fiber reinforced epoxy composites. The factors affecting the mode II delamination of woven carbon/epoxy composites were determined by analyzing the fracture surface of carbon/epoxy composites following the crack path with scanning electron microscopy. In particular, the characteristic difference between the woven and unidirectional geometry were also one of our main research concerns.

2. Experimental
2.1. Materials
Tetrafunctional type epoxy resin, N,N,N',N'-tetraglycidyl-α,α'-bis-(4-amino-phenyl)-p-diisopropylbenzene (TGBAP) and amine type curing agent, α,α'-bis-(4-amino phenyl)-p-diisopropylbenzene (BAP) were used as a matrix. The chemical structures of TGBAP and BAP are illustrated in Fig. 1. These materials were supplied by Shell Chemical Co. and used as supplied without further purification. The physical properties of these materials are presented in Table I.

Carbon fiber used in this study was Torayca T300. Its physical form was plain fabric. Table II shows the

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TABLE I Typical properties of TGBAP epoxy compound and BAP curing agent

	Epoxy compound	Curing agent
Physical form	Dark solid	Free flowing solid powder
Epoxy equivalent weight	150–170	
Melting point[a]	50 °C	161–164 °C
Melting viscosity (110 °C)[b]	1.8–2.2 Pa s	
Equivalent weight per active hydrogen		86
T_g[c]	22.8 °C	

[a]ASTM D3461, Mettler, 1 °C/min.
[b]Brook Field viscometer.
[c]Differential scanning calorimetry.

TABLE II Typical properties of carbon fiber[a]

Number of filament	Tensile strength	Tensile modulus	Ultimate strain	Density
3k	3.53 GPa	230 GPa	1.50%	1.77 g/cm

[a]Trade name: Torayca T300.

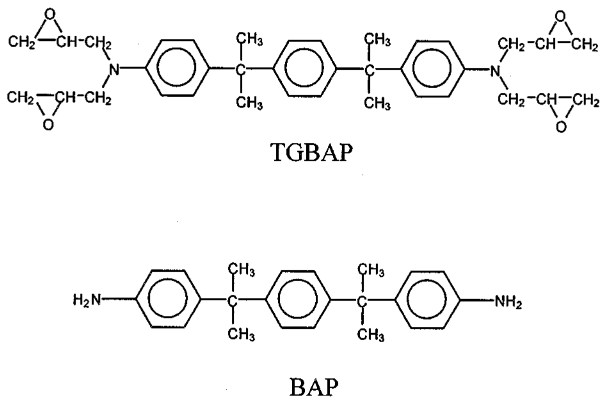


Figure 1 Chemical structures of TGBAP and BAP reagent.

basic properties of Torayca T300. Carbon fiber was used after desizing. Fiber desizing was carried out as follows. Carbon fiber was refluxed in dichloromethane for 5 days and in distilled water for 2 days, consecutively. Desized carbon fiber was dried at 120 °C for more than 5 days.

2.2. Composite manufacturing

Carbon/epoxy prepreg was made by hand lay-up method. The composition of resin bath used for prepreg preparation is given in Table III. Carbon/epoxy prepreg was dried in a hood at room temperature for 1 day and then dried *in vacuo* at 80 °C for 3 h.

Carbon/epoxy composites were manufactured by vacuum bag moulding technique according to the moulding cycle represented in Fig. 2. In vacuum bag moulding, 10 Torr of vacuum was applied through out the manufacturing process. The average fiber volume fraction of manufactured composite was 65%.

TABLE III The composition of resin bath for carbon/epoxy prepreg

Component	TGBAP resin	BAP curing agent	Acetone (solvent)
Weight(g)	21	9.45	60

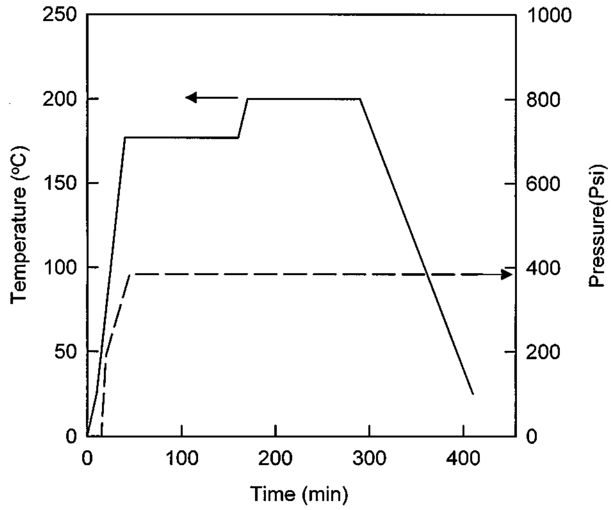


Figure 2 Moulding cycle of woven fabric carbon/epoxy composites.

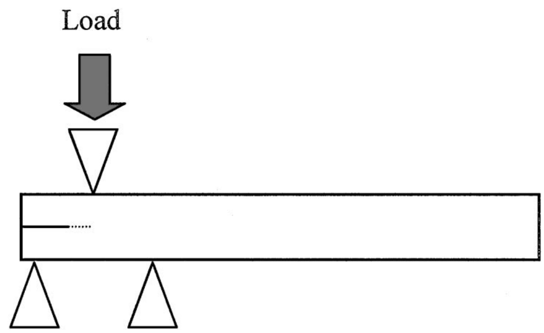


Figure 3 Crack introduction of ENF test specimen.

2.3. Measurement

The specimens for ENF test were made with inserting 25 μm of polyimide film whose dimension was 20 × 20 mm to the center of specimen as a crack starter. The dimension of ENF specimen was 100 × 20 × 2.5 mm. Sharp crack was introduced in advance to ENF test to facilitate the ENF fracture of carbon/epoxy composite. This action was performed using a universal testing machine model Lloyd LR10k as shown in Fig. 3. In this step, crosshead speed was 1.3 mm/min. The ENF test of carbon/epoxy composite including sharp crack was carried out by moving the crosshead at the speed of 2.5 mm/min. At least 5 specimens were tested in this measurement. The detailed test information was given in other reference [10, 11].

The analysis on the fractured surface of carbon/epoxy composite was performed with scanning electron microscopy (SEM) model JEOL-840A. Samples for SEM analysis were prepared from the fractured specimens using a diamond coated saw. Prior to gold coating, saw debris of surface was removed by pressurized air. 100 Å of gold layer was coated on the fractured surface of carbon/epoxy composites to enhance the resolution. In this experiment, acceleration voltage was 15 kV.

SEM micrographs of various features were taken from the fractured surface of carbon/epoxy composite along the crack path. In all micrographs, crack propagates from the bottom to the above.

3. Results and discussion

In order to elucidate the mode II delamination mechanism of woven fabric composites, the geometrical characteristics of woven fabric and fracture surface of woven fabric reinforced composites were examined in this study.

3.1. Difference between unidirectional fiber and woven fabric

Woven fabrics are composed of warp and fill yarns, which are interlaced in an alternative over-and-under pattern. The typical appearance of plane type woven fabric is illustrated in Fig. 4. There is one warp yarn for one fill yarn without skipping. Compared with unidirectional fiber, woven fabric has different features due to its peculiar physical form. It is worthwhile to note that two unique positions exist in woven fabric. One is the interstitial position, which is surrounded with 4 different yarns, and the other is undulated position, which is defined as the intersection point of warp and fill yarns. Compared with the other positions, these two positions become resin rich region in the fiber reinforced polymer composite.

Considering the geometry of woven fabric, crack propagation in woven fabric composites is rather complex than that of unidirectional composites. In the case of unidirectional composites, crack propagates along the either of following two directions. One is parallel to fiber direction and the other is perpendicular to the fiber direction. Crack propagation pattern is determined by the relative direction of crack path to fiber alignment. In woven fabric composites, however, above two different crack paths happen together due to the woven geometry. Two different crack propagation patterns appear repeatedly at an interval of yarn width. Therefore, it is easily expected that weaving type of fabric can affect the crack propagating pattern.

3.2. Fractographical analysis

Fig. 5 shows the schematic diagram of two possible crack paths observed in woven fabric composites. Crack

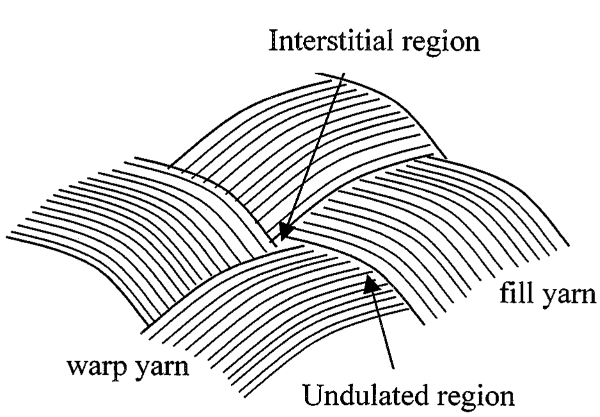


Figure 4 Sketch of typical plane type woven fabric.

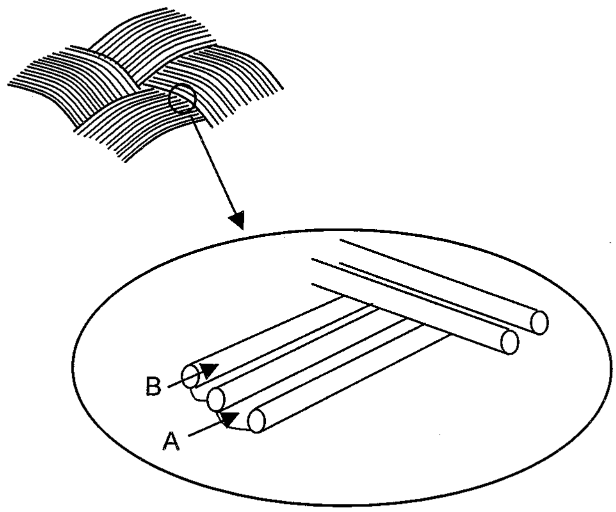
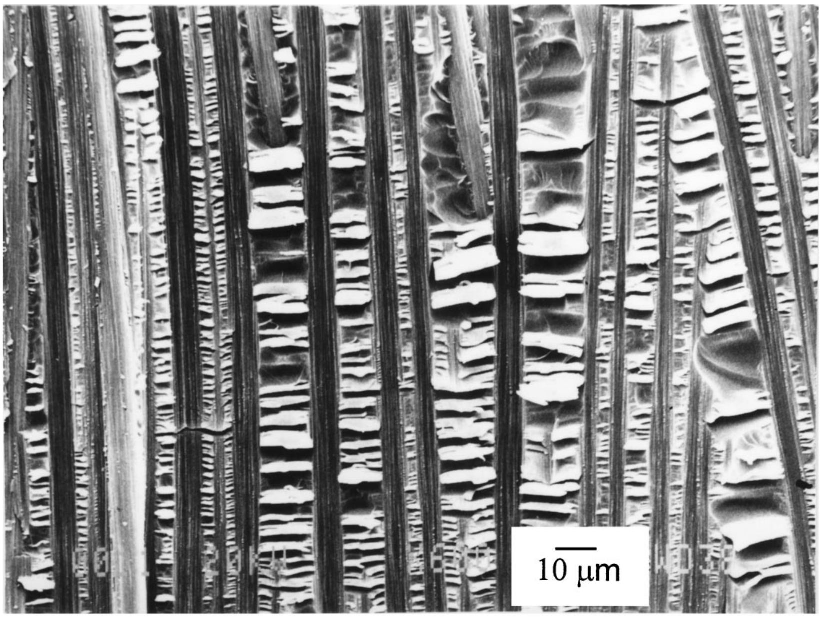


Figure 5 Two possible crack paths in woven fabric (A: matrix → fill filament, B: fiber/matrix interface → fill filament).

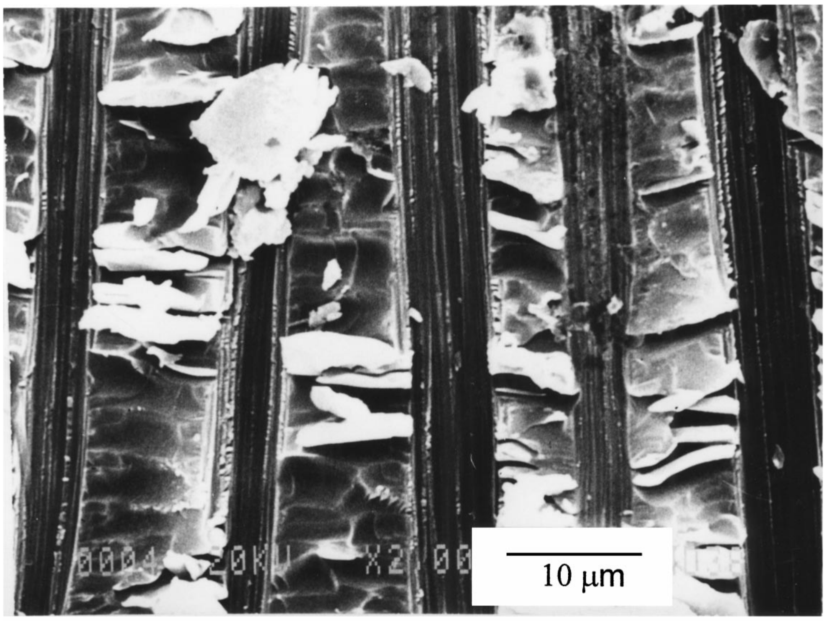
propagates from warp yarn to fill yarn through the matrix region (path A) and the fiber/matrix interface (path B). Therefore, the path A and B are distinguished from each other in that fracture phenomena occurred in warp yarn region are different. Fig. 6 represents the fracture surface of warp yarn region of woven fabric carbon/epoxy composite. When crack propagates along the path A, matrix part shows a hackle structure, which is commonly observed in mode II failure. The size of hackle structure seems to depend on the interspace between fibers. In path B, however, it is difficult to observe this hackle pattern. These phenomena can be explained by understanding the mode II stress state of fiber/matrix composite.

The schematic diagram of fiber/matrix composite, which is deformed by pure shear stress, is displayed in Fig. 7. As each end deforms in the opposite direction, the forced stress is concentrated at these ends. If the fiber/matrix interface is strong enough to endure this stress, the microcrack is formed at the crack front as the applied shear stress increases. When the maximum effective stress at crack tip reaches the critical value that causes microcrack to coalesce to macrocrack, the macroscopic crack advances [12–14]. Therefore, the typical morphology caused by shear stress, which is called shear band, is observed in this fracture surface. However, if fiber/matrix composite has weak interfacial strength, the adhesive failure occurs at this weak interface within small deformation prior to coalescence of microcracks and the shear band was not formed in this case. Considering the crack propagating pattern, it could be suggested that the propagation rate of crack accompanying the shear band be slower than that of crack without shear band.

On the other hand, the fracture phenomena occurred at the fill yarn region are remarkably different from those of warp yarn region. These differences are well understood by Fig. 8. This represents the fracture surfaces of fill yarn region. Compared with warp yarn region, the fill yarn region does not show the shear band and its morphology is similar to that of interfacial failure. This means when the propagating crack reaches



(A)



(B)

Figure 6 SEM photographs of fracture surface of warp yarn region in carbon/epoxy composites (many shear bands are observed in this region): (A) ×600, (B) ×2000.

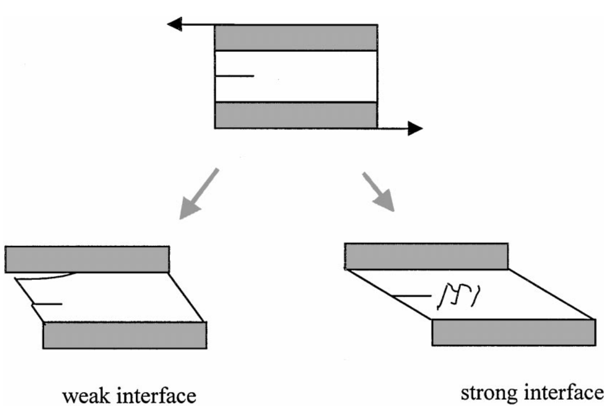
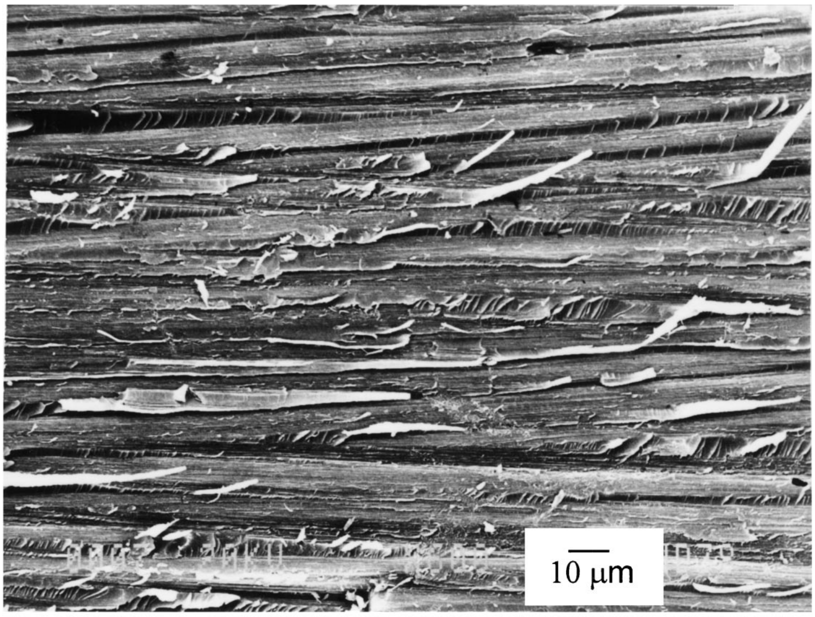


Figure 7 Schematic diagram of fiber/matrix composite deformed by pure shear.

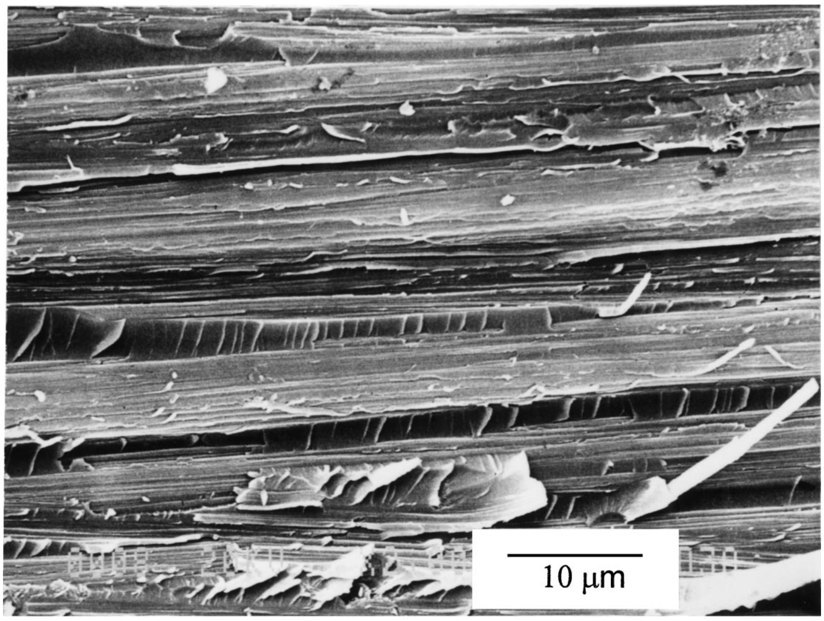
the fill yarn, the crack goes over the fill yarn with the interfacial breakage without forming a shear band.

As seen in Fig. 8, fill yarn region is not divided into two crack paths, which is different from warp yarn region. In other words, fill yarn region has the uniform property to the direction of crack propagation. In this region, because fiber is aligned perpendicular to the crack propagation, crack starts at the interface of fill yarn and matrix before the matrix deformation approaches the level that forms a shear band. Therefore, the crack propagates along the fiber/matrix interface and transfers to the neighboring matrix.

Considering the both fracture phenomena observed in warp and fill yarn region, two different crack paths can be suggested as shown in Fig. 9. Judging from these



(A)



(B)

Figure 8 SEM photographs of fracture surface of fill yarn region in carbon/epoxy composites (it is difficult to observe the shear band): (A) ×600, (B) ×2000.

fracture phenomena, fiber/matrix interfacial strength and the shear property of matrix are important factors affecting the mode II delamination resistance of woven fabric composites.

Another factor, which should be considered seriously in woven fabric composites, is the resin rich region. The effect of resin rich region on the mode II delamination was not considered in Fig. 9. As previously mentioned, the interstitial and undulated regions of woven fabric become resin rich region. Fig. 10 shows the fracture surface of undulated region of woven fabric composite. It is interesting to observe the debris of upper laminate at the boundary of warp and fill yarns. This debris implies that crack propagated to the fiber/matrix interface of upper laminate at this point. If the crack propagates only along the fiber/matrix interface of lower laminate, this debris could not be formed. This fracture pattern comes from the fact that warp yarns go down the fill yarns at this undulated region. As the crack approaches the undulated region, crack propagating plane gradually recedes from the fiber/matrix interface due to the geometrical characteristic of this region. Considering this fact, the schematic diagram of crack path at the undulated region could be suggested as Fig. 11. As a result, the fracture pattern of this region means that the geometry of undulated region affects the crack path of woven fabric composites.

Fig. 12 shows the fracture surface of interstitial region, another resin rich region of woven fabric carbon/epoxy composite. As seen in Fig. 12, the interstitial

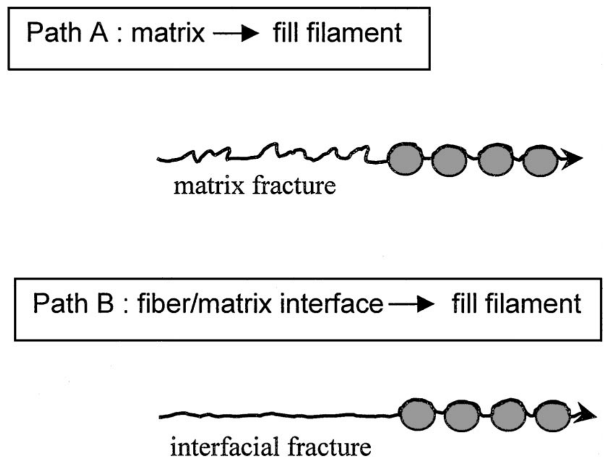


Figure 9 Schematic diagram of crack path occurred in woven fabric carbon/epoxy composites (intersection region is not considered).

region was mainly composed of matrix resin, therefore matrix fracture was mainly observed here. With detailed examination, it could be known that a part of laminate, which was closed to interstitial position, was disjoined with matrix fracture. Considering the morphologies of fracture surface, this rather complex fracture pattern in interstitial region seems to originate from the fact that massive matrix fracture was predominant in this region and crack path became complicate, as seen in undulated region, due to the geometrical characteristic. Although it was difficult to know the exact cause of this fracture morphology, it was obvious that the fracture surface of this region was greatly affected by the matrix fracture. Therefore, it could be concluded that the delamination resistance of woven fabric composites is considerably enhanced by the modification of brittle matrix fracture occurred in this interstitial region.

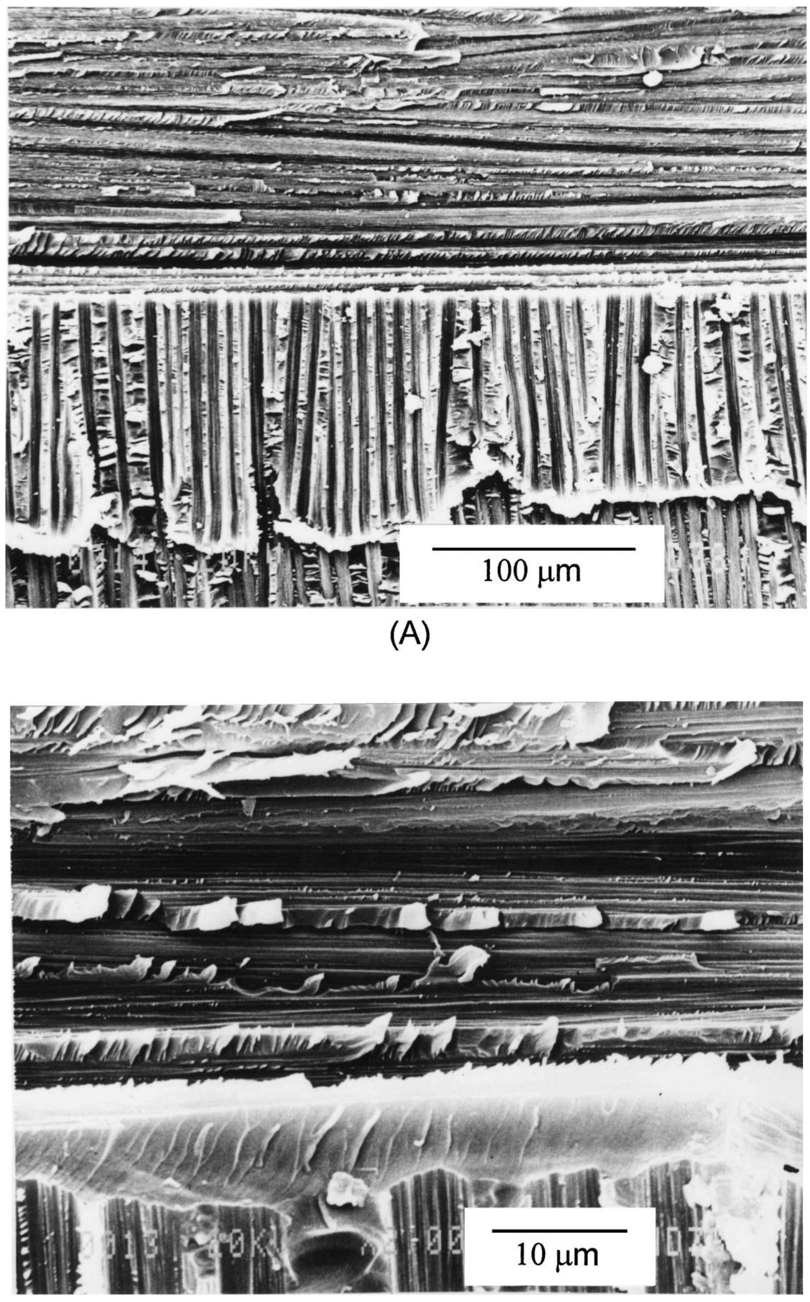


Figure 10 SEM photographs of fracture surface of undulated region in woven fabric carbon/epoxy composites: (A) ×300, (B) ×2000.

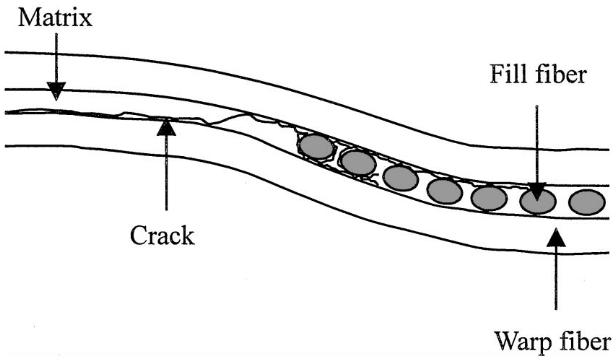


Figure 11 Schematic diagram of crack path at the undulated region of woven fabric composite.

4. Conclusions

From the fractographical study on the mode II fracture of woven fabric carbon/epoxy composites, the following conclusions were obtained.

As for woven fabric composites, their mode II delamination fracture was different from that of unidirectional composites. From SEM analysis on the fracture surface of woven fabric composites, two different crack paths were observed due to the geometrical factor of fabric. One was that crack propagated to the fill yarn region undergoing matrix fracture in the warp region. The other was that crack propagated along the fiber/matrix interface in the warp region and then it advanced to the fill yarn region. Considering these crack

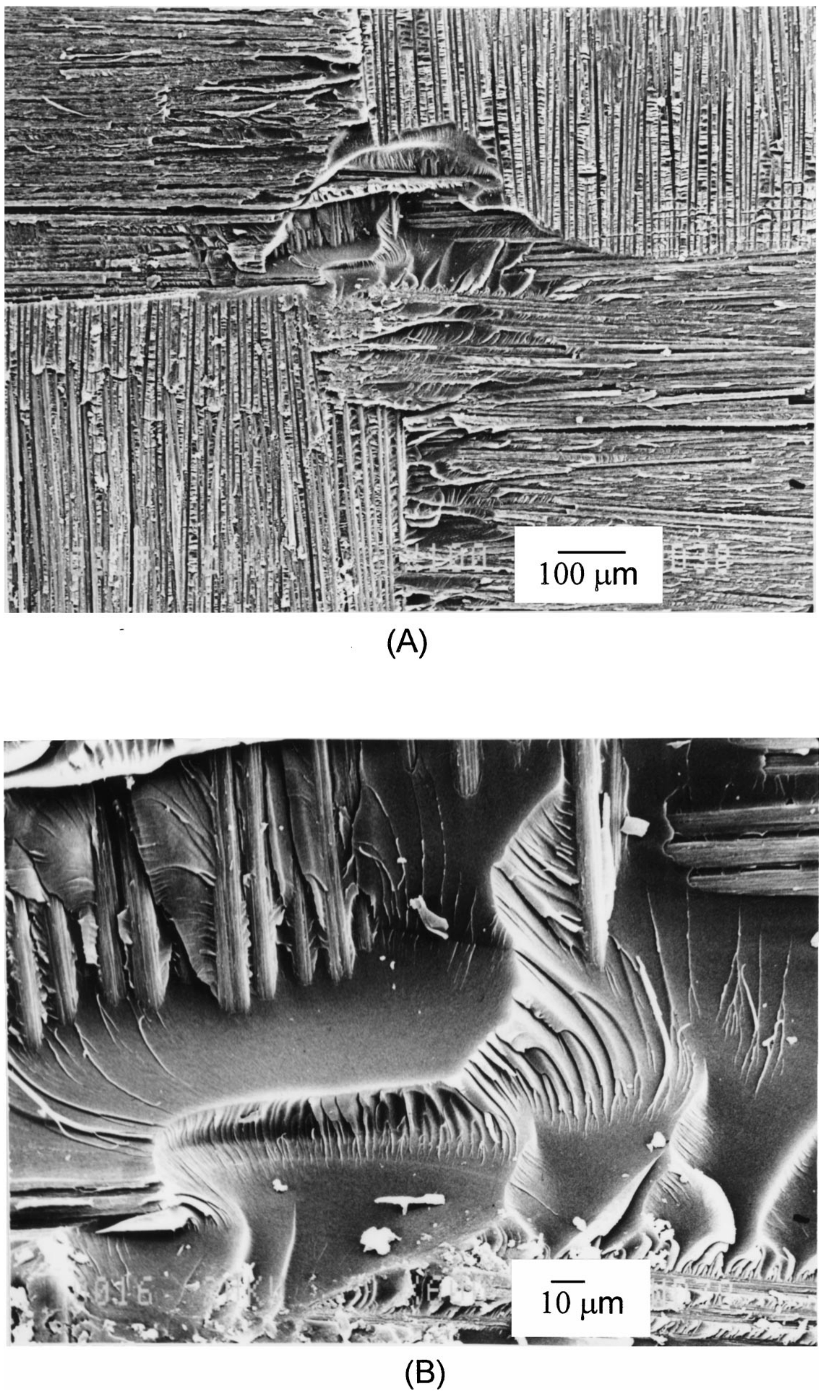


Figure 12 SEM photographs of fracture surface of interstitial region in woven fabric carbon/epoxy composites: (A) ×100, (B) ×600.

paths, it could be known that the shear property of matrix and the fiber/matrix interfacial strength of woven fabric composite were crucial factors in controlling the delamination.

Interstitial and undulated regions, which were peculiar parts in woven fabric composite, yielded in the form of brittle matrix fracture and showed complex crack pattern due to their geometrical characteristics. Therefore, it can be concluded that the suppression of the matrix fracture at these regions due to the modification of matrix resin will increase the delamination resistance of woven fabric composite.

As a result, the modification of matrix and the control of fiber/matrix interfacial strength will change the mode II delamination resistance of carbon/epoxy composites.

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