Study on Properties of Interlayer Short Fiber Reinforced Carbon Fiber/Epoxy Composite Laminates

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Abstract. In order to improve the interlaminar delamination resistance of the carbon-fiber/epoxy composite laminates, short fibers were applied between layers. The effects of length, density, type of short fiber on mode I fracture toughness ($G_{IC}$) and mode II fracture toughness ($G_{IIC}$) were investigated by three-factor and four-level orthogonal test combined with range analysis and scanning electron microscope observation. Finally, parameter optimization and test verification. The results show that the order of factors influencing $G_{IC}$ and $G_{IIC}$ within the selected parameters are: fiber type, fiber density and fiber length. When Kevlar fibers with a length of 11 mm and a density of 20 g·m$^{-2}$ were laid between layers, the $G_{IC}$ was 451.4 J·m$^{-2}$, which was 82.2% higher than that of the specimens without short fibers in the layers. Kevlar fibers with a length of 11 mm and a density of 5 g·m$^{-2}$ were layered with a $G_{IIC}$ of 2034.3 J·m$^{-2}$, which was increased by 240.5% compared with the specimens without short fibers in the interlayer.

1. Introduction
With the development in the field of aerospace, the material requirements are increasingly strict, and the traditional metal materials can not fully meet the performance requirements. Therefore, lightweight and high-strength composite materials are increasingly used in the aerospace industry, of which carbon fiber reinforced plastics (CFRP) laminates account for a large proportion[1]. At present, most of CFRP laminates are laid by prepreg, then cured and molded. It is easy to process and design. But the composite laminates have no reinforcing fibers in the thickness direction and are only bonded by the resin, and the interlaminar properties are much lower than the in-plane mechanical, which limits the application range of the laminates.
In view of the shortcomings of poor interlaminar performance, the interlaminar reinforcement technology is adopted to form the microstructure that hinders the interface fracture by introducing the reinforcing material and improve the fracture toughness. Interlaminar reinforcement technology can be divided into two categories: one is the use of fibers throughout the structure, such as "Z-pin"[2], stitching[3]. While the overall fixed technology to introduce defects, reduce damage tolerance, has complex process and narrow scope of application[4-6]. Another type of reinforcement technique involves the addition of a ductile material such as short fiber, particle[7], nanomaterial[8][9], or thermoplastic resin film between layers to form a second phase with a toughened structure between the layers. The technology is low cost, wide applicability. But, a good enhancement effect requires adding a larger amount of particles, films or nanomaterials, which leads to an increase in the quality of the composite laminates and the loss of in-plane mechanical properties. Compared with the addition of particles, nanomaterials and thermoplastic resin film between the layers or physical macroscopic Z-pin and stitching method, short fiber reinforced technology has many advantages such as good enhancement effect, less impact on the structure weight, simple process and low cost[10].
At present, a large number of studies have been made on the technology of interlaminar short fiber reinforcement and significant achievements have been achieved. In 1994, MS Sohn and XZ Hu
[11][12] proposed for the first time that Kevlar chopped fibers were added between carbon fiber/epoxy composite layers to reinforce the composite. During the lamination process, a small amount of kevlar fibers, short cut 5-7 mm in length, were uniformly and non-directional dispersed between the prepreg layers and were press-cured. So that chopped fibers in the resin-rich layer between the layers formed a very thin reinforced interlayer, interwoven with prepreg sheets. The experimental results showed that the $G_{IC}$ and $G_{IIC}$ of the test specimen both increased more than one time. Subsequently, Sohn et al [13] added Kevlar fibers between the laminates layers for impact testing. The results showed that under impact load, Kevlar fiber had poor reinforcing effect and fiber breakage was not obvious. Yasaee et al [14] compared the reinforcing effect of aramid short fiber, carbon short fiber, thermoplastic resin film and other materials. The results showed that aramid short fiber achieved excellent enhancement effect on both $G_{IC}$ and $G_{IIC}$ and resulted in the least structural weight gain. Choi et al [15] added aramid fiber to the surface of carbon fiber resin-based composites to improve the bonding properties. ST Cholake et al [16] modified structural epoxy resin by adding short milled carbon fiber (SMCF) particles. Subsequently, the SMCF modified epoxy resins were used to fabricate unidirectional carbon fibre reinforced laminates and tested under Mode I crack opening. The SMCF reinforcement showed 50% and 64% improvement in the laminates $G_{IC}$ after adding 5% and 10% SMCF by wt. Z Mo et al [17] made the interlayer-toughed carbon fiber/epoxy resin (CF/EP) composite laminates with short ramie fiber (SRF) through hot press laminated molding process. The test results indicated that the insert of SRF significantly improved the CF/EP composites $G_{IC}$ and $G_{IIC}$. The maximum increase of $G_{IC}$ was 34.24%, and the maximum increase of $G_{IIC}$ was 69.54%.

Currently, there is a certain research foundation for interlaminar short fiber reinforced laminates. The research directions mainly focus on the test of short fiber reinforced specimen and the establishment of mechanical model. But there are few researches on the selection of short fiber reinforcement parameters. Therefore, in this paper, in view of the poor interlaminar properties of the CFRP laminates, short fiber reinforcement was carried out. The types of short fibers, short fiber length and short fiber density were studied. Taking $G_{IC}$ and $G_{IIC}$ as targets, the parameters were optimized and the influence of short fiber parameters on the composite laminates was analyzed, which lays the foundation for the extensive application of CFRP laminates.

2. Test preparation

2.1. Specimen preparation

The raw material is T800 carbon fiber reinforced epoxy composite unidirectional prepreg tape. The prepreg tapes are cut by hand, laminated and autoclaved (figure 1). Then the laminates are cut by special diamond grinding wheel to meet the standard requirements.

![Figure 1. Solidified laminates (It has not been cut into a standard test specimen).](image1)

As shown in figure 2, the double cantilever beam (DCB) test according to HB7402-1996 standard, the specimen length of 180mm, width 25mm, 24-ply of unidirectional (0° oriented) plies. A Teflon film of thickness 20μm and length 50mm was used as a crack starter, placed on the mid-plane of the laminates. The end notched flexure (ENF) test according to the standard of HB7403-1996, the test specimen is 140mm long and 25mm wide with 24-ply of unidirectional (0° oriented) plies. A 20μm thick, 40mm
long Teflon film was placed on the mid-plane of the laminates. Hand-cut short fibers were added evenly between the two types of specimens, the use of electronic scales to control the spread of staple fiber surface density.

2.2. Orthogonal design
As shown in table 1, taking three factors of fiber length, fiber density and fiber type of staple fiber, taking four levels for each factor, an orthogonal experiment table of three factors and four levels was established.

| Number | Fiber length (mm) | Fiber density (g · m⁻²) | Fiber type   |
|--------|------------------|------------------------|--------------|
| 1      | 3                | 5                      | Kevlar       |
| 2      | 3                | 10                     | carbon fiber |
| 3      | 3                | 15                     | glass fiber  |
| 4      | 3                | 20                     | basalt fiber |
| 5      | 7                | 5                      | carbon fiber |
| 6      | 7                | 10                     | Kevlar       |
| 7      | 7                | 15                     | basalt fiber |
| 8      | 7                | 20                     | glass fiber  |
| 9      | 11               | 5                      | glass fiber  |
| 10     | 11               | 10                     | basalt fiber |
| 11     | 11               | 15                     | Kevlar       |
| 12     | 11               | 20                     | carbon fiber |
| 13     | 15               | 5                      | basalt fiber |
| 14     | 15               | 10                     | glass fiber  |
| 15     | 15               | 15                     | carbon fiber |
| 16     | 15               | 20                     | Kevlar       |

3. Test and data processing
The DCB tests and the ENF tests all adopt the electronic universal testing machine CSS-88010 manufactured by Changchun Testing Machine Research Institute. The maximum tensile load is 10KN, and it is loaded by controlling the displacement.

3.1. DCB Test
The DCB test requires gluing hinges on both sides of the composite laminates, with the center of the hinge 25 mm from the top of the prefabricated crack, as shown in figure 3.

With the intersection of the two hinge centers and the composite plate as the starting point for effective cracking, the initial crack is 25 mm. To facilitate the observation of cracks, apply a white correction fluid on the side of the specimen and mark lines on the side. Prefabricated crack tip as line 0, line 1 from the 0th line for the 20mm, then draw a line per 10mm, a total of 7 lines. The test loading rate was set at 1 mm/min and the unloading rate was set at 20 mm/min. The experiment used a high-definition digital microscope to observe the crack propagation, using a magnification of 25 times.

![Figure 3. DCB specimen.](image)

![Figure 4. DCB test.](image)
When the effective crack length is 45mm, that is, when the crack propagates to the line 1, unload immediately and record the load and displacement at this moment. When unload to load and displacement is 0, pause. And continue to re-load until the crack growth to the second line (an effective crack length of 55mm), immediately unload. Then when the load and displacement of 0, reload. This operation has been repeated until the effective crack reaches 95mm distance, the line 6, while stopping the test. The test process is shown in figure 4.

3.2. ENF Test

To facilitate the observation of crack propagation, similar to the DCB test, a white correction fluid was applied to the side of the test piece and a marking line was made at the tip of the prefabricated crack, 45 mm away from the edge of the test piece, as shown in figure 5.

![Figure 5. ENF specimen.](image)

First put the specimen into the support. The center of the supporting cylinder on the side of the initial crack is 15 mm from the edge of the specimen. Adjust the span between the supports to 70mm and ensure that the loading head is in the middle of the support. Span between the support refers to the distance between two cylindrical centers of the support.

The effective crack distance is calculated from the center of the bearing cylinder, at which point the effective crack length is 25 mm. Load was started and crack propagation was observed by an electron microscope. When the crack spreads to the mark line, it is unloaded and stops when the load drops to zero. Adjust the span between the support to 100mm, to ensure that the loading head is in the middle of the support, and adjust the position of the test piece so that the center of the support cylinder is 20mm from the edge of the test piece. Due to the crack extension of 5mm, but also adjusted 5mm, then the effective crack length continues to 25mm. Load head reload, when the load drops, stop the test and unloaded, record the maximum load and displacement. The test process is shown in figure 6.

![Figure 6. ENF test.](image)

3.3. Experimental data processing

$G_{IC}$ calculated according to equation (1).

$$G_{IC} = \frac{mP\delta}{2Wa} 	imes 10^3$$  \hspace{1cm} (1)

Where $G_{IC}$ is mode I fracture toughness, and the unit is J·m$^{-2}$. $P$ is the load when reaching the mark line, the unit is N. $\delta$ is the displacement when reaching the marking line in mm. $W$ is the width of the specimen in mm; $a$ is the effective crack length in mm. And $m$ is the fitting coefficient of the compliance curve, calculated using equation (2).

$$m = \frac{\sum_{i=1}^{5}(\frac{\sum_{i=1}^{5}\lg(a_i)\cdot\lg(C_i)}{\sum_{i=1}^{5}\lg(a_i)}) - \frac{1}{5}(\sum_{i=1}^{5}\lg(a_i))((\sum_{i=1}^{5}\lg(C_i))}{\sum_{i=1}^{5}(\lg(a_i))^2 - \frac{1}{5}(\sum_{i=1}^{5}\lg(a_i))^2}$$  \hspace{1cm} (2)
i represents the i-th line, \( k \) is the number of valid data points. \( a_i \) represents the effective crack length before the i-th loading, in millimeters. \( C_i \) is the compliance, and the calculation formula is \( \delta / P \), the unit is mm/N. \( G_{IC} \) calculated according to equation (3).

\[
G_{IC} = \frac{9P\delta a^2}{2W(2L + 3a^2)} \times 10^3 \tag{3}
\]

Where \( G_{IC} \) is mode II fracture toughness in J·m\(^{-2}\). \( P \) is the maximum load in N. \( \delta \) is the displacement at maximum load in mm. \( a \) is the effective crack length in mm. \( W \) is the width of the test piece, the unit is mm. \( L \) is half the span of the support, the unit is mm.

4. Results Analysis and Discussion

4.1. Test results

The \( G_{IC} \) of the laminates without short fibers is 247.7 J·m\(^{-2}\), and the \( G_{IC} \) is 597.5 J·m\(^{-2}\). The results of the laminates with interlaminar short fiber reinforcement are shown in table 2.

According to table 2, get \( G_{IC} \) range analysis table and \( G_{IC} \) range analysis table, as shown in table 3 and table 4. The M value is the average of the corresponding experimental values at different levels. For the fiber length, the corresponding levels of M1 to M4 are 3mm, 7mm, 11mm and 15mm respectively, and for the fiber density, the corresponding levels of M1 to M4 are 5 g·m\(^{-2}\), 10 g·m\(^{-2}\), 15 g·m\(^{-2}\), 20 g·m\(^{-2}\) respectively. The different levels of fiber types corresponding to M1 to M4 are Kevlar, carbon fiber, glass fiber and basalt fiber.

It can be seen from the R value in table 3 that the fiber type has the greatest influence on the \( G_{IC} \), followed by the fiber density, and the fiber length has the least impact.

It can be seen from the R values in table 4 that the fiber type has the greatest influence on the \( G_{IC} \), followed by the fiber density, and the fiber length has the least effect.

| Number | \( G_{IC} \) (J·m\(^{-2}\)) | Increase percentage (%) | \( G_{IC} \) (J·m\(^{-2}\)) | Increase percentage (%) |
|--------|-----------------|------------------------|-----------------|------------------------|
| 1      | 260.9           | 5.3                    | 1389.5          | 132.6                  |
| 2      | 242.5           | -2.1                   | 1339.3          | 124.2                  |
| 3      | 222.5           | -10.2                  | 1131.7          | 89.4                   |
| 4      | 204.7           | -17.4                  | 1426            | 138.7                  |
| 5      | 203.8           | -17.7                  | 1595            | 166.9                  |
| 6      | 321.6           | 29.8                   | 1349.9          | 125.9                  |
| 7      | 233.9           | -5.6                   | 1278.2          | 113.9                  |
| 8      | 233             | -5.9                   | 1283.8          | 114.9                  |
| 9      | 206.7           | -16.6                  | 1360.5          | 127.7                  |
| 10     | 210.5           | -15.0                  | 1343.3          | 124.8                  |
| 11     | 336.7           | 35.9                   | 1615.9          | 170.4                  |
| 12     | 272             | 9.8                    | 1391.6          | 132.9                  |
| 13     | 191.2           | -22.8                  | 1389.9          | 132.6                  |
| 14     | 216.6           | -12.6                  | 1430.1          | 139.3                  |
| 15     | 224             | -9.6                   | 1245.3          | 108.4                  |
| 16     | 345.1           | 39.3                   | 1555.5          | 160.3                  |
Table 3. $G_{IC}$ range analysis.

| Fiber length | Fiber density | Fiber type |
|--------------|---------------|------------|
| M1           | 232.7         | 215.7      | 316.1      |
| M2           | 248.1         | 247.8      | 235.6      |
| M3           | 256.5         | 254.3      | 219.7      |
| M4           | 244.2         | 263.7      | 210.1      |
| R            | 23.8          | 48.0       | 106.1      |

Table 4. $G_{IIC}$ range analysis.

| Fiber length | Fiber density | Fiber type |
|--------------|---------------|------------|
| M1           | 1321.6        | 1433.7     | 1477.7     |
| M2           | 1376.7        | 1365.7     | 1392.8     |
| M3           | 1427.8        | 1317.8     | 1301.5     |
| M4           | 1405.2        | 1414.2     | 1359.4     |
| R            | 106.2         | 115.9      | 176.2      |

The results of range analysis show that the order of influence of various factors on $G_{IC}$ and $G_{IIC}$ is: fiber type, fiber density and fiber length.

According to the range analysis, the optimum level of $G_{IC}$ in the selected parameters range: length 11mm, density 20 g·m$^{-2}$, fiber type Kevlar fiber. The optimal combination of $G_{IIC}$: Length 11mm, density 5 g·m$^{-2}$, fiber type Kevlar fiber.

4.2. The effect of short fiber length

Through the results in table 3 and table 4, the influence of fiber length on $G_{IC}$ and $G_{IIC}$ is obtained as shown in figure 7.

![Figure 7. Effect of fiber length on $G_{IC}$ and $G_{IIC}$.](image)

$G_{IC}$ first increases and then decreases with the increase of fiber length. When the fiber length is 11mm, the $G_{IC}$ is most improved. When the fiber length is 3mm, the $G_{IC}$ increase is the least. When the fiber length is 7mm and 15mm, the difference of $G_{IC}$ value is not obvious. When the short fiber length is small, the short fibers can’t enter into the resin matrix to form a bridging effect, and the force of fiber pulling out is relatively small, and its $G_{IC}$ is relatively low. When the fiber length is too short, the fibers form an impurity for the composite laminate, reducing the $G_{IC}$. When the fiber length is longer, it can’t completely enter the resin matrix, and can only be simply lapped in the in-plane direction. As the crack propagates, longer short fibers do not provide good bridging and assist in crack propagation. The $G_{IC}$ of 15mm short fiber is not high.

With the increase of fiber length, $G_{IIC}$ firstly increases and then decreases. $G_{IIC}$ reaches its maximum at a fiber length of 11mm. For $G_{IIC}$, shear stress is applied to the composite laminates. When the fiber length is too short, the contact area between the resin and the short fiber is too small, and the $G_{IIC}$ is not significantly improved.

When the length of the short fiber is too long, the resin can not all wrap the short fiber, the short fiber can not fully infiltrate, and the enhancement effect of the interlaminar performance is not significant.
4.3. The effect of short fiber density

Through the results in table 3 and table 4, the influence of fiber density on $G_{IC}$ and $G_{IIC}$ is obtained as shown in figure 8.

As the fiber density increases, the $G_{IC}$ increases, but the increase rate decreases. For the DCB test, the load direction is perpendicular to the crack plane and the improvement of $G_{IC}$ only depends on the bridge force. Short fibers between layers are disordered. When the short fibers and crack surface are not parallel, short fibers provide bridge force. As the short fiber density increases, the number of short fibers that are not parallel to the crack surface also increases, providing greater bridging forces, impeding the cracking, and thus increasing the $G_{IC}$.

With the increase of fiber density, $G_{IIC}$ firstly decreases and then increases, and $G_{IIC}$ is larger when fiber density is smaller and larger. At the fiber density of 5 g·m$^{-2}$, $G_{IIC}$ reaches the maximum. With the increase of the density of short fibers, the amount of interlaminar resin is certain, and the amount of short fibers is greater than the amount of resin able to be infiltrated, so interlaminar resin can't combine well with short fibers, resulting in a decrease of $G_{IIC}$. When the fiber density reaches 20 gm$^{-2}$, the short fibers form simple laps, because of the large number of interlaminar short fibers. Resin plays the role of glue between short fibers. This increases the friction at crack propagation and increases $G_{IIC}$.

4.4. The effect of short fiber type

Through the results in table 3 and table 4, the influence of fiber type on $G_{IC}$ and $G_{IIC}$ is obtained as shown in figure 9.

![Figure 9](image1)

**Figure 9.** Effect of fiber type on $G_{IC}$ and $G_{IIC}$.

![Figure 10](image2)

**Figure 10.** Scanning electron microscope image of Kevlar short fibers (high magnification).

It can be seen from the figure that Kevlar fiber has better enhancement effect on $G_{IC}$ and $G_{IIC}$. Carbon fiber secondly, the reinforcing effect of glass fiber and basalt fiber is poor.

In order to further study the influence of fiber types, the fracture surface of DCB test specimens were observed by scanning electron microscopy (SEM). Kevlar images at different magnifications are shown in figures 10 and 11; carbon fibers are shown in figure 12; glass fibers are shown in figure 13 and basalt fibers are shown in figure 14.

Figure 10 shows the high-magnification interlaminar short Kevlar image. In the figure, the carbon fibers are neatly arranged, while one end of the Kevlar fiber is embedded in the inside of the substrate, and the end of the other end is severely bifurcated, which is a marked trace of fiber tensile fracture. This shows that the Kevlar fiber plays a good bridging role in the propagation of mode I cracks, and eventually tensile failure, thus increasing the interlaminar fracture toughness.
Figure 11 is a scanning electron microscope image of Kevlar short fiber at low magnification. It can be seen that there are many traces of fiber pull-out and tensile fracture. And in the layer form a random messy distribution of short fiber toughened structure. Kevlar fiber embeds in the resin matrix, resulting in anchoring. Not only the fracture energy of the resin, but also entanglement between the Kevlar fibers and the energy extracted by the Kevlar fibers from the resin increase the interlaminar fracture toughness.

In figure 12, figure 13 and figure 14, the fiber topography is almost the same. And a large amount of entanglement between the fibers does not occur. Kevlar fiber has better toughness and can produce greater bridging displacement. However, the interlaminar carbon fiber, glass fiber and basalt fiber are fractured under a small bridging displacement, and a large number of effective bridging fibers can not be observed during experiments. Therefore, Kevlar fibers can produce more energy consumption than the other three fibers and significantly improve the interlaminar fracture toughness.

The effects of adding three short fibers on interlaminar performance are different and related to their resin compatibility. Carbon fiber has good compatibility with epoxy resin. Glass fiber is an inorganic surface, which has poor affinity with resin. Basalt fiber contains a lot of calcium oxide components, but too much calcium oxide causes poor fiber toughness, smooth texture and small surface roughness, so it is poor in combination with resin. For the addition of short fiber layer technology, it is recommended to choose Kevlar, and try not to choose carbon fiber, glass fiber and basalt fiber.
4.5. Parameter optimization and verification

When the length of Kevlar fiber was 11 mm and the density was 20 g·m⁻², the $G_{IC}$ increased most. The optimum level combination was tested. The average value of $G_{IC}$ was 451.4 J·m⁻². The $G_{IC}$ increased by 82.2% over the composite laminate without the addition of short fibers.

When the length of Kevlar fiber was 11 mm and the density was 5 g·m⁻², the $G_{IIC}$ was the most improved. The average $G_{IIC}$ value was 2034.3 J·m⁻². Compared with the composite without the addition of short fibers, $G_{IIC}$ increased by 240.5%.

5. Conclusions

The results show that the order of influence of various factors on the $G_{IC}$ and the $G_{IIC}$ is: fiber type, fiber density and fiber length.

The addition of Kevlar fibers enhances the $G_{IC}$ most dramatically. And $G_{IC}$ increases with increasing fiber density. With the increase of the length of short fibers, $G_{IC}$ first increases and then decreases. For $G_{IIC}$, adding Kevlar fibers increases the most. With the increase of fiber density, $G_{IIC}$ first decreases and then increases. As the fiber length increases, $G_{IIC}$ first increases and then decreases.

Within the parameters of the study when Kevlar fibers with a length of 11 mm and an areal density of 20 gm⁻² were added into the interlaminar layers of composite laminates, the $G_{IC}$ was increased by 82.2% compared with the non-reinforced composite laminates. When Kevlar fibers with a length of 11 mm and an areal density of 5 g·m⁻² were added into the interlamimates, the $G_{IIC}$ increased by 240.5% compared with the non-reinforced composite laminates, which significantly improved the interlaminar properties of the composites laminates.

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