 한국동력기계공학회지 제7권 제1호 pp.25~33 2003년 2월

Study on the Evaluation of the Interfacial Strength in the Fiber Reinforced Composites
섬유강화 복합재료에서 계면강도의 평가에 관한 연구

D. B. Lee and C. K. Moon
이덕보 · 문창권

Key Words : Interfacial Properties(계면특성), E glass Fiber/Epoxy Composites(E 글래스/에폭시 복합재료), Interfacial Shear Strength(계면전단강도), Stress Distribution(응력분포)

요약 : 섬유강화 복합재료의 계면 강도는 강화재와 메트릭스간의 계면특성, 강화용 섬유의 표면처리 및 섬유간의 거리 등에 많은 영향을 받는다. 본 연구에서는 섬유간의 거리가 섬유강화 복합재료의 계면특성에 미치는 영향을 고찰하기 위해, E glass fiber/epoxy 복합재료의 시험편을 제작하고, 섬유의 표면처리 및 섬유파괴가 이웃하는 섬유파괴에 영향을 미치는 거리에 대해 고찰하였다. E glass fiber/epoxy 복합재료의 계면 전단강도는 섬유간 거리 0~50 ㎛ 사이에서는 섬유의 표면처리와는 관계없이 섬유간 거리가 증가할수록 증가하였고, 섬유간 거리 50 ㎛ 이상에서는 섬유간거리에 관계없이 계면전단강도는 일정하였다.

1. Introduction

The interface between the reinforcing fibers and the polymer matrix plays a very important role in determining the final performance of a composite. Especially, the interfacial shear strength is one of the most fundamental factors in evaluating the mechanical properties and durability on the specific environment of the fiber reinforced composites. If the interfacial shear strength in fibrous composites is too low, its mechanical properties are controlled mainly by the interface of low shear strength; hence, it is hard to expect that the performance of reinforcing fiber is reflected in composites, even using the high performance fiber.

On the other hand, if the interfacial shear strength is too high in fibrous composites, there is a fear of a decrease in fracture toughness of composites because of the resistance against the crack propagation. Therefore, it is necessary that the interfacial shear strength of the fiber reinforced composites is controlled in accordance with the performance of material demanded for the final purpose.

In principle, the interfacial shear strength will be able to be controlled by the suitable combination of fiber, matrix resin and fiber surface modification, etc.

Then, it is also very important to evaluate exactly the interfacial shear strength thus controlled. For the last a few decades, several techniques such as the pull out[1-3], microbond[4-8], fragmentation[9-12] and indentation[13] test methods have been developed to try to measure the interfacial shear strength correctly. Among the number of techniques mentioned previous, one popular technique is single fiber fragmentation test. The merits of this method are that a lot of data are generated from one sample test, and easy to prepare sample comparing the other techniques. But currently, it takes about 5hrs to test a single sample. Because of this problem, a new approach has been developed to overcome, many works[9,21] have been done exploring the effects of testing multiple fibers in a fragmentation test.

The objective of this paper is to study

접수일: 2002년 9월 29일
문창권(책임저자) : 부경대학교 재료공학부
E-mail : moonck@pknu.ac.kr Tel. 051-620-1654
이덕보 : 부경대학교 재료공학부

whether the results gained at previous work might be applied in two dimension fragmen tation test.

In this paper, multiple fibers fragmentation test specimens with two dimension were fabricated. We have investigated the effect of interfiber distance on the interfacial properties in two dimension arranged multi E glass fiber/ epoxy resin composites. In additions, the effect of sized and desized fiber in model composites on the interfacial properties has been studied.

2. Experimental

2.1 Materials

The materials used in this study are as follows. The fibers used were sized and desized E glass fiber (Owens Corning). Sized fiber was coated with 3 aminopropyltriethoxysilane(A 1100) and desized fiber meant the sized fiber washed by water.

The matrix resin used was epoxy (diglycidl ether of bisphenol A, DGEBA, Epon 828, shell co.). The harder used was meta phenylene diamine (m PDA, Fluka chemical co.)

2.2 Single fiber tensile test

The tensile strength was measured on single fiber with gage length of 20 mm at a crosshead speed of 2.0 mm/min. using a tensile tester equipped with a load cell of 100g. Before tensile testing, the diameter of every sample was measured with an optical micrometer (VIA 100, Boeckeler).

2.3 Preparation of fragmentation test sample

The sample preparation for single and multi fiber testing was similar to that described by Drzal at al.[10], and a brief description is as follows.

The silicone (GE silicone RTV 664) mold with eight dogbone shaped cavities (see Fig.1) was used for the preparation of fragmentation test samples.

Each cavity in the mold has sprue slots with width of 400 m in the center of each end to aid in aligning the fiber in the center of the cavity. We placed single and multi fibers of E glass through the sprue slots of a silicone mold. Single fiber was placed by hand and multi fibers with two dimension were placed using specific designed device as can be seen in Fig. 1. And then fixed them in place by putting a small drop of five minutes epoxy resin (Hardman Adhesives) at the far end of each sprue slot.

The multi fiber specimens were prepared with an epoxy resin cured using meta phenylene diamine. One hundred grams of DGEBA and 14.5 g of m PDA were weighed out in separate beakers. To lower the viscosity of the resin and melt the m PDA crystals, both beakers were placed in a vacuum oven (Fisher Scientific Isotemp Vacuum Oven, model 281 A) set at 6 5℃. After the m PDA crystals were completely melted, the silicone rubber mold containing the fibers was placed into another vacuum oven (Fisher Scientific Isotemp Vacuum Oven, model

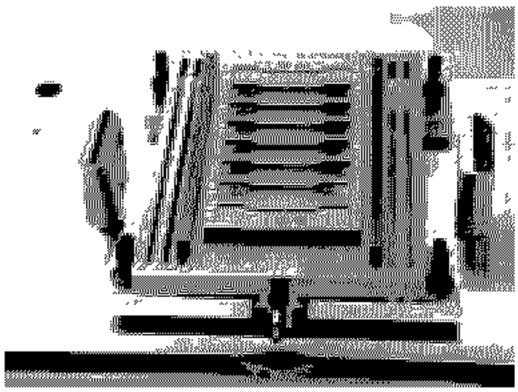


Fig. 1 Apparatus of fiber arranging for multi fiber fragmentation test specimen

281 B) that was preheated to 75 ℃ at 20 kPa, for 20 min. This last procedure dries the mold and minimizes the formation of air bubbles during the curing process. At approximately 9 min before the preheated molds were removed from the oven, the m PDA is poured into the DGEBA and mixed thoroughly. The mixture was placed into the vacuum oven and degassed for approximately 7 min. After 20 min, the preheated molds were removed from the oven and filled with the DGEBA/m PDA resin

mixture using 10 ml disposable syringes. The filled molds were then placed into a programmable oven (Blue M, General Signal, model MP 256 1, GOP). A cure cycle of 2 h at 75 ℃ followed by 2 h of post curing at 125 ℃ was used.

2.4 Fragment test

Fragmentation test was carried out using fragmentation test machine as shown in Fig. 2. Before testing, samples were polished to avoid stress concentration at the edge part of sample by emery paper of # 800 and # 2400. And then samples were marked the standard gauge length of about 10 mm to measure real strain by permanent pen with blue color. When specimen was installed, we adjusted that grip was not much tight. The specimen was loaded in tension by the sequential application of step strain. The number of total strain step was 28 and total strain was 2.4 mm. The strain rate was 85 ㎛ /sec and the average deformation at each step was 85.7 ㎛ through the whole sample length between the sample holders at both sides. The delay time between the applications of successive step strain was 10 min. The image was scanned at before loading and every strain step using movable camera and then it was saved automatically in a computer.

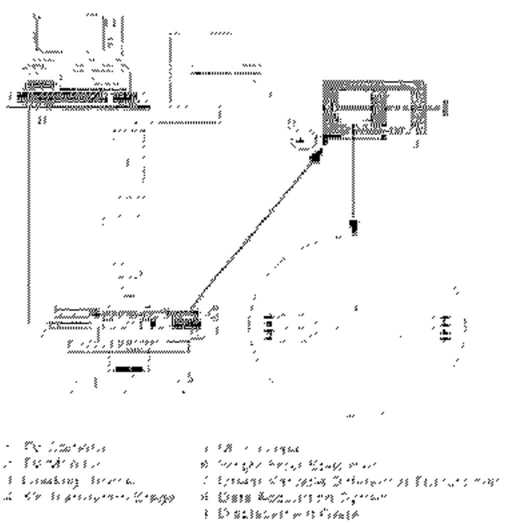


Fig. 2 Schematic of single fiber fragmentation test Machine

The image scanning length was 23 mm. After 28 steps, sample was uninstalled. Real strains at each step were calculated using scanned images of each step as the standard gauge length of about 10 mm marked on the sample before testing and the real strain of every sample was about 6.0%.

To measure fragment length, the sample was installed by translating frame under the microscope with a micrometer. The image was scanned using a CCD camera (Optronics LX 450 RGB Remote Head microscope camera) and monitor (Sony, PVM 1344Q).

The position is monitored by a linear variable differential transformer (LVDT) (Trans Tek, Inc. model 1002 0012) connected to an A to D board (Strawberry Tree, Inc.) in a computer. The location was aligned with a cross hair in the microscope as seen on the video monitor, and the position of the LVDT was digitized into the computer.

The standard uncertainty in relocating a point reproducibly is +1.1 m. The bridge is attached to the same computer via a serial connection. A custom program was developed to continuously record changes in the displacement.

2.5 Interfacial shear strength calculation

We calculated the interfacial shear strength using following equation (1)[10].

The distribution of fragment lengths have been determined to be satisfactorily described by a two parameter Weibull analysis, causing the expression for the interfacial shear strength τ,

$$\tau = \frac{\sigma}{2\beta}\,\Gamma\left(1 - \frac{1}{\alpha}\right) \qquad (1)$$

to be come where α and $\beta$are the shape and scale parameters, respectively. $\Gamma$was the Gamma function.

In eq.(1), $\sigma$is the average fiber tensile strength at the critical fiber length needed to calculate interfacial shear strength. However, in this equation, we used to the single fiber tensile strength at 20 mm of gauge length. One goal of this study is to discuss about the effect of interfiber distance on the interfacial properties in fragmentation test.

3. Results and discussion

3.1 Single fiber tensile test

The table 1 shows the tensile strengths of sized and desized E glass single fiber. The tensile strength of sized fiber is shown stronger than one of desized fiber. This is probably that desized fiber is easier to get damage when they rub against each other during processing operations. Generally it is known that the most important factor determining the tensile strength of the glass fiber is the damage of fiber surface.

Fig. 3 shows the relationship between failure probability and single fiber tensile strength. Solid lines are plotted by the Weibull distribution equation with two parameters. We can see that the experimental values are according to the Weibull distribution equation with two parameters.

3.2 Single fiber fragmentation test

Fig. 4 represents the plot of a failure probability as a function of aspect ratio of the fiber fragment in the sized single fiber fragm entation test. Solid lines are plotted by the Weibull distribution equation with two parame ters. We can see that experimental values and Weibull distribution equation with two param eters are good accordance. It is shown that the range of aspect ratio is 15 to 37.

Fig. 5 shows the plot of a failure probability as a function of aspect ratio of the fiber

Table 1 Tensile strength of single fiber

Fiber	Sized fiber	Desized fiber
Tensile strength (GPa)	2.10+0.57	1.63+0.44

Table 2 Interfacial shear strength of E glass fiber/epoxy resin

Fiber	Sized fiber	Desized fiber
Interfacial shear strength (MPa)	46.70+2.59	40.69+2.60

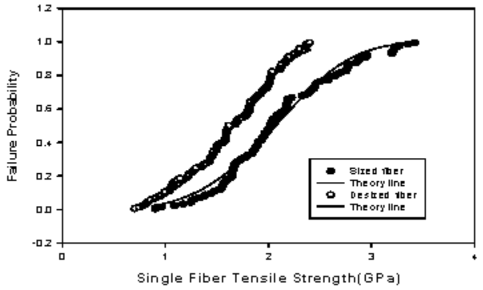


Fig. 3 Plot of a failure probability vs. single fiber tensile strength

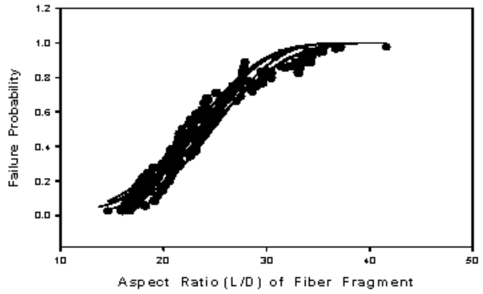


Fig. 4 Failure probability vs. aspect ratio of fiber fragment in the sized single fiber fragmentation test

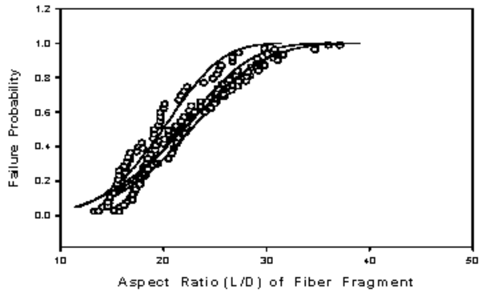


Fig. 5 Failure probability vs. aspect ratio of the fiber fragment in the desized single fiber fragmentation test

fragment in the desized single fiber fragmen tation test. Solid lines are plotted by the Weibull distribution equation with two param eters. We can see that the experimental values and the Weibull distribution equation with two parameters are good accordance. It is shown that the range of aspect ratio is 12 to 33. These aspect ratio are not much important to compare for different kind of fiber in fragmentation test owing to it depend on such as the fiber tensile strength, diameter and interfacial shear strength.

Fig. 6 represents the normalized number of fiber breaks as a function of applied strain in sized and desized single E glass fiber /epoxy resin fragmentation test. We can see that after the number of desized fiber breaks was saturated, the number of sized fiber breaks was saturated. Also we can see that the extent of sized fiber strain from initial fiber break to saturation was smaller than that of desized fiber. And the strains of initial fiber break were about 2 and 3 % of desized and sized fiber, respectively.

Fig. 7 shows the polarized transmitted light micrographs of E glass fiber/epoxy resin fragmentation test. In the figure 5, (a) is shown the sized fiber and (b) is shown desized fiber In this figure, we can see that the stress distribution pattern of sized was very similar with that of desized fiber. This reason is that the interfacial shear strengths of sized and desized fiber were not marked difference an can be se

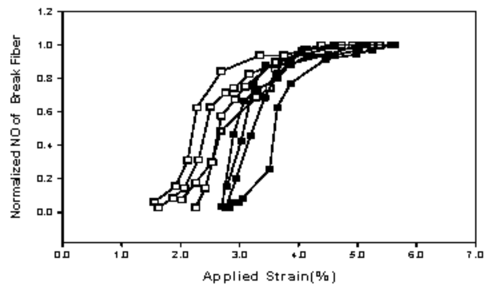
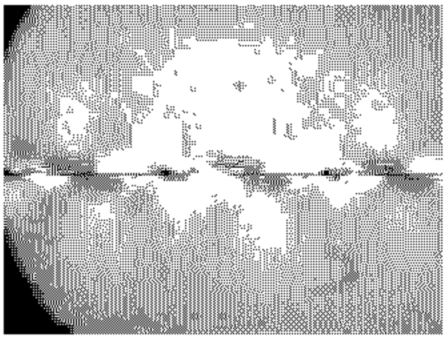


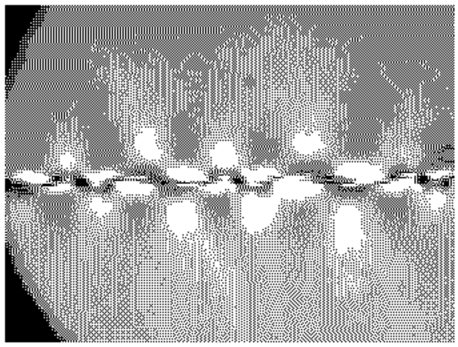
Fig. 6 Plot of normalized number of fiber breaks as a function of applied strain in fragmentation test of single E glass fiber/epoxy resin(solid ; sized, open ; desized)

3.3 Two fibers fragmentation test

Fig. 8 shows the plot of a failure probability as a function of aspect ratio of the fiber fragment in the sized two fibers fragmentation test. Solid lines are plotted by the Weibull distribution equation with two parameters.



(a) Sized fiber



(b) Desized fiber

Fig. 7 Polarized transmitted light micrographs of E glass fiber/epoxy resin fragmentation test at saturation

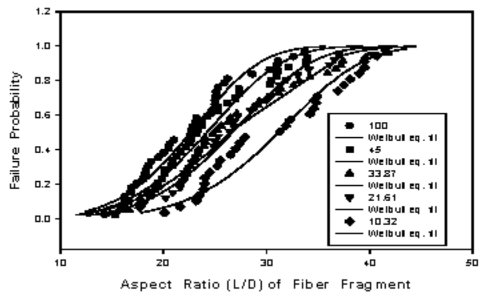


Fig. 8 Failure probability vs. aspect ratio of the fiber fragment in the two fibers fragmentation test

We can see that experimental values and two parameters Wibull distribution equation are good accordance. It is shown that the range of aspect ratio became high according to decreasing of interfiber distance and when the interfiber distance is over 45 ㎛, the range of aspect ratio is very similar with the result of sized single fiber fragmentation.

Fig. 9 represents the plot of a failure probab ility as a function of aspect ratio of the fiber fragment in the desized two fibers fragmentation test. Solid lines are plotted by two parameters Weibull distribution equation. We can see that experimental values and two parameters distribution equation are good accordance. It is also shown that the range of aspect ratio became high according to decreasing of the interfiber distance and when the interfiber distance is over 54.9 ㎛, the range of aspect ratio is similar with the result of desized single fiber fragmentation.

Fig. 10 reveals the normalized number of fiber breaks as a function of applied strain in sized two E glass fiber/epoxy resin fragmentation test. We can see that initial fiber crack began to occur at strain of about 3 %, the number of fiber breaks was saturated at strain of 6 %. Also we can see that the effect of interfiber distance did not exist in this graph.

Fig. 11 shows the normalized number of fiber breaks as a function of applied strain in desized two E glass fiber /epoxy resin fragmentation test. We can see that initial fiber crack began to occur at strain of about 2 %, the number of fiber breaks was saturated at strain of 6 %. We also can understand that the effect of interfiber distance did exist in this graph.

In Fig. 10 and 11, we can realize that the plots of normalized number of fiber breaks as a function of applied strain in two E glass fiber/epoxy resin fragmentation were similar

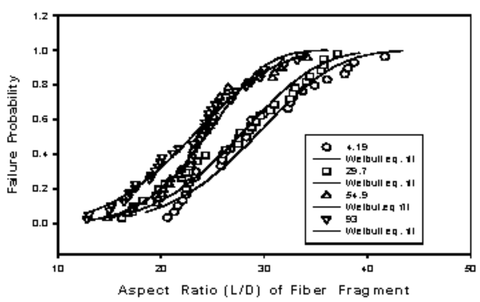


Fig. 9 Plot of failure probability vs. aspect ratio of fiber fragment in the desized two fibers fragmentation test

with the results of single E glass fiber/epoxy fragmentation test.

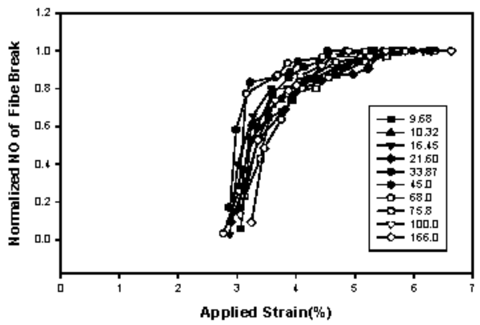


Fig. 10 Plot of normalized number of fiber breaks as a function of applied strain in fragmentation test of sized two

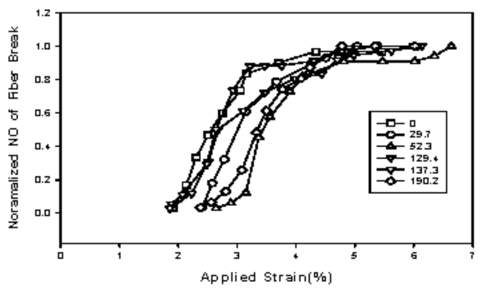


Fig. 11 Plot of normalized number of fiber breaks as a function of applied strain in fragmentation test of desized two E glass fiber /epoxy resin

Fig. 12 represents the plot of interfacial shear strength according to interfiber distance in the two E glass fiber/epoxy resin fragmentation test. It was shown that the interfacial shear strength increased until interfiber distance became about 50 ㎛, and then the ones were saturated regardless of sized and desized fiber. In the other word, in case of the interfiber distance of under 50 ㎛, the interfacial shear strength decreased with decreasing of the interfiber distance. In case of the interfiber distance of over 50 ㎛, the interfacial shear strengths became a constant value. This is probably that the interface between the fiber and the resin was damaged by the adjacent fiber breaks and the damage increased with being close of interfiber spacing. The damage factors considered owing to fiber break are strain energy release, stress transfer and stress concentration, etc. The extent of stress concentration depends on mainly exiting matrix crack in case of sized fiber sample. Therefore, it was shown that when the interfiber distance is small, the decreasing of interfacial shear strength in sized fiber fragmentation was more serious than the decreasing of the one in the desized fiber fragmentation test.

From Fig. 12, when the interfiber distance was small, the interfacial shear strength decreased with decreasing interfiber distance and the stress distribution pattern is shown like one fiber with big fiber diameter, we could find a good agreement with the result of the interfacial shear strength decreased with the increasing fiber diameter[7].

It is also shown that when the interfiber distance is small, the crack positions of two fibers were almost same. In fact, when the interfiber distance was 9.68 and 10.32 ㎛, the cracks location of two fibers were same. In case of the distance of 16.45 and 21.6 ㎛, different fiber crack position was only one. In case of the distance of 33.87 ㎛, different fiber crack positions were six. But when the interfiber distance was over 45 ㎛, the crack positions were [illegible] ation even [illegible] n.

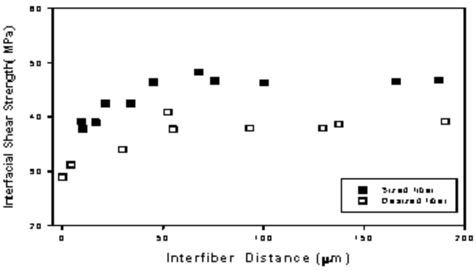
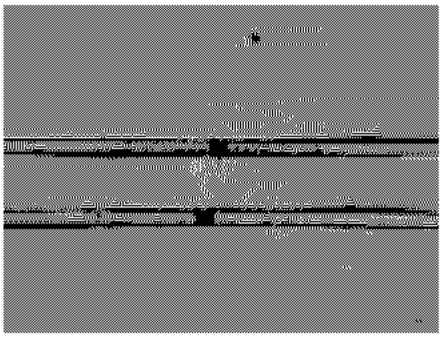
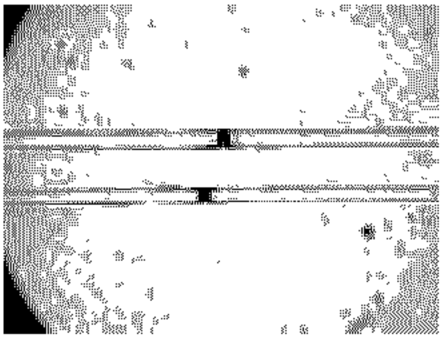


Fig. 12 Plot of interfacial shear strength according to interfiber distance in the two fiber fragmentation test



(a) Sized fiber



(b) Desized fiber

Fig. 13 Micrographs of the E glass fiber/epoxy resin fragmentation test

From these results, we could realized that the when stress distribution pattern was like one fiber at two fiber fragmentation test, it could make fiber break at same location between two fibers. We could observe that some of crack positions were same even though the interface distance became longer. This might be able to be described in Fig. 18. That is, fiber breaks might be able to happen the part of the stress overlap.

Fig. 13 shows the micrographs of the E glass fiber/epoxy resin fragmentation test. There are sized and desized fiber samples. We can see that sized fiber had more damaged interface around fiber breaks than desized fiber. This is probably one of the reason mentioned previous in Fig. 12 that when the interfiber distance is small, the decreasing of interfacial shear strength in sized fiber fragmentation was more serious than the decreasing of the one in the desized fiber fragmentation test.

We considered to two parts, low strain and high strain, about fiber break happen to be made almost same position when interfiber distance is under 50 ㎛. At low strain; A few cracks happened on one fiber when strain reached ultimate strain value. After fiber cracks on the one fiber, it might be considered as damage factors owing to fiber break such as strain energy release, stress transfer and stress concentration. The interface of the other fiber at around fiber break was damaged mainly by damage factors and might become weak. Therefore, fiber break of the other fiber could occur within damaged region. At high strain; A few fiber breaks of two fiber were at same location were occurred at low strain, and then at high strain, the stress pattern must be redistributed like one fiber. Therefore, fiber cracks happen the same position at a time. And there are overlapping of stress and then fiber crack might happen at the region of stress overlapping.

Finally, this phenomenon shows more serious as the interfiber distance is decreasing. Therefore, Interfacial shear strength increased with decreasing interfiber distance when the interfiber distance is under 50 ㎛.

4. Conclusion

The effect of interfiber distance and fiber surface treatment on the interfacial properties in E glass fiber/epoxy resin composites has been studied and the findings made from this study can be summarized as follows.

1. When the interfiber distance is small, the crack positions of two fibers were almost same. But when the interfiber distance was over 50 ㎛, the crack positions were independent of adjacent fiber crack location even though some cracks were same position.
2. The interfacial shear strength increased with increasing the interfiber distance at the range of the interfiber distance between 0 and 50 ㎛ and then the ones were saturated regardless of sized and desized fibers. The interfacial shear strengths saturated were in close agreement with those of the single fiber fragmentation test.
3. When interfiber distance was small, the stress distribution pattern was shown like one fiber and when interfiber distance was large, over 50 ㎛, the stress distribution pattern was independent on between fibers.
4. The interfacial shear strength evaluated using two E glass fibers/epoxy resin fragmentation test is shown as real values in site regardless of fiber surface treatment and interfiber distance.

Acknowledgments

This work was supported by Pukyoung Resesrch Abroad Fund in 2001.

References

1. A. Takaku and R. G. G. Arridge, "The Effect of Interfacial Radial and Shear Stress on Fibre Pull out in Composite Materials", J. Phys. D: Appl. Phys., Vol. 6, pp. 2038~2047, 1973
2. J. Bowling and G. W. Groves, "The Debonding and Pull out of Ductile Wires from a Brittle Matrix", Journal of Materials Science, Vol. 14, pp. 431~442, 1979
3. J. Favre and M. C. Merienne," Character ization of Fibre/Resin Bonding in Composites Using a Pull out test", Int. J. Adhesion and Adhesives, Vol. 1, pp. 311~316, 1981
4. B. Miller, P. Muri, and L. Rebenfeld, "A Microbond Method for Determination of the Shear Strength of a Fiber/Resin Interface", Composites Science and Technology, Vol. 28, pp. 17~32, 1987
5. U. Gaur and B. miller, "Microbond Method for Cetermination of the Shear Strength of a Fiber/Resin Interface: Evaluation of Exper imental Parameters", Composites Science and Technology, Vol. 34, pp. 35~51, 1989
6. C. K. Moon, H. H. Cho, J. O. Lee and T. W. Park, "A Solution Microbond Method for

Determination of the Shear Strength of a Fiber/Thermoplastic Resin Interface", Journal of Applied Polymer Science, Vol 44, pp. 561~563, 1992
7. C. K. Moon, J. O. Lee, H. H. Cho and K. S. Kim, "Effect of Diameter and Surface Treatment of Fiber on Interfacial Shear Strength in Glass Fiber/Epoxy and HDPE", Journal of Applied Polymer Science, Vol. 45, pp. 443~450, 1992
8. C. K. Moon, "The Effect of Interfacial Microstructure on the Interfacial Strength of Glass Fiber/Polypropylene Resin Composites", Journal of Applied Polymer Science, Vol. 54, pp. 73~82, 1994
9. C. K. Moon and W. G. McDonough, "Multiple Fiber Technique for the Single Fiber Fragmentation Test", Journal of Applied Polymer Science, Vol. 67, pp.1701~1709, 1998
10. L. T. Drzal, M. T. Rich, J. D. Campaing and W. J. Park, " Interfacial Shear Strength and Failure Mechanisms in Graphite Fiber Composites", In Proceedings of the 35th Annual Technical Conference, Reinfored Plastics/ Composites Institute, Part 20c 1, pp. 1~7, 1980
11. L. T. Drzal, "Composite Interphase Characterzation", SAMPE Journal, pp. 7~13, 1983
12. M. C. Waterbury and L. T. Drzal, "On the Determination of Fiber Strengths by In Situ Fiber Strength Testing", Journal of Composites Technology & Research, Vol. 13, pp. 22~28, 1991
13. J. F. Mandell, J. H. Chen and F. J. McGarry, "A microdebonding test for in situ assessment of fibre/matrix bond strength in composite materials", Int. J. Adhesion and Adhesives, Vol. 5, pp. 40~44, 1980