Arch. Mech., **52**, 3, 429–442, Warszawa 2000

Effect of fiber treatment on bending strength of aramid short fiber reinforced polyester

A. K. M. MASUD (1), K. ISOGIMI (2) and J. SUZUKI (2)

(1) *Dept. of Industrial Production Eng.*
Bangladesh University of Eng. and Tech.,
Dhaka-1000, Bangladesh

(2) *Dept. of Mechanical Eng., Mie University,*
Kamihama-cho 1515, Tsu 514-8507, Mie, Japan

EFFECT OF FIBER TREATMENT on the bending strength of the aramid short fiber composite is studied in this paper. Interfacial shear strength and fiber strength are determined for different fiber treatments by performing single-fiber pull-out test. Effect of the fiber treatments on the property of the composite depends both on the interfacial shear strength and the fiber strength, simultaneously. Thus, to evaluate quantitatively the effect of the treatments on the strength of the composite a parameter "critical embedded length" is introduced that covers both the interfacial shear strength and the fiber strength. It is a parameter that defines the maximum embedded length during pull-out test, above which the shear force in the fiber/matrix interface exceeds the value of fracturing force of the fiber. The result shows that decrease of the critical embedded length causes increase in the bending strength of the composite, for the cases of the treatments employed in this research. Finally, an empirical equation is developed to calculate the bending strength of the aramid short fiber composite for the fiber treatments employed in this research.

1. Introduction

IT IS REPORTED [1, 2] that reinforcement by 1 mm aramid short fiber does not cause an increase of the bending strength of the composite if compared to that of the plain Polyester resin. On the other hand, bending strength of the composite with 3 mm long fiber increases a little (only in the case of high fiber content) compared to that of the resin. Therefore in this research, we have tried to analyse interfacial property of the composite by performing several fiber treatments. We have tried to introduce an epoxy interface in between the fiber and matrix by treating the fiber with epoxy. In case of the fiber treatments applied in this paper, it is found that the treatment changes interfacial property and strength of the fiber.

http://rcin.org.pl

Fiber/matrix interface is a crucial region in the composite and usually considered as responsible for transmitting the load from matrix to fiber. Therefore, the interface has great influence on mechanical properties of the aramid short fiber composites. In case of aramid fiber and the treatments employed in this research, it is found that the interfacial shear strength and fiber strength vary simultaneously with the treatments. So far, the change of the fiber strength due to fiber treatment is often neglected in many studies on composite interface. Therefore, conclusion on the effect of the interface in mechanical properties of composite cannot be drawn correctly, and thus a complete understanding of the effect of fiber treatment is not possible. The change of mechanical properties of the composite may not only depend on the interfacial property but also depends on the fiber strength. In an extreme case of very short fiber, the fiber strength might not be an important factor. In this research, experimental studies have been made to explain the effect of fiber treatment on the bending strength of the aramid short fiber composite.

Single-fiber pull-out test is a simple means to determine interfacial property of fiber and matrix. Here, the classical single-fiber pull-out test is employed to determine the interfacial property of the aramid fiber and polyester matrix and we can also determine the fiber strength during the pull-out test. It is found that the interfacial strength and the fiber strength are different for different fiber treatment. In this paper, a parameter "critical embedded length" is introduced that corresponds both the interfacial shear strength and the fiber strength. It is a parameter that defines the maximum embedded length during pull-out test, above which the shear force in the interface exceeds the value of fracturing force of the fiber. The result shows that decrease of the critical embedded length causes to increase the bending strength of the composite for the cases of the treatments employed in this research. Finally, an empirical equation is developed to calculate the bending strength of the aramid short fiber composite for the fiber treatments employed in this paper.

2. Experimental

Aramid (Kevlar 29 of type T950 and T965A, made by Toray Du Pont Co., Ltd., Japan) short fibers are used as filler. These short fibers are 1 and 3 mm in length. Unsaturated Polyester (8285AP type, made by Takeda Yakuhin Co., Ltd., Japan) resin is used as matrix. Permek-N (made by Nihon Yushi Co. Ltd., Japan) is used as hardener for the polyester resin.

Fabrication process of short fiber composite is similar to that employed in previous paper [1, 2]. Aramid short fiber is mixed into the polyester resin and degassed by a vacuum pump. After proper mixing of the hardener (1 wt% of the resin), the mixture is poured into a mold and compressive force is applied on it. The short fiber composite was cured under compression for 24 hr at room temperature (298 K). After releasing the composite from the mold, post-curing of the fabricated composite is done at 353 K for 3 hr in a drying oven.

Fabricated short fiber composite (without any machining) is used as test specimen for three point bending test. Figure 1 shows the nominal dimensions of the specimen and set-up of the bending test. Response of the load is measured and recorded by a $x - t$ recorder. The cross head speed is kept constant at 0.027 mm/sec during the test.

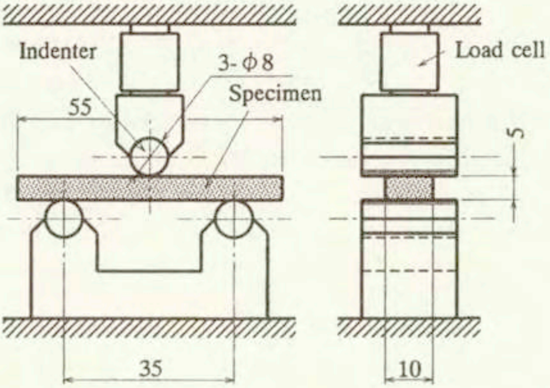

FIG. 1. Set-up of the three point bending test and nominal dimension of the specimen.

2.1. Fiber treatments

It is reported in previous paper [1, 2] that the bending strength of the composite of 1 mm short fresh aramid fibers does not increase compared to that of the resin even at very high fiber content. Only in case of high fiber content, bending strength of the composite of 3 mm short fresh aramid fiber increases a little compared to that of the resin. Therefore, in order to modify the interfacial property following fiber treatments are done.

Two kinds of solutions are prepared by mixing epoxy into water and toluene solvent where amount of the epoxy is 1% of the solution. Two types of Kevlar 29 fibers (T950 and T965A type) are immersed into the epoxy solutions and dried at 513 K. Another fiber treatment is done by heating Kevlar 29 (T950 type) fiber at 353 K for 60 days in a drying oven. In this paper, these above mentioned treatments are defined by EW-T950, EW-T965A, ET-T950, ET-T965A and H-T950 accordingly and are also shown in Table 1. In order to investigate the effect of the treatments on the strength of the composites, short fiber composites are produced by each treated fiber (1 and 3 mm long) and performed three-point bending test.

Table 1. Identification of the fiber treatments.

Type of fibers Type of treatments	Kevlar 29 T950	Kevlar 29 T965A
Epoxy-Water solution treatment	EW-T950	EW-T965A
Epoxy-Toluene solution treatment	ET-T950	ET-T965A
Heating at 353 K for 60 days	H-T950	——

It is assumed that not only the interfacial properties affect the strength of the short fiber composite but tensile strength of the fiber also affects the strength property of the composite. Therefore, in order to investigate the effect of the treatments on the bending strength of the short fiber composite, interfacial property and fiber strength of each type of fibers are measured. Single-fiber pull-out test is a simple means for determining the interfacial property and in this research we have also performed this test to investigate the interfacial property.

2.2. Pull-out test

Figure 2 shows a schematic diagram of the specimen of single-fiber pull-out test. A construction paper was selected to prepare the pull-out test specimen. Two trapezoid shaped holes were cut off leaving a space in between them for embedment as shown in Fig. 2. Width of this space ultimately controls the embedded length of the specimen. A fiber was carefully placed along the center of the holes and was temporally fixed. A drop of polyester was placed on the space in between the trapezoids by a needle-like bow. Then the polyester was hardened for 24 hours. Embedded region was carefully observed and embedded length L was measured by an optical microscope. Enlarged drawing of the embedded region is also shown in Fig. 2. Middle portion of the construction paper is cut off after mounting the specimen on the test machine, so that, the embedded length would be directed towards the loading direction.

Response of the load cell is passed through an amplifier and is recorded by a $x - t$ recorder. P_t is maximum load required to pull-out the fiber from the resin. At a certain value of the embedded length, the load P_t required to pull out the fiber exceeds the fracturing load of the single fiber. This value of embedded length is defined as critical embedded length L_c. Most of the fibers are found to be fractured out when the embedded length is greater than the critical embedded length. This fracturing load of the fiber is used to calculate the strength of the fiber.

http://rcin.org.pl

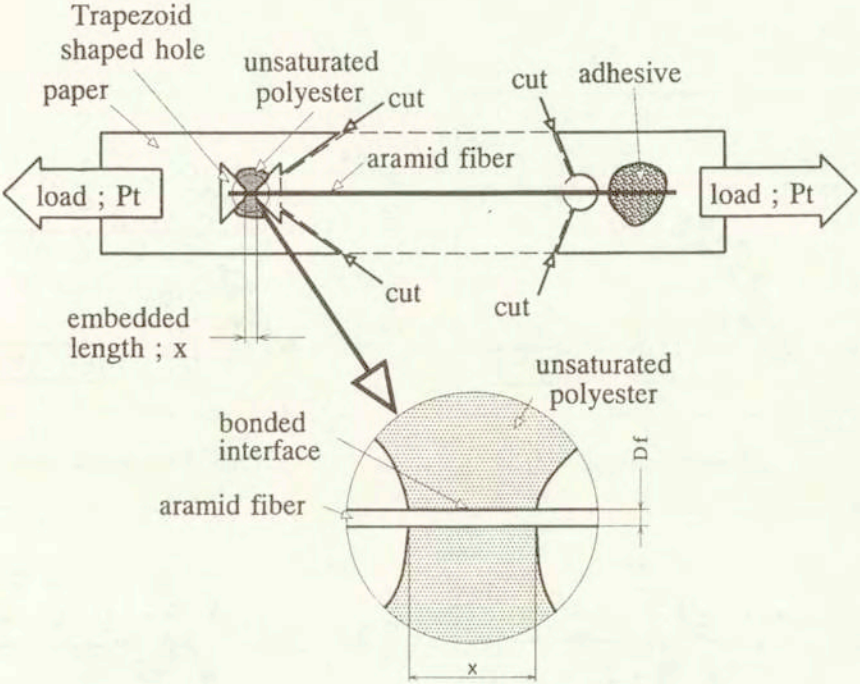

FIG. 2. A pull-out test specimen.

3. Results and discussion

Figure 3 shows the results of pull-out test of the aramid fresh fiber and the treated fibers. At small and large embedded length, fibers are usually found to be pulled out from the matrix and torn, respectively. From the results we could find a transient region where some fibers are torn and some are pulled out and usually this transient region is around the critical embedded length L_c. Figure 3 shows that the load required to pull out the fiber from the matrix increases with the embedded length. Here, it is assumed that the pull-out load increases linearly with the embedded length. Therefore, the data of pull-out region are fitted by the method of least squares and the line is defined as pull-out load line. On the other hand, mean value of the load required to fracture the fiber is also plotted in Fig. 3 and this line is defined as fiber fracture load line. However due to the presence of the transient region, it is difficult to find a unique critical embedded length from the results of the pull-out test. Therefore we have defined the critical embedded length L_c as the embedded length at which the pull-out load line and the fiber fracture load line intersect each other. High inclination (P_t/L) of the pull-out load line and mean fiber fracture load correspond high interface strength and

http://rcin.org.pl

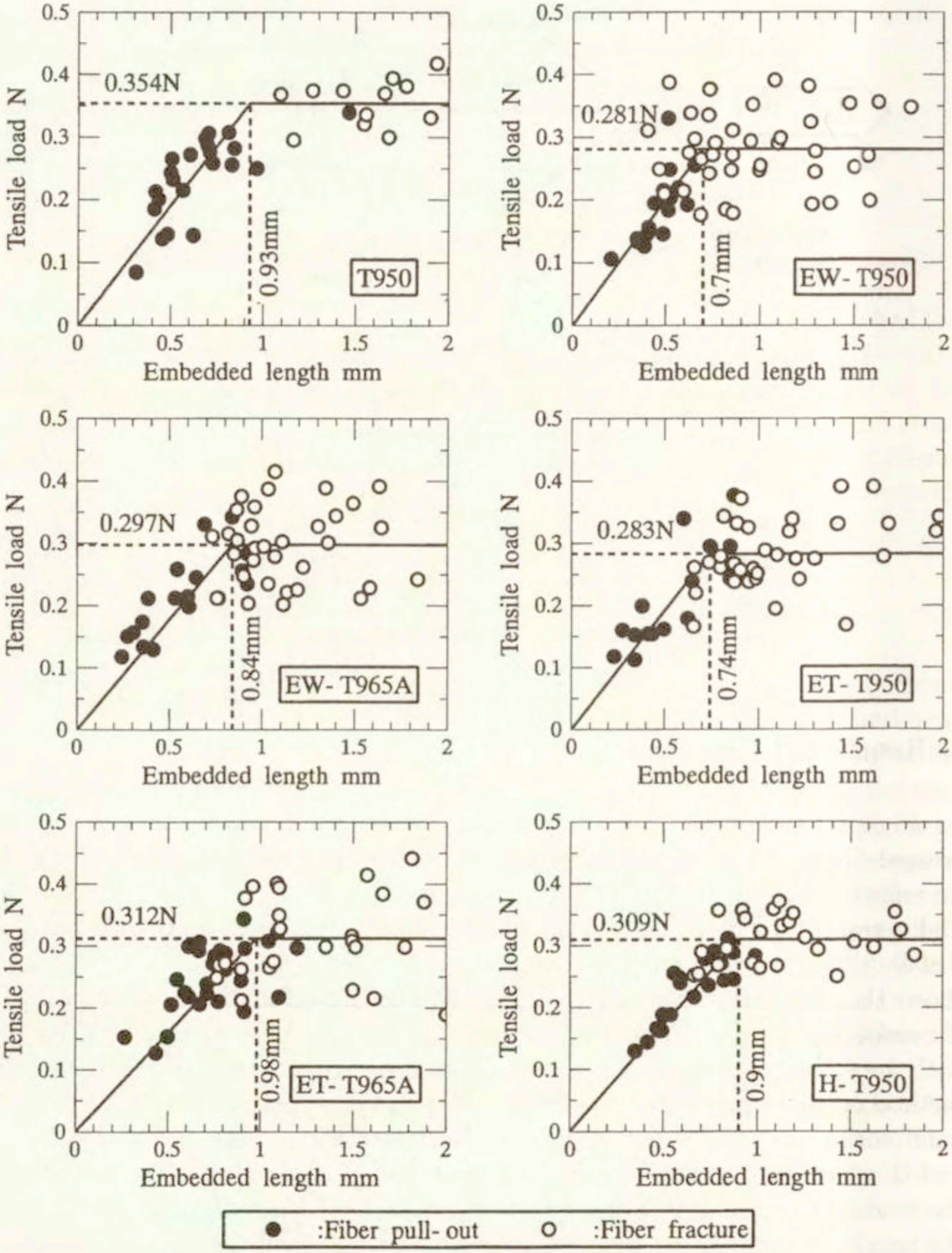

FIG. 3. Pull out test results of the aramid fresh fiber and the treated fibers.

http://rcin.org.pl

fiber strength, respectively. From Fig. 3, we could find that inclination (P_t/L) of the pull-out load lines and mean value of the fiber fracture loads is different for different fiber treatment. Therefore, the critical embedded length L_c also differs from treatment to treatment. As this critical embedded length depends both on the pull-out and fiber fracture property, we have considered this length an important parameter in defining the fiber treatments.

Interfacial shear strength for each treatment was calculated using following Eq. (3.1), with assumption that shear stress is distributed uniformly in the part of fiber/matrix interface.

$$\tau_i = \frac{P_t}{\pi D_a L}, \tag{3.1}$$

where, τ_i and D_a is defined as interfacial shear strength and average measured diameter of each type of fiber, respectively. Diameters of the fiber are measured at more than 20 points along the fiber length by using Scanning Electron Microscope (SEM) and compared with the results obtained by an optical microscope. The standard deviation is obtained as about $0.9 \sim 1.2$ μm. In Eq. (3.1) P_t/L is the inclinations of the pull-out load lines for each fiber treatment. On the other hand, average fiber fracture load is divided by cross sectional area of the fiber to calculate average fiber strength σ_f. Result obtained from the pull-out test is summarised in Table 2.

Table 2. Fiber fracture strength and interfacial property of the aramid fresh fiber and the treated fibers.

Type of fiber	Average diameter D_a μm	Inclination P_t/L N/mm	Interfacial shear strength τ_i MPa	Critical embedded length L_c mm	Average fiber fracture load P_f N	Average fiber strength σ_f MPa
T950	13.31	0.381	9.10	0.93	0.354	2544.2
EW-T950	12.45	0.401	10.25	0.70	0.281	2308.2
EW-T965A	12.20	0.355	9.26	0.84	0.297	2540.7
ET-T950	12.62	0.383	9.66	0.74	0.283	2262.4
ET-T965A	12.35	0.319	8.22	0.98	0.312	2604.5
H-T950	12.17	0.344	9.00	0.90	0.309	2656.4

The critical embedded length L_c depend on the inclination of the pull-out load line as well as the interfacial shear strength and the fiber strength, for all the cases of the fiber treatments. Moreover, it can be concluded that decrease of the fiber strength reduces the critical embedded length and increase of that strength makes the length longer, and increase of the interfacial shear strength reduces the critical embedded length and decrease of that strength makes the length longer. However, H-T950 treatment seems somewhat different. Therefore,

http://rcin.org.pl

we assumed that the critical embedded length is a single parameter to define the fiber treatment for aramid fibers.

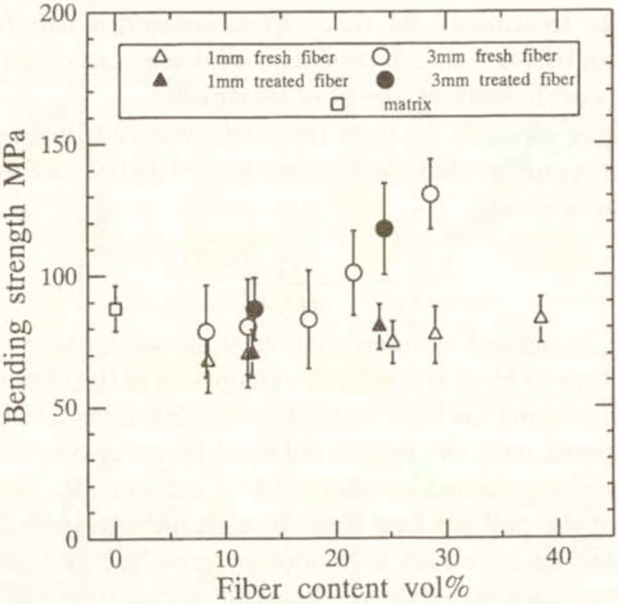

FIG. 4. Bending strength of the composite of treated and untreated fiber with respect to the content of the fiber.

Three point bending test is carried out in order to investigate the effect of the treatments on the bending strength of the short fiber composite. Figure 4 shows the bending strength of the composite of the aramid fresh fiber and the treated fibers. In case of the fresh fibers, we could find that the bending strength of the composite of 1 mm fiber does not increase compared to that of the matrix even at very high fiber content, but the strength of the composite of 3 mm fiber increases compared to that of the matrix when the fiber content is above 20 vol% [1]. Two typical fiber contents 12 vol% and 24 vol% are selected and investigated the effect of the fiber treatments on the composite in detail. Filled circle and triangle shown in Fig. 4 indicate the average strength of the composites of all the five fiber treatments. We could expect from Fig. 4 that the strength of the composite increases a maximum of about 10% with the treatments employed in this research, except in case of the composite of 1 mm fiber and about 12 vol%.

Therefore, the strength of the composite depends on the content and length of the fiber and the fiber treatment. If we concentrate our discussion on the individual treatment, we could find the bending strength of the composite of different treated fiber as shown in Table 3 and 4.

Table 3. Bending strength of composite of 1 mm fiber.

Type of fiber	Fiber content V_f vol%	Average bending strength σ_c MPa	Standard deviation MPa
T950	11.98	70.02	12.48
EW-T950	12.49	72.16	9.17
EW-T965A	12.02	68.72	8.74
ET-T950	11.68	68.02	8.34
ET-T965A	12.38	63.69	6.15
H-T950	12.24	79.23	13.35
T950	25.16	74.48	8.15
EW-T950	23.96	85.84	6.81
EW-T965A	23.24	69.49	6.69
ET-T950	24.31	91.35	12.95
ET-T965A	23.58	72.57	6.83
H-T950	24.52	83.69	9.66

Table 4. Bending strength of composite of 3mm fiber.

Type of fiber	Fiber content V_f vol%	Average bending strength σ_c MPa	Standard deviation MPa
T950	12.00	80.72	18.2
EW-T950	12.04	86.29	7.84
EW-T965A	12.04	85.30	12.88
ET-T950	12.29	84.35	8.34
ET-T965A	12.42	74.60	19.74
H-T950	14.18	107.37	10.32
T950	21.62	101.00	15.99
EW-T950	24.00	110.10	8.7
EW-T965A	24.20	115.56	22.1
ET-T950	23.98	125.6	13.55
ET-T965A	25.98	119.89	15.2
H-T950	24.00	116.96	26.7

Concerning to the composites of 1 mm fiber shown in Table 3, in case of the composite of fiber content of about 24 vol%, bending strength of the composite of EW-T950, ET-T950 and H-T950 fiber increase about 13% $\sim$ 17% compared to that of the T950 fiber.

On the other hand, bending strengths of the composite of 3 mm fiber are shown in Table 4. In case of the composite of fiber content of about 12 vol%, bending strength do not vary significantly with the treatments except H-T950 fiber. Bending strength of the composite of H-T950 fiber increase exceptionally about 33% compared to that of the T950 fiber. Moreover in case of the composite of fiber content of about 24 vol%, bending strength of the composite of all the treated fibers increase about 9% ∼ 16% compared to that of the T950 fiber. However, this increment and decrement of the strength depend not only on the treatments but also on the content of the fiber into the composite.

After all we can summarise that these variations of the strength of the composite of different fibers depend on the fiber content, fiber length, interfacial shear strength and fiber strength as well. Therefore, in order to define the effect of those factors and fiber treatments on the strength, we have developed an empirical equation.

Tensile strength of the short fiber composites σ_t is usually calculated by the following equation [3].

$$\sigma_t = \Phi\eta\sigma_f V_f + (1 - V_f)\sigma_m, \tag{3.2}$$

where, σ_m is the strength of the matrix material. The factor η is considered the orientation efficiency of the reinforcing fibers and Φ is the length efficiency factor. Here, the first term of the Eq. (3.2) explains the effect of the short fiber on the strength of the composite. Interfacial property of the fiber and matrix is not directly considered in the Eq. (3.2), and thus the construction of this equation can not represent the effect of fiber treatment on the strength of the composite. Therefore, we have derived an empirical equation that can represent the effect of the fiber treatment on the strength.

As during the bending test, maximum tensile force would act in a very thin layer on the bottom surface and the direction of the tensile force is along the neutral axis, we could consider this thin layer a tensile test specimen. Fracture would occur in this thin layer at an imaginary plane perpendicular to the direction of the tensile force. This imaginary plane would divide the distributed fibers into two portions and smaller one would act as embedded length. When the embedded length is smaller than corresponding critical embedded length then the fiber would pull out. When the embedded length is longer than corresponding critical embedded length, then the fiber would fracture out. Therefore, it is assumed that the first term of Eq. (3.2) could be considered to define the effect of the short fibers on the bending strength of the composite.

However, in order to define the bending strength of the composite made by different fibers, we have introduced a parameter ξ as a ratio of the strength of the composite to the effect of the short fibers into the composites. Basic idea of the effect of the short fiber into the composite is taken from the first part of the Eq. (3.2). This parameter ξ might be called as relative bending strength of the composite with respect to the effect of the short fibers into the composite and is shown below

http://rcin.org.pl

$$\xi = \frac{\sigma_c}{\sigma_f V_f \eta \left(\dfrac{L_f}{L_0}\right)}, \tag{3.3}$$

where, σ_c, σ_f, V_f and L_f are defined as experimental value of the bending strength of the composite, fiber strength, fiber volume fraction in the composite and length of the short fibers into the composite. Here, the term L_f/L_0 is considered as fiber length efficiency factor and L_0 is ideal length of the short aramid fiber for reinforcement in the composite. Then the value of length L_0 can be obtained as follows,

$$L_0 = 2\frac{\Sigma L_c}{N}, \tag{3.4}$$

where, L_c is the critical embedded length of fibers of each treatment that are shown in Table 2 and N is number of fiber treatments. Then value of the length L_0 is found approximately 1.7 mm. In considering the effect of the short fibers into the composite, it is assumed that both of the fiber content V_f and the length efficiency factor L_f/L_0 are directly contributing to the strength as shown in the denominator of the right hand side of the Eq. (3.3).

As described above, the maximum tensile force would be induced in a very thin layer on bottom surface under the bending loading. And the fracture or the pull-out of the reinforcing fiber is decided by the relationship between the embedded length and the critical embedded length. Further, the critical embedded length can well define the fiber treatments as described in the discussion of Table 2. Therefore, in this paper the critical embedded length is employed as a unique parameter to define effectiveness of the fiber treatments.

In order to define the fiber treatments, another parameter ζ is introduced and defined as a ratio of the critical embedded length of the treated fiber to that of the fresh fiber, that is, value of the parameter ζ is shown below

$$\zeta = L_c/L_{c0}, \tag{3.5}$$

where, L_{c0} is defined as critical embedded length of untreated fiber. The parameter ζ is a non-dimensional parameter that describes the fiber treatments.

Figure 5 shows the effect of the treatments on the relative bending strength of the composite with respect to the effect of the short fiber into the composite. From Fig. 5, we find that the data of 1 mm fiber show higher value of the relative bending strength than those of 3 mm fiber. Moreover, higher fiber content (24 vol%) also reduces the relative bending strength in both the cases of 1 and 3 mm fiber. These results mean that the increase of the length and content of the fibers do not cause sufficient increase of the bending strength of the composite. Decrease of the parameter ζ defines developed fiber treatment. Therefore, an increase of the parameter ζ causes a decrease of the relative bending strength for each of the cases of the fiber length and fiber content.

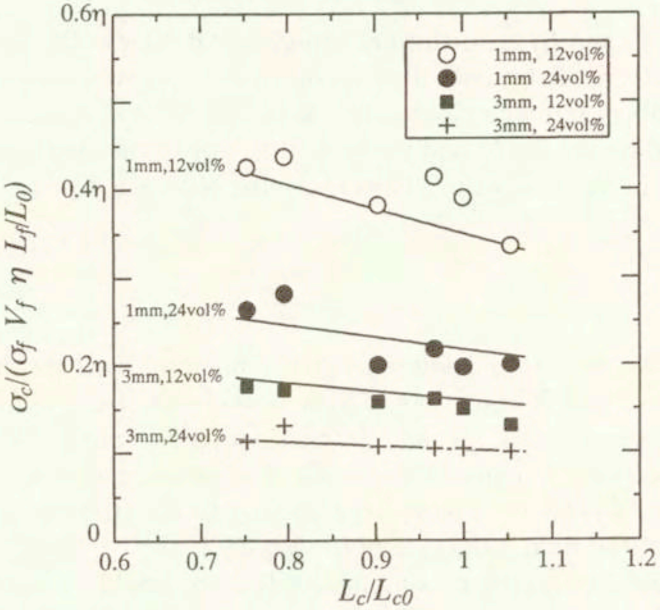

FIG. 5. Effect of the non-dimensional critical embedded length on the relative bending strength of the composite with respect to effect of the short fibers into the composite.

We have developed an equation to explain the result described in Fig. 5. Here, it is assumed that the relative bending strength of the composite decreases linearly with the parameter ζ, for each of the cases of the fiber length and fiber content. Then generalised linear equation can be expressed as follows,

$$\xi = A\zeta + B, \tag{3.6}$$

where, A and B are defined as constants depend on the length and content of the fiber. Value of A and B is calculated such that sum of square of error of each plot of Fig. 5 is minimum. Substituting the value of ξ and ζ from Eqs. (3.3) and (3.5) into the Eq. (3.6), the empirical equation of the bending strength of the composites can be derived as shown below,

$$\sigma_c = 0.075\sigma_f \left(V_f \frac{L_f}{L_0}\right)^{1/5} - 0.02\sigma_f \frac{L_c}{L_{c0}}. \tag{3.7}$$

The first term of the Eq. (3.7) explains the effect of the short fiber on the strength of the composite, and the second one of the Eq. (3.7) explains the development of the fiber treatment.

http://rcin.org.pl

The fiber content V_f and length efficiency factor L_f/L_0 are powered by 1/5 in the first term. The constant 0.075 of the first term might depend on the distribution of the fiber. In some research works [3, 4], fiber orientation parameter f_p is introduced as follows,

$$f_p = 2\cos\phi - 1, \tag{3.8}$$

where, ϕ is the average angle between distributing fiber and the loading direction. If we consider the constant 0.075 of the first term of Eq. (3.7) as an orientation parameter f_p, then the average orientation angle ϕ would become 42.8 degree. On the other hand, we know that the average orientation angle is 45 degree, in case of random orientation [4]. As this average angle does not define adequately the distribution of the fibers into the composite, it might not be quite reasonable to say that the distribution is very close to random. However, the assumption that the distribution of the fibers is three dimensional proposed in previous discussions [1, 2] would be closely compromised by the average orientation angle obtained.

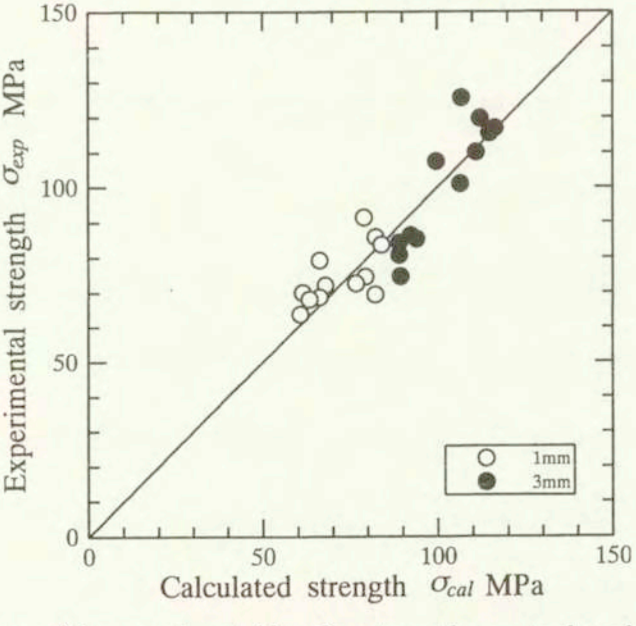

FIG. 6. Comparison of the experimental bending strength σ_{exp} to the calculated bending strength σ_{cal}.

The second part of the Eq. (3.7) defines the interfacial property as well as the fiber treatments. If L_c decreases keeping the fiber strength constant, that means an increase of interfacial shear strength, then the strength of the composite calculated by Eq. (3.7) would increase. This is the same conclusion obtained through the discussion about the results of Table 2 and 3, 4.

http://rcin.org.pl

Figure 6 shows the comparison of the bending strength of the composite calculated by the empirical Eq. (3.7) to the practical bending strength obtained in our experiment. All the plots are almost very close to the diagonal line. Therefore, the empirical equation is justified well under the experimental conditions employed in this research.

References

1. A. K. M. MASUD, J. SUZUKI and K. ISOGIMI, *Fabrication of advanced FRP with aramid short fiber and its properties*, Int. Conf. Adv. Tech. in Exp. Mechanics, Wakayama, Japan, 181–186, 1997.
2. A. K. M. MASUD, J. SUZUKI and K. ISOGIMI, *Effect of the fiber surface treatment on the mechanical behavior of aramid short fiber reinforced polyester*, First Asian-Australasian Conference on Composite Materials (ACCM-1), Osaka, Japan, 611–1/4, 1998.
3. H. VOSS and K. FRIEDRICH, *Influence of short fiber reinforcement on the fracture behaviour of a bulk liquid crystal polymer*, J. Mater. Sci., **21**, 2889–2900, 1986.
4. K. FRIEDRICH, *Fracture mechanical behavior of short fiber reinforced thermoplastics*, Fortschr.-Ber. VDI-Z., **18**, 8, 1984.

Received Juni 26, 1999; new version April 4, 2000

http://rcin.org.pl