Interfacial Evaluation of Kenaf and Ramie Fibers/Epoxy Composites using Micromechanical Technique

Tran Quang Son[*], **Joung-Man Park**[*,†], **and Byung-Sun Hwang**[**]

Micromechanical 시험법을 이용한 Kenaf 와 Ramie 섬유강화 에폭시 복합재료의 계면 물성 평가

트란콩손[*] · 박종만[*,†] · 황병선[**]

KEY WORDS: green composite, natural fiber, acoustic emission, interfacial properties, interfacial shear strength (IFSS), fragmentation test.

ABSTRACT

Interfacial shear strength (IFSS) of environmentally-friend natural fiber reinforced polymer composites play a very important role in controlling the overall mechanical properties. In this work the IFSS of Ramie and Kenaf fibers/epoxy systems were evaluated using the combination of micromechanical technique, microdroplet test to find out an optimal condition in accordance with final purpose by comparing to each other. Clamping effect on fiber elongation was determined as well. In addition, the mechanical properties of the natural fibers were investigated using single fiber tensile test and analyzed statistically by both uni- and bimodal Weibull distributions. Microfailure modes of different natural fiber structures were observed using optical microscope.

Nomenclature

τ : Interfacial shear strength (IFSS)
ΔL : Fiber extension
L_o : Original length of fiber
D : Fiber diameter
l : Embedded length
α : Scale parameter
β : Shape parameter
p : Low strength population
q : High strength population

1. INTRODUCTION

Recently, natural fiber reinforced polymer composites have attracted an considerable attention in research and industrial fields due to some advantages such as low density, high specific strength, their abundance, low cost, and together with biodegradable ability [1-4]. However, the natural fibers exhibit high moisture absorption, which can be a major problem for many applications and especially for interfacial adhesion between the hydrophilic natural fibers and resin matrices [4]. It is necessary to extensively investigate the interfacial adhesion because the interface between the natural fibers and resin matrix plays a very important role in controlling the overall properties of the composites such as off-axis strength, fracture toughness, environmental stabilities by transferring the load from matrix material through the interface/interphase onto the fibers which have the high tensile strength. There are several methods available to quantify the interfacial adhesion in composite materials. In the case of single fiber composite system, some techniques such as the pullout test, the single fiber fragmentation test have been used. In this work, the mechanical properties of the natural fibers were

---

[*] Department of Polymer Science and Engineering,
Engineering Research Institute,
Gyeongsang National University
[**] Composite Materials Group
Korea Institute of Machinery and Materials
[†] To whom correspondence should be addressed.

investigated using single fiber tensile test method and statistically analyzed by both uni- and bimodal Weibull distributions. Acoustic emission (AE) technique can be also used as a nondestructive evaluation (NDE) for detecting microfailure mechanism of the composite materials in the future [5,6].

2. EXPERIMENTAL

2.1. Materials

Ramie and Kenaf fibers were supplied from Yamaguchi University, Japan as reinforcing fibers for green composites. Epoxy resin (YD-128, Kukdo Chemical Co., Korea) based on diglycidyl ether of bisphenol-A was used as a matrix. Flexibility of the epoxy matrix was controlled by changing the ratio of Jeffamine (polyoxypropylene diamine, Huntsman Petrochem. Co.) D400 *versus* D2000 in the curing mixture. Epoxy was pre-cured at 80°C for 2 hours and then post-cured at 120°C for 2 hours

2.2. Methodologies

2.2.1. Measurement of single fiber tensile strength Ramie and Kenaf fibers were fixed on the paper frame using Kapton tape with various gauge lengths, respectively. The tensile strength of biodegradable fibers with the various gauge lengths were obtained using about sixty specimens for meaningful value and statistically analyzed using both uni- and bimodal Weibull distribution. The fiber failure process of the unimodal cumulative Weibull distribution function based on one type defect is

$$F(t) = 1 - \exp\left[-\left(\frac{t}{\alpha}\right)^{\beta}\right] \qquad (1)$$

Where α and β are the shape and scale parameters, respectively. The cumulative bimodal Weibull distribution function based on the presence of two kinds of defect is described as

$$F(t) = 1 - \left\{p\exp\left[-\left(\frac{t}{\alpha_1}\right)^{\beta_1}\right] + q\exp\left[-\left(\frac{t}{\alpha_2}\right)^{\beta_2}\right]\right\}$$

$$p + q = 1 \qquad (2)$$

Where p and q are the portions of low and high strength population, and β_1, α_1, β_2, and α_2 are the shape and scale parameters for the low and high strength portions, respectively.

2.2.2. Specimen Preparation and IFSS Measurement: Natural fibers were fixed with regularly separated distance in a steel frame. Microdroplet of epoxy matrix was formed on each the natural fiber using carbon fiber of 8 μm in diameter as a tip pin. Microdroplet specimens were cured thermal conditions such as pre-curing at 80°C for 2 hours and post-curing at 120°C for 2 hours and then cooled at slowly speed of 0.5°C/min. A microdroplet specimen was fixed by the microvice using a specially designed micrometer. The IFSS was calculated from the measured pullout force, *F* using the following equation,

$$\tau = \frac{F}{\pi D_f L} \qquad (3)$$

Where D_f and L are fiber diameter and fiber embedded length in the matrix, respectively.

3. RESULTS AND DISCUSSION

3.1. Morphology and structure of natural fibers. Figure 1 shows morphology and structure of natural fiber bundles are composed of several high oriented elementary fibers embedded by some matrix materials such as lignin, hemicellulose, water, and extractives. The elementary fiber is based on cellular material, which is defined as a linear, crystalline polymer composed of (1-4) linked β-D-glucopyranose and can respond to high stress. Whereas hemicelluloses, wax, lignin have low tensile strength and make adhesion between the fiber surface and matrix materials poor due to high hydrophilic property. Figure 2 shows morphology in diameter direction and crossed section of Kenaf and Ramie fibers, for the natural fibers its diameter is not circular. The natural fibers contain two types of material structure such as an amorphous region and crystal region. Figure 2 shows that the amorphous region corresponded to hemicelluloses, lignin is a white part of the fibers, whereas the crystal region is a dark part of the fibers.

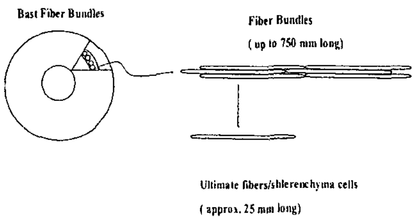

Figure 1. Morphology and structure of natural fibers

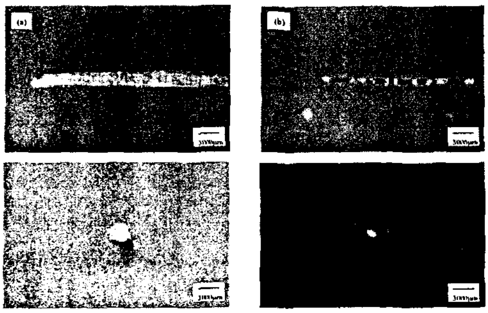

Figure 2. Morphology and crossed section: (a) Kenaf fiber, (b) Ramie fiber

3.2.Clamping Effect. Figure 3 shows clamping effect on elongation of the natural fiber. It is because a part of the fiber can be pulled out from the clamp during testing. The part of the fibers slipping ΔL_c contributes to the total extension ΔL_{tot} in the stress-strain experiment with the various gauge length of natural fibers. If the lines is extrapolated to L_o =0, one gets the clamping effects,

$$\Delta L_{tot} = \Delta L + \Delta L_c \qquad (4)$$

Where ΔL is the real elongation which can was valuated from the above equation

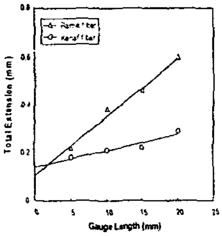

Figure 3. The relationship between the total extension and the gauge length

Table 1 shows calculation of the clamping effect on the elongation of both Kenaf and Ramie fibers.

Table 1. The clamping effect for two natural fibers

Parameter	Non-orrected elongation ε_n (%)	Total extension ΔL_{tot} (mm)	Clamping effect ΔL_c (mm)	Corrected elongation ε_c (%)
Ramie	0.60	3.00	0.11	2.45
Kenaf	0.29	1.45	0.14	0.75

3.3. Mechanical properties and the gauge length. Figure 4 shows the change in the tensile strength and Young's modulus with the various gauge length of Kenaf and Ramie fibers. The tensile strength for the both fibers decreases when the gauge length increases and follows the same principle as conventional fibers such as glass fiber, carbon fiber. It may be because when the gauge length increases, a number of defects increase along with its length. The tensile strength and modulus of Ramie fiber were significantly higher than that of Kenaf fiber, it may be because of a difference of their structure and chemical Composition. The cellular composition of Ramie fiber is significantly higher than that of Kenaf case. The change in the mechanical properties with the various gauge length were detailed in Table 2 for both Ramie and Kenaf fibers

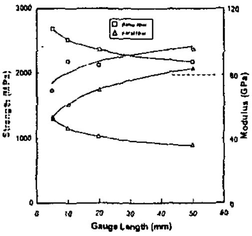

Figure 4. Mechanical properties – the gauge length curves

Table 2. Mechanical properties of Kenaf and Ramie fibers

Gauge length (mm)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation (%)	COV[b] (%)	α[c]	β[d]
Ramie						
5	2684 (714)[a]	69.9 (29.8)	4.1 (0.8)	27	2805	4.5
10	*2517(468)*	*88.2 (18.6)*	*3.5 (0.4)*	19	2575	7.1
20	2380 (469)	85.8 (33.9)	3.9 (0.9)	20	2615	5.0
50	2172 (534)	95.4 (27.7)	2.7 (0.5)	25	2331	5.6
Kenaf						
5	1316 (249)[a]	54.1(7.8)	2.5 (0.5)	19	1421	5.0
10	1137 (255)	61.2 (17.0)	2.1 (0.4)	23	1229	4.4
20	1010 (309)	70.3 (8.3)	1.2 (0.3)	31	1128	3.1
50	888 (239)	84.0 (12.1)	1.1 (0.2)	27	954	3.7

a Standard deviation (SD)
b Coefficient of variation (COV) for tensile strength = SD/mean*100
c Scale parameter for fiber strength
d Shape parameter for fiber strength

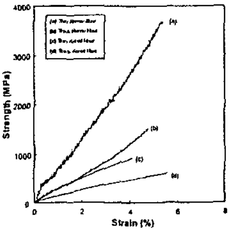

Figure 5. Mechanical properties-various diameter curves

3.4. Mechanical properties and diameter Figure 5 shows the change in the tensile strength and elongation with the diameter. The tensile strength decreases with an increase in the diameter for both Ramie and Kenaf fibers. It may be because high orientation of elementary fibers in fiber bundles will decrease with increase in the diameter. Whereas the elongation increases when the diameter increases because under load the elementary fibers slips together before fiber breakage happens.

3.5. Microdroplet test Figure 6 shows the plots of pullout force versus embedded length for (a) the untreated Ramie and (b) Kenaf fibers/epoxy composites in microdroplet test, respectively. Critical embedded area means the intimate contacting area between fiber and matrix. The critical embedded area was used instead of critical embedded length due to the different diameter for both fibers. The critical embedded area was obtained by intersection of two linear regression lines: one is the fiber pullout linear regression line, whereas another is the fiber fracture linear regression line. The critical embedded area can be correlated to IFSS values. The narrower the critical embedded area, the higher the IFSS value.

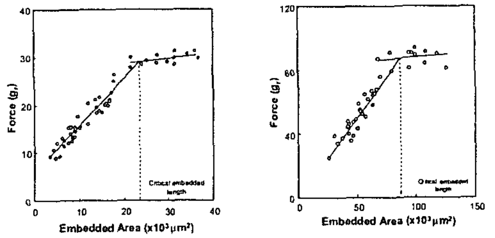

Figure 6. Debonding force – embedded area curves: (a) for Ramie fiber; (b) for Kenaf fiber.

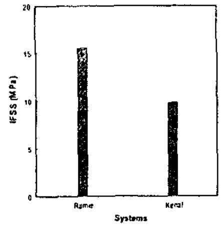

Figure 7. IFSS of Ramie and Kenaf fibers/epoxy composites

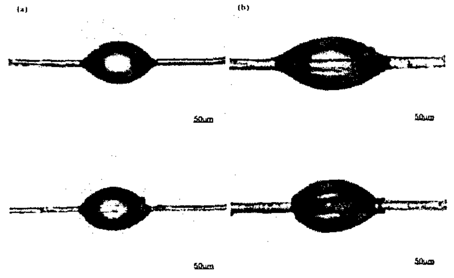

Figure 8. Typical microfailure modes of the natural fiber/epoxy: (a) Ramie fiber; (b) Kenaf fiber.

3.6. IFFS measurements. Figure 7 shows the IFSS of both untreated Ramie and Kenaf fibers/epoxy systems calculated for each sample according to the equation (1). For Ramie fiber case, the IFSS is higher than that of Kenaf fiber. It is consistent with their structures and chemical composition. Kenaf fiber contains some more chemical compositions such as hemi-celluloses, lignin, and wax than Ramie fiber. These chemical agents make adhesion between the fiber surface and matrix material to be poor based on their hydrophilic properties. Figure 8 shows the microfailure modes of Ramie and Kenaf fibers/epoxy systems before and after pulling out.

CONCLUSION

Interfacial evaluation of Ramie and Kenaf fibers/epoxy composites was performed using microdroplet test. The IFSS of Ramie fiber/epoxy system was significantly higher than that of Kenaf fiber/epoxy system. It may be because of the difference of their structure and chemical composition. The clamping effect on the real elongation of the natural fibers during testing process was determined for both Ramie and Kenaf fibers using single fiber tensile test. The change in the mechanical properties of Ramie and Kenaf fibers such the tensile strength, tensile modulus, and the elongation with the various gauge lengths and diameters were obtained using single fiber tensile test and statistically analyzed using both uni- and bimodal Weibull distribution. The tensile strength and tensile modulus of Ramie fiber was significantly higher than that of Kenaf fiber, and decrease with increase in the gauge lengths and the diameters. The morphology, structure of Ramie and Kenaf fibers, and the microfailure modes were clarified in this work using optical microscope

ACKNOWLEDGMENT: This study was financially supported by National R&D Program by KISTEP.

REFERENCES

(1).A. Valadez-Gonzaleza, J.M. Cervantes-Uca, R Olayob, P.J. Herrere-Francoa, *Compos. Part B, 30, pp. 309-320, 1999.*

(2) N.E. Zafeiropoulosa, D.R. Williams, C.A. Baillie, F.L. Matthews, *Compos. Part A, 33, pp. 1083-1090, 2002.*

(3) R. Joffea, J. Andersonsb, L. Wallstroma, *Compos. Part A, 34, pp. 603-612, 2003.*

(4) N.E. Zafeiropoulos, C.A. Baillie, J.M. Hodgkinson, *Compos. Part A, 33, pp. 1185-1190, 2002.*

(5) J.M. Park, J.O. Lee, C.W. Park, *Poly. Compos., 17(3), p. 375, 1996.*

(6) J.M. Park, E.M. Chong, D.J. Yoon, J.H. Lee, *Poly. Compos., 19(6), p.747, 1998.*