Damage Behavior of Hemp Fiber Reinforced Poly(L-Lactic Acid) Composites under Fatigue Loading*

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Abstract
Green composites, such as natural fiber reinforced naturally-derived plastics attract much attention because of reducing CO₂ emission. Many studies about green composites have enabled the materials substitutes as glass fiber reinforced composites. However, lack of investigations about fatigue properties restricts the actual usage of the composites. In the present study, we investigate fatigue properties of green composites. A hemp fiber yarn reinforced poly(lactic acid) composite was selected as a green composite. Unidirectional (UD) and textile (Textile) composites were fabricated using micro-braiding technique. Fatigue tests results indicated that fatigue damages in UD composites was splitting which occurred just before the final fracture, while matrix crack and debonding between matrix and fiber yarn occurred and accumulated stably in Textile composites. These results were consistent with modulus reduction and acoustic emission measurement during fatigue tests.

Key words: Green Composites, Fatigue, Acoustic Emission, Hysteresis

1. Introduction
Advances in science and technology pose new challenges in relation to certain environmental issues, such as depletion of final disposable area and CO₂ emission. In this point of view, biodegradable, recyclable and eco-friendly materials have been necessary to preserve and protect our environments. Composite materials from the annually renewable natural fibers and biodegradable matrices have been developed in the past decade in an attempt to find alternatives to the fossil fuel-based polymeric materials in the automotive and packaging industries. Especially, continuous fiber yarns are expected as reinforcements instead of short fibers, because of lower mechanical properties of natural fibers. As a matrix, thermoplastic poly(lactic acid) (PLA) is also expected, since PLA is one of biodegradable and plant-derived plastic and has good mechanical properties.

Composites developed based on thermoplastic matrix materials have several advantages over those based on thermoset materials, such as storage stability without freezing device, higher stability in mechanical performance, recycling usage ability, in situ adaptability (e.g., through a hot treatment), and so on, and hence have received more attention in recent years. Unlike thermoset polymers, thermoplastic polymers have relatively high viscosity in melt state. Therefore, composites based on thermoplastic polymers are difficult to fabricate because of lower impregnation of the matrix to the reinforcement fiber yarns.

Previously, Sakaguchi et al. developed a micro-braiding method to supply flexible material design on continuous fiber reinforced thermoplastic composites [1]. The developed...
method allows many design choices of commingled yarns. Furthermore, high matrix impregnation and good fiber dispersion can be expected since the reinforcing fibers and the matrix fibers can be combined as one braided yarn. Recently, a micro-braiding technique was used for fabricating a continuous natural fiber reinforced biodegradable plastic composite [2].

From the viewpoint of actual usage, it is important to know fatigue characteristics of a material. Some investigations are conducted on fatigue properties of continuous natural fiber composites. Towo and Ansell [3, 4] conducted fatigue tests on sisal/epoxy and sisal/polyester composites. Liang et al. [5] studied fatigue behavior of flax/epoxy composites. These studies treated continuous natural fiber reinforced thermoset composites. Matrices of these composite are petroleum-based, so these are not complete renewable composites. Furthermore, they have no biodegradability. As a fatigue behavior of a complete naturally-derived and biodegradable composite, Katogi et al. [6] investigated a unidirectional jute/PLA composite. In the practical usage, a multidirectional composite, such as a textile composite is desired because of significant anisotropy of a unidirectional composite. Previously, many works were conducted on microscopic damage behavior in carbon or glass fiber reinforced composites subjected to fatigue loading. For example, microscopic fatigue damage initiation and progress in UD [7] and plain woven [8] composites were observed closely. However, knowledge on microscopic damage in natural fiber composites is limited. In this study, fatigue behavior of unidirectional and plain woven hemp fiber/PLA composites was investigated. Fatigue Life and modulus reduction were measured and macroscopic observation was also conducted. From the results obtained, micro-fracture process was deduced.

2. Experimental Method

A reinforcement used in this study was hemp spun yarn (tex590). The matrix material used in this study was biodegradable PLA fiber yarn (tex55) from TORAY, Japan. An intermediate material -micro-braided yarn (MBY)- was fabricated using Micro-Braiding technique. A continuous hemp fiber yarn was used as the straightly inserted axial yarn, and matrix fibers were braided around the reinforcing hemp fiber yarn. Figure 1 shows fabrication of micro-braided yarn and micro-braided yarn obtained.

Fabrication of unidirectional hemp fiber reinforced PLA (UD) composites was twofold process. The first step was performed by initially winding MBY 20 times in a parallel configuration onto a metallic frame. The second step involved placement of the metallic frame containing intermediate material in a pre-heated molding die for consolidation by compression molding to produce composite specimens. Molding conditions of processing temperature 190 °C, pressure 1MPa and time 4 min were selected, which was obtained the optimum condition in the previous study [9]. Static tensile strength of this specimen was 205 MPa.

In the present study, plain woven hemp fiber reinforced PLA (Textile) were also fabricated. First, plain woven fabrics were made using a loom. The geometry of fabrics was 150 mm × 150mm. Next, four fabrics were set in a pre-heated molding die for consolidation by compression molding. Molding conditions were same with those for UD. Warp and fill fiber yarn density is 3.8 and 2.2 /10mm, respectively. Static tensile strength of this specimen was 60 MPa.

Tensile specimens of 180 mm (UD) or 150 mm (Textile) × 20 mm in nominal dimensions, with the fiber axis along the loading direction, were used for this study. Specimens were clamped over an area of 30 mm × 20 mm at each end leaving a gauge length of 120 mm. Aluminum tabs were glued on the clamped area. Stress ratio, which is the ratio of minimum to maximum stress during tests, and frequency were selected as 0.1 and 5Hz. Sinusoidal wave form was also selected. Fatigue tests were conducted up to
1,000,000 cycles. In order to evaluate microscopic damages, acoustic emission (AE) was monitored using a AE sensor (PICO, Physical Acoustics) glued on the surface of specimen and an AE tester (AE9501A, NF). Threshold level was 50 μV.

During tests, load and displacement were measured to obtain stiffness reduction. Stiffness, $k$, was calculated as,

$$k = \frac{\Delta F}{\Delta \delta}$$

where, $\Delta F$ and $\Delta \delta$ are amplitude of load and displacement, respectively. In the present study, normalized stiffness $\Delta k(i)$ was used to investigate microscopic damage during fatigue loading.

$$k_n(i) = \frac{k(i)}{k(1)}$$

where $i = N/N_f$, $N$ is number of cycle and $N_f$ is life at the stress level, $k(i)$ is stiffness at $i$. In case of no failure up to 1,000,000, $N_f$ is set to 1,000,000.

3. Results and Discussion

Figure 2 shows a relation between number of cycles to failure and maximum stress level. Fatigue life decreased with increasing stress level. At maximum stress level of 80 MPa, no UD specimens failed until 1,000,000 cycles. Textile specimens do not failed at maximum stress level of 10 MPa. In order to discuss the effect of stress level compared to static strength, maximum stress ratio is defined as maximum stress over static tensile strength. Figure 3 shows the relation between maximum stress ratio and cycle to failure. At the same maximum stress ratio, cycle to failure is larger for UD composites. In addition, the slop for Textile specimen was steeper than that for UD specimen. These indicate damage initiation and progress were severer in Textile composites.

![Fabrication of micro-braided yarn](image1.png)

(a) Fabrication of Micro-braided Yarn

![Micro-braided Yarn](image2.png)

(b) Micro-braided Yarn

Figure 1 Fabrication of micro-braided yarn.

Figure 2 Relation between cycle to failure and maximum stress.

![Relation between cycle to failure and maximum stress](image3.png)

Figure 2 Relation between cycle to failure and maximum stress.
Figure 3 Relation between cycle to failure and maximum stress ratio.

Figure 4 shows normalized stiffness as a function of normalized number of cycles. For UD composites, gradual and continuous stiffness reduction was observed at stress level of 150 and 120 MPa, whereas sudden decrease was detected at stress level of 100 MPa. At lower stress level, such as 100MPa, microscopic damage initiated before final fracture might grow slowly comparing with higher stress level of 120 and 150 MPa. At higher stress level, once microscopic damage initiated, it grew rapidly to final fracture.

For Textile composites, stiffness degradation was severer than for UD composites, which also indicated the microscopic damage accumulation was significant in Textile composites. Stiffness degradation was larger with maximum stress level. At lower stress level, such as 20 MPa, sudden decrease in stiffness was measured just before final fracture. This is due to mild microscopic damage progress at lower stress level similar to UD composites.

Figure 4 Relation between normalized stiffness and normalized cycles.

Figures 5 and 6 show appearances of UD and Textile composites under fatigue loading, respectively. Unlike the case of CFRP or GFRP, macroscopic views of UD and Textile composites after fracture are independent from maximum stress levels. Thus, fracture process was not affected by maximum stress level. This result is consistent with Fig. 4 especially for Textile, where stiffness reductions were almost the same at final fracture irrespective of maximum stress level. In UD composites, no macroscopic damage was observed during the whole of fatigue loading other than maximum stress 100MPa and splittings initiated just before final fracture. Then, fiber fracture followed and specimens failed in a catastrophic manner. At maximum stress 100MPa, splitting initiated at $N/N_f =$
0.92, which corresponded to larger stiffness decrease. In Textile composites, whitening of matrix rich region at the surface of specimen was observed with increasing number of cycles. This might correspond to matrix cracking and debonding between fiber yarn and matrix. With the accumulation of those damages, stress in the warp fiber yarn became larger, which damaged slivers in warp fiber yarn and yarns themselves. Finally, warp fiber yarn failed. Comparing to UD composites, fracture process was stable in Textile composites, which was consistent with modulus reduction shown in Fig. 4.

Figure 5 Appearance of UD composite under fatigue loading (Maximum stress 150 MPa).

(a) N/Nf=0          (b) N/Nf=0.07         (c) N/Nf=0.98          (d) N/Nf=1.00

Figure 6 Appearance of Textile composite under fatigue loading (Maximum stress 40 MPa).

(a) N/Nf=0          (b) N/Nf=0.24         (c) N/Nf=0.62         (d) N/Nf=0.97

Figure 7 shows the relation between normalized stiffness and AE count rate measured during fatigue tests. Maximum stress levels are 150MPa for UD and 40MPa for Textile, respectively. AE count rate remained lower level at the early stage of fatigue loading and suddenly increased just before the fracture. On the other hand, larger AE count rate was generated in Textile composite during the whole of fatigue loading and AE count rate increased at the final stage of loading. Increasing in AE count rate at final stage of loading in both composites might correspond to severe damage initiation such as breakage of fiber yarns. These results were consistent with macroscopic observation results.

Figure 7 Normalized stiffness and AE count rate as a function of normalized number of cycles.
Figure 8 shows the relation between stress and nominal displacement during fatigue loading. Hysteresis in stress displacement kept constant in UD composites, while it became larger with increasing number of cycles in Textile composites. In general, hysteresis in stress and displacement corresponds to internal friction of materials. In Textile composites, a lot of damages occurred during the whole of fatigue loading, as shown in Fig. 6. Friction between damage surfaces dissipated energy which result in larger hysteresis. These results were also consistent with the result of AE measurement, especially at the early stage of the loading.

Hysteresis of UD composites remained the same until final fracture. This means that microscopic damages do not affect the displacement especially in UD composites. Considering the results, AE measurement is more appropriate for health monitoring of hemp fiber composite structures.

4. Conclusion

In order to attain sustainable society, continuous hemp fiber reinforced PLA composites were fabricated using micro-braided yarn to replace oil-based fiber composites. Fatigue behaviour of Textile composites as well as UD composites were evaluated from the view point of actual usage. For UD composites, no microscopic damage was observed and final fracture occurred in a catastrophic manner. On the other hand, matrix cracks and matrix/fiber yarn debondings grew stably in Textile composites. These fracture manners were also confirmed stiffness reduction during cyclic loading, acoustic emission measurement and hysteresis measurement in stress-displacement curves. It is clarified that, acoustic measurement is more appropriate for damage monitoring than hysteresis measurement.

References