Effect of addition of fine sub-micron sized and thick glass fibers into epoxy matrix on fatigue life of plain-woven textile carbon fiber composite

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Abstract. This study investigated the mechanism of the effect of addition of fine sub-micron sized (close to nano-sized) and thick (11µm) glass fibers into epoxy matrix on the fatigue life of plain-woven textile carbon fiber composite. Static bending strength and fatigue life of the coupon shaped composite with the modified matrix were investigated by three-point static and cyclic loading test. The resistance of interlaminar delamination was characterized with double cantilever beam of Mode-I fracture test and ENF (end notch flexure) of Mode-II fracture test. The effect of the size of the added glass fibers on those mechanical properties was discussed. To explain the effect of the modification on the observed interfacial strength around the intertwining point of the reinforcing textile cloth, model specimens were prepared where an embedded carbon fiber bundle was put between other two carbon fiber bundles oriented to the perpendicular direction in the modified resin. Test results in current study showed that the addition of glass fibers with fine size was rather effective to dramatically improve the fatigue life. The fatigue life was dramatically increased 10-100 times by the addition of nano-sized glass fibers into the matrix in contrast to the static case. Fracture toughness in Mode-II was also improved by the modification of the matrix, while that in Mode-I was improved slightly. Macroscopic crack propagation in fatigue was totally prevented when the matrix was modified with fine glass fibers of 500nm in diameter. Those effects in improvement of mechanical properties of the textile carbon fiber composite were explained by the prevention of interfacial crack propagation with large resistance between carbon fiber bundles in Mode-II and modified matrix around the texture points where comparatively large strain energy would be released, if the epoxy matrix was adequately modified with the fine glass fibers.

1. Introduction

Textile carbon fiber composites with polymer matrix have superior advantages for many applications with attracting attentions ([1]). Generally, the interlaminar fracture toughness of the material with a layered structure is poor and cracks between the layers easily lead to fatal result. The interlaminar fracture toughness and interfacial debonding are important considerations at discussing the long-term reliability of the textile composites. Studies have been conducted to investigate the interlaminar fracture toughness and crack propagation of the composites ([2]-[9]). To improve the fatigue life and mechanical properties of the composites, various studies, such as the modification with micro and nano-sized fibers have also been conducted. It has been experimentally shown that the fatigue life and mechanical propirieted of textile composite is improved by the addition of tiny fibers into the matrix ([10]-[14]). Although the effect of added fibers on fatigue life of the textile composite may depend on the diameter of the added fibers, the effect of the size on the fatigue life of the composite is not well be cleared. In
addition, the behavior of crack propagation around carbon fiber bundle under Mode-I and Mode-II condition should be discussed to establish the efficacy of these fibers on the fatigue life of the composite. This study investigated the mechanism of the effect of addition of fine sub-micron sized (close to nano-sized) and thick (11µm) glass fibers into epoxy matrix on the fatigue life of plain-woven textile carbon fiber composite. Static bending strength and fatigue life of the coupon shaped composite with the modified matrix were investigated by three-point static and cyclic loading test. The resistance of interlaminar delamination was characterized with double cantilever beam of Mode-I fracture test and ENF (end notch flexure) of Mode-II fracture test. The effect of the size of the added glass fibers on those mechanical properties was discussed. To explain the effect of the modification on the observed interfacial strength around the intertwining point of the reinforcing textile cloth, model specimens were prepared where an embedded carbon fiber bundle was put between other two carbon fiber bundles oriented to the perpendicular direction in the modified resin.

2. Materials and testing machine

2.1 Materials
Plain-woven carbon fiber cloth with 3GPa of tensile strength and 240GPa of Young’s modulus (Pyrofil TR-3110-MS: Mitsubishi Chemical Co., Ltd., Japan) was used as reinforcement. Epoxy resin (Epikote 828: Mitsubishi Chemical Corporation, Japan) and modified aliphatic polyamines (JER Cure 113: Mitsubishi Chemical Corporation), were used as matrix and curing agent, respectively. To enhance the matrix, in this study, conventional glass fibers with 11µm (EFH150-01: Central Glass Co., Ltd., Japan) (Figure 1) and thin 500 nm (FM1700: Nippon Muki Co., Ltd.) (Figure 2) in diameters were utilized as enhancing materials, respectively. The length of glass fiber with 11µm in diameter was 150µm and with 500 nm in diameter was 200~2,000µm. The weight fraction of the glass fibers was 0.2 wt%.

2.2 Fabrication of composite plate
The composite plate was obtained after laminating with 8 cloths by conventional hand lay-up method and being cured at 80 degree-C for 1 hour and then at 150 degree-C for 3 hours under 5MPa on heating press machine (HB200HB: KAWANAKA SANGYO CO., LTD., Japan). Then heat was radiated slowly for 4 hours. The volume fraction of the fibers was set to 60%. After curing, the composite plate was cut into the dimension of specimen.

3. Test procedure
3.1 Static three point bending test and cyclic three point bending test
Static three point bending tests and cyclic three point bending tests were conducted to investigate the effect of the addition of two different sized glass fibers on the bending strength and fatigue life. The dimension of the composite specimen for measuring bending strength and fatigue life was 100×15×2mm (Figure 3). The three point bending strength of the specimen was determined using a universal testing machine (Servo-pulser EHF-EA5: SHIMADZU CORPORATION, Japan) under displacement controlled condition according to ASTM D790. The span length was 80mm and testing speed was 5.0mm/min. Test data were obtained for five runs under the same test conditions. The fatigue lives of the composite specimens were also measured using an electro hydraulic material testing machine.
under load controlled conditions at 2Hz of frequency and 0.1 of stress ratio under 80% of maximum cyclic loading to the ultimate strength. The wave profile of cyclic loading was set to sinusoidal wave, and gage length was 80mm.

![Figure 3. Specimen for static and fatigue bending test.](image1)

![Figure 4. Specimen for DCB test.](image2)

3.2 Mode-I interlaminar fracture toughness test
The double cantilever beam (DCB) Mode-I fracture specimens were employed to characterize the resistance of interlaminar delamination according to ASTM D5528-01. The dimension of the DCB specimen was $162 \times 25 \times 2\text{mm}$ (Figure 4). The initial crack was introduced on interlayer of the DCB specimen by inserting a Teflon-sheet. Aluminium End-Blocks with the same width as the specimen were attached at the end of specimens by an epoxy adhesive to allow load application. The compact table-top universal tester (EZ test, load rating 500N: SHIMADZU CORPORATION) was used under displacement controlled test. The Mode-I interlaminar fracture toughness $G_{IC}$ was calculated by the following equation.

$$G_I = \frac{3}{2(2H)} \left( \frac{P}{B} \right)^2 \left( \frac{B \lambda}{2} \right)^{3/2}$$

where $G_{IC}=G_I$ at critical state, $P$, $\lambda$, $B$, $2H$, and $\alpha_1$ denote the Mode-I interlaminar fracture toughness, the critical load, the compliance corresponding to crack length, the specimen width, the thickness in eqation, and $\alpha$ the end-block correction factor, respectively.

3.3 Mode-II interlaminar fracture toughness test
End-Notched Flexure (ENF) tests were applied to determine the Mode-II interlaminar fracture toughness of the specimens. The dimensions of the ENF specimen were $45 \times 15 \times 2\text{mm}$ (Figure 5). Pre-crack was introduced by inserting a Kapton film of approximately with 30µm in thickness (Kapton, Du Pont-Toray Corporation, Japan). The Mode II inter-lamina fracture toughness was calculate by

$$G_{IIc} = \frac{9a_2^2F_c^2C_1}{2B(2L^3 + 3a_2^3)}$$

$$\alpha_1 = \left[ \frac{C_1}{C_0} a_3^3 + \frac{2}{3} \left( \frac{C_1}{C_0} - 1 \right) L \right]^{1/3}$$

where, $a_0$, $F_c$, $C_0$, $C_1$, $L$ and $B$ denote initial crack length, critical load, load point compliance at initial elastic part, load point compliance at critical load, span length and specimen width, respectively.

3.4 Measuring of crack propagation under fatigue loading
Notched coupon specimen was also prepared (Figure 6). Pre-crack with 2mm was initiated on the side surface of specimen by a razor after conventional mechanical cutting. In this test, cyclic simple tensile
Load was nominal applied to the specimen, where cyclic applied nominal load, cyclic stress ratio and loading frequency were 13kN, 0.1 and 5Hz, respectively.

![Figure 5. Specimen for ENF test.](image)

![Figure 6. Notched specimen for measuring macro crack propagation.](image)

3.5 Estimating interfacial strength and frictional force around texture point.

Model specimen was originally prepared to estimate the interfacial strength and frictional force around texture point. One longitudinal carbon fiber bundle was passed between two transverse carbon fiber bundles which were all embedded into the unmodified and modified epoxy resin, respectively (Figure 7). Tensile force was applied to the longitudinal fiber bundle while the part of epoxy resin was hooked at an exit of tiny hole to fix the displacement.

![Figure 7. Model specimen and its testing machine.](image)

4. Result and Discussion

4.1 Static strength and fatigue life

Figure 8 shows the test results of static three point bending strength of plain-woven fiber composite modified with nano-sized glass fibers with 11µm and 500nm in diameter. The static three point bending strength was about increased 17.8-29.9% by the addition of glass fibers into the matrix. These result shows that the static bending strength of composite was slightly increased with a reduction in the diameter of additional glass fibers. Figure 9 shows the change of the bending fatigue lives defined as the number of cycles to failure of the composite specimen subjected to 80% of cyclic maximum stress of static strength. The fatigue life was dramatically increased 10-100 times by the addition of nano-sized glass fibers into the matrix in contrast to the static case. After the cyclic three point bending test, the state of the side surface of the specimen was observed by microscope. Figure 10 shows an example of the damage state of the specimen after failure. It was shown that the fatigue crack of composite was grown along the carbon fiber bundle under cyclic bending loading.
4.2 Mode-I interlaminar fracture toughness

Figure 11 shows the change of Mode-I interlaminar fracture toughness by the modification proposed in this study. When composite was modified with additional glass fibers with 11 \( \mu \)m in diameter, the Mode-I internal fracture toughness (\( G_{IC} \)) was increased about 26\%. The \( G_{IC} \) was also increased about 21\%, when the composite was modified with glass fibers of 500nm in diameter. These result showed that the \( G_{IC} \) was basically increased due to addition of micro and nano-sized glass fibers into the matrix. However, the improvement in \( G_{IC} \) was comparatively slight with that in Mode-II shown in next session.
4.3 Mode II inter-lamina fracture toughness
Figure 12 shows the change of Mode-II interlaminar fracture toughness by the modification. The Mode-II fracture toughness between the carbon fiber bundle and epoxy matrix was significantly improved about 4-6 times due to the addition of micro or nano-sized glass fibers compared with that of unmodified samples. The Mode-II fracture toughness of the specimen modified with fine glass fibers of 500nm in diameter was about 1.5 times higher than modified with 11µm in diameter. The fracture toughness in Mode-II was remarkably improved by the modification of the matrix, while that in Mode-I was improved slightly.

4.4 Macroscopic crack propagation under fatigue loading
Figure 13 shows the measurement result of the macroscopic crack length of the composite specimens with unmodified and modified matrix. Macroscopic crack propagation in fatigue was totally prevented when the matrix was modified with fine glass fibers of 500nm in diameter, while relatively high speed crack propagation was shown in the unmodified case especially in early stage of fatigue. This means that remarkable damage was prevented by the modification with fine glass fibers.

4.5 Observed interfacial strength and frictional force around texture point.
Figure 14 shows an example test result showing the loading force applied to longitudinal carbon fiber bundle passing between two transverse carbon fiber bundles, as explained above. Loading force was linearly increased with the displacement of loading point, before a sharp peak of maximum, and then showed an almost plateau response with pulling behavior. These means the observed shear strength
between carbon fiber bundle and matrix, and observed frictional force around the crossing point, respectively. Figure 15 shows the change of observed shear strength and frictional force by the modification. The observed frictional force was improved about 2.5 times due to the addition of fine glass fibers compared with that of unmodified samples, while the improved ratio was 28% in observed shear strength. Figure 16 shows the comparison of states of fracture and sliding surfaces of the tested longitudinal carbon fiber bundles embedded in the model specimen. Lots of particles of the resin were remained, if the matrix was modified with fine glass fibers. It was suggested that those effects in improvement of mechanical properties of the textile carbon fiber composite were explained by the prevention of interfacial crack propagation with large resistance between carbon fiber bundles in Mode-II and modified matrix around the texture points where comparatively large strain energy would be released, if the epoxy matrix was adequately modified with the fine glass fibers.

![Figure 15. Change of observed shear strength and frictional force](image1.png)

![Figure 16. Comparison of states of fracture and sliding surfaces of tested longitudinal carbon fiber bundles embedded in model specimen.](image2.png)

5. Conclusion
5.1 Test results in current study showed that the addition of glass fibers with fine size was rather effective to dramatically improve the fatigue life. The fatigue life was dramatically increased 10-100 times by the addition of nano-sized glass fibers into the matrix in contrast to the static case.
5.2 Fracture toughness in Mode-II was also improved by the modification of the matrix, while that in Mode-I was improved slightly. The Mode-II fracture toughness between the carbon fiber bundle and epoxy matrix was significantly improved about 4-6 times due to the addition of micro or nano-sized glass fibers compared with that of unmodified samples. The fracture toughness in Mode-II was remarkably improved by the modification of the matrix, while that in Mode-I was improved slightly.
5.3 Macroscopic crack propagation in fatigue was totally prevented when the matrix was modified with fine glass fibers of 500nm in diameter
5.4 The observed frictional force of one longitudinal carbon fiber bundle passed between two transverse carbon fiber bundles embedded into the modified epoxy resin was improved about 2.5 times due to the
addition of fine glass fibers compared with that of unmodified samples, while the improved ratio was 28% in observed shear strength.

5.5 Those effects in improvement of mechanical properties of the textile carbon fiber composite were explained by the prevention of interfacial crack propagation with large resistance between carbon fiber bundles in Mode-II and modified matrix around the texture points where comparatively large strain energy would be released, if the epoxy matrix was adequately modified with the fine glass fibers.

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