

EFFECTS OF FIBER SURFACE TREATMENT ON THE FIBER BREAK BEHAVIOR IN SINGLE FIBER COMPOSITE

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SUMMARY: A single fiber fragmentation test is performed to investigate the effects of fiber surface treatment on fiber break behavior. Single glass fiber/epoxy composites with five different surface treatments are loaded in tension in the fiber direction. In-situ observation of the fiber fragmentation process is conducted by using an optical microscope equipped with a loading device. Fiber break density is measured as a function of applied strain. The microfailure around fiber breaks is also observed. It is seen that the fiber break behavior and microdamage are different with the fiber surface treatments. To interpret the experimental data, the authors' analysis of the stress transfer between the fiber and matrix in a single fiber reinforced composite with fiber breaks is used. The analysis is based on a concentric cylinder model considering an interphase layer between the fiber and matrix. By using the stress transfer analysis, fiber break density as a function of strain is predicted. By comparing between the experimental results and the analytical predictions, the validity of the analytical prediction is discussed.

KEYWORDS: single fiber composite, fiber surface treatment, interfacial strength, stress transfer, interphase, fiber break

INTRODUCTION

It is well known that effects of the properties of the fiber/matrix interface on the mechanical properties of fiber reinforced composites is very significant. In order to develop high performance composite materials, it is very important to understand the relation between the interface properties and composite macroscale properties. In this point of view, many methods to evaluate the interface properties are introduced such as the fragmentation test, the fiber pull-out test, the indentation test and microdroplet test. Especially, the fragmentation test is widely used [1-3] to estimate the fiber/matrix interfacial shear strength.

The authors have been investigating the relation between the mechanical properties of single fiber composites (model composites) and actual composites [4-8]. The relation between the interface properties obtained by the single fiber composites and the macroscopic properties (strength and toughness) of actual composites have been discussed. Two material systems, glass/nylon6 and glass/epoxy, have been investigated experimentally.

Recently, McCartney [9] developed a method to analyze the stress transfer between the fiber and matrix in a single fiber composite with fiber breaks or matrix cracks. It is a two-dimensional approximate elastic analysis which considers the mismatches of thermal expansion coefficients and Poisson's ratios of the fiber and matrix. In the analysis, stress, strain and corresponding displacement fields are obtained.

The authors have extended McCartney's analysis to consider the interphase layer between the fiber and matrix [10]. Stress, strain and displacement fields in a single fiber composite with fiber breaks are analyzed. Young's modulus reduction in a single fiber composite due to fiber breaking is also analyzed.

In the present study, a single fiber fragmentation test is performed to investigate the effects of fiber surface treatment on fiber break behavior. Single glass fiber/epoxy composites with five different surface treatments are loaded in tension in the fiber direction. In-situ observation of the fiber fragmentation process is conducted by using an optical microscope equipped with a loading device. Fiber break density is measured as a function of applied strain. The microfailure around fiber breaks is also observed. To interpret the experimental data, the authors' analysis of the stress transfer between the fiber and matrix in a single fiber reinforced composite with fiber breaks is used. By using the stress transfer analysis, fiber break density as a function of strain is predicted. By comparing between the experimental results and the analytical predictions, the validity of the analytical prediction is discussed.

EXPERIMENT

Materials

Five types of fibers with different surface treatments as shown in Table 1 are used. The glass fibers are E-glass fibers whose diameter is 13 μ m. The five surface treatments are called Untreated (not treated), Methacryl-silane (treated only with γ -methacryloxypropyltrimethoxysilane), Film former (treated only with urethane film former), Epoxy-silane (treated only with γ -glycidoxypropyltrimethoxysilane) and Mixture (treated with γ -aminopropyltrimethoxysilane, urethane film former and paraffin lubricant). Bisphenol-A type epoxy resin (Epokote 828) and triethylenetetramine (TETA) as hardener are used in 100:11 weight ratio as matrix resin. The resin is cured at 50 $^{\circ}$ C for 60 min and post cured at 100 $^{\circ}$ C for 80 min.

Table 1. Fiber surface treatments

	Coupling Agent			Film former	Lubricant
	Amino-silane	Epoxy-silane	Methacryl-silane	Urethane	Paraffin
Untreated	-	-	-	-	-
Methacryl-silane	-	-	o	-	-
Film former	-	-	-	o	-
Epoxy-silane	-	o	-	-	-
Mixture	o	-	-	o	o

o:used - :not used

A single glass fiber is extracted randomly from the roving and mounted on a Teflon mold. The resin mixed with hardener is poured into the mold to obtain a single fiber reinforced epoxy composite specimen. Specimen size is shown in Fig.1.

Figure 1 Single fiber composite specimen

Fragmentation test

The single fiber composites are loaded in tension in fiber direction. In-situ observation of damage process is conducted with an optical microscope equipped with a loading device. Fiber break density is measured as a function of applied strain. The damage process around fiber breaks is also observed. Figure 2 shows the schematic illustration of the in-situ observation system.

Figure 2 Schematic illustration of the system for in-situ observation of fragmentation process in single fiber composites

ANALYSIS

The authors [10] conducted a two-dimensional stress transfer analysis of a single fiber composite with fiber breaks based on McCartney's analysis [9]. We extended McCartney's analysis to consider the effect of interphase layer between the fiber and matrix. Consider a concentric cylinder model as shown in Fig.3. The single fiber composite is subjected to tensile stress σ in z direction. It is assumed that equally spaced parallel array of fiber breaks occur and that the fiber break crack surface is perpendicular to the fiber axis. It is also assumed that the fiber break crack edge lies on the fiber/interphase interface. By using the analysis, stress, strain and displacement fields in the fiber, matrix and interphase are obtained.

Figure 3 Schematic of model for analysis

By applying the stress criterion whose assumption is that new fiber breaks occur at the midway between the existing fiber breaks when the tensile stress in fiber axial direction

reaches a critical value, the relation between the composite strain ε and fiber break spacing L becomes

$$(1)$$

where σ_c is the critical stress, E , ν , α , V are Young's modulus, Poisson's ratio, thermal expansion coefficient and volume fraction, respectively, and $T=(\text{testing temperature})-(\text{curing temperature})$. Subscripts f and m denote fiber and matrix, respectively. The details of the parameter λ and the function $S(L)$ are expressed in elsewhere [11]. In the present study, the effect of the statistical nature of fiber strength is considered by assuming that the fiber strength distribution obeys Weibull distribution.

RESULTS AND DISCUSSION

It is found that the fiber break behavior is different with different fiber surface treatment. Figure 4 shows fiber break density as a function of applied strain for single fiber composites with five different fiber surface treatments. Fiber break density is defined as the number of fiber breaks per unit fiber length (/cm). It is seen that both the fiber break onset strain and fiber break multiplication behavior are different with fiber surface treatments. This is due to the difference in the fiber strength and stress transfer between the fiber and matrix.

Figure 4 Fiber break density as a function of applied strain in single fiber composites with five different fiber surface treatments

In the present study, microfailure behavior around fiber breaks is also observed. Three types of microdamages are observed. Figure 5 shows schematic of the microdamages observed. Type A involves a circular matrix crack and fiber/matrix interfacial debonding whose tip penetrates into matrix like a cone. In Type B, a circular matrix crack and debonding are observed. In Type C, only debonding is observed.

In Epoxy-silane specimens, Types A and B are observed. In Untreated and Methacryl-silane specimens, Types B and C are observed. In Film former and Mixture specimens, only Type C is observed. It is expected that the fiber/matrix interfacial strength is relatively higher in specimens with Types A and B microdamages than those with Type C microdamage.

Figure 6 shows comparisons between the analytical predictions and the experimental results of fiber break behavior as a function of applied strain. The material properties used in the prediction are listed in Table 2. The outer diameter of the interphase layer is assumed to be 14 μ m. Interphase Young's modulus and the fiber strength are determined to fit the experimental data. In the predictions assuming the statistical fiber strength, the shape parameter of Weibull distribution is assumed to be 6. The scale parameter for the fiber length of 14mm is determined to fit the experimental results. The parameters determined in the prediction are listed in Table 3. In Fig.6, the solid lines show the prediction using the statistical fiber strength and the dotted lines show the predictions using the constant fiber strength. It is seen that by selecting the proper Interphase Young's modulus and the fiber strength, the fiber fragmentation behavior can be predicted by using the present analysis. This implies that the analysis can be applied to a method for evaluation of the interface properties using fragmentation results.

Figure 5 Schematic of microfailure around a fiber break

Table 2 Material properties used in the analysis

	Young's modulus (GPa)	Poisson's ratio	Thermal expansion coefficient ($\times 10^{-6}/^{\circ}\text{C}$)
Glass fiber	73.5	0.25	5.0
Matrix resin	3.66	0.35	60
Interphase	E_i	0.35	60

Table 3 Parameters used in the prediction of fiber break density

	Interphase Young's Modulus (GPa)	Fiber strength (constant fiber strength model) σ_c (GPa)	Fiber strength (statistical fiber strength model) σ_L (GPa) ($L=14\text{mm}$)
Untreated	1.82×10^{-1}	2.20	1.35
Epoxy-silane	3.64	2.70	1.60
Methacryl-silane	1.82×10^{-1}	2.30	1.40
Film former	0.92×10^{-2}	3.40	2.30
Mixture	1.82×10^{-2}	2.90	1.80

Figure 6 Comparison between analytical predictions and experimental results of the fiber break density as a function of applied strain

CONCLUSIONS

A single fiber fragmentation test is performed to investigate the effects of fiber surface treatment on fiber break behavior. Single glass fiber/epoxy composites with five different surface treatments are loaded in tension in the fiber direction. In-situ observation of the fiber fragmentation process is conducted by using an optical microscope equipped with a loading device. Fiber break density is measured as a function of applied strain. The microfailure around fiber breaks is also observed. Differences in the fiber break behavior and microdamage are different with the fiber surface treatments are clarified experimentally. To interpret the experimental data, the authors' analysis of the stress transfer between the fiber and matrix in a single fiber reinforced composite with fiber breaks is used to predict the fiber break density as a function of strain is predicted. It is found the fiber break behavior is well predicted by selecting proper value of interphase Young's modulus and the fiber strength. The present analysis will be useful to evaluate the interface properties using fragmentation results.

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