

EXPERIMENTAL AND ANALYTICAL CHARACTERIZATION OF SILICONCABIDE FIBER-REINFORCED PLASTICS

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Abstract

In order to characterize the fiber/matrix interfacial shear strength and the effect of them on the tensile strength of the unidirectional composite, microbond tests and tensile tests were conducted on silicon carbide (SiC) fiber reinforced bismaleimide and epoxy composites. The interfacial shear strength is larger in bismaleimide composites. However, no difference in the tensile strength was confirmed between bismaleimide and epoxy.

Monte Carlo simulation considering the change in the stress concentration along fiber direction. Size scaling was conducted to compare the simulation results with experiments. Simulation results are much larger than the experiments.

1 Introduction

The effect of fiber/ matrix interfacial properties on the mechanical properties of composite materials is significant and it is important to understand the relation between interfacial properties and mechanical properties of composite materials.

There have been many studies on analytical prediction of the unidirectional composite strength [1-3]. Rosen developed a model using both the chain-of-bundles and the equal load sharing (ELS) concepts [1]. Zweben modified the model considering the local load sharing (LLS) concept [2]. Batdorf proposed a model which did not use the chain-of-bundles concept [3]. Recently, many attempts have been made to clarify the characteristics of statistical composite strength using the Monte Carlo simulation [4, 5]. In many approaches of Monte Carlo simulation for composite strength, the unidirectional composites are regarded as a chain of links where the fiber strength and fiber stresses are constant in the fiber direction. In these approaches, the resulting fracture surface of composites is considered to be flat and the distribution of the fiber pull-out cannot be discussed.

In the present study, the interfacial properties of siliconcarbide fiber reinforced plastics are investigated using microbond tests. Two types of thermosetting resins, such as bismaleimide and epoxy are used. The tensile tests on the unidirectional composite is also performed to investigate the effect of matrix resin on the composite strength. In order to discuss the experimental results, a Monte Carlo simulation considering the stress concentration in the fiber direction [6] is also conducted.

2 Experimental Procedure

2.1 Single Fiber Tensile Tests

Siliconcarbide fiber used in the present study is NICALON (Nippon Carbon). Since the strength distributions of ceramic fibers were varied by the introduction of initial flaw introduced in the manufacturing process, two lots of fibers manufactured at the different times were used. Single fiber tensile tests were conducted based on the Japan Industrial Standard (JIS) R7501. Gage length and tensile speed are 25 mm and 0.1mm/min, respectively. The fiber diameter of each specimen was measured with a video microscope.

2.2 Microbond Tests

In this study, the interfacial shear strength of fiber/matrix is evaluated by microbond tests. The schematic illustration of the microbond tests was shown in Fig. 1. Interfacial shear strength, τ , is calculated as,

$$\tau = \frac{F}{\pi d_f l} \tag{1}$$

where d_f is fiber diameter, l is embedded fiber length and F is the maximum load.

Fibers used were NICALON, as mentioned above. A single fiber was bonded to the specimen holder without a slack. Two kinds of matrix resins, such as bismaleimide (BMI) (5250-4, Cytec) and bisphenol-A type epoxy resin (Epikote 828) were used. Bismaleimide resin was supplied in the solid state. The resins were dry ground and kept in the oven at 125°C for 10 min. Then the resin droplet was bonded to the fiber with a needle. BMI resin was cured at 177°C for 360 min and at 210°C for 360°C. The hardener for the epoxy resin used was triethylenetetramine in 100:11 weight ratios. The blend of epoxy resin and hardener was mixed and the mixture was vacuum-deformed for 10 min. Then the droplet was bonded in the same way with BMI. Epoxy resin was cured at 50°C for 60min. Microbond tests were conducted at 0.125mm/min.



Figure 1 Schematic view of a microbond test.

2.3 Unidirectional Composite Tensile Tests

The specimens for unidirectional composites tensile tests is fabricated with a bundle of 500 NICALON fibers and the same BMI and epoxy resin as microbond tests. The bundles were dipped in the resin and they were evacuated for 10 min. Then the bundle was pulled up and went through the PTFE die with a hole (0.75mm) to control the shape of a cross-section and to remove the excess resin. The curing conditions for both resins were same to the microbond tests. The specimen geometry is shown in Fig. 2. The aluminum tabs were glued on the end of the specimen. The gage length of the specimen was 100mm. The tensile tests were conducted at a cross-head speed of 1mm/min.



Figure 3 Schematic view of unidirectional composite specimen.

3 Experimental Results

The cumulative fracture probability of the fiber is shown the Weibull-type form as,

$$F(\sigma) = 1 - \exp\left\{-\frac{L}{L_0}\left(\frac{\sigma}{\sigma_0}\right)^p\right\}$$
(2)

where ρ and σ_0 are the shape and scale parameters, respectively. In the present study, the gage length L_0 is 25mm.

Figure 3 shows the Weibull plot of single fiber tensile strength from lot A and B. Weibull parameters obtained from Fig. 3 are shown in Table 1. There is no difference between lot A and lot B. So only the fibers from lot A was used in the following tests.



Figure 3 Weibull plots of single fiber strength.

Table 1 Weibull parameter of the SiC fibers.

	Lot A	Lot B
Shape Parameter	4.83	4.91
Scale Parameter (GPa)	3.02	2.92
Mean Strength (GPa)	2.77	2.79

Figure 4 shows the relation between the embedded length, l, and the maximum load, F. Solid line is the result of the least-square method. Large scattering are observed for both material systems. This is due to the fracture of droplet by the contact stress at the knife edges. Table 2 shows the interfacial shear strength obtained from Fig. 4. Interfacial shear strength is larger with BMI resin.

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Figure 4 Relation between maximum load and embedded length.

The Weibull plot of the strength obtained from the unidirectional composite tensile tests were shown in Fig. 5. The Weibull parameters obtained were shown in Table 3. No differences in the bismaleimide and epoxy composites were observed, which indicated the no effect of the fiber/matrix interfacial strength on the composite strength. On the other hand, fiber pull-out length in epoxy composites was larger than in bismaleimide composites, as shown in Fig. 5. These results agree with the tendency obtained in the microbond tests. From these results, the fracture process of the unidirectional composites is expected as follow. Multiple fiber fractures occurred the and fiber/matrix interfacial shear stress becomes larger at the vicinity of the fiber fracture. At the initial loading, the shear stress does not reach the interfacial shear strength and fiber/matrix interfacial debondings do not occur. The composite separates with matrix fracture when the arbitrary cross section of the composites cannot sustain the loading. The fiber/matrix interfacial fractures also occur instantaneously. The lower interfacial strength in epoxy composites results in the larger pull-out length. It is very important to evaluate the fiber/matrix interfacial shear strength and the stress distribution around the fiber breakages.



Figure 5 Weibull plots of unidirectional composite strength.

Table 3 Weibull parameter of unidirectional composites.

•	Bismaleimide	Epoxy	
Shape Parameter, p	12.90	13.72	
Scale Parameter, σ_0 (GPa)	0.62	0.63	
Mean Strength (GPa)	0.60	0.61	



(a) Bismaleimide Composite (b) Epoxy Composite

4 Discussion

In order to estimate the strength of unidirectional composite, Monte Carlo simulation [6] was conducted. In this simulation, fiber distributions around a breaking fiber are assumed as hexagonal array, shown in Fig. 6. The unit link length, which is assumed as the maximum pull-out length in the present study, for bismaleimide and epoxy composites are assumed as 0.47 and 0.28 mm, respectively. The composite is assumed to consist of 469 fibers. In the present study, to consider the change in stress concentration due to fiber break

Figure 5 Fracture surfaces observed by scanning electron microscopy.

along fiber direction, the unit link is divided into sublinks. In the simulation, the experimentallyobtained fiber strength distribution is used to assign the fiber strength in each sublink. The stress concentrations around the breaking fibers are calculated with shear-lag analysis [7. 8]. In the shear-lag analysis, the fiber carries the normal stress and matrix carries only shear stress. The assembly of shear-lag model results in a governing differential equation for the fiber displacement, u, as a function of the distance from the break, ξ .

$$\frac{\partial^2 u_{n,m}}{\partial \xi^2} + \left(u_{n+1,m} + u_{n,m+1} + u_{n+1,m-1} + u_{n+1,m} + u_{n-1,m+1} - 6u_{n,m} \right) = 0$$
(3)

$$p_{n,m}(\xi) = 1 + \sum_{i=0}^{r-1} \sum_{j=0}^{r-1} q_{n-i,m-i}(\xi) u_{i,j}(0)$$
(4)

u is displacement, *p* is axial stress, *r* is number of fiber breaks, *q* is influence function and (i, j) is fiber break point. Influence function *q* is equation (5) at $\xi=0$.

$$q_{n,m}(0) = -\frac{1}{4\pi^2} \int_{-\pi}^{\pi} \int_{-\pi}^{\pi} d\theta d\phi \lambda \cos(n\theta + m\phi) \quad (5)$$

$$\lambda = \sqrt{2(3 - \cos\theta - \cos\phi - \cos(\theta - \phi))} \tag{6}$$

Size scaling was conducted on the results of the simulation in order to evaluate the strength of the unidirectional composite with 100mm gage length. The failure probability of unidirectional composite which have gage length of n times sublink length is expressed as

$$W(\sigma) = 1 - \{1 - F(\sigma)\}^n \tag{7}$$

In the present study, 30 simulations were conducted on both composites. Figures 7 show the results of Monte Carlo simulation. The Weibull parameters obtained were shown in Table 4. In this simulation the number of sublink used is 30, because the simulation results converge [6]. Comparing the simulation results with experiments, the simulation results are much larger than the experiments. The unidirectional composites tested have 26.8% of fiber volume fraction. That is, the load capacities of the resin are not neglected in such larger fiber volume fraction. In addition, the resin rich region tends to crack at the early stage of the loading, which result in the fiber breakages. Adequate fiber volume fraction and uniform fiber arrangements must be necessary for the high performance composites.



Figure 6 Shear-lag model of hexagonal array.



(b) Epoxy Composite

Figure 7 Weibull plot of unidirectional composite strength distribution (Simulation and size scaling results.)

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composites.				
	Bismaleimide		Epoxy	
length (mm)	0.46	100	0.28	100
Shape Parameter, p	17.83	10.02	34.60	47.89
Scale Parameter, σ_0 (GPa)	1.577	1.046	1.443	1.263
Mean Strength (GPa)	1.530	0.995	1.420	1.248

Table 3 Weibull parameter of unidirectional composites.

3 Conclusion

Two types of SiC fiber reinforced plastics were characterized experimentally. Microbond tests indicated the interfacial shear strength is larger in bismaleimide composite. On the other hand, the tensile strength of bismaleimide and epoxy composites is almost the same values. Monte Carlo simulation considering the change in the stress concentration along the fiber direction was conducted. The results of the simulation were much higher than experiments, which is due to the difference in fracture process between simulation and experiments. Adequate fiber volume fraction and uniform fiber arrangements must be necessary for the high performance composites.

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