Tensile Characteristics of Silicon Carbide Fiber in Fabrication

Process of FRM by Liquid Infiltration Method *

(Part 1; Strength and Fracture Mode)

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The present paper deals with the tensile strength and fracture mode of SiC fiber exposed to a hydrostatic pressure, isothermal heating and liquid aluminum environment. These conditions are sometimes encountered in FRM fabrication by a liquid infiltration method. The results of the tensile-test showed no fiber degradation in a given isothermal heating and hydrostatic pressurizing, but the fiber exposed to the liquid aluminum environment degraded with an increasing exposure time. And a fractographic observation revealed the following. As for the original fiber, the isothermally heated fiber and the hydrostatically pressurized fiber, the cracking occurred from the inner defect of the fiber or from the surface defects. Most of the fibers failed by the inner defect are stronger than those by the surface defect. On the other hand, for the liquid aluminum exposure, the fibers which the fracture originates from the reaction product formed on the fiber surface with the aluminum increased in number as the exposure time increased, and after 2min exposure all the fibers fractured by the reaction product.

Key Words: Composite Materials, Silicon Carbide Fiber, Tensile Strength, Fractography, Fracture Mode, Bi-modal Weibull Distribution

1. Introduction

There are an increasing number of reports on FRM, in particular, on coreless silicon carbide fiber ⁽¹⁾ reinforced aluminum ⁽²⁾⁻⁽⁷⁾, which has a light weight and a high strength as well as an excellent thermal resistance. It is necessary to make clear the statistical strength and the fracture mechanism of the reinforcing fiber in order to improve the mechanical properties and the reliability of FRM. In general, the fibers are exposed to several environments injured thermally, chemically and mechanically during FRM fabrication process, so the tensile characteristics and the fracture mode of them are changed. There are already some reports on the tensile characteristics of coreless silicon carbide fiber ⁽⁸⁾ and the effect of environmental factor on the fiber ⁽⁶⁾, but few reports on the relation between the fiber strength and the inherent defects.

In the present work, for the purpose of investigating the change in strength of the coreless silicon carbide fiber encountered during the process of FRM fabrication by squeeze casting, the fibers were exposed to

isothermal heating, hydrostatic pressure and liquid aluminum environments, respectively. The exposed fibers and the as-received fibers were tensile-tested, and the fracture surfaces of every tensile-tested fiber were observed by SEM. And the relations between the fiber strength and several types of defects causing the failure were described.

2. Experimental Procedure

2.1 Test materials

Continuous coreless silicon carbide fibers (Trade name : Nicalon, hereinafter referred to as "SiC fiber") supplied by Nippon Carbon Co., Ltd. were employed as test materials. It has mass-productivity and the excellent chemical and mechanical properties. A bundle of the fibers consists of 500 filaments with an organic sizing. In the pre-treatment of experiment, the organic sizing in the bundle was fully removed by heating at 450°C for 30 minutes in air. Hereinafter, the stripped fibers are referred to as "virgin fibers".

2.2 Exposed conditions of fibers to environments

Supposing that reinforcing fiber is exposed to several environments injured thermally, physically and chemically during the process in FRM fabrication by a liquid infiltration method, the following three kinds of exposure conditions are selected.

2.2.1 Effect of isothermal heating at elevated temperatures

In general, the reinforcing fiber is exposed to isothermal heating at elevated temperatures in the pre-process and the

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in-process of the fabrication. In order to investigate independently the thermal effect on the tensile characteristics of SiC fiber, the fibers were isothermal-heated for 1, 10 and 100hs at 700°C, respectively. Hereinafter, the fibers subjected to those exposure conditions are referred to as "heated fiber".

2.2.2 Effect of hydrostatic pressurizing

The reinforcing fiber is often exposed to hydrostatic pressure through a medium of molten metal by squeeze casting process⁽²⁾⁽⁵⁾. Therefore, the fibers were hydrostatically pressurized in oil using a universal pressurizing tester at 49 and 196 MPa, respectively, for 10 min. Hereinafter, the fibers are referred to as "pressurized fiber".

2.2.3 Effect of coating with liquid aluminum

SiC fiber is containly exposed to liquid aluminum environment during fabrication of FRM by squeeze casting. So, the fibers were coated one by one with aluminum by vapour deposition in a vacuum of about 1.33×10^{-3} Pa, after that, the filaments were isothermally heated for 0.5, 2 and 10 min at 700°C, respectively. The aluminum layer was dissolved in the 10% sodium hydroxide solution after the heating. Hereinafter, such fibers are referred to as "Al treated fiber".

2.3 Evaluation of tensile characteristics

Tensile characteristics of the virgin fiber and each exposed fiber were evaluated by the tensile test. The specimen was prepared with a 10 mm gage length on the basis of Japan Industry of Standards R 7601 (Testing methods for carbon fibers), and the fiber diameter was measured using an optical microscope with the micrometer eyepiece. The test was carried out using an Instron-type testing machine (Tensilon, STM-T-50BP, Toyo Baldwin) under 0.2 mm/min in crosshead speed, and the number of test fibers was from 40 to 60 per each exposure condition.

Primary fracture surfaces are often lost because of the fragmentation occurring at the failure, so a fractography technique⁽⁹⁾⁽¹⁰⁾ was used, that is the method of coating the test filament with a jellied dressing pack to prevent the tips from flying away. The pack is so soft that the load caused by a viscosity of the pack is negligible compared with the load necessary to break the fiber itself.

3. Experimental Result

3.1 Strength and diameter distribution of virgin fibers

Figures 1(a) and (b) show histograms of the diameter and the tensile strength of virgin fibers, respectively. As shown in Fig.1(a), the fiber diameter distribution deviates somewhat to the left side from the center. The average diameter and the CV value (coefficient of variation) were 14.1 μm and 13.5%, respectively. This CV value means a considerably large scatter. As seen in Fig.1(b), the strength distribution has a long tail and a narrow half-width, that is

Fig.1. Histograms of fiber diameter and tensile strength of virgin SiC fiber. (a) Diameter. (b) Tensile strength.

Mean :Average value. SD :Standard Deviation. CV :Coefficient of variation.

to say, a large asymmetry to a minus side. The average strength is 3.51GPa, which value is nearly equal level to other ceramic fibers⁽¹¹⁾⁽¹²⁾, but the 23.2% CV value is somewhat large. In this case, a total test number of fibers was 58, and the mean fiber diameter was 14.2 μm .

3.2 Tensile strength of exposed fibers

Figures 2(a), (b) and (c) show the tensile characteristics of the heated, pressurized and Al treated SiC fibers, respectively. In these figures, the open circles denote an average tensile strength, and the solid line's half width does a range of standard deviations. And the open triangles denote CV value. In Figs.2(a) and (b), the mean strengths of heated and pressurized fibers seem to rather increase with an increase in time and pressure, respectively. On the other hand, the CVs showed almost the same level within a variation between 20 and 25%.

As shown in Fig.2(c), however, Al treated fibers decrease in the mean strength and in the CV value with an increase in exposure time. And finally, the strength and the CV value diminish to one-third and two-third, respectively, after the exposure time of 10 min.

3.3 Fracture modes of SiC fiber

Figure 3 illustrates the typical electron-fractographs of the SiC fibers after tensile-testing. Figures 3(a) and (b) show the fracture modes initiated at an inner defect and a surface defect, respectively. Figures 3(c) and (d) show the fracture modes initiated at a reaction

Fig.2. Tensile strength of SiC fibers exposed to each environment.
 (a) Isothermal heating at 700°C. (b) Hydrostatic pressurizing for 10 min.
 (c) Liquid aluminum coating at 700°C.

○:Average strength. △:CV value.
 ●:Average strength for the specified fibers between 13.6 and 14.5 μm in diameter.

Fig.3. SEM photographs of fracture surfaces of SiC fiber.
 (a) Initiated at inner defect. (b) Initiated at surface defect.
 (c) Initiated at reaction product with spotted pattern.
 (d) Initiated at reaction layer.

product formed on the fiber surface. Each fracture initiation is indicated by a white arrow. The product is assumed to occur through the reaction between the fiber and the liquid aluminum. The products in Figs. 3(c) and (d) are characterized by the morphology of a spotted pattern and a layer on the fiber surface, respectively. Two types of fracture modes (a) and (b) were observed in the virgin, heated and pressurized fibers. In the Al treated fibers after 0.5min, all kinds of fracture modes were observed. On the other hand, in the Al treated fibers after 2min, only one type of fracture mode (d) was observed. Here, it can be safely said that the fracture mode of the fiber was changed from either (a) or (b) to (d) by the liquid aluminum environment. This inevitably induced a change of tensile characteristics.

4. Discussion

4.1 Tensile characteristics of virgin SiC fibers

Virgin fibers were observed to exhibit two kinds of fracture mode initiated at either inner defect or surface defect as shown in Fig.3. The inner defects are probably developed in the fabrication process of fiber itself. The surface defects contain also a flaw caused by a secondary factor involved in a handling process of the

mono-filament. Other ceramic fibers, for example carbon fibers, exhibit the same fracture mode⁽¹³⁾⁽¹⁴⁾. So, the strength distribution function of SiC fiber is similarly expected to be bi-modal distribution given by Weibull's link model⁽¹⁵⁾. Table 1 shows a relation between the fracture modes and the strength of SiC fiber. The mean strength of the fibers failed by the inner defect is high and its variation is low. On the other hand, the mean strength of the fibers failed by the surface defect is low and the CV value is considerably high. So, the strength distribution of virgin fibers is assumed to be well described by the mixture of the intrinsic probabilities of each defect based on Weibull's weakest link model. In this case, however, the strength distribution characteristics, as shown in Fig.1(b), are strongly dependent on the surface defect, since the surface defect failed fibers are numerous.

Table 1. Relation between the defect and the strength of virgin SiC fibers.

Defects	N	Mean (GPa)	C.V. (%)	Photo.
Inner defect	12	4.87	9.0	Fig.3(a)
Surface flaw	23	3.33	27.2	Fig.3(b)

N : Number of observed fibers. Mean : Average strength. CV : Coefficient of variation.

4.2 Effect of heated and pressurized treatment on tensile strength of SiC fiber

The mean strength of heated and pressurized fibers showed a little increase as illustrated in Fig.2. SiC fiber was already shown to have a large scatter of diameter (see Fig.1(a)), and it has been reported that the fiber strength decreased as the diameter increased⁽⁶⁾⁽⁸⁾ because of the size effect. After checking the diameter tested, every average diameter under a given exposed condition came between 12 and 13 μm . To discuss the strength under a constant average diameter, fibers which had the specified diameter between 13.6 and 14.5 μm were extracted, and the average strength was replotted with solid circles on Fig. 2(a) and (b). In both cases, the levels are nearly equal to the virgin one, so it is concluded that the fiber has no change in strength after heating and pressurizing.

4.3 Effect of Al treatment on tensile characteristics of SiC fiber

Al treated SiC fibers decreased in the mean strength and the CV value as shown in Fig.2(c). The SEM observations gave, as shown in Table 2, the number of the defects causing a fracture, and the mean strength and CV value due to the defect, respectively.

The Al treatment for 0.5 min of the fiber is recognized to transit the fracture modes from the virgin fiber mode of two defect type to the reaction fiber mode. As shown in the table, the fractured fibers due to the surface defect account for twenty (74%) of all. The defect of Fig.3(c) type is observed only in five (18%), and the mean strength does not decrease so much because the reaction products are assumed to be very thin.

On the other hand, the Al treatment for 2 min of the fibers drastically changes the fracture mode of fiber. All the fibers fracture at the reaction layer, and the mean strength and the CV value decrease remarkably. The possible reason is as follows. Ochiai et al.⁽¹⁶⁾ and Baker et al.⁽¹⁷⁾ have reported that the fracture mechanism due to the reaction layer is attributed to a stress concentration due to a fiber caused by a premature failure of the layer. The stress concentration factor becomes larger with the thickness of the layer, depending on the exposure time, so that the fiber strength is lowered significantly. The reason why the CV

Table 2. Relation between the defect and the strength of Al treated SiC fibers.

Time (min)	Defects	N	Mean (GPa)	C.V. (%)	Photo.
0.5	Inner defect	1	4.16	—	Fig.3(a)
	Surface flaw	20	3.27	26.6	Fig.3(b)
	Reaction layer (1)	5	3.11	12.3	Fig.3(c)
	Reaction layer (2)	1	1.78	—	Fig.3(d)
2	Reaction layer	24	1.87	14.7	Fig.3(d)

N :Number of observed fibers. Mean :Average strength. CV :Coefficient of variation.

value decreases is assumed as follows. The layer consisting of the reaction products⁽⁵⁾ is more brittle than SiC fiber itself, and the thickness of the layer does not vary so widely as the depth of the surface defect.

5. Conclusions

Coreless SiC fibers were exposed to three kinds of environments which were often encountered during FRM fabrication by a liquid infiltration method, and the tensile strength of the fibers and the fracture surfaces were investigated. The results obtained are as follows;

(1) The fibers had no change in the tensile strength and fracture mode after isothermal heating at 700°C for 100h and also after the hydrostatic pressurizing at 196MPa for 10min.

(2) The fibers decreased in the average strength and the coefficient of variation with the exposure time after exposure to the liquid aluminum environment.

(3) Two types of fracture modes due to an inner defect and a surface defect were observed in the as-received fibers. On the other hand, all the Al treated fibers after 2min were limited to the one fracture mode due to the reaction layer. This induced a decrease of tensile strength. The fibers failed by the inner defect have the highest strength, and the fibers failed by the surface defect have the second highest strength. The fibers failed by the reaction layer have the weakest strength of all.

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