Bias in the Weibull Strength Estimation of a SiC Fiber for the Small Gauge Length Case*

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It is known that the single-modal Weibull model describes well the size effect of brittle fiber tensile strength. However, some ceramic fibers have been reported that single-modal Weibull model provided biased estimation on the gauge length dependence. A hypothesis on the bias is that the density of critical defects is very small, thus, fracture probability of small gauge length samples distributes in discrete manner, which makes the Weibull parameters dependent on the gauge length. Tyranno ZMI Si-Zr-C-O fiber has been selected as an example fiber. The tensile tests have been done on several gauge lengths. The derived Weibull parameters have shown a dependence on the gauge length. Fracture surfaces were observed with SEM. Then we classified the fracture surfaces into the characteristic fracture patterns. Percentage of each fracture pattern was found dependent on the gauge length, too. This may be an important factor of the Weibull parameter dependence on the gauge length.

Key Words: Weibull Model, Tyranno ZMI, Gauge Length, Fracture Surface

1. Introduction

Ceramic Matrix Composites (CMCs) have excellent heat resistance and specific tensile strength. However, it is difficult to design reliable CMC components due to the wide distribution in the strength. Mechanical properties of reinforcement fibers have major effect on the strength distribution of CMCs. Thus, ceramic fibers must be evaluated not only in the mean strength but also in the dispersion, beforehand the CMCs production(1), (2).

It is known that the strength distribution of brittle material is described well with single-modal Weibull model. Ceramic fibers are brittle, thus the single-modal Weibull model may fit well to the strength. However, some ceramic fibers have been reported that single-modal Weibull model provided biased estimation on the gauge length dependence(3), (4). A hypothesis on the bias is that the density of critical defects is very small thus fracture probability of small gauge length samples distributes in discrete manner, which makes the Weibull parameters dependent on the gauge length. The objectives of this study is thus to assess if the Weibull parameters are dependent on the gauge length for the case of small gauge length samples.

To analyze the dependence of the Weibull parameters on the gauge length, we made single fiber tensile tests on the gauge lengths of 200 mm, 100 mm, 50 mm, and 20 mm on Tyranno ZMI Si-Zr-C-O monofilaments (UBE Industry Co.) as an example ceramic fiber.

SEM fractography was conducted on the fracture surfaces. The relations between the number of characteristic fracture patterns and gauge length were discussed.

2. Experiment

2.1 Sample

Table 1 shows typical properties of Tyranno ZMI fiber supplied by UBE INDUSTRIES LTD.

2.2 Diameter measurements along the fiber axes

Laser Scan Micrometer type LSM-500 (MITUTOYO, Corp.) has been applied for the diameter measurements of the sample fibers along each 1 mm of the 500 mm gauge length, as some ceramic fibers have been reported to show variable diameters both along the gauges and between each fibers at a bundle(5) – (8).

It implies that to calculate fracture stress with a diameter may provide biased data. Thus, to measure diameters in gauge section beforehand the tensile tests is indispensable to derive accurate Weibull parameters.
Table 1 Typical properties of Tyranno ZMI fiber

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filament Diameter (μm)</td>
<td>11</td>
</tr>
<tr>
<td>Filament Number ( )</td>
<td>800</td>
</tr>
<tr>
<td>tex (g/1000m)</td>
<td>200</td>
</tr>
<tr>
<td>Tensile Strength (GPa)</td>
<td>3.4</td>
</tr>
<tr>
<td>Tensile Modulus (GPa)</td>
<td>200</td>
</tr>
<tr>
<td>Elongation at Break (%)</td>
<td>1.7</td>
</tr>
<tr>
<td>Density (g/cm^3)</td>
<td>2.48</td>
</tr>
<tr>
<td>Composition (wt.%): Si</td>
<td>56</td>
</tr>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td></td>
<td>O</td>
</tr>
<tr>
<td></td>
<td>Zr</td>
</tr>
<tr>
<td>Thermal Expansion (10^-4/K)</td>
<td>4.0</td>
</tr>
<tr>
<td>Coefficient (R.T.-1000°C)</td>
<td></td>
</tr>
<tr>
<td>Thermal Conductivity (W/m·K)</td>
<td>2.5</td>
</tr>
</tbody>
</table>

2.3 Tensile Test

2.3.1 Specimen

For the single fiber tensile tests, tensile specimens were prepared as depicted in Fig. 1. A paper holder has a slot in the central part for the gauge lengths of 200 mm, 100 mm, 50 mm or 20 mm. However, 5 mm was added at both ends of the slot to avoid the error due to the clamp effect. Thus, the data of the samples that fractured within the 5 mm were neglected in the statistical analyses.

Complete form of specimen is as depicted in Fig. 2. At tensile test, we filled protection films up with viscous liquid of 20 wt% alpha-olefin sulphonate (AOS) based surfactant and 80 wt% water for the ease of fragment recovery. Note that the protection films did not touch the fiber by the paper thickness, thus fiber strength was measured without the friction bias.

2.3.2 Tensile Test

Universal Tensile Test System type 5542 (INSTRON, Corp.) with 10 N load cell was used with the crosshead speed of 0.1 mm/min.

2.3.3 SEM Fractography

Fracture surfaces were observed with SEM, model S-4700 (HITACHI, Corp.). Then we classified the fracture surfaces into typical fracture patterns.

3. Statistical Analyses

Single-modal Weibull distribution function is given by:

\[ F(\sigma) = 1 - \exp \left( \frac{V}{V_0} \left( \frac{\sigma}{\sigma_0} \right)^m \right) \]  (1)

Where, \( V_0, V, m, \) and \( \sigma_0 \) are standard volume, volume of specimen, shape parameter and scale parameter, respectively. The \( V/V_0 \) is proportional to the gauge length \( l \) of the fiber if the diameters are uniform along the gauge(9), (10). Thus, Eq. (1) may be rewritten as follows.

\[ F(\sigma) = 1 - \exp \left[ -l \left( \frac{\sigma}{\sigma_0} \right)^m \right] \]  (2)

However, the Eq. (2) is not suitable for the strength estimation of variable diameter fibers. For long fibers of variable diameters, several modifications have been reported for the exact strength scalings(11), (12). For the short gauge length fibers, however, a new modification is essential to apply the single-modal Weibull model. Thus, in this study, a gauge section was divided into short length “Δℓ” sections at which stress is considered to be uniform. Then cumulative fracture probability at each section was calculated. The fracture probability of whole gauge section was then derived by multiplying the fracture probabilities of “Δℓ” sections.

The whole gauge length had been divided as the cylinders of “Δℓ= 1 mm” as schematically shown in Fig. 3. Stress of each cylinder was calculated with the fracture load and the diameter of each cylinder, then the fracture probability of i_th cylinder, which is defined as \( F_i(\sigma_i) \) was assigned.

Then, cumulative fracture probability of whole gauge length \( F(\sigma) \) was given as following.

\[ F(\sigma) = 1 - \prod_{i} \left[ 1 - F_i(\sigma_i) \right] \]  (3)

The cumulative fracture probabilities calculated with this method were of hypothetical \( m \) and \( \sigma_0 \). The calculations with the Eq. (3) provide \( n \) cumulative probabilities if the sample number is \( n \). By ordering the results from the lowest probability to the highest probability, the ith result \( F_i \) in the set of \( n \) samples may be reassigned a cumulative probability of failure \( F_{i,rank} \) by the estimators such as...
Fig. 3 A model for the Weibull analysis of variable diameter fiber

\[ F_{i,\text{rank}} = i/(n + 1). \]

If a set of Weibull parameters \( m \) and \( \sigma_0 \) is ideal and the sample number \( n \) is large enough, the \( F_i \) and \( F_{i,\text{rank}} \) must be equal. Thus, we set a factor \( G \) that is defined by Eq. (4) and select a set of \( m \) and \( \sigma_0 \) when the \( G \) is minimum.

\[
G = \sum_i \left( F_i(\sigma_i) - F_{i,\text{rank}} \right)^2
\]  

(4)

In this calculation, \( V_0 \) is given as \( 1.9 \times 10^{-12} \text{ m}^3 \), which equals to the cylinder volume of 11 \( \mu \text{m} \) in the diameter of manufacture’s data sheet and 20 mm in the length.

4. Results and Discussion

The axial diameter distributions have been measured on 200 pieces of Tyranno ZMI Fiber along 500 mm gauge. Five examples of diameter data along each 1 mm of the 500 mm gauge length are shown in Fig. 4. The results showed that the diameters vary widely along the gauges. Thus, we must take into account the fact that the tensile stresses are not uniform along the gauge length. Thereby, the Weibull plot using a diameter yields excessive error that may bias the size effect of Tyranno ZMI Fiber.

Statistical data of tensile tests are given in Table 2. Mean fracture stress in Table 2 was defined as the quotient of the fracture load over the cross sectional area at the fracture point.

Table 3 shows shape parameter \( m \) and scale parameter \( \sigma_0 \) calculated with layered cylindrical model. Shape parameter \( m \) has a tendency to become smaller with longer gauge length, and scale parameter \( \sigma_0 \) becomes larger with longer gauge length.

The parameters of single-modal Weibull model are shown to be dependent on the gauge lengths. Thus, single-modal Weibull model may provide biased size effect in scaling the fiber strength.

It was found that there were four kinds of fracture pattern as depicted in Fig. 5. The pattern (A) was defined as fracture surface with a very small particle at the fracture origin, or the particle was invisible. Fracture pattern (A) may include a case of particle drop off from the fracture surface. The fracture pattern (A) was named ‘normal’ as it was found the most often on the fracture surfaces. Fracture pattern (B) was defined as fracture surface with an agglomerate of particles at the origin. Fracture pattern (C) was defined as fracture surface with a crack at the origin. In pattern (A) to (C), defects were found close to the circumference of cross section. Fracture pattern (D) was defined as fracture surface that has the fracture origin inside of the circumference.

Table 4 shows the percentage of each fracture pattern at each gauge length. The number of fracture pattern (A) decreases with increasing gauge length. On the other hand, the number of fracture pattern (B) or (C) tends to increase with increasing gauge length. The fracture pattern (D) is found very rare.
Table 4 Percentage of each fracture pattern

<table>
<thead>
<tr>
<th>Gauge length [mm]</th>
<th>20</th>
<th>50</th>
<th>100</th>
<th>200</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A) normal [%]</td>
<td>98</td>
<td>94</td>
<td>72</td>
<td>47</td>
</tr>
<tr>
<td>(B) agglomerate [%]</td>
<td>4</td>
<td>9</td>
<td>9</td>
<td>27</td>
</tr>
<tr>
<td>(C) crack [%]</td>
<td>16</td>
<td>6</td>
<td>16</td>
<td>27</td>
</tr>
<tr>
<td>(D) central part [%]</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Numbers of samples</td>
<td>25</td>
<td>32</td>
<td>32</td>
<td>30</td>
</tr>
</tbody>
</table>

Figure 6 shows Stress-Diameter plots at gauge length 20 mm. The fracture stress in Fig. 6 was defined as the quotient of the fracture load over the cross sectional area at the fracture point. It SEM fractography indicated that the defect responsible for fracture pattern (A) has the highest probability of existence in sample fibers. In contrast, fracture pattern (B) and (C) are anticipated that they have lower probabilities of existence in sample fibers. However, defects of pattern (B) and (C) have prominent effects on fiber fracture, as is indicated in Fig. 6, too.

These results show that there are several kinds of fracture origin and the percentages change with the gauge length. This may be an important factor of the Weibull parameter dependence on the gauge length.

5. Conclusions

The following conclusions may be drawn from the studies on Tyranno ZMI fiber.

1) The parameters of single-modal Weibull model are dependent on the gauge length when gauge length is small.

2) Single-modal Weibull model provides a biased size effect of Tyranno ZMI fiber strength.

3) The percentage of typical fracture pattern is de-
dependent on the gauge length.

References


