Strength Improvement on an Imaginary SiC Fiber of Ideal Diameter and Reduced Internal Defects Estimated from the Weibull Scaling of Tyranno ZMI Fiber*

Tetsuya MORIMOTO**, Koji YAMAMOTO*** and Shinji OGIHARA****

The size effect of the diameter has been assessed for the tensile strength of Tyranno ZMI fiber. Uniform diameter section in sample fibers has been selected as tensile test sample. The single fibers of measured diameters have been tensile tested to provide two groups of data, i.e., “small diameter” group and “large diameter” group. The parameters of single-modal Weibull model showed inconsistency on the two groups, thus the Weibull parameters have shown the dependence on the sample diameter. Scanning electron microscope (SEM) analyses had revealed characteristic fracture patterns of “extremely weak” samples only in “large diameter” group. The key information for improving the reliability was then discussed through coupling the Weibull scaling and the fracture surface analyses. The potential in the strength improvement has been assessed for an imaginary fiber, which does not contain the sources of the characteristic fracture patterns.

Key Words: Size Effect, Weibull Model, Strength Improvement, Fracture Sources, Fractography

1. Introduction

Ceramic Matrix Composites (CMCs) have been studied for gas-turbine applications due to the excellence in the mechanical properties and heat resistance. Tyranno fiber (UBE INDUSTRIES, LTD.), which is one of the reinforcements for CMCs, has good oxidation resistance and it doesn’t show notable strength reductions after a heat treatment of 1 500° for 1 000 h, following the supplier’s report(1). However, the strength of Tyranno fiber shows a large dispersion, which limits the advantage in the applications(2).

Tyranno ZMI fiber has been known to show variable diameter both along the gauge length and between fibers at a bundle(3). Thus, the parameters of single-modal Weibull model may vary as functions of fiber diameter due to the diameter-related material inhomogeneity. The strength estimation may be thus more reliable by correlating the diameter factor. In addition, key information for improving the reliability may be derived through coupling the Weibull scaling and the fracture surface analyses. In this work, the interaction between fiber diameter and Weibull parameters was studied with an emphasis on understanding the important role of the fracture sources.

2. Experimental

2.1 Diameter measurement

For diameter measurements, a laser scan micrometer LSM-6000 (MITUTOYO, Corp.) of ±0.1 μm accuracy has been applied along the gauge length of 500 mm in 1 mm steps. Figure 1 shows an example of the measurements. As shown in this figure, Tyranno ZMI fiber shows widely variable diameter along the gauge.

The uniform diameter sections such as a section “A” are suitable for the tensile test for the exclusion of the diameter distribution bias. The tolerance of diameter was set ±1% in selecting the uniform diameter sections, thus, for example, a section of the diameter from 9.9 μm to 10.1 μm was assumed of 10.0 μm.

2.2 Tensile test

Each sample fiber was glued on the paper holder with
elastic adhesive across the 30 mm slot, as shown in Fig. 2. However, the samples fractured within the 5 mm ends were not used for the following Weibull analyses\(^{(4),(5)}\) in order to avoid the bias by the glue and grip stress concentration. Thus, the gauge length was the central 20 mm in the 30 mm paper slot.

The sample fiber was covered with protection films except lower 3 mm. Beforehand the tensile test, the shaded area in Fig. 2 was filled with a surfactant for the 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.

Each sample was attached to an Instron Universal Tensile Test System model 5 542 of 10 N load cell, and then the paper holder was cut at the perforation in Fig. 2. Cross head speed was set 0.1 mm/min., which may be slow enough to assimilate a quasi static loading.

2.3 Weibull analysis

The single-modal Weibull model was set as follows

\[
F(\sigma) = 1 - \exp \left[ -\frac{D}{D_0} \left( \frac{\sigma}{\sigma_0} \right)^m \right]
\]

(1)

where \(F\) is the fracture probability of the fiber under an uniaxial tensile stress \(\sigma\), \(m\) is the shape parameter and \(\sigma_0\) is the scale parameter of the distribution. \(D\) is the diameter of each sample and \(D_0\) is an imaginary standard diameter\(^{(6),(7)}\). Note that the critical cracks were assumed to nucleate from the fiber surface, thus the population was proportional to the diameter. The mean diameter of the “small diameter” group was selected as the standard diameter \(D_0\).

Equation (1) was modified as follows for the Weibull plots of the each group.

\[
\ln \left( \frac{1}{1 - F(\sigma)} \right) - \ln \left( \frac{D}{D_0} \right) = m \ln \sigma - m \ln \sigma_0
\]

(2)

2.4 Fractography

The fracture surfaces of recovered samples were analyzed using a scanning electron microscope (SEM), model S-4700 (HITACHI, Corp.).

3. Results and Discussion

3.1 Tensile test

Figure 3 shows the tensile tests results. The 60 tensile test results are divided into two groups of 30 samples, i.e., a “small diameter” group and a “large diameter” group at the border diameter of 11.5 µm. Most samples have shown the strength from 3 to 4 GPa. However, several samples in the “large diameter” group have showed extremely low strength of 1 to 2 GPa.

3.2 Weibull analysis

Two Weibull plots for the two groups of a “small diameter” group and a “large diameter” group have shown the inconsistency as depicted in Fig. 4. In addition, the shape parameter \(m\) of the small diameter group was larger...
than that of the large diameter group, implying that the strength of small diameter fibers have smaller dispersion than that of large diameter fibers (Table 1).

Table 1 Weibull parameters for “large diameter” and “small diameter” groups

<table>
<thead>
<tr>
<th>Diameter</th>
<th>m</th>
<th>( o_0 )</th>
<th>Mean strength (GPa)</th>
<th>Variance (( \times 10^{15} ))</th>
<th>Standard deviation (( \times 100 )MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Small diameter</td>
<td>6.51</td>
<td>3.44</td>
<td>3.21</td>
<td>3.14</td>
<td>5.60</td>
</tr>
<tr>
<td>Large diameter</td>
<td>3.73</td>
<td>3.77</td>
<td>3.18</td>
<td>5.32</td>
<td>7.29</td>
</tr>
</tbody>
</table>

However, the small shape parameter of “large diameter” group was due to the extremely weak fibers. Following fractography analysis shows the sources of these extremely weak samples.

### 3.3 Fractography analysis

SEM micrographs showed the fiber fracture origins, which the authors classified as is shown in Fig. 5. Table 2 shows the results of single fiber tensile tests, and the classifications. Each factor symbol is defined as following.

“(a) No trace” was defined as “there is no trace at fracture starting point”. “(b) Particle on side face” was as “there is a particle on the fiber side face”. “(c) Particle in cross-sectional surface” was as “there is a particle

Table 2 Single fiber tensile test results

<table>
<thead>
<tr>
<th>Small diameter Diameter (( \mu m ))</th>
<th>Strength (GPa) Factor group</th>
<th>Large diameter Diameter (( \mu m ))</th>
<th>Strength (GPa) Factor group</th>
<th>Large diameter Diameter (( \mu m ))</th>
<th>Strength (GPa) Factor group</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.3</td>
<td>3.22 B</td>
<td>10.4</td>
<td>3.42 A</td>
<td>11.5</td>
<td>2.90 C</td>
</tr>
<tr>
<td>7.7</td>
<td>2.37 D</td>
<td>10.5</td>
<td>4.29 A</td>
<td>11.5</td>
<td>3.57 A</td>
</tr>
<tr>
<td>8.3</td>
<td>3.34 B</td>
<td>10.5</td>
<td>3.13 A</td>
<td>11.6</td>
<td>2.94 A</td>
</tr>
<tr>
<td>8.6</td>
<td>3.63 A</td>
<td>10.6</td>
<td>3.53 B</td>
<td>11.7</td>
<td>2.98 F</td>
</tr>
<tr>
<td>9.1</td>
<td>3.25 B</td>
<td>10.6</td>
<td>3.86 C</td>
<td>11.7</td>
<td>2.99 A</td>
</tr>
<tr>
<td>9.6</td>
<td>3.88 B</td>
<td>10.8</td>
<td>3.06 C</td>
<td>11.8</td>
<td>2.66 B</td>
</tr>
<tr>
<td>9.6</td>
<td>2.49 C</td>
<td>10.9</td>
<td>3.55 C</td>
<td>11.9</td>
<td>2.71 B</td>
</tr>
<tr>
<td>9.8</td>
<td>4.12 B</td>
<td>11.2</td>
<td>2.95 A</td>
<td>12.1</td>
<td>4.27 A</td>
</tr>
<tr>
<td>9.9</td>
<td>2.48 E</td>
<td>11.2</td>
<td>3.26 B</td>
<td>12.2</td>
<td>6.94 E</td>
</tr>
<tr>
<td>9.9</td>
<td>3.00 B</td>
<td>11.2</td>
<td>2.65 B</td>
<td>12.2</td>
<td>3.43 A</td>
</tr>
<tr>
<td>10</td>
<td>3.07 A</td>
<td>11.2</td>
<td>2.85 B</td>
<td>12.2</td>
<td>4.55 A</td>
</tr>
<tr>
<td>10.1</td>
<td>3.87 B</td>
<td>11.3</td>
<td>3.00 A</td>
<td>12.3</td>
<td>3.55 C</td>
</tr>
<tr>
<td>10.2</td>
<td>2.82 A</td>
<td>11.4</td>
<td>3.83 A</td>
<td>12.3</td>
<td>3.21 A</td>
</tr>
<tr>
<td>10.3</td>
<td>2.17 F</td>
<td>11.4</td>
<td>2.36 E</td>
<td>12.4</td>
<td>3.74 A</td>
</tr>
<tr>
<td>10.4</td>
<td>3.66 A</td>
<td>11.4</td>
<td>3.34 B</td>
<td>12.5</td>
<td>3.43 A</td>
</tr>
</tbody>
</table>
embedded in the cross sectional surface”. “(d) Crack at side face” was as “there is a crack on the fiber side face”. “(e) Inhomogeneous structure” was as “there is a foamed structure at fracture starting point”. “(f) Internal starting point” was as “fracture starting point was not on the circumference”.

4. Estimation of the Strength Improvement

4.1 Influence of D and E factors

Fractography analysis showed that the “extremely weak” samples (in Fig. 3) were categorized as D or E factor groups (Fig. 6). In other words, the groups D and E possessed extremely harmful factors to the fiber strength.

SEM fractography have revealed that these three “Extremely Weak” samples exhibited relatively larger mirror zone than other samples, as depicted in Fig. 7. In addition, the mirror zones of low strength case, D and E, were larger than the case of relatively higher strength case of A, B, C and F. Thus, the “larger mirror zones” may imply that some factors of lower strength are also the factors of larger mirror zone.

It has been reported with the basis of fracture mechanics that the product of strength, $\sigma$, and the square root of the distance of the mirror radius from the origin of the fracture, is a constant\(^{[8]}\). The relationship between the mirror size and the fracture stress was reproduced also in this study, as depicted in Fig. 8. The fracture factors of $D$ and $E$ may also be indicated in Fig. 8 to possess distinct effect both on the mirror constant, which is the slop of the plot, and on the low strength.

4.2 Estimation of the Weibull parameters

Although there are several kinds of fracture sources, the ones of $D$ and $E$ are detrimental to the strength, especially of larger diameter fibers, as was indicated through the SEM fractography. The fracture model on Tyranno ZMI fiber may thus require a multi-modal function. This may be the key factor of the inconsistency of single-modal Weibull analyses between the large diameter group and small diameter group, as seen in Fig. 4 and Table 1. Thus, it is rational to deduce that the strength of Tyranno ZMI fiber drastically improves if the fracture factors in $D$ and $E$ are cancelled by the refinement of the production process. We have thereby conducted a Weibull scaling on the case without the data of $D$ and $E$, as depicted in Fig. 9 and Table 3.

The results revealed a larger Weibull shape parameter $m$ as depicted in Table 3. What is more, the Weibull plots showed good agreement between the “large diameter
Table 3  Weibull parameters (D and E: removed)

<table>
<thead>
<tr>
<th>Group</th>
<th>m (with D)</th>
<th>m (with E)</th>
<th>( \mu ) (GPa)</th>
<th>( \sigma_0 ) (GPa)</th>
<th>Mean strength (( \times 10^3 )) (GPa)</th>
<th>Variance (( \times 10^3 )) (GPa)</th>
<th>SD (GPa)</th>
<th>MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Small</td>
<td>8.59</td>
<td>6.51</td>
<td>3.54</td>
<td>3.35</td>
<td>2.07</td>
<td>4.55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Large</td>
<td>7.95</td>
<td>3.73</td>
<td>4.06</td>
<td>3.96</td>
<td>2.34</td>
<td>4.83</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Therefore, it was indicated that if the fracture factors of \( D \) and \( E \) are cancelled, the strength of Tyranno ZMI fiber improves, becomes less distributed, and is insensitive to the diameter.

5. Conclusions

The authors have investigated the influence of the diameter of Tyranno ZMI fiber on the statistical strength, through single fiber tensile testing, SEM analysis, and Weibull strength scaling. Following conclusions are drawn from the results.

(1) Weibull parameters of Tyranno ZMI fiber are dependent on the fiber diameter. Shape parameter \( m \) decreases with increasing fiber diameter. Thus, the strength distribution of large diameter fiber group is larger than that of small diameter group.

(2) The diameter dependence of the Weibull parameters due to extremely weak samples. Fractography showed the fracture factors of the extremely weak samples are categorized as a limited number of distinct fracture sources. Thus, the dependence may disappear and the strength of Tyranno ZMI fibers may be improved by the refinement of the production process.

References