INFLUENCE OF THE DIAMETER OF TYRANNO ZMI FIBER ON THE STATISTICAL STRENGTH

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Abstract

The size effect on the diameter has been assessed for the tensile strength of Tyranno ZMI Si-Zr-C-O fiber (UBE INDUSTRIES, LTD.), which shows variable diameter along the gauge. 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 potential in the strength improvement has been discussed for an imaginary fiber, which does not contain the characteristic crack sources.

1 Introduction

Ceramic Matrix Composites (CMCs) have been studied for gas-turbine applications due to the excellence in the mechanical properties and heat resistances. Tyrannofiber (UBE INDUSTRIES, LTD.), which is one of the reinforcement for CMCs, has good oxidation resistance and it doesn't show notable strength reductions after a heat treatment of 1500℃ for 1000h, 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. 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 explored with an emphasis on understanding the important role of the fracture sources.

2 Experimental

2.1 Sample fiber

A bundle of previously desized Tyranno ZMI Si-Zr-C-O fiber was used in this study. Table 1 shows the mechanical properties and the chemical compositions reported by the supplier [3].

Table 1 Mechanical properties and chemical compositions of Tyranno ZMI fiber (UBE INDUSTRIES REPORT)

Fiber diameter		11（μm）
Tensile strength		3.4（GPa）
Young's modulus		200（GPa）
Strain		1.7（%）
Chemical compositions	Si	56（wt.%）
	C	34（wt.%）
	O	9（wt.%）
	Zr	1（wt.%）

2.2 Diameter measurements

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

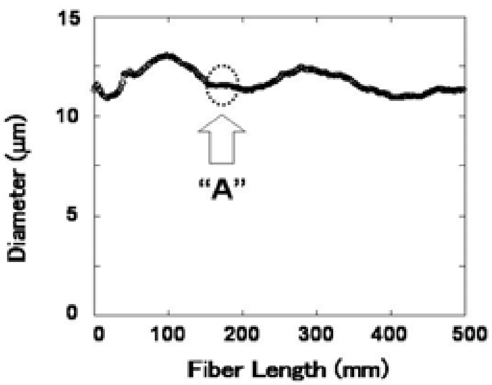


Fig.1 Result of LSM measurement (Example)

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 9.9μm to 10.1μm in the length of 20 mm was assumed of 10.0μm.

2.3 Tensile test

Each sample fiber was glued on the paper holder with elastic adhesive across the 30mm slot, as is shown in Fig.2. However, the samples fractured within the 5mm 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 20mm of the paper slot of 30mm.

The sample fiber was covered with protection films except lower 3mm. 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.

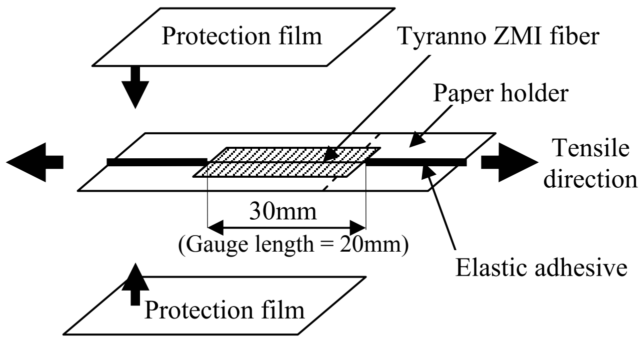


Fig.2 Tensile test specimen

Each sample was attached to an Instron Universal Tensile Test System model 5542 of 10N 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.

2.4 Weibull analysis

The single-modal Weibull model was set as follows

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

where F is the fracture probability of the fiber under an uniaxial tensile stress σ, m is the shape parameter and σ_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.

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

$$\ln \ln\left(\frac{1}{1 - F(\sigma)}\right) - \ln\left(\frac{D}{D_0}\right) = m \ln \sigma - m \ln \sigma_0 \qquad (2)$$

2.5 SEM

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

Fig.3 shows the tensile tests results. The 60 tensile test results were divided into two groups of 30 samples, i.e., a "small diameter" group and a "large diameter" group at the border diameter of 11.5μm.

Most samples have shown the strength from 3 to 4GPa. However, several samples in the "large diameter" group have showed extremely low strength of 1 to 2GPa.

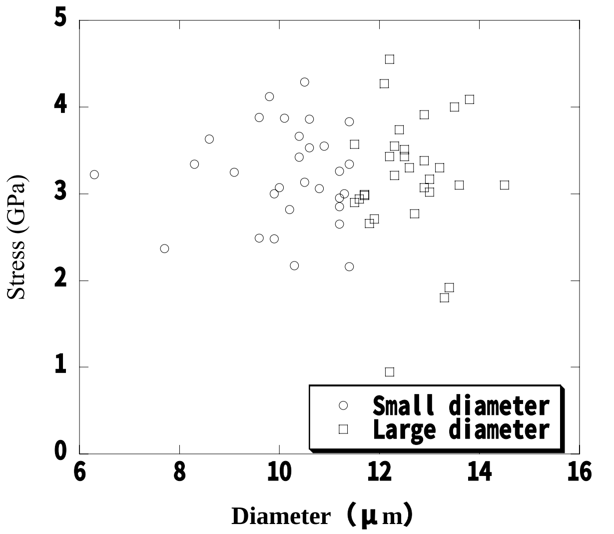
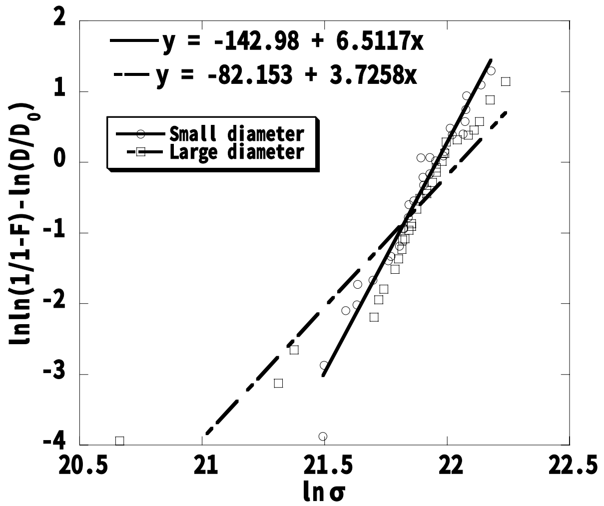


Fig.3 Single fiber tensile test results

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.



Diameter	m	σ_0 (GPa)	Mean stress (GPa)	Variance ($\times 10^{17}$)	Standard deviation (×100MPa)
Small diameter	6.51	3.44	3.21	3.14	5.60
Large diameter	3.73	3.77	3.18	5.32	7.29

Fig.4 Weibull plots for "large diameter" and "small diameter" groups

However, the small shape parameter of "large diameter" group was due to the several extremely weak fibers.

3.3 SEM 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.

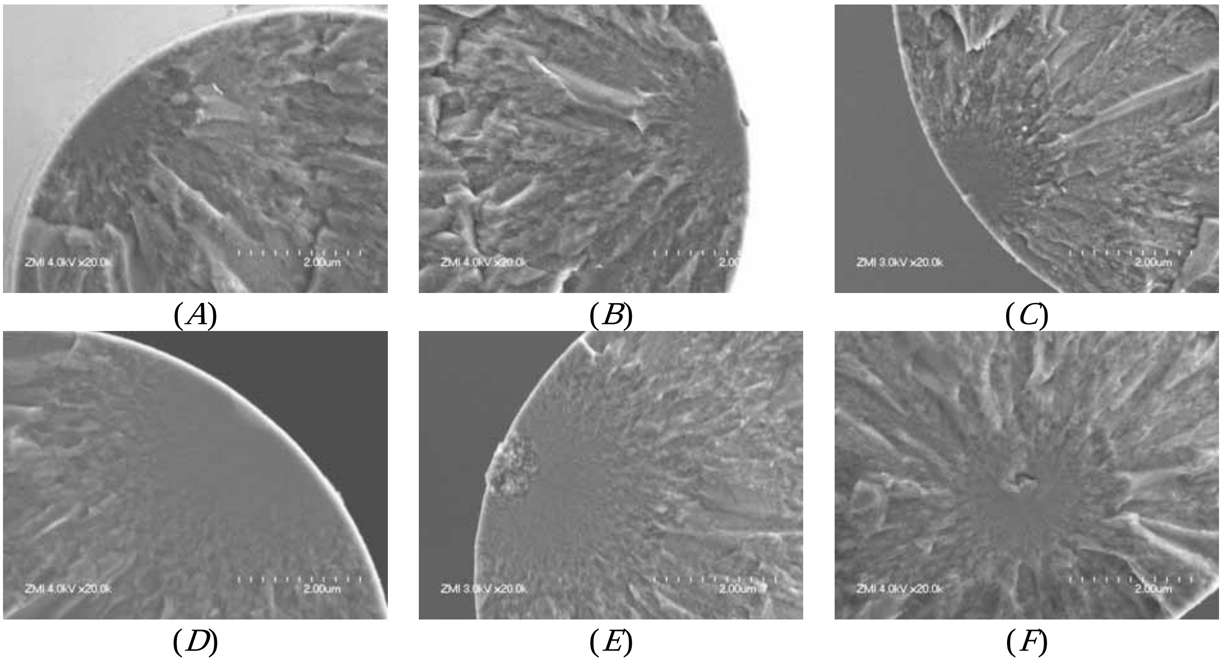


Fig.5 SEM micrographs of each factor group (Example) (*A*)no trace (*B*)particle on side face (*C*)particle in cross-sectional surface (*D*)crack at side face (*E*)unhomogeneous structure (*F*)internal starting point

Table 2 Single fiber tensile test results

Small diameter						Large diameter					
Diameter (μm)	Stress (GPa)	Factor group	Diameter (μm)	Stress (GPa)	Factor group	Diameter (μm)	Stress (GPa)	Factor group	Diameter (μm)	Stress (GPa)	Factor group
6.3	3.22	*B*	10.4	3.42	*A*	11.5	2.90	*C*	12.5	3.51	*A*
7.7	2.37	*Dmir.*	10.5	4.29	*A*	11.5	3.57	*A*	12.6	3.3	*B*
8.3	3.34	*B*	10.5	3.13	*A*	11.6	2.94	*A*	12.7	2.77	*C*
8.6	3.63	*A*	10.6	3.53	*B*	11.7	2.98	*F*	12.9	3.91	*A*
9.1	3.25	*B*	10.6	3.86	*C*	11.7	2.99	*A*	12.9	3.07	*C*
9.6	3.88	*B*	10.8	3.06	*C*	11.8	2.66	*B*	12.9	3.38	*B*
9.6	2.49	*C*	10.9	3.55	*C*	11.9	2.71	*B*	13.0	3.02	*A*
9.8	4.12	*B*	11.2	2.95	*A*	12.1	4.27	*A*	13.0	3.17	*C*
9.9	2.48	*Emir.*	11.2	3.26	*B*	12.2	0.94	*Emir.*	13.2	3.30	*A*
9.9	3.00	*D*	11.2	2.65	*B*	12.2	3.43	*A*	13.3	1.8	*Dmir.*
10	3.07	*A*	11.2	2.85	*B*	12.2	4.55	*A*	13.4	1.92	*Emir.*
10.1	3.87	*B*	11.3	3.00	*A*	12.3	3.55	*C*	13.5	4.00	*F*
10.2	2.82	*A*	11.4	3.83	*A*	12.3	3.21	*A*	13.6	3.10	*D*
10.3	2.17	*Emir.*	11.4	2.16	*Emir.*	12.4	3.74	*A*	13.8	4.09	*A*
10.4	3.66	*A*	11.4	3.34	*B*	12.5	3.43	*A*	14.5	3.10	*B*

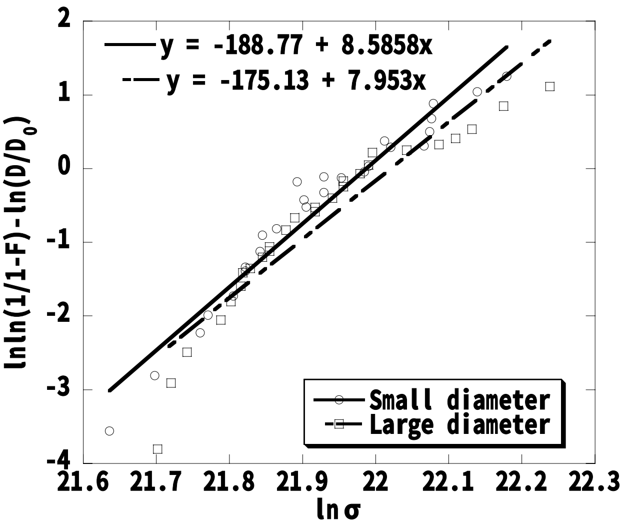
"(*A*) No trace" was defined as "there is no trace at fracture starting point". "(*B*) Particle on side face" was as "there is a particle on the fiber side face". "(*C*) Particle in cross-sectional surface" was as "there is a particle imbedded in the cross sectional surface". "(*D*) Crack at side face" was as "there is a crack on the fiber side face". "(*E*) Unhomogeneous structure" was as "there is a foamed structure at fracture starting point". "(*F*) Internal starting point" was as "fracture starting point was not in/on the circumference". "(*mir.*) Large mirror zone" was as "the mirror zone extend more than 10 % of the cross sectional surface", like Fig.5 (*D*). "(*Dmir.*)" and "(*Emir.*)" were as "(*D*) and (*mir.*)" and "(*E*) and (*mir.*)", respectively.

It was observed that (*mir.*) factors were found only in (*D*), and (*E*) groups. The number of (*mir.*) fibers is not concerned with the fiber diameter. Fibers of (*Dmir.*) or (*Emir.*) groups in large diameter group show a very low strength.

3.4 Strength improvement estimation

SEM analysis implied that the groups *D* and *E* possessed extremely harmful factors to the fiber strength. In reality, the two Weibull plots, which do not contain (*Dmir.*) and (*Emir.*) data, show good agreement of improved strength as is seen in Fig.6.

Therefore, the authors estimate that the strength of Tyranno ZMI fibers may be improved and becomes more reliable if the factors in the group *D* and *E* are removed by improving the fiber production process.



Diameter	m	σ_0 (GPa)	Mean stress (GPa)	Variance ($\times 10^{17}$)	Standard deviation ($\times$100MPa)
Small diameter	8.59	3.54	3.35	2.07	4.55
Large diameter	7.95	3.66	3.36	2.34	4.83

Fig.6 Weibull plots (*Dmir.* and *Emir.* data removed)

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 may be 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. It shows the strength distribution of large diameter group is larger than that of small diameter group.
2. The diameter dependence of the Weibull parameters dues to extremely weak samples, which appear only in large diameter fibers. Thus, the dependence may disappear and the strength of Tyranno ZMI fibers may be improved by the refinement of the production process.

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