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MODIFIED FRACTURE MIRROR METHOD AND ITS APPLICATION IN SILICON CARBIDE MATRIX COMPOSITES

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ABSTRACT: A fracture mirror method was modified and applied to evaluation of the in-situ strength of fibers in ceramic composites. The most significant modifications include (1) the mirror constant replies on the tensile test of fiber bundles instead of single fiber test and (2) the gauge length parameter is redefined. Traditionally the gauge length parameter was considered as mean fiber pull out length. In this study, by analyzing the mechanism of fiber pullout, the parameter was defined as the sum of double pullout length and double ineffective length of the broken fibers and the random parameter was then graded statistically. Based on this method, Weibull parameters and mean in-situ strength of the M40JB fiber embedded in SiC matrix composites produced from polymer precursor were obtained. The experimental results show that the Weibull shape parameters of fabricated fibers and in-situ ones are nearly identical, but the scale parameter of in-situ fiber strength is much lower than that of the fabricated one. That means position distribution of the flaws in both fibers does not vary obviously, however, the flaws are deeper in in-situ fiber than in fabricated one and that lead the strength of the former fiber decreases markedly.

KEYWORDS: silicon carbide matrix composites, carbon fiber, in-situ strength, gauge length

INTRODUCTION

The strength of fibers is usually considered as the most important factor in the design of high performance fiber reinforced composites. However, in the case of ceramic matrix composites, the properties of the composites obtained is always much lower than expectation. This may be result of the degradation of in-situ strength of fibers. The direct determination of fibers in-situ strength in high performance ceramic matrixes, such as SiC, Si_3N_4 and carbon, is very difficult because the fibers cannot be extracted from composites by burning or corroding as in the case of polymer, metal and glass composites. Many studies[1-7] applied fracture mirror method to determine the in-situ strength of fibers in ceramic composites, but most of the studies aimed at the system of SiC fiber[4-7] and no report can be found about that of carbon fiber. In the traditional fracture mirror method, the steps are followed. (1) The fracture mirror radii and the pullout length of single fiber on a composite fractograph are measured by SEM. (2) The in-situ strength of single fiber is calculated by the empirical relationship between the fracture mirror radii and fiber strength. (3) Weibull statistical analysis is carried out based on large amount of measuring data, where the average value of fiber pullout length (over whole data) is considered as the gauge length. Thus the relative statistical parameters and the mean in-situ strength are obtained. From the concept of Weibull statistical distribution, the in-situ strength and Weibull parameters obtained following the above steps should correspond to a specific gauge length L. However, the pullout lengths of fibers are usually random on a true composite fractograph, so the assumption that the average pullout length equals to the gauge length L may be incorrect for the purpose. This is a problem that makes the theory less accurate. On the other hand, no fiber pullout can be observed on the fractographs of some brittle failure composites, that means the pullout length of the fibers equals to zero. Obviously, it is not correct to regard the zero pullout length as the gauge length.

In this paper, we focus our attention on solving these two problems by finding a statistic method to treat the scattered pullout length and establishing the relationship between the pullout length and the gauge length. The modified method is used to investigate the in-situ strength of M40JB fiber embedded in SiC matrix composite.

MODIFIED FRACTURE MIRROR METHOD

FRACTURE MIRROR METHOD

The in-situ strength of fibers can be characterized by the fracture mirror method. The schematic of fracture mirror in the cross-section of a fiber is shown in Fig.1. The cross-section consists of four parts: origin of failure, mirror, mist and hackle. Many papers showed that the nominal fracture stress is related to the dimensions of the mirrored section of the glass fracture. Smekal[8] and his co-workers found out that when circular glass rods are broken in tension, the normal breaking stress in the section outside the mirror is essentially constant over a fairly wide range of mirror sizes. Terao[9] reported that the breaking stress varies roughly with the reciprocal square root of the mirror radius. This relation has been confirmed by Levengood[10] for specific types of fracture. Shand[11,12] studied the local stress conditions which occur during the fracture process and derived the experimental relation of Smekal by analytical methods.

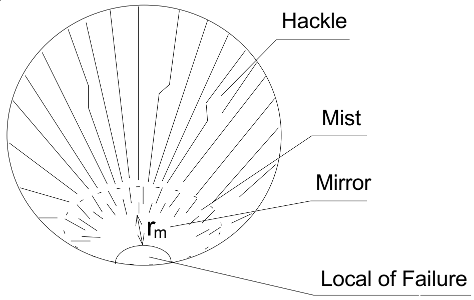


Fig.1 Schematic of fracture mirror in a fiber section

The in-situ strength of fiber can be determined by fracture mirror method, which is used to investigate the strength of the Nicalon fiber by Sawyer[13] and Jamet[14]. The empirical relations between in-situ strength of fibers σ_f and radius of the fracture mirror r_m is

$$\sigma_f r_m^{1/2} = A_m \tag{1}$$

where A_m is mirror constant. Rice[15] pointed out that fracture toughness of fibers K_f and r_m has a following relation

$$K_f \approx \sigma_f r_m^{1/2} / 3.5 \tag{2}$$

Comparing the eqns(1) and (2), and one has

$$A_m \approx 3.5K_f \tag{3}$$

So A_m can be determined as K_f is known. But it is difficult to obtain K_f in practice. Thus one has to determine A through eq.(1) by measuring strength of fiber and corresponding dimension of fracture mirror. Once the A_m is obtained, the eqn.(1) can be applied to the in-situ strength of fiber in composites.

Assuming that the in-situ strength of the fiber σ_f obeys Weibull distribution[13]

$$P_f(L,\sigma) = 1 - exp\left[-L\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{4}$$

where $P_f(L,\sigma)$ is the failure probability of a fiber with a length L loaded to stress level σ.

When the gauge length L is constant, one has

$$P_f(\sigma) = 1 - exp\left[-L\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{5}$$

where σ_0 is scale parameter and *m* is shape parameter. In reference[7], L was treated as average pullout length, so the σ_0 and m can be obtained based on eqn(5).

In order to evaluate the parameters m and σ_0, eqn(5) can be rearranged into a linear form as

$$\ln\ln\left(\frac{1}{1-p_f}\right) = m\ln\sigma + m\ln\left(\frac{L^{1/m}}{\sigma_0}\right) \tag{6}$$

Thus a plot of $\ln\ln(1/(1-p_f))$ verse $\ln\sigma$ will be a straight line whose slope and intercept are m and σ_0 respectively.

MODIFIED FRACTURE MIRROR METHOD

In order to determine the gauge length of the broken fiber in a composite, a model shown in Fig.2[16] is uesd. Assuming that the composite element is loaded by a tensile stress $\bar{\sigma}$ along the fiber, when a brittle matrix transverse crack develops and reaches the interface of the fiber/matrix, interface debond will occure. The debond will propagate symmetrically along the interface as $\bar{\sigma}$ becaomes larger and larger. The fiber will break along with the increasing of both the debond areas and the stress level in fiber, meanwhile the fiber pullout ocures. The pullout length of the fiber is represented by Lp. At the breaking point of the fiber, the stress in the fiber is zero and then it builds up with increasing distance from the break point until the stress reaches to its original value $\bar{\sigma}$. This buildup length, on each side of the break, is the ineffective length l_s of the fiber[17]. According to the cumulative weakening model of composites[17], there is same strength in the fiber in length of $2l_s$. Thus the gauge length corresponding to the in-situ strength determined by the fracture mirror data should be L= 2Lp+2 l_s. If there is no fiber pullout such as in brittle failure composites, the gauge length is 2 l_s.

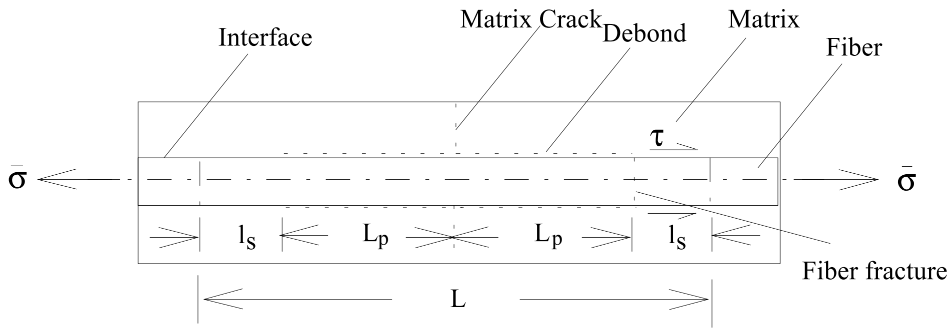


Fig.2 Analysis model of gauge length

Because of the large dispersion of the gauge length measured in practice and L is far from a constant, the direct application of statistical analysis based on eqn(5) is impossible. Grading statistical analysis is then proposed in this paper. The different L is graded as n levels, i.e. $L_1 \pm \Delta L$, $L_2 \pm \Delta L$, **, $L_n \pm \Delta L$, where $L_1 \langle L_2 \langle ......\langle L_n$, and $\frac{\Delta L}{L_i}\langle\langle 1$. In any of a level, or *i*th level, the gauge length is a constant with $\frac{\Delta L}{L_i} \to 0$. The mean strength $\bar{\sigma}_{fi}$ and L_i in this level must satisfy[18]

$$\bar{\sigma}_{fi} = \sigma_0 L_i^{-1/m}\Gamma\left(1+\frac{1}{m}\right) \tag{7}$$

where Γ is Gamma function.

The logarithm form of the eqn(7) is

$$ln\bar{\sigma}_{fi} = -\frac{1}{m} ln L_i + ln\sigma_0 + ln\Gamma\left(1+\frac{1}{m}\right) \tag{8}$$

Because of σ_0 and m are constants，eqn(8) represents a linear equation on logarithm coordinate（$\ln\bar{\sigma}_f, \ln L$）. The Weibull parameters σ_0 and m of in-situ strength can be obtained according to the eqn(8) based on a series of $\bar{\sigma}_{fi}$，L_i.

THE DETERMINATION OF MIRROR CONSTANT BY FIBER BUNDLE TEST

According to the classical theory of the statistical strength of fiber[19], the fiber is linear elastic until rupture under quasi-static loading. Hence, the stress/strain relationship is

$$\sigma_f = E_f \varepsilon_f \tag{9}$$

where E_f is the static elastic modulus of the fiber.

The failure strain of fiber, ε_f^s, corresponding to σ_f^s satisfies the following formula:

$$\sigma_f^s = E_f \varepsilon_f^s \tag{10}$$

The σ_f^s obeys the Weibull distribution, so the eqns(4)-(8) are valid for filament fiber at gauge length L. Now we establish the relationship of parameters between filament fiber and fiber bundle.

The fiber bundle model[19] under static loading is shown in Fig.3. In this model, parallel fibers of the same length, L, cross-sectional area, A, and elastic modulus E_f are rigidly fixed between the two ends. The deformation of the fibers is linear elastic until rupture, and there is no interaction between fibers. When some fibers break, the residual load is equally allotted to the unbroken fibers. This fiber bundle model can be regarded as a continuum unit if it has a large number of fibers, therefore the stress/strain relationship can be be expressed as

$$\sigma = E_b(1-\omega)\varepsilon \tag{11}$$

where E_b is the initial elastic modulus of the fiber bundle and ω is the damage parameter. In this model, one has the following equetions.

$$\begin{cases} E_b = E_f \\ \varepsilon = \varepsilon_f \end{cases} \tag{12}$$

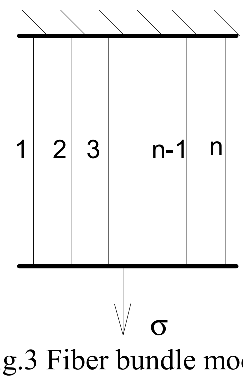


Fig.3 Fiber bundle model

According to the definition of the damage parameter as following equation and eqn(5)

$$\begin{aligned} \omega &= \frac{ineffective\ \ area}{total\ \ area} \\ &= \frac{broken\ \ fibers}{total\ \ fibers} \\ &= P_f(\sigma) \end{aligned} \tag{13}$$

$$= 1 - exp\left[-L\left(\frac{E_b \varepsilon}{\sigma_0} \right)^m \right]$$

Substituting this equation into eqn(11), the stress/strain relationship is denoted by

$$\sigma = E_b \varepsilon \; exp\left[-L\left(\frac{E_b \varepsilon}{\sigma_0} \right)^m \right] \tag{14}$$

A double logarithms form of eqn(14) is

$$ln\left[-ln\left(\frac{\sigma}{E_b \varepsilon} \right) \right] = m \; ln\left(E_b \varepsilon \right) - m \; ln\left(\sigma_0 \right) + ln \; L \tag{15}$$

From eqn(15), a Weibull plot can be obtained from an integral stress/strain curve, so σ_0 and m are calculated from slope and intercept of the linear equation.

In order to obtain A_m, equation (1) can be rewriten as

$$\sigma_{fi} = \frac{A_m}{r_{mi}^{1/2}} \tag{16}$$

where σ_{fi} is strength of *i*th filament and r_{mi} is radium of fracture mirror correspending to the filament. Then

$$\begin{aligned} \bar{\sigma}_f &= \frac{1}{n}\sum_{i=1}^{n} \sigma_{fi} \\ &= \frac{1}{n}\sum_{i=1}^{n} \frac{A_m}{r_{mi}^{1/2}} \\ &= \frac{A_m}{n}\sum_{i=1}^{n} \frac{1}{r_{mi}^{1/2}} \end{aligned} \tag{17}$$

i.e.

$$A_m = n\bar{\sigma}_f \Big/ \sum_{i=1}^{n} \frac{1}{r_{mi}^{1/2}} \tag{18}$$

where n is number of tested filament fiber.

IN-SITU STRENGTH OF M40JB FIBER IN SIC MATRIX COMPOSITES

The unidirectional M40JB fiber/SiC composite, with approximately 38 vol% fiber, is manufactured by dipping and pyrolysis of polycarbosilane precursor. The in-situ strength of M40JB fiber will be determined by modified fracture mirror method described in Section 2.2.

THE DETERMINATION OF A_M

The fiber bundle tensile test[19] was performed on MTS-810 universal testing machine with loading rate of 0.5mm/min. The tensile stress-strain curve is shown in Fig.4. The average values of maximum stresses σ_{max} , which was determined by Fig.4, corresponding strains ε_b and E_b are listed in Table.1. According to the tensile stress-strain curve of the fabricated fiber bundle, a Weibull plot can be obtained on the coordinate of ($ln\left[-ln\left(\frac{\sigma}{E_b \varepsilon} \right) \right]$, $ln(E_b \varepsilon)$) . From the straight line shown in Fig.5, σ_0 and m can be calculated from slope and intercept respectively. According to equation (14), the simulated σ~ε curve is obtained and shown in Fig.4. The simulated results are well consistent with the experimental data.

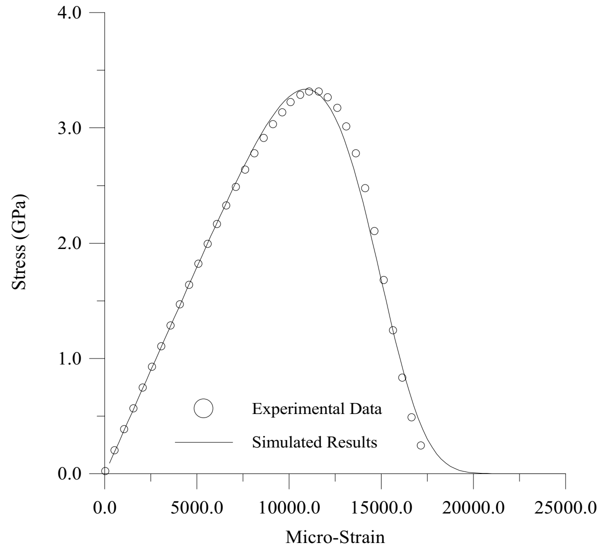


Fig.4 The tensile stress-strain curve

Table.1 Mechanical and Weibull distributing parameters for fibers and fiber bundles (L=10.0mm)

σ_{max}(GPa)	ε_b (%)	E_b(GPa)	L(mm)	m	σ_0(GPa • mm$^{1/m}$)	$\bar{\sigma}_f$(GPa)
3.34	1.10	359.0	10.0	6.08	2.53	5.06

Then the mean strength of the filament fiber with gauge length L=10.0mm can be calculated by eqn(7), that is $\bar{\sigma}_f$=5.06GPa.

The dimension of the fracture mirror of M40JB fiber, r_{mi} , can be directly measured with SEM, then A_m =4.114GPa.µm$^{1/2}$ can be calculated by eqn(18). A typical SEM photo of fracture mirror of the fiber is shown in Fig.6.

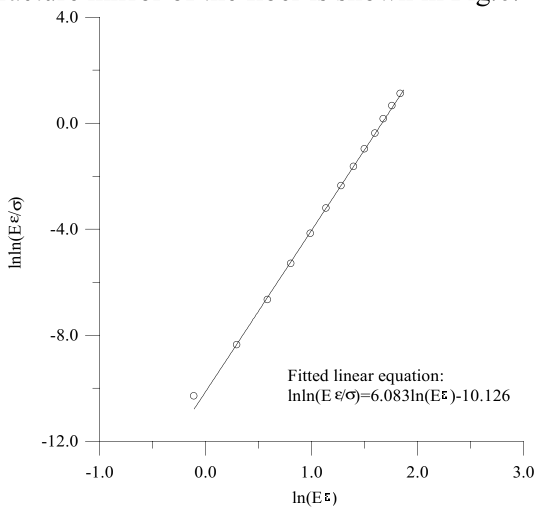


Fig.5. Weibull plots of the strength of the fibers

Fig.6 SEM photograph of fracture mirror

COMPOSITE SAMPLE

The M40JB/SiC composite sample, 2×5×50mm, was fractured in a three-point bending test of a chevron-notch specimen with the span of three points bending of 40 mm. The measurement was carried out on an MTS-810 tester with loading rate of 0.5 mm/min. The fracture sample exhibited extensive fiber pullout as shown in Fig.7.

THE IN-SITU STRENGTH OF THE M40JB FIBER

We have investigated 238 individual fibers in a single composite specimen. Measurements of the fracture mirror radii and pullout length of individual fiber were performed directly on SEM fractographs. The pullout length, Lp, distributes between 6.0μm and 280.0μm as observed. The ineffective length, ls=60.0μm was determined by numerical analysis[20]. In this paper, the gauge length of individual fiber is determined based on relation $L=2Lp+2l_S$ as mentioned above. The gauge length is graded and the corresponding average in-situ strength $\overline{\sigma}_{fi}$ is calculated. Those data are listed in Table 2 and also plotted on logarithmic coordinate system of $(ln\sigma, ln L)$ as shown in Fig.8, while σ_0 and m are calculated from the slope and intercept of the linear equation. The mean strength at 10.0mm gauge length can also be calculated by equation (7). Those data are listed in Table 3. The mean in-situ strength obtained by the modified method is 48.8 % of fabricated fiber strength (gauge length L=10.0mm). This indicates that fibers in this composite are probably weaker than that before incorporated into the composite structure.

As a comparison, the Weibull parameters and mean in-situ strength based on the unmodified (traditional) method are calculated and also listed in Table 3. Those results indicate that the Weibull shape parameters obtained by two methods are nearly same. However the scale parameter obtained by unmodified method is less than that of modified method. Thus the unmodified method may underestimate the in-situ strength of the fibers.

Table 2 The graded pullout length and in-situ strength

No.	Pullout length(μm)	In-situ strength(GPa)	No.	Pullout length(μm)	In-situ strength(GPa)	No.	Pullout length(μm)	In-situ strength(GPa)
1	340.0	3.92	**11**	149.8	4.71	**21**	89.9	4.45
2	330.0	3.36	**12**	138.2	4.31	**22**	87.0	4.43
3	300.0	3.36	**13**	129.6	4.41	**23**	84.0	4.73
4	272.5	3.50	**14**	120.8	4.80	**24**	82.0	4.07
5	237.5	5.03	**15**	114.8	4.18	**25**	80.1	4.57
6	220.0	3.45	**16**	108.5	4.60	**26**	78.0	4.53
7	209.0	4.49	**17**	105.1	4.51	**27**	76.1	4.55
8	193.3	4.47	**18**	100.6	4.45	**28**	72.7	4.11
9	175.0	4.53	**19**	95.8	4.05	**29**	69.6	4.21
10	163.8	4.47	**20**	93.0	4.43	**30**	66.0	4.17

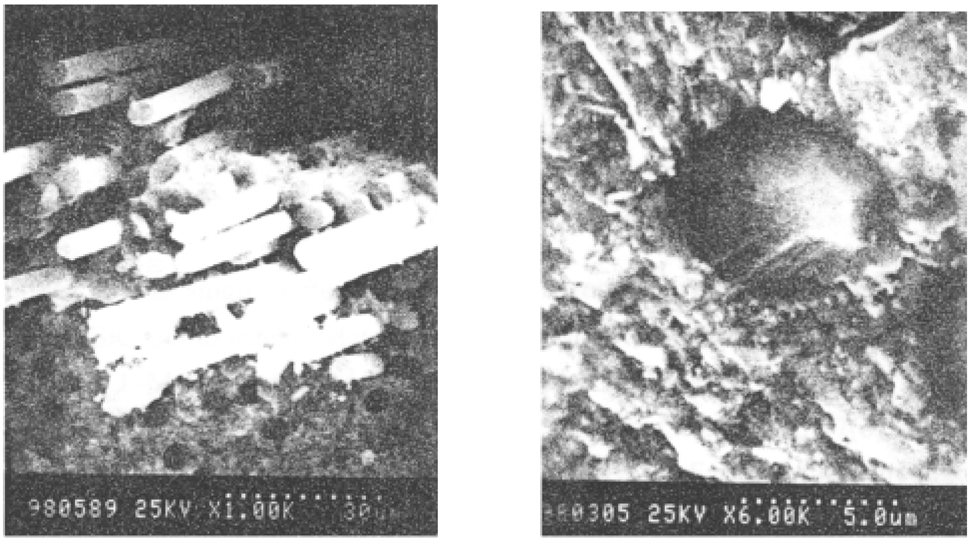
Table3 The comparison of two different assessments at same gauge length L=10.0mm

	M	σ_0(GPa • $\mu m^{1/m}$)	$\overline{\sigma}$(GPa)
Modified method	6.57	10.71	2.47
Unmodified method	6.33	8.61	1.89

CONCLUSIONS

(1) In this paper, the traditional fracture mirror method is modified by new definition of gauge length and grading statistic method of fiber pullout length. Thus the modified fracture mirror method can be also used to evaluate the in-situ strength of fibers in brittle failure composite which there is no fiber pullout and may obtain more reasonable results for the case of large dispersion of fiber pullout length.

(2) The Weibull shape parameters of fabricated M40JB fiber and in-situ fiber embedded in SiC matrix composite are nearly same, this indicates that the flaws distributions in the two conditions do not change obviously. The scale parameter of in-situ fiber strength is largely lower than that of the fabricated fiber, this indicates that the flaws are deeper in in-situ fiber than in fabricated fiber. It leads the degradation of in-situ strength.



(a) (b)

(a) The distribution of pullout lengths of fiber (b) Fracture mirror on break surface of fiber

Fig.7. The randomly distribution of pullout lengths and fracture mirror of fiber

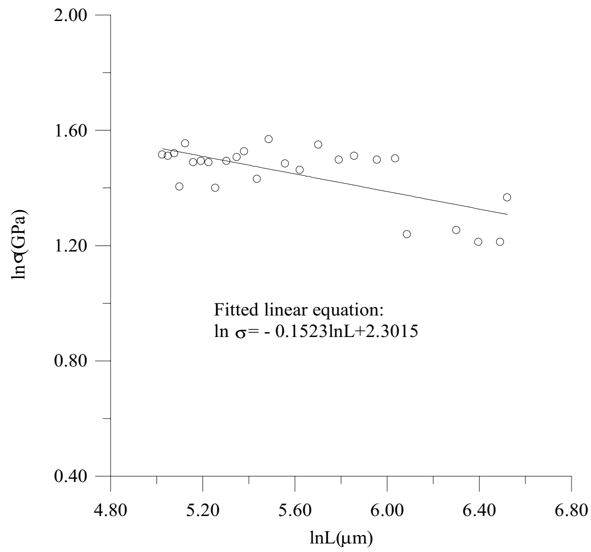


Fig.8. The relationship between in-situ strength and gauge length

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