JMEPEG (2006) 15:712-716
DOI: 10.1361/105994906X150768

1059-9495/$19.00

Flexural Strength Distribution of 3D SiC/SiC Composite

S. Wu, L. Cheng, N. Dong, L. Zhang, and Y. Xu

(Submitted December 3, 2005; in revised form December 18, 2005)

Flexural strength of a four-step, three-dimensional (3D) braiding SiC/SiC composite was tested at room temperature. The strength distribution was studied based on Weibull distribution and Normal distribution as well as examined by the Kolmogorov test. The results indicated that the flexural failure behavior of the composite was rather brittle with a small displacement. And the statistical strength distribution of the 3D SiC/SiC composite was in agreement with two-parameter Weibull distribution of the Weibull modulus, *m* = 8.1545 and normal distribution. And the predicated mean flexural strength of the 3D SiC/SiC composite by the two-parameter Weibull distribution was consistent with the tested value.

Keywords Kolmogorov test, normal distribution, SiC/SiC composite, strength distribution, Weibull distribution

1. Introduction

Continuous fiber reinforced ceramic matrix composites (CFCC) are considered as the most promising thermal structural materials due to their high toughness, good resistance to thermal shock, and good mechanical properties at high temperature, especially improved flaw tolerance and noncatastrophic mode of failure (Ref 1-3). High reliability is the primary requirement for CFCCs components, and therefore requires data and design methods based on reliability. Distribution functions are the fundamental component of reliability.

Strength distribution of ceramics or ceramic fibers has been studied widely. The failure probability of these brittle materials is normally described by the Weibull distribution (Ref 4-10). As for brittle fiber reinforced plastic matrix composites or plastic fiber reinforced brittle matrix composites, studies indicate that the strength distribution could be described by two-parameter Weibull distribution (Ref 10-12). Cattell and Kaushik (Ref 10) found that the strength data of E-glass epoxy composite fit a two-parameter Weibull distribution. Singh et al. (Ref 11) carried out a study on the fatigue strength of steel fiber reinforced concrete (SFRC) and demonstrated that the statistical distribution of equivalent fatigue-life of SFRC is in agreement with the two-parameter Weibull distribution.

Few works have been conducted on the strength distribution of two dimensional (2D) SiC/SiC composites (Ref 13, 14). These works demonstrated that the strength distribution of 2D SiC/SiC composites could be described using a Weibull distribution, but the strength distribution of three-dimensional (3D) SiC/SiC composites have rarely been reported. In the present study, the flexural strength of a 3D SiC/SiC composite prepared by a four-step three-dimensional (4-step 3D) braiding method was tested at room temperature, and the strength distribution of the composite was investigated based on the Weibull distribution and the normal distribution. The goodness of fit was then examined by means of the Kolmogorov statistical test.

Shoujun Wu, Laifei Cheng, Ning Dong, Litong Zhang, and **Yongdong Xu,** National Key Laboratory of Thermostructure Composite Materials, Northwestern Polytechnical University, Xi'an Shaanxi 710072, China. Contact e-mail: shoujun_wu@163.com.

2. Experimental Procedure

2.1 Preparation of 3D SiC/SiC Composite Specimens

Hi-Nicalon silicon carbide fiber from Japan Nippon Carbon (Tokyo, Japan) was used. The fiber preform was prepared using 4-step 3D braiding method, and was supplied by the Nanjing Institute of Glass Fiber, People's Republic of China. A low pressure chemical vapor infiltration (LPCVI) process was used to deposit pyrolytic carbon interphase and the silicon carbide matrix, which has been described earlier in detail (Ref 15, 16). The volume fraction of fibers was about 40% and the braiding angle was about 20°. The interfacial layer of pyrocarbon (PyC) was deposited for 1 h at 870 °C and 5 kPa with C_3H_6. The deposited PyC interphase layer was about 0.2 μm. Methyltrichlorosilane (MTS, CH_3SiCl_3) was used for the deposition of the SiC matrix. MTS vapor was carried by bubbling hydrogen. The conditions for deposition of SiC matrix were as follows: the deposition temperature 1100 °C, pressure 5 kPa, time 120 h, and a molar ratio of H_2 to methyltrichlorosilane (MTS) of 10. Argon was used as the dilute gas to slow down the chemical reaction rate of deposition. Specimens with dimension of 2.45 × 4.30 × 40.30 mm were machined from the as-received composite and then polished. The density of the as-received composites samples was 2.67 g/cm^{-3} as determined by Archimedes' method. A chemical vapor deposition (CVD) SiC coating was prepared on the substrates for 20 h to seal the open ends of the fibers after cutting from the prepared composite.

2.2 Three-Point Flexural Strength Test

Three-point flexural tests were carried out on SANS CMT4304 universal testing machine (Shenzhen SANS Testing Machine Co. Ltd., Shenzhen Guang Dong, China) at room temperature. The span dimension was 30 mm and the loading rate was 0.5 mm/min^{-1}. The flexural strength σ was calculated by:

$$\sigma = \frac{3}{2}\frac{P_{\max} L}{bh^2} \qquad \text{(Eq 1)}$$

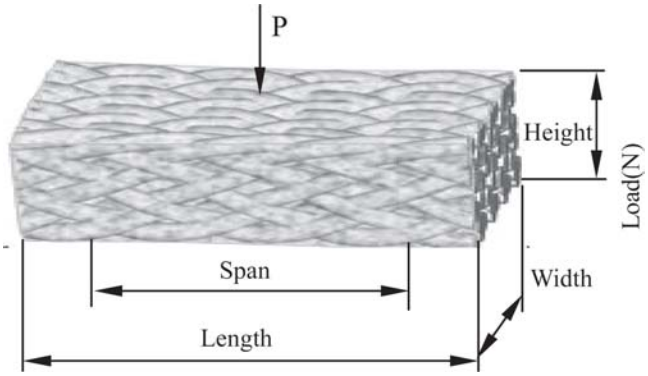

Fig. 1 Schematic diagram of the specimen for flexural strength test

where L, b, and h are the span, thickness, and height of specimens, respectively. P_{max} is the maximum flexural load during the test. A schematic diagram of the specimen for flexural strength test is shown in Fig. 1. The micromorphologies of the composite before and after the flexural test were observed using the scanning electron microscope (SEM).

3. Strength Distribution Model for the 3D SiC/SiC Composite

To investigate the strength distribution of the as-received 3D SiC/SiC composite, Weibull distribution and normal distribution were selected. The parameters of each distribution model were defined by a graphical method for Weibull distribution and the maximum likelihood method for normal distribution. Their suitability to describe the properties was tested by the Kolmogorov test.

3.1 Parameters Estimation

To obtain the parameters of each distribution and to examine the validity of the distribution by the Kolmogorov test, the test data were arranged in ascending order. Then the failure probability could be estimated from (Ref 12, 17):

$$S = \frac{n - 0.5}{N} \quad \text{(Eq 2)}$$

where n is the ranking number of the strength and N the number of specimens.

For Weibull distribution, the failure probability is given by the relation (Ref 5):

$$S = 1 - \exp\left[-V \times \left(\frac{\sigma - \sigma_u}{\sigma_0}\right)^m\right] \quad \text{(Eq 3)}$$

where S is the failure probability, σ is the failure stress; σ_u is the threshold stress below which the samples will not fail; σ_0 is the characteristic stress. The characteristic strength is a normalizing parameter that relates strength for a given probability of survival to the effective volume under load. V is the effective volume of the samples that indicates those segments that may fail under the failure stress σ. For three-point flexural test, V is described as follows (10):

$$V = \frac{V_t}{2(m+1)^2} \quad \text{(Eq 4)}$$

where V_t is the whole volume of the sample, m is the Weibull modulus. Higher values of the modulus, m, indicate less scatter in the data and greater confidence in the reliability of the material.

Taking the logarithm twice of both sides of the Eq 3:

$$\text{lglg}\,\frac{1}{1-S} = \text{lg}0.4343 + \text{lg}V + m\text{lg}(\sigma_n - \sigma_u)\text{-}m\text{lg}\sigma_0 \quad \text{(Eq 5)}$$

According to Eq 5, if the strength distribution agrees with Weibull distribution, the lglg 1/1 − S will be a straight line when plotted against lg(σ_n−σ_u), and the slope of the line is the Weibull modulus, m. The intercept in lglg 1/1 − S is equal to lg0.4343 + lg$V$–$m$lgσ_0.

For the normal distribution, the failure probability is given by:

$$S = 1 - \int_{-\infty}^{+\infty} \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(x-\mu)^2}{2\sigma^2}\right] dx = 1 - N(\mu, \sigma) \quad \text{(Eq 6)}$$

and the parameters obtained by the maximum likelihood method are given by:

$$\mu = m' \qquad \sigma = S_n \quad \text{(Eq 7)}$$

where m' and S_n is the sample mean and standard deviation, respectively, and n is the sample size.

3.2 Kolmogorov Test

To test the validity of the approximation of each distribution, the Kolmogorov test is used. The basic procedure of the test involves the comparison between the experimental cumulative frequency and the assumed theoretical cumulative distribution function. If the discrepancy between these two is large, compared with what is normally expected from the given sample size, the theoretical model is rejected. A numerical algorithm was used to derive the sample statistic D_n and this value was then compared with the critical value $D_{a,n}$ which is defined by the probability:

$$P(D_n \leq D_{a,n}) = 1 - a \quad \text{(Eq 8)}$$

where a is the significance level, and n is the sample size. If the observed D_n exceeds $D_{a,n}$ then the tested distribution is rejected at the specified significance level a.

4. Results and Discussion

4.1 Flexural Failure Behavior of the 3D SiC/SiC Composite

Figure 2 shows the typical failure behaviors of the 3D SiC/SiC composite in flexural strength tests at room temperature. It can be seen that the failure behavior of the SiC/SiC composite was rather brittle and exhibited steep stress drops after the maximum stress point with a relatively small displacement. Figure 3 shows the typical cross-section morphology of the 3D

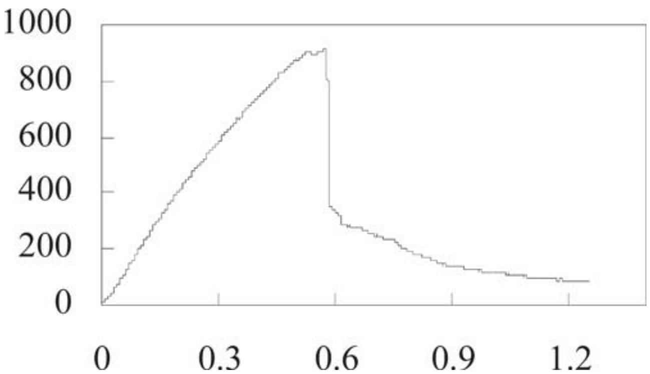

Fig. 2 Typical failure behaviors of the 3D SiC/SiC composite in flexural strength tests

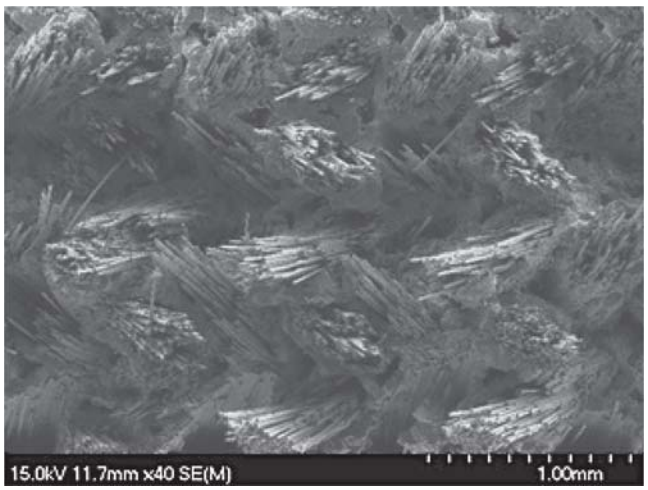

Fig. 3 Typical cross-section morphology of the 3D SiC/SiC composites after flexural test at room temperature

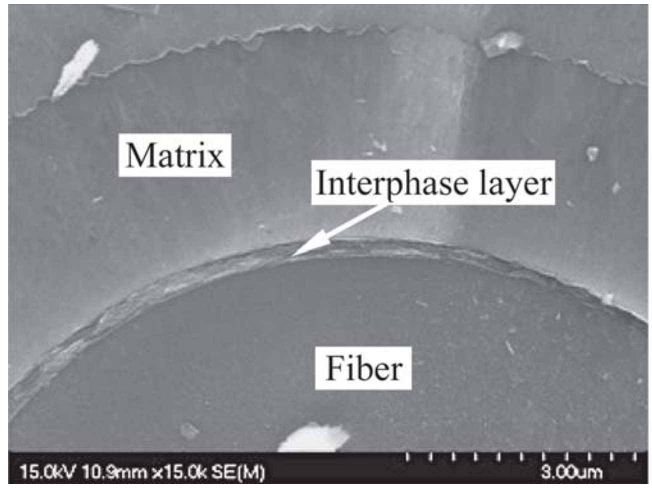

Fig. 4 Typical micromorphology of fiber/interphase/matrix in the as-received 3D SiC/SiC composite

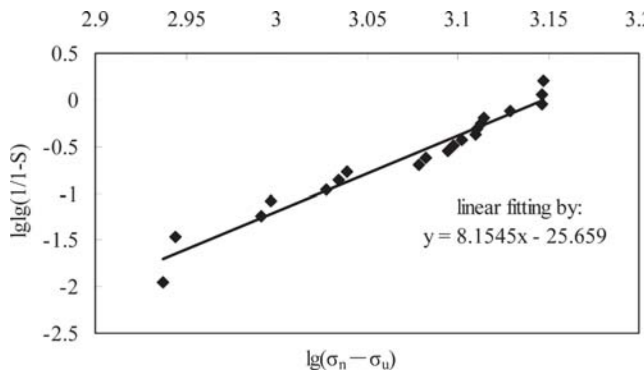

Fig. 5 Plot of lglg 1/1 − S to lg(σ_n–σ_u) at $\sigma_u = 0$ MPa

SiC/SiC composites after flexural testing at room temperature. The reinforcement fiber pullout is mainly in fiber bundles rather than in single fibers.

The coefficient of thermal expansion (CTE) of Hi-Nicalon SiC fiber and SiC matrix was about 3.1~3.5 × 10^{-6}/°C and 4.6 × 10^{-6}/°C (Ref 2, 18, 19), respectively. Hence, a compressive stress within the interfacial phase along the fiber radial direction was generated after the composite cooled from the infiltration temperature (1100 °C) to room temperature. From the typical micromorphology of fiber/interphase/matrix in the as-received 3D SiC/SiC composite as shown in Fig. 4, it was clear that the PyC interphase was closely bonded to both CVI SiC matrix and Hi-Nicalon fiber. It was difficult for the Hi-Nicalon SiC fiber to debond and pull away from the silicon carbide matrix as single fibers. Thus, the failure of the 3D SiC/SiC composite in flexural strength tested at room temperature exhibited a small displacement.

4.2 Graphical Method for Weibull Distribution Analysis

Figure 5 shows the plots of experimental flexural strength data at $\sigma_u = 0$ MPa. It can be seen that the data points fall approximately along a straight line, which indicates that the two-parameter Weibull distribution is a reasonable assumption for the flexural strength distribution of as-received 3D SiC/SiC composites. Table 1 presents the basic calculations to plot the experimental flexural strength data of the as-received 3D SiC/SiC composite. From Fig. 5 the Weibull modulus (m) is 8.1545, and the intercept (lglg 1/1 − S) is −25.659. The calculated parameters: $\sigma_0 = 1418.035$ MPa, $V = 2.53303$ mm^3, so the distribution function is:

$$S = 1 - \exp\left[-2.533\,03 \times \left(\frac{\sigma - 0}{1418.035}\right)^{8.1545}\right] \qquad \text{(Eq 9)}$$

The Weibull modulus of the SiC/SiC composite is 8.1545, which is somewhat small for fiber-reinforced composite. This value also indicates that the flexural strength of the SiC/SiC composite has large scatter. In the SiC/SiC composite, the PyC interphase was closely bonded to both CVI SiC matrix and Hi-Nicalon fiber. It is hypothesized that this microcharacter of the SiC/SiC composite was also responsible for the low Weibull modulus.

4.3 Kolmogorov Test and Strength Prediction

The Kolmogorov test was performed for a significance level $a = 5\%$. Table 2 presents the calculated D_n of the Kolmogorov test for the Weibull distribution and normal distribution. For the significance level $a = 5\%$ and sample size $n = 20$, the critical value $D_{0.05,20}$ is 0.29403, which is larger than the cal-

Table 1 Basic calculations to Weibull parameters estimation by graphical method at $\sigma_u = 0$ MPa

Number n	Break strength σ_n, MPa	$\lg\lg\frac{1}{1-S}$	$\lg(\sigma_n - \sigma_u)$	Number n	Break strength σ_n, MPa	$\lg\lg\frac{1}{1-S}$	$\lg(\sigma_n - \sigma_u)$
1	865.9	−1.95879	2.937468	11	1252.7	−0.49039	3.097847
2	879.4	−1.47034	2.944186	12	1265.5	−0.42991	3.102262
3	981.6	−1.23663	2.991935	13	1288.1	−0.37062	3.10995
4	993.6	−1.07807	2.997212	14	1291.1	−0.31148	3.11096
5	1066	−0.95586	3.027757	15	1296.9	−0.25129	3.112906
6	1081.5	−0.85492	3.034027	16	1300.9	−0.18855	3.114244
7	1093.9	−0.76778	3.038978	17	1346.5	−0.12093	3.129206
8	1199	−0.69011	3.078819	18	1401.7	−0.04427	3.146655
9	1208	−0.61919	3.082067	19	1402.1	0.051129	3.146779
10	1242.7	−0.55309	3.094366	20	1403.6	0.204679	3.147243

Table 2 Basic calculations to Kolmogorov test for Weibull and normal distribution at $a = 5\%$, $n = 20$

Number n	Break strength σ_n, MPa	Empirical failure probability $S\ \frac{n-0.5}{N}$	Weibull distribution		Normal distribution	
			Failure probability S_W	$\lvert S_W - S\rvert$	Failure probability S_N	$\lvert S_N - S\rvert$
1	865.9	0.025	0.044358	0.019358	0.022846	0.002154
2	879.4	0.075	0.05017	0.02483	0.027699	0.047301
3	981.6	0.125	0.118531	0.006469	0.098267	0.026733
4	993.6	0.175	0.130039	0.044961	0.112196	0.062804
5	1066	0.225	0.219019	0.005981	0.218869	0.006131
6	1081.5	0.275	0.242768	0.032232	0.245274	0.029726
7	1093.9	0.325	0.263007	0.061993	0.272413	0.052587
8	1199	0.375	0.475244	0.100244	0.514574	0.139574
9	1208	0.425	0.496095	0.071095	0.53638	0.11138
10	1242.7	0.475	0.57828	0.10328(D_n^W)	0.619181	0.14418(D_n^W)
11	1252.7	0.525	0.602169	0.077169	0.642248	0.117248
12	1265.5	0.575	0.632634	0.057634	0.670951	0.095951
13	1288.1	0.625	0.685538	0.060538	0.719235	0.094235
14	1291.1	0.675	0.692429	0.017429	0.725375	0.050375
15	1296.9	0.725	0.705636	0.019364	0.737122	0.012122
16	1300.9	0.775	0.71465	0.06035	0.745041	0.029959
17	1346.5	0.825	0.810017	0.014983	0.825729	0.000729
18	1401.7	0.875	0.900208	0.025208	0.898782	0.023782
19	1402.1	0.925	0.900742	0.024258	0.899197	0.025803
20	1403.6	0.975	0.90273	0.07227	0.900798	0.074202

culated $D_n^W = 0.10328$ for Weibull distribution and $D_n^N = 0.144181$ for normal distribution. The Kolmogorov test indicates that both distributions could be used to describe the flexural strength distribution of the as-received composite.

From Eq 1, the density distribution function of Weibull distribution is:

$$f(\sigma) = \frac{dS}{d\sigma} = \frac{m\,v}{\sigma_0} \cdot \left(\frac{\sigma - \sigma_u}{\sigma_0}\right)^{m-1} \exp\left[\left(\frac{\sigma - \sigma_u}{\sigma_0}\right)^{m}\right] \quad \text{(Eq 10)}$$

and the mean strength value calculated via Weibull distribution Eq 3 is:

$$\begin{aligned}\overline{\sigma} &= \int_{\sigma_u}^{\infty} \sigma \cdot f(\sigma) \cdot d\sigma \\ &= \sigma_0 V^{-\frac{1}{m}} \Gamma\left(1 + \frac{1}{m}\right) - \sigma_u \int_{\sigma_0}^{\infty} e^{-x}\, dx \end{aligned} \quad \text{(Eq 11)}$$

With the parameters $m = 8.1545$, $\sigma_0 = 1418.035$ and $V = 2.53303$, according to Eq 11, the mean strength from Weibull distribution is 1192.7 MPa, which is close to experimental mean value.

5. Conclusions

According to the Kolmogorov test combined with the flexural strength value by test data and predicted from the conformable distribution, strength distribution of a 3D SiC/SiC composite was investigated. The results indicated that the flexural failure behavior of the composite is rather brittle with a small displacement and the strength of it in three-point flexural tests could be described by either the two-parameter Weibull distribution or the normal distribution. The corresponding Weibull parameters for the composite are as follows: $m = 8.1545$, $\sigma_0 = 1418.035$. And the flexural strength of 3D SiC/SiC composite at room temperature could be accurately predicted using the two-parameter Weibull distribution model.

Acknowledgments

The authors acknowledge the support of the Chinese National Foundation for Natural Sciences under Contract No.90405015 and the NSFC Distinguished Young Scholar under Contract No.50425208, 2004.

References

1. B.N. Cox and F.W. Zok, Advances in Ceramic Composites Reinforced by Continuous Fibers, *Curr. Opin. Solid State Mater. Sci.,* 1996, **1**(5), p 666-673
2. R. Naslain, Design, Preparation and Properties of Non-Oxide CMCs for Application in Engines and Nuclear Reactors: An Overview, *Compos. Sci. Technol.,* 2004, **64**(2), p 155-170
3. S. Schmidt, S. Beyer, H. Knabe, H. Immich, R. Meistring, and A. Gessler, Advanced Ceramic Matrix Composite Materials for Current and Future Propulsion Technology Applications. *Acta Astronaut.,* 2004, **55**(3-9), p 409-420
4. S.L. Fok, B.C. Mitchell, J. Smart, and B.J. Marsden, A Numerical Study on the Application of the Weibull Theory to Brittle Materials, *Eng. Fract. Mech.,* 2001, **68**(10), p 1171-1179
5. J. Lamon, Statistical Approaches to Failure for Ceramic Reliability Assessment, *J. Am. Ceram. Soc.,* 1998, **71**(2), p 106-112
6. J.C. Fernandes, P.M. Amaral, L.G. Rosa, and N. Shohoji, Weibull Statistical Analysis of Flexure Breaking Performance for Alumina Ceramic Disks Sintered by Solar Radiation Heating, *Ceram. Int.,* 2003, **26**(2), p 203-206
7. C.-Y. Hui, S.L. Phoenix, and D. Shia, The Single-Filament-Composite Test: A New Statistical Theory for Estimating the Interfacial Shear Strength and Weibull Parameters for Fiber Strength, *Compos. Sci. Technol.,* 1998, **57**(12), p 1707-1725
8. J. Andersons, R. Joffe, M. Hojo, and S. Ochiai, Glass Fibre Strength Distribution Determined by Common Experimental Methods, *Compos. Sci. Technol.,* 2002, **62**(1), p 131-145
9. K.L. Pickering and T.L. Murray, Weak Link Scaling Analysis of High-Strength Carbon Fiber. *Compos. Part A-Appl. S.,* 1999, **30**(8), p 1017-1021
10. M.K. Cattell and K.A. Kibble, Determination of the Relationship between Strength and Test Method for Glass Fibre Epoxy Composite Coupons using Weibull Analysis, *Mater. Des.,* 2001, **22**(4), p 245-250
11. S.P. Singh and S.K. Kaushik, Fatigue Strength of Steel Fibre Reinforced Concrete in Flexure, *Cement. Concrete. Comp.,* 2003, **25**(7), p 779-786
12. T.P. Philippidis, D.J. Lekou, and D.G. Aggelis, Mechanical Property Distribution of CFRP Filament Wound Composites, *Compos. Struct.,* 1999, **45**(1), p 41-50
13. V. Calard and J. Lamon, A Parabolic-Statistical Approach to the Ultimate Failure of Ceramic-Matrix Composite-Part I: Experimental Investigation of 2D Woven SiC/SiC Composites, *Compos. Sci. Technol.,* 2002, **62**(3), p 385-393
14. H. Araki, T. Noda, W. Yang, and A. Kohyama, Homogeneity and Flexural Properties of SiC/SiC Composites Prepared by CVI Method, *J. Nucl. Mater.,* 2002, **307-311**(part 2), p 1210-1214
15. Y.D. Xu, L.T. Zhang, L.F. Cheng, and D.T. Yan, Microstructure and Mechanical Properties Of Three-Dimensional Carbon/Silicon Carbide Composites Fabricated by Chemical Vapor Infiltration, *Carbon,* 1998, **36**(7-8), p 1051-1056
16. Y.D. Xu and L.T. Zhang, Three Dimensional C/SiC Composites Prepared by Chemical Vapor Infiltration, *J. Am. Ceram. Soc.,* 1997, **80**(7), p 1897-1900
17. M.F. Burrow, D. Thomas, M.V. Swain, and M.J. Tyas, Analysis of Tensile Bond Strengths Using Weibull Statistics, *Biomaterials,* 2004, **25**(20), p 5031-5035
18. H. Ichikawa, Recent Advances in Nicalon Ceramic Fibres Including Hi-Nicalon Type S, *Ann. Chim. Sci. Mat.,* 2000, **25**(7), p 523-528
19. W. Yang, A. Kohyama, T. Noda, Y. Katoh, T. Hinoki, H. Araki, and J. Yu, Interfacial Characterization of CVI-SiC/SiC Composites, *J. Nucl. Mater.,* 2002, **307-311**(part 2), p 1088-1092