PREDICTION OF THE MECHANICAL BEHAVIOR OF A MINICOMPOSITE BASED ON GREY VERHULST MODELS

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Submitted April 13, 2017; accepted June 2, 2017

Keywords: C/SiC, Tensile behavior, Acoustic emission, Grey systems

The tensile behavior of a C/SiC minicomposite fabricated by chemical vapor infiltration was examined and the associated damage evolution was monitored by using acoustic emission (AE) technique. Both the tensile behavior and the associated AE evolution of the minicomposites can be well characterized by three regimes including matrix cracking emergence, multiplication followed by matrix cracking saturation. The grey Verhulst model and inverse Verhulst model were established for the evolution of the cumulative AE energy and the stress-strain behavior, respectively. Excellent agreement between predictions and experimental results was obtained for both the grey models, which indicates the nature of intraspecific competition for these matrix cracks in the minicomposites studied. Such robust relationships can be further applied to model the constitutive behaviour of other ceramic composite systems.

INTRODUCTION

Ceramic matrix composites (CMCs) have been proposed for decades as thermostructural materials suitable for aerospace and aeronautical engine hot-section components like nozzle extensions, air mixers, etc. [1, 2]. However, most current CMCs consist of multiple tows structured in different directions and complex arrangements such as woven, braided, needled and stitched to meet the mechanical requirements along certain directions [3]. The densification of these multi-dimensional fiber performs is essentially time consuming, regardless of the fabrication techniques, e.g. chemical vapour infiltration (CVI) or polymer impregnation and pyrolysis (PIP). Thus the mechanical modeling and processing optimization for these CMCs seems to be very inconvenient.

Fortunately, a cost-effective approach has been developed most recently by using minicomposites [4]. A large number of specimens can be fabricated at relatively low cost with short time, which makes it attractive for obtaining a robust set of experimental data. Since the minicomposite represents a subelement (infiltrated longitudinal bundles) of these real macrocomposites in loading direction, the tensile behaviour of minicomposites is believed to mimic that of their multidirectional counterparts, because the interfacial and elastic properties of the constituents are the same.

EXPERIMENTAL

Composite fabrication

The carbon fiber utilized was T300 (Nippon Toray Co., Japan), and each bundle contained 3000 fibers (with a filament diameter of 7 μm). The continuous
as-received multifilament bundle was wrapped on a graphite frame with a width of 80 mm to ensure that the gauge length of minicomposites is at least 50 mm. The C/SiC minicomposite was fabricated by isothermal CVI to deposit the pyrocarbon (PyC) interphase and subsequent SiC matrix. Methyltrichlorosilane (MTS, CH₃SiCl₃) was used for the deposition of the SiC matrix. MTS vapor was carried by bubbling hydrogen. The processing conditions for minicomposites are the same as for the real macrocomposites, except that the infiltration time of SiC matrix is about 1/4 of that of macrocomposites in order to avoid over-thick SiC sheath around the minicomposites. The volume fractions of fiber of each specimen were determined from the weights measured on minicomposites and on reference fiber bundles, leading to an average volume fraction of fibers \( V_f \approx 0.3 \).

The ends of specimens were mounted with epoxy to aluminum tabs. An alligator clip on every side of the tabs was used to facilitate handling of specimens until its removal before tensile tests. Much care was taken to avoid the premature or unexpected failure of the specimens.

**RESULTS AND DISCUSSION**

Microstructure characterization

A typical cross-section of C/SiC minicomposites is shown in Figure 1. It can be seen that the cross-section is close to elliptical, with the relatively thick CVI-SiC sheath surrounding the bundle. It can also be seen from the longitudinal surface as shown in Figure 2 that the outer SiC sheath was actually formed by relatively uniform SiC particles with the diameter of about 100 μm which were finally deposited by the current CVI process. Based on the image analysis using Photoshop software, the minicomposites studied have a cross-sectional area of 0.35 - 0.42 mm². This is consistent with the results determined from the weights measured on minicomposites and on reference fiber bundles.

After tensile tests, an example of polished longitudinal gauge sections of C/SiC minicomposites was shown in Figure 3. It is evident that the multiple matrix cracks crossing the entire minicomposites are densely spaced and of relatively uniform intervals, which is indicative of the matrix cracking saturation achieved for the minicomposites studied.

![Figure 1. Typical micrograph of the cross-section of minicomposites.](image-url)
matrix cracks newly formed and/or propagating through-the-diameter of the minicomposites, i.e. multiplication of the matrix microcracking and associated interface debonding until saturation.

The last part of the stress-strain curve displays a slope recovery and the rate of cumulative AE energy actually starts to diminish to nearly plateau with increasing applied load until fracture. This corresponds to the matrix cracking saturation and elongation of the remained intact fibers, which are carrying the entire load. Rupture then occurs at an average stress of 350 MPa for a corresponding average strain of 0.66%.

The above-mentioned domains exhibited in both the stress-strain curve and the accumulation of AE energy curve including matrix cracking emergence, multiplication and saturation, are very similar to the development of the self-limiting growth of a biological population, which is often described by the Verhulst equation, namely the law of population growth as follows

\[
\frac{dN}{dt} = rN - aN^2
\]

where \( N \) represents number of individuals at time \( t \), \( r \) the intrinsic growth rate, and \( a \) is the density-dependent crowding effect (also known as intraspecific competition). In this equation, the population equilibrium (the so-called carrying capacity) \( K \) can be derived as

\[
K = \frac{L}{a}
\]

In the minicomposites studied, the early, unimpeded matrix crack growth rate can be modeled by the first term \( rN \). The value of the rate \( r \) represents the proportional increase of the crack number \( N \). Later, as the damages evolve, the effect of the second term becomes almost as large as the first, as some members of the matrix cracks interfere with each other by competing for the critical matrix space. This antagonistic effect is called the bottleneck, and can be modeled by the value of the parameter of the carrying capacity. The competition...
diminishes the combined growth rate, until the number of the matrix cracks ceases to grow, i.e. the domain of the matrix cracking saturation corresponding to the so-called maturity of the population.

Since the amount of cumulative AE energy is nearly directly related to the number of matrix cracks formed, it can also be described by the Verhulst equation as follows:

\[ \frac{dE(t)}{d\varepsilon} = aE(t) - bE(t)^2 \]  \hspace{1cm} (3)

where \( E(t) \) is the amount of cumulative AE energy, \( \varepsilon \) is the tensile strain. Thus its grey difference equation in the discrete form can be described as

\[ E^{(i)}(k) = aE^{(i)}(k-1) - bE^{(i)}(k-1)^2 \]  \hspace{1cm} (4)

where \( E^{(i)}(k) = [E^{(i)}(k) - E^{(i)}(k-1)]/(\varepsilon(k) - \varepsilon(k-1)) \), \( k \) is the sequential number of measured data and \( k = 2, 3, ..., n \). And the sequence \( Z(k) \) can be obtained by using the mean consecutive neighbors operator for \( E(t) \) as follows

\[ Z(k) = \frac{1}{2} [E^{(i)}(k) + E^{(i)}(k-1)], \quad k = 2, 3, n. \]  \hspace{1cm} (5)

The parameters of \( a \) and \( b \) can be estimated as follows by minimizing the squared-errors, i.e., least squares method:

\[
\begin{bmatrix} \hat{a} \\ \hat{b} \end{bmatrix} = (B^T B)^{-1} B^T Y
\]  \hspace{1cm} (6)

where

\[
B = \begin{bmatrix} z(2) & -z(2)^2 \\ \vdots & \vdots \\ z(n) & -z(n)^2 \end{bmatrix}
\]  \hspace{1cm} (7)

\[
Y = \begin{bmatrix} E^{(0)}(0) \\ E^{(0)}(1) \\ \vdots \\ E^{(0)}(n-1) \end{bmatrix}
\]  \hspace{1cm} (8)

Based on the estimated parameters, the AE energy response function can be obtained by solving Equation (3), as shown below:

\[ E^{(i)} = \frac{a}{1 + \left( \frac{a}{1 + bE^{(i)}} - 1 \right) e^{-a(\varepsilon(k) - \varepsilon(1))}} \]  \hspace{1cm} (9)

where the initial condition of the model is set as \( \varepsilon = \varepsilon(1), \quad E = E^{(i)}(1) \), namely using the first measured data point.

The measured cumulative AE energy versus strain at fixed intervals are listed in Table 1. The strain can be normalized by the true tensile strain divided by 0.05 % for mathematical convenience, leading to \( \varepsilon(k) \) equal to the sequence number \( k \).

Therefore the response of the cumulative AE energy versus strain can be predicted as follows based on the grey verhulst model above and experimental data in Table 1,

\[ E^{(i)}(k) = \frac{6.81}{1 + 22.49 e^{-0.4838(k-1)}} \]  \hspace{1cm} (10)

where \( a = 0.4838, \quad b = 0.071 \).

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**Table 1.** Measured cumulative AE energy at regular strain intervals.

<table>
<thead>
<tr>
<th>Sequence number</th>
<th>Tensile strain (%)</th>
<th>AE energy ((\times 10^5 \text{ mV}^2 \text{s}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.1</td>
<td>0.29</td>
</tr>
<tr>
<td>2</td>
<td>0.15</td>
<td>0.81</td>
</tr>
<tr>
<td>3</td>
<td>0.2</td>
<td>1.48</td>
</tr>
<tr>
<td>4</td>
<td>0.25</td>
<td>2.12</td>
</tr>
<tr>
<td>5</td>
<td>0.3</td>
<td>2.78</td>
</tr>
<tr>
<td>6</td>
<td>0.35</td>
<td>3.51</td>
</tr>
<tr>
<td>7</td>
<td>0.4</td>
<td>4.18</td>
</tr>
<tr>
<td>8</td>
<td>0.45</td>
<td>5.14</td>
</tr>
<tr>
<td>9</td>
<td>0.5</td>
<td>5.59</td>
</tr>
<tr>
<td>10</td>
<td>0.55</td>
<td>5.93</td>
</tr>
<tr>
<td>11</td>
<td>0.6</td>
<td>6.23</td>
</tr>
<tr>
<td>12</td>
<td>0.65</td>
<td>6.53</td>
</tr>
</tbody>
</table>

---

The predicted cumulative AE energy curve is shown in Figure 5 along with the experimental data. It can be seen that an evident deviation exits between the prediction and experimental data except the first point. This is due to an improper setting of initial condition to Equation 3, since there is no sufficient evidence that using the first raw data would lead to higher prediction accuracy. Consequently the initial condition should be optimized to achieve a better agreement. For convenience the Equation 9 can be rewritten as

\[ \hat{E}^{(i)}(k) = \frac{1}{b + c e^{-a(\varepsilon(k) - \varepsilon(1))}} \]  \hspace{1cm} (11)

where \( \hat{E}^{(i)}(k) \) is the predicted cumulative AE energy, \( c \) is the parameter reflecting the initial condition, which can be optimized by minimize the sum of squared errors of the reciprocals as follows
min \( Q = \min \sum_{k=2}^{n} \left( \frac{1}{E^{(1)}_{0(k)}} - \frac{1}{E^{(1)}_{0(k)}} \right)^2 \) (12)

\[ = \min \sum_{k=2}^{n} \left( \frac{b}{a} + c e^{-a(\epsilon(k) - \epsilon(1))} - \frac{1}{E^{(1)}_{0(k)}} \right)^2 \]

The optimized parameter \( c \) can be obtained through

\[ \frac{dQ}{dc} = 0 \] (13)

And the result is

\[ c = \sum_{k=2}^{n} \frac{1}{E^{(1)}_{0(k)}} \frac{b}{a} e^{-a(\epsilon(k) - \epsilon(1))} \]

\[ \sum_{k=2}^{n} e^{-2a(\epsilon(k) - \epsilon(1))} \]

(14)

Based on the measured data in Table 1, the parameter \( c \) is finally optimized as 1.6289, and the corresponding curve is also shown in Figure 5. It can be seen that the predicted curve exhibits an excellent agreement with the actual data. Furthermore, the prediction accuracy can also be improved by another simple method, where the initial condition was optimized and set as the inverse function of Verhulst equation and described as

\[ \sigma = 50 \left[ \frac{1}{a} \ln \left( \frac{a - b \epsilon^{(1)}_{0}}{a - b \epsilon^{(1)}_{0}} + 1 \right) \right] + \sigma(4) \] (17)

where \( k \) is also the sequential number and \( \sigma_{(k)} \) was normalized by the true tensile stress divided by 50 MPa, leading to \( \sigma_{(0)} \) equal to \( k \).

<table>
<thead>
<tr>
<th>Sequence number</th>
<th>Tensile strain (%)</th>
<th>Tensile stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.03</td>
<td>50</td>
</tr>
<tr>
<td>2</td>
<td>0.07</td>
<td>100</td>
</tr>
<tr>
<td>3</td>
<td>0.13</td>
<td>150</td>
</tr>
<tr>
<td>4</td>
<td>0.34</td>
<td>200</td>
</tr>
<tr>
<td>5</td>
<td>0.49</td>
<td>250</td>
</tr>
<tr>
<td>6</td>
<td>0.58</td>
<td>300</td>
</tr>
<tr>
<td>7</td>
<td>0.66</td>
<td>350</td>
</tr>
</tbody>
</table>

Based on the measured sequential strain-stress data in Table 2, the parameters of \( a \) and \( b \) were estimated as

\[ \left[ \begin{array}{c} \hat{a} \\ \hat{b} \end{array} \right] = \left[ \begin{array}{c} 1.0382 \\ 1.5262 \end{array} \right] \]

Finally, the stress-strain curve can be obtained by the inverse function of Verhulst equation and described as

\[ \sigma = 50 \left( \frac{1}{a} \ln \left( \frac{a - b \epsilon^{(1)}_{0}}{a - b \epsilon^{(1)}_{0}} + 1 \right) \right) + \sigma(4) \] (17)

where the initial condition was optimized and set as the fourth measured data point.

Both the predicted strain-stress curve and the stress-strain curve are shown in Figure 6, together with the increase of AE energy corresponds to the first domain of the stress-strain curve with a slightly nonlinear behavior. The domain of matrix cracking multiplication with a rapid increase of AE energy corresponds to the middle domain of the stress-strain curve with a largely nonlinear behavior. And the domain of matrix cracking saturation with a near plateau of AE energy corresponds to the final domain of the stress-strain curve with an obvious slope recovery. Consequently, the constitutive law of the minicomposites in this paper is also subject to the Verhulst equation, which is confirmed by strains plotted as a function of tensile stress as shown in Figure 6. It is evident that the strain-stress curve exhibits the very similar evolution to that of the AE energy curve. Therefore the grey Verhulst method can be also used to model the constitutive law as follows

\[ \frac{d\epsilon^{(1)}_{k}}{d\sigma} = a \frac{\epsilon^{(1)}_{k}}{a - b \epsilon^{(1)}_{k}} \]

And the final constitutive law can be described as

\[ \epsilon^{(1)}_{k} = \frac{a \epsilon^{(1)}_{k}}{1 + \left( \frac{a}{b \epsilon^{(1)}_{k}} - 1 \right) e^{-a(\epsilon(k) - \epsilon(1))}} \] (16)

Figure 6. Prediction and measured constitutive behavior of the minicomposites. Experimental data are represented by solid lines, while curves with dash lines indicate the predictions by grey Verhulst model and inverse model. Also shown is the predicted carrying capacity of strain.

Table 2. Measured tensile strain-stress data in equidistant stress sequence.
experimental data. It is clear that excellent agreements were achieved by both the predicted curves, thus validating the proposed constitutive law based on the grey Verhulst model. Moreover, the carrying capacity of strain derived from Equation 16, is estimated as $a/b = 0.68$, corresponding the infinite stress by Equation 17. This is also consistent with the fact that the minicomposites studied has the largest ultimate tensile strain of about 0.7% at the final failure.

CONCLUSIONS

A C/SiC minicomposite was fabricated by chemical vapor infiltration and its microstructure and tensile behavior were investigated, as well as the associated damage evolution with the aid of acoustic emission technique. The largely nonlinear tensile behavior and the associated acoustic emission evolution of the minicomposites can be generally divided into three regimes including matrix cracking emergence, multiplication followed by matrix cracking saturation. Both the tensile behavior and the cumulative AE energy curve can be well predicted by the grey Verhulst models, indicating the nature of intraspecific competition for these matrix cracks in the minicomposites studied. Such relationships can be further applied to other ceramic composite systems to establish the constitutive law and optimize material parameters by the corresponding damage evolution.

Acknowledgements

The authors would like to acknowledge the financial support of the Scientific Research Foundation of Civil Aviation University of China under Grant No. 3122016B003, and Civil Aviation Administration of China under Grant No. MHRD20160105.

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