Evaluation of interphase mechanical properties by embedded single fiber transverse tensile test
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Abstract

This paper describes the modeling methodology on composite interphase, which takes the interfacial region into consideration and deals with tensile and shear moduli for the region independently. From in-situ SEM observation results by the embedded single fiber transverse tensile test, it has been revealed that the fiber surface treatment conditions have an influence on the generation and propagation of the interfacial debonding and matrix cracking around a single fiber. Damage simulation in this test is carried out by a finite element analysis. Hoffman’s failure criterion is used for a judgment of damage in the interphase and the matrix. The reasonable elastic properties for the interphase are obtained by comparison of the computational with the experimental results. The effect of surface treatment conditions is investigated by changing the tensile strength of the interphase. The qualitative validity of this analytical methodology is verified by comparing the analytical results with the experimental ones.

1 Introduction

The fiber-matrix interface can be modified or controlled by surface treatment conditions of the fiber. [1] The denatured matrix region exists around a surface-treated fiber. Such an interfacial region is called “interphase”. The relationship between chemical surface
treatment conditions of the fiber and mechanical properties of the interphase should be clarified both qualitatively and quantitatively. Some experimental methods have been proposed for an evaluation of the interfacial mechanical properties, such as the fragmentation test, pullout test, push-out test, and micro droplet test, etc. However, the material constants of interphase have not been clarified yet. We think also that it is more necessary for practical laminated composite to evaluate the transverse microscopic damage behaviors, such as the generation or propagation of interfacial debonding or matrix cracking occurred around a single fiber. We have proposed the embedded single fiber transverse tensile test (ESFTT). \[2\][3] This test is carried out by in-situ SEM in order to investigate the microscopic behaviors of damage, where the tensile load is applied transversely to single fiber embedded into the matrix.

In this study, a simulation of damage behavior for our proposed ESFTT test carried out by the finite element analysis with a consideration of the interphase property. The reasonable material elastic constants for the interphase have been decided by parameter survey comparing the analytical results with the experimental ones. The effect of surface treatment conditions is also investigated by changing the tensile strength of the interphase.

## 2 Embedded single fiber transverse tensile test

### 2.1 Test specimen

The configuration of test specimen is shown in Fig.1. The single fiber used in the experiment is E-glass filament of 17μm in diameter. The filament is obtained from a fiber strand (1700 filaments) supplied by Nippon glass fiber Co. The filaments are treated by silane coupling agent (γanilino-propyl-tri-methoxy silane (APS), 0.1% in concentration) or not treated. The single filament has been embedded into Epoxy resin (Shell, Epikote828) cured by Tri-ethylen-tetramine (TETA). The observation section of test specimen has been polished by Al₂O₃ (0.05μm), and has been sputtered by Pt.
2.2 Experiment

The semiconductor-loading cell is installed in the tensile device of specimen into SEM (Hitachi, S-2460N). The SEM image in tensile loading is recorded in video. Test specimen is applied the tensile displacement at constant speed. The influence of fiber surface treatment conditions on the generation and propagation of damage has been investigated.

3 Finite element analysis

3.1 Modeling

The fiber, the matrix and the interphase referring to the specimen size (Fig. 1) are modeled with 4-node quadrilateral plane strain element as shown in Fig. 2. The diameter of the fiber \(d_f\) is set to be 17\(\mu\)m, and the interphase thickness \(t_i\) is assumed to be 0.2\(\mu\)m. The region for calculation is the part of 1/4 whole for symmetry of the model. A constant displacement is given to the edge of the matrix and all of the stresses occurred at the neighborhood of single fiber are calculated by a linear finite element analysis using the commercial FEM code, MARC® K6.
3.2 Elastic properties

The material properties used for the analysis are summarized in Table 1. The fiber and the matrix are E-glass fiber and epoxy resin, respectively. They are isotropic materials. Material properties of the interphase have not been clarified yet. Their reasonable constants are estimated by the parameter survey. The other researcher [4][5] have been handled the interphase as isotropic, and the interfacial property was expressed by changing only tensile modulus of the interphase. We have handled them to be orthotropic. Thus, tensile and shear moduli of the interphase are treated independently, so that the role of interphase can be expressed separately for stress bearing and stress transfer. [6][7] Tensile modulus ($E_i$) and shear modulus ($G_i$) of the interphase is changed widely on basis of that of the matrix ($E_m$, $G_m$). The effect of elastic constants of the interphase on stresses occurring in both the interphase and the matrix near the fiber is investigated.

Table 1 Elastic properties for finite element analysis.

<table>
<thead>
<tr>
<th></th>
<th>Tensile modulus, $E$</th>
<th>Poisson’s ratio, $\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber</td>
<td>$E_f = 71.43$ GPa</td>
<td>$\nu_f = 0.22$</td>
</tr>
<tr>
<td></td>
<td>$G_f = E_f / (2(1+\nu_f))$</td>
<td></td>
</tr>
<tr>
<td>Matrix</td>
<td>$E_m = 3.3$ GPa</td>
<td>$\nu_m = 0.33$</td>
</tr>
<tr>
<td></td>
<td>$G_m = E_m / (2(1+\nu_m))$</td>
<td></td>
</tr>
<tr>
<td>Interphase</td>
<td>$E_i^{xx} = E_i^{yy} = E_i^{zz} = 0.001 \sim 1E_m$, $\nu_i^{xy} = \nu_i^{yz} = \nu_i^{zx} = 0.33$, $G_i^{xy} = G_i^{yz} = G_i^{zx} = 0.001 \sim 10G_m$</td>
<td></td>
</tr>
</tbody>
</table>
3.3 Judgment for damage

Hoffman's failure criteria [8] has used for judging the damage of the interphase and the matrix. Though each strength of the matrix can be obtained from experiment, all strengths of the interphase have not been clarified satisfactory. Accordingly only tensile strength of the interphase was set to be lower than that of the matrix, because the interfacial debonding occurs initially at lower than the tensile stress for which the damage for matrix occurs in the transverse tensile test. However, compressive and shear strengths of the interphase are set to be the same values as the matrix.

Table 2 Strengths for each material.

<table>
<thead>
<tr>
<th></th>
<th>Tensile strength, T (MPa)</th>
<th>Compressive strength, C</th>
<th>Shear strength, S</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Matrix</strong></td>
<td>$T_m (X_m^t=Y_m^t=Z_m^t)=35$</td>
<td>$C_m (X_m^c=Y_m^c=Z_m^c)=45$</td>
<td>$S_m (S_{m}^{xy}=S_m^{yz}=S_m^{zx})=35$</td>
</tr>
<tr>
<td><strong>Interphase</strong></td>
<td>$T_i (X_i^t=Y_i^t=Z_i^t)=10,20,30,35$ ($T_i/T_m=0.29~1$)</td>
<td>$C_i (X_i^c=Y_i^c=Z_i^c)=45$</td>
<td>$S_i (S_i^{xy}=S_i^{yz}=S_i^{zx})=35$</td>
</tr>
</tbody>
</table>

3.4 Damage simulation

When the failure index value of Hoffman's criteria for element of the interphase ($F_i$) reaches more than 1, it will be judged that the interphase is damaged. The interphase element after damaged is expressed by reducing the tensile and shear moduli up to 0.001 times as origin. Practically, when the interfacial debonding occurs, a high stress concentration will be occurred near the tip of crack. A crack model has not been applied for the damage simulation, though much finer mesh division is necessary near the tip of crack. The flow chart of damage simulation is shown in Fig.3. Failure indexes in the interphase ($F_i$) and the matrix ($F_m$) are obtained respectively. When $F_i$ is larger than $F_m$, the damage transition angle ($\theta$) where the damage transfer from interphase to matrix is obtained.
Fig. 3 Flow chart of damage simulation.

4 Analytical results

4.1 Stress distribution

Effects of the interphase elastic properties on stress distribution have been investigated. When the interphase tensile modulus (E_i) is the same as the matrix tensile modulus (E_m), E_i/E_m=1, and the interphase shear modulus (G_i) changes from 0.001 to 1 times for the matrix shear modulus (G_m), the distributions of axis stress (σ_i^{xx}) and shear stress (τ_i^{xy}) occurred at the interphase around the single fiber are shown in Fig. 4. As shown in Fig.4 (a), σ_i^{xx} occurring near θ=0° tends to concentrate with a decrease of G_i/G_m. It is suggested that the interfacial debonding is likely to initiate locally at θ=0° when G_i is lower. As shown in Fig.4(b), peak position of τ_i^{xy} moves to smaller θ site as G_i/G_m decreases. For example, the place of peak shear stress occurs at θ=65° when G_i/G_m=1, at θ=45° when G_i/G_m=0.1, and at θ=30° when G_i/G_m=0.01~0.001. It is evident that the stress distribution around the single fiber is affected largely by the shear modulus of the interphase.
4.2 Initial damage

In order to judge the generation of damage, the failure index \( F \) is calculated by Hoffman's rule. Maximum values of interphase failure index \( F_i \) and matrix failure index \( F_m \) in the each element are shown in Fig.5, separately, where the tensile strength ratio of the interphase to the matrix \( T_i/T_m \) is assumed to be about 0.86, and both \( E_i/E_m \) and \( G_i/G_m \) were changed from 0.001 to 1. As shown in Fig.5 (a), \( F_i \) tends to increase with increasing of \( E_i/E_m \) in any \( G_i/G_m \). It is obvious that the initial damage occurs in the interphase near \( \theta=0^\circ \), because the interphase axis stress at \( \theta=0^\circ \) behaves critical to damage. On the other hand, the location of initial matrix damage changes remarkably by \( E_i/E_m \), in particular when \( E_i/E_m \) is smaller, \( F_m \) changes largely depending on \( G_i/G_m \) as shown in Fig.5 (b). By using the results of Fig.5 (a) and (b), the ratio of the interphase failure index to the matrix ones \( F_i/F_m \) is plotted as a function of \( E_i/E_m \) in Fig.6. When \( F_i/F_m \) is more or less than 1, the initial damage is possible to occur in the interphase or the matrix, respectively. In almost any \( G_i/G_m \), \( F_i/F_m \) is larger as \( E_i/E_m \) increases. In more than \( E_i/E_m=0.01 \) roughly, \( F_i/F_m \) is more than 1, and the interphase might damage initially. Therefore it is found that the interphase tensile modulus influences significantly on initial damage behavior. It is thought that the reasonable interphase tensile modulus can be estimated by referring the initial damage behavior obtained in the experiment.
4.3 Damage progression

The damage progressive behavior is investigated by iteration analysis of damage. Micro cracking changes from the interphase to the matrix at the certain angle, which is called the damage transition angle ($\theta_t$), for the region of $E_i/E_m=0.1$ up to 1. Fig. 7 shows the effect of shear modulus ratio ($G_i/G_m$) on damage transition angle ($\theta_t$) when the ratio of interphase tensile strength to the matrix ones ($T_i/T_m$) is 0.86. $\theta_t$ tends to decrease with increasing of $G_i/G_m$. By comparing the analytical value ($\theta_t$) with the experimental ones, the reasonable shear modulus of the interphase can be obtained. It will be useful to evaluate the interphase properties by the damage transition angle, which depends on the
elastic constants and strengths of the interphase.

Fig. 7 Effect of shear modulus ratio \( (G_i/G_m) \) on damage transition angle \( (\theta) \).

5 Comparison of experimental and analytical results

Reasonable elastic constants of the interphase are assumed from the analytical results as \( E_i/E_m = 0.1, G_i/G_m = 0.01 \). Fig. 8 shows the effect of tensile strength ratio \( (T_i/T_m) \) on failure index ratio \( (F_i/F_m) \), initial failure load \( (P_i) \) and damage transition angle \( (\theta) \). It is evident that \( P_i \) tends to increase with increasing of \( T_i/T_m \). \( F_i/F_m \) is plotted for the model of non-damage or initial damage. \( F_i/F_m \) tends to decrease with increasing of \( T_i/T_m \). It is found for \( T_i/T_m = 0.86 \) that the initial damage occurs in the interphase, and the following damage changes into the matrix damage. Accordingly, the resultant damage transition angle is obtained as \( \theta = 50^\circ \) from analytical results. However, for less than \( T_i/T_m = 0.57 \), it is obtained as \( \theta = 90^\circ \), thus the interphase damage does not change into the matrix damage. Conversely, for \( T_i/T_m = 1 \), \( F_i/F_m \) becomes less that 1, thus the matrix damage occurs predominantly. By comparing \( \theta \) obtained by analysis with the crack transition angle obtained by the experiment, the validity of this analytical methodology can be checked. For \( T_i/T_m = 0.8 \), \( \theta \) can be obtained easily as \( 65^\circ \) from Fig. 8. This value of transition damage angle is well agreed with damage transition point of SEM image shown in Fig. 9 (a) for APS treated fiber. On the other hand, the interfacial debonding has around the single
Accordingly, from analytical results, $T_i/T_m$ for non-treated fiber is thought to be less than $T_i/T_m=0.6$. From above mentioned comparison, the analytical results agree qualitatively well with the experimental results by using the elastic properties and strength properties of the interphase, which are suitable for two surface treatment conditions used in the experiment.

![Graph showing the effect of tensile strength ratio ($T_i/T_m$) on failure index ratio ($F_i/F_m$), initial failure load ($P_i$), and damage transition angle ($\theta_i$).](image)

**Fig. 8** Effect of tensile strength ratio ($T_i/T_m$) on failure index ratio ($F_i/F_m$), initial failure load ($P_i$), and damage transition angle ($\theta_i$).

![SEM images showing damage propagation.](image)

**Fig. 9** SEM images during damage propagation.

**5 Conclusion**

In order to estimate the material properties of composite interphase and to establish the modeling methodology on composite interphase, we have investigated the effect of the interphase properties on microscopic damage behaviors from both experimental and analytical approaches. The effects of surface treatment conditions on microscopic damage are observed by the
transverse tensile test of single fiber composites. The effects of the interphase properties on stress distribution are investigated by FEM, where the interfacial properties are expressed by changing tensile modulus and shear modulus of the interphase. By comparison of the damage behaviors observed in the experiment and the analytical results, the following points have obtained:
(1) The interfacial debonding is likely to initiate locally at outer edge of single fiber, when the interphase shear modulus is lower.
(2) The analytical results agree qualitatively with the experimental ones.
(3) The elastic constants and tensile strength of the interphase can be estimated roughly for the treated fiber or not treated fiber.

From above-mentioned things, the validity of the analytical methodology in this paper has been verified. By using the proposed modeling methodology on composite interphase, the damage simulation will be able to apply in practical composites, such as woven cloth laminates

References