Time-dependent and Temperature-dependent Fatigue Strength under Open Hole Compression for Quasi-isotropic CFRP Laminates with Toughened Interlayer

by

Masayuki NAKADA, Yasushi MIYANO
(Materials System Research Laboratory, Kanazawa Institute of Technology, Hakusan, Ishikawa, Japan)

Takayuki MATSUMOTO
(ENEOS Corporation, Tokyo, Japan)

Abstract
Recently, novel CF/benzoxazine prepregs for aircraft structures have been developed by ENEOS Corp. in Japan. Thermoplastic particles are dispersed on the surface of these prepregs. This study examines the applicability of our proposed formulations for the statistical time-dependent and temperature-dependent fatigue strength under open hole compression (OHC) for quasi-isotropic CFRP laminates using CF/benzoxazine prepreg. Results demonstrated that the statistical fatigue OHC strength of quasi-isotropic CFRP laminates with and without toughened interlayer can be formulated using the statistically determined static and fatigue OHC strengths. Furthermore, the characteristics for fatigue OHC strength of quasi-isotropic CFRP laminates with toughened interlayer were clarified compared with those of CFRP laminates without a toughened interlayer.

Keywords: Benzoxazine resin, Carbon fiber reinforced plastics, Fatigue strength, Interlaminar toughened layer, Open hole compression, Statistical life prediction, Viscoelasticity

1. Introduction
Carbon fiber reinforced plastics (CFRP) have been used for primary structural members of airplanes, ships, automobiles, and other vehicles for which high reliability must be maintained during long-term operation. Therefore, an accelerated testing methodology is strongly anticipated for long-term life prediction of CFRP structures exposed to actual environmental temperatures, water, and other influences.

The mechanical behavior of matrix resin of CFRP exhibits time and temperature dependence, so-called viscoelastic behavior, not only above the glass transition temperature \( T_g \), but also below \( T_g \). Consequently, it can be inferred that the mechanical behavior of CFRP depends strongly on the time and temperature [1–5]. Our earlier reports have proposed formulations of statistical static, creep, and fatigue strengths of CFRP based on matrix resin viscoelasticity [6,7].

Tensile strength along the longitudinal direction of unidirectional CFRP constitutes important and basic data for the reliable design of CFRP structures. The authors developed a test method to assess creep and fatigue strengths and the static strength at elevated temperatures using resin-impregnated carbon fiber strands (CFRP strands) [8]. Our recent study undertook the prediction of statistical creep and fatigue strengths under tension loading along the longitudinal direction of unidirectional CFRP. The examination was performed using CFRP strands of T300-3000 with thermostet epoxy resin as the matrix [9,10]. The statistical creep strength of CFRP strands at any constant load and temperature was predicted using the viscoelastic behavior of matrix resin and statistical results obtained for static tensile strengths of CFRP strands measured at various temperatures. Furthermore, the statistical fatigue strength of a CFRP strand at any cyclic load and temperature was predicted using the measured data described above and S–N curves at a cyclic load and temperature. The predicted results quantitatively agree well with experimentally obtained results measured using creep and fatigue tests for CFRP strands.

Recently, novel CF/benzoxazine prepregs for aircraft structures have been developed by ENEOS Corp. in Japan. The thermoplastic particles are dispersed on the surface of this prepreg. The CFRP laminates using this prepreg showed superior strength of open hole compression, compression after impact, and so on compared with those using conventional epoxy prepreg [11].

In our previous paper, the open hole compression (OHC) fatigue tests were conducted for quasi-isotropic CFRP laminates using CF/benzoxazine prepreg with and without toughened interlayer. The fracture mechanisms for these CFRP laminates under OHC fatigue loading were clarified by damage observations [12].

This study examines the applicability of our proposed formulations [10] for the statistical OHC fatigue strength of quasi-isotropic CFRP laminates using CF/benzoxazine prepreg with toughened interlayer. First, the formulations for the statistical time-dependent and temperature-dependent fatigue strength are introduced. Second, the test method for static and fatigue OHC strengths is explained. Third, the viscoelasticity of matrix resin and the statistical static OHC strengths of quasi-isotropic CFRP laminates were measured at various constant temperatures under a constant strain rate for ascertaining the parameters in the formulations. Additionally, the statistical fatigue OHC strength for quasi-isotropic CFRP laminates are measured at a constant temperature for the formulations. Finally, the master curve of fatigue OHC strengths of quasi-isotropic CFRP laminates is constructed by substituting the measured data into the formulation and is investigated through comparison with that of quasi-isotropic CFRP laminates without a toughened interlayer.

2. Formulations
We have proposed the formulation of statistical time-dependent and temperature-dependent static strength \( \sigma \) of CFRP based on the matrix resin viscoelasticity, as presented in the following equation in our earlier paper [10] as

\[
\log \sigma = \log \sigma_0 + \frac{1}{\alpha} \log \left( \frac{1}{1 - P_f} \right) + \eta \log \left( \frac{E(t, T)}{E(0, T_0)} \right)
\]

where \( P_f \) signifies the failure probability, \( t \) denotes the failure time, \( \tau_0 \) represents the reference time, \( T \) is the temperature, \( T_0 \) stands for the reference temperature, \( \sigma \) and \( \alpha \) respectively...
denote the scale parameter and the shape parameter on the Weibull distribution of static strength. In addition, \( m \) is the viscoelastic parameter. Also, \( E_i \) and \( E_i^* \) respectively represent the relaxation and viscoelastic moduli of matrix resin. The viscoelastic modulus \( E_i^* \) for the static load with a constant strain rate is calculated as shown below.

\[
E_i^*(t, T) = E_i(t/2, T) \tag{2}
\]

We proposed the formulation of statistical time-dependent and temperature-dependent fatigue strength of CFRP \( \sigma \) with fatigue degradation parameter \( F_i \) based on the matrix resin viscoelasticity, as shown below [10].

\[
\log \sigma_i = \log \sigma_0 + \frac{1}{\alpha} \log \left(-\ln(1 - P_i)\right) + n_i \log \left[ \frac{E_i(t, T)}{E_i(t_0, T_0)} \right] - F_i \log(2N_i) \tag{3}
\]

The viscoelastic modulus \( E_i^* \) is calculated using the following equation for the cyclic load for the case in which the stress ratio of the minimum stress / the maximum stress is zero, assuming that the matrix resin deformation in CFRP during cyclic loading is perfectly constrained by the carbon fiber rigidity.

\[
E_i^*(t, T) = \frac{1}{2} \left[ E_i \left(\frac{1}{4}, T\right) + E_i \left(\frac{1}{1 - 4T}, T\right) \right] , \quad N_i = f t \tag{4}
\]

Fatigue degradation parameter \( F_i \) is obtainable as a function of the number of cycles to failure \( N_i \) by the following polynomial function of \( \log(2N_i) \), which is found from experimentation.

\[
F_i \log(2N_i) = a \log(2N_i) + b[\log(2N_i)]^2 + c[\log(2N_i)]^3 \tag{5}
\]

The fatigue strength at \( N_i = 1/2 \) is equal to the static strength when failure time \( t \) is equal to \( 1/(2f) \).

3. Experiments

3.1 Specimen preparation

Quasi-isotropic CFRP laminates of two kinds are prepared as shown in Table I. One is un-toughened CFRP laminates T800S/BXZ (S specimen) using carbon fiber T800S and benzoxazine resin. The other is inter laminar toughened CFRP laminates T800S/BXZ_ILT (T specimen) using the same carbon fiber and matrix with toughened interlayers where thermoplastic particles are dispersed on the surface of the prepreg, as shown in Figure 1. The stacking sequence of these CFRP laminates is [45/0/-45/90]s. These CFRP laminates' thickness is 2.24–2.50 mm.

Table I. Quasi-isotropic CFRP laminates of two kinds.

<table>
<thead>
<tr>
<th></th>
<th>T800S/BXZ</th>
<th>T800S/BXZ_ILT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(S specimen)</td>
<td>(T specimen)</td>
</tr>
<tr>
<td>Toughened layer</td>
<td>No exist</td>
<td>Exist</td>
</tr>
<tr>
<td>Carbon Fiber</td>
<td>T800S</td>
<td></td>
</tr>
<tr>
<td>Matrix Resin</td>
<td>Benzoxazine</td>
<td></td>
</tr>
<tr>
<td>Molding method</td>
<td>Autoclave</td>
<td></td>
</tr>
<tr>
<td>Curing conditions</td>
<td>185 °C × 2 h</td>
<td></td>
</tr>
<tr>
<td>Stacking sequence</td>
<td>[45/0/-45/90]s</td>
<td></td>
</tr>
<tr>
<td>Thickness [mm]</td>
<td>2.24–2.50</td>
<td></td>
</tr>
</tbody>
</table>

The length, width, and thickness of the OHC specimen are, respectively, 305 mm, 38.1 mm and 2.24–2.50 mm (ASTM D 6484) shown in Figure 2. Test specimens are cut from quasi-isotropic CFRP laminates using diamond wheel blade. Furthermore, the center hole of 6.35 mm diameter is opened using a drilling machine (YDS-94CTN; DMG MORI Co., Ltd.). A diamond-coated carbide drill (D-GDN 6.2 mm; OSG Co., Ltd.) is used to open the hole. A carbide straight reamer (CRM 6.4 mm, OSG Co., Ltd.) is used to finish the hole.

![Fig.1 Cross-section of CFRP laminates with toughened interlayer, T800S/BXZ_ILT (T-specimen).](image1)

3.2 Test method

The static OHC tests were conducted for S specimens and T specimens. These tests were conducted according to ASTM D 6484. The electro-hydraulic servo testing machine (EHF-EB10; Shimadzu Corp.) was used for static OHC tests. The cross-head speed was \( f = 1.27 \) mm/min. The test temperatures were 25, 80, 120, and 150 °C. The static OHC strength was calculated using the following equation as

\[
\sigma = P_{\text{max}} / (bt) , \tag{6}
\]

where \( P_{\text{max}} \) represents the maximum load, and where \( b \) and \( t \) respectively denote the specimen width and thickness.

Fatigue OHC tests were conducted using the same test machine and same test fixture for static test. The loading frequency was \( f = 2 \) Hz. The test temperatures were room temperature and 80 °C. Stress ratio \( R \) was 0.05.

To evaluate the viscoelastic properties of matrix resin, dynamic mechanical analyses (DMA) were conducted for neat benzoxazine resin.

![Fig.2 Configuration of OHC specimen (unit: mm) and test fixture according to ASTM D 6484.](image2)
4. Results and Discussion

4.1 Relaxation modulus of matrix resin

The storage moduli $E'$ for neat benzoxazine resin were measured under various temperatures and frequencies using DMA. The relaxation moduli $E_r$ at various temperatures are shown on the left side of Figure 3 obtained from the storage modulus based on the linear viscoelasticity. Long-term $E_r$ is obtained by shifting those horizontally and vertically at various temperatures based on the time–temperature superposition principle, as shown on the right side of Figure 3. The reference temperature and time were selected as $T_0= 25 ^\circ C$ and $t_0= 1$ min. The time–temperature (horizontal) shift factor and temperature (vertical) shift factor are presented in Figure 4.

4.2 Static OHC strength of CFRP laminates

Figure 5 portrays the Weibull distributions of static OHC strengths at room temperature for two quasi-isotropic CFRP laminates: S specimen and T specimen. The shape parameter $\alpha$ and scale parameter $\beta$ are almost identical, respectively, for the S specimen and T specimen. The shape and the scale parameters in this figure are selected as parameters $\alpha$ and $\sigma_0$ (scale parameter at $T_0= 25 ^\circ C$ and $t_0= 1$ min) for the S specimen and T specimen in Equations (1) and (3), as listed respectively in Table II.

Figure 6 presents static OHC strength versus temperature for S specimen and T specimen. These strengths decrease with increasing temperature.

Figure 7 portrays the dimensionless static OHC strengths of quasi-isotropic CFRP laminates $\sigma_s/\sigma_0$ against the dimensionless viscoelastic moduli of matrix resin $E_s^*/E_0$. The relation of $\sigma_s/\sigma_0$ against $E_s^*/E_0$ can be represented as two straight lines with slope of $n_R$, which is the viscoelastic parameter in Equations (1) and (3). Results clarified that the existence of toughened interlayer increases the viscoelastic parameter $n_R$ in Equations (1) and (3).

Table II. Parameters for statistical static and fatigue OHC strengths of CFRP laminates.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Parameter $\alpha$</th>
<th>Parameter $\beta$</th>
</tr>
</thead>
<tbody>
<tr>
<td>S specimen</td>
<td>321</td>
<td>24</td>
</tr>
<tr>
<td>T specimen</td>
<td>320</td>
<td>23</td>
</tr>
<tr>
<td>$n_R$</td>
<td>0.93</td>
<td>1.27</td>
</tr>
<tr>
<td>$a$</td>
<td>0.021</td>
<td>0.017</td>
</tr>
<tr>
<td>$b$</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>$c$</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

Fig.5 Weibull distributions of static OHC strength of CFRP laminates at room temperature.

Fig.6 Static OHC strength of CFRP laminates versus temperature.

Fig.7 Determination of viscoelastic parameter.
4.3 Fatigue OHC strength of CFRP laminates

Figure 8 presents the fatigue OHC strength versus the number of cycles to failure for quasi-isotropic CFRP laminates of two kinds: S specimen and T specimen at room temperature 25 °C and at 80 °C. The fatigue strengths of both CFRP laminates decrease markedly with the increased number of cycles to failure and temperature.

The dimensionless fatigue strength is defined as shown by the following equation.

\[
\log S = \log \frac{\sigma}{\sigma_0} - n_R \log \frac{E_1(t,T)}{E_0(T_0,T_0)} = \frac{1}{\alpha} \left[ \log(-\ln(1-P)) - F_\text{f} \log(2N_f) \right]
\]

(7)

Figures 9 and 10 respectively present the relation between the dimensionless fatigue strength \( S \) and the number of cycles to failure \( N_f \) at 25 °C and 80 °C for S specimen and T specimen. This relation clarifies only one curve for each specimen. It is independent of temperature. It is definable that this curve is the S–N master curve for each CFRP laminate. These curves are approximated by the polynomial function of Equation (5). Parameters \( a \), \( b \), and \( c \) for each CFRP laminate are also shown in Table II. Therefore, the statistical fatigue strength at an arbitrary time, temperature, and frequency under a pulsating load can be predicted clearly from the long-term relaxation modulus of matrix resin shown in Figure 3 and the parameters for statistical static and fatigue strengths of CFRP laminates shown in Table II. Figure 10 also presents the degradation rate against the number of cycles to failure for T specimen is smaller than that for S specimen. Results clarified that the existence of the toughened interlayer decreases fatigue parameter \( a \) in Equation (3).

5. Conclusion

The statistical fatigue OHC strengths for quasi-isotropic CFRP laminates with and without toughened interlayer were evaluated based on our proposed formulations by measuring the statistical static and fatigue OHC strengths of quasi-isotropic CFRP laminates and the matrix resin viscoelasticity at various temperatures. Results demonstrated that the statistical fatigue OHC strength of CFRP laminates with and without interlaminar toughened layer can be formulated using the statistically determined static and fatigue OHC strengths. Furthermore, the characteristics for fatigue OHC strength of CFRP laminates with toughened interlayer were clarified by comparison with those of CFRP laminates without a toughened interlayer. Concretely, the existence of toughened interlayer increases the viscoelastic parameter \( n_R \) which is the viscoelastic sensitivity in the formulations. On the other hand, the existence of the toughened interlayer decreases the fatigue parameter \( a \) which is the damage sensitivity by cyclic load in the formulations.